

# **Final Program**

# The Seventh Pacific Rim International Conference on Advanced Materials and Processing

August 2-6, 2010 Cairns, Australia

Organized by Sponsored by

Materials Australia (MA) The Chinese Society for Metals (CSM) The Japan Institute of Metals (JIM) The Korean Institute of Metals and Materials (KIM) The Minerals, Metals and Materials Society, USA (TMS)

# WELCOME TO PRICM-7



Dear Participants,

On behalf of the International and Local Organising Committees of the 7<sup>th</sup> Pacific Rim International Conference on Advanced Materials and Processing (PRICM-7), I express my warmest welcome to all participants of the PRICM-7 conference. It is a great honour for us to host this international event on 2-6 August, 2010 in Cairns, Australia.

The Pacific Rim International Conference on Advanced Materials and Processing is held every three years, jointly sponsored by the Chinese Society for Metals (CSM), The Japan Institute of Metals (JIM), The Korean

Institute of Metals and Materials (KIM), Materials Australia (MA), and The Minerals, Metals and Materials Society (TMS) and organised in rotation. The purpose of PRICM is to provide an attractive forum for the exchange of scientific and technological information on materials and processing. The first Conference was held in Hanzhou, China, in 1992, followed by the second in Kyongju, Korea, in 1995, the third in Hawaii, USA, in 1998, the fifth in Beijing, China, in 2004, the sixth in Jeju Island, Korea, in 2007. This is the first time for the PRICM to be held in Australia.

The PRICM-7 conference represents one of the largest-scale interactions in the Asia-Pacific region that crosses traditional discipline boundaries. It brings together leading scientists, technologists and engineers from the Asia-Pacific region and around the world to discuss contemporary discoveries and innovations in the rapidly evolving field of materials and processing. This event is also intended to foster stronger and closer interactions between materials practitioners and their international counterparts. As on previous occasions, this Conference covers most aspects of advanced materials and the processes by which they are produced. Particular emphasis is placed on 13 symposia, covering over 1250 presentations and over 720 papers in the proceedings. A truly unique feature the PRICM-7 conference is the large number, over 150 plenary, keynote and invited speakers, of internationally distinguished scientists, engineers and technologists. It would be difficult to make this large-scale event successful without the active participation of these distinguished experts. I gratefully appreciate their tremendous support.

I would like to express my deep appreciation of the great efforts made by the In-Country Technical Representatives, Symposia Chairs, members of the Organising Committees and the International Advisory Committee, and the support of the International Organisation of Materials, Metals and Mineral Societies (IOMMMS). I wish to thank the Air Force Office of Scientific Research, Asian Office of Aerospace Research and Development, the U.S. Army International Technology Centre-Pacific, the Office of Naval Research Global for their contribution to the success of this conference. We also acknowledge gratefully the financial support from the Australian Synchrotron, CAST CRC, Defense Materials Technology Centre (DMTC), The University of Queensland, and Trans Tech Publications Inc.

Cairns is a tropical city in the northern part of Queensland. It is an excellent base for exploring the Great Barrier Reef, the largest coral reef system in the world and one of the seven wonders of the natural world. I wish the participants will take advantage of the conference break to explore this beautiful region of the world and to enjoy the sceneries.

I wish all of you an enjoyable time at this conference and in Cairns.

Jian-Feng Nie Chairman, International Organising Committee The 7<sup>th</sup> Pacific Rim International Conference on Advanced Materials and Processing (PRICM-7)

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# **Symposia Chairs**

# Symposium A: Advanced Steels and Processing

Professor Elena Pereloma, University of Wollongong, Australia Dr Han Dong, China Iron and Steel Research Institute Group, China Professor Zhigang Yang, Tsinghua University, China Dr Kaneaki Tsuzaki, National Institute of Materials Science, Japan Dr Sung Joon Kim, KIMS, Korea Professor Greg Olson, Northwestern University, USA

# Symposium B: Advanced High Temperature Structural Materials

Dr Matthew Dargusch, CAST CRC, Australia Dr Yuansheng Yang, Institute of Metal Research, China Dr Guoqing Zhang, Beijing Institute of Aeronautical Materials, China Professor Yoshinao Mishima, Tokyo Institute of Technology, Japan Professor Kyung Tae Park, Hanbat National University, Korea Professor Fernand Marquis, Naval Postgraduate School, USA

## Symposium C: Light Metals and Alloys

Professor Jian-Feng Nie, Monash University, Australia Dr Baiqing Xiong, General Research Institute for Nonferrous Metals, China Dr Yongqing Zhao, Northwest Institute for Nonferrous Metal Research, China Professor Yoshihito Kawamura, Kumamoto University, Japan Professor Kwang Seon Shin, Seoul National University, Korea Dr Kumar Jata, AFOSR/AOARD, USA

## Symposium D: Bulk Metallic Glasses and Nanomaterials

Professor Michael Ferry, The University of New South Wales, Australia Professor Zhaoping Lu, University of Science and Technology Beijing, China Professor Yue Zhang, University of Science and Technology Beijing, China Professor Akihiro Makino, Tohoku University, Japan Professor Do Hyang Kim, Yonsei University, Korea Professor Peter Liaw, University of Tennessee, USA

# Symposium E: Solidification, Deformation and Related Processing

Professor Yuri Estrin, Monash University & CSIRO Materials Science & Technology, Australia Professor Wanqi Jie, Northwestern Polytechnical University, China Professor Jishan Zhang, University of Science and Technology Beijing, China Professor Zenji Horita, Kyushu University, Japan Professor Hyoung Seop Kim, POSTECH, Korea Dr Chandra Pande, Naval Research Laboratory, USA

## Symposium F: Modelling and Simulation of Microstructures and Processes

Dr Shouyi Sun, CSIRO Minerals, Australia Professor Baicheng Liu, Tsinghua University, China Professor Tetsuo Mohri, Hokkaido University, Japan Professor Sun Keun Hwang, Inha University, Korea Professor Naresh Thadhani, Georgia Institute of Technology, USA

## Symposium G: Thin Films and Surface Engineering

Professor Fanxiu Lu, The University of Science and Technology Beijing, China Professor Chuang Dong, Dalian University of Technology, China Professor Shigeaki Zaima, Nagoya University, Japan Dr Kyung Ho Shin, KIST, Korea Professor David Young, University of New South Wales, Australia Professor Enrique Lavernia, University of California, Davies, USA

# Symposium H: Advanced Ceramics

Dr Nigel Stone, CSIRO Materials Science and Technology, Australia Professor Yong Huang, Tsinghua University, China Professor Katsutoshi Komeya, Yokohama National University, Japan Professor Hyung Sun Kim, Inha University, Korea Professor Eugene Olevsky, San Diego State University, USA

## Symposium I: Biomaterials, Smart Materials and Structures

Professor Chengbao Jiang, Beijing Univ. Aeronautics & Astronautics, China Professor Takao Hanawa, Tokyo Medical and Dental University, Japan Professor Byong Taek Lee, Soonchunhyang University, Korea Professor Joanna Mckittrick, University of California, San Diego, USA

# Symposium J: Materials Characterisation and Evaluation

Professor Jin Zou, The University of Queensland, Australia Professor Ze Zhang, Beijing University of Technology, China Professor Hiroyuki Toda, Toyohashi University of Technology, Japan Professor Dong Il Kwon, Seoul National University, Korea Professor Miguel Yacaman, University of Texas at San Antonio, USA

## Symposium K: Composites and Hybrid Materials

Professor Mark Hoffman, The University of New South Wales, Australia Dr Xiaosu Yi, Beijing Institute of Aeronautical Materials, China Professor Yutaka Kagawa, University of Tokyo, Japan Dr Soon Hyung Hong, KAIST, Korea Professor John Lewandowski, Case Western Reserve University, USA

# Symposium L: Energy Generation, Harvesting and Storage Materials

Dr Lyndon Edwards, ANSTO, Australia Dr Shaoxiong Zhou, China Iron and Steel Research Institute Group, China Professor Akihiko Kimura, Kyoto University, Japan Dr Young Whan Cho, KIST, Korea Dr Deryn Chu, Army Research Laboratory, USA

# Symposium M: IOMMMS Global Materials Forum

Professor David StJohn, Materials Australia, Australia Professor George Collins, Materials Australia, Australia Professor Xishan Xie, University of Science and Technology Beijing, China Dr Yoshimasa Kajiwara, JIM, Japan Dr Oh Joon Kwon, RIST, Korea Dr Robert Shull, NIST, USA

# **CONFERENCE PLENARY SPEAKERS**

# **Functional Materials Session**

Time:09:15 AM, Monday, 2 August, 2010Topic:Nanostructured Oxides as Catalysts for Clean Water and EnergySpeaker:Professor Max Lu, Deputy Vice Chancellor (Research), The University of Queensland, Australia



Nanoparticles and nanocrystals of semiconducting oxides constructed from nanoscale building locks often possess unique and much improved properties. Such materials are promising in enabling innovative technologies for conversion and storage of renewable energies for the future. This paper highlights the latest developments in oxides such as ZnO, TiO2, and ZrO2 as catalysts.

Max is Director for the ARC Centre of Excellence for Functional Nanomaterials, and Deputy Vice-Chancellor (Research) at the University of Queensland. His expertise is in nanoparticles and nanoporous materials for clean energy and environmental technologies. He co-authored 350 journal publications with over 6000 citations (and h-index of 41). He has received numerous awards including Australian Research Council's Federation Fellowships (twice) and Fellowship of the Australian Academy of Technological Sciences and Engineering.

## Time: 09:50 AM, Monday, 2 August, 2010

Topic:Research Activities and Industrial Development of the Advanced Electronic Materials in ChinaSpeaker:Dr. Hailing Tu, National Engineering Research Center for Semiconductor Materials, General<br/>Research Institute for Nonferrous Metals, China



China's electronic industry has the largest share in the world market and its revenue reached 6.3 trillion RMB last year. Out of economical and technical considerations, research and development of advanced electronic materials have been the major concern of China. This review describes the industrial development of large diameter Si wafers and Si-based materials, such as Si-on-insulator, strained Si and SiGe, and the compound semiconductor materials GaAs and GaN that are massively manufactured as the substrates for microwave electronic devices and light-emitting-diodes. In addition, defect engineering and impurity control to improve the performance of these materials will be presented. The research activities in high dielectric constant (high-k) materials, including Hf based rare earth complex oxides, and the phase change materials for the next generation of memory devices, will also be discussed.

Dr. Hailing Tu is an expert in semiconductor materials. He received his Ph.D. in solid state physics from Bath University, UK in 1983. Dr. Tu was the President of General Research Institute for Nonferrous Metals from 1995 to April 2009, and he is the Vice President of Chinese Materials Research Society, The Nonferrous Metals Society of China, and The Chinese Society of Rare Earths. Dr. Tu has been devoted to research in silicon, compound semiconductor, rare earth semiconductor crystal growth technology, impurities and defects behavior in the semiconductor materials, interfaces, surface physical-chemistry of semiconductor materials and their links with the device performances. He has been leading the Chinese special engineering programs of 200 mm and 300 mm silicon polished wafers. Dr. Tu published 8 books, 200 papers and 16 patents. He is the recipient of 10 National and Ministerial Science and Technology Prizes, and the Scientific and Technological Progress Prize of Ho Leung Ho Lee Foundation in 2004. He was elected as a member of Chinese Academy of Engineering in 2007.

# **Structural Materials Session**

Time: 10:55 AM, Tuesday, 2 August, 2010

Topic:Stronger, Tougher Steels: Potential of Nano-structured SteelsSpeaker:Dr. Kaneaki Tsuzaki, Managing Director of Structural Metals Center, National Institute for<br/>Materials Science, Japan



Stronger, tougher steels are needed to realize a low-carbon society. Toughness of steels decreases as the strength and the impurities increase and the temperature decreases. This talk introduces recent research activities on enhanced toughness through multi-scale structure control, and shows how we can overcome ductile-brittle transition and embrittlement by hydrogen and phosphorous.

Kaneaki Tsuzaki received his Ph.D. in 1983 from the Department of Materials Science and Technology, Kyoto University. He was Postdoctoral Fellow at Massachusetts Institute of Technology, USA from 1983 to 1985, Research Associate and Associate Professor at Kyoto University, 1985-1997. He joined National Institute for Materials Science (NIMS) in 1997 and has been in the current position since 2006. He is also working as a professor at University of Tsukuba since 2007.

## Time: 11:30 AM, Monday, 2 August, 2010

Topic:An Internal Variable Theory for Inelastic Deformation – A Unified ApproachSpeaker:Professor Young Won Chang, Pohang University of Science and Technology (POSTECH),<br/>Korea



An internal variable theory has been developed to account for the evolution of microstructures during inelastic deformation. The framework of the theory was built on the basis of well known dislocation dynamics to provide the concept of an internal strain tensor as the most fundamental deformation state variable. The theory has consequently been applied successfully to the various physical phenomena including the structural superplasticity, creep deformation, and deformation induced phase transformation. The various application aspects of the theory will be presented and discussed in this talk.

Young Won Chang is a SeAH Chair Professor in the Department of Materials Science and Engineering, POSTECH, Korea. Prof. Chang has mostly worked on developing an internal variable theory for inelastic deformation and its applications to various mechanical responses of crystalline materials. He received his B.Sci. in 1970 from Seoul National

University and his Ph.D. in 1979 from Brown University, USA. He worked at Cornell University as Research Associate and Michigan Technological University as Assistant Professor before returning to Korea in 1987 as one of the founding faculty members of the POSTECH. He was Head of Dept. of MS&E (1989-1994), Dean of Academic Affairs (1996-1997), and Vice-President (2003) during his tenure at POSTECH. He was the president of the Korean Society for Technology of Plasticity from 2003 to 2004. He was the President of the Korean Institute of Metals & Materials in 2007, during which period he successfully organized the PRICM-6 conference in Korea. He is currently elected fellow members of the National Academy of Engineering of Korea and the Korean Academy of Science and Technology.

Time:12:05 PM, Monday, 2 August, 2010Topic:High Temperature Magnesium: What are the Alloy Design Issues?Speaker:Professor Tresa M. Pollock, University of California, Santa Barbara, U.S.A.



With emphasis on vehicle weight reduction in the automotive industry, interest has grown in magnesium alloys with improved high temperature properties. Approaches to strengthening Mg alloys at ambient and elevated temperatures will be discussed. Issues with regard to system thermodynamics and partitioning of solute in cast materials will be addressed with emphasis on Mg-Al-Ca and rare-earth containing materials. The mechanistic contributions of elements in solution to creep resistance with consideration of partitioning are evaluated. The possible role of grain boundary sliding and intermetallic phases will be considered. Current challenges in alloy design and the development of integrated models for prediction of the properties of high temperature Mg alloys will be highlighted.

Tresa Pollock is a Professor of Materials at the University of California, Santa Barbara. She graduated with a B.Sci. from Purdue University in 1984, and a Ph.D. from MIT in 1989. Dr. General Electric Aircraft Engines from 1989 to 1991, where she conducted research and

Pollock was employed at General Electric Aircraft Engines from 1989 to 1991, where she conducted research and development on high temperature alloys for aircraft turbine engines. She was a professor in the Department of Materials Science and Engineering at Carnegie Mellon University from 1991 to 1999 and the University of Michigan from 2000 - 2010. Her current research focuses on the processing and properties of structural materials and coatings and on the use of ultrafast lasers for microfabrication and materials diagnostics. Professor Pollock was elected to the U.S. National Academy of Engineering in 2005, is a Fellow of TMS, ASM International, Associate Editor of Metallurgical and Materials Transactions and was the 2005-2006 President of The Minerals, Metals and Materials Society.

# **CONFERENCE LUNCHEON LECTURERS**

Time: 13:10 PM, Monday, 2 August, 2010

Topic: Materials in Military Systems

Speaker:

Dr. Ian Sare, Deputy Chief Defence Scientist (Platform and Human Systems), Defence Science and Technology Organisation, Australia



The performance of military platforms (aircraft, ships, submarines and land vehicles) is highly dependent upon their materials of construction, and the materials which make up the on-board systems that deliver the required capability. These materials, whether they are the aluminium alloys or composites of aircraft skins, the steel of submarine hulls, the coatings designed to resist corrosion or the armour designed to resist fragment penetration, must perform for long periods of time in often very arduous conditions. Another dimension to materials in a military context lies in the protective equipment worn by individual soldiers. There have been many developments in improving camouflage properties, flammability resistance, heat load management and ballistic penetration resistance which have been integral to reducing the vulnerability of soldiers in hostile environments. Materials designed with specific properties to go into military platforms cannot, however, be treated in isolation.

They invariably form elements of a complex system, and the integration of the parts of a system into the unitary whole must also receive attention if maximum performance is to be achieved. Examples will be described of developments in materials science and technology employed across a range of military systems. They will be developments that have delivered either better performance or reduced through-life costs, and they will show how military systems continue to push the boundaries of the properties available in materials.

Dr. Ian Sare trained as a metallurgist, gaining a BSc from UNSW and a PhD from the University of Cambridge. He joined the CSIRO Division of Tribophysics in Melbourne in 1975 where he initiated research into the wear properties of cast ferrous alloys used in mining and minerals processing applications. In 1984 he became Officer-in-Charge of the Adelaide Laboratory of CSIRO's Division of Manufacturing Technology and then, in 1995, Chief of Division. In 1997 he became the first Chief of CSIRO Manufacturing Science and Technology, formed from a merger of the Division of Manufacturing Technology with the Division of Materials Science and Technology. In 2002 he was appointed to a newly-created position within CSIRO as Sector Leader – Manufacturing. He joined DSTO in 2004 as Director of the Platforms Sciences Laboratory and Corporate Leader – Air. He is now Deputy Chief Defence Scientist, with responsibility for guiding the development of technologies underpinning military platforms, as well as those supporting defence in the chemical, biological, radiological and nuclear environment, and in the performance of Australian Defence Force members in military tasks. He is a Fellow of the Australian Academy of Technological Sciences and Engineering, the Institution of Engineers Australia and the Australian Institute of Company Directors. He was awarded a Centenary Medal for service to Australian Society in Metallurgical Science and Engineering.

#### Time: 13:10 PM, Tuesday, 3 August, 2010

Opportunities for Innovation in the Australian Building and Construction Industry Topic: Speaker: Mr. Paul O'Keefe, Chief Executive, Australian Coated and Industrial Markets, BlueScope Steel, Australia



The building and construction industry is a key market for BlueScope Steel branded products such as COLORBOND and ZINCALUME steel. This industry is also a significant employer and driver of economic growth in the Australian economy. The building industry is often seen as mature, with slow change in the adoption of new materials and practices. There are however notable examples where innovation is leading to significant improvements in efficiency of construction, and ultimately to reduced use of operational energy for building maintenance and heating/cooling purposes. The use of new coating materials that provide additional functionality is a key enabler for this innovation activity.

Paul O'Keefe was appointed Chief Executive Australian Coated and Industrial Markets, BlueScope Steel in November 2007. In this role, he is responsible for the sales, marketing, customer service and supply chain activities of the Company's domestic and export markets

across its Australian coated and industrial products. This business focuses on customers in the major building products, industrial, automotive and mining sectors. Paul joined BlueScope Steel in January 2007 as Vice President, Finance for the Australian Manufacturing Markets business. Before joining BlueScope Steel, he held the position of General Manager, Finance for Smorgon Steel Reinforcing and Steel Products, and immediately prior to that was a senior executive in the Operations division of Simplot Australia, the multinational food and agri-business. Paul has almost twenty years steel industry experience, having held a number of financial and accountancy roles in the Smorgon Steel Group from 1989 to 2000, and again from 2001 to 2006.



# Symposium A: Advanced Steels and Processing: Mechanical Properties of Steels

Monday PM August 2, 2010 Room: 5 Location: Cairns Convention Centre

Session Chairs: Setsuo Takaki, Kyushu University; Hu-Chul Lee, Seoul National University

#### 2:00 PM Keynote

The Effect of Inclusions on Mechanical Behaviour in Ultra-High Strength Alloy Steels: Xishan Xie<sup>1</sup>; Yanping Zeng<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing

Inclusions are un-avoidable even in super-clean engineering alloy steels because of the necessary melting process. These inclusions (such as TiN, AlN etc) are considered as harmful phases especially for ultra-high strength alloy steels. The unique experiments (in-situ tension and in-situ fatigue tests) have been conducted in a loading chamber of scanning electron microscope. TiN often characterizes with large blocky cubic morphology. Cracks easily initiate at the sharp corners of TiN cubic particles or sometimes directly initiate in TiN particles because of its brittleness. These cracks propagate to the matrix and to introduce early failure. AlN small particles (in several microns) often distribute as inclusion chains in steels. At tensile and fatigue tests cracks very often initiate at the inclusion chains among AlN small particles and line up to develop voids, which rapidly propagate to the matrix till early failure. These important results reveal the harmful effect of inclusions in micro-scale and can be connected with tensile and fatigue loading processes for understanding the early failure mechanisms.

#### 2:20 PM

The Effects of Holding Time at the Intercritical Temperature on the Mechanical Properties of a Dual Phase Steel: Xinsheng Liao<sup>1</sup>; Zhenghong Guo<sup>1</sup>; Xiaodong Wang<sup>1</sup>; Yonghua Rong<sup>1</sup>; <sup>1</sup>Shanghai Jiao Tong University

The mechanical property of a dual phase steel, which is subjected to different holding times at the intercritical temperature of 750°C followed by water quenching, is investigated. Tensile tests show that both yield strength and ultimate tensile strength increase with the increase of holding time, while elongation decreases. A very low ratio of yield strength to ultimate tensile strength is obtained at a relative longer holding time. Microstructural observation indicates that the volume fraction of martensite increases with the increase of holding time, but lower than 25% in the present experiments. The analysis of fracture surface shows that the low volume fraction of martensite corresponds to the ductile fracture while the high volume fraction of martensite corresponds to the brittle feature, i.e., fracture surface feature changes from the deep dimple-like to slight smooth cleavage type. The relationship between mechanical property and holding time was discussed.

#### 2:35 PM

#### Effect of Grain Size on Formability of SUS 304 Stainless Microtube Press Bending Process: *Cho-Pei Jiang*<sup>1</sup>; Yi-Syun Wu<sup>1</sup>; <sup>1</sup>National Formosa University

One of the most troublesome problems that are facing tubing production industry is wall thickness change in press bending process. With the ongoing miniaturization in products, thickness change is a dominant effect because material behaviour greatly varies in microtube press bending process. The present investigation addresses the influence of grain size on the press bending process of SUS 304 microtube numerically and experimentally. The problem was approached in two different ways: firstly, by reduction of the wall thickness of the microtube at a constant gran size, and secondly, by changing the grain size at a constant wall thickness. The experimental results show that the yield strength as well as tensile strength decrease with a decreasing T/D (wall thickness/average grain diameter) ratio. A T/D ratio of 8.9 is optimal for eliminating the springback angle and wall thickness change. Furthermore, the simulation prediction of springback associated with press bending angle using finite element method agrees well with the experimental results.

#### 2:50 PM

Effects of Carbide on Hydrogen Delayed-Fracture for 1GPa Tensile Strength Steel Wire: *Jin-Beom Lee*<sup>1</sup>; Nam Hyun Kang<sup>1</sup>; Kyung-Mox Cho<sup>1</sup>; Sun-Tae Ahn<sup>2</sup>; Ji-Tae Park<sup>2</sup>; Yeong-Do Park<sup>3</sup>; <sup>1</sup>Pusan National University; <sup>2</sup>Samhwa Steel; <sup>3</sup>Dong-Eui University

In modern times, automobile industry tries to reduce the weight of automobile by using high-strength steels. But, the high-strength steels are highly susceptible to delayed-fracture. And, the mechanism of delayed-fracture and the relationship with the microstructure and alloying elements are still ambiguous. This study analysed the effect of the carbides size and the spheroidization rate on hydrogeninduced delayed fracture properties for the 1GPa tensile strength wire. The four

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 of spheroidization larger than 80%. And, the 0.2%C-Cr steel had smallest size of the carbides. The hydrogen emitted was associated with the factors such as the amount, the spheroidization, and the size of the carbides.

 **gh Strength 3:05 PM Evaluation of Formability with Servo Die Cushion in Deep Drawing**: *Akihiro Watanabe*<sup>1</sup>; Yuji Kotani<sup>1</sup>; Hisaki Watari<sup>1</sup>; Takehiro Shimizu<sup>2</sup>; <sup>1</sup>Gunma University; <sup>2</sup>Komatsu Industries Corp.

 wanufacturing process using press machine has been substantially essential process in auto manufactures and many industries. It is needed to establish the

process in auto manufactures and many industries. It is needed to establish the press working process for manufacturing high accurate products of great extra value Introduction of servo press machine has been increasing with development of servo motors that are small and high torque. The main features for introduction of servo press machine manufacture high accurate products with high strength materials. By introducing a servo press, improvement of working technique for unworkable materials which have light weight and high mechanical strength, such as high tensile steel or magnesium has been focused. Traditional methods by using hydraulic pressure or pneumatic die cushion have been so far established under condition that BHF was constant value. In recent years, with the development of servo motor press machines, servo press machine on board servo die cushion have been introducing. It is expected that variable BHF improves the cup height and prevents materials from wrinkling and tearing. Our aim is to propose utilizing variable BHF to increase cup height and to control occurring wrinkling and tearing. By using simulation and experiments, the influence of variable BHF of servo die cushion on products is performed.

alloys were used for the specimen as a function of the alloying elements. The specimens were produced with a specific diameter (6.5mm) post to the deformation

(0, 10, 20 and 30%). And, the hydrogen was injected during the 2 days. The

emission of hydrogen was measured using the gas chromatography. The more

hydrogen was emitted for the high-carbon steels (0.45%C and 0.35%C steel) than the low-carbon steels (0.2%C-Cr and 0.2%C-Cr-Mo steel). Furthermore

the 0.45%C steel, the 0.35%C steel and the 0.2%C-Cr-Mo steel exhibited the

crack for the 30% deformed specimen. All of the specimens had the percentage

#### 3:20 PM

# Experimental and Numerical Investigations on Springback Variation in Stamping of Advanced High Strength Steels (AHSS): *Muammer Koc*<sup>1</sup>; <sup>1</sup>CPF/ VCU

Use of Advanced High Strength Steels for automotive body structures is a prominent method of reducing vehicle weight as an alternative to aluminum and magnesium alloys due to lower cost and higher formability at low temperature. However, AHSS parts demonstrate more springback. Moreover, variations in the incoming material, friction and other process conditions cause variations in the springback characteristics, which prevent the practical applicability of the springback prediction and compensation techniques. As a result, it leads to increased levels of variations and quality issues during assembly. The objective of this study is to investigate and gain an understanding of the variation of springback in the forming of AHSS both numerically and experimentally. First, two sets of experiments were conducted to analyze the influence of the material property, lubrication and blank holder pressure on the springback variation. The experiment results showed that the variation in the incoming blank material is the most important factor. Second, numerical simulations of a stamping process on an experimental tooling with 15 different flange configurations were performed. Effects of blank holder force (BHF), friction  $(\mu)$ , initial blank thickness (t) and hardening exponent (n) on the springback and its variation were investigated numerically.

#### 3:35 PM

#### Influence of Thermal History on Microstructure and Mechanical Properties of Steels for Hot Stamping: *Takehide Senuma*<sup>1</sup>; Yoshito Takemoto<sup>1</sup>; <sup>1</sup>Okayama University

Hot stamping is now drawing attention for producing high strength automotive components. In the conventional hot stamping, the furnace heating is employed and the heating rate is quite low. To improve the productivity of the hot stamping technology, the time reduction of the heating process is required. In this study, the influence of heating rate in a range up to 200°C/s, heating temperatures between 700°C and 950°C and cooling condition on microstructure and mechanical properties of 0.22% C steels with a Mn content varying between 1.2% and 3% has been investigated. Steels with Mn up to 2.5% reveal a martensite microstructure even though air cooling is employed. These steels are reaustenized at low temperature and the specimens heat treated at high heating rate, for short time holding at low heating temperature show significantly fine prior austenite microstructure and high strength and high toughness. In the paper, the  $\alpha > \gamma$  transformation behavior after intercritical annealing are discussed to explain the evolution of microstructures.

#### 3:50 PM Tea Break

**Symposium A:** 

**Advanced Steels and Processing:** 

**Mechanical Behaviour of Steels** 

Location: Cairns Convention Centre

Room: 5

Session Chair: Xishan Xie, University of Science and Technology Beijing

Review on the Hall-Petch Relation in Ferritic Steel: Setsuo Takaki<sup>1</sup>; <sup>1</sup>Kyushu

point followed by unstable Lüders deformation and such a vielding behavior is

taken over to fine grained steel with the grain size of  $1\mu m$  or less. Yield strength of

ferritic steel increases with grain refinement standing on the Hall-Petch relation;  $\sigma y [MPa]=100+600 \times d[\mu m]^{-1/2}$  in the relation between yield strength  $\sigma y$  and grain

size d. In low carbon steel, it might be concluded that the Hall-Petch coefficient

(Ky) is around 600MPa•µm<sup>1/2</sup>. However, the Ky value of interstitial free steels

is substantially small as 130-180MPa• $\mu m^{1/2}$  and it can be greatly increased by

a small amount of solute carbon less than 20ppm. It was also cleared that the

disappearance of yield point by purifying is due to the decrease in the Ky value.

On the other hand, the Ky value is changeable depending on heat treatment

conditions such as cooling condition from an elevated temperature and aging

treatment at 90°C. These results suggest the contribution of carbon segregation

at grain boundary in terms of the change in the Ky value. On the contrary,

substitutional elements do not give large influence to the Ky value in comparison

Yielding of polycrystalline low carbon steel is characterized by a clear yield



# 5:20 PM

Influence of Strain Path Changes on Microstructure Inhomogeneity and Mechanical Behaviour of Wire Drawing Products: Krzysztof Muszka<sup>1</sup>; Michal Wielgus<sup>1</sup>; Karolina Doniec<sup>1</sup>; Monika Stefanska Kadziela<sup>1</sup>; Janusz Majta<sup>1</sup>; <sup>1</sup>AGH University of Science and Technology

Cold drawn low carbon steel wires are widely used in several engineering applications where a proper combination of strength and ductility is of the paramount importance. In the present paper, the multi-pass angular accumulative drawing (AAD) is proposed as a new forming process where the high strain accumulation is used as a way to achieve much higher microstructure refinement level compared to the conventional wire drawing process. This process is characterised by a complex strain path history, being an effect of wire diameter reduction, bending, tension and torsion, what directly affects the microstructure changes in the final product. This process also evolves high inhomogeneity of microstructure, that if properly controlled, can lead to further properties improvement - what can be especially beneficial for alloys that are not characterized by complex compositions. In the present paper, special emphases is given on the inhomogeneity of both deformation and microstructure and resulted mechanical properties. After drawing and annealing (at 500°C) mechanical properties measurements and microstructure analysis on the cross sections of the wires were performed to assess the differences existing with respect to the conventional wire drawing process.

#### 5:35 PM

Influence of Initial Microstructure on Mechanical Properties of Cu Bearing Extra Low Carbon Steel Sheets: *Sim-Kun Min*<sup>1</sup>; Sung-Il Kim<sup>1</sup>; Jong-Sang Kim<sup>1</sup>; Moon-Hi Hong<sup>1</sup>; Sangho Han; <sup>1</sup>POSCO

To increase energy efficiency, thinner sheet with high strength require automotive steel sheet. So, extra low carbon(ELC) steel sheet with high strength and bake hardenability(BH) is mainly used automotive outer panels. Cu bearing ELC steel sheet which is high BH without Ti and Nb is matched this requirement. However, Cu bearing ELC steel has higher planar anisotropy( $\Delta r$ ) than IF steel because it has low r-value to diagonal direction(r45°). In this study, we investigated effect on microstructure of  $\Delta r$  by means of cooling condition change after hot rolling. The hot rolled sheets were performed cold rolling, annealing process and temper rolling in sequence. To evaluate deep drawability, R-bar and  $\Delta r$  were measured by uniaxial tensile tests with 3-direction (0°, 45°, 90° to rolling direction). Microstructures were observed using the optical microscopy and the EBSD technique. Chang of cooling delay time that it means water spray starts time shows change of the ferrite grain size(FGS) of hot and cold rolling sheets. Fine FGS condition shows lower  $\Delta r$ . Microstructure formed less than 0.5sec cooling delay time has more dense {111} texture than over 1sec.

#### 5:50 PM

**Deformation Behavior of Ferrite-Base Lightweight Fe-Mn-Al-C Steel:** *Chang-Hyo Seo*<sup>1</sup>; Ka Young Choi<sup>1</sup>; Hakcheol Lee<sup>2</sup>; Jai-Hyun Kwak<sup>3</sup>; Kwang-Geun Kwang-Geun Chin<sup>3</sup>; Nack Joon Kim<sup>1</sup>; <sup>1</sup>Graduate Institute of Ferrous Technology, Pohang University of Science and Technology; <sup>2</sup>Plate Research Group, Technical Research Laboratories, POSCO; <sup>3</sup>Automotive Steels Research Group, Technical Research Laboratories, POSCO

Al containing lightweight steels have excellent combinations of specific strength and ductility, which can meet the demands for energy conservation and environmental protection. There are two variants of lightweight steels; ferrite-base and austenite-base, depending on the type and amounts of alloying elements added. Although there has been rather extensive research on the austenite-base lightweight steels, ferrite-base lightweight steels, have not received much attention. In the present study, deformation behavior of ferrite-base Fe-Al-Mn-C steel containing various types of second phases has been investigated. Three different types of second phases, austenite,  $\kappa$ -carbide, and martensite, have been obtained by controlling the annealing conditions. It shows that the present steel shows quite different deformation behavior of the steel with different types of second phases. The deformation behavior of the steel with different types of second phases were investigated by electron back-scattered diffraction and transmission electron microscopy analyses on the specimens subjected to interrupted tensile tests.

#### 6:05 PM

Effect of Aging on the Microstructure and Tensile Properties of Lightweight Fe-Mn-Al-C Steel: *Kayoung Choi*<sup>1</sup>; Chang-Hyo Seo<sup>1</sup>; Hakcheol Lee<sup>2</sup>; Kyung-Tae Park<sup>3</sup>; Nack J. Kim<sup>1</sup>; <sup>1</sup>POSTECH; <sup>2</sup>POSCO; <sup>3</sup>Hanbat University

Recently, there has been a growing interest in the so-called lightweight steels, which have high specific strength and stiffness, excellent ductility and formability. One of lightweight steels developed is the austenite base steel containing nanosized  $\kappa$ -carbide particles as strengthening precipitates. Deformation behavior of the steels can be very complex, including TRIP, TWIP, shear band induced plasticity and microband induced plasticity. Although the steels can contain

## 4:50 PM Invited

with the effect by carbon.

Monday PM

August 2, 2010

4:30 PM Keynote

University

Effect of Austenite on Drawing Limit of Ferrite-Austenite Dual Phase Steel Wire: Seung-Hyun Lee<sup>1</sup>; *Hu-Chul Lee*<sup>2</sup>; <sup>1</sup>Hyundai Motor Company; <sup>2</sup>Seoul National University

The effect of austenite volume fraction on the strength and drawability of ferrite-austenite dual phase (DP) steel wire was investigated. Low carbon high manganese alloys were solution treated and tempered at between 525°C and 700°C. The volume fraction of austenite was varied from 20 to 40% depending on the alloy composition, tempering temperature, and tempering time. The drawability of the wires was decreased with the increase in austenite volume fraction as well as with the decrease of the mechanical stability of austenite. The interface of the strain induced martensite and ferrite was identified as the void nucleation site and the number density of voids was increased with the increase in the austenite volume fraction. Plastic incompatibility at the interface was assumed to be the main reason of void nucleation. Ferrite-austenite DP steels could be drawn to the true strain of 8.0 at maximum without intermediate heat treatment. The tensile strength of drawn wires was increased with the increase in austenite volume fraction, in other words, with the increase in the volume fraction of transformed martensite.

#### 5:05 PM

Room Temperature Tensile Defromation of High Mn-Al-C Steels: Si Woo Hwang<sup>1</sup>; Jung Hoon Ji<sup>2</sup>; *Kyung-Tae Park*<sup>2</sup>; <sup>1</sup>Steel Research Institute, Yonsei University; <sup>2</sup>Hanbat National University

Room temperature tensile behavior of high Mn-Al-C steels in the solid solution state was correlated to the microstructures developed during plastic deformation in order to clarify the dominant deformation mechanisms. The steels were as fully austenitic with the fairly high stacking fault energies. The tensile behavior of the steels was manifested by an excellent combination of strength and ductility over 80,000 MPa•% in association with continuous strain hardening to the high strain. In addition, the austenite phase was very stable during deformation. The high stacking fault energy and firm stability of austenite were attributed to the high Al content. In spite of the high stacking fault energy, deformed microstructures exhibited the planar glide characteristics, seemingly due to the glide plane softening effect. In the process of tensile straining, the formation of crystallographic microbands and their intersections dominantly occurred instead of mechanical twins or  $\varepsilon/\alpha'$  martensite. Microbands consisting of geometrically necessary dislocations led to the high total dislocation density state during deformation, resulting in continuous strain hardening. This microband induced plasticity is to be the origin of the enhanced mechanical properties of the steels.



a significant volume fraction of  $\kappa$ -carbide precipitates, their effects on the deformation behavior and tensile properties of austenite base lightweight steels are not clear. In the present study, deformation behavior and tensile properties of austenite base Fe-Mn-Al-C steels has been investigated, with particular emphasis on the effect of  $\kappa$ -carbide precipitates. Aging conditions were varied to have variations in the size and volume fraction of  $\kappa$ -carbide precipitates based on the hardness test results. Detailed deformation behavior of the steel with various morphologies of  $\kappa$ -carbide precipitates has been investigated by TEM and EBSD and correlated with tensile properties.

#### 6:20 PM

**On the Stretch-Flangeability of High Mn TWIP Steels**: *Lei Chen*<sup>1</sup>; Jinkyung Kim<sup>1</sup>; Sungkyu Kim<sup>2</sup>; Kwanggeun Chin<sup>2</sup>; Bruno De Cooman<sup>1</sup>; <sup>1</sup>Materials Design Lab; <sup>2</sup>Technical Research Laboratories

It is well known that second generation AHSS (Advanced High Strength Steel) TWIP (TWinning Induced Plasticity) steels achieve high strengths (800-1200MPa) and large uniform elongations (45%-60%) in uni-axial tension tests. In sheet deformation operations involving cut edge stretching, however, the deformation properties of TWIP steels are poor. It is usually assumed that it is a combination of (1) an increased tensile strength and (2) a multi-phase microstructure with phases of different hardness usually cause poor stretch-flangeability. High Mn TWIP steel, however, is a single phase austenitic steel and the reasons for its low HER are therefore not obvious. The present contribution reports on a detailed comparative study of the edge formability of FeMnCAl TWIP steel and three other single phase steels including the austenitic AISI 304 steel, the ferritic AISI 409 and Ti IF steels. Two types of Hole Expansion (HE) test were carried out: the KWI flat bottom HE test and the ISO/TC 16630 conical punch HE test. The punch force, and the sample strain and temperature were continuously monitored, by means of in situ Infrared (IR) thermo-graphy and optical strain analysis. The edge characteristics of specimen before and after edge cracking were studied by Scanning Electron Microscopy (SEM).

# Symposium A: Advanced Steels and Processing: Steel Production/Steelmaking

Monday PM	Room: A/B
August 2, 2010	Location: Cairns Convention Centre

Session Chair: Rian Dippenaar, University of Wollongong

#### 2:00 PM Keynote

**On the Performance Improvement of Steels through M<sup>3</sup> Structure Control**: *Han Dong*<sup>1</sup>; Maoqiu Wang<sup>1</sup>; Yuqing Weng<sup>1</sup>; <sup>1</sup>Central Iron & Steel Research Institute

A basic research project on high performance steels has been granted to 8 research institutes and universities by National Basic Research Program of China (973 Program) since 2010. The program aims at the investigation into the potential microstructure evolution phenomena in steels and to form the new microstructure control technologies for high performances, by which the safety of steels in service could be eventually improved remarkably. The issues such as steel metallurgy for ultra clean and ultra homogeneous microstructures, phase transformation mechanism of meta-stable austenite subject to temperature and load changes, carbon diffusion and partitioning during transformation, multiscale characterization of transformed microstructures, and microstructure stability subject to temperature and load changes will be focused on by the researchers. It is expected to develop the theory of microstructure control featured by multi-phase. meta-stable and multi-scale (M3). It is also to form the prototype technologies for manufacturing high performance steels such as the third generation high strength low alloy steels with improved toughness and/or ductility, the third generation advanced high strength steels for automobiles with improved ductility and low cost, and the third generation heat resistant martensitic steels with improved creep strength. It will provide the fundamentals for improving safety and efficiency of steels in service for infrastructures, automobiles and fossil power station.

#### 2:20 PM

**Study on the Production of Ti Microalloyed High Strength Hot Rolled Steel by CSP Process**: *Guanggiang Li*<sup>1</sup>; Aida Xiao<sup>1</sup>; Dezhi Wen<sup>2</sup>; Guohua Jiao<sup>2</sup>; Baiping Zheng<sup>2</sup>; Jie Fu<sup>1</sup>; <sup>1</sup>Wuhan University of Science and Technology; <sup>2</sup>Hunan Valin Lianyuan Iron and Steel Co., Ltd.

Ti microalloyed high strength hot rolled steel was developed in Valin Lianyuan Steel in the CSP line. The cleanliness of liquid steel was good enough for thin slab casting after LF refining. The basicity, viscosity, and melting temperature of mould powder were adjusted for stabilizing the heat flux of thin slab continuous casting mould. The banded structure, mixed grain size, and surface coarse grain were well controlled by the improving of rolling process. Homogeneous microstructure consisting of ferrite and pearlite was obtained in the hot rolled steel plates. The composition and quantity of inclusions in the slab were analyzed. The nano-sized precipitation of Ti(C,N) and Nb(C,N) is the main strengthening mechanism. The yield strength of developed hot rolled plate is higher than 660 MPa and the tension strength is 760 MPa. The ductile-brittle transition temperature is below -60 degree Celsius. No cracking happened during face bending and back bending test for weld samples. The fracture of tension test for weld samples appeared in base metal. The developed Ti alloyed steel with designed composition fulfils the requirements of 600 MPa grade steel for engineering machinery.

#### 2:35 PM

**Development of Steel Plate for One Kind of Heavy-Duty Truck Carriage and Its Structure Optimization**: *Di Hongshuang*<sup>1</sup>; Wang Xiaonan<sup>1</sup>; Liang Bingjie<sup>1</sup>; Du Linxiu<sup>1</sup>; <sup>1</sup>Northeastern University

In order to realize the weight reduction of the carriage of heavy-duty truck, the 700MPa hot-rolled high-strength C-Mn steel plate were successfully developed on 1750mm hot-strip mills to replace the Q345 steel plate which is originally used for carriage. The microstructure of the plate is mainly composed of finegrained ferrite and carbides distributed along the ferrite grain boundaries, and a little pearlite with 40~50nm lamellar spacing. The yield and tensile strengths of the plate are about 650 MPa and 740MPa, respectively, and the value of n, r and hole expansion rate( $\lambda$ ) are 0.12, 0.8 and 60%, respectively. Also the plate has great low temperature impact toughness and weldability. The strengthening mechanism of the plate are fine-grained strengthening and (Ti,Nb)C nanoprecipitates strengthening. If the carriage is constructed by using the new plate, the weight of it may reduce about 20% due to the thickness reduction. In order to further reduce the weight of the carriage, the optimization design of it was carried out by using the FEM method and the static structural strength and rigidity of carriage were checked. The weight of the carriage after structure optimization may reduce about 13%

#### 2:50 PM

# **Research and Production Practice of DR Material in Baosteel**: *Xiujun Li*<sup>1</sup>; Yunpeng Chen<sup>1</sup>; <sup>1</sup>Baosteel, China

In recent years, along with the development of the industries such as computer, mobile telephone, household electrical appliances and continuous expansion of pop canning industry market, the double cold Reduction Material (DR material), which is characterized by its very thin production specification, strong intensity, high board shape and surface quality, was developed rapidly. In China,only Baosteel has ability to product using the double cold reduction(DCR)technology. After five years reaserch and production Practice since 2005,DR material of tinplate serise from DR7 to DR9 have been recognized by users.In this paper, discussing Experimental Study, production commissioning and Product performance optimization methods at baosteel. In order to promote the development and progress of DR Matierial Producting Process, especially taken the Baosteel 1220 DCR unit as the example, combined with the field equipment and technology characteristic, the unit thinner production practice was taken out the detailed analysis and expositions by every aspects such as the model choice, roller seam lubrication system as well as the control mode, the actual production operation and technology parameter set, and so on, which offered beneficial reference for the technologist on the spot.

#### 3:05 PM

Smelting Technology and Final Product Quality of Steel Rails Used for Passenger Special Line with 350 km/h: Si Yongtao<sup>1</sup>; Li Chunlong<sup>1</sup>; *Zhi Jianguo<sup>1</sup>*; Zhao Dianqing<sup>1</sup>; Wang Bingyi<sup>1</sup>; Li Degang<sup>1</sup>; Jing Yinong<sup>1</sup>; Zhang Xiaoguang<sup>1</sup>; Liu Jianguo<sup>1</sup>; Liu Yanjun<sup>1</sup>; Zhang Zhi<sup>1</sup>; <sup>1</sup>Baotou Iron and Steel (Group) Corp.

The railway transportation with Chinese characteristics is developing towards the direction of higher speed, greater axle load and traffic volume so that demands for steel rails are higher. Baotou Steel (Group) Corp. is one of the important production bases for steel rails in China. In order to meet the development of railway transportation, steel rails used for passenger special line with 350km/h are developed. The technical specifications of it show that it is very strict on internal qualities such as composition, oxygen, hydrogen, inclusion, organization and structure, as well as surface qualities such as cross-section dimension, straightness and torsion, and performances of steel rails. The steel rails are produced with Bessemer zing, deoxidizing, refining, electromagnetic stirring of continuous casting, soft reduction for continuous casting, heating, universal rolling and straightening. In the paper, it is mainly introduced the smelting technology of manufacturing the steel rails, with which the goals of accurate composition of steel, control of narrow range, low content of inclusion and gas such as [O] and [H] in steel, as well as high quality of continuous casting blooms are achieved.



#### 3:20 PM

The Effects of the Ultrasonic Treatment on Degassing Rates in RH Water Model System: Yong-Tae Kim<sup>1</sup>; Kyoung-Woo Yi<sup>1</sup>; <sup>1</sup>Seoul National University

Ultra-low carbon steel is used for both automobile applications requiring high formability and electrical steel requiring high electric conductivity, and can be produced through RH decarburization processing. As demands for ultra-low carbon steel increase, the decarburization rates must grow higher to decrease decarburization processing times and to gain higher yields. In RH degasser, decarburization reactions are classified as three types according to the reaction sites: surface, plume, and inner site reactions. Among these, ultrasonic treatment can influence reaction rates at inner sites, because inner site reaction is sensitively dependent on pressure in melt. We investigated the effects of ultrasonic treatment on reaction rates at inner sites according to the pressure in vessel and degassing rates during RH processing. In order to study this phenomenon, we made a 1/8 scale RH water model and suspended the ultrasonic transducer by metal bar from top of the model system. Dissolved gas removal rates were higher when ultrasonic treatment was used during degassing processing than when the treatment was not. Also, based on observations of variations depending on different frequencies, larger frequency of transducer resulted in higher dissolved gas removal rates.

#### 3:35 PM

# Thermodynamics of Carbon and Silicon in Manganese Alloy Melts: M.K. Paek<sup>1</sup>; W.K. Lee<sup>1</sup>; J.H. Park<sup>1</sup>; J. Jinan<sup>1</sup>; Y.E. Lee<sup>2</sup>; *J.J. Pak*<sup>1</sup>; <sup>1</sup>Hanyang University; <sup>2</sup>Dongbu Metal

Thermodynamics of carbon and silicon in manganese alloy melts are important to predict the refining limit of carbon in manufacturing low carbon ferromanganese and silicomanganese alloys. The effect of silicon on the carbon solubility in Mn-Si melts was determined as a function of melt temperature. The critical silicon and carbon contents to form a SiC phase in Mn-Si-C melts were also determined. The equilibrium carbon and silicon contents in Mn-C and Mn-Si-C melts were determined in the presence of pure solid MnO and liquid MnO-SiO<sub>2</sub> slags at controlled oxygen potentials in the temperature range from 1673 to 1773 K. The activities of carbon and silicon in manganese alloy melts of various compositions were determined from the experimental results and available thermodynamic data for MnO-SiO<sub>2</sub> slag system. Using thermodynamic relations, the standard Gibbs free energy changes for the dissolution of carbon and silicon in manganese melt and the interaction parameters of carbon and silicon in manganese melt have been determined as a function of temperature.

## 3:50 PM

#### Mechanism of Leaching of Calcium Ion (Ca<sup>2+</sup>) from Commercial Calcium Silicate; Its Implementation to Blast Furnace Slag: Vandana Mehrotra<sup>1</sup>; <sup>1</sup>GIFT.POSTECH

Blast furnace slag is a valuable secondary resource containing about 46% percent of CaO similar to commercially bought Calcium Silicate (CCS). In this paper a process and mechanism of leaching Ca<sup>2+</sup> Ion first from CCS with Hydrochloric acid is discussed and Importance of this work is also highlighted when it can be converted into Calcium Hydroxide along with later implementation of same process to the blast furnace slag which will be an important step for effective use of slags to decrease CO<sub>2</sub> emission and energy consumption in the Steel Industry. Mechanism of reaction is also described. Under laboratory conditions the rate is controlled by a chemical reaction. 100% Leaching of Calcium Ion is projected till now from BF Slag.

4:05 PM Tea Break

## Symposium A: Advanced Steels and Processing: Welding of Steels

Monday PMRoom: A/BAugust 2, 2010Location: Cairns Convention Centre

Session Chair: Sung-Mo Jung, Pohang University of Science and Technology

#### 4:30 PM

Effect of Cr Contents on Microstructural and Hardness in the Resistance Spot Welded of Dual Phase Steel for Automotive Body Applications: *Jong Pan Kong*<sup>1</sup>; Tae Jun Park<sup>1</sup>; Tae Kyo Han<sup>2</sup>; Kwang Geun Chin<sup>2</sup>; Chung Yun Kang<sup>1</sup>; <sup>1</sup>Pusan National University; <sup>2</sup>POSCO Technical Research Laboratories

Increasing demands regarding improve the fuel efficiency, crash safety in the automotive industry. So, extend usage of Advanced high strength steels (AHSS). Many researchers studied the effect of processing parameters on the welding of AHSS in the spot welding process. However, the effect of alloy elements was less

revealed on the literature. Especially, Cr is expected to influence the weldability because it increased strength by increasing the hardenability of the dual phase(DP)steel. In this research, the effect of Cr contents on the microstructural and hardness in the resistance spot welded of dual phase steel were observed. Material used in the study were DP steel. Spot welding was performed using AC resistance spot welding machine. Electrode pressure and welding, holding and squeeze times were fixed to 4kN and 17, 17 and 40 cycles respectively. However, welding current was changed form 5.2kA to 9.6kA. As the results, the maximum hardness had at the HAZ of spot welded without Cr contents. Significant HAZ softening was occurred at the HAZ near BM. As the Cr contents increased, the HAZ softening is caused by increased martensite tempering through the HAZ approaching the weld.

#### 4:45 PM

#### Fundamental Studies on Hydrogen Dissolution in Welding Type Molten Fluxes: Jin Kyun Park<sup>1</sup>; Wan Wook Huh<sup>1</sup>; Il Sohn<sup>1</sup>; <sup>1</sup>Yonsei University

Mon. PN

Minute amounts of hydrogen less than 10 ppm can have a serious impact on the properties of steel welds. In particular, hydrogen induced cracking can be a serious problem when uncontrolled amounts of hydrogen is contained in and near the heat affected zone of the weld. There may be several sources of hydrogen during welding including the atmosphere, the filler metal, and the cored flux, but the final concentration of hydrogen can be directly affected by the control of molten flux or slag chemistry. In this study, the hydroxyl capacity of  $TiO_2$ -SiO<sub>2</sub>-MnO type slags and the  $TiO_2$ -SiO<sub>2</sub>-FeO type slags have been studied to identify and compare the hydrogen dissolution behavior in molten slags at high temperatures above 1500°C. The slag structure has also been analyzed using an FTIR to attempt to determine the effect of slag structure on the hydroxyl capacity. In addition, a comparison is made between the typical thermo-gravimetric method used in this study and an infra-red detection method typically used for metal samples.

#### 5:00 PM

Hardness Characteristics and Carbon Equivalent Equation of Resistance Spot Beam Welded Advanced High Strength DP Steels: Jong Pan Kong<sup>1</sup>; Tae Kyo Han<sup>2</sup>; *Chung Yun Kang*<sup>3</sup>, <sup>1</sup>School of Materials Engineering; <sup>2</sup>POSCO Technical Research Laboratories; <sup>3</sup>Pusan National University

Recently, in order to improve fuel efficiency and collision safety, advanced high strength steels were used as auto body and resistance spot welding was applied to assemble them. In this case, good formability and mechanical properties of the weld should be secured. For that purpose hardening characteristics of the weld should be analyzed and carbon equivalent equation which can predict the hardened state of the weld should be led out. In this study, resistance spop welding was applied out to 30 kinds of dual phase steels and hardness distribution of weld was analyzed as a function of modifying elements (Cr, Si, C, Al, P, S, B and Ti) and welding conditions. In order to predict hardening characteristics of weld, known carbon equivalent equation was estimated and new equation was suggested. From the corelation between carbon equivalent and maximum hardness of the weld, empirical equation to predict the maximum hardness of the spot welded DP steel was led out.

#### 5:15 PM

#### Influences of Welding Parameters and Heat Treatment on Microstructure and Tensile Properties of Welded Joints of Ni-Base Superalloy: *Dong Sun*<sup>1</sup>; Jing Wang<sup>1</sup>; Y. Na<sup>1</sup>; Y. Zhou<sup>1</sup>; <sup>1</sup>Harbin Institute of Technology

The influences of parameters of tungsten inert gas arc welding on the morphology, microstructure, tensile property and fracture of welded joints of Nibase superalloy have been studied. Results show that the increase of welding current or decrease of welding speed brings about the large amount of heat input in the welding pool and the enlargement of width and deepness of the welding pool. The increase of impulse frequency has the same effect on the microstructure compared with the increase of welding current. The effect of welding parameters on the tensile strength and fracture was analysed. It is found that the root of welding joint is unwelded when the welding current is lower, so that the strength and elongation of welded joint are inferior, and the more welding defects form in the welding zone and the more hard and brittle  $\gamma$ " phase precipitates in the overheated zone when the welding current is too high. Consequently, the strength and plasticity go up first and then go down, that is, have a peak value, with welding current increasing. In addition, the decrease of impulse frequency is beneficial to improve the strength of the welded joint.

#### 5:30 PM

Preliminary Investigation into Repair of High Strength Steel Using Laser Cladding: *Reza Mohammed*<sup>1</sup>; Qianchu Liu<sup>2</sup>; Madabhushi Janardhana<sup>3</sup>; Graham Clark<sup>1</sup>; <sup>1</sup>RMIT University; <sup>2</sup>DSTO; <sup>3</sup>Directorate General Technical Airworthiness

High-strength steels, such as the AISI 4340 adopted for this study, are used in several critical aerospace applications such as aircraft landing gear, primary



structure and piston rods. These steels are prone to in-service damage from impact or corrosion, and are fracture-sensitive, which makes attempts to repair the damage particularly difficult. One potential repair method uses laser assisted metal deposition (LAMD or 'laser cladding'-LC): a process where a high powered laser is used to fuse an alloy layer or coating, with enhanced mechanical properties, onto the surface of a substrate with minimal dilution of the substrate into the alloy cladding. The overall goal of this project is to investigate the possibility of restoring components which suffer damage exceeding permissible limits, to the required minimum level of performance using LC. Different strengths of 4340 were laser clad with an appropriate powder, with various combinations of powder feed rate, laser power, and laser traverse rate. The microstructure of the LC-repaired region at various points across, and around the repair, are presented and discussed, including features such as bonding and possible defects (e.g. porosity and cracking). The feasibility of using LC to repair 4340 or similar steels such as AeroMet100 is discussed.

# Mon. P

## 5:45 PM

#### **The Oxide Inclusion and Heat-Affected Zone Toughness of Low Carbon Steels**: *Xuemin Wang*<sup>1</sup>; Shurui Li<sup>2</sup>; Wei Shu<sup>1</sup>; Chengjia Shang; Xinlai He<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing; <sup>2</sup>Wuhan Iron and Steel Company

The oxide metallurgy technique has attract more and more interests around the world since this technique can improve the HAZ toughness of steels after high heat input welding. The low carbon steels are smelted with special oxide introduction technique and the HAZ properties has been studied with thermosimulation. The optical microscope, SEM and TEM were used to analyse the composition, size and distribution of the inclusions and the mechanical properties after thermo-simulation was also analysed. The results show that after the smelting the inclusion with 1 micron and they are oxide with Ti. These inclusions distribute homogeneously and there is no obvious congregation. After the thermo-simulation with the  $t_{8/5}$  increasing the toughness of HAZ decreased. The toughness of samples at -20° after a heat input of 100kJ/cm is over 80J. The microstructure is composed of intergranular ferrite with intragranular acicular ferrite. And the acicular ferrite nucleated at the inclusion and the inclusions accelerate the formation of acicular ferrite.

#### 6:00 PM

#### Welding Technology of Ultra-Low Carbon and Nitrogen Ferrite Stainless Steel: *Baosen Wang*<sup>1</sup>; Shuangchun Zhu<sup>1</sup>; Xiaoning Ye<sup>1</sup>; <sup>1</sup>Baosteel Iron and Steel Co., Ltd

Ultra low carbon and nitrogen 12% chromium ferrite stainless steel (FSS) is a new atmospheric corrosion resistance, wear resistance high strength structure steel developed in 2005 in China. It has been applied in body structure for lorry. Weldability of ultra low carbon and nitrogen low chromium FSS is analysed by using Thermol-cal software and welding metallurgy, embrittlement of welding joint is the failure reason during ultra low carbon and nitrogen 12% chromium FSS's application. Comparing welding joint performance of different welding process, gas metal arc welding (GMAW), laser arc welding (LAW) and friction stir welding (FSW), GMAW with high toughness welding material and proper welding heat input is economic and feasible welding process. Controlling growth of ferrite grain is the key to improve toughness of heat affected zone (HAZ) through analysing microstructure of HAZ. Titanium carbide or nitride and content of martensite along ferrite grain intergranular are very important to toughness of HAZ in low chromium FSS. The best size of Ti(C,N) grain is 2-5µm and content of martensite is 40%.

#### 6:15 PM

# **Improving Machinability of Plane Carbon Free Cutting Steels**: *Rajkumar Singh*<sup>1</sup>; <sup>1</sup>Bharat Forge Ltd

Most of the steels are rated on the their strength / toughness and corrosion resitance. In case of engineering steel for manufacturing components, machining accounts for as much as 50% of the cost. Steels commonly designated as free cutting or machining steels, where machining is easier, contain special additives for improving their machining characteristics. The additives, most commonly used for this purpose, are sulphur and lead. Addition of sulphur, which is the cheapest available free machining additive, impairs not only transverse toughness and ductibility, but also corrosion resitance. Modification of sulphur inclusions in steel to a globular morphology gives improvement in product quality. Tellurium is now recognized as a potent sulphur modifier as well as free machining additive when used in combination with lead and sulphur. The present paper is based on experimental heats made at Sunflag Iron and Steel Co Limited, Bhandara with tellurium addition in the plane carbon free cutting steel and tests conducted on them. Efforts of these various elements in the steel composition and its microstructure on machinability were studied and correlations were derived based on test data

## Symposium C: Light Metals and Alloys: Titanium Alloys I

Monday PM August 2, 2010 Room: C Location: Cairns Convention Centre

Session Chairs: Graham Schaffer, The University of Queensland; Xinhua Wu, The University of Birmingham

#### 2:00 PM Keynote

Building Capability through Benchmarking and Technology Transfer: Mark Hodge<sup>1</sup>; S. Palanisamy<sup>1</sup>; M.S. Dargusch<sup>1</sup>; <sup>1</sup>DMTC Ltd.

Production, sustainment and repair technologies for light alloy components in the defence applications account for a significant proportion of Defence Materials Technology Centre (DMTC) activities. Key challenges in this regard include the affordable manufacture and repair and sustainment of Titanium components for new and legacy platforms. The DMTC research program portfolio incorporates collaborative technology development activities between industry and the research sector within a framework of a partnership model that includes input from the Defence customer on likely procurement opportunities for industry, and longer term strategic requirements of Defence. The technological focus is particularly on the benchmarking of strategies, technologies and manufacturing techniques associated with the manufacture of aerospace components via high speed machining. Developing affordable, cost-effective and best of breed machining processes and practices among a broad range of Australian manufacturing organisations is a key goal of the DMTC.

#### 2:20 PM

Surface Modification and Repair for Life Enhancement and Structural Restoration: *Qianchu Liu*<sup>1</sup>; Pud Baburamani<sup>1</sup>; Wyman Zhuang<sup>1</sup>; Darren Gerrad<sup>1</sup>; Madabhushi Janardhana<sup>1</sup>; Khan Sharp<sup>1</sup>; <sup>1</sup>DSTO

Maintenance of ageing military aircraft structures encompasses both engineering and scientific management. In support of this, surface modification and repair methods are used on an opportunity basis to extend the life of aircraft in terms of fatigue and safety. Often, certain surface modification technologies have proved to be both cost effective and amenable for safe application. Some candidate and proven technologies include shot peening, cold spray, deep surface rolling, friction stir welding, laser shock peening, and laser cladding. Some technologies have been successfully applied in F/A-18 fighter. Cold spray technology has been recently approved for application in helicopter gear box. This paper briefly summarises the research work of these technologies at DSTO and discusses potential applications for aircraft components in near future. It also provides an appreciation of technologies – which may have potential advantages and disadvantages from a fatigue and safety aspects. A brief overview on certification methods and initiatives undertaken by RAAF is provided.

#### 2:35 PM

#### Assessment of Durability and Damage Tolerance of ß-Annealed Ti-6Al-4V: *Alexandra Shekhter*<sup>1</sup>; Simon Barter<sup>1</sup>; Marcus McDonald<sup>1</sup>; Russell Wanhill<sup>2</sup>; <sup>1</sup>DSTO; <sup>2</sup>National Aerospace Laboratory NLR

ß-annealed Ti-6Al-4V has a special chemical composition and manufacturing process, intended to optimise its fatigue and fracture properties and is found in primary fatigue-critical structures of advanced military aircraft. However, little has been published about the fatigue and crack growth behaviour under service spectrum loading, and the ability of constitutive and crack growth models to predict this behaviour. To assess durability and damage tolerance, two fatigue analysis methods were considered. The first was the strain-life based fatigue crack initiation, which is used to estimate durability lifetime. Coupons were loaded with flight manoeuvre spectra and the results were compared to predictions based on constant amplitude strain-life tests. Secondly, fatigue crack growth analysis method was used for life prediction. Constant amplitude fatigue crack growth and threshold tests were conducted. The data obtained was used to compare the crack growth models based not only the potential drop method but also quantitative fractographic analysis of short crack growth. Metallurgical assessment of the Ti-6Al-4V thick plate was also performed to compliment the mechanical testing. The results presented here, were used to assess and calibrate the predictive models for durability and damage tolerance assessment of B-annealed Ti-6Al-4V fatiguecritical structures under fighter aircraft representative loading.



#### 2:50 PM

Influence of Heat Treatment on the Pseudoelastic Behaviour of a ß Ti-25Nb-3Zr-3Mo-2Sn Alloy: *Damon Kent*<sup>1</sup>; Gui Wang<sup>1</sup>; Li-Hui Zheng<sup>2</sup>; Matthew Dargusch<sup>1</sup>; Zhentao Yu<sup>3</sup>; <sup>1</sup>The University of Queensland; <sup>2</sup>CAST CRC, School of Mechanical and Mining Engineering, The University of Queensland; <sup>3</sup>Biomaterial Research Centre, Northwest Institute for Nonferrous Metal Research

Due to their high strength to weight ratio, excellent toughness and corrosion resistance ß titanium alloys are suitable for a wide range of applications in aerospace, military, industrial and biomedical fields. They have a broad property spectrum which can be controlled through processing and heat treatment conditions. Metastable (or near) ß titanium alloys are stabilised by alloying with transition metal elements to the right of Ti in the periodic table. The stability of the ß phase is determined by the alloy composition and influences the deformation behaviour. The purpose of this paper is to characterise the elastic response and pseudoelastic behaviour of a Ti-Nb-Zr-Mo-Sn alloy. The effects of solution and age hardening heat treatments upon the microstructure and mechanical behaviour of the alloy are assessed. Significant pseudoelasticity, associated with the formation of the martensitic  $\alpha$ " phase, was observed. The solution treated condition displayed the greatest proportion of pseudoelastic behaviour. Detailed studies of the structure and deformation behaviour of the alloy obtained from tensile testing, optical microscopy, SEM, TEM and XRD studies are presented. The relationship between processing, phase composition and mechanical behaviour, and their implications for the performance of the alloy are discussed.

#### 3:05 PM

Plastic Deformation Characteristics and Evaluation of Press Formability for Ti-6Al-4V Sheet at Warm Temperature: *Jingee Park*<sup>1</sup>; Nhokwang Park<sup>2</sup>; Youngsuk Kim<sup>1</sup>; <sup>1</sup>Kyungpook National Univ.; <sup>2</sup>Korea Institute of Materials Science

Titanium alloy sheets have excellent specific strength and corrosion resistance as well as good performance at high temperature. Recently, titanium alloys are widely employed not only aerospace parts but also prosthetics and motorcycle. However, titanium and its alloys are difficult-to-form materials due to limited slip system and plastic anisotropy. Titanium alloy sheets were usually formed by slow forming or hot forming with heating die and specimen. In the sheet metal forming area, FE simulation technique to optimize forming process is widely used. To achieve high accuracy FE simulation results, identification of material properties and deformation characteristics such ad yield behaviors are very important. In this study, the yield locus of Ti-6Al-4V sheet was obtained at warm temperature. The experimental results are compared with the theoretical predictions. Also, the Forming Limit Curves (FLC) was achieved at warm temperature. The experimental results were compared with theoretical predictions based on M-K theory.

#### 3:20 PM

**Drawability of Ti-6Al-4V Sheet at Elevated Temperatures**: *Nhokwang Park*<sup>1</sup>; Jingee Park<sup>2</sup>; Sanghyun Seo<sup>1</sup>; Jeounghan Kim<sup>1</sup>; <sup>1</sup>Korea Institute of Materials Science; <sup>2</sup>Kyungpook National Univ.

Titanium and its alloys are difficult-to-form materials due to limited slip system and plastic anisotropy. Titanium is also prone to change in color due to oxidation at high temperatures during deformation. It is thus advisable to conduct sheet metal forming on titanium and its alloys at temperatures between RT and 500°C. In this study, the drawability of Ti-6Al-4V sheet is estimated in respect to the process parameters such as forming speed, lubricant, and blank holding force at elevated temperatures. It is shown that the limit drawing ratio (LDR) increases with increasing temperature, but varies little with forming speed. Using FE simulation, the earing formation is evaluated based on the Balat's yield criteria.

#### 3:35 PM

The Role of Interfacial Precipitates on Creep Behaviour of Power Metallurgy (PM) Ti-48Al-2Cr-2Nb+1W Alloy: *Dongyi Seo*<sup>1</sup>; Scott Bulmer<sup>1</sup>; Henry Saari<sup>1</sup>; Hanliang Zhu<sup>1</sup>; Peter Au<sup>1</sup>; <sup>1</sup>Institute for Aerospace Research, National Research Council Canada

Pre-alloyed powder having nominal composition of Ti-48Al-2Cr-2Nb-1W was produced by a gas atomization process. The gas atomized powders were consolidated by hot isostatic processing (HIP) at 1200°C, 200 MPa in an Ar atmosphere for 2 hours. After HIP, a step cooled heat treatment (SCHT) with carefully controlled cooling rate was applied to homogenize the HIP'ed microstructure and produces the fully lamellar microstructure while avoiding massively transformed  $\gamma$ . Following the SCHT, various aging schemes such as isothermal aging at 950°C were applied for times ranging from 8 up to 144 hours in conjunction with a stepped aging process. The stepped aging consisted of increasing the temperature in steps and holding for a specific length of time. After aging, interfacial precipitates formed at the lamellar interfaces and the morphology of the precipitates depended on the aging condition. Creep tests were carried out in air at 760°C and 276 MPa to investigate the effect of the interfacial precipitate. The creep properties of the aged conditions were compared with

those for the SCHT condition. The deformed microstructures were examined using transmission electron microscopy (TEM). The creep behaviours of these microstructures were elucidated in terms of lamellar morphology, especially the interfacial precipitates.

#### 3:50 PM

Improvement in Hot Workability of Titanium Matrix Composites by Thermohydrogen Treatment: *Weijie Lu*<sup>1</sup>; Junqiang Lu<sup>1</sup>; Di Zhang<sup>1</sup>; Hongliang Hou<sup>2</sup>; <sup>1</sup>State Key Laboratory of Metal Matrix Composites, Shanghai Jiao Tong University; <sup>2</sup>Beijing Aeronautical Manufacturing Technology Research Institute

Ti-6Al-4V matrix composite reinforced with TiB plus TiC was prepared and hydrogenated. The phase transformation and microstructures were studied. Isothermal compression tests and high temperature tensile tests were carried out to study the effect of the hydrogen on hot deformation and superplastic deformation. The flow behaviour and microstructure evaluation of hot deformation was investigated. And the processing map was constructed. The results show that hydrogen can decrease the flow stress, lower the deformation temperature or increase the strain rate at the same stress level, and reduce the instability zone. The result of superplastic deformation indicates hydrogen decreases the optimum superplastic temperatures and increases the optimum superplastic strain rate. The elongations of hydrogenated composites were significantly enhanced at appropriate deformation conditions. The microstructures observation was combined with the calculated activity energies to discuss the superplastic deformation mechanism. Hydrogen decreasing the transformation temperature. increasing ß phase, promoting dynamic recrystallization (DRX) and improving the accommodation deformation between matrix and reinforcements were considered as the main reasons for hydrogen-induced plasticity.

4:05 PM Tea Break

## Symposium C: Light Metals and Alloys: Titanium Alloys II

Monday PM	Room: C
August 2, 2010	Location: C

Location: Cairns Convention Centre

Session Chairs: Barry Muddle, Monash University; Dongyi Seo, Institute for Aerospace Research, National Research Council Canada

#### 4:30 PM Keynote

Microstructure Evolution and the Interrelationship between Microstructure and Properties of Titanium Alloys: *Hamish Fraser*<sup>1</sup>; Rajarshi Banerjee<sup>2</sup>; Peter Collins<sup>1</sup>; Soumya Nag<sup>2</sup>; Santhosh Koduri<sup>1</sup>; Brian Welk<sup>1</sup>; Dan Huber<sup>1</sup>; Robert Williams<sup>1</sup>; <sup>1</sup>The Ohio State University; <sup>2</sup>University of North Texas

This paper describes the results of research performed aimed at understanding the evolution of microstructure in Ti alloys, and then describes efforts to provide models for predicting the interrelationship between microstructure and mechanical properties. There has been a specific focus on determining the mechanism(s) of nucleation of the alpha phase. Here, the influence of beta phase separation and the formation of the omega phase as potential heterogeneous nucleation agents has been assessed. The roles of these competing structural instabilities will be highlighted. Regarding the prediction of mechanical properties, neural networks relating microstructure to various properties have been developed. These networks have been trained and tested using databases relating microstructure to properties, and the combinatorial means that have been developed to produce these databases in a rapid fashion will be outlined. New materials design tools that have been developed based on these developed neural networks will be demonstrated. It will be shown how these networks can be used to perform virtual experiments that can provide new insights into microstructural and compositional factors that control these properties. This research has been supported in part by the US Office of Naval Research (D3D Program) and the National Science Foundation.

## 4:50 PM Keynote

**Progress of Research and Development on Titanium Alloys in China**: *Yongqing Zhao*<sup>1</sup>; Henglei Qu<sup>1</sup>; <sup>1</sup>Northwest Institute for Nonferrous Metal Research

TC21 alloy with high strength, high toughness and damage tolerance is designed and developed by the authors in China. It has been used in aircrafts. Its 25kg, 350kg and 1ton ingots were used and bars of 20mm,90mm,130mm,180mm and 300mm in diameter were made. The effect of heat treatment on their microstructures and properties was researched. The results show that TC21 alloy has stable mechanical properties (UTS:1100MPa, YS:1000MPa, EI:8%, RA:12%, KIC:70MPa.m<sup>1/2</sup>). After solution treatment and ageing, its microstructure is bimodal one, which has good match among mechanical properties.



#### 5:10 PM

**Net Shape HIPping of Ti Alloys for Aeroengine Application**: Kun Zhang<sup>1</sup>; Junfa Mei<sup>1</sup>; Nick Wain<sup>1</sup>; *Xinhua Wu*<sup>1</sup>; <sup>1</sup>The University of Birmingham

Net shape Hot Isostatically Pressing(HIPping) has been used to manufacture Ti and Ni components for aeroengine applications. This study investigates the influence of HIPping conditions on microstructure and mechanical properties of HIPped Ti64 powder. The mechanical properties, including that from samples machined from the HIPped powder and from samples which contain the as-HIPped surface, have been reported. The fatigue limit of samples which contained the as-HIPped surface was improved by using a new HIPping procedure. The machined samples which had been HIPped at 930°C exhibited a better balance of properties than those HIPped at 880°C or 1020°C. The fine microstructure, formed from the martensitic structure of the atomised powder, coarsens with increase of temperature or time during HIPping. These changes have been correlated with the corresponding changes in properties and with the fracture surfaces. The significance of these observations, especially the fatigue properties of samples which contain the as-HIPped surface, is discussed in terms of the properties of net-shape HIPped components.

#### 5:25 PM

**Composition Design of BCC Ti-Based Solid Solution Alloys Using a Cluster Structure Model:** *Qing Wang*<sup>1</sup>; Rentao Ma<sup>1</sup>; Yingmin Wang<sup>1</sup>; Chuang Dong<sup>1</sup>; <sup>1</sup>Dalian University of Technology

A composition design of BCC Ti-based solid solution alloys is carried on by using a cluster-plus-glue atom model. This model regards the alloy structure as isolated clusters linked with glue atoms, which is expressed with a composition formula [cluster](glue atom)x, x denoting the number of glue atoms matching one cluster. In Ti-Mo-Nb ternary system, the negative enthalpy of mixing between Ti and Mo favors Ti-Mo clustering and a Mo-centered CN14 Mo-Ti14 cluster is constructed. While Nb serves as glue atoms due to the positive enthalpy of mixing between Ti and Nb. A series of alloy compositions [MoTi14]Nbx (x=1, 2, 3) are thus designed and they possess single BCC structure both under suction-casting and solid solution treatment. In addition, the binary alloy compositions [MoTi14]Ti1 and [TiTi14]Nb3 given by the cluster model are consistent with those alloys given by Mo equivalent method, which correspond to the lowest limit for forming BCC solid solutions. All of these cluster composition alloys have low tensile Young's modulus, high yield strengths and good plasticities in tension.

#### 5:40 PM

**The Effect of Temperature on Microstructure of a Metastable ß Ti Alloy**: *Gui Wang*<sup>1</sup>; Weiqi Wang<sup>2</sup>; Yulan Yang<sup>2</sup>; Damon Kent<sup>1</sup>; Matthew Dargusch<sup>1</sup>; <sup>1</sup>The University of Queensland; <sup>2</sup>BaoTi Group Ltd

A metastable ß titanium alloy, BTi-6554 (Ti-6Cr-5Mo-5V-4Al) has been developed for application in aircraft structures because of its high strength and high toughness. This paper reports on investigations into the effect of heat treatment on microstructure and hardness of the alloy. It has been shown that in the as forged condition, the alloy consists of the  $\beta$  phase. Heat treatment for 30 minutes at 350°C results in the presence of the  $\omega$  phase. Heat treatment between 350-650°C results in the gradual transformation of the  $\alpha$  phase back to  $\beta$  phase with larger grain sizes resulting from higher heat treatment temperatures.

#### 5:55 PM

Phase Constitution and Heat Treatment Behavior of Ti-7mass% Mn-Al Alloys: Masahiko Ikeda<sup>1</sup>; <sup>1</sup>Kansai University

Titanium exhibits many attractive properties. It is considered to be ubiquitous since it has the 9th-highest Clarke number of all the elements. However, the principal beta-stabilizing elements for titanium can be very expensive, making many titanium alloys expensive. Manganese is a beta stabilizer for titanium alloys and it is also considered to be ubiquitous since it has the 11th-highest Clarke number of all the elements. The behavior of Ti-Mn alloys during heat treatment has been investigated and it was found that in some allovs the isothermal omega phase is precipitated. Because this phase can lead to brittleness, it is very important to suppress its precipitation. Since it is well-known that aluminum suppresses isothermal omega precipitation, we investigated the effect of adding aluminum using Ti-7mass% Mn-0, 1.5, 3.0 and 4.5mass% Al alloys by performing electrical resistivity, Vickers hardness, and X-ray diffraction measurements. In solutiontreated and water-quenched 0 and 1.5 alloys, only beta phase was identified. while hcp martensite and bate phase were identified in 3.0 and 4.5Al alloys. The resistivities at room and liquid-nitrogen temperatures were found to increase monotonically with increasing Al content. Details regarding the heat treatment behaviors of these alloys will be presented in the conference.

## 6:10 PM

Aging Response of TB-13 Titanium Alloy: *Zhongbo Zhou*<sup>1</sup>; Jinshan Li<sup>1</sup>; Hongchao Kou<sup>1</sup>; Zhishou Zhu<sup>2</sup>; Bin Tang<sup>1</sup>; Hui Chang<sup>1</sup>; <sup>1</sup>Northwestern Polytechnical University; <sup>2</sup>Beijing Institute of Aeronautical Materials

At present, with increasing applications in aircrafts and nonaerospace military applications, metastable ß type titanium alloys with superior mechanical properties have been required largely. TB-13 alloy is a new metastable ß type titanium alloy which possesses an attractive combination of high strength and good ductility, making it a potential material candidate for aerospace applications. In order to develop a perspective of this new alloy, the influence of ageing treatments on the microstructure and properties has been studied. The results show that TB-13 alloy displays high age strengthening effect and fine precipitation. During the one-step aging process, the higher the aging temperature, the coarser the a plate thickness. As a result, TB-13 alloy shows the lower the strength and the higher the ductility. The best combination of strength and ductility has been acquired in the TB-13 alloy after solution treated at 860°C following aging at 540°C for 12h. The ultimate strength is near 1400MPa and the specific elongation is 7.0%.

#### 6:25 PM

**The Ageing Behavior of Titanium Alloy Ti-10V-2Fe-3Al**: *Huda Al-Salihi*<sup>1</sup>; Colleen Bettles<sup>1</sup>; Barry Muddle<sup>1</sup>; <sup>1</sup>Monash University

A good combination of high strength and hardenability make the alloy Ti 10V-2Fe-3Al a prime candidate for applications in the aerospace arena. However, these properties are very dependent on a post-forming heat treatment. The overall objective of this work is to determine the effect of prior deformation on the aging behaviour. In this particular study, the influence of the heat treatment, either solution and/or aging, on the microstructures, and consequently on the mechanical properties, without introduced strain is reported. Various solution heat treatments have been conducted, either in the  $\beta$  phase or in the ( $\alpha+\beta$ ) phase field, followed by rapid quenching or slow cooling, and aging treatments at different temperatures (250,350,400,500 C°) above and below the  $\omega$ -transus temperature. Vickers hardness indentations were used to follow the precipitation hardening behaviour. and mechanical properties were determined using a shear punch test. The aging response is dependent not only on the presence of the athermal  $\omega$  phase but also on the proximity of the aging temperature to the  $\omega$ -transus. Most treatments showed an unusual initial softening behaviour prior to age hardening, however this appears to be related again to the composition and fraction of the  $\beta$  phase retained after solution treatment.

# Symposium D: Bulk Metallic Glasses and Nanomaterials: Nanomaterials - Properties and Processing I

Monday PMRoom: 4August 2, 2010Location: Cairns Convention Centre

Session Chair: Robert O'Donnell, CSIRO

#### 2:00 PM Keynote

Performance and Damage of Nanodevices Based on One-Dimensional ZnO Nanomaterials: Yousong Gu<sup>1</sup>; Yue Zhang<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing

We have investigated the semiconducting nanodevices (Photodiodes, electromechanical devices, piezoelectric switches, and piezotronic strain sensors) based on one-dimensional ZnO nanomaterials and damage of ZnO nanomaterials/ nanodevices. The blue-light-emitting diode was constructed by using a ZnO-Nanowire array grown on p-GaN thin film. The electromechanical devices based on single Sb-doped ZnO nanobelts can be used to measure the nano-Newton forces from 20 nN to 60 nN. The piezoelectric switch based on single polar-surface dominated ZnO nanobelts shows a high on/off current ratio up to  $1.6 \times 104$  and a low threshold force of about 180 nN. The piezotronic strain sensor based on single bridged ZnO nanowires has a sufficiently high sensitivity of about 200% and a fast response time to the tensile strains. The damage threshold voltage is about 7 V for the 5 nm ZnO nanowires.

#### 2:20 PM Keynote

Applications of In-situ TEM Method on Nano Research: *Xuedong Bai*<sup>1</sup>; <sup>1</sup>Institute of Physics, Chinese Academy of Sciences

The in-situ TEM method has been developed to probe novel properties of nanomateials. It is powerful in a way that it can directly correlate the microstructure of nanomaterials with their physical properties. In this talk, I will report on the construction and applications of our home-made in-situ TEM nanomanipulation and nanomeasurement system, and the nanomaterials are focused on nanotubes. The electronic structures of carbon nanotube varying



from metallic to semiconducting are highly sensitive to their chiral indices (n, m). So far, it seems to have a long way to go to fully control nanotube chirality. We performed physical property measurements on individual nanotubes insitu TEM, inside which the chiral indices were simultaneously obtained using electron diffraction method. Thus, the properties of nanotubes can be directly correlated with their chiral structures. Here, field electron emission properties of the individual single-walled carbon nanotubes are studied. The metallic and semiconducting nanotubes showed quite different field emission behaviors, resulted from their different electronic states. Here I will report results on the electrical transport measurements of double-walled carbon nanotubes with known indices of each layer. Also, the manipulation and physical measurements on boron nitride (BN) nanotubes were performed in-situ TEM. I will also report on our recent studies on the electrically driven redox process in cerium oxides by using the in-situ TEM method.

#### 2:40 PM

#### **Carbon Nanotube Nanofluids Systems for Multifunctional Applications**: *Fernand Marquis*<sup>1</sup>; 'Naval Postgraduate School

The need for powerful and reliable thermal management systems has increased exponentially in the last two decades in order to sustain the performance of a very wide range of systems. Conventional heat transfer fluids such as water, ethylene glycol, water/ethylene glycol mixtures and lubricating oils are poor heat transfer fluids due to their low thermal conductivity. Carbon nanotube nanofluids have a much higher thermal conductivity then those based on metal and oxide particles. better stability, increased lubricity, good fluidity, non-clogging properties and low chemical reactivness. The values for the thermal conductivity of carbon nanotube nanofluids covers a wide range, depending on the base fluid, additional nanoadditives, nano chemistry, processing routes and temperatures. Typical top range increments can exceed 175% for a 1vol% load. Higher increments have been achieved at higher loads but with significant increase in the viscosity. Some of these systems exhibit considerable additional lubricity increments which makes them well suit for a wide range of applications. This paper presents the nanotechnology of carbon nanotube nanofluids and discusses current challenges and potential applications.

#### 2:55 PM

# **Ductility of Bulk Nanostructured Materials**: Yonghao Zhao<sup>1</sup>; *Enrique Lavernia*<sup>1</sup>; <sup>1</sup>University of California-Davis

The limited ductility of bulk nanostructured materials has evolved as one of major hurdles limiting widespread application of these materials, despite their relatively high strength. The low ductility of bulk nanostructured materials is determined by their limited plasticity and deformation mechanisms. In this talk, we will first review microstructure (including grain size, gain size distribution, dislocation density)-ductility relationship and external factors (including strain rate, temperature, specimen dimensions, processing artifacts) that influence the ductility of bulk nanostructured materials. Finally we will report on recent efforts in our laboratories to implement these strategies in nanostructured Cu, Ni, Ti to obtain both high strength and ductility.

#### 3:10 PM

Ultrafine-Grained/Nanostructured Metastable Bcc Beta-Titanium Alloys and Their Deformation Behaviors: *Wei Xu*<sup>1</sup>; Xiaolin Wu<sup>1</sup>; Mihai Stoica<sup>2</sup>; Mariana Calin<sup>2</sup>; Roberto Figueiredo<sup>3</sup>; Jürgen Eckert<sup>2</sup>; Terence Langdon<sup>4</sup>; Kenong Xia<sup>1</sup>; <sup>1</sup>University of Melbourne; <sup>2</sup>IFW Dresden; <sup>3</sup>University of Southampton; <sup>4</sup>University of Southern California

In metallic materials, the length scale, physical and mechanical properties of grains/constituent phases are key factors determining resultant overall mechanical properties. In this study, severe plastic deformation (SPD) techniques in the form of equal channel angular pressing (ECAP) and high pressure torsion (HPT) have been utilized to realize substantial grain refinement. A series of metastable bcc beta Ti-Nb-Zr-Sn alloys have been designed in terms of phase stability. The correlation between the extent of grain refinement and phase stability has been analyzed with respect to operative deformation mechanisms upon SPD processing. For instance, in a Ti67.4Nb24.6Zr5Sn3 alloy room-temperature HPT processing for five turns resulted in a uniform nanostructure mainly composed of beta-Ti grains of ~20-60 nm. By comparison, an ultrafine grain structure (~410 nm) was attained after ECAP at 903 K for four passes. The pronounced grain refinement is proposed to be facilitated by a stress-/strain-induced martensitic transformation. On the other hand, the presence of a large fraction of beta-Ti phase is possibly attributed to a reverse martensitic transformation stimulated by the applied shear stress. As a result, yield strength increased from 560 MPa for solution-treated condition to about 710 and 900 MPa for ECAP and HPT processed samples, respectively.

# 3:25 PM

**Processing and Excellent Oxidation Resistance of Nanocrystalline Fe-Cr Alloys:** Rajeev Gupta<sup>1</sup>; *Mahesh B.V.*<sup>1</sup>; R.K. Singh Raman<sup>1</sup>; Carl Koch<sup>2</sup>; <sup>1</sup>Mechanical and Aerospace Engineering, Monash University; <sup>2</sup>Materials Science and Engineering, North Caroline State University

This paper presents the description of the processing of nanocrystalline Fe-Cr alloy powders produced by mechanical alloying. Nanocrystalline alloy powders thus produced were successfully compacted and sintered to nearly 100% of the theoretical density while retaining nanocrystalline structure. When oxidation and aqueous corrosion resistance of nanocrystalline and microcrystalline alloys of same composition were compared, the nanocrystalline structure was found to provide superior oxidation/corrosion resistance.

#### 3:40 PM

#### Preparation of Photoluminescent Silicon Nanowires Based on Multicrystalline Silicon Wafers: Xianzhong Sun<sup>1</sup>; Jiayou Feng<sup>1</sup>; <sup>1</sup>Tsinghua University

Ion. PN

The single crystalline (c-Si) wafers are widely used as the precursors to prepare the silicon nanowires (SiNWs) by employing a silver-assisted chemical etching process. In this work, we obtained SiNWs arrays using polycrystalline silicon (Poly-Si) wafers. Firstly, the silver nanoparticles were deposited on the textured solar-grade poly-Si wafer by a galvanic displacement process; secondly, the SiNWs arrays were formed by a silver-assisted chemical etching process conducted in the HF-H<sub>2</sub>O<sub>2</sub> aqueous solution. The etching process indicated that the growth of SiNWs is independent on the orientation of the Si wafer. The diameter of SiNWs is much smaller than the ones obtained from c-Si wafers. The TEM images and selected area electron diffraction (SAED) patterns showed that the SiNWs were single crystalline structures. The photoluminescence (PL) spectra of SiNWs showed a broad visible emission centred around 690 nm, which is attributed to the emission properties of silicon nanocrystallites in SiNWs. We also modified the microstructure and the geometry of SiNWs, and obtained stronger PL intensity. This work may contribute to the application development of SiNWs on optoelectronic devices, solar energy conversion devices, chemical sensors, and lithium secondary batteries, etc.

#### 3:55 PM

#### Tensile Properties of Bulk Nanocrystalline Ni and Ni-W Fabricated by Sulfamate Bath: *Isao Matsui*<sup>1</sup>; Yorinobu Takigawa<sup>1</sup>; Tokuteru Uesugi<sup>1</sup>; Kenji Higashi<sup>1</sup>; <sup>1</sup>Osaka Prefecture University

Recently, nanocrystalline materials with high strength have been reported in large numbers. In particular, there has been considerable research on electrodeposited nanocrystalline Ni (nc-Ni) and nc-Ni alloys. However, reported data vary widely especially in ductility. Therefore, it is necessary to obtain the true characteristic value of nc-Ni and nc-Ni alloys. In the present study, nc-Ni and nc-Ni-W was electrodeposited on different conditions in order to obtained bulk nc-Ni and nc-Ni-W with high tensile strength and good ductility. At first, homogeneous bulk nc-Ni-W with thickness of 0.38-0.54 mm was fabricated by sulfate bath. However, fabricated specimens were curved shaped and brittle. We consider the brittleness of nc-Ni-W as the influence of internal stress. So, nc-Ni and nc-Ni-W was fabricated by sulfamate bath that has lower internal stress than that by sulfate bath. As a result, plastic deformation was observed in all specimens. Additionally, bulk nc-Ni obtained from sulfamate bath with grain size of about 60 nm exhibited tensile strength of about 900-1000 MPa and ductility of 4.8-8.8 %. Moreover, grain size of nc-Ni could be refined by addition bright agent. We are going to show tensile test result of bulk nc-Ni and nc-Ni-W with grain size of about 20 nm.

4:10 PM Tea Break

## Symposium D: Bulk Metallic Glasses and Nanomaterials: Nanomaterials - Properties and Processing II

Monday PM	Room: 4	
August 2, 2010	Location:	Cairns Convention Centre

Session Chair: Fernand Marquis, Naval Postgraduate School

#### 4:30 PM Keynote

**Optical Properties of Graphene and Graphene Nanoribbons**: *Chao Zhang*<sup>1</sup>; <sup>1</sup>University of Wollongong

Graphene is a sheet of carbon, just one atom thick. They possess the strongest mechanical strength and can achieve the highest carrier mobility, several times higher than that of the best semiconductor structures. It is a promising new material for the next generation of electronic devices and for many non-



electronics applications. Understanding the optical properties of graphene and graphene nanostructures is of importance in the development of graphene-based optoelectronic devices. We have carried out a theoretical and computation study of optical response of graphene over a wide frequency range, from terahertz to ultraviolet. It is found that the optical response is highly anisotropic at high frequencies. This property can be useful for developing polarization devices. At low frequencies, the graphene is nearly transparent. This weak response can be significantly enhanced by several mechanisms, (i) nonlinear absorption for fields greater than 1200 V/cm, (ii) magneto-electron absorption, and (iii) geometry effect such as using nanoribbons of specific geometry and chirality. We have identified a class of ribbon structures whose optical response can be enhanced by two orders of magnitude. The result removes a significant obstacle in potential application of graphene.

#### 4:50 PM

PM

Effect of Sol-Gel Synthesis on the Structural and Photoluminescence Properties of Magnetoplumbite-Type Strontium Ferrite: *Geok Bee Teh*<sup>1</sup>; Yat Choy Wong<sup>2</sup>; James Wang<sup>2</sup>; Seng Gee Tan<sup>3</sup>; Balakrishnan Samini<sup>3</sup>; <sup>1</sup>Tunku Abdul Rahman College; <sup>2</sup>Swinburne University of Technology; <sup>3</sup>Universiti Tunku Abdul Rahman

Magnetoplumbite-type (M-type) strontium ferrite particles with two stoichiometric ratios (SrFe<sub>x</sub>O<sub>19</sub>; x = 9.2 and 11.6) have been synthesized via the sol-gel technique employing ethylene glycol as the gel precursor. Structural properties were investigated via XRD, TGA and photoluminescence (PL) spectrophotometry. X-ray powder diffraction patterns showed that the samples were single-phase with the space group of P63/mmc and cell parameter values of a = 5.88 Å and c= 23.03 – 23.04 Å. Energy dispersive spectroscopy confirmed the composition being mainly of M-type SrFe<sub>12</sub>O<sub>19</sub>. The photoluminescence property of strontium ferrite was examined at excitation wavelength of 260 - 290 nm and significant PL emission peaks centered at 334 nm were detected. Both as-prepared strontium ferrites exhibited significant oxygen vacancies which were detectable via TGA where the sample with the Sr/Fe ratio of 1:11.6 exhibited the highest oxygen vacancies in its structure.

#### 5:05 PM

#### Interfacial Effects on the Electrical Characteristics of Carbon Nanotube Nanocomposites: *Michael Njuguna*<sup>1</sup>; Cheng Yan<sup>1</sup>; John Bell<sup>1</sup>; Prasad Yarlagadda<sup>1</sup>; <sup>1</sup>Queensland University of Technology

Carbon nanotubes (CNTs) are molecular scale tubes of graphitic carbon with unique mechanical, transport and electromechanical properties. Incorporating CNTs in polymers has dramatically improved mechanical and transport properties in these polymer nanocomposites for a wide range of applications. It is known that the surface energy plays a key role in determining the electrical properties of carbon based fillers such as polyacrylonitrile (PAN) carbon fibers and nickel coated PAN fibers. However, the effects of interfacial energy on the electrical properties of CNT polymer composites have not been well explored. In this work, the electrical characteristics of CNT nanocomposites with surface modified multiwall carbon nanotubes (MWCNTs) were investigated. The surface energy of acid treated, covalent and non-covalent functionalized, metal particle decorated and mechanically milled MWCNTs was evaluated through extensive measurement of surface tension. The interfacial effects were also modelled using thermodynamic percolation theory.

#### 5:20 PM

Modelling the Crystallization Reactions of Amorphous Precursors in Fe<sub>3</sub>B/ Nd<sub>2</sub>Fe<sub>14</sub>B Nanocomposite Magnets: *Vanalysa Ly*<sup>1</sup>; Stéphane Gorsse<sup>2</sup>; Kiyonori Suzuki<sup>1</sup>; Christopher Hutchinson<sup>1</sup>; <sup>1</sup>Monash University; <sup>2</sup>CNRS, Universite de Bordeaux, ENSCBP-IPB, ICMCB

A physically-based model is proposed for the precipitation of multiple phases (bcc-Fe, t-Fe<sub>3</sub>B, Nd<sub>2</sub>Fe<sub>14</sub>B, Nd<sub>2</sub>Fe<sub>23</sub>B<sub>3</sub>, NdFe<sub>4</sub>B<sub>4</sub> and Fe<sub>2</sub>B) from an amorphous Fe-Nd-B metallic matrix. The model considers the competition in nucleation, growth and dissolution between these six phases with an emphasis on the important role of the metastable phases. The approach draws on the field of computational thermodynamics and kinetics and makes use of a recently developed thermodynamic assessment of this system. The main advantage of the model is to deepen the understanding of the phase transformation sequence in this system, which influences strongly the performance of the resulting nanocomposite magnets. The novelty of the work is that it is the first precipitation model that simultaneously considers the competitive nucleation and growth of multiple phases in this alloy system. The approach naturally captures changes in bulk alloy composition and non-isothermal annealing conditions. Comparisons between model calculations and experiment suggest that the key thermodynamic aspects of phase transformation of this system have been captured and sheds light on the dependence of the magnetic properties of these materials on the crystallization conditions employed.

# 5:35 PM

**Soft Magnetic Nanocrystalline Fe-Ni-Nb-B Alloys with Improved Bend Ductility:** *Ivan Skorvanek*<sup>1</sup>; Jana Turcanova<sup>1</sup>; Jozef Marcin<sup>1</sup>; Jozef Kovac<sup>1</sup>; Peter Svec<sup>2</sup>; <sup>1</sup>Institute of Experimental Physics; <sup>2</sup>Institute of Physics

The influence of Fe replacement by Ni on the magnetic and mechanical properties was studied in (Fe<sub>1-x</sub>Ni<sub>x</sub>)<sub>81</sub>Nb<sub>7</sub>B<sub>12</sub> (x=0, 0.14, 0.25, 0.33, 0.5 and 0.67) nanocrystalline alloys. The analysis of phase evolution performed by XRD and TEM revealed that the nanocrystalline grains with an increase of Ni exhibit transition from BCC-phase to a mixed state with coexistence of BCC- and FCCphase and finally the FCC-FeNi phase is formed. The presence of ultrafine grains from 5 to 15 nm was evidenced by TEM. The addition of Ni has a beneficial effect on magnetic softness of the optimally heat treated nanocrystalline samples. The lowest coercivity value Hc ~ 1A/m was found for the  $(Fe_{0.5}Ni_{0.5})_{81} Nb_7B_{12}$ annealed for 1 hour at 773 K. An increase of Ni concentration towards the x=0.66 leads to a drastic increase of coercivity due to an appearance of big grains of the (Fe,Ni)<sub>23</sub>B<sub>6</sub> phase. Striking differences in the magnetic hardening regime at elevated temperatures have been observed for the nanocrystalline samples with different Ni concentration. The ductile/brittle behavior of thermally treated samples was determined by simple bending and miniaturized Erichsen tests. The Ni-rich compositions exhibit improved bend ductility as compared to the Fe-rich ones.

#### 5:50 PM

Nanocrystalline Phosphors for Lighting and Detection Applications: *Christopher Summers*<sup>1</sup>; Hisham Menkara<sup>1</sup>; Won Park<sup>2</sup>; <sup>1</sup>PhosphorTech Corp; <sup>2</sup>University of Colorado

We report the development of new nanoparticle phosphors and quantum dot structures designed for applications to enhance the color rendering and efficiency for high brightness white LEDs, and the upconversion of infrared radiation to improve lamp efficiency and for bio-sensing applications. The intrinsic problem of self-absorption, high toxicity, and high sensitivity to thermal quenching of conventional quantum dot systems has prevented their adoption to LED devices. Doped Cd-free quantum dots may circumvent these issues due to their distinct Stoke's shift and improved stability at high temperature. We report modification of Mn-doped ZnSe/ZnS core-shell quantum dots for application to (blue diode + yellow emitter) white LED system. Band gap tuning for 460 nm excitation, inorganic shell growth and in-situ monitoring for enhanced efficiency, and analysis of thermal stability will be reported. The infrared radiation emitted by incandescent/fluorescent lamps and sunlight makes materials for transforming the color or frequency of light extremely important in enhancing the efficiency of lamp and solar cell technologies. Extensive modelling and recent experimental studies show that novel doping schemes have high potential for improving upconversion efficiency. Further applications of these structures to new medical detection and disease diagnostic strategies are discussed.

#### 6:05 PM

# Development of High Strength and Ductile Alloys Based on Modulation of Ultrafine Eutectic Structure: *Ki Buem Kim*<sup>1</sup>; <sup>1</sup>Sejong University

Bulk metallic glasses often undergo inhomogeneous plastic flows at room temperature. In order to overcome such critical disadvantage of the nc and BMGs, it is necessary to design novel microstructure containing both structural i.e. phase selection and spatial i.e. length-scale heterogeneities as a source to cause homogeneous plastic flows. Along the line to design the heterogeneous microstructure, we have fabricated a series of ultrafine eutectic alloys in Ti-Fe-Sn and Mg-Cu-Zn alloys. The detailed microstructural investigations on a series of Ti-Fe-Sn alloy point out that an interesting microstuctural evolution occurs to form bimodal ultrafine eutectic structure consisting of a mixture of two eutectic colonies with different morphology and lamellar spacing by controlling Sn content. Similarly, the formation of the bimodal eutectic structure in a series of Mg-Cu-Zn alloys also reveals a strong enhancement of the strength with decent plasticity ep of  $\sim$  5%. Based on these results, it is feasible to interpret roles of the structural and spatial heterogeneities to control the strength and plasticity of the Ti- and Mg-based bimodal ultrafine eutectic alloys.

#### 6:20 PM

# Surface Layer Deposition of Nanostructured Materials onto Support Particles: Jonian Nikolov<sup>1</sup>; Seng Lim<sup>1</sup>; <sup>1</sup>CSIRO - Minerals

Nanostructured materials exhibit many unique functional properties and have been extensively investigated for the last three decades. One of the main reasons impeding their current uptake into wider usage is their high production cost. This limits their usage to only a few high-end applications. A new cost-effective method for producing a wide range of nanomaterials using modified flame spray pyrolysis in a fluidized bed has been developed which would enable larger scale production of nanomaterials with selectively tailored properties. The technique may offer promising opportunities for future development of a new generation of fully-recyclable catalytic materials. The preliminary results of surface coating



of a nanostructured layer onto support-mobile particles will be discussed in the paper. The coated layers were analyzed using SEM-EDS and XRD. The results showed that the layers were homogeneous in morphology and had an average size of around 10 nm. There were also no traces of secondary phases or other impurities.

#### 6:35 PM Invited

Low Temperature Synthesis of Silicon Nanocrystals Fabricated by PECVD and Their Optical Properties: Donghyun Jahng<sup>1</sup>; Gil Ho Gu<sup>1</sup>; *Chan Gyung Park*<sup>2</sup>; <sup>1</sup>Deptartment of Materials Science and Engineering, Pohang University of Science and Technology (POSTECH); <sup>2</sup>Deptartment of Materials Science and Engineering, Pohang University of Science and Technology (POSTECH), National Center for Nanomaterials Technology (NCNT)

Silicon nanostructures have attracted considerable interest due to their potential applications to optoelectronic devices such as Si-based light emitting diodes (LEDs) and solar cells. In order to fabricate the silicon nanocrystals, non-thermal plasma synthesis has been applied. Mono-dispersed silicon nanocrystals with a size ranging from 2 to 30nm have been successfully synthesized without agglomeration from the gas phase of silane. Slightly oxidized silicon nanocrystals with a size of  $2 \sim 6$ nm revealed the photoluminescence of  $648 \sim 729$ nm wavelengths. In order to enhance the quantum yield for photo-emission from silicon nanocrystals, nitrogen plasma treatment have been applied. The effects of nitrogen plasma on compositional, electrical and optical properties of silicon nanocrystals have been investigated by high resolution electron microscopy (HREM), secondary ion mass spectrometry (SIMS), X-ray photoelectron spectroscopy (XPS) and photoluminescence (PL). The results revealed that the optimum nitrogen plasma treatment enhanced the quantum yield of photoemission from silicon nanocrystals.

# Symposium E: Solidification, Deformation and Related Processing: Ultrafine-Grained Materials I

Monday PM	Room: 2
August 2, 2010	Location: Cairns Convention Centre

Session Chair: Minoru Umemoto, Toyohashi University of Technology

#### 2:00 PM

Evaluation of Plastic Work Density, Strain Energy and Slip Multiplication Intensity at Some Typical Grainboundary Triple Junctions: *Tetsuya Ohashi*<sup>1</sup>; Michihiro Sato<sup>1</sup>; Yuhki Shimazu<sup>1</sup>; <sup>1</sup>Kitami Institute of Technology

When polycrystalline metals deform plastically, various kind of inhomogeneous deformation evolve inside the microstructure. If we observe and consider the deformation at the length scale of crystal grain size, such inhomogeneities are related to the accumulation of geometrically necessary (GN) dislocations, but quantitative understandings of the accumulation of GNDs are not yet sufficient. Inside the polycrystal microstructure, grainboundary triple junctions and guadruple points are important sites to be studied in detail. In this communication, we analyze plastic slip deformation in two models of tricrystals by a crystal plasticity FE code and discuss the plastic slip and dislocation accumulation. Two tricrystal models are designed so as that they exhibit wedge- or twist-disclination type deformation field after slip deformation on the primary slip systems in three crystal grains. Distributions of the total slip, plastic work density and GN dislocations on slip systems, as well as some quantities for the intensity of slip multiplication are evaluated. Results suggest that the total slip or the plastic work density could not be used as a measure for the generation of embryos of recrystallisation but GNDs or coexistence of GNDs on different slip systems are better ones for that

#### 2:15 PM Keynote

Texture Change of Severe Plastic Deformed Al Alloy Sheets: Insoo Kim<sup>1</sup>; Su

Kwon Nam<sup>1</sup>; Saidmurod Akramov<sup>1</sup>; <sup>1</sup>Kum Oh National Institute of Technology Aluminum alloys have good potential to replace the low carbon steel in automotive industry. However most of aluminum alloys have lower formability (plastic strain ratio, r-value, Lankford parameter) than low carbon steel in fully annealed condition. The texture of fully annealed aluminum alloy sheet mainly consists of cube component {001}<100> which has low formability, whereas γ-fiber component <111>//ND has high formability of fully annealed Al alloy sheets. In this paper, development of textures after the severe deformation and subsequent heat treatment in Al sheet was observed. In the present study, Al alloy sheets have been severe plastic deformed and heat treated. The studies on the texture changes of the samples after the severe deformation and subsequent heat treatment have been carried out. The Al specimens after the severe deformation showed nano-size grains. The specimens after the severe deformation and subsequent heat treatment of Al alloy sheets were observed the changes of texture components. The changes of the mechanical property after the severe deformation and subsequent heat-treated Al sheets have been analyzed with the change of texture through the severe deformation and subsequent heat treatment in Al alloy sheets.

#### 2:35 PM

Susceptibility to Hydrogen Embrittlement of IF Steel with Ultrafine-Grained Microstructure Produced by Accumulative Roll-Bonding Process: *Takumi Haruna*<sup>1</sup>; Yuichi Nakagawa<sup>1</sup>; Daisuke Terada<sup>2</sup>; Naoki Takata<sup>3</sup>; Nobuhiro Tsuji<sup>2</sup>; <sup>1</sup>Kansai University; <sup>2</sup>Kyoto University; <sup>3</sup>Tokyo Institute of Technology

We have investigated susceptibility of hydrogen embrittlement to interstitialfree (IF) steel with ultrafine-grained microstructure produced by accumulative roll-bonding (ARB) process. The as-received IF steel was ARBed at 773 K, and repeated to five cycles. The as-received and the ARBed steels were cut into tensile specimens, and then hydrogen was charged to the specimens in an sulfuric acid solution of pH 2.5 at a cathodic current density of 50 Am<sup>-2</sup> for several charging times. Immediately after the hydrogen-charging process, tensile tests were conducted at ambient temperature and an initial strain rate of 3.3 x 10<sup>-4</sup> s<sup>-1</sup> Besides, state and amount of hydrogen charged to the specimen were determined with a thermal desorption gas analyzer (TDA) at a heating rate of 5.6 x 10<sup>-2</sup> Ks<sup>-1</sup>. As a result, almost no hydrogen was entered in the as-received steel charged for a long time of c.a. 300 ks, and a fracture strain of the steel was independent of the charging time. On the other hand, amount of hydrogen in the 5-cycle ARBed steel increased with an increase in the charging time, and the fracture strain decreased with an increase in the charging time. This indicates that the ARBed steel exhibits susceptibility of hydrogen embrittlement.

#### 2:50 PM

Effects of Phase Transformation Temperature on Formation of Wear-Induced Layer in Fe-Ni Alloys: *Hisashi Sato*<sup>1</sup>; Yuichi Kubota<sup>1</sup>; Eri Fujiwara<sup>1</sup>; Yoshimi Watanabe<sup>1</sup>; <sup>1</sup>Nagoya Institute of Technology

When metallic material is worn, wear-induced layer with fine grain is formed on its worn surface. This comes from the severe plastic deformation (SPD) due to wear. But microstructure of wear-induced layer is affected by not only SPD but also frictional heat. In case of Fe alloy, the microstructure of the wear-induced laver would change depending on its phase transformation temperature. However, these influences for the wear-induced layer are still unclear. In this study, effects of the transformation temperature on formation of the wear-induced layer in Fe allovs are investigated using Fe-33mass%Ni and Fe-30mass%Ni alloys. Martensitic transformation temperature and reverse transformation temperature (As) of Fe-33mass%Ni alloy are lower than those of Fe-30mass%Ni alloy. Microstructure of the wear-induced layer in Fe-33mass%Ni alloy was single austenite phase with fine grain. On the other hand, wear-induced layer in Fe-30mass%Ni alloy consists of martensite and austenite with fine grain. This difference is due to the difference of As between these Fe-Ni alloys. Also, it is found that the microstructure of the wear-induced layer has no dependence of the distribution of martensite in initial microstructure. Therefore, it is concluded that the formation of the wear-induced layer in Fe alloys is affected by As.

#### 3:05 PM

Dynamic Recrystallization Behaviour in Cu-Sn-P Alloy for High Strength Copper Tube: *Masato Watanabe*<sup>1</sup>; Takashi Shirai<sup>1</sup>; Akihiko Ishibashi<sup>1</sup>; Hiromi Miura<sup>2</sup>; <sup>1</sup>Kobelco and Materials Copper Tube, Ltd.; <sup>2</sup>UEC Tokyo (The University of Electro-Communications)

As a high strength copper tube, Cu-Sn-P alloy is developed and has been already employed as heat exchangers and the tubes. For further strengthening and better deformability, microstructural control of the alloy is necessary. Especially, grain refinement by a mechanism of dynamic recrystallization (DRX) is of great importance for the microstructural control. Cu-0.025P and Cu-0.6Sn-0.025P (in mass%) alloy with grain sizes of 5.3 mm and 200 µm were deformed in compression at various temperatures from 1023K to 1253K and at strain rates between 2×10<sup>-4</sup>s<sup>-1</sup> and 2×10<sup>-1</sup>s<sup>-1</sup>. DRX fully occurred at all the testing conditions independent of grain size. The onset of DRX was more advanced with increasing temperature and with decreasing strain rate and grain size. The occurrence of full DRX was, however, much delayed even at high temperature and low strain rate with increasing grain size and Sn content. This tendency was reasonably understood from i) decrement of DRX nucleation sites in the coarser grained structure and ii) slower rate of grain boundary migration by increment of Sn content. The Sn addition caused finer grain size and higher flow stress. These experimental results indicate the important role of Sn for strengthening and microstructural control.



#### 3:20 PM

**Mechanisms of Dynamic Recrystallization in Cu-Sn-P Alloy**: *Hiromi Miura*<sup>1</sup>; Masato Watanabe<sup>2</sup>; Takashi Shirai<sup>2</sup>; Akihiko Ishibashi<sup>2</sup>; <sup>1</sup>UEC Tokyo; <sup>2</sup>Kobelco and Materials Copper Tube, Ltd.

Mechanisms of dynamic recrystallization (DRX) behavior in a Cu-0.65Sn-0.025P (mass%) alloy (Cu-Sn-P) was systematically investigated by compression tests at 1073 K and at true strain rates from  $2 \times 10^{-3} \text{ s}^{-1}$  to  $2 \times 10^{-1} \text{ s}^{-1}$  in vacuum. As a model sample of as-casted alloy having coarse columnar grains, orientation-controlled bicrystals were employed. Appearance of the peaks stress, where DRX onsets, was much delayed in Cu-Sn-P alloy compared with that in Cu. Nucleation of new grains tended to take place preferentially at grain boundary with decreasing strain rate. This tendency was more evident at higher angle grain boundaries. The misorientation angle of the initial grain boundary gradually changed with increasing strain by viscous sliding to reduce plastic incompatibility of the component single crystals. Almost all the new grains were annealing twins formed behind the migrating grain boundary. Because grain boundary migration was observed to take place more easily with increasing misorientation angle and with decreasing strain rate, the preferential nucleation at grain boundary became more significant.

#### 3:35 PM

Dynamic Recrystallization Behavior of Biomedical CCM Alloys in Hot Compression Process: *Yunping Li*<sup>1</sup>; Singo Kurosu<sup>1</sup>; Emi Onodera<sup>1</sup>; Hiroaki Matsumoto<sup>1</sup>; Akihiko Chiba<sup>1</sup>; <sup>1</sup>Tohoku University

Co-Cr-Mo (CCM) alloys have been considered to be surgical implant materials for the use of artificial hip and knee joint due to their excellent wear resistance, corrosion resistance, and biocompatibility. In order to improve the elongation of this material, it has been found that N addition is greatly helpful in maintaining the high temperature  $\gamma$  phase at room temperature. In the current research, dynamic recrystallization behavior of CCM alloys with and without N addition was analyzed in details. Materials used are Co-29Cr-6Mo and Co-29Cr-6Mo-0.16N. Compression tests were carried out in vacuum from 1000 to 1200°C in a computer aided Thermacmastor- Z hot forging simulator. Strain rates were chosen from 0.01 to 30 /s. The results showed that uniformly distributed superfine grain size in both kinds of alloys could be obtained by continuous dynamic recrystallization (DRX) process; however, it was found that the dynamic recrystallization mechanism is different due to the increasing in stacking fault energy (SFE) after adding N element. Approximately 50 percent of twinning boundaries was observed in the DRXed grain of CCM-N alloy, while no such phenomenon existed in the CCM alloy without N addition.

#### 3:50 PM

# Texture and Substructure Development during Post-Dynamic Annealing in Ni-30%Fe Austenite: *Hossein Beladi*<sup>1</sup>; Pavel Cizek<sup>1</sup>; Peter Hodgson<sup>1</sup>; <sup>1</sup>Deakin University

The texture and substructure development during post-dynamic annealing of an austenitic Ni-30%Fe model alloy was investigated using electron back-scattered diffraction (EBSD) and transmission electron microscopy (TEM). The changes observed within the fully dynamically recrystallized microstructure during post-dynamic annealing have provided a basis to suggest a novel mechanism of metadynamic softening for the current experimental conditions. It was proposed that the initial softening stage involves rapid growth of the dynamically formed nuclei and migration of the mobile boundaries. The sub-boundaries within dynamically recrystallized (DRX) grains progressively disintegrated through dislocation climb and dislocation annihilation, which ultimately led to the formation of dislocation-free grains, and the grain boundary migration gradually became slower. As a result, the DRX texture largely remained preserved throughout the annealing process.

#### 4:05 PM Tea Break

Symposium E:
Solidification, Deformation and Related Processing:
<b>Ultrafine-Grained Materials II</b>

Vonday PM	Room: 2
August 2, 2010	Location: Cairns Convention Centre

Session Chair: Helena Van Swygenhoven, Paul Scherrer Institute

#### 4:30 PM Keynote

Work-Softening, High Pressure Phase Formation and Powder Consolidation by HPT: *Minoru Umemoto*<sup>1</sup>; Yoshikazu Todaka<sup>1</sup>; Koichi Tsuchiya<sup>2</sup>; Bui Duc Long<sup>3</sup>; <sup>1</sup>Toyohashi University of Technology; <sup>2</sup>National Institute for Materials Science; <sup>3</sup>Toyohashi University of Technology and Universiti Sains Malaysia

Various severe plastic deformation (SPD) processes are developed and substantially unique phenomena were reported. In this presentation, the following unique phenomena introduced by high pressure torsion technique are reviewed. 1. Strain induced transformation in stainless steels; 2. Amorphization in TiNi; 3. Dissolution of cementite and pearlite; 4. Mechanical alloying of elemental powder mixture of Cu, Nb and C; 5. Stabilization of high pressure phase in pure Ti and pure Zr; and 6. Structure refinement. Grain refinement by SPD will be discussed for bulk SPD and surface SPD. In bulk SPD, grain size saturates at around 0.2  $\mu$ m while nano-grained structures with 10 to 20 nm can be produced by surface SPD. The deformation conditions to reach nano-grained structure by surface SPD are large strain, large strain gradient, high strain rate, dynamic phase transformation, solute elements and/or precipitation, multi directional deformation. Among these, high strain rate at low deformation temperature and large strain gradient are considered to be most important factors to obtain nano-grained structure.

#### 4:50 PM

Visualization and Quantification of Severe Internal Deformation on Compressive Torsion Process: Yuji Kume<sup>1</sup>; Masakazu Motohashi<sup>1</sup>; Makoto Kobashi<sup>1</sup>; Naoyuki Kanetake<sup>1</sup>; <sup>1</sup>Nagoya University

Compressive torsion process (CTP) which was developed by authors is effective process for grain and precipitates refinement of metallic materials with a severe plastic deformation. In the CTP, a cylindrical specimen is subjected to simultaneous compressive and torsional loading without change in its shape. However, a metal flow and a strain distribution in the processed specimen are not cleared, because the deformation is very large and inhomogeneous. In the present work, visualization of internal deformation of specimen processed by CTP was investigated using dual alloy etching technique. Two kinds of aluminum alloy were prepared by cutting on fun-like shape and alternately placed to a cylindrical shape. After CTPing contrasts in the specimen were observed by polishing and etching. The internal distribution of shear strain was quantified by measuring the displacement of interface between the alloys. As a result, the visualization and quantification of internal deformation was successfully carried out using the technique. The internal strain distribution was varied not only in radial direction but also in longitudinal direction because of frictional constrain on the lateral face. A laminate structure of the allovs observed on the vertical cross section was well related with the strain distribution in the specimen.

#### 5:05 PM Keynote

Processing Design of Groove Pressing for Homogeneous Ultrafine Grained Materials: *Hyoung Seop Kim*<sup>1</sup>; A. Krishnaiah<sup>2</sup>; <sup>1</sup>POSTECH; <sup>2</sup>Osmania University

Manufacturing bulk nanostructured materials with least grain growth from initial powders is challenging because of the bottle neck of bottom-up methods using the conventional powder metallurgy of compaction and sintering. In this study, bottom-up type powder metallurgy processing and top-down type SPD (Severe Plastic Deformation) approaches were combined in order to achieve both real density and grain refinement of metallic powders. Elasto-plastic finite element analysis for plastic deformation behavior of sheet workpiece during groove pressing up to 2 cycles was employed. Deformation localization was studied in terms of strain variations along the longitudinal direction. One cycle of groove pressing is subdivided into five stages; 1st. pressing, 1st. flattening, die shifting, 2nd. pressing and 2nd. flattening. Shearing regions at the first pressing stage become no-shearing flat regions at the second pressing stage, and vice versa. Plastic strain is minimum in the local boundary areas, where plastic deforming zones don't cover during the whole cycle, between shearing and flat regions. A new design of groove pressing for manufacturing ultrafine grained metallic materials was proposed and investigated using the finite element method in associated with a dislocation-based microstructural constitutive model.



#### 5:25 PM

High Strength and Ductility in Ball-Milled Titanium Powders Consolidated by High-Pressure Torsion: *Kaveh Edalati*<sup>1</sup>; Zenji Horita<sup>1</sup>; Hiroshi Fujiwara<sup>2</sup>; Kei Ameyama<sup>2</sup>; <sup>1</sup>Kyushu University; <sup>2</sup>Ritsumeikan University

In the present work, pure Ti (99.5%) powders were subjected to ball milling using a planetary device for 50 hours. High-pressure torsion (HPT) was applied to the ball-milled Ti powders under the pressures of 2 and 6 GPa to introduce severe plastic strain and subsequent consolidation. It is found that the hardness at the steady state under both pressures reaches 330 and 370 Hv, respectively, and the grain size is reduced to ~150 nm. It is shown that consolidation is improved with an increase in the pressure and strain. X-ray diffraction analysis shows that a phase transformation occurs from alpha phase to omega phase during HPT under 6 GPa and this phase transformation leads to an enhancement of the hardness but to a decrease in ductility. A high density (99.9%) and good mechanical properties are well attained by ball milling and subsequent processing by HPT when compared with those of other consolidation methods and of other severe plastic deformation processes.

#### 5:40 PM

Aging Behavior of Al-Li-Cu-Mg Alloy Processed by High-Pressure Torsion: *Seungwon Lee*<sup>1</sup>; Daichi Akama<sup>1</sup>; Zenji Horita<sup>1</sup>; Tetsuya Masuda<sup>2</sup>; Shoichi Hirosawa<sup>2</sup>; Kenji Matsuda<sup>3</sup>; <sup>1</sup>Kyushu University; <sup>2</sup>Yokohama National University; <sup>3</sup>University of Toyama

This study presents an application of high-pressure torsion (HPT) to an Al-Li-Cu-Mg alloy (2091:Al-2.09mass%Li-1.99mass%Cu-1.55mass%Mg-0.12mass%Zr-0.03mass%Si-0.05mass%Fe-0.03mass%Ti). The alloy was subjected to solid solution treatment at 778 K for 30 minutes and was processed by HPT under 6 GPa for 5 revolutions at room temperature. The hardness increased with straining and saturated to a constant level at 225 Hv. Aging was undertaken on the HPT-processed alloy at 373 K for the total period up to 9 days. The aging treatment led to a further increase in the hardness to ~280 Hv. It is shown that the simultaneous strengthening of the alloy due to grain refinement and age hardening was successfully achieved by application of HPT and subsequent aging treatment. The enhancement of the strength is prominent when compared with the application of a conventional rolling process.

#### 5:55 PM Invited

Effects of Grain Morphology on Quasistatic and Dynamic Defromation of Ultrafine Grained OFHC Cu: *Kyung-Tae Park*<sup>1</sup>; Leeju Park<sup>2</sup>; Hyung-Won Kim<sup>2</sup>; Youngil Son<sup>2</sup>; Chong Soo Lee<sup>3</sup>; <sup>1</sup>Hanbat National University; <sup>2</sup>Agency for Defense Development; <sup>3</sup>POSTEC

The quasi-static tensile tests and dynamic compression tests were carried out on ultrafine grained (UFG) oxygen free high conductivity copper (OFHC Cu) having two different grain morphologies. The present study was intended to rationalize the effects of the grain morphology on plastic flow of UFG materials. Two different grain structures, one the lamellar and the other the equiaxed, were prepared by 8 passes equal channel angular pressing (ECAP) with routes A and Bc, respectively. The results of the quasi-static tensile tests at the strain rates of  $10^{-3}$  s<sup>-1</sup> and 1 s<sup>-1</sup> and dynamic compression tests at the strain rates of  $\sim 10^{3}$  s<sup>-1</sup> order revealed that the equiaxed UFG Cu exhibited higher strength and less ductility compared to the lamellar UFG Cu at all strain rates regardless of tension or compression. In order to explain the flow difference between the two, a geometrical orientation relationship between the slip plane and the stress axis was considered.

# Symposium F: Modelling and Simulation of Microstructures and Processes: Materials Modelling I

Monday PM August 2, 2010 Room: 6 Location: Cairns Convention Centre

Session Chair: Khershed Cooper, Naval Research Laboratory

#### 2:00 PM

Modeling of Grain Growth Phenomena: A Critical Review: Chandra Pande<sup>1</sup>; <sup>1</sup>Naval Research Lab.

Grain growth is the well known phenomenon of the evolution of microstructure in a deformed polycrystal after recrystallization resulting in the increase in average grain size, by the motion of grain boundaries due to annealing at a certain temperature and time. Current situation in the modeling of grain growth using both analytical and computational methods is critically reviewed. The models of Hillert, Lucke et al and Mullins are reviewed in detail and shown to be inadequate in describing one or more features of Grain Growth phenomenon. An approach based on the stochastic methods due to Pande (Pande CS (1987) Acta Metall 35:2671) describing main features of Grain Growth phenomenon is presented. It is shown that the model explains most of the observed features of grain growth at least in principle. Finally a brief response to the criticism of this model by Mullins (Mullins WW (1998) Acta Metall 46:6219) is provided.

#### 2:15 PM

#### A Multi-Scale Constitutive Model in High Temperature Deformation of near Alpha Ti-5.6Al-4.8Sn-2.0Zr Alloy: *Miaoquan Li*<sup>1</sup>; <sup>1</sup>Northwestern Polytechnical University

Mon. PM

Isothermal compression of near alpha Ti-5.6Al-4.8Sn-2.0Zr alloy is conducted on a Thermecmaster-Z simulator at the deformation temperatures ranging from 1233 K to 1333 K, the strain rates ranging from 0.001 s<sup>-1</sup> to 10.0 s<sup>-1</sup> at an interval of an order magnitude and the height reductions ranging from 50% to 70%. The primary alpha grain size is measured at an OLYMPUS PMG3 microscope with the quantitative metallography SISC IAS V8.0 image analysis software. A multiscale constitutive model coupling the grain size, volume fraction and dislocation density is established to represent the deformation behavior of near alpha Ti-5.6Al-4.8Sn-2.0Zr allov in high temperature deformation, in which the flow stress is decomposed a thermal stress and an athermal stress. A Kock-Mecking model is adopted to describe the thermally activated stress, and an athermal stress model accounts for the working hardening and Hall-Petch effect. A genetic algorithm (GA)-based objective optimization technique is used for determining material constants in this study. The mean relative difference between the predicted and experimental flow stress is 5.98%, thus it can be concluded that the multi-scale constitutive model with high prediction precision can efficiently predict the deformation behavior of near alpha Ti-5.6Al-4.8Sn-2.0Zr alloy in high temperature deformation.

#### 2:30 PM

Numerical Simulation of Deformation during Hot Procedure for Large Hydraulic Turbine Runner Blade: *Pei Wang*<sup>1</sup>, Namin Xiao<sup>1</sup>; Dianzhong Li<sup>1</sup>; Yiyi Li<sup>1</sup>; <sup>1</sup>Institute of Metal Research, Chinese Academy of Sciences

Francis hydraulic turbine runner blade has a complex profile, which always causes severe and unpredictable deformation during the hot procedure. In this paper, an integral finite element method model based on ProCast software was developed to simulate the stress and deformation of the blade during the whole hot procedure including casting, shake-out of the mould, cutting-off the gating system and heat treatment. And the model predictions were validated by the experimental measurements. Based on the predictions a reverse deformed mould was designed by adding inverse displacements to the initial mould. During the calculation of the inverse displacements, a relaxation coefficient was introduced into the system. The relaxation coefficient at one point is inversely proportional to the restraint intensity at the point, which can be calculated from the ratio of the stress at the point to the maximum stress. Finally the reverse deformed mould was used to produce a huge hydraulic turbine runner blade with uniform and reasonable machining allowance successfully.

#### 2:45 PM

Simulation and Analysis of Plastic Deformation in a Three-Dimensional BCC Microstructure: *Alexis Lewis*<sup>1</sup>; M A Qidwai<sup>2</sup>; Andrew Geltmacher<sup>1</sup>; <sup>1</sup>Naval Research Laboratory; <sup>2</sup>SAIC

An experimentally measured three-dimensional reconstruction of a polycrystalline beta-Ti microsctructure was used as input for simulation and analysis of plastic deformation. The dataset contains the 3D grain morphology and crystallography, as measured using serial sectioning with Electron Backscatter Diffraction (EBSD). The reconstructed microstructure was used as input to a crystal plasticity finite element simulation of deformation in the BCC material. Analysis of the grain morphology and crystallography data in conjunction with the simulated response, particularly plastic strain on the 48 individual BCC slip systems, showed that initiation of plastic strain takes place at certain grain boundaries, and the propagation of strain through the grains and across boundaries depends on the active slip systems, the crystallographic alignment of a grain and its neighbors with respect to the axis of applied load, and the character of the grain boundaries. Data from selected volume elements of approximately 100 grains is compared to data from larger (500 grain) models to determine statistically significant structure-property relationships.

#### 3:00 PM

Simulation of Dislocation Accumulation in ULSI Cells of Reduced Gate Length: *Michihiro Sato*<sup>1</sup>; Tetsuya Ohashi<sup>1</sup>; Keisuke Aikawa<sup>2</sup>; Takuya Maruizumi<sup>3</sup>; Isao Kitagawa<sup>4</sup>; <sup>1</sup>Kitami Institute of Technology; <sup>2</sup>Alps Electric Co., Ltd.; <sup>3</sup>Tokyo City University; <sup>4</sup>Hitachi Ltd.

Length scale of the next-generation type semiconductor devices is going to the range of nano-meter order and, several atomic level problems such as



unevenness of the oxidation film or generation of lattice defects is taking place in the fabrication processes. Among them, dislocation accumulation in the silicon substrate of devices is a serious cause which hinders mass production of the nextgeneration type semiconductor devices. In this study, we numerically evaluate the accumulation of dislocations in periodic structure of the shallow trench isolation (STI) type ULSI cells which has generally been adopted as the latest semiconductor device structure. STI type ULSI cells with gate length less than 62 nm and various trench depths are employed and subjected to a temperature drop from the initial value of 1000 degrees. Dislocation accumulation is simulated by a technique of crystal plasticity analysis. Relations between the geometry of the STI type ULSI cells and dislocation accumulation are discussed.

# 3:15 PM

#### **Statistics for Quantifying the Mechanical Properties of Nanomaterials**: *Chunsheng Lu*<sup>1</sup>; <sup>1</sup>Curtin University of Technology

As the dimensions of materials and devices approach nano-scales, one of the most challenging issues is whether the existing models or theories can still be applied to explain the unique phenomena observed. It is shown that, in most cases, we cannot easily resort to these traditional models because some of underlying assumptions may be unsuitable in nano-scales. Using the statistical analysis of strengths by Weibull distribution as an example, the feasibility of the weakest link principle for nano-structured materials is usually in question, where there are a few rather than numerous defects. Simple and uncritical extrapolation may result in overestimation on strengths of nano-materials and even a misunderstanding on their fracture mechanisms. In this paper, the up-to-date advances in the statistical analysis of statistical methods, the intrinsic mechanical properties can be quantified and optimal information can be extracted from those imperfect experimental data obtained with recently available nano-mechanical testing techniques.

#### 3:30 PM

Strain Localization during Tension and Compression Test and the Plasticity of Cu-Zr Model Amorphous Alloy: *Kyoung-Won Park*<sup>1</sup>; Eric Fleury<sup>1</sup>; <sup>1</sup>KIST (Korea Institute of Science and Technology)

Elastostatic and quasistatic compression tests performed in a wide compositional range of the binary Cu-Zr amorphous alloys have demonstrated that the difference in the structural disordering (or softening, shear localization and energy absorption) during deformation control the plasticity of amorphous alloys. If the binary Cu-Zr amorphous alloys can be deformed plastically with a few percent under compression mode, these alloys break without any plasticity when deformed under uniaxial tensile mode. To investigate these phenomena, we explore the deformation behavior of  $Cu_{65}Zr_{35}$  during tension and compression tests using molecular dynamics simulations. The results demonstrated that when tensile stress is applied to the alloy, the degree of structural softening (strain localization) is high and the yield strength decreased when comparing to the compressive stress. We clarify this different deformation behavior during tension and compression tests, by exploring the relationship between the stress state during tension and compression modes and its effect on the strain localization.

#### 3:45 PM

Studies on Structural and Mechanical Properties of Al<sub>x</sub>FeNiCrCuCo High-Entropy Alloys by DFT Calculation: *Shaoqing Wang*<sup>1</sup>; <sup>1</sup>Institute of Metal Research, Chinese Academy of Sciences

High-entropy alloys (HEAs) have many exceptional properties, such as strong work-hardening capacity, wear resistance, very high mechanical strengths, and satisfied oxidation resistance. It is expected the high-entropy alloys will eventually replace the traditional alloys as the main structure materials in future. The novel properties of HEAs are originated from the following four effects: the high-entropy effect, slow-diffusion effect, lattice-distortion effect and Cocktail effect. It is essential to understand the mechanisms of these effects in atomistic and electronic scales for HEAs' composition design and experimental fabrication. In this work, an elabrate study on the structural and mechanical properties of the Al<sub>x</sub>FeNiCrCuCo HEAs is carried out by DFT first-principles calculation. The combination application of plane-wave pseudopotentials, alchemical pseudoatoms, and the special-quasirandom structure techniques are realized to imitate the random elemental lattice occupation in the alloys. The effects of lattice distortion and Al composition variation to the lattice structure, mechanical strength, and elastic parameters of the alloys are investigated.

#### 4:00 PM Tea Break

Symposium F: Modelling and Simulation of Microstructures and Processes: Solidification Modelling

Monday PMRoom: 6August 2, 2010Location: Cairns Convention Centre

Session Chair: Chandra Pande, Naval Research Laboratory

#### 4:30 PM Keynote

Dendritic Grain Growth Simulation in Weld Molten Pool during Weld Solidification Process: *Dong Wenchao*<sup>1</sup>; Liu Dongrong<sup>1</sup>; Lu Shanping<sup>1</sup>; Li Dianzhong<sup>1</sup>; Li Yiyi<sup>1</sup>; <sup>1</sup>Institute of Metal Research, Chinese Academy of Sciences

In this study, the solidification microstructures of the weld molten pool at different locations along the fusion boundary are simulated during gas tungsten arc welding of SUS304 stainless steel using the CA (cellular automaton) model. Determination of the solidification conditions (temperature gradient, local solidification rate) is carried out with a numerical macroscopic modeling calculation of the weld pool fluid flow and of the temperature distribution. The weld solidification microstructure of SUS304 stainless steel obtained using different welding speeds are simulated. The simulated results reproduced the grain morphology evolution as well as the core concentration distribution of dendrites during the weld solidification process and compared with the theoretical models. It is indicated that the complicated thermal field and solute field can lead to complex grain morphologies in weld molten pool. The influence of the welding speed on the grain boundary segregation is important. When the other parameters are constant, the grain boundary segregations become more severe with increasing welding speed.

#### 4:50 PM

**Thermal Modeling of Direct Digital Melt Deposition Processes**: *Khershed Cooper*<sup>1</sup>; Samuel Lambrakos<sup>1</sup>; <sup>1</sup>NRL

Additive manufacturing involves creating three-dimensional objects by depositing materials layer-by-layer. Examples of additive processes for metals are laser and e-beam deposition. The freeform nature of the method permits the production of components with complex internal and external geometries. Deposition processes provide one more capability, which is the addition of multiple materials in a discrete manner to create heterogeneous objects with local control of composition and microstructure. The result is direct digital manufacturing (DDM) by which different materials are added voxel-by-voxel following a predetermined tool-path. A typical example is functionally-gradient materials such as a gear with a tough core and a wear-resistant surface. The inherent complexity of DDM processes is such that process modelling based on direct physics-based theory is difficult, especially due a lack of temperaturedependent thermo-physical properties. To overcome this difficulty we propose the inverse problem approach to develop thermal models for multi-material deposition processes. Our approach is based on the construction of a numericalalgorithmic framework for modelling anisotropic diffusivity such as that which would occur during energy deposition within a heterogeneous workpiece. This framework consists of path-weighted integral formulations of heat diffusion according to spatial variations in material composition and requires consideration of parameter sensitivity issues.

#### 5:05 PM

A Cellular Automaton Model with the Lower Mesh-Induced Anisotropy for Dendritic Solidification of Pure Substance: Xin Lin<sup>1</sup>; Lei Wei<sup>1</sup>; Meng Wang<sup>1</sup>; Weidong Huang<sup>1</sup>; <sup>1</sup>Northwestern Polytechnical University

A cellular automata model for describing the dendritic solidification morphology of pure substance was developed. Instead of using high mesh anisotropy capture rules, such as Von Neumann's and Moore's method, a new capture rule - random zigzag method was developed, which greatly reduced the mesh anisotropy. The method for the calculation of solid/liquid interface curvature was also improved. The characteristic of dendritic solidification was compared between the simulation results and theoretic model.

#### 5:20 PM

A New Porosity Prediction Criterion for Solidification Simulation of S.G. Iron Casting: *Zheng Hongliang*<sup>1</sup>; Sun Yucheng<sup>2</sup>; Zhang Ning<sup>2</sup>; Tian Xuelei<sup>2</sup>; <sup>1</sup>Mechanical Engineering Post-doctoral Research Station Shandong University; <sup>2</sup>Key Laboratory of Liquid Structure and Heredity of Materials Ministry of Education Shandong University

Although the volume expansion due to graphite formation in spheroidal graphite cast iron greatly affects the porosity defects, most of the conventional methods for



predicting the porosity defects do not include it. With the mold strong enough, the graphite expansion will bring a pressure to the liquid melt and push them to the last solidification field and reduce the porosity formation. Based on these ideas, a new method is proposed with the graphite expansion and liquid pressure included in order to predict porosity more accurately. In particular, the porosity defects are caused by volume change. In this method, the graphite and austenite volume changes during solidification are calculated with the assumption of instaneous nucleation event using experiment data and quasi-steady state diffusion of carbon through the austenite shell. The porosity of vertical board and step castings are calculated with this method. And the calculated results are compared with the experimental results. It is shown that the predicted porosity size, shape and distribution of the method are in good agreement with the measured results.

#### 5:35 PM

#### Modeling of Microstructure Development in a Continuously Solidified Immiscible Alloy: *Jiu Zhao*<sup>1</sup>; Hai Li<sup>1</sup>; Qingliang Wang<sup>1</sup>; <sup>1</sup>Institute of Metal Research, Chinese Academy of Sciences

A model is developed to analyze the microstructure evolution in a continuously solidified immiscible alloy. The model takes into account the common actions of the nucleation and the diffusional growth/shrinkage of the minority phase droplets, the spatial phase segregation and the convections of the melt. The microstructure formation in a continuously solidified immiscible alloy is calculated. The numerical results demonstrate that the convective flow has great effect on the microstructure evolution. The convective flow against the solidification direction causes an increase in the nucleation rate while the convective flow along the solidification direction causes a decrease in the nucleation rate of the minority phase droplets. The convective flow leads to a more nonuniform distribution of the minority phase droplets in the melt. It causes an increase in the size of the largest minority phase droplets and is against the obtaining of the immiscible alloys with a well dispersed microstructure.

#### 5:50 PM

#### Three-Dimensional Modeling and Simulation of Dendrite Morphology of Cast Mg Alloys: Liang Huo<sup>1</sup>; *Zhiqiang Han*<sup>1</sup>; Baicheng Liu<sup>1</sup>; <sup>1</sup>Tsinghua University

A three-dimensional (3-D) cellular automaton (CA) model for simulating the dendrite growth in solidification of magnesium alloy castings has been developed. In the model a technique based on two sets of mesh is utilized to perform the simulation reproducing the morphology of Mg alloy dendrites. The CA calculations are performed using a set of mesh that is defined by the hexagonal close-packed (HCP) crystal lattice, and other computations are carried out by using a cubic mesh. The two sets of mesh are coupled by employing an interpolation method. The kinetics of the solid-liquid interface is obtained directly by the difference between the local equilibrium composition and the local actual composition provided by solving the solute transport equation, which eliminates the necessity for calculating the interface velocities and reduces the complexity of the 3-D computations. The model is used to simulating the 3-D columnar growth of several grains and 3-D equiaxed growth of a single dendrite of Mg alloy AZ91D. The microstructure of the samples obtained from AZ91D Mg alloy castings was studied by optical metallographic examinations and the simulation was compared with the metallographic results.

#### 6:05 PM

# Numerical Simulation of Bubbles Expansion and Solidification of Metal Foams: *Hai Hao*<sup>1</sup>; Mouhamadou Diop<sup>1</sup>; Shan Yao<sup>1</sup>; Xingguo Zhang<sup>1</sup>; <sup>1</sup>Dalian University of Technology

Metal foams have both structural and functional properties, offer advantages in different fields such as automotive and aircraft etc., and thus gain more and more technical interest. In this study, a computational model is presented which allows the numerical simulation of the bubble expansion and solidification during metal foaming processes. Mathematical modelling of the different relevant physical properties and effects during the metal foam expansion and solidification is discussed in this paper. This model considers the broad variety of the complex boundary conditions, the simple and multiphase fluid flow with complex geometries, the stabilization of the foams, the influence of the gravity on the metal foams and the mutual interactions between the bubbles. The Navier-Stokes equation is applied to elucidate the fluid flows in the liquid and in the mushy zone. And the diffusion equation for the hydrogen transport, the distribution functions of the temperature, the density and kinetic equations are also be proposed and discussed.

#### 6:20 PM

#### Mathematical Model on Relationship between Nuclei Number and Cleanness: Cheng Huang<sup>1</sup>; <sup>1</sup>WISCO Engineering and Technology Corporation

The nucleation area law of heterogeneous nucleation during the solidification of liquid metal is analyzed, the relationship between undercooling and solidification time is discussed too, based on which, a mathematical model of relationship between grain number and cleanness is developed, the results showed that solidification time decreased with the increase of undercooling and the heterogeneous nucleation area increased during the solidification period. The mathematic model of relationship between cleanness and grain number developed in this work indicated that when initial cleanness of Fe-C melt was small, the total grain number per unite volume increased with the increase of cleanness at first, and then decreased with the further increase of cleanness. When the initial cleanness was too high, the grain number would decreased continuously with the increase of cleanliness. The solidification experiment was carried out with electrolyte iron, and the experiment results is agreement with the results of the model of this paper.

# Symposium G: Thin Films and Surface Engineering: Thin Films - Preparation and Properties I

Monday PM	Room: 8	
August 2, 2010	Location:	Cairns Convention Centre

Session Chairs: Marcela Bilek, University of Sydney; Chuang Dong, Dalian University of Technology

#### 2:00 PM Keynote

**Superconducting YBCO Thin Films: Progress and Applications**: *Alexey Pan*<sup>1</sup>; Serhiy Pysarenko<sup>1</sup>; Alexandre Weikhard<sup>1</sup>; Sergey Fedoseev<sup>1</sup>; Shixue Dou<sup>1</sup>; <sup>1</sup>University of Wollongong

The progress on the development, fundamentals, properties, and applications of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub> high temperature superconducting films, achieved with pulsed laser deposition (PLD) technique at Institute for Superconducting and Electronic Materials, will be reviewed. An advanced PLD system is used for the epitaxial growth of the films, which is capable of growing various films and hybrid multilayers on the broad range of up to 10-15 cm long single crystal and polycrystalline substrates. The tunable properties of the films and interfaces are optimised to the extent to suit both high power and electronic applications, which demonstrate superior performance, as well assubstantial energy savings. Current-carrying limitations and vortex pinning mechanism have been established and quantitatively developed to describe the behaviour of the critical current density and the parameters of film microstructure.

#### 2:20 PM Keynote

# Epitaxial Growth and Properties of Ferromagnetic Thin Films on Group-IV Semiconductors: *Masanobu Miyao*<sup>1</sup>; <sup>1</sup>Department of Electronics, Kyushu University

Research and development for new semiconductor devices which enable ultrahigh speed operation and ultralow power dissipation are strongly required to overcome a scaling limit of CMOS performance. In line with this, SiGebased heterostructure technologies have been widely developed in a quarter century. These efforts achieved significant enhancement of carrier mobility and resonant tunneling transport. What is the next jump? New functions created by spin injection from ferromagnetic electrodes into semiconductor channels are big candidates to be used for this purpose. To combine such spintronics with SiGe-based heterostructure technologies, it will become important to explore ferromagnetic materials which can be integrated with group-IV-semiconductor devices from now on. In line with this, we have been developing heteroepitaxy of ferromagnetic Heusler alloys on the SiGe platform. Present paper reviews recent progress of the author's group in this field. Main subjects to be discussed in this symposium are as follows: (1) Atomically controlled heteroepitaxy and electrical properties of a ferromagnetic silicide (Fe3Si) on SiGe substrates. (2) Successful spin injection into SiGe channel and prospect for spintransistor (3) Advanced heteroepitaxy of half-metallic alloys for high-efficiency spin-injection.

#### 2:40 PM Invited

# **Epitaxial Nature and Transport Properties in Highly Ionic Conductive** (LaBa)Co<sub>2</sub>O<sub>5</sub> **Epitaxial Thin Films**: *Chonglin Chen*<sup>1</sup>; <sup>1</sup>University of Texas at San Antonio

Mixed ionic/electronic conductive (LaBa) $Co_2O_5$  thin films were epitaxially grown on various single crystal substrates by using pulsed laser deposition. Microstructure characterizations from x-ray diffraction and electron microscopy indicate that the films are highly c-axis oriented with cube-on-cube epitaxy. Transport property measurements indicate that the films have typical semiconductor behavior with a novel phase transition and hysteresis phenomena at 540K for the films grown on (001) LaAlO<sub>3</sub>. The chemical dynamic studies reveals that the resistance of the film changes drastically with the change of redox environment, i.e., the magnitude of resistance changes, delta  $R=10^2$  to  $10^6$  Ohm is



found within a short response time (~700 ms). These phenomena suggest that the as-grown (LaBa)Co<sub>2</sub>O<sub>5</sub> film have extraordinary sensitivity to reducing-oxidizing environment and the exceedingly fast surface exchange rate. These interesting results suggested that the LBCO films can be used for cathode materials, high temperature chemiscal sensors, etc. Details will be discussed in the talk.

#### 2:55 PM

# **Deposition of Superconducting Thin Films of Magnesium Di-Boride on Various Substrates by Electroless Plating Process**: *Susil Putatunda*<sup>1</sup>; <sup>1</sup>Wayne State University

Superconducting thin films of magnesium diboride has been synthesized on various substrates such as gold, silver .copper and silicon using a novel electroless plating technique. The microstructures and the superconducting properties of these films have been characterized using X-ray diffraction, scanning electron microscopy and temperature dependent magnetometry. X-ray diffraction measurements confirm that the films are crystaline magnesium diboride with some impurity phases. Clear evidence for a superconducting transition in the magnetization measurements was observed.

#### 3:10 PM

Investigation on the Mechanical Properties of Molybdenum-Doped Zinc Oxide Transparent Thin Film by Sputtering Technique: *Tao-Hsing Chen*<sup>1</sup>; Po-Tsung Hsieh<sup>1</sup>; Chao-Yu Huang<sup>2</sup>; Ji-Quan Wang<sup>2</sup>; Ricky Wenkuei Chuang<sup>3</sup>; <sup>1</sup>Center for Micro/Nano Science and Technology, National Cheng Kung University; <sup>2</sup>Department of Mechanical Engineering, National Cheng Kung University; <sup>3</sup>Department of Electrical Engineering and Institute of Microelectronics, National Cheng Kung University

Zinc oxide thin films were prepared on the glass substrate by rf-magnetron sputtering technique. The structural, optical characteristics and mechanical properties were then investigated. According to the SEM images, it can be clearly recognized that the average grain size of ZMO thin film are influenced by pressure and sputter power and the value of the average grain size in this study are about 30~50 nm. The EDAX analysis also revealed that Mo was apparently doped into ZnO thin film. The transmittance property of ZMO thin film exhibited an excellent transparency in the visible light range. The transmittance was about 90% for ZMO film with Mo. Moreover, the transmittance also showed a good performance in the range of 350nm to 400nm (UV area). The nano-mechanical properties of ZMO thin films are investigated using a nanoindentation technique. The results show that the Young's modulus and hardness increase with decreasing pressure. However, the hardness increase with increasing sputtering power.

#### 3:25 PM

#### Technological Parameters and Electrical Properties of Ti/TiN Multilayer Films Prepared by Magnetron Sputtering: *Liu Ying*<sup>1</sup>; Hu Min<sup>1</sup>; Lai Zhenquan<sup>1</sup>; <sup>1</sup>Nanchang University

A series of Ti/TiN multilayer films was deposited on Si substrates by DC reactive magnetron sputtering process. The influence of sputtering current density and substrate temperature on cycle membrane structure and its electrical properties was studied. When sputtering current density is less than 0.25A, there is no sputtering; when it is greater than 0.45A, the produced films come off; When it is 0.4A, the sheet resistance and electrical resistivity of the film are minimum value. The sheet resistance and electrical resistivity of the film decrease with the increase of substrate temperature. Therefore, sputtering current density should be controlled between 0.3-0.4A, while substrate temperature should be above 400°C. For a given modulation period and modulation ratio, with the change of number of cycles the films can present a unique set of colours, and its electrical resistivity decreases with the increase of the number. When number of cycles is greater than 3, the sheet resistance is significantly reduced, while the number is greater than 15, the produced films come off. To keep number of cycles at five and change the modulation period, it is shown that colour of the films is golden yellow, and a minimum electrical resistivity exists.

#### 3:40 PM

# Thin Film Coating Thickness Measurement: *Frank Edwards*<sup>1</sup>; <sup>1</sup>KKS Instruments

With the advent of thin film technology the measurement of very thin coatings becomes more and more important in practical applications. With the various coatings applied to those manufactured items a necessity exists to examine the coating thickness and composition, for example, CIS/CIGS PV solar cells, and thin Au/Pd/Ni/CuFe coating on lead frame. Three categorical approaches have emerged so far using evaporation, nano-particles and PVD "sputtering" with targets. In order to maintain the specified electrical features of a photovoltaic panel, the manufacturer must ensure that the solar cell process is kept within a narrow tolerance band. Further work has been conducted with EDXRF on the assaying of precious metals. Finally, the methods of testing CIGS solar cells, found that EDXRF evaluation maintains the solar cell manufacturing under control.

3:55 PM Tea Break

# Symposium G: Thin Films and Surface Engineering: Characterisation and Properties of Engineered Surfaces I

Monday PM	
August 2, 2010	

Room: 8 Location: Cairns Convention Centre

Session Chairs: Xin Jiang, University of Siegen; Yaogen Shen, City University of Hong Kong

#### 4:30 PM Keynote

Plasma Modified Polymeric Surfaces for Covalent Immobilisation of Active Biomolecules without Linker Molecules: Mechanisms for Immobilization: *Marcela Bilek*<sup>1</sup>; Alexey Kondyurin<sup>1</sup>; Yongbai Yin<sup>1</sup>; Neil Nosworthy<sup>1</sup>; Daniel Bax<sup>1</sup>; David McKenzie<sup>1</sup>; Cristobal dos Remedios<sup>1</sup>; Anthony Weiss<sup>1</sup>; <sup>1</sup>University of Sydney

Plasma modification and plasma polymer deposition are valuable technologies for the preparation of surfaces for the covalent binding of biomolecules for applications such as biosensors, medical prosthetics and diagnostic microarrays. The use of linker chemistry to space the molecules from the surface is in some cases beneficial. However, we have shown that linkers are not necessary to retain the function for long periods when the polymer surface is modified by energetic bombardment. The energetic bombardment enables the retention of hydrophilicity of the modified surface by inducing cross linking below the surface which facilitates retention of protein function when stored in buffer solution or freeze dried. Analysis of the modified surfaces shows that the covalent binding mechanism is related to the presence of free radicals on the surface and in the subsurface regions. The presence of structures which allow long range mobility of the radicals and a sufficient number of free radicals capable of accessing the surface during the incubation time is required to enable covalent attachment of a protein monolayer from solution. In this presentation, we present the energetic ion based treatment processes developed in our group and provide evidence for our model of protein attachment to the treated surfaces. Finally we propose novel approaches for obtaining contrast in the protein binding characteristics on a polymeric surface.

#### 4:50 PM

**Characterisation of Bio-Compatible Engineered Surfaces by Neutron and X-Ray Reflectometry**: *Michael James*<sup>1</sup>; Andrew Telford<sup>2</sup>; Chiara Neto<sup>2</sup>; <sup>1</sup>ANSTO; <sup>2</sup>University of Sydney

Examination of nanoscale thin-films and surfaces in Australian using neutron and X-ray reflectometry has entered a "golden age", with the start-up of the Platypus time-of-flight neutron reflectometer at the 20 MW OPAL Research Reactor in Sydney. These techniques allow the study of engineered surfaces with angström-precision and excellent sensitivity to the lightest elements such as hydrogen. This presentation will concentrate on studies of engineered nanoscale thin-films and surfaces generated by microphase separated diblock copolymers as well as polymer hetero-structures. In addition, we present a simple new technique for generating robust, bio-compatible surface coatings without the need to resort to chemical cross-linking agents. We demonstrate excellent anti-fouling properties which correlate with the chemical and physical structure of these materials. Critical examination of these complex nanostructured systems involves a range of surface sensitive and analytical techniques including atomic force microscopy, X-ray photoelectron spectroscopy, quartz crystal microbalance, as well as X-ray and neutron reflectometry.

#### 5:05 PM

# Composition Design and Laser Cladding of Cu-Zr-Al Alloy Coating on the Magnesium Alloy: *Cunshan Wang*<sup>1</sup>; <sup>1</sup>Dalian University of Technology

The cluster line criterion was used for optimized design of a Cu -Zr-Al alloy used as coating on the AZ91HP magnesium alloy by laser cladding. Results show that the coating mainly consists of an amorphous, two ternary intermetallic phases with  $Cu_8Zr_3$ , and  $Cu_{10}Zr_7$  phases type structures, of which the relative content of amorphous phase is about 61%. The formation of the amorphous and intermetallics composite structure results in high hardness, good wear resistance and corrosion resistance. The interface between the clad layer and the substrate has good metallurgical bond.



#### 5:20 PM

Determination of Mechanical Properties of TiN Coating Using a Notched Cylindrical Stainless Steel Substrate: Joe Elambasseril<sup>1</sup>; Raafat Ibrahim<sup>1</sup>; Raj Das<sup>2</sup>; <sup>1</sup>Monash University; <sup>2</sup>CSIRO, Clayton

Comprehensive study of the fracture morphology and the sequence of fracture patterns during tensile loading are important to understand the performance of ceramic coatings during its operation. The ceramic coatings are applied to relatively rough surfaces with edges and corners. When the substrate is not perfectly smooth and flat, large stress concentrations are generated at the coated system. The stress concentrations due to the shape of the substrate also affect the initiation and propagation of cracks in the coatings. The mechanical integrity of TiN and TiAIN coatings are studied using a new test specimen, known as the Cylindrically-Notched-Tensile (CNT) specimen that can mimic the real life applications. The coated specimens under tensile loading are then examined using a scanning electron microscope to study the response of the coatings to the applied loads. The interfacial shear strength of the coatings was determined using the Raj-Agrawal model. The interfacial bonding strength of hard coatings were analysed on both non-precracked and precracked substrates to investigate the effect of the pre-crack on fracture strength. Multiple cracks were observed in the coating in the perpendicular direction to the applied load, and the fracture morphology was found to depend on the stress concentration at notch corners.

#### 5:35 PM

#### Effect of Iodine Doping on Surface and Optical Properties of Polyterpenol Thin Films: *Kateryna Bazaka*<sup>1</sup>; Mohan Jacob<sup>1</sup>; <sup>1</sup>James Cook University

Plasma polymers have attracted much attention due to their unique structure, low-cost and the ability to deposit on a broad range of substrates, including flexible plastics, suitable for application in flexible electronics and biotechnology. As the conductivity of this class of polymers is very low in pristine state, doping with strong acceptors or donors, such as iodine, is frequently used to lower the energy gap and thereby increase the conductivity of the polymer. However, the practical application of iodine doped films is frequently hindered by the decreased transparency and overall stability of the films. This study presents the effect of iodine doping on optical and surface properties of polyterpenol thin films deposited from non-synthetic precursor by means of plasma polymerisation. Spectroscopic ellipsometry studies showed iodine doping reduced the optical band gap from 2.82 eV to 1.50 eV for pristine and doped samples respectively. Higher levels of doping notably reduced the transparency of films, an issue if material is considered for applications that require high transparency. Contact angle studies demonstrated higher hydrophilicity for films deposited at increased doping levels, results confirmed by XPS Spectroscopy and FTIR. Doping had no significant effect on the surface profile or roughness of the film.

#### 5:50 PM

Effects of Mg Doping on the Electrical and Optical Properties of ZnO:Al Transparent Conductive Oxide (TCO): *Sung Min Park*<sup>1</sup>; Gil Ho Gu<sup>1</sup>; Chan Gyung Park<sup>2</sup>; <sup>1</sup>Pohang University of Science and Technology (POSTECH); <sup>2</sup>Pohang University of Science and Technology (POSTECH) & National Center for Nanomaterials Technology (NCNT)

Transparent conductive oxide (TCO) films have been widely used as electrodes for solar cell, light emitting diodes and display devices. In order to achieve good conductivity and high transmittance of TCO, we have tried to fabricate Mg-doped ZnO:Al films and to observe their structural, electrical and optical properties. Mg-doped ZnO:Al films were successfully deposited on top of glass substrates by using ion beam sputter system. The Mg concentration was controlled by varying the number of MgO chips attached on the ZnO:Al target. Energy Dispersive analysis by X-ray (EDAX) was used to determine the concentration of Mg incorporated to ZnO:Al films. Structure properties, electrical properties and optical properties of the Mg-doped ZnO:Al TCO film depending on the Mg concentration have been also analysed. As the concentration of Mg increased, the optical band gap increased due to the replacement of Mg to Zn site in TCO film. Detail will be discussed.

#### 6:05 PM

#### Fundamental Property of Metals – Grain Boundaries Phase Transition as a Basis of Nanostructured Layers, Materials and Composites Production: Yury Minaev<sup>1</sup>; <sup>1</sup>Russian Academy of Natural Science

The right description of coatings technology methods controlled by diffusion require a fundamental understanding of the process or phenomena which define the physic-chemical mechanism of masstransfer. For a development of a technology author focus on the interfacial surfaces and defects on a grain boundaries. The film thermodynamics have been used for description of fundamental property of solid crystalline materials i.e. a grain boundary first-order phase transition with formation of two-dimensional liquid. The calculating of grain boundary phase transition temperature of any metals gives value which lies in range 0.55 - 0.86 of melting point. The described phenomenon reduces in a radical modification

of existing process engineering of handling of metals. It was employed for development of a coatings production for wear improving by gas nitriding. It was studied the hard metal for cutting tools, steel tools for drilling pipes and elements for forcing-lift of diesel. The mechanical behavior of new tools with high surface resistance to wear and to fragile break-down are defined of combination of high microhardness (700 – 2100 Hv) of surface coatings (enriched of nanosize nitrides) with high impact toughness.

#### 6:20 PM

#### Investigation on Interfacial Bonding Strength of Anisotropic Conducive Adhesive with a New Cohesive Zone Model: Jun Zhang<sup>1</sup>; <sup>1</sup>Zhengzhou University

This paper focuses on the bonding strength and lifecycle prediction of the microelectronic products connected by anisotropic conductive film (ACF). Considering the conditions of high temperature and humidity, temperature cycle, a new cohesive zone interface model that has a damage factor couple with the thermal cycle and humidity aging was proposed. The damage factor not only can change the cohesive zone strength acting but also can effect on the energies of separation. The Needleman constitutive equation for the interface model was modified by a damage factor. The thermal damage factor and humidity damage factor can be derived from the experiments. The cohesive zone interfacial model with damage factor used to simulate the specimen bonding by anisotropic conducive adhesive film (ACF) that experience the thermal cycle and humidity tests. The simulation performed by ABAQUS using the user-defined elements. In these calculations, the interface connecting was replaced by user-defined elements that simulated an appropriate traction-separation relationship. The simulated results were compared with the result of experiments.

## Symposium I: Biomaterials, Smart Materials and Structures: Biomaterials

Monday PM	Room: 3	
August 2, 2010	Location:	Cairns Convention Centre

Session Chairs: Hideshi Miura, Kyushu University; Raman Singh, Monash University

#### 2:00 PM

Grain Refining Technique and Mechanical Properties of the Biomedical Co-Cr-Mo Alloys: *Shingo Kurosu*<sup>1</sup>; Yunping Li<sup>1</sup>; Hiroaki Matsumoto<sup>1</sup>; Akihiko Chiba<sup>1</sup>; <sup>1</sup>Institute for Materials Research, Tohoku University

Grain refining is one of the most effective methods achieving high mechanical reliability of materials, because it can enhance the strength without sacrificing other properties. Conventional grain refining techniques for biomedical Co-Cr-Mo alloys involve thermomechanical treatment through recrystallization, such as hot forging, swaging, and rolling etc. and greatly improvements in mechanical properties have widely reported. However, due to the friction between sample surfaces and jig inhomogeneous strains inside of the sample are more or less introduced in the above processes, leading to non-uniform distribution of grains finally. Recently, phase transformation of Co-Cr-Mo-N alloys through eutectoid reaction from fcc phase to hcp phase and M<sub>2</sub>N (M=Cr, Co, Mo) was reported in our recent research. On the basis of this phenomenon, in the current by using the reverse phase transformation from hcp and M2N to fcc above 1273 K, advanced grain refinement technique without deformation was developed and the uniformly distributed fine grains were produced. The grain refinement without plastic deformation is considered to be a very useful way further improving mechanical properties of Co-Cr-Mo products from precision casting or metal injection mold method, in which the fine grain can not be obtained by the conventional methods.

#### 2:15 PM

# **Biodegradable Mg-Ca and Mg-1Ca-1Y alloys for Regenerative Medicine**: *Yuncang Li*<sup>1</sup>; Meiheng Li<sup>2</sup>; Wangyu Hu<sup>2</sup>; Peter Hodgson<sup>1</sup>; Cui<sup>2</sup>e Wen<sup>1</sup>; <sup>1</sup>Deakin University: <sup>2</sup>Hunan University

Mg-Ca alloys with Ca contents from 0.5 to 20.0 wt. % and Mg-1Ca-1Y (wt.%) alloy were prepared by casting from the melt of pure magnesium (Mg), calcium Ca and yttrium (Y) under an atmosphere of pure argon using a steel crucible, aiming to develop new biodegradable bone implant materials. The microstructures and mechanical properties of Mg-Ca and Mg1Ca1Y alloys were investigated. The in vitro cytotoxicities of these alloys were assessed using osteoblast-like SaSO<sub>2</sub> cells. The corrosion of these alloys in simulated body fluid (SBF) and Dulbecco's modified Eagle's medium (DMEM) was evaluated using the concentrations of the released Mg and Ca ions for various immersion intervals. The solutions of SBF



and DMEM with the immersion of Mg-Ca alloys and Mg-1Ca-1Y alloy showed strong alkalisation. Results indicated that the compressive strength, corrosion rate and biocompatibility of the alloys decreased with the increase of the Ca content. It has also been demonstrated that the yttrium does not improve either the corrosion resistance or the biocompatibility of the Mg-1Ca-1Y alloy as expected compared to the Mg-1Ca alloy. It is suggested that Mg-Ca alloys with Ca additions less than 1.0 wt. % exhibited good biocompatibility and low corrosion rate.

#### 2:30 PM

Calcium Phosphate Deposition on Magnesium Alloy for Bioimplant Applications: *Bobby Kannan Mathan*<sup>1</sup>; Yinghe He<sup>1</sup>; Andrew Sandham<sup>1</sup>; <sup>1</sup>James Cook University

In recent years, magnesium and its alloys have been researched extensively for their potential application as biodegradable biomaterials. Magnesium is biodegradable, biocompatible and exhibits mechanical properties similar to natural bone. Magnesium degradation forms a non-toxic soluble product that tends to excrete harmlessly with the urine. In fact, magnesium is essential to human metabolism, engaging in DNA/RNA stabilisation and also acting as a cofactor for enzymes. However, applications of magnesium implants are not common in surgery, primarily because the rate of magnesium degradation is extremely high in physiological conditions i.e., pH levels of 7.4-7.6 and high chloride concentration, and hence the implant materials will dissolve completely much before the expected service life. Various magnesium alloys have been tested for their use as biodegradable implants with varying degrees of improvement in the degradation resistance. However, for significantly higher degradation resistance, biocompatible coating on magnesium-alloy might be more appropriate. Calcium phosphate is biocompatible and has very low degradation rate. This paper investigates a calcium phosphate coating on a magnesium alloy and considers the parameters associated with the application of the coating and the resultant morphology of the deposited layer.

#### 2:45 PM

Formation of Hydroxyapatite on Titanium Oxides in Simulated Body Fluid under UV Irradiation: Masato Ueda<sup>1</sup>; Hiroki Sai<sup>1</sup>; Masahiko Ikeda<sup>1</sup>; Michiharu Ogawa<sup>2</sup>; <sup>1</sup>Kansai University; <sup>2</sup>Daido Steel Co., Ltd

Hydroxyapatite (HAp) is known to precipitate on bioactive materials by soaking in simulated body fluid (SBF). The formation of HAp on TiO, surfaces under continuous ultraviolet (UV) irradiation was investigated in this study. Pure Ti substrates were chemically treated with H<sub>2</sub>O<sub>2</sub>/HNO<sub>2</sub> solution at 353 K for 20 min to form a TiO<sub>2</sub> gel layer. The specimens were then hydrothermally treated with an aqueous NH<sub>3</sub> solution in an autoclave at 453 K for 12 h. An adhesive and sufficiently crystallized anatase-type TiO, film could be synthesized on the Ti surface. The specimens were immersed in SBF in darkness or under UV irradiation with a centered wavelength of  $\lambda$ = 365 nm. Under dark conditions, a thin homogeneous HAp film was formed, with just a few spherical clusters of HAp. The UV irradiation promoted the formation of HAp clusters, which may be due to the generation of functional Ti-OH or Ti-O<sup>-</sup> groups on the TiO<sub>2</sub> surface. On the other hand, the UV light produced electron-hole pairs in the TiO<sub>2</sub>, and the photogenerated holes that migrated to the surface repelled the Ca2+ ions in the solution. As a consequence, the UV irradiation suppressed the formation of a HAp thin film.

#### 3:00 PM

Effect of Cu Content on Unique Hardening Behavior of Dental Ag-Pd-Au-Cu System Alloy Subjected to Solution Treatment: *Yonghwan Kim*<sup>1</sup>; Mitsuo Niinomi<sup>2</sup>; Toshikazu Akahori<sup>2</sup>; Masaaki Nakai<sup>2</sup>; Harumi Tsutsumi<sup>2</sup>; Hisao Fukui<sup>3</sup>; <sup>1</sup>Graduate School of Engineering, Tohoku University; <sup>2</sup>Institute for Materials Research, Tohoku University; <sup>3</sup>Aichi-Gakuin University

Ag-20Pd-12Au-xCu (mass%) alloys have been widely used for dental applications because of lower cost and higher mechanical strength as compared with those of gold-type alloys. It is well-known that the mechanical strengths of Ag-20Pd-12Au-xCu (mass%) alloys increase by aging after a solution treatment. However, it has been reported that the hardness of a certain Ag-20Pd-12AuxCu (mass%) alloy subjected to a solution treatment at a temperature over 1073 K followed by water quenching increases drastically. This unique hardening behavior has been explained by two hardening mechanisms. One is solid solution hardening mechanism, and the other is precipitation hardening mechanism. However, the mechanism of this unique hardening behavior is still unclear because of the complex microstructure with a, a1, a2, and ß phases. Ag-20Pd-12Au-xCu (mass%) alloys with various Cu/Ag ratios fabricated by liquid rapid solidification (LRS) method were prepared for having single a phase in this study. Then, changes in microstructure and mechanical properties of Ag-20Pd-12AuxCu (mass%) alloys with various Cu/Ag ratios subjected to a solution treatment were investigated systematically.

#### 3:15 PM

The Armadillo Osteoderm: Irene Chen<sup>1</sup>; Victor Correa<sup>1</sup>; Po-Yu Chen<sup>1</sup>; Maria Lopez<sup>1</sup>; Marc Meyers<sup>1</sup>; Joanna McKittrick<sup>1</sup>; <sup>1</sup>UC San Diego

The armadillo has a unique protective shell-like armor, called the osteoderm. It has bony components within the osteoderm which result in distinctive mechanical properties. The pectoral and pelvic shields of the osteoderm have hexagonal tiles (5mm in diameter) with composition same as bone (collagen and hydroxyapatite apatite which is the substance found in human bone and teeth); the banded and tail shields of rectangular tiles are also composed of the bone material as well. Optical microscopy reveals that laterally oriented osteons are found in the papillary region within the skin layer of 2 mm in depth and are mainly collagen fibers. The surface layer of the epidermis is approximately 120  $\mu m$  and is mainly keratin. The tough and highly mineralized polygonal tiles underneath the keratin layer have a tensile strength of approximately 23 MPa and toughness of around 0.57 MJ/m<sup>3</sup>. The tiles are bridged together with collagen fibers, called Sharpey fibers. The non-mineralized Sharpey fibers have tensile strength of approximately15 MPa, whereas the tensile strength of the hexagonal tiles are 8 MPa higher than the Sharpey fibers. The mechanical properties (tensile and impact) of the osteoderm and Sharpey fibers are discussed.

#### 3:30 PM

Nano-Hardness Testing of Wear Particles in Sheep Knee Joints: Christopher Allen<sup>1</sup>; Zhongxiao Peng<sup>1</sup>; *Ling Yin*<sup>1</sup>; Emma Carmichael<sup>1</sup>; <sup>1</sup>James Cook University

Osteoarthritis is a common synovial joint disease which affects many people. While the reasons for the occurrence of the disease are not fully understood it is known that articular cartilage of the synovial joint deteriorates with increasing grades of osteoarthritis. The focuses of this study were (a) to establish a suitable procedure for establishing wear particle hardness, and (b) to determine if cartilage wear particle hardness increases with increasing grades of osteoarthritis. Fresh sheep knee joints were obtained and consequently worn in a specially designed wear simulator. Wear particles were then removed from the joint using a syringe and prepared for hardness testing. In order to test the hardness of the wear particle samples nanoindentation was used. Once completed statistical analyses and correlation analyses were performed in order to find any relationships present. This study has found that there seems to be a general relationship between the hardness of wear particles and the grade of osteoarthritis. This relationship shows an increase in wear particle hardness which corresponds to an increase in the grade of osteoarthritis. This is the first time that the hardness of wear particles in knee joints has been tested and obtained.

#### 3:45 PM

Effects of DSS Peptide on Nano-Mechanical Behaviors of Demineralized Human Enamel after Different Aineralization Treatments: *ChiaChan Hsu*<sup>1</sup>; Hsiu-Ying Chung<sup>2</sup>; Wenyuan Shi<sup>1</sup>; Jenn-Ming Yang<sup>1</sup>; Ben Wu<sup>1</sup>; <sup>1</sup>UCLA; <sup>2</sup>Feng Chia University

Aspartate-serine-serine (DSS) repeats are abundant in naturally occurring proteins that are critical for tooth formation. We recently developed aspartateserine-serine peptides to promote hydroxyapatite nucleation from free ions. In this study, we report a possible role of DSS in promoting mineral deposition onto human enamel. Nanoindentation results show that remineralization treatments effectively improve the mechanical and elastic properties for demineralized enamel. The difference in surface roughness and DSS binding affinity among the native and demineralized enamel surfaces may account for this result. The hardness and elastic modulus for the demineralized enamel remineralized with DSS peptide are higher than those remineralized without the addition of DSS peptide. Moreover, the demineralized enamel remineralized with DSS peptide in two times concentration of SBF solution possesses the highest hardness and elastic modulus. This is most likely, a consequence of the uniform calcium phosphate carbonate and HA formation, which creates a smooth surface.

#### 4:00 PM Tea Break



## Symposium I: Biomaterials, Smart Materials and Structures: Ti-Based Biomaterials I

Monday PM	Room: 3	
August 2, 2010	Location:	Cairns Convention Centre

Session Chairs: Rajarshi Banerjee, University of North Texas; Akira Ishida, National Institute for Materials Science

#### 4:30 PM Keynote

Laser Forming of Ti-6Al-7Nb Alloy Powder Compacts for Medical Devices: *Hideshi Miura*<sup>1</sup>; Kenta Okawachi<sup>1</sup>; Hyun Kang<sup>1</sup>; Fujio Tsumori<sup>1</sup>; Kosaku Kurata<sup>1</sup>; Nobuhiro Arimoto<sup>1</sup>; <sup>1</sup>Kyushu University

Titanium and it's alloys have been widely used for medical and aerospace applications because of their excellent attributes of light metal, high strength, high corrosion resistance and high biocompatibility. Especially, Ti-6Al-7Nb alloy has been developed as a more suitable biomaterial to replace Ti-6Al-4V alloy, because the vanadium is toxic to the human body. However, it is not easy to produce the complicate shaped and precise parts due to their poor castability and macinability. In this study, laser forming technique has beem applied for fabricating the complex shaped and precise Ti-6Al-7Nb alloy compacts. The relative density and strength were improved by optimizing the laser forming parameters such as laser power, pulse frequency, scan rate and so on. Moreover, a honeycomb structure by laser forming was designed in order to grow the neighboring tissue and also encourage osseointegration by increasing the surface area. Their results will be discussed at the conference

#### 4:50 PM Keynote

Calcium Phosphate Coating on Titanium Using Dry Process: Takayuki Narushima<sup>1</sup>; <sup>1</sup>Tohoku University

Coating titanium implants with calcium phosphate is one of the techniques used to improve the osseointegration of titanium implants. The radiofrequency (RF) magnetron sputtering has several advantages such as low processing temperature and excellent adhesion of the coating film to metallic substrates. In the fabrication of calcium phosphate films on titanium, the phase, crystallinity and preferential crystallographic orientation of the films can be controlled by changing the sputtering parameters. In our group, oxyapatite and amorphous calcium phosphate films were obtained on the titanium plates by RF magnetron sputtering, and their properties were evaluated in vivo and in vitro. The bonding strength between the calcium phosphate films and the titanium plates was greater than 60 MPa. This value is greater than the bonding strength reported in the case of plasma-sprayed calcium phosphate films fabricated on titanium substrates. The removal torque of the coated titanium implants in the femurs of Japanese white rabbits increased with the duration of implantation, and the removal torque of the coated implants is higher than that of the uncoated implants. In vitro and vivo studies indicate that coating titanium implants with calcium phosphate films by RF magnetron sputtering is effective in improving the biocompatibility of titanium implants.

#### 5:10 PM

**Design of a New Biocompatible Ti-Based Shape Memory Alloy and Its Superelastic Deformation Behaviour**: Jianyu Xiong<sup>1</sup>; Yuncang Li<sup>1</sup>; Peter Hodgson<sup>1</sup>; *Cui'e Wen*<sup>1</sup>; <sup>1</sup>Deakin University

Titanium-nickel (Ti-Ni) shape memory alloys have been widely used for biomedical applications in recent years. However, it is reported that Ni is allergic and possibly carcinogenic to the human body. Therefore, it is desirable to develop new Ni-free titanium-based shape memory alloys for biomedical applications. In the present study, a new Ti-18Nb-5Mo-5Sn (wt.%) alloy, containing only biocompatible alloying elements, was designed with the aid of molecular orbital method and produced by vacuum arc melting. Both  $\beta$  and  $\alpha$ " martensitic phases were found to coexist in the alloy after ice-water quenching, indicating the martensitic transformation of the alloy. The phase transformation temperatures of the Ti-18Nb-5Mo-5Sn alloy were Ms = 7.3°C, Mf = -31.0°C, As = 9.9°C, and Af = 54.8°C. Superelasticity was observed in the alloy at a temperature high than the Af temperature. A totally recovered strain of 3.5% was achieved for the newly designed Ti-based shape memory alloy with a pre-strain of 4%.

# 5:25 PM

Effect of Aging on Mechanical Properties of Ti-Mo-Al Biomedical Shape Memory Alloy: *Hideki Hosoda*<sup>1</sup>; Makoto Taniguchi<sup>2</sup>; Tomonari Inamura<sup>1</sup>; Hiroyasu Kanetaka<sup>3</sup>; Shuichi Miyazaki<sup>2</sup>; <sup>1</sup>Tokyo Institute of Technology; <sup>2</sup>University of Tsukuba; <sup>3</sup>Tohoku University

Ti-Mo-Al is one of Ni-free Ti-based shape memory alloys (SMAs) which are expected to replace Ti-Ni for medical applications. A drawback of such Tibased SMAs is imperfect shape recovery, especially in solution treated alloys. This is because slip deformation is easily introduced due to low critical stress for slip. For the case of Ti-Ni, cold work and aging at intermediate temperatures are utilized to improve shape memory effect through hardening. Therefore, in this work, effect of aging on mechanical properties Ti-Mo-Al with or without cold rolling was studied in the similitude of Ti-Ni. A difference is that brittle omega phase possibly forms at intermediate temperature. Ti-6mol%Mo-8mol%Al alloy was fabricated by Ar arc melting method, homogenized, cold rolled with 95% thickness reduction and homogenized at 1273K for 3.6ks in vacuum. A piece of the sheet was additionally cold rolled with 30% thickness reduction. Then, tensile tests were performed at room temperature. It was found that plateau region, which corresponds to reorientation of martensite variants in stress-strain curves, vanished by cold rolling. And also, the materials became severely brittle by aging at 773K due to omega embrittlement. Effect of multistep aging will be also noted.

#### 5:40 PM

Effect of Oxygen Addition on Isothermal Omega Phase Stability in Ti-29Nb-13Ta-4.6Zr: *Masaaki Nakai*<sup>1</sup>; Mitsuo Niinomi<sup>1</sup>; Toshikazu Akahori<sup>1</sup>; Harumi Tsutsumi<sup>1</sup>; <sup>1</sup>Tohoku University

A peculiar effect of oxygen on  $\omega$ -phase stability, i.e., the enhancement of the isothermal  $\omega$ -phase formation during aging due to oxygen addition was observed in a metastable  $\beta$ -type titanium alloy, Ti-29Nb-13Ta-4.6Zr (mass%); this effect is contradictory to that reported conventionally. The effect was analyzed from a viewpoint of distribution of alloying elements. Oxygen or/and zirconium may dissolve in the  $\omega$  phase during aging, resulting in the stabilization of the  $\omega$  phase in this alloy.

#### 5:55 PM

Effect of Y<sub>2</sub>O<sub>3</sub> on Mechanical Properties of Ti-29Nb-13Ta-4.6Zr for Biomedical Applications: *Xiu Song*<sup>1</sup>; Mitsuo Niinomi<sup>1</sup>; Harumi Tsutsumi; Toshikazu Akahori<sup>1</sup>; Masaaki Nakai<sup>1</sup>; Lei Wang<sup>2</sup>; <sup>1</sup>Institute for Materials Reserch, Tohoku University; <sup>2</sup>The Key Laboratory for Anisotropy and Texture of Materials, Northeastern University

Ti-29Nb-13Ta-4.6Zr (TNTZ) composed of non-toxic and allergy-free elements is expected to be applicable for biomedical applications. Low Young's modulus and excellent biocompatibility are obtained, while mechanical properties subjected to solution treatment are less than those of conventional titanium alloy, Ti-6Al-4V ELI. Mechanical properties of TNTZ are improved by thermomechanical treatments, but Young's modulus becomes high. Therefore, improvements in mechanical properties of TNTZ with keeping low Young's modulus are required. The mechanical properties of TNTZ with different Y2O3 additions were investigated with relating to microstructures in this study. TNTZ with  $Y_2O_3$  additions are all found to be composed of  $\beta$  phase. The grain size of TNTZ with Y<sub>2</sub>O<sub>2</sub> additions becomes smaller than that of TNTZ without Y<sub>2</sub>O<sub>2</sub> although the grain size seems to be nearly the same with the increase in the amount of Y2O3. Y2O3 precipitates can prevent the texture movement and with the increase in the amount of Y<sub>2</sub>O<sub>3</sub>, this effect becomes more obvious. Young's moduli of TNTZ with Y<sub>2</sub>O<sub>3</sub> additions are all kept low, and are almost same as that of TNTZ. The tensile strength of TNTZ is successfully improved by adding Y2O3. TNTZ with 0.2mass% and 1.0mass% Y show good balance of tensile strength and elongation.



## Symposium J: Materials Characterisation and Evaluation: Nanostructured Materials

Monday PMRoom: 1August 2, 2010Location: Cairns Convention Centre

Session Chair: Dongil Kwon, Seoul National University

#### 2:00 PM Keynote

III-V Compound Semiconductor Nanowires for Optoelectronic Applications: Chennupati Jagadish<sup>1</sup>; Qiang Gao<sup>1</sup>; Hannah Joyce<sup>1</sup>; Suriati Paiman<sup>1</sup>; Jung Hyun Kang<sup>1</sup>; Hoe Tan<sup>1</sup>; <sup>1</sup>The Australian National University

Various semiconductor nanowire (NW) photonic and electronic devices have been demonstrated in recent years and continue to attract intensive attention. A highly controllable growth of nanowire in terms of crystal phase, crystal quality and uniform morphology is critical for successful nanowire devices. In addition, a variety of nanowire heterostructures have to be grown in order to satisfy the requirement for device design. However, III-V semiconductor nanowires grown via vapor-liquid-solid (VLS) mechanism often exhibit several problems: for example, tapered morphology, high density of planar defects, mixed WZ and ZB crystal structures, kinked heterostructures and low light emission efficiency. These problems have to be resolved before the commercial device applications for nanowires. In this talk, I will present the research activities in our group at the Australian National University to tackle these problems and integrate III-V semiconductor NWs on Si platform. Results have shown great success in improving morphology, crystal quality and photoluminescence efficiency of III-V NWs and integration of III-V NWs with Si. I will also present an innovative hetero-homostructure with a type-II band alignment by utilising different crystal phases in a single InP NW.

#### 2:20 PM

# Temperature and Magnetic Field Dependence of Electrical Resistance in Trilayer and Fourlayer Graphene Interconnects: *Yanping Liu*<sup>1</sup>; Wen Siang Lew<sup>1</sup>; <sup>1</sup>NTU

In this letter we have investigated the temperature and magnetic field dependence electrical resistance behaviour of trilayer and fourlayer graphene interconnects. It was found that as temperature increases from 5K to 340K the resistance of the multilayer graphene interconnects drops follow intrinsic semiconductors and also found that the two-terminal trilayer and fourlayer graphene-Metal interface resistance increase with magnetic field between from 0T to 6T. In spite of the few layer graphene (FLG) with semimetal zero band gap in the zero field, FLG resistors behaviour more similar to intrinsic semiconductors. The magnetic field dependence of the resistance can be explained by the Landau level splitting was induced by the magnetic field. The energy gap is predicted to be of excitonic nature and to increase with squr(B), according to recent theoretical reported. This gap should be thermally activated and may contribute in first order a component proportional to exp (squr(B)/kT)to R . The obtained results are important for the demonstrated that magnetic field induced low mobility in trilayer and fourlayer graphene. The temperature dependence of the resistance is ascribed to the defect scattering in the low temperature and the thermal generation of the electron-hole pairs and acoustic phonon scattering effect in the high temperature.

#### 2:35 PM

#### **Polarity Induced Growth of Dual-Phase ZnS Tetrapod Heterostructures:** *Zhigang Chen*<sup>1</sup>; Jin Zou<sup>1</sup>; <sup>1</sup>The University of Queensland

Rational design and fabrication of nanostructures as building blocks for functional materials and devices is the key to precise control of their properties for many applications in electronics, optics, biomedicine, and micro/nanosystems. Ionic crystals typically consist of alternating layers of positively charged cations, or, on the opposite side, negatively charged anions, which have characteristically polarized surfaces, resulting in dipole moments and spontaneous polarization along particular directions. These dipole moments and divergence in surface energies may be compensated by creating facets and/or massive surface reconstructions, which can significantly contribute to the asymmetric growth of unique nanostructures. The driving force for the formation of these unique morphologies is the minimization of the electrostatic interaction energy of the polar surface. To date, different compositive tetrapod nanostructures have been reported, but patterned or ordered growth of tetrapods as building block for devices remains a great challenge. Here, we show that the surface polarity can induce novel asymmetric growth of ZnS dual-phase tetrapod heterostructure (a single compound with two phases). Dual phase ZnS tetrapod tree-like heterostructures consist of two phases: zinc blende for the trunk and hexagonal wurtzite for the branch, which demonstrated their TEM and SAED characterization. Direct evidence is presented through quenched experiments. SAED pattern shows the coexistence of the WZ structure and the ZB structure with the orientation relations between the two phases: (2110)//(011) (the directions parallel to the electron beam) and (0001)//{111}.CBED analysis of the trunk and tip observation at the top of brancheds demonstrated that the polarity induced growth of tetrapod ZnS trees through high-resolution electron microscopy study, that is Zn-terminated ZnS (111)//(0001) polar surface is chemically active and S-terminated (111)//(0001) polar surface is inert in the growth of tetrapod ZnS trees.

#### 2:50 PM Keynote

Mechanical Properties Evaluation of Nano-Structured Materials in Scanning Electron Microscope: Seung Hoon Nahm<sup>1</sup>; Hoon-Sik Jang<sup>1</sup>; Sang Koo Jeon<sup>1</sup>; Hak Joo Lee<sup>2</sup>; <sup>1</sup>Korea Research Institute of Standards and Science; <sup>2</sup>Korea Institute of Machinery and Materials

To apply nano-structured materials in micro/nano system, comprehension for the mechanical properties of nano-structured materials is required. In order to perform the mechanical test of nano-structured materials, the mechanical testing system was installed in a scanning electron microscope (SEM). The nanomanipulator was set up in the SEM, and the force sensor, which is formed as a cantilever, was mounted on the nano-manipulator. Then, the force sensor can be controlled by using the nano-manipulator. The nano-structured materials were dispersed on the transmission electron microscope (TEM) grid, and the both end of the nano-structured materials were welded on the TEM grid and the tip of force sensor by E-beam. The mechanical tests of the nano-structured materials were carried out in the SEM. The load response during the mechanical test was obtained by force sensor. The fractured area of nano-structured materials was observed by the TEM, and also the dimension of nano-structured materials was obtained by using the TEM. And then, strain-stress curve was obtained. Here, the mechanical properties for several the nano-structured materials such as carbon nanotubes, ZnO nanorods and ZnS nanowires were evaluated and compared with each other. Finally we suggested standardization method for mechanical properties measurement of nano-structured materials.

#### 3:10 PM

#### Pattern Formation of Copper Nanopowder Deposition Using Nano-Particle Deposition System (NPDS) for the Application of Direct Printing Technology: *Woojin Song*<sup>1</sup>; Kyubong Jung<sup>1</sup>; Doo-Man Chun<sup>2</sup>; Sung-Hoon Ahn<sup>2</sup>; Caroline Lee<sup>1</sup>; <sup>1</sup>Hanyang University; <sup>2</sup>Seoul National University

Nano-Particle Deposition System (NPDS) is a recently developed powder system where both ceramic and metallic powders are deposited on a substrate at room temperature. Previously, cold gas dynamic spray (CGDS) and aerosol deposition method (ADM) have been used to deposit powders on substrate, but they have limitation on the type of powders the equipment can deposit. This drawback eventually causes limitation on powder spraying, including composite powder deposition. On the contrary, NPDS takes the structural advantages from both CGDS and ADM where any type of powders can be deposited at low processing temperature. In this study, copper nanopowders were deposited on a flexible substrate using NPDS. A line pattern was formed on the SU-8 substrate. After the powder deposition, heat treatment step was taken at 250°C to sinter the deposited pattern. After that, the adhesion and electrical properties of copper patterns were investigated, and were compared to that of bulk copper to assure that the pattern was successfully deposited for the application of direct printing technology.

#### 3:25 PM

# **High Throughput Transmission X-Ray Diffraction Analysis**: *David Hay*<sup>1</sup>; Natasha Wright<sup>1</sup>; <sup>1</sup>CSIRO Materials Science and Engineering

The paper describes development of a dedicated high-throughput X-ray microdiffractometer for rapid analysis of crystalline powder samples using transmission Debye-Scherrer geometry. This instrument is one arm of a high throughput X-ray diffraction analysis package utilising both laboratory instrumentation and the Powder Diffraction beamline (10-BM-1) at the Australian Synchrotron. The sample cassette is designed to hold 36 powdered samples in 2.5mm diameter wells, sandwiched between thin polyester films, and it locates precisely, both on laboratory instruments, and the Australian Synchrotron beamline stage. In this way both laboratory and synchrotron data can be collected from identical samples. To date, the methodology has been used successfully for the analysis of various materials in particular metal-organic frameworks with the capability to analyse up to 600 samples per day at the Australian Synchrotron Powder Diffraction beam line.



#### 3:40 PM

Structural Changes Produced by Low Number of ECAP Passes in Coarse Grained Copper: *Ivan Saxl*<sup>1</sup>; Vaclav Sklenicka<sup>1</sup>; Petr Kral<sup>2</sup>; Lucia Ilucova<sup>2</sup>; Milan Svoboda<sup>2</sup>; Jiri Dvorak<sup>2</sup>; <sup>1</sup>Charles University; <sup>2</sup>Institute of Physics of Materials, Academy of Sciences of the Czech Republic

The equal channel angular pressing (ECAP) technology is well known for its grain refinement and considerable improvement of material properties. However, the creep tests revealed that if the original material is coarse grained (the intercept count of the order of several millimetres, hence the grain boundary area per unit volume (area intensity S) is of the order of 0.1[mm<sup>-1</sup>]]), then the greatest improvement of the time to fracture follows the first ECAP pass and then gradually deteriorate after subsequent passes. The purpose of this paper is a detailed examination (by means of EBSD) of copper subboundaries and grain boundaries after the first two ECAP passes. It occurs [101] that the intensity S is roughly 10<sup>3</sup> higher, but the boundary and subboundary structure is extremely inhomogeneous (it is quantitatively characterized by the profile area coefficient of variation CV a) and is not much homogenized even after long time annealing. Examined are also triple grain and subgrain junctions. A peculiar feature is the presence of small dispersed grains inside large grains and subgrains. Relations of such structures to the high temperature properties are discussed.

#### 3:55 PM

**Precipitation Behavior and Magnetic Properties of Nano-Scale Particles in a Cu–Fe–Ni Alloy**: *Sung Kang*<sup>1</sup>; Mahoto Takeda<sup>1</sup>; Masaki Takeguchi<sup>2</sup>; <sup>1</sup>Yokohama National University; <sup>2</sup>National Institute for Materials Science(NIMS)

The microstructural evolutions of nano–scale magnetic particles formed in a Cu–Fe–Ni alloy on isothermal annealing at 873K and 1073K have been investigated by means of transmission electron microscopy (TEM), electron dispersive X–ray spectroscopy (EDS), electron energy–loss spectroscopy (EELS) and field–emission scanning electron microscopy (FE–SEM). Nano–scale magnetic particles were formed randomly in the copper matrix after receiving a short annealing due to phase decomposition in the alloy. With increasing the isothermal annealing time, however, the striking features that two or more nano– scale particles with a cubic shape and precipitates with a needle shape were aligned linearly along <100> directions were observed on isothermal annealing at 873K and 1073K, respectively. To investigate the relationship between microstructures and magnetic properties of precipitates in a Cu–Fe–Ni alloy were also carried out the superconducting quantum interference device (SQUID) magnetometer. The present study revealed several significant influences to magnetic properties were induced during the precipitation process in a Cu–Fe–Ni alloy.

4:10 PM Tea Break

## Symposium J: Materials Characterisation and Evaluation: Mechanical Properties I

Monday PMRoom: 1August 2, 2010Location: Cairns Convention Centre

Session Chairs: J. Wayne Jones, University of Michigan; David Hay, CSIRO Materials Science and Engineering

#### 4:30 PM Keynote

Multiscale Mapping of Mechanical Properties by Instrumented Indentation Test: Kug-Hwan Kim<sup>1</sup>; Young-Cheon Kim<sup>1</sup>; Seung-Kyun Kang<sup>1</sup>; Kwang-Ho Kim<sup>2</sup>; *Dongil Kwon*<sup>1</sup>; <sup>1</sup>Seoul National Univ.; <sup>2</sup>Frontics Inc.

The instrumented indentation test (IIT) is a mechanical testing method to determine the hardness and elastic modulus of materials by putting an indenter into a material surface. This technique has now gone beyond normal hardness tests by evaluating additional properties of materials and by allowing testing at much lower forces and indentation depths (micro/nano ranges). This study presents analytic models and procedures for evaluating tensile flow properties and residual stress state using IIT; the tensile flow properties are treated by defining a representative stress/strain beneath a spherical indenter and the residual stress by using a stress-insensitive contact hardness model. The IIT results are compared with those from conventional methods such as uniaxial tensile test and X-ray diffraction. In addition, IIT can be used as a multiscale mapping tool for the mechanical properties of composite materials and constituent phases by using macro/micro/nano indentation system: we made a hardness map of multiphase steel and measured the strength/residual stress distributions of welded pipeline.

## 4:50 PM

Microstructure and Mechanical Properties of High Strength Brass Alloy with Some Elements: *Haruhiko Atsumi*<sup>1</sup>; Hisashi Imai<sup>1</sup>; Shufeng Li<sup>1</sup>; Yoshiharu Kousaka<sup>2</sup>; Akimichi Kojima<sup>2</sup>; Katsuyoshi Kondoh<sup>1</sup>; <sup>1</sup>Osaka University; <sup>2</sup>Sanetsukinzoku

The purpose of this research was the development of the high strength  $\alpha/\beta$  brass (Cu-40Zn) with the elements of small solid solubility. The high strength brass alloy was extruded by using cast ingot with 0.73 wt% Chromium (Cr), 0.51 wt% Iron (Fe), and 0.66 wt% Tin (Sn). (Cu-40Zn-CrFeSn) The ultimate tensile strength of extruded Cu-40Zn-CrFeSn was 600MPa, 36% higher than that of extruded binary Cu-40Zn without the elements. Micro Vickers hardness of 158Hv was higher than that of extruded Cu-40Zn-CrFeSn was 35%. The strengthening mechanism of this alloy was considered as follows; solid solution elements of 0.14 wt% Cr, 0.12wt% Fe and 0.69 wt% Sn in  $\alpha$  and  $\beta$  phases identified by energy dispersive X-ray spectroscopy. The other was an increase of the area ratio of  $\beta/\alpha$  phase measured by X-ray diffraction integral intensity.

#### 5:05 PM

# **Tensile Properties of Processed FDM Polycarbonate Material**: *Syed Masood*<sup>1</sup>; Kalpeshkumar Mau<sup>1</sup>; Wei Song<sup>1</sup>; <sup>1</sup>Swinburne University of Technology

Knowledge of the mechanical properties of parts processed by Fused Deposition Modelling (FDM) rapid prototyping process is essential for engineering applications of such parts as the mechanical strength of parts depends heavily on the FDM process parameters selected during part fabrication. Little knowledge is available for the Polycarbonate (PC) material used in the FDM systems. This paper presents results of the experimental work on the effect of the FDM process parameters such as air gap, raster width, raster angle and build orientation on the tensile properties of PC and compare them with the published properties of extruded and moulded PC material. Results show that FDM made parts have tensile strength in the range of 70 to 75% of the moulded and extruded PC parts. This shows that the FDM parts are comparatively good enough as functional parts and if it is hard to make intricate shapes by moulding and extrusion, then it is over come by the rapid prototype technology. The results will be valuable for different functional applications of FDM produced parts and assemblies.

#### 5:20 PM

# Effects of Strain Rate on Mechanical Properties and Microstructure of V-5Cr-5Ti Alloy: *Wenjun Hu*<sup>1</sup>; Xicheng Huang<sup>1</sup>; <sup>1</sup>China Academy of Engineering Physics

Vanadium-base alloys are promising candidate materials for application in fusion first-wall and blanket structure because of their high-temperature capability and their potential for low neutron activation and rapid activation decay, good mechanical properties, good compatibility with lithium, and good resistance to irradiation-induced damage. In the present work, the uniaxial tension mechanical responses of V-5Cr-5Ti were experimentally measured on a strain-rate range of  $3.3 \times 10^{-5}$ /s to  $1.2 \times 10^{2}$ /s with split Hopkinson tensile bar and MTS. Microstructure study was performed using the optical microscopy, SEM and TEM. The experimental results show that the tensile strength of V-5Cr-5Ti alloy increase with strain rate, the ductile-to-brittle transient strain rate was about 200/s. The fracture surface of V-5Cr-5Ti at room temperature shows a mixture of microvoid coalescence and intergranular fracture, The grain size is not uniform, and average size is about 500 µm, the grain size in the equiaxial is also not uniform, TEM analysis revealed the precipitation of Ti(O,C,N) on the grain boundaries, and martensite organization exists in all of the samples microsturcture.

#### 5:35 PM

Influence of Mechanical Loading on Failure of Aircraft Protective Coatings: *Ung Hing Tiong*<sup>1</sup>; Graham Clark<sup>1</sup>; Bruce Crawford<sup>2</sup>; <sup>1</sup>RMIT University; <sup>2</sup>Defence Science and Technology Organisation

The degradation and failure of protective coatings (paints and sealants) is a key element influencing the service life of aircraft. Such degradation is influenced by the coatings response to environmental factors such as high temperatures, and exposure to UV radiation, as well as chemical factors. However, the effect of loading and load history on coating durability has received little attention, and is known to be a factor in determining failure sites (such as joints) and the rate of degradation. This paper describes the key characteristics of coatings at aircraft joints, and the complex strain cycling and deformation history experienced by coatings in locations influenced by in-service loads. It also outlines the role of modelling such additional strain as part of developing a prognostic capability for the service life of aircraft coatings. The configuration of coating layers at different joints is important and this research has considered simple generic lap and butt joints; predictions of critical displacements are compared against experimental results obtained using laser speckle interferometry and high resolution microscopy. A reasonably good correlation is observed, and the paper also discusses the



potential interactions between the through-life deformation history of the coating and other environmental factors (eg. temperature, radiation).

#### 5:50 PM

Synthesis, Characterization and Analytical Modelling of Mechanical Behaviour of a Conducting Polymer Actuator: Akif Kaynak<sup>1</sup>; *Chunhui Yang*<sup>1</sup>; Abbas Kouzani<sup>1</sup>; <sup>1</sup>Deakin University

# Electrochemically synthesized tri-layer polypyrrole (PPy) based actuator was optimized for performance and stability. 0.05 M pyrrole and 0.05 M tetrabutylammonium hexaflurophosphate in propylene carbonate (PC) yielded the optimum performance and stability. The force produced ranged from 0.2 to 0.4mN. Cyclic deflection tests on PC based actuators for a duration of 3 hours indicated that the displacement decreased by 60%. PC based actuator had a longer operating time, exceeding 3 hours, compared to acetonitrile based actuators. A triple-layer model of the polymer actuator was developed based on the classic bending beam theory by considering strain continuity between PPy and polyvinylidene fluoride (PVDF). The force was inversely proportional to the length and proportional to the curvature of the deformed actuator. The output forces of the actuator predicted by using the proposed analytic model had a good agreement with the maximum force, which was measured in the range of 0.2-0.5N.

#### 6:05 PM

**Circumferential Notch Testing of Metal Matrix Composites**: *Jonathan Mak*<sup>1</sup>; Wing Yeung<sup>1</sup>; Richard Wuhrer<sup>1</sup>; Di Zhang<sup>2</sup>; <sup>1</sup>University of Technology, Sydney; <sup>2</sup>Shanghai Jiao Tong University

The standard test method for determination of plane strain fracture toughness,  $K_{IC}$  values often require a large specimen size. This restricts applications of fracture toughness tests on materials with limited availability, such as performing continuous monitoring of operating component parts of power plants and assessing properties without affecting their structural integrity. Circumferential notch testing (CNT) is an alternative test technique which is able to use small sized specimens to determine the plane strain fracture toughness values. In this study, CNT was applied to determine the fracture toughness of a series of particle reinforced Ti-6Al-4V metal matrix composites (MMCs) which contained 5 vol. % and 10 vol. % of TiB and TiC particulates respectively. A comparison test on the Ti-6Al-4V base alloy was also performed. Fracture analysis of the materials was performed using scanning electron microscopy (SEM). The distribution and location of the particulates in the metal matrix composites were examined using quantitative x-ray mapping (XRM) technique and their effects on the fracture toughness of the materials were studied and analysed.

#### 6:20 PM

Mechanical Properties of 5083 Aluminium Welds after Manual and Automatic Pulsed Gas Metal Arc Welding Using ER5356 Filler Wire: *Kalenda Mutombo*<sup>1</sup>; Madeleine Du Toit<sup>2</sup>; <sup>1</sup>CSIR; <sup>2</sup>University of Pretoria

Magnesium-alloyed 5083-H111 displays good weldability, although the welded joints often have inferior mechanical properties. Plates prepared with double-V or square butt joint, were joined using manual or automatic pulsed gas metal arc welding (GMAW or MIG) and ER5356 filler wire. Micro-hardness measurements revealed considerable softening in the weld metal and heat affected zone. The tensile strength of the dressed automatic welds was very similar to that of the base metal, but the tensile strength of undressed welds was reduced of about 20%. Undressed manual welds exhibited reduced tensile strength corresponding to about 77% of the tensile strength of the base metal. Automatic dressed weld specimens displayed longer fatigue lives than the manual dressed welds. Failure occurs in the weld metal of all the tensile and fatigue specimens. Fatigue cracks initiated preferentially at gas pores, lack of fusion defects and non-metallic inclusions in the dressed welds, and at weld toes in the undressed welds.

# Symposium L: Energy Generation, Harvesting and Storage Materials: Solar Cell and Catalyst

Monday PMRoom: 7August 2, 2010Location: Cairns Convention Centre

Session Chairs: Dan Li, Monash University; Yan Xiang, Beihang University

#### 2:00 PM Invited

Design of Layered Metal Oxide Photocatalysts Enabling Visible Light Photoactivities: *Lianzhou Wang*<sup>1</sup>; <sup>1</sup>The University of Queensland

Aimed at developing new nanostructures for visible light driven photcatalytic air/water pollutant decomposition, the synthesis, structural modification and exfoliation behaviour of layered transition metal oxides including titanate, tantalates and niobate-based pervoskite were systematically studied. The successful exfoliation of these layered structures led to the formation of colloidal suspension containing paper-like individual metal oxide nanosheets. These unique nanosheets can be surface and/or structural modified into ideal twodimensional building blocks for new nano-architecture fabrication. The layer-bylayer self-assembly and flocculation of nanosheets via electrostatic interaction led to multilayer ultrathin films and restacked nanoporous structures. These newly-developed nanostructures showed excellent visible light photocatalytic performance and promising electrochemical conversion activities.

#### 2:15 PM

**Cyanine and Phycobiliprotein Photosensitizer for Dye-Sensitized Solar Cell**: *Li Qiu Wang*<sup>1</sup>; Yue Wei<sup>1</sup>; Xiao Fei Lv<sup>1</sup>; Ming You Wang<sup>1</sup>; Wei Wei Nie<sup>1</sup>; Li Hui Zheng<sup>1</sup>; Chao Xu<sup>1</sup>; Min Shou Zhao<sup>1</sup>; <sup>1</sup>Yanshan University

Solar cells are important for development and utilization of solar energy. This paper has synthesized a linear trimethine cyanine dye containing p-carboxybenzyl groups on the nitrogen atoms in the heterocyclic rings under ultrasonic wave for dye-sensitized solar cells (DSSC). The cyanine was confirmed by 1HNMR, MS, and its maximum UV absorption and fluorescence emission wavelengths were 551 nm and 565 nm, respectively. Photosynthesis efficiency of spirulina is great due to its phycobiliprotein. This paper cultivated spirulina using mixture of wastewater and seawater. This method made not only cultivating cost of spirulina reduced but also the wastewater purified. Phycobiliprotein was extracted from spirulina by expansion in water under 35°C. Nanometer TiO, film was prepared on glass electrode by sol-gel from butyl titanate, and was sensitized with the cyanine, phycobiliprotein or cyanine-phycobiliprotein by impregnated method, respectively. The results of testing performance of the dye-sensitized TiO, photo anode showed that cyanine-phycobiliprotein had relatively best efficiency for making UV light absorption and fluorescence emission of TiO, film widen and strengthen, the impedances decreased, and photovoltage and photocurrent of its DSSC improved.

#### 2:30 PM

Water-Splitting Photocatalytic Activities of Carbon and Nitrogen Doped TiO<sub>2</sub>/Cr<sub>2</sub>O<sub>3</sub> Nanotube Composites: *Se Im Kim*<sup>1</sup>; Seung-uk Lee<sup>1</sup>; Ji-hun Hwang<sup>1</sup>; Bee Lyong Yang<sup>1</sup>; <sup>1</sup>Kumoh National Institute of Technology

Over the past several years increasing interest has been focused on research of TiO, which has great potential in applications such as photo-catalyst, solar cell, and gas sensor. However, it operates in UV range only due to its large band-gap. For higher efficiency, the optical absorption to visible light, which accounts for 51% of the incoming solar energy, is important. This study has aimed for controlling of redox potential for water splitting through N/C doping and heterojuction to improve the photo-conversion efficiency. The TiO, nanotube arrays with ~7µm length and ~100nm diameter were fabricated by an anodization technique of Ti plates using FA-based electrolytes. The amorphous samples after the anodization were annealed to crystallize at 550°C in air ambient for 4hr. Then TiO, nanotubes were annealed at elevated temperatures in CO and NH<sub>3</sub> gas ambient to dope carbon and nitrogen. The TiO<sub>2</sub>/Cr<sub>2</sub>O<sub>2</sub> nanotube composites were fabricated by dipping the TiO<sub>2</sub> nanotube arrays into liquid sources of Cr<sub>2</sub>O<sub>2</sub>, and then by annealing at high temperatures. Results of microstructural analysis for TiO, nanotube composites by FE-SEM, XRD, and TEM, and results of photocurrent and GC measurements during water-splitting tests under visible light will be also discussed.



## 2:45 PM

Synthesis of Ni-Al Intermetallic Nanoparticle Catalysts by Vacuum Arc Plasma Evaporation: Ya Xu<sup>1</sup>; Junyou Yang<sup>2</sup>; Masahiko Demura<sup>1</sup>; Toru Hara<sup>1</sup>; Toshiyuki Hirano<sup>1</sup>; <sup>1</sup>National Institute for Materials Science; <sup>2</sup>Huazhong University of Science and Technology

Recently, we found that Ni,Al intermetallic compound shows catalytic activity and selectivity for methanol decomposition and methane steam reforming, indicating a possibility to develop Ni-Al intermetallic compounds as catalysts for hydrogen production. In the present study, in order to achieve high catalytic performance of Ni-Al intermetallic compounds for hydrogen production, we synthesized Ni<sub>2</sub>Al-based nanopowders by vacuum arc plasma evaporation technique for the first time. The microstructure and morphology of the synthesized nanopowders were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), energy dispersive X-ray spectroscopy (EDS). The surface area of the nanopowders was analyzed using nitrogen adsorption (Brunauer-Emmett-Teller (BET) method). The catalytic properties of the nanopowders for methanol decomposition were evaluated using a conventional fixed-bed flow reactor. It was found that the particle size of the nanopowders was in the range of 1-100 nm. The nanopowders had a large BET surface area (in the range of 50-110 m<sup>2</sup>/g), and showed very high catalytic activity and selectivity for methanol decomposition at temperatures below 673 K.

#### 3:00 PM

Monodisperse Porous Titania Microspheres and Their Photovoltaic Application: *Dehong Chen*<sup>1</sup>; Fuzhi Huang<sup>2</sup>; Yi-Bing Cheng<sup>2</sup>; Rachel Caruso<sup>1</sup>; <sup>1</sup>Melbourne University; <sup>2</sup>Monash University

The effectiveness of materials in practical application varies considerably with its specific surface area and mesoporosity, composition, crystallinity and, more importantly, unique morphology and texture of the material. Controlled synthesis of titania materials with defined structural architectures has drawn lots of research attention in recent years. In this presentation, monodisperse mesoporous anatase titania microspheres with high surface areas, tunable pore sizes and particle diameters prepared through a combined sol-gel and solvothermal process will be presented. Morphologies and monodispersity of the resultant titania beads can be controlled by the amounts of structure-directing agent involved in the sol-gel process. The diameter of the titania beads, from ~320 to 1150 nm, is tunable by altering the hydrolysis and condensation rate of the titanium alkoxide, while the crystallite size, specific surface area and pore size distribution of the resultant materials can be varied through a mild solvothermal treatment. The proposed formation mechanism of such monodisperse precursor microspheres will also be discussed. Such mesoporous titania microspheres have been used to prepare the working electrodes for dye-sensitized solar cells and an overall photon-to-current conversion efficiency of 8.84% has been achieved based on these unique titania structures, indicating their highly promising application in the photovoltaic field

#### 3:15 PM

Structural Design for the Development of the Floating Type Photovoltaic Energy Generation System: *Hoon Choi*<sup>1</sup>; Hyung-Joong Joo<sup>1</sup>; Jeong-Hun Nam<sup>1</sup>; Kyoung-Soo Kim<sup>1</sup>; Soon-Jong Yoon<sup>1</sup>; <sup>1</sup>Hongik University

This study was focused on the structural design for the development of floating type photovoltaic generation system using pultruded FRP members. Pultruded FRP has superior material properties compared with those of conventional structural materials. Especially, pultruded FRP has an excellent corrosion-resistance and it has high specific strength and stiffness, which is highly appreciated for the design and fabrication of floating type photovoltaic generation system. In the paper, we present the results of investigations pertaining to the mechanical and structural behaviors of FRP structural members based on the experiments. In addition to the experimental investigations, finite element analysis on the floating type photovoltaic generation system using the results obtained from the experiment and finite element analysis.

#### 3:30 PM

Surface Modification of Ni ( $\Gamma$ )-Ni<sub>2</sub>Al( $\Gamma$ ') Two-Phase Foils for Catalytic Activity Enhancement: *Hye-Youn Lee*<sup>1</sup>; Masahiko Demura<sup>2</sup>; Ya Xu<sup>2</sup>; Dang-Moon Wee<sup>1</sup>; Toshiyuki Hirano<sup>2</sup>; <sup>1</sup>KAIST; <sup>2</sup>NIMS

Ni<sub>2</sub>Al foils show catalytic activity for hydrogen production reactions. The activity is expected to be higher on a rough surface having larger surface area compared with a flat surface. In this study, we tried to roughen the surface of Ni( $\gamma$ )-Ni<sub>2</sub>Al( $\gamma'$ ) foils using the selective etching of  $\gamma$  phase. The microstructures of the foils were refined by heavy cold-rolling and subsequent heat-treatment at temperatures 873, 1073 and 1273K. The selective etching of  $\gamma$  phase was conducted by electrochemical processing in the distilled water with 1wt.% (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and 1wt.% citric acid. After the selective etching of  $\gamma$  matrix,  $\gamma'$ 

precipitates were left behind, yielding rough surfaces. The Brunauer-Emmett-Teller results revealed that the surface area increased after the selective etching. In particular, the specimen heat-treated at 1073K had the largest surface area which was expected to show the highest catalytic activity. The large surface area was ascribed to the formation of a large number of fine  $\gamma'$  particles with the size of 10~100nm on the surface. The result of the catalytic activity for methane steam reforming will be reported in the presentation.

3:45 PM Tea Break

# Symposium L: Energy Generation, Harvesting and Storage Materials: Fuel Cell

Monday PM	Room: 7
August 2, 2010	Location: Cairns Convention Centre

Session Chairs: Yoshisato Kimura, Tokyo Institute of Technology; Lianzhou Wang, The University of Queensland

#### 4:30 PM Keynote

Fabrication of Solid Oxide Fuel Cells via Thin Film Techniques: Youngseok Jee<sup>1</sup>; Ikhwang Chang<sup>1</sup>; Suk Won Cha<sup>1</sup>; <sup>1</sup>Seoul National University

Since many researchers highlighted SOFCs as the next generation power source, the first obstacle for the commercialization has been its high operation temperature. We decided to use thin film techniques to solve this problem. Among various methods of lowering the operation temperature for SOFCs, adopting bilayer electrolyte has shown the potential though its concept is relatively simple. Especially the functional layer via thin film deposition could guarantee minimum power density loss and stable operating circumstance. After the study of ALD condition to deposit YSZ on GDC substrate, GDC/YSZ bi-layered electrolyte button cells, showed higher OCV and larger limiting current with 100nm YSZ ultra thin film. The performance improvement might be attributed to the function of electron blocking and cutting off the reducible gas. Another try to lower the operation temperature of SOFCs was thin proton conducting electrolyte using PLD. Because the major polarization portion of the SOFC is ohmic loss, we chose direct solving methodology - proton conducting material that has the conductivity of a couple of orders higher than oxygen conducting one and minimized thickness of the ceramic electrolyte. We investigated the optimization of manufacturing the MEA employing BYZ thin film electrolyte via PLD.

#### 4:50 PM Keynote

The Challenges of Portable Fuel Cell System for Commercial Applications: Lessons Learned and the Road Ahead: *Deryn Chu*<sup>1</sup>; <sup>1</sup>U. S. Army Resaerch Laboratory

The efficient polymer electrolyte membrane fuel cell systems (PEMFCs) are already in development to meet the need for portable applications. The main difficulty, however, remained to be overcome is the development of safe and compact units for hydrogen storage or the on-demand production of this gas. Therefore, direct methanol fuel cell (DMFC) becomes very attractive for an alternative portable fuel cell system. Many advanced engineering design prototypes of direct methanol fuel cell systems have been developed and manufactured by the Smart Fuel Cell (SFC) in Germany under the U.S. Army program. The systems were evaluated at U. S. Army Research Laboratory (ARL) and performance was excellent. However, the DMFC system is still facing many challenges issues such as (1) overpotential losses at methanol electro-oxidation anode and oxygen electro-reduction cathode; (2) methanol crossover through the polymer electrolyte membrane; (3) water management; (4) system complexity and overall size; (5) performance stability; and (6) cost. More detailed results will be presented at the conference. Recently, we initial alkaline anion exchange membrane electrolyte fuel cell. More detailed research and development results will be presented and discussed at the Conference.

#### 5:10 PM

Synthesis of Nanoporous Sinter-Active Layer for Low Temperature Solid Oxide Fuel Cells: Dehua Dong<sup>1</sup>; Dongyuan Zhao<sup>1</sup>; Chun-Zhu Li<sup>2</sup>; *Huanting Wang*<sup>1</sup>; <sup>1</sup>Monash University; <sup>2</sup>Curtin University of Technology

Low temperature solid oxide fuel cells (SOFC) have attracted tremendous attention because inexpensive metals can be employed as interconnect and structural components for SOFCs stack. However, as the operating temperature decreases to below 600°C, the electrode (cathode and anode) polarization resistances substantially increase, and thus dominates total cell resistances. The electrode polarization resistances are largely determined by the interfaces



between the electrolyte and the electrodes, at which electrochemical reactions occur. In this presentation, we report a new synthesis method developed for forming a nanoporous metal oxide layer using a thermosetting polymer template. A nanoporous  $Ce_{0.8}Sm_{0.2}O_{1.9}$  (SDC) layer with high sinterability was prepared on an anode-supported electrolyte film at 900°C, and used as sinter-active layer in cell fabrication to improve the interface between the inert electrolyte surface and the cathode layer of solid oxide fuel cell (SOFC). The resulting interface led to an increase in the maximum powder density of the cell by 51% at 600°C and 162% at 500°C compared with that of the unmodified cell. The cell with the modified electrolyte-cathode interface exhibited lower electrode polarization resistance than those reported in the literature, and such improvement was much more significant at reduced operating temperatures (400-500°C).

#### 5:25 PM

Md

## Solution Processable Graphene: Dan Li1; 1Monash University

Graphene, a new class of two-dimensional carbon nanostructure, has attracted tremendous attention from both the experimental and theoretical scientific community in recent years. This unique nanostructure holds great promise for potential applications in many technological fields such as nanoelectronics, sensors, nanocomposites, batteries and supercapacitors. However, a lack of an efficient approach to producing processable graphene sheets in large quantities has been a major obstacle to exploiting most proposed applications. In this talk, we report that chemically converted graphene sheets obtained from graphite can readily form stable aqueous colloids without the need for any surfactants. We will further discuss how graphene nanosheets can be assembled into strong freestanding films and how their hierarchical structures can be controlled to enhance their electrochemical properties in energy storage and conversion devices.

#### 5:40 PM

Development of High-Power Density Solid Oxide Fuel Cells (SOFCs) for Long-Term Operation: *Norbert Menzler*<sup>1</sup>; Wolfgang Schafbauer<sup>1</sup>; Feng Han<sup>1</sup>; Oliver Büchler<sup>1</sup>; Robert Mücke<sup>1</sup>; Hans Peter Buchkremer<sup>1</sup>; Detlev Stöver<sup>1</sup>; <sup>1</sup>Forschungszentrum Jülich

Solid oxide fuel cells (SOFCs) offer the possibility to produce environmentally friendly energy with high efficiency. The market entry of SOFC systems depends on the functionality of the components and the costs. Up to now the SOFC has not reached market status. The presentation focusses on the manufacturing possitbilities for SOFCs with high power output, long-term durability and by using manufacturing technologies which are near to industry. Forschungszentrum Jülich has developed in the past 15 years so called anode-supported SOFCs in big sizes (up to 200 x 200 mm<sup>2</sup>) and with reproducibly high power output (> 2 A/cm<sup>2</sup> at 800°C). Novel technologies for high capacity manufacturing like tape casting and roller coating have been introduced. Additionally newly developed thin film techniques led to power output of more than 3 A/cm<sup>2</sup> at 800°C or more than 1.5 A/cm<sup>2</sup> below 700°C. These high power densities push open the door for operation of SOFCs at low temperatures to ensure low degradation and therefore long lifetime.

#### 5:55 PM Keynote

Recent Advances in Fabricating Planar SOFC Stack of Novel Design with Higher Thermo-Mechanical Stability: *Hae-Weon Lee*<sup>1</sup>; Jong-Ho Lee<sup>1</sup>; Ji-Won Son<sup>1</sup>; Hae-Ryoung Kim<sup>1</sup>; Byung-Kook Kim<sup>1</sup>; <sup>1</sup>KIST

Commercialization of SOFC technology requires the development of high performance SOFC stack, combined with an engineering focus on stack reliability and cost. Particularly in the viewpoint of ceramic materials, the reliability of SOFC stacks is likely to suffer from the statistical nature of component reliability such that all the SOFC components should be developed based on engineering reliability as well as processing reliability. Recently KIST was successful to greatly improve the performance and reliability of SOFC stack based on newly designed large area planar-type anode-supported cells and glass-based compression-seal gaskets. In this presentation, recent effort to improve thermomechanical stack reliability will be dealt with in terms of microstructural design of the critical stack components and their relevant fabrication processes. Current status of planar SOFC stacks in progress at KIST will be presented in terms of structural reliability and stack scale-up.

#### 6:15 PM

**Ni-Based Supercapacitors for Electrical Energy Storage**: *Hao Gong*<sup>1</sup>; Guangxia Hu<sup>1</sup>; Chunghua Tang<sup>1</sup>; Huimin Li<sup>1</sup>; <sup>1</sup>National University of Singapore

Supercapacitors have attracted more and more attention due to its performance in electric energy storage. In this work, Ni(OH)<sub>2</sub> based supercapacitor electrode films with interconnected nanoflakes was deposited directly on nickel foam substrate, and a high specific capacitance up to 2200 Fg<sup>-1</sup> was achieved. Nanoporous Ni(OH)<sub>2</sub> coated on nickel foam by using chemical bath deposition method shows a high specific capacitance of 2200 Fg<sup>-1</sup> at a discharging current density of 1Ag<sup>-1</sup>. After 500 charging-discharging cycles, the specific capacitance is stabilized at 1470 Fg<sup>-1</sup>, and there is only 5% specific capacitance drop after the following 1500 cycles. The relationship between the capacitance decay and the changes of the microstructure and morphology of nanoporous  $Ni(OH)_2$  is investigated.

#### 6:30 PM

#### A Problematic Issue Regarding Graphene-Based Electrode in Supercapacitor: *Dawei Wang*<sup>1</sup>; Ian Gentle<sup>1</sup>; Max Lu<sup>1</sup>; <sup>1</sup>The University of Queensland

Graphene is the elementary building block of graphite. The successful mass production of exfoliated nanographite flakes from bulk graphite with diverse resources has enabled the wide application of the graphene-based nanoflakes in either electronic fields or energy storage area. Although certain attentions have been devoted to exploring the usage of pure graphene powders or graphenebased composite for supercapacitor electrode applications, little revisit has been focused on the structural feature of graphene-based electrode. We engineered the number of layers of graphene sheets by selective treatments. The number of layers of graphene was determined according to specific surface area. The interfacial capacitance of multilayer graphene sheets is found to depend on the number of layers. This result is attributed to the dependence of space charge layer capacitance of graphene on the number of layers, where the two factors of screening length and stacking thickness play dominantly. This work opens up the understanding of the electronic structure of multilayer graphene via an electrochemical way, and probably initiates interests in the field of electrochemical electronics of graphene.

#### 6:45 PM

Development of Inorganic High Temperature Proton Exchange Membranes for Direct Methanol Fuel Cells: San Ping Jiang<sup>1</sup>; Jinlin Lu<sup>1</sup>; Jie Zeng<sup>1</sup>; <sup>1</sup>Nanyang Technological University

High temperature (100-200°C) proton exchange membrane fuel cells (HT-PEMFCs) have received worldwide attention as a result of several advantages over current PEMFC technology operating at low temperatures (= 100°C). For fuel cell systems directly coupled to a reformer, the primary advantage of high temperatures is the elimination of CO poisoning. Direct methanol fuel cells (DMFCs) benefit greatly from enhanced oxidation kinetics at elevated temperatures. State-of-the-art proton exchange membrane based on perfluorosulfonic acid (PFSA) such as Nafion cannot be operated above 100°C because these membranes require humid operating conditions. The development of hybrid membranes such as Nafion/silica, Nation/TiO, and polybenzimidazole (PBI)/phosphoric acid as high temperature PEMs is not very successful due to the low thermal stability of the polymeric matrix and the leaching of phosphoric acid dopant. Here we report the successful development of a novel HT-PEM based on ordered 12-tungstophosphiric acid (H<sub>3</sub>PW<sub>12</sub>O<sub>40</sub> or HPW)/silica mesoporous nanocomposite prepared by impregnation method. The conductivity and stability of HPW-meso-silica electrolyte membranes were investigated in details. The performance of cells in H<sub>2</sub>/O<sub>2</sub> and in methanol/O<sub>2</sub> at different temperatures based on HPW-meso-silica membrane was presented. The results demonstrate the promising potential of the HT-PEMFCs based on HPW-meso-silica membranes in DMFCs.



# Symposium A: Advanced Steels and Processing: Solidification and Casting of Steels

Tuesday AM	Room: A/B
August 3, 2010	Location: Cairns Convention Centre

Session Chairs: Han Dong, Central Iron and Steel Research Institute; Chung Yun Kang, Pusan National University

#### 8:30 AM Keynote

# Continuous Casting of Advanced Steels of near-Peritectic Composition: Rian Dippenaar<sup>1</sup>; <sup>1</sup>University of Wollongong

The automotive industry is increasingly utilizing advanced high-strength steels, primarily to reduce the mass of motor vehicles. These new types of steel exhibit high strength combined with excellent ductility and impact strength. However, many of these steels fall within the peritectic composition range, which are notoriously difficult to cast by continuous casting techniques. Although the rate and mechanism of the peritectic phase transition in binary iron-carbon alloys are reasonable well understood, the impact of the peritectic phase transition occurring in the newly developed advanced steel designs has not received much attention. A brief review will be given of our current understanding of the peritectic reaction as such and the subsequent peritectic phase transition. New in-situ observations of the peritectic composition seem to be specifically susceptible to transverse surface cracks and this phenomenon as well as the underpinning causes of this type of defect will be discussed.

#### 8:50 AM

#### Mechanism and Kinetics of the Peritectic Phase Transition Relevant to High-Speed Continuous Casting of Steel: Suk-Chun Moon<sup>1</sup>; Rian Dippenaar<sup>1</sup>; Sang-Hyeon Lee<sup>2</sup>; <sup>1</sup>University of Wollongong; <sup>2</sup>POSCO

Recent advances in steel industry have been made by the development of thin-slab casting&hot-rolling processes beneficial for the production of thinner, high quality gauge. It is possible to broaden steel grade selection by utilizing the advantages of these processes, for example in the production of HSLA and AHSS(Advanced High Strength Steel). In thin-slab casting process, due to its high-speed casting characteristics, detailed knowledge of initial solidification is more important than in conventional casting process because operational stability and slab surface quality are determined in this region. Moreover, many advanced steels are frequently designed to have peritectic composition. Volume contraction associated with peritectic transition results in solidified shell detachment from mould and this incurs decrease in heat flux leading to hot spots, which significantly increases the risk of breakouts or surface cracking. These risks are higher in thinslab casting process than in conventional casting process.An attempt is made to obtain fundamental understanding of the peritectic transition occurring in high-strength steels under industrially relevant high-speed continuous casting conditions. The use of high-temperature laser-scanning confocal microscopy for in-situ observations and a levitation droplet technique to study early solidification phenomena is being made. In addition, phase-field modelling is used to interpret experimental observations.

#### 9:05 AM

# Mold Thermocouple Locations and Their Impact on Prevention of Caster Breakouts: *Il Sohn*<sup>1</sup>; Asish Sinha<sup>2</sup>; <sup>1</sup>Yonsei University; <sup>2</sup>United States Steel Corporation

With advances in mold instrumentation, high performance mold fluxes, better reliability maintenance procedures, and improved operating practices, there has been a significant decline in the number of unplanned caster breakouts experienced at U. S. Steel. The typical breakouts of stickers and flux entrapments that were frequently observed in the past are often detected using embedded thermocouples in the mold. However, in-mold events still occur resulting in caster downtime increasing operating costs. Many of these occasional events have been linked to bleeders along the slab corners, slab joint defects during tundish changes, and longitudinal-face-cracking. Considering the potential costs associated with breakouts, improvements have been made in the existing breakout prevention system(BPS) to better detect these events. Major modifications of the existing BPS included addition of edge thermocouples and changes in the location of existing embedded thermocouples to a staggered-design extended the detection range of the BPS without increasing the total number of thermocouples. Furthermore, the implementation of the U.S. Steel longitudinal face cracking detection logic using the temperature measurements from the embedded thermocouples have allowed

strand operators to determine more effectively thermal issues in the mold that can lead to cracking type breakouts below the mold.

#### 9:20 AM

#### Recent Product Developments with Ultra-Thin Cast Strip Products Produced by the CASTRIP® Process: Chris Killmore<sup>1</sup>; Dan Edelman<sup>2</sup>; Kristin Carpenter<sup>1</sup>; Harold Kaul<sup>1</sup>; James Williams<sup>1</sup>; Peter Campbell<sup>2</sup>; Walter Blejde<sup>3</sup>; <sup>1</sup>BlueScope Steel; <sup>2</sup>Nucor Steel - Indiana; <sup>3</sup>Castrip LLC

The CASTRIP® facility at Nucor Steel's Crawfordsville, Indiana plant is the world's first commercial installation for the production of Ultra-Thin Cast Strip (UCS), via twin-roll strip casting. A range of structural grades are in regular production utilising a plain, low-carbon steel type, offering yield strengths covering the range 275 to 380 MPa and strip thicknesses from 0.9 to 1.5 mm. Recent product development has focussed on developing a range of higher strength structural grades utilising microalloying with Nb and V. Niobium was found to retard austenite grain refinement and enhanced hardenability, which produced higher strength levels compared to a plain low C, UCS steel, due to significant microstructural hardening. The Nb and V UCS steels were age hardened using the annealing furnaces of a conventional hot dip galvanising line, achieving further significant strengthening. Strength levels in excess of 550 MPa, combined with good ductility, have been achieved from a low C, lean alloy content, steel type. The effect of microalloying content and processing conditions on the microstructure and final mechanical properties of UCS products produced by the CASTRIP process are described. In addition, recent product development experiences with medium carbon steels and higher residual levels will be discussed.

#### 9:35 AM

Test of the SHTT Method to Simulate the Crystallization of Inclusions in Semi-Killed Steels during Heat Treatment Prior to Hot Rolling: Chong Zhao<sup>1</sup>; *Sung-Mo Jung*<sup>2</sup>; Yoshiaki Kashiwaya<sup>3</sup>; Henri Gaye<sup>2</sup>; Hae-Geon Lee<sup>2</sup>; <sup>1</sup>POSCO China; <sup>2</sup>Pohang Univ. of Science and Technology; <sup>3</sup>Hokkaido University

SHTT (Single Hot Thermocouple Technique) has been used to determine the crystallization behavior of a composition of the CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> system, representative of inclusions in semi-killed steels. XRD and SEM have been employed, respectively, to detect the crystalline phases and to analyze the morphology, quantity and properties of crystals. The same composition has also been studied by quenching the oxides in iron crucibles, a method which had been proven reliable to simulate the actual behavior of inclusions in steel samples during heat treatment prior to hot rolling. The TTT diagrams obtained by using the above two techniques are almost identical. The observed crystalline phases for this composition are gehlenite (2CaO•Al<sub>2</sub>O<sub>3</sub>•SiO<sub>2</sub>) and anorthite (CaO•Al<sub>2</sub>O<sub>2</sub>•2SiO<sub>2</sub>). The TTT diagram from SHTT gives the nose position of gehlenite and anorthite of, respectively, 1200s at 1200°C and 4800s at 1300°C. Thus, the Hot Thermocouple Technique (DHTT or SHTT), already largely used to study the crystallization behavior of CC mold slags is shown to be suitable to simulate the crystallization behavior of inclusions with much higher SiO, contents.

#### 9:50 AM

#### **Interfacial Properties Prediction of Liquid Iron Si Inclusion MgO Refractory**: D. Pradhan<sup>1</sup>; *Ramana Reddy*<sup>1</sup>; <sup>1</sup>The University of Alabama

A thermodynamic model for the prediction of interfacial tension of liquid iron, inclusion and different solid oxide substrates/refractories was evaluated. The combined Good's and Young's equations were used for high temperature liquid metal-solid oxide substrate-inclusion system to evaluate the interfacial tensions. The study predicts the liquid silicon (as model inclusion/impurity) adherence on the solid oxide substrates/refractories (MgO) in a liquid iron melt. The calculated results for interfacial tension between liquid iron-MgO were in good agreement with the experimental results. The Gibbs energy of adhesion for liquid silicon-MgO substrate shows that silicon adhesion to MgO substrate increases with decreasing Gibbs energy of adhesion and temperature. The results shows that silicon adherence to MgO is highly dependent on temperature. It was concluded that MgO is a suitable solid oxide substrates for silicon attachment.

#### 10:05 AM Tea Break



# Symposium A: Advanced Steels and Processing: Bainitic and Martensitic Steels

Tuesday AM August 3, 2010 Room: A/B Location: Cairns Convention Centre

Session Chairs: Tadashi Furuhara, Institute for Materials Research, Tohoku University; David Edmonds, University of Leeds, Institute for Materials Research

#### 10:50 AM Keynote

**Characterization of Substructure Evolution in Ferrous Lenticular Martensite**: *Akinobu Shibata*<sup>1</sup>; Shigekazu Morito<sup>2</sup>; Tadashi Furuhara<sup>3</sup>; Tadashi Maki<sup>4</sup>; <sup>1</sup>Tokyo Institute of Technology; <sup>2</sup>Shimane University; <sup>3</sup>Tohoku University; <sup>4</sup>Nippon Steel Corporation

The morphologies of ferrous martensite are classified into several kinds, i.e., lath, butterfly, lenticular and thin plate, depending on the formation temperature. Among these morphologies, lenticular martensite exhibits a complicated substructure. The substructure of lenticular martensite changes greatly during growth, and consists of three different regions: the midrib, the twinned region and the untwinned region. The present study elucidates the formation mechanism of each substructure. The martensitic transformation of lenticular martensite starts from the midrib, and the midrib is thin plate martensite itself. Substructure of lenticular martensite changes from fine transformation twins in the midrib and twinned region to several sets of screw dislocations in the untwinned region. The substructural change during growth is attributed to the change in the lattice invariant deformation mode due to the local temperature rise inside the martensite plate. Tangled and curved dislocations also appeared near martensite - austenite interface, because the dislocations in the surrounding austenite were inherited by martensite. In the case of lenticular martensite with relatively high Ms temperature, tangled and curved dislocations appeared in the entire untwinned region. This is because the higher Ms temperature facilitates the plastic deformation in the surrounding austenite.

#### 11:10 AM

**Evidence of Lath Martensite in High-C Japanese Sword Produced from Tamahagane Steel by Tatara Process**: Takuya Ohba<sup>1</sup>; *Ananda Das*<sup>1</sup>; Shigekazu Morito<sup>1</sup>; Muneo Yaso<sup>2</sup>; <sup>1</sup>Shimane University; <sup>2</sup>Wakoh Museum

Field Emission Scanning Electron Microscopy with Electron Back-Scattering Diffraction (SEM- EBSD) and Optical microscopy were used to point out the microstructural features of a Japanese sword prepared from tamahagane steel using traditional tatara method. A lath martensite structure, which is usually characterized by packet and block in a prior austenite grain, existed both on the surface and in the cross-section of the sword. SEM-EBSD study revealed that the development of prior austenite grain and packet were not much distinctive but the blocks within the packets were fairly observed. It was found that the packet size increased with the prior austenite grain size but the increment was small. Vickers micro-hardness measurement revealed that the sharp end was comparatively harder than other sections of the sword. EPMA study showed that the average carbon content of the sword was around 1 mass% along with a variety of nonmetallic inclusions. Formation of lath martensite structure in such high carbon steel is remarkable but comparable to 0.6 mass% carbon ordinary steel. It was realized that the traditional method of preparation using tamahagane as well as the higher content of carbon provided the extraordinary features to the Japanese sword different from the ordinary steel.

#### 11:25 AM

#### Isothermal Martensitic Transformation in a Sensitized SUS304 Stainless Steel under Magnetic Field: Ju Young Choi<sup>1</sup>; Takashi Fukuda<sup>1</sup>; Tomoyuki Kakeshita<sup>1</sup>; <sup>1</sup>Osaka University

We have investigated effect of magnetic field at cryogenic temperature on the stability of austenitic phase in a sensitized SUS304 stainless steel. The steel was solution-treated at 1323K for 0.5 h followed by sensitization heat-treatment at 973K for 100 h. We found that the sensitized SUS304 exhibits isothermal martensitic transformation and the *TTT* diagram corresponding to the formation of 0.5 vol. % of  $\alpha$ '-martensite shows a double-C curve with two noses located at about 100 and 200 K. In-situ optical microscope observation reveals that the upper nose is due to the  $\gamma$  (fcc)– $\alpha$ ' (bcc) transformation and the lower nose is due to the  $\gamma$  (fcc)– $\epsilon$ ' (hcp) transformation. We also found that the nose temperature and the incubation time of  $\gamma$ – $\alpha$ ' transformation become lower and shorter, respectively, with increasing magnetic field. On the other hand, the nose temperature of the  $\gamma$ – $\epsilon$ 'transformation does not change by the application of magnetic field.

#### 11:40 AM Relationship between I

Relationship between Particle Size and Martensitic Transformation in Fe-30at%Ni Alloy: *Jung-min Nam*<sup>1</sup>; Masashi Mino<sup>1</sup>; Yoshikazu Aikawa<sup>1</sup>; Tomoyuki Terai<sup>1</sup>; Tomoyuki Kakeshita<sup>1</sup>; <sup>1</sup>Osaka University

The martensitic transformation characteristics are reported to be strongly influenced by particle size. However, up to now, these experiments were made by using polycrystalline particles, and the effect of particle size has not been well understood yet. In the present study, therefore, we investigated the effect of particle size on martensitic transformation by using single crystalline particles of an Fe-30at%Ni alloy exhibiting a martensitic transformation from f.c.c. structure to b.c.c. structure. The alloy was prepared by melting high purity iron and nickel by the arc-melting method and then the alloy powder was fabricated by the gas atomizing method. The microstructures were observed by SEM. The martensitic transformation was detected by an X-ray diffraction and the amount of martensite was evaluated by M-H curves. Following results are obtained: i) The athermal martensitic transformation changes to an isothermal one with decreasing particle size. The *TTT* diagram shows a C-curve with nose temperature about 170 K; ii) Based on a phenomenological theory derived by the authors, the size of cluster for nucleation is calculated to be about 0.9 nm<sup>3</sup>.

#### 11:55 AM

Tensile Deformation Behavior of Tempered Martensitic Steel Produced in CGL Lines: *Taejin Song*<sup>1</sup>; Jaehyen Kwak<sup>2</sup>; B. C. De Cooman<sup>3</sup>; <sup>1</sup>POSTECH; <sup>2</sup>POSCO; <sup>3</sup>Graduate Institute of Ferrous Technology

Thermal cycles of conventional galvanizing and galvannealing processes were applied to low carbon martensitic steels to examine the mechanical property of martensitic steels after their processing in conventional CGL lines. During the thermal cycle simulation, tempering phenomena occurred resulting in changes of microstructure and mechanical properties. In this study, the tensile deformation behavior of martensitic steel was studied in detail in order to understand the tempering phenomena occurring during their processing. Conventional uniaxial tensile tests were carried out and the samples were observed by Infra Red Thermography. It was found that, after tempering process, strain hardening ability decreased drastically and that the plastic flow became localized. An experimental analysis of this phenomenon will be presented based on TEM microstructural observation and Internal Friction measurements.

#### 12:10 PM

Nucleation of Bainite on Allotriomorphic Ferrite/Austenite Interface in a Low Carbon Steel: *Hui Guo*<sup>1</sup>; Yin Bai<sup>1</sup>; Shanwu Yang<sup>1</sup>; Xinlai He<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing

The low carbon bainitic steels are applied widely due to their good combination of strength, toughness and weldability. The bainite is suggested to nucleate at the grain boundaries of prior austenite. In a low carbon steel, however, the allotriomorphic ferrite is usually the first phase formed during the cooling of the austenite. The allotriomorphic ferrite/austenite interface may influence the nucleation of the bainite and the final size of bainite packets, which indicates the effective grain size and decides the strength and toughness. The nucleation of bainite on allotriomorphic ferrite/austenite interface in a low carbon steel is studied in this paper. The orientation of the allotriomorphic ferrite and bainite nearby is measured by EBSD. The influence of the nature of the allotriomorphic ferrite/austenite interface on the nucleation of bainite is investigated.

#### 12:25 PM

Nano-Scale Analysis of Nano-Bainite Formed in Advanced High Strength Steels: *Ilana Timokhina*<sup>1</sup>; Hossein Beladi<sup>1</sup>; Peter Hodgson<sup>1</sup>; Elena Pereloma<sup>1</sup>; Xiang Xiong<sup>1</sup>; <sup>1</sup>Deakin University

Bainite is a phase formed in a variety of high strength steels as a deliberate attempt to obtain a particular combination of strength, ductility and toughness. The contribution of bainite to the structure-property relationship depends on its morphology. The effect of composition and processing schedule on the morphology of carbide-free bainite was studied in 0.79C-1.5Si-1.98Mn-0.24Mo-1.06Al, 0.26C-1.96Si-2Mn-0.31Mo and 0.2C-1.5Mn-1.2Si-0.3Mo-0.6Al-0.02Nb (wt%) steels using transmission electron microscopy (TEM) and atom probe tomography (APT). All these steels offered a unique combination of ultimate tensile strength and elongation due to the formation of non-carbide bainite with retained austenite. The microstructural characteristics of bainite, such as dislocation density and thickness of bainitic ferrite laths and retained austenite islands, crystallographic analysis of bainitic ferrite and retained austenite, as a function of steel composition and processing was studied using TEM. The average carbon content of bainitic ferrite and retained austenite was determined using APT. The carbon content of bainitic ferrite was higher than expected from paraequilibrium level of carbon in ferrite. The formation of fine Fe-C carbides and clusters in bainitic ferrite was observed in all steels. The distribution of substitutional elements within bainitic ferrite, and retained austenite also was studied for all steels by APT.



#### 12:40 PM

New Type Bainitic Steel for Grade R5 High Performance Offshore Mooring Chain: Anxiang Tao<sup>1</sup>; Weixin Zhang<sup>1</sup>; *Jiang Yin*<sup>2</sup>; <sup>1</sup>Jiangsu Asian Star Anchor Chain Co. Ltd.; <sup>2</sup>Baoshan Iron and Steel Co. Ltd.

A new type bainitic steel for grade R5 (typical composition: 0.21C-0.90Mn-0.25Si-1.90Cr-0.9Ni-0.45Mo-microalloy) high performance offshore mooring chain welded by flash butt with tensile strength of 1000MPa is presented in this paper. In this work we focus on the high performance of the new type bainitic steel from the following aspects: composition design, control of structure and property, the mechanical properties, the corrosion performance, environment assisted cracking (EAC). Experimental data show that: 1) New type R5 mooring chains have high performance and reliability that is high strength, high toughness and low EAC sensibility, fine and uniform structure from clean steel with low inclusion and low harmful elements. 2) The yield to tensile ratio is controlled to guarantee low brittle failure sensibility of new type R5 chain to prevent nonductile fracture while un-controlled. 3) The product of the strength multiplied by the elongation of new type R5 chain is very high. The difference of the strength of the base and the weld of the links is very small and change of the strength of the links is very narrow.

## Symposium A: Advanced Steels and Processing: Precipitation in Steels/Hypereutectoid Steels

Tuesday AMRoom: 5August 3, 2010Location: Cairns Convention Centre

Session Chair: Michael Miller, Oak Ridge National Laboratory

#### 8:30 AM Keynote

#### Investigation of Experiment and Theory on Precipitation Transition Behaviors in Reduced Activation Steels: *Chi Zhang*<sup>1</sup>; <sup>1</sup>Tsinghua University

Experiments and thermodynamic calculations were performed on reduced activation steels containing 0 to 9 wt% of chromium and 0.15wt% of tantalum. Detailed microstructure analyses indicated that Ta did form TaC in reduced activation steels. After austenitizing at 1050°C for 1 hour, the larger spherical particles had an average diameter of 150 nm, what were believed to be TaC. The undissolved TaC exerted strong pinning force on migrating packet and prior austenite grain boundary. The smaller TaC particles had an average diameter from 15 to 45 nm during holding at 750° for a series of durations. In the process of the re-dissolution of the precipitated TaC particles during tempering, the dissolved atoms were consumed in precipitating new particles but not in coarsening the preexisting particles, resulting in the coarsening rate of MX particles slowed down. The especial atoms plane (200)TaC, (111)TaC existed in TaC with matrix during precipitation and coarsening. The chromium carbides precipitation sequence M3C->M7C3->M23C6 occured in reduced activation steels during precipitation and coarsening, and M23C6 was the stable chromium carbides in reduced activation steels. The thermodynamic modeling model of dissolved carbides was also developed at austenitizing process, it was coincide well with experimental results

#### 8:50 AM Keynote

**The Study on the Precipitation and Mechanical Properties of Bainitic Steels**: Chuang Li<sup>1</sup>; *Xinlai He*<sup>1</sup>; Xuemin Wang<sup>1</sup>; Chengjia Shang<sup>1</sup>; Yu He<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing

The properties and precipitation behaviour of Cu-bearing bainitic steels has been studied. The optical microscope and HRTEM were employed to study the influence of cooling rate on the precipitation process. Also, the properties of steels with different processes are tested. The results show that when the steels was cooled at a cooling rate between 0.1-1°C/s with the cooling rate increasing the second phase precipitates become finer but the precipitates become denser. When the cooling rate is 1°C/s the density of the second phase precipitates are the largest. When the cooling rate is quicker than 1°C/s as the cooling rate increase the precipitates become finer and fewer. The hardness tests also show that the sample will get the highest hardness. During the continuous cooling a large quantity of fine precipitates are found in ferrite and granular bainite. With the cooling rate larger than 3°C/s the microstructure is mainly lath-like bainite and there are few precipitates.

#### 9:10 AM

In-Situ TEM and APT Analysis on the Dislocations Associated with Solute Carbons in Strain-Aged Low Carbon Linepipe Steels: *Hyoung Seok Park*<sup>1</sup>; Ju Seok Kang<sup>1</sup>; Jang Yong Yoo<sup>2</sup>; Chan Gyung Park<sup>3</sup>; <sup>1</sup>Pohang University of Science and Technology (POSTECH); <sup>2</sup>POSCO; <sup>3</sup>Pohang University of Science and Technology (POSTECH) & National Center for Nanomaterials Technology (NCNT)

Low carbon (~0.34 at.%) linepipe steels with high strength and high toughness are widely used for the transportation of petroleum and natural gas. These linepipes usually experience UOE piping followed by FEB (Fusion Epoxy Bond) coating at 250°C, which usually cause strain aging phenomenon with discontinuous yielding in practical field. In order to understand the major cause of the strain aging in these low carbon linepipe steels, the dislocation associated with solute carbons have been investigated in three different types of steels (plate, pipe, and coated pipe). In-situ TEM heating up to 250°C for 30min did not cause any change of structure in both steel plate and pipe. However, APT results revealed the segregation of solute carbon along dislocation lines in ferrite region of both UOE pipes and coated pipes. We believe this solute segregation along the dislocations is the major cause of strain aging of low carbon linepipe steels.

#### 9:25 AM

Influence of Silicon on the Spheroidization of Cementite in Hypereutectoid Bearing Steels: *Kwanho Kim*<sup>1</sup>; Jaeseung Lee<sup>1</sup>; Duklak Lee<sup>1</sup>; <sup>1</sup>POSCO

The influence of silicon on the spheroidization of cementite in hypereutectoid

1.0C-1.45Cr bearing steels has been investigated, on the basis of microstructural analysis by scanning electron microscopy and thermodynamic calculations by Thermo-Calc. The silicon content was varied 0.25 to 2.00 in weight percent. Before spheroidizing, it was confirmed that the interlamellar spacing in asrolled conditions was almost the same as 0.30 µm with varying silicon content. Annealed at 790-850°C for 6 hr. the 0.25Si and 1.00Si steels were entirely spheroidized at 790°C, while 1.50Si and 2.00Si steels at 830°C, respectively. This implies that the increase of silicon content in hypereutectoid steels retards the spheoridization of cementite, therefore requiring higher annealing temperature to assure the complete spheroidization. The thermodynamic calculations revealed that silicon atoms were partitioned into not cementite but austenite at annealing temperatures, and the increase of silicon content led to the increase of activities of carbon atoms. As a result, it is suggested that the increase of silicon content can raise the chemical potential of carbon atoms within austenite at austenite/ cementite interfaces, causing the decrease of driving force for the diffusion of carbon atoms from cementite to austenite.

#### 9:40 AM

Effect of Aluminum Content on the Microstructure and Mechanical Properties of Hypereutectoid Steels: Yoon Soo Jang<sup>1</sup>; Phaniraj Madakashira<sup>2</sup>; *Dong-Ik Kim*<sup>2</sup>; Jae-Hyeok Shim<sup>2</sup>; Moo-Young Huh<sup>1</sup>; <sup>1</sup>Korea University; <sup>2</sup>Korea Institute of Science and Technology

Hypereutectoid(1.15%C) steels with 0, 0.69, 1.29 and 1.95 wt. % aluminum were prepared. The samples were hot rolled at 1100°C followed by cooling in air. The microstructure of the as-rolled samples was characterized using field emission-scanning electron microscopy (FE-SEM). The electron back scattered diffraction (EBSD) technique was used to identify the grain boundary phases. The steels have a pearlitic microstructure with different amounts of grain boundary cementite. A continuous grain boundary cementite network is present in the 0 wt. % Al steel. Grain boundary cementite formation is completely suppressed in the 1.29 wt. % Al steel. Phase diagram calculations show that aluminum increases the eutectoid temperature. However, the interlamellar spacing and pearlite colony size decrease with increase in aluminum content. Dilatometry measurements show that aluminum addition increases the undercooling below the eutectoid temperature.

#### 9:55 AM

# **Evolution of Inclusions in High Mn-Al Alloyed Steels at 1873 K**: Dong Jin Kim<sup>1</sup>; Chan Ik Park<sup>1</sup>; Geun Ho Park<sup>1</sup>; *Joohyun Park*<sup>1</sup>; <sup>1</sup>University of Ulsan

High manganese (~30wt%) steels have been significantly issued in view of their good mechanical properties such as high strength and good ductility. At present, twin-induced plasticity (TWIP) aided steels satisfy a mechanical demand by tensile strength of ~900MPa and extended ductility over ~50%. However, weight savings of TWIP steels for automotive applications are not effective because of the atomic weight of Mn comparable to that of Fe. Thus, several investigations recently revealed that the Al addition up to ~10wt% into the high Mn steels is beneficial for achieving not only remarkable weight savings but also mechanical properties comparable to or better than those of TWIP steels. Even though several types of steel compositions are designed, there are few studies for the formation behavior of inclusions in high Mn-Al alloyed steels at high temperatures. Thus, in the present study, the effect of Al addition (1~9wt%) on the formation behavior of inclusions in the Fe-Mn steels of which Mn content



from 10 to 20wt% is investigated as a function of reaction time at 1873 K. The changes in the composition, morphology and the size distribution of inclusions are discussed based on the thermodynamics of Mn and Al in Fe-Mn-Al system.

#### 10:10 AM

In Situ Observation of Microstructure Evolution in Low Carbon Bainite Steels Isothermally Held below A1 Temperature: Rui Zhang<sup>1</sup>; *Shanwu Yang<sup>1</sup>*; Chao Sun<sup>1</sup>; Xinlai He<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing

Due to their excellent mechanical properties and weldability, low carbon bainite steels have been widely applied in bridges, ships, construction, etc in recent decades. The dominant microstructures in the steels such as bainitic ferrite, granular bainite and acicular ferrite are nonequilibrium phases, which will probably evolve towards equilibrium microstructure, i.e. polygonal ferrite and cementite, when the steels are subjected to thermal disturbance. The evolution will result in marked decreasing of strength of the steels. In the present investigation, in situ observation by optical and scanning electron microscopy was carried out to track the evolution when the steels were isothermally held below A1 temperature. It was found that the evolution progresses in a way similar to recovery and recrestallization of deformed metals. Different microstructures in the steels exhibit obviously different thermal stability and evolution behavior. The prior interfaces in the steels provide nucleation sites for recrystallization. Pre-deformation will promote the evolution to occur.

Tue. AM

10:25 AM Tea Break

# Symposium A: Advanced Steels and Processing: Q&P

Tuesday AM August 3, 2010 Room: 5 Location: Cairns Convention Centre

Session Chairs: Bruno De Cooman, Pohang University of Science and Technology; Ilana Timokhina, Deakin University

#### 10:50 AM Keynote

Application of Quenching and Partitioning to Improve the Ductility of Ultra High Strength Low Alloy Steel: *Wenquan Cao*<sup>1</sup>; Jie Shi<sup>1</sup>; Han Dong<sup>1</sup>; <sup>1</sup>National Engineering Research Center of Advanced Steel Technology, Central Iron and Steel Research Institute

In this study Quenching and Partitioning (Q&P)as proposed by Speer was applied to improve the ductility of C-Mn high strength Low Alloy steel (HSLAs). The microstructures of processed steels were examined by scanning electron microscopy equipped with electron back scattered diffraction(SEM/ EBSD), transmission electron microscopy(TEM) and x-rays diffraction(XRD). Mechanical properties were measured by uniaxial tensile testing. Microstructural observations revealed a multiphase microstructure including first martensite, fresh martensite and retained austenite in the Q&P processed steel. During tensile process, the austenite volume fraction gradually decreased with strain increasing, suggesting the phase transformation induced plasticity for the Q&P processed steel. Ultrahigh strength about 1300-1800MPa and tensile elongation about 22-16% were obtained after Q&P processing at specific conditions. This improved mechanical properties were related to the ductility contribution from phase transformation induced plasticity of the retained austenite and strength contribution from the hard martensitic matrix. At last it was turned out that the Q&P process is a promising way to produce ultrahigh strengthed steel with relative high ductility.

#### 11:10 AM Invited

**Microstructural Evolution during the Novel Quenching and Partitioning** (Q&P) Heat Treatment of Steel: Timothy D. Bigg<sup>1</sup>; *David Edmonds*<sup>1</sup>; <sup>1</sup>University of Leeds, Institute for Materials Research

The novel non-equilibrium heat treatment procedure known as Quenching and Partitioning (Q&P) may offer the prospect of higher strength steel products with enhanced formability based upon martensitic microstructures containing controlled quantities of carbon-enriched retained austenite. The Q&P process requires an interrupted quench and isothermal annealing (partitioning) step at intermediate temperatures, whereby untransformed austenite can be thermodynamically stabilised by carbon migration from supersaturated martensite regions. The concept is comparable to that producing carbide-free bainite, for example, in TRIP-assisted steel, although Q&P allows separation of the ferrite formation and austenite enrichment stages of the process. However, although the Q&P concept is readily understood, evolution of the microstructure during interrupted quenching and partitioning has been inferred indirectly from dilatometer studies and metallographic examination after final quenching to room temperature. Consequently, a model alloy was developed in which the sequential steps of heat treatment could be separated for direct inspection by conventional metallography, X-ray diffraction and neutron diffraction techniques.

#### 11:25 AM Keynote

Study of a Novel Ultra-High Strength Steel with Adequate Ductility and Toughness by Quenching–Partitioning–Tempering Process: Ying Wang<sup>1</sup>; Shu Zhou<sup>1</sup>; Zhenghong Guo<sup>1</sup>; Yonghua Rong<sup>1</sup>; <sup>1</sup>Shanghai Jiao Tong University

According to the design principle of microstructures for high strength steel and a new quenching-partitioning-tempering (Q-P-T) process recently proposed by Hsu, a microalloying Fe-Mn-Si base steel by the Q-P-T process has been designed. The results indicate that the Q-P-T steel exhibits ultra-high tensile strength combining with good ductility and toughness, and it is a new family of advanced high-strength steels. The microstructures of samples by different Q-P-T processes were characterized by means of optical microscopy, scanning electron microscopy, X-ray diffraction and transmission electron microscopy, and the relation between microstructures and mechanical properties was analyzed.

#### 11:45 AM

The Effect of Mn and Si on the Properties of Advanced High Strength Steels Processed by Quenching and Partitioning: Bohuslav Masek<sup>1</sup>; *Hana Jirkova*<sup>1</sup>; Danuse Klauberova<sup>1</sup>; Ludmila Kucerová<sup>1</sup>; Daniela Hauserova<sup>2</sup>; <sup>1</sup>University of West Bohemia in Pilsen, Research Centre of Forming Technology; <sup>2</sup>COMTES FHT a.s.

The concepts of the new types of materials are for the economical reasons focused mainly on low alloyed steels with a good combination of strength and ductility. Suitable heat and thermo-mechanical treatment play an important role for the utilization of these materials. Different alloying strategies are used to influence phase transformations during the processing. Quenching and partitioning process (Q&P Process) presents one of the methods of the heat treatment which can result in a high ultimate strength as well as a good ductility. However, these good properties can be obtained only if a sufficient amount of retained austenite is stabilized during the processing and carbide precipitation is avoided. The influence of different contents of manganese, silicon and chromium on a microstructural development and mechanical properties were experimentally tested. The above mentioned alloying elements were used to stabilize the retained austenite in the final microstructure and also to strengthen a solid solution. The ultimate strengths over 2000MPa with the ductility over 10% were obtained after the optimization of Q&P Process parameters.

#### 12:00 PM

Characterization of Microstructure and Mechanical Properties of a Nb-Microalloyed Steel after Quenching-Partitioning-Tempering Process: *Xiaodong Wang*<sup>1</sup>; Zhenghong Guo<sup>1</sup>; Yonghua Rong<sup>1</sup>; <sup>1</sup>Shanghai Jiao Tong University

A novel heat treatment manner, that is, quenching-partitioning-tempering (Q-P-T) process, is developed based on the quenching and partitioning process proposed by Speer et al. In order to display the merit of Q-P-T process, a medium carbon Nb-microalloyed steel is treated by Q-P-T 1-step process and Q-P-T 2-step process, as well as treated by the transformation induced plasticity (TRIP) heat treatment process and quenching and tempering process, respectively. The results show that the samples after Q-P-T process possess better mechanical properties than those after other heat treatment processes. The origins of the good mechanical properties are analyzed based on the phase and microstructure characterization using X-ray diffraction, scanning electron microscopy and transmission electron microscopy.

#### 12:15 PM

Microstructural Evolution Analysis of Medium Carbon Steels during the Quenching and Partitioning Process: Hongyan Li<sup>1</sup>; *Xuejun Jin*<sup>1</sup>; <sup>1</sup>Shanghai Jiao Tong University

The "Quenching and Partitioning" (Q&P) process is a novel heat treatment designed for processing new generation advanced high strength steels (AHSS) with substantial ductility. This treatment consists of full austenitization and subsequent rapid cooling to a quenching temperature in the range between martensite start temperature (Ms) and martensite finish temperature (Mf), followed by an isothermal treatment at the quenching temperature (one-step Q&P treatment) or above Ms temperature (two-step Q&P treatment). In this paper, characterization and evolution of complex microstructure for medium carbon steels during the Q&P process have been discussed in detail. Such steels have shown a complex multiphase microstructure consisted of fresh lath-martensite, fresh plate-martensite, transition carbide and/or cementite, isothermal martensite/lower bainite, and second twin-martensite after the one-step Q&P process (with the identical quenching and partitioning temperature). The morphology for the microstructure at room temperature after the two-step Q&P process (with different quenching and partitioning temperatures) demonstrated a little different.



The formation of different microstructure for these two processes and their correlation with the mechanical properties are discussed. Microstructure analysis is favorable for optimization of the Q&P process.

#### 12:30 PM

**The Isothermal Transformation of Low-Alloyed Low-C CMnSi Steels below MS**: *Donghwi Kim*<sup>1</sup>; John G. Speer<sup>2</sup>; B. C. De Cooman<sup>1</sup>; <sup>1</sup>Graduate Institute of Ferrous Technology; <sup>2</sup>ASPPRC

In the martensite-start (MS) and martensite-finish (Mf) temperature range, an isothermal transformation was observed in dilatometry when a low-alloyed low-carbon steel austenitized at a temperature above the A3 temperature was quenched and held at a quenching temperature below MS. The precise nature of this isothermal transformation in the MS-Mf temperature range is still unclear. The present contribution is a pioneering comprehensive comparison of the main difference between the isothermal transformation in the MS-Mf temperature range and athermal martensite or lower bainite transformation using FE-SEM, EBSD, internal friction (IF) and TEM microstructural analysis. The effect of transformation product on mechanical property was also considered. The observations revealed that the isothermal transformation product in the MS-Mf temperature range has its own characteristic microstructure with a Kurdjumov-Sachs (K-S) orientation relationship with the parent austenite and without carbide precipitation.

## Symposium C: Light Metals and Alloys: Powder Metallurgy of Light Alloys

Tuesday AM August 3, 2010 Room: 6 Location: Cairns Convention Centre

Session Chairs: Yongqing Zhao, Northwest Institute for Nonferrous Metal Research; Roger Lumley, CSIRO Light Metals Flagship

#### 8:30 AM Keynote

The Role of the Atmosphere and the Importance of the Formation of AlN on the Powder Processing of Aluminium: *Graham Schaffer*<sup>1</sup>; <sup>1</sup>The University of Queensland

Sintering of metals is typically conducted in a controlled atmosphere, which is used to sweep away volatile compounds, to reduce surface oxides or prevent further oxidation. However, the oxide on aluminium is thermodynamically very stable and cannot be reduced in conventional furnaces. Consequently, the role of the atmosphere in the sintering of aluminium is often seen as simply limiting further oxidation. However, it has recently emerged that nitrogen is not inert but plays an active role in the sintering and infiltration processing of aluminium powder. A key feature in the use of nitrogen is the formation of aluminium nitride which has now been directly observed and characterised to form in association with the sintering of aluminium under nitrogen. The aluminium nitride is believed to enhance pore filling during sintering and forms a rigid skeleton during infiltration of loose or selective laser sintered powder. This paper describes the various effects of nitrogen and the formation of aluminium nitride and presents a detailed study of the microstructure of aluminium nitride, which has a fine columnar structure and grows both into and outward from the aluminium surfaces.

#### 8:50 AM

Production of Ti-6Al-4V Strip by Direct Rolling of Blended Elemental Powder: Gersende Cantin<sup>1</sup>; Nigel Stone<sup>1</sup>; David Alexander<sup>2</sup>; Mark Gibson<sup>1</sup>; David Ritchie<sup>1</sup>; Robert Wilson<sup>1</sup>; Merchant Yousuff<sup>1</sup>; Raj Rajakumar<sup>3</sup>; Kevin Rogers<sup>3</sup>; <sup>1</sup>CSIRO Light Metals Flagship, Process Science and Engineering; <sup>2</sup>CSIRO Light Metals Flagship, Mathematical and Information Sciences; <sup>3</sup>CSIRO Light Metals Flagship

A significant research effort within the CSIRO Light Metal Flagship is aimed at the development of new processes for the manufacture of (semi-finished) titanium products based on a powder metallurgy approach. The main driver in considering alternative processing and consolidation techniques to conventional ingot metallurgy is improved techno-economics associated with a reduction in processing steps and increased productivity via rapid consolidation of parts. In this respect, CSIRO has developed a process to manufacture sheet products utilising direct powder rolling; the process consists of cold rolling the powder feedstock to a green sheet, which is then rapidly heated and hot rolled to consolidate the material completely. The work reported here has investigated the feasibility of fabricating Ti-6AI-4V sheet by a blended elemental powder metallurgy route. The development of microstructures occurring during the processing and heat treatment steps has been studied. The generic roles of some process, material and heat treatment variables on the tensile properties and chemical homogeneity of the final material have been assessed and are discussed in this paper.

#### 9:05 AM

Mechanical Properties of Oxide Dispersion Strengthened Pure Titanium Produced by Powder Metallurgy Method: *Tomohiro Yoshimura*<sup>1</sup>; Thotsaphon Thrirujirapaphong<sup>1</sup>; Hisashi Imai<sup>2</sup>; Katsuyoshi Kondoh<sup>2</sup>; <sup>1</sup>Osaka University; <sup>2</sup>JWRI, Osaka University

Pure titanium has good specific properties i.e. low density of 4.5g/cm<sup>3</sup>, extremely high resistance for corrosion and good elongation. However, its mechanical properties are not enough to employ as structural parts of mechanical products. Accordingly, titanium alloys are often applied for various fields of industries due to their high specific strength. However, the application is limited to highperformance products because of expensive their material cost and poor plastic formability. From a view point of cost reduction, pure titanium was employed as starting material. To improve the poor mechanical strength of pure titanium, the materials design by oxide dispersion strengthening (ODS) was employed. TiO, powder was used as dispersoids because of its easily obtainable and low material cost. Powder metallurgy (P/M) method was employed to disperse TiO, particles in titanium matrix. Pure titanium powder and TiO<sub>2</sub> particles are elementally mixed by conventional mixing process. The elemental mixture powders were consolidated by using spark plasma sintering (SPS) equipment to serve a high density billet. Subsequently, hot extrusion process was applied to the billet to prepare a full density rod specimen. The evaluation of mechanical properties at room temperature showed high tensile strength of 1040MPa and good elongation of 25% (1.5 mass% TiO<sub>2</sub>).

#### 9:20 AM

**Evaluation of Sintering Properties with Al-14Si-2.5Cu-0.5Mg Powders**: *Yong-Jin Kim*<sup>1</sup>; Haris Rudianto<sup>1</sup>; Sangsun Yang<sup>1</sup>; <sup>1</sup>Korea Institute of Materials Science

Aluminum-Silicon P/M alloys have drawn much attention in automobile and aerospace industries due to their good properties, such as wear resistance, high strength, good temperature resistance, and a low coefficient of thermal expansion. In this presentation, the densification behavior and mechanical properties of P/M Al-Si alloys(Al-14Si-2.5Cu-0.5Mg) will be reported. Al-Si powders were compacted with the compaction pressure of 300-700MPa to analyze influence compaction pressure on sintering density. Sintering of green compacted Al-Si powders was carried out the temperature of 550-600°C for 1 hour under the ultra high purity nitrogen gas atmosphere. Before reaching the sintering temperature, compacted powders were de-waxed at 400°C for 20 minutes. T6 treatment was used to enhance the mechanical properties of sintered body. Highest sintered density was achieved with the sintering temperature of 580°C and it was more than 98% of theoretical density. Various kinds of mechanical properties were measured, such as sintered density, hardness, microstructures and tensile strength depending on experimental conditions.

#### 9:35 AM

**Spark Plasma Sintering of Ti-4.5Al-3V-2Fe-2Mo (SP-700)**: *Genki Kikuchi*<sup>1</sup>; Yuya Takahashi<sup>1</sup>; Shota Fujino<sup>1</sup>; Hiroshi Izui<sup>1</sup>; <sup>1</sup>Nihon University

Ti alloy Ti-4.5Al-3V-2Fe-2Mo (SP-700) was fabricated by spark plasma sintering (SPS) in vacuum. The effects of sintering conditions on microstructures, relative density, and tensile properties of the compacts were investigated. The relative density increased with sintering temperature increase. The compact sintered at 900°C for 10 minutes had the highest tensile strength. It will be discussed the fatigue resistance of the compacts.

#### 9:50 AM

**Sintering Characteristics of Pre-Alloyed Ti-6Al-4V**: *Ya-Feng Yang*<sup>1</sup>; G. B. Schaffer<sup>1</sup>; Qian Ma<sup>1</sup>; <sup>1</sup>University of Queensland, ARC Centre of Excellence for Design in Light Metals

Powder metallurgy offers a promising route for the near net shape manufacturing of titanium components. Ti-6Al-4V is a workhorse alloy. However, it has proved difficult to sinter pre-alloyed Ti-6Al-4V powder. This work assesses the sintering characteristics of pre-alloyed Ti-6Al-4V in vacuum as a function of the powder size in the ranges of 45-75; 75-105 and 105-150 µm and sintering temperature ranging from 1200 to 1350°C, with a fixed compaction pressure of 500 MPa. Differential Scanning Calorimetry (DSC) analyses suggest a solid-state sintering process. Dilatometer curves revealed that the abrupt sintering shrinkages start from ~ 891°C irrespective of the powder size. The sintering of pre-alloyed Ti-6Al-4Al thus occurs primarily in the β region. The sintering activation energy data obtained suggest that the sintering process is controlled by the lattice selfdiffusion of titanium. The maximum relative density attained is 85% after sintering the fine powder (45-75 µm) compacts at 1350°C for 2 hr. Sintered samples made from the fine powder show typical lamellar microstructures while those made from the coarse powder (105-150 µm) show a distinctly different microstructure. Recommendations are made of enhancing the sinterability of pre-alloyed Ti-6Al-4V based on the experimental findings.


#### 10:05 AM

Effects of Impurity Elements on Green Strength of Titanium Powder Compacts: Ju Beom Lim<sup>1</sup>; Colleen Bettles<sup>1</sup>; Barry Muddle<sup>1</sup>; Nho-Kwang Park<sup>2</sup>; <sup>1</sup>Monash University; <sup>2</sup>Korea Institute of Materials Science

The green strength of a powder compact results from the mechanical interlocking of the irregularities on the particle surfaces. During compaction, particle rearrangement, plastic deformation and particularly surface deformation of powders occur. Titanium powder is susceptible to interstitial element contamination, which may lead to solid solution strengthening of the particles and/or the formation of non-metallic compounds on the surface. However, the influence of these various impurities, namely oxygen, nitrogen and hydrogen, on the green strength has not been investigated. This work investigates and quantitatively evaluates the factors influencing the green strength of the powder compacts, and test results were compared to that of a more conventional 3 point bending test. The substantial dependence of green strength on both the amount of impurity element in the core of the surface condition of the powder particles and the compaction pressure is demonstrated. The effect of the surface condition of the powder particles on green strength is also reported.

10:20 AM Tea Break

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### Symposium C: Light Metals and Alloys: R&D of Magnesium Alloys

Tuesday AM August 3, 2010 Room: C Location: Cairns Convention Centre

Session Chairs: Karl Kainer, GKSS Research Centre Geesthacht; Kwang Seon Shin, Seoul National University

#### 10:50 AM Keynote

**Thixomolded and Thermomechanically Processed Fine-Grained Mg Alloys:** *Raymond Decker*<sup>1</sup>; J. Huang<sup>1</sup>; S. Kulkarni<sup>1</sup>; J. Wayne Jones<sup>2</sup>; <sup>1</sup>Thixomat, Inc; <sup>2</sup>University of Michigan

Thixomolding of Mg alloys produces fine microstructure of about 5-10 micron alpha phase grain size, surrounded by divorced eutectic phases. During the period from 1995 to 2009, this process and microstructure has captured broad applications around the globe - in markets such as electronics (lap-tops, cameras and cell phones), autos, sports and hand tools. Properties so molded will be related to microstructure. Thermomechanical processing has been applied recently to the Thixomolded precursor to further refine the grain size and eutectic phases - providing yield strength above 300 MPa, fatigue strength of 150 MPa along with elongation of 10%. Alloys studied include AM60, AZ61L and Thixoblended alloys of higher Zn content. Microstructure is related to processing and properties.

#### 11:10 AM Keynote

#### Development of High Strength Magnesium Alloys by Thermomechanical Treatment: *Shigeharu Kamado*<sup>1</sup>; Tomoyuki Honma<sup>1</sup>; <sup>1</sup>Nagaoka University of Technology

Applications of extruded Mg alloys to structural components are still limited due to the fact that strengths of Mg alloys are generally insufficient compared with those obtained in steels and Al alloys. In addition, the compressive proof strengths (CPS) of conventional extruded Mg alloys are remarkably smaller than the tensile proof strengths (TPS). A combination of grain refining and precipitation hardening through thermomechanical treatment is very important in order to improve mechanical properties including the ratio of CPS to TPS representing the yielding anisotropy in wrought Mg alloys. In this study, effects of alloying elements, process conditions of extrusion and rolling and heat treatment on microstructures and mechanical properties of Mg-Al-Ca and Mg-Zn-Ca based alloys have been investigated in order to improve mechanical properties of wrought magnesium alloys by utilizing dynamic phenomena such as dynamic recrystallization assisted second phases, for example, crystallized compounds and dynamically-precipitated phases.

#### 11:30 AM Keynote

Development of Magnesium Alloys and Their Processing Technologies for Large Size Profiles: *Fusheng Pan*<sup>1</sup>; Dingfei Zhang<sup>1</sup>; Jian Peng<sup>1</sup>; Mingbo Yang<sup>2</sup>; Peidao Ding<sup>1</sup>; <sup>1</sup>Chongqing University; <sup>2</sup>Chongqing University of Science and Technology

Large size magnesium profiles may be widely used in transportation industry, especially in rail transportation, in which there is a huge market. However, it is very difficult for producing large size magnesium profiles due to large size and very complex structure. A lot of work on the development of magnesium alloys and their processing technologies for large size profiles has been carried out, with more attention to composition modification of wrought magnesium alloys and innovation of processing technologies. Some innovative processing technologies for large size magnesium profiles have been developed and the magnesium profiles with size of 360 mm have been successfully fabricated. The results showed that AZ alloys micro-alloyed by Sr,Ca and C and ZK alloys containing rare earth elements could be used to fabricate the large size profiles with complex structure. The homogenization parameters, extrusion temperature and extrusion speed were found to have very important influences on the fabrication of large size magnesium profiles. The effects of Sr addition and key processing parameters on microstructure and mechanical properties are discussed in detail.

#### 11:50 AM Keynote

Development of New Magnesium Alloys with Consideration of Castability and Formability in IMR: *Rongshi Chen*<sup>1</sup>; Enhou Han<sup>1</sup>; <sup>1</sup>Institute of Metal Research Chinese Academy of Sciences

A consideration of castability, heat treatability or formability, as well as mechanical properties, for development of cast alloys or wrought alloys are very important issues in magnesium alloys. The solidification pathways and phase equilibrium of Mg-Al-Ca and Mg-Zn-Al alloys has been investigated. Two-step solid solution treatment, which can completely dissolve the secondary phases into the matrix and spontaneously avoid the occurring of inclusive liquid, is designed based on thermodynamic calculations for Mg-Zn-Al alloys. The relationship between hot tearing and alloy compositions in Mg-Al-Ca were discussed in terms of strength of mushy zone, solidification pathways and feeding mechanisms, et. al. Metallurgical Criteria for thixoforming are summarized and the thixoformability of Mg-Al-Ca based alloys are evaluated using the thermodynamic calculations. The rolled Mg-1-2%Zn-1Gd sheets were found to exhibit an excellent ultimate elongation of nearly 36% and an uniform elongation greater than 15% with a very low planar anisotropy. These rolled sheets have a random basal texture and the basal pole is tilted by about 30° from the normal direction towards the transverse direction, these orientation is favorable for both basal slip and tensile twining because of a high Schmid factor and may imply excellent room temperature formability

#### 12:10 PM

A Technology of Twin Roll Casting and Differential Speed Rolling for Producing Magnesium Alloy Sheets: *Suk-Bong Kang*<sup>1</sup>; Jae Hyung Cho<sup>1</sup>; Xibing Gong<sup>2</sup>; Saiyi Li<sup>2</sup>; <sup>1</sup>Korea Institute of Materials Science; <sup>2</sup>South China University of Technology

Magnesium alloys are becoming increasingly attractive for potential use in a wide range of structural applications, particularly in automobile industry, because of their high specific strength, high specific stiffness, excellent damping capacity. However, high manufacturing cost and limited formability at room temperature are two major barriers to the wide industrial applications of Mg alloys. Twin-roll casting (TRC) process is a good way to manufacture magnesium alloy sheets economically due to near net shaping by combining the casting and hot rolling into a single step. It is well known that the conventional warm rolling of Mg alloys induces to form a strong basal texture, which results in a very limited formability near the room temperature and restricts the applications of Mg alloys. The differential speed rolling (DSR) can have more shear deformation and improve the formability of Mg alloys by grain refinement and weakening the basal texture. It is thus of great interest to perform the combined TRC and DSR process for Mg alloy sheets with more improved formability. The objective of this study was to explore the possibility of further enhancement of strength and formability of Mg alloys through the TRC and DSR technique.

#### 12:25 PM

Fabrication of High-Alloyed Magnesium Alloy Plate via Horizontal Continuous Casting Process: *Bong Sun You*<sup>1</sup>, Chang Dong Yim<sup>1</sup>; Young-Min Kim<sup>1</sup>; Sung Soo Park<sup>1</sup>; Ha Sik Kim<sup>1</sup>; <sup>1</sup>Korea Institute of Materials Science

High alloyed magnesium plate of 300 mm width and 40 mm thickness was produced by horizontal continuous casting, and its microstructure was analyzed on the surface and in the plate interior. The defect formation on the surface and subsurface, such as side cracks, buckling, pores and segregation, was very sensitive to the casting condition and processing parameters. Especially, a secondary cooling was effective in refining the microstructure and controlling the grain size distribution across the plate thickness, which means that the cooling rate and curvature of the solid/liquid interface in the mold changed. Since several defects caused side cracks during rolling and survived even after rolling, pretreatment of the as-cast plate such as homogenization and surface machining is necessary to produce sound thin sheet by hot rolling. Particularly, in the case of high alloyed magnesium alloys, it is hard to produce thin sheet without severe side crack by hot-rolling process. In order to improve rollability of high alloyed

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magnesium plate, therefore, grain refining technique of cast magnesium alloy was tried to be applied to horizontal continuous casting process. It could be found that fine and equi-axed grain structure considerably improves the rollability of high alloyed magnesium alloys.

#### 12:40 PM

# LCA Study of Rare Earth Metals for Magnesium Alloy Applications: Paul Koltun<sup>1</sup>; Rajah Tharumarajah<sup>1</sup>; <sup>1</sup>CSIRO

High strength properties combined with low density has made magnesium alloys a highly attractive structural material, in particular where weight savings is of concern. In air and ground transport these alloys are used as alternative material in place of heavier ferrous or aluminium alloys. In this respect, much research has been directed at developing and deploying superior magnesium alloys using Rare Earth Elements (REEs), an example is the Mg-RE (Ce, Y, Nd) alloys for drive train components.With the overall aim of ascertaining the environmental impact of employing REEs as alloying agents in producing superior Mg-RE alloys, it is paramount that a fundamental understanding of the environmental burden imparted by the extraction and production of REEs be determined. This study reports on such an assessment of REEs by conducting a detailed life cycle assessment (LCA) study of the environmental impact from mining to production of REEs.

## Symposium C: Light Metals and Alloys: Titanium Alloys III

Tuesday AMRoom: CAugust 3, 2010Location: Cairns Convention Centre

Session Chairs: Nhokwang Park, Korea Institute of Materials Science; Alexandra Shekhter, DSTO

#### 8:30 AM

#### Recent Developments in Mechanisms and the Kinetics of Deformation Twinning in Titanium Alloys: Zane Wyatt<sup>1</sup>; Sreeramamurthy Ankem<sup>1</sup>; <sup>1</sup>University of Maryland

Within the past decade, it has been shown that twinning in alpha, beta, and alpha+beta titanium alloys can occur at speeds much lower than the speed of sound by many orders of magnitude. This is related to the twinning deformation mechanisms controlled by the diffusion of oxygen as compared to simply a shear process. Very recent developments, such as a strain-rate effect on twinning, supports a recent hypothesis that the twinning in these materials is controlled by a slow diffusion process, resulting in time-dependent twinning. These recent developments, along with the ramifications of the findings will be outlined in this presentation.

#### 8:45 AM

Characterisation of Cold Spray Titanium Coatings: *Stefan Gulizia*<sup>1</sup>; Mahnaz Jahedi<sup>1</sup>; Ciaxian Tang<sup>1</sup>; <sup>1</sup>CSIRO

Cold Spray is a solid state deposition process utilising a converging-diverging de Laval nozzle to accelerate small powder particles to supersonic velocities in a gas stream. Particles bond to the substrate and to each other on impact to form strong bonds. In this study, we examine the microstructure and mechanical properties of cp-titanium structures directly fabricated using Cold spray technique in a nitrogen gas stream. In general, it was found that the tensile strengths could be achieved comparable to wrought titanium, but the maximum elongation was less than expected. Chemical and microstructural analysis indicated that the process does not add oxygen to the material, suggesting that the use of higher purity powder and further process optimisation should lead to significant improvements in ductility.

#### 9:00 AM

#### Microstructural Evolution in Chips during Machining of Commercially Pure (Grade 2) Titanium: *Shoujin Sun*<sup>1</sup>; Milan Brandt<sup>1</sup>; Wei Qian Song<sup>1</sup>; Matthew Dargusch<sup>2</sup>; <sup>1</sup>Swinburne University of Technology; <sup>2</sup>University of Queensland

Development of microstructure in chips during machining of alpha titanium at different cutting speeds has been investigated. The morphology of the chip changes from the continuous chip to the irregular segmented and the regular segmented chip with increasing cutting speed. The deformation in continuous and segmented chips is characterized as continuous shear and localized shear respectively. Deformation twining was observed inside the segment adjacent to the shear band. These deformation twins are responsible for the hardening near the shear bands. The twin density is a function of the cutting speed in the speed region governing the transition from the irregular segmented chip to the regular segmented chip.

#### 9:15 AM

Solid State Recycling of Ti Machining Chips by Back Pressure Equal Channel Angular Pressing (BP-ECAP): *Peng Luo*<sup>1</sup>; Wei Xu<sup>1</sup>; S Palanisamy<sup>2</sup>; MS Dargusch<sup>2</sup>; K Xia<sup>1</sup>; <sup>1</sup>University of Melbourne; <sup>2</sup>University of Queensland

With a view to reducing waste from Ti manufacturing and to adding value, severe plastic deformation (SPD) consolidation has been developed to recycle CP-Ti and Ti-6AI-4V machining chips into fully dense bulk material. As one of the most widely used SPD techniques, equal channel angular pressing (ECAP) was combined with a back pressure to realize upgrade solid state recycling, leading to superior mechanical properties due to refinement of microstructure as well as potentially low energy consumption by virtue of avoidance of remelting and casting. The recycling was conducted at temperatures between 400 and 600°C with the application of a back pressure from 50 to 200 MPa. Fully dense bulk samples were obtained after a single pass or multiple passes of ECAP. The strengths of the recycled materials are comparable or higher than those of the commercial CP-Ti and Ti-6AI-4V alloy, respectively. It is demonstrated that BP-ECAP is a promising method for recycling and value-adding to Ti machining chips.

#### 9:30 AM

**Modelling the High Temperature Deformation of Ti-6Al-4V**: *Jikang Zhong*<sup>1</sup>; Matthew Dargusch<sup>2</sup>; Chris Davies<sup>1</sup>; <sup>1</sup>CAST, Department of Materials Engineering, Monash University, Australia; <sup>2</sup>CAST and DMTC, School of Mechanical and Mining Engineering, The University of Queensland, Australia

lue. AM

The hot deformation behaviour of alpha / beta Ti-6Al-4V alloy was investigated at various temperatures and strain rates by means of compression and torsion tests. A high logging rate thermocouple was employed to measure the sample temperature during the experiments. As expected, the peak flow stress increased with increasing strain rate and decreased as the initial sample temperature was increased. The different flow behaviours observed are discussed in terms of the volume fraction of each phase. The dual phase Ti-6Al-4V alloy was assumed to be a composite material containing a soft phase and a hard phase. By taking into consideration the phase-to-phase interaction and volume fraction change with temperature, constitutive models (iso-strain, iso-stress and self-consistent) were proposed to simulate the deformation behaviours. By application of the rule of mixtures the simulated stress-strain curves showed good agreement with the experiment results.

#### 9:45 AM

# Varying Behavior of Shot Peened TC4-DT Titanium Alloy: Xiaonan Mao<sup>1</sup>; <sup>1</sup>NIN

Shot peening is an effective surface treatment method, which can improve the fatigue properties of metallic materials. In the present paper, the residual compressive stress field induced in TC4-DT titanium alloy after different shot peening conditions and thermal relaxation behavior were discussed. The process can cause residual compressive stress on the material surface compared to unpeened materials. The subsurface residual stress distributions of original and shot peened specimens were measured by an X-ray diffraction method. It was demonstrated that the residual compressive stress field induced by shot peening. Surface roughness was also increased with increasing shot peening intensity. Thermal relaxation behavior of residual stress was studied at 150, 300°C with different ageing times. Temperature and time influences on the residual stress relaxation during ageing are controlled by thermally activated processes. Thermal residual stress relaxation process was discussed and analyzed by applying the Zener-Wert-Avrami function.

#### 10:00 AM

Improved Properties of Boron-Modified Titanium Alloys: Masuo Hagiwara<sup>1</sup>; Tomoyuki Kitaura<sup>1</sup>; Yoshinori Ono<sup>2</sup>; Tetumi Yuri<sup>2</sup>; Toshio Ogata<sup>2</sup>; <sup>1</sup>Kyushu Institute of Technology; <sup>2</sup>National Institute for Material Science

The effect of minor boron (B) addition (less than 0.12 wt.% B) on the microstructures and mechanical properties of titanium alloys such as Ti-6Al-4V, Ti-6.8Mo-4.5Fe-1.5Al and Ti-22Al-22Nb-2Mo-1Fe was investigated. These B-modified alloys showed typical colony microstructures when they were slowly cooled from the high temperature single phase region. Grain size was reduced from 1 mm to 80  $\mu$ m by the addition of 0.05 wt.% B due to the pinning effect of TiB against grain growth, and accordingly the colony size within the grains was greatly reduced. The tensile elongation of these B-modified alloys at room and higher temperatures increased remarkably compared to alloys without B. These alloys also showed improved room temperature high cycle fatigue (HCF) strength. Fatigue crack initiated neither from the TiB/matrix interface nor from the TiB but rather from the matrix itself. The reduced colony size was thought to be responsible for the significantly higher HCF strength exhibited by the B-modified alloys.



### 10:15 AM

Effect of Pore Size on Mechanical Properties of Titanium Foams: Yang An<sup>1</sup>; Chunhui Yang<sup>1</sup>; Peter Hodgson<sup>1</sup>; Cuie Wen<sup>1</sup>; <sup>1</sup>Deakin University

Recently metal foams are becoming popular due to their excellent physical and mechanical properties. However the size effects of internal pores on mechanical properties of such materials are still ambiguous in literatures. On the one hand, Gibson et al. claimed that the collapse stress of a porous metal is not affected by the pore size. Controversially, Miyoshi et al. found that a porous material with a small pore size shows higher collapse stress. In the study, to investigate the pore size effects on mechanical properties, both experimental work and numerical modeling are performed. Titanium foams with different pore sizes are fabricated through powder metallurgy. Then the cylindrical samples are manufactured and the foam samples are tested through compression tests to determine their mechanical properties. To investigate foam microstructures, the SEM are used to determine the relationship among pore size, pore distribution and microstructural geometry of pores, e.g., the ratio of the length to the width of pores. Finally an attempt of applying finite element modeling is carried out. We found different deformation modes of cell walls during compression could be generated and the mechanical properties of titanium foams depend on pore microstructures greatly.

#### 10:30 AM

Study of Thermal Behavior in a Kroll Reactor for the Optimization of Ti Sponge Production: *Hyun-Na Bae*<sup>1</sup>; Seon-Hyo Kim<sup>1</sup>; Go-Gi Lee<sup>2</sup>; Sung-Koo Jo<sup>2</sup>; Jae-Young Jung<sup>2</sup>; <sup>1</sup>Pohang University of Science and Technology; <sup>2</sup>Research Institute of Industrial Science and Technology

The titanium reduction from titanium tetrachloride  $(\text{TiCl}_4)$  by molten magnesium pool, called Kroll process, is regarded as a well-known process for the commercial-scale production of sponge titanium. Purified titanium tetrachloride vapor reduced by magnesium forms sponge titanium with generating excessive heat. The heat transfer phenomena in a Kroll reactor should be thoroughly understood for productivity and quality enhancement. In this work, a computational modeling method to describe the thermal behavior in the TiCl4 reduction reactor was investigated and validated with the measured temperature distribution in a 500kg titanium sponge-capacity pilot-scale reactor in terms of various reduction ratios. The approach model for heat flow phenomenon in a reduction reactor could be utilized as a tool to predict the influence of operating process parameters on the optimization of Kroll process.

10:45 AM Tea Break

### Symposium C: Light Metals and Alloys: Welding and Joining of Light Alloys

Tuesday AMRoom: 6August 3, 2010Location: Cairns Convention Centre

Session Chair: Sri Lathabai, CSIRO Process Science and Engineering

#### 10:50 AM Keynote

Microstructure and Joint Strength of Similar and Dissimilar Lap Joints Fabricated by Several Advanced Solid-State Welding Methods: *Shinji Kumai*<sup>1</sup>; Mitsuhiro Watanabe<sup>1</sup>; Keyan Feng<sup>1</sup>; <sup>1</sup>Tokyo Institute of Technology

Both similar- and dissimilar metal joints, which are difficult to be welded by using ordinary fusion welding methods, were successfully obtained by using several advanced high speed solid-state joining methods. (1) Al/Al, Al/Fe, Al/ Cu and Al/Mg lap joints were magnetic pulse welded by means of mutual highspeed oblique collision of metal sheets at a high speed of about 500m/s. (2) 2xxx aluminum alloy pins were stud-welded to 5xxx alloy aluminum sheets and several kinds of plated steel sheets at a high speed by using a specially designed discharge circuit. The welding was achieved within a few milliseconds without producing any weld marks on the back surface of the plate. (3) 6022 aluminum alloy sheets were friction stir spot welded to steel sheets and various kinds of galvanized and aluminum-plated steel sheets. The welding was achieved within a few seconds. For those joints, joint strength and characteristic joint interface morphology were investigated.

#### 11:10 AM

Characterization of the Mechanical Behaviour of Both Fusion Zone and Base Metal of Electron Beam Welded TA6V Titanium Alloy: Julitte Huez<sup>1</sup>; Christophe Buirette<sup>1</sup>; Eric Andrieu<sup>1</sup>; Sylvain Audion<sup>2</sup>; Simon Perusin<sup>2</sup>; <sup>1</sup>Université de Toulouse; <sup>2</sup>Airbus Operations SAS

The fusion zone of an electron beam welded TA6V titanium alloy presents a alpha prime martensitic structure which leads to a loss of mechanical properties. Starting from two processing routes for the alloy (1) a beta processing followed by the weld, (2) an alpha+beta processing followed by welding and a post weld heat treatment (PWHT), the microstructure can be adjusted to find the best combination of strength, fatigue properties and impact toughness. The present work investigates the tensile and impact properties and the damage process of both base metal and fusion zone in regards to the microstructure and to the heat treatment parameters. The first results pointed out that a supertransus PWHT on the alpha + beta weld allows to homogenise the entire microstructure and enhance the impact toughness simultaneously in the fusion zone and the base metal. The thickness of the alpha platelets and the size of the alpha colonies seem to be the most important microstructural features associated to the crack path morphology. In order to progress in the comprehension of the implied damage mechanisms, a particular attention is given to the specimen orientation and crack propagation in relation to the texture of the alloy.

#### 11:25 AM

Welding Interface in Magnetic Pulse Welded Joints: Mitsuhiro Watanabe<sup>1</sup>; Shinji Kumai<sup>1</sup>; <sup>1</sup>Tokyo Institute of Technology

Magnetic pulse welding was applied to the lap joining of similar (Al/Al and Cu/Cu) and dissimilar materials (Al/Steel, Al/Cu, Al/Ni and Al/Metallic glass). The welding was achieved within 10 microseconds with a negligible temperature increase. The welding interface exhibited a characteristic wavy morphology, which was similar to that of the explosive welding. Wavelength and amplitude of the interfacial wave were not constant through the interface. In the Al/Fe, Al/Cu, Al/Ni, Al/Metallic glass joints, an intermediate layer which consists of fine crystal grains and amorphous phase was produced along the wavy interface. In order to investigate the formation manner of such a characteristic interfacial morphology, insitu observation of the magnetic pulse welding process was performed using a high speed video camera. A series of images obtained by the in-situ observation revealed that collision angle between the plates gradually increased during the welding. Such a characteristic oblique collision behavior between the plates is considered to result in the wavy interface with fluctuated wavelength and amplitude.

#### 11:40 AM

Joint Interface Morphology of Friction Stir Spot Welded Aluminum Alloy Sheets and Plated Steel Sheets: *Keyan Feng*<sup>1</sup>; Mitsuhiro Watanabe<sup>1</sup>; Shinji Kumai<sup>1</sup>; <sup>1</sup>Tokyo Institute of Technology

Friction stir spot welding (FSSW) was applied for lap joining of aluminum alloy sheets and steel sheets. A 1.2 mm-thick non-plated carbon steel sheet and plated steel sheets with zinc alloy (ZAM), pure zinc (GI), zinc alloy including Fe (GA) and Al-Si alloy (AS) were prepared. The melting temperature of the plated layer is 330, 420, 880 and 640°C, respectively. A 1.1 mm-thick 6022 aluminum alloy sheet was overlapped on the steel sheet. A rotating tool was inserted from the aluminum alloy sheet side and the probe tip was kept at the position of 0.2 mm above the lapped interface for 3 seconds. For ZAM and GI, original plated layers were removed from the interface and intermediate layers were formed at the joint interface. This is because melting temperature of the plated layer was lower than the interface temperature under the rotating probe tip during the FSSW. While, the partial original plated layer remained after welding and additional layer formed at the plated layer, which was formed at the original Al-Si alloy plated steel surface remained.

#### 11:55 AM

**3D** Transient Thermal Modelling and Experimental Validation of the Temperature Distribution during Laser Heating of Ti<sub>6</sub>Al<sub>4</sub>V Alloy: *Jihong* (*Nancy*) Yang<sup>1</sup>; Shoujin Sun<sup>1</sup>; Milan Brandt<sup>1</sup>; Wenyi Yan<sup>2</sup>; <sup>1</sup>CAST Cooperation Research Centre, Swinburne University of Technology; <sup>2</sup>Monash University

Titanium alloys have been widely used in the aerospace, biomedical and automotive industries because of their high strength-to-weight ratio and superior corrosion resistance at room and elevated temperature. However, titanium alloys are difficult to machine due to their high strength, low thermal conductivity and high chemical reactivity. This means that conventional machining of titanium alloys is a low productivity process with high materials running costs. Laser assisted machining (LAM) offers ability to machine difficult to cut materials more efficiently and economically. Normally, measurement of temperature variation during laser surface treatment is not possible due to the high temperature variation rate. The aim of this work is to develop 3D transient finite element model to predict 3D temperature distribution in Ti<sub>6</sub>Al<sub>4</sub>V workpiece, optimize the laser parameters and tool position during LAM process. Also the experimental observations have been conducted to determine the emissivity, absorptivity and temperature distribution on workpiece material. The simulation results are compared with the results produced by experimental work, showing good agreement. The influence of laser parameters on the temperature distributions in the Ti<sub>x</sub>Al<sub>4</sub>V alloy workpiece was also investigated, which provides important information to optimize and improve the LAM technique.



### 12:10 PM

A Study on Optimum Welding Conditions for Friction Stir Welded Aluminum Plates: Byung Chul Kim<sup>1</sup>; Tae Jin Yoon<sup>1</sup>; *Bong Gyu Park*<sup>1</sup>; Jung Yoon Kang<sup>1</sup>; <sup>1</sup>Busan National University

Friction stir welding is an advanced technology to join aluminum plates which is known to be difficult to join them by a fusion welding technique. Using extruded AA6005 alumimun plates which have no uniform thickness, authors tried several friction stir welding trials and explored to find optimum welding conditions. At first, varying parameters were welding speed, and load and revolution speed of tool. Next, the length of the tool was changed and finally, tool shape was changed. Finishing each trial, macro-structure of the weld was observed using an optical microscope and SEM and the existance of defects such as lack of penetration and tool wear was analysed.

#### 12:25 PM

# Study on Tensile Property and Fracture of Welded Joint of Ti-6.5Al-2Zr-1Mo-1V Alloy by Hydrogen Processing: *Qing Wang*<sup>1</sup>; <sup>1</sup>Harbin Institute of Technology

The microstructural evolution of TIG welded joint in Ti-6.5Al-2Zr-1Mo-1V alloy treated by thermo hydrogen processing was investigated by optical microscopy, transmission electron microscopy and scanning electronic microscopy, and the mechanical properties of welded joints were tested and analysed. The results showed that d-hydride formed after hydrogenising at 700°C for 3 hours. A lot of rhombic martensite phase a" generated in the area of weld joint. Metastable B(H) phase and a" phase wre gradually decomposed to a and d phases during aging. Hydrides was dissolved, the hydrogen was removed from the alloy and the recrystallization accured in the process of annealing in vacuum. After hydrogen treatment, the refinement effect of grains in the fusion area was very obviously, that is, coarse columnar grains changed to finer equiaxed grains. Non-equilibrium phase had reduced in grain. After hydrogenization at 700°C for 3 hours, eutectoid at 300°C for 8 hours and annealing at 750°C for 8 hours in vacuum, the tensile strength of welded samples increased by 25.7%, and the elongation increased by 37.6% compared with welded samples without hydrogen treatment. After hydrogen treatment, the surface morphology of tensile fracture was intercrystalline fracture.

#### 12:40 PM

**Durability of Aluminum Alloy/Rubber Joints in Corrosive Environment:** *Masatoshi Mori*<sup>1</sup>; Nguyen The<sup>2</sup>; Goroh Itoh<sup>3</sup>; Nobuhide Itoh<sup>3</sup>; Yasuhiro Shimada<sup>4</sup>; <sup>1</sup>Graduate Student, School of Science and Engineering, Ibaraki University; <sup>2</sup>Undergraduate Student, Department of Mechanical Engineering, Ibaraki University; <sup>3</sup>Department of Mechanical Engineering, Ibaraki University; <sup>4</sup>Yamashita Rubber Co., Ltd.

In automobiles, a suspension bush, a set of aluminum parts connected to each other via rubber, is mounted into the suspension members to link the body to the wheels, and realizes hard and soft mounts contributing both to the stabilization during high speed driving and to absorbing the shock from the road. However, decohesion of the aluminum part from the rubber occurs occasionally, arising from the interface corrosion of aluminum, which deteriorates the performance of the vehicle directly. In this study, effects of alloy composition and processing route on this kind of corrosion will be investigated. Also, development of a new corrosion test that shortens the time for the test will be attempted.

# Symposium D: Bulk Metallic Glasses and Nanomaterials: Design and Production of BMGs

Tuesday AM	Room: 4
August 3, 2010	Location: Cairns Convention Centre

Session Chair: Eun Soo Park, Seoul National University

#### 8:30 AM Keynote

Design of Metallic Glass Matrix Composites Using Phase Separation Phenomena: Do Hyang Kim<sup>1</sup>; <sup>1</sup>Yonsei University

Recent research results show that addition of alloying element having positive enthalpy of mixing with constituent elements can induce the two-glass phase separation in glass forming alloys. In the present study, results on the phase separation in (Ti, Zr)-Y-Al-Co, Cu-(Zr, Hf)-(Y, Gd)-Al, Gd-(Zr, Ti)-Al-Co alloy systems will be presented, showing that phase separating metallic glass system can offer a unique opportunity for designing composites with hierarchical microstructure with different length scales. In particular, we report novel core shell and hierarchical structures of spherical glassy droplets, resulting from critical wetting behavior and limited diffusion. We will also show that compositional inhomogeneity resulting from the addition of positive heat of mixing between binary pairs can play a role in enhancing plasticity of the bulk metallic glasses. For example, Ni-Nb-Zr Cu-Zr-Be ternary bulk metallic glasses show enhanced plasticity in the limited composition range, indicating that addition of alloying element having positive enthalpy of mixing with the constituent elements can lead to the enhancement of plasticity.

#### 8:50 AM

Al-Ni-Zr Alloy Phases and Metallic Glasses Understood via Cluster Formulas: *Chuang Dong*<sup>1</sup>; Jixiang Chen<sup>1</sup>; Qing Wang<sup>1</sup>; Yingmin Wang<sup>1</sup>; Jianbing Qiang<sup>1</sup>; <sup>1</sup>Dalian University of Technology

Via examples of Al-Ni-Zr alloy phases, simple and universal composition formulas for alloy phases are developed using 1st-neighbor coordination polyhedra plus their connections. The resultant cluster formulas [cluster](glue atom)x, similar to molecular formulas for chemicals, contain key structure and composition information of the alloy phases. By avoiding cluster sharings, good glass-forming compositions are reached using periodicity-incompatible clusters. For instance a bulk metallic glass  $Al_{20}Ni_{20}Zr_{60}$  is expressed with  $[Ni_3Al_3Zr_6]Zr_3 = Al_3Ni_3Zr_9$ .

#### 9:05 AM

Amorphous Magnesium Sheet Produced by Twin Roll Casting: Daniel East<sup>1</sup>; Michael Kellam<sup>1</sup>; Mark Gibson<sup>1</sup>; Aaron Seeber<sup>1</sup>; Daniel Liang<sup>1</sup>; Jian-Feng Nie<sup>2</sup>; <sup>1</sup>CSIRO; <sup>2</sup>Monash University

Twin roll strip casting was used to produce bulk amorphous sheets of Mg<sub>60</sub>Cu<sub>29</sub>Gd<sub>11</sub>. To produce quality amorphous sheet material, the casting speed needs to be controlled in order to control the exit temperature and thickness of the sheet at the crystallisation temperature (Tx). At the point of glass transition the material should be below the critical casting thickness for the alloy as measured by the wedge casting technique. For casting speeds higher than the optimum range the alloy will be insufficiently cooled, and strip exiting the caster will be crystalline in nature. However, casting at slower than optimum roll speeds leads to the exit temperature of the strip falling below the lower limit of the supercooled liquid region, which causes the strip to shatter due to the extremely brittle nature associated with these bulk metallic glasses. Therefore, if a suitable casting speed is selected for the alloy system such that the exit temperature of the strip is within the supercooled liquid region, then a malleable amorphous sheet with no surface defects is produced. This work shows that twin roll strip casting is a viable process for producing magnesium based bulk amorphous or composite structures as a continuous production method.

#### 9:20 AM

**Design of In-situ Ductile Ti-Based Bulk Glassy Matrix Composites**: *Jin Man Park*<sup>1</sup>; Norbert Mattern<sup>1</sup>; Jurgen Eckert<sup>1</sup>; Ka Ram Lim<sup>2</sup>; Do Hyang Kim<sup>2</sup>; <sup>1</sup>Leibniz Institute for Solid State and Materials Research Dresden: <sup>2</sup>Yonsei University

Recently, highly toughened glassy matrix composites with different length scale heterogeneity have been developed in Zr-, Ti-, La-based glassy alloys. These heterostructured composites can improve the macroscopic plasticity by controlling the shear band formation and preventing the rapid propagation of major shear bands. In this study, we developed micrometer size secondary phase reinforced composites including the several types of constituent phases, i.e. quasicrystalline (icosahedral), crystalline (α-Ti, β-Ti, Ti,Cu) phases in Ti-Zr-Be-Cu-Ni-(Nb, Ta, V) system. Although in-situ composites have been successfully formed by optimizing the alloy composition and cooling rate, plasticity does not always occur. Only if, size, distribution, and elastic constants of the dendrites were properly controlled, i.e. dendrites with lower shear modulus than glassy matrix and homogeneously distributed in the glassy matrix, they show large plasticity. By controlling the microstructural length scale and tuning the intrinsic elastic constants of constituent phases a Ti-based bulk glassy matrix composites with high yield strength of ~1.7 GPa and large plasticity of ~25% have been achieved.

#### 9:35 AM

**Effects of Ni Addition on Mg<sub>65</sub>Zn<sub>30</sub>Ca<sub>5</sub> Bulk Metallic Glass**: *Dorna Heidari*<sup>1</sup>; Ming Jen Tan<sup>1</sup>; Anders Eric Wollmar Jarfors<sup>2</sup>; <sup>1</sup>Nanyang Technological University; <sup>2</sup>Singapore Institute of Manufacturing and Technology (SIMTech)

Mg-based metallic glasses (BMGs) have high strength, low density, and corrosive and wear resistance compared to conventional Mg alloys. Research shows Mg-based metallic glasses have very good mechanical properties, low mass density, and so can be used as low-density structural materials. Mg<sub>65</sub>Zn<sub>30</sub>Ca<sub>5</sub> has been found to have a very good high glass forming ability (GFA), because at this composition the difference between the glass transition temperature (T<sub>g</sub>) and the first crystallization temperature (T<sub>x1</sub>) is more than that in other compositions. The present paper reports on a study into the effects of Ni as an additional element on this composition by partial substitution of Zn by Ni (Mg<sub>65</sub>Zn<sub>30-x</sub>Ca<sub>5</sub>Ni<sub>x</sub>, where x= 0.5, 1, 3, 5, 10). The samples are fabricated by copper mold casting method in shape of cylindrical rods. The formation of the amorphous phase in all solidified



as-cast samples is determined using XRD (Cu K $\alpha$  radiation). Differential scanning calorimetry (DSC) at a constant heating rate of 20 K/min is done to determine glass transition (T<sub>g</sub>), crystallization (T<sub>x</sub>), melting (T<sub>m</sub>) and liquidus (T<sub>1</sub>) temperatures with the aim to calculate usual GFA indicators:  $\Delta T = T_x - T_g$  and  $\gamma = T_x'(T_g + T_1)$ .

### 9:50 AM

Formation of Amorphous Fe-Cr-P-C Alloy Coating Films by a Newly Developed Thermal Spraying Technique: *Masahiro Komaki*<sup>1</sup>; Tsunehiro Mimura<sup>1</sup>; Yuji Kusumoto<sup>1</sup>; Ryurou Kurahashi<sup>1</sup>; Masahisa Kouzaki<sup>1</sup>; Tohru Yamasaki<sup>2</sup>; <sup>1</sup>Nakayama Steel Works Ltd.; <sup>2</sup>University of Hyogo

# lue. AM

Thermal spraying method is a superior technique for producing amorphous alloy coating films with large area on the various industrial materials. However, Fe-based amorphous films having high melting temperatures of about 1500 K have never been produced by previous methods. In the present study, formation of some amorphous Fe-Cr-P-C coating films having high hardness and high corrosion resistance have been demonstrated by newly developed thermal spraying techniques. In order to control the temperatures of powder particles in the flame spray and substrate, newly developed cylindrical nozzle with external cooling nitrogen gas was fitted to the front end of the thermal spraying gun. Cooling rates of the spraying samples on the SUS316L substrates were estimated to attain about 106 K/s by measuring the temperature gradient of the spraying flame. Structure of the coating films was observed by using SEM, XRD and TEM. Corrosion-resistance of the films was evaluated by the immersion test using various corrosive liquids. X-ray diffraction patterns of the sprayed  $Fe_{70}Cr_{10}P_{13}C_7$  films with various external cooling gas velocities between 20 m/s and 40 m/s exhibited entire amorphous structure without oxides and/or unmelted particles. More detailed results about the corrosion resistance will be presented at the conference.

#### 10:05 AM

Synthesis and Mechanical Properties of New Cu-Based Cu-Zr-Al Glassy Alloys with Critical Diameters up to Centimeter Order: *Wei Zhang*<sup>1</sup>; Bingwen Zhou<sup>2</sup>; Xingguo Zhang<sup>3</sup>; Hisamicni Kimura<sup>1</sup>; Akihisa Inoue<sup>1</sup>; <sup>1</sup>Institute for Materials Research, Tohoku University; <sup>2</sup>Graduate school, School of Materials Science and Engineering, Dalian University of Technology; <sup>3</sup>School of Materials Science and Engineering, Dalian University of Technology

It has been reported that Cu-based Cu-Zr-Al bulk glassy alloys (BGAs) exhibit excellent mechanical properties, large supercooled liquid region and low materials cost. However, their glass-forming ability (GFA) were not so high as compared with the Zr-based ternary alloys, the critical diameters (dc) for formation of a single glassy phase were below 5 mm. For broader engineering applications as well as scientific studies on the glassy alloys, it is important to develop the Cu-based BGAs with high GFA through the optimization of the alloy composition. Recently, we systematically investigated the thermal stability, melting behavior, and GFA of Cu-based Cu-Zr-Al glassy alloys. The fully glassy samples with dc of over 10 mm were obtained by copper mold casting. The BGAs showed high reduced glass transition temperature of over 0.565 and large supercooled liquid region of  $65 \sim 85$ K. The mechanical tests on these alloys show high compressive fracture strength of over 2000 MPa. The effect of the addition elements on the thermal stability, GFA and mechanical properties of the Cu-based ternary BGAs was also investigated.

#### 10:20 AM Tea Break

Symposium D: Bulk Metallic Glasses and Nanomaterials: BMGs - Properties and Processing I

Tuesday AM August 3, 2010 Room: 4 Location: Cairns Convention Centre

Session Chair: Chuang Dong, Dalian University of Technology

#### 10:50 AM Keynote

Investigation of Shear Band Evolution in Metallic Glasses: Eun Soo Park<sup>1</sup>; Frans Spaepen<sup>2</sup>; <sup>1</sup>Seoul National University; <sup>2</sup>Harvard University

The metallic glasses, a fully non-periodic structure, have strengths close to Frenkel's theoretical limit of a tenth of the shear modulus, which makes these glasses candidates for structural application. However, their Achilles heel is the deformation-induced shear bands that render these materials mechanically unstable at high stress. Thus, understanding how shear bands form and propagate in metallic glasses is essential for the eventual use of these materials in structural applications. Although it is generally known that the bands behave like fastmoving shear cracks, very little quantitative information has been collected on their dynamics. Furthermore, the structure and density of these shear bands has been studied mostly after the fact. We have developed observational techniques for studying the nucleation and propagation of shear bands in bend test on thin ribbons. Effects of strain rate and alloy composition will be reported. As a result, nucleation and density of shear bands during bend test can be clearly observed. In particular, it can be understood that the structural heterogeneity increase the nucleation sites of shear bands. Hence, the instability of ribbons evaluated from dynamic behaivor can be closely related to the mechanical properties in bulk metallic glass-forming alloys.

#### 11:10 AM

**Devitrification Studies of Mg<sub>60</sub>Cu<sub>29</sub>Gd<sub>11</sub> Bulk Metallic Glass**: Daniel East<sup>1</sup>; Mark Reid<sup>2</sup>; Mark Gibson<sup>1</sup>; Rian Dippenaar<sup>2</sup>; *Robert O'Donnell*<sup>1</sup>; <sup>1</sup>CSIRO; <sup>2</sup>University of Wollongong

The application of moderate cooling rates to metal alloys of certain composition can generate metals that exhibit an amorphous microstructure on a bulk scale. This phenomenon is related to the avoidance of the nucleation of the competing crystalline phases associated with the alloy during solidification. This work describes the devitrification behaviour of the bulk glass forming  $Mg_{60}Cu_{29}Gd_{11}$  system through the use of a number of analytical techniques including DSC, laser confocal microscopy, XRD and conventional metallography. Attention is drawn to the correlation between the more common analytical techniques and the observation of phase transformations on the surface of the metal, evident using a laser confocal microscope fitted with a heating stage.

#### 11:25 AM

Effect of Residual Stress on Mechanical Property of Monolithic Bulk Metallic Glass: *Min Ha Lee*<sup>1</sup>; Joong-Hwan Jun<sup>1</sup>; Jürgen Eckert<sup>2</sup>; <sup>1</sup>Korea Institute of Industrial Technology; <sup>2</sup>IFW Dresden

Mechanical surface treatments such as deep rolling are known to affect the strength and toughness of metallic glass due to the residual stress. It is well known that compressive residual stress states usually enhance the mechanical properties in conventional metallic materials. We present investigations on the change of fracture behavior related with mechanical properties of "brittle" bulk metallic glass by cold rolling at room temperature. Improvement of the intrinsic plasticity is observed not only after constrained cyclic compression but also after cold rolling. Moreover, neither nanocrystallization nor phase separation occurs during deformation. By these findings we provide an unique fundamental basis by considering the introduction of structural inhomogeneity and ductility improvement in metallic glasses. The experimental evidence clearly supports that such an inhomogeneous glassy can be produced by residual stress in well known "brittle" bulk metallic glasses, and does not depend on a specific pinpointed chemical composition.

#### 11:40 AM

Local Atomic Arrangements of Pd-Based Bulk Metallic Glasses of the Metal-Metalloid Type Demonstrated by Molecular Dynamics Simulations: *Akira Takeuchi*<sup>1</sup>; Akihisa Inoue<sup>1</sup>; <sup>1</sup>Tohoku University

The local atomic arrangements of Pd-based bulk metallic glasses (BMGs) in metal-metalloid type were analysed by molecular dynamics (MD) simulations based on a plastic crystal model (PCM). The feature of compositions of  $Pd_{40}Ni_{40}P_{20}$ , which can be approximated with the value of Golden Mean ( $\phi \cong$ 1.618) as  $Pd\phi^{-2}Ni\phi^{-2}P\phi^{-3}$ , was utilized to relate the compositions of the BMGs with critically-percolated cluster-packed structure. It was found that the local atomic arrangements of the  $Pd_{40}Ni_{40}P_{20}$  BMG can have an ensemble of clusters with 18 and 7 atoms where the former and latter clusters have cubeoctahedra capped with four half octahedral sites without an atom at a center site and trigonal prism sites with a center site, respectively. The MD-PCM revealed that the random rotations of clusters followed by structural relaxation lead to the formation of the noncrystalline structure. The reason for the  $Pd_{40}Ni_{40}P_{20}$  alloy to have high glass-foming ability is due the critically-percolated cluster-packed structure, in which the latter-type clusters form a network structure. In addition to  $Pd_{40}Ni_{40}P_{20}$ BMG, it was found that  $Pd_{40}Cu_{30}Ni_{10}P_{20}$  BMG also exhibits the same features. This critically-percolated cluster-packed structure is a unique characteristic for both metal-metalloid and metal-metal type BMGs.

#### 11:55 AM

New Crystallographic Textures of Nd<sub>2</sub>Fe<sub>14</sub>B/A-Fe Nanocomposite Materials Prepared by Controlled Melt Spinning: *Xierong Zeng*<sup>1</sup>; Hongchao Sheng<sup>2</sup>; Jizhao Zou<sup>3</sup>; Shenghui Xie<sup>3</sup>; <sup>1</sup>college of Materials Science and Engineering, Shenzhen University; Shenzhen Key Laboratory of Special Functional Materials; <sup>2</sup>School of Materials Science and Engineering, Northwestern Polytechnical University; <sup>3</sup>College of Materials Science and Engineering, Shenzhen University; Shenzhen Key Laboratory of Special Functional Materials

In Nd<sub>95</sub>Fe<sub>84</sub>B<sub>65</sub> melt-spun ribbon, the quenching temperature is found to be effective for the texture development of Nd<sub>2</sub>Fe<sub>14</sub>B nanocrystals. For a relatively



low quenching temperature of 1250°Ca (00l) texture of  $Nd_2Fe_{14}B$  crystals was found on the free-side surface of the ribbons. At a higher quenching temperature of 1350°C, the microstructure of the free-side surface of the ribbons switches into (320) and (517) texture, and A good rectangular-like shape of hysteresis loops has been observed with excellent magnetic properties of Jr=1.07T, Hc =509kA/m and (BH)max=135kJ/m3. It is believed that the transformation of the melt at higher temperature triggers the switch of the texture.

#### 12:10 PM

**Phase Transformation and Electron Transport Properties of** a Zr<sub>61.75</sub>Al<sub>9.5</sub>Ni<sub>9.5</sub>Cu<sub>14.25</sub>Nb<sub>5</sub> Bulk Metallic Glass: *Yingmin Wang*<sup>1</sup>; Qing Wang<sup>1</sup>; Jianbing Qaing<sup>1</sup>; Chuang Dong<sup>1</sup>; <sup>1</sup>Dalian University of Technology

The phase transformation and the electrical transport properties of a Zr<sub>61.75</sub>Al <sub>9.5</sub>Ni<sub>9.5</sub>Cu<sub>14.25</sub>Nb<sub>5</sub> bulk metallic glass (BMG) have been studied using differential scanning calorimetry and transmission electron microscopy, and a physical properties measurement system, respectively. The BMG exhibited a two-stage crystallization process during continuous heating at a rate of 20 K/min. In the first stage crystallization, the BMG transformed into a nanometer scaled P-type icosahedral quasicrystal (aR = 0.54 nm) via a polymorphic mechanism. An Avrami exponent range from 1.8 to 2.5 was derived from the isothermal transformation kinetics experiments conducted within the undercooled liquid temperature span of 683-713 K. The effective activation energy for quasicrystallization was determined to be about 369 kJ/mol. Both the BMG and its quasicrystalline counterpart were demonstrated to be d-electron strong scattering alloys. They showed negative temperature coefficients of resistivity within a wide temperature span of 2.5-330 K. The structural evolution and the electron transport behavior are discussed in light of the icosahedral glass model proposed for P-type metastable icosahedral quasicrystal.

# Symposium E: Solidification, Deformation and Related Processing: Ultrafine-Grained Materials III

Tuesday AM	Room: 2	
August 3, 2010	Location:	Cairns Convention Centre

Session Chair: Yuri Estrin, Monash University

#### 8:30 AM Keynote

Nanostructure Formation during Deep Wire-Drawing of Copper: Nobuhiro Tsuji<sup>1</sup>; Ken-ichi Hanazaki<sup>2</sup>; <sup>1</sup>Kyoto Univ; <sup>2</sup>Yazaki Corporation

Copper and copper alloys are frequently used as fine wires for electric applications. Deep drawing process to produce fine wires of copper alloys can act as a kind of severe plastic deformation process, though the total process is carried out in multi passes. High purity copper was deeply wire-drawned up to equivalent strain of 9, and microstructure evolution and change in mechanical properties were investigated. It was confirmed that deep wire-drawing process produced nanostructures composed of fiber-shaped ultrafine grains with a diameter of about 300 nm. The copper wires having nanostructures showed tensile strength of 480MPa, which was two times higher than that of the starting material. Characteristic microstructure, texture and mechanical properties of the nanostructured copper fabricated by deep wire-drawing are presented in the paper.

#### 8:50 AM Keynote

# Microstructure and Texture Evolution during Friction Stir Processing of AZ31 Mg Alloy: *Qing Liu*<sup>1</sup>; Renlong Xin<sup>1</sup>; Bo Li<sup>1</sup>; <sup>1</sup>Chongqing University

AZ31 Mg alloy sheets with different initial textures were chosen for friction stir processing (FSP). Different characterization techniques including optical microcopy (OM), scanning electron microscopy (SEM) and electron back scatter diffraction (EBSD) technique were used to investigate the microstructure and texture evolution during FSP of AZ31 Mg alloy. Micro-hardness and tensile tests were used to measure the mechanical behavior of the processed samples. It was found that the FSP resulted in a very strong texture and a fine grain-size structure by a significant texture evolution and dynamic recrystallization within the stir zones (SZ), and there is no obvious difference of both micro-texture and grain size for the samples with different initial textures. However, from the heat affected zone (HAZ) to the thermo-mechanical affected zone (TMAZ), there is a continuous and different texture evolution from the initial texture to the texture of the stir zone for the samples with different initial textures. Effect of the initial texture on the mechanism of the microstructure and texture evolution during FSP of AZ31 Mg allov was discussed based on the structure characterization results and the related mechanical behavior of the materials.

### 9:10 AM

Application of SPH for Modelling Heat Transfer and Residual Stress Generation in Arc Welding: *Raj Das*<sup>1</sup>; Paul Cleary<sup>1</sup>; <sup>1</sup>CSIRO Mathematical and Information Sciences

An approach for three-dimensional modelling of thermo-mechanical responses in an arc welding process is developed using the Smoothed Particle Hydrodynamics (SPH) method. It is demonstrated for a simple arc welding configuration by solving the fully coupled three-dimensional elastoplastic and heat transfer analysis. The temperature distribution of the metal in the weld pool and the surrounding parent material are analysed using SPH, and the resulting residual thermal stresses are evaluated. This work establishes the capability of SPH as a tool for simulating the long-term thermo-mechanical responses, including heat transfer and residual stresses in a welded joint, and gaining insights into post-welding structural behaviour of joints during cooling stages.

#### 9:25 AM Keynote

Atomic-Scale-Deformation Dynamics (ASDS) of Nanowires and Nanofilms: Ze Zhang<sup>1</sup>; <sup>1</sup>Beijing University of Technology

Nanowires and nanofilms are fundamental building blocks of micro and nanoelectronics for both of bottom-up and top-down technologies. Monitoring and recording the mechanical property dynamics at atomic scale are important to understand the atomic mechanism of new and surprising nano-phenomena and design new applications. Through years' endeavours, we have developed tensile and/or bending in-situ atomic-lattice resolution electron microscopy methods and equipments for nanowires and successfully conducted atomic-lattice resolution mechanical tests on individual nano-objects. With this, we observed that brittle materials SiC, Si and even Si<sub>x</sub>O<sub>1-x</sub>-nanowires (NWs) become highly ductile at room temperature. The metallic nanowires show unusual deformation mechanisms for large strain elasticity and plasticity. The crystalline structural evolution processes corresponding to the occurrence of unusual plasticity includes the dislocation initiation, dislocation accumulation and interaction as well as the necking of the one dimensional nanowires were fully recorded at atomic-scale and in real time. Further, we expand the experimental methods and equipments to two-dimensional nanofilms. An example of tensile experiment on Au films will be presented. The deformation and failure mechanisms of nano-crystalline Au films were observed at the atomic-scale and in real-time. In addition, we dynamically and directly observed intergranular crack nucleation and propagation along tri-boundaries. At the mean time, an atomic-scale single crystalline necking process was captured with the original grain diameter being 15 nm and a final 1nm before fracture which depress the emission of dislocations. In larger grains, the dislocation nucleation and propagation, deformation twins, grain rotations and grain boundary sliding were observed and in-situ recorded at atomic-scale.

#### 9:45 AM Invited

In-Situ Mechanical Testing during X-Ray and Neutron Diffraction: *Helena Van Swygenhoven*<sup>1</sup>; Alex Evans<sup>1</sup>; Steven Van Petegem<sup>1</sup>; <sup>1</sup>Paul Scherrer Institute

The microstructure of today's metals is steadily increasing in complexity because of the increasing demands on performance. Classical characterization and mechanical testing techniques can not provide all necessary input parameters for adapting predictive engineering models for such complex microstructures. Advances in X-ray/neutron technologies have increased beam intensities, improved detector efficiency/speed and provided better focusing techniques. In-situ mechanical testing under X-ray and neutron diffraction has become now an interesting research method to address load-sharing and phase transforming mechanisms in complex multiphase structures or degradation phenomena in fatigue and creep processes. Another interesting aspect resulting from microfocusing capacities at synchrotron sources is the renewed application of white beam Laue diffraction in addition to powder diffraction methods. Laue diffraction is the oldest diffraction method for crystallography and can be now applied insitu to study the enhanced strengthening observed in spatially confined volumes or to follow the dynamics of sub-grain formation. This talk will discuss a few in-situ studies that have provided unprecedented details on the microstructure and deformation mechanisms, information that is essential for the understanding of the mechanical behavior. The examples will range from advanced steels to micron-sized single crystal pillars.

#### 10:00 AM

Formation of Deformation Twins and Related Shear Bands in Copper Single Crystals Pressed by ECAP: *Takumi Ikeda*<sup>1</sup>; Hiroyuki Miyamoto<sup>1</sup>; Toshiyuki Uenoya<sup>1</sup>; Satoshi Hashimoto<sup>2</sup>; Alexei Vinogradov<sup>2</sup>; <sup>1</sup>Doshisha University; <sup>2</sup>Osaka City University

The prominent feature of ECAP is that intense shear strain is imposed along a well-defined plane around the intersection of the input and output channels. It means that the plastic deformation of the materials along the other directions will be severely restricted by the channel wall. Therefore, this special deformation mode of ECAP may provide a possibility to effectively suppress the activity



of slip systems for some specific oriented single crystals. Recently, twinning deformations were observed using pure copper single crystal having an initial orientation with the {111} slip plane parallel to the shear plane and the [112] twinning direction parallel to the shear direction at room temperature and low strain rate, because the formation of deformation twinning depends on an initial crystallographic orientation. We found that a shear band having a high angle boundaries and similar orientation relationship with twining relation formed in a specific condition. Deformation twinning presumably have great influence on the formation of such shear bands because they were observed with deformation twins. In this study, we pressed some copper single crystals by ECAP for one passes, focusing on the influence of the deformation twins on development of shear band in a crystallographic aspect.

10:15 AM Break

# Symposium E: Solidification, Deformation and Related Processing: Deformation Processing and Mechanical Properties I

Tuesday AM	Room: 2
August 3, 2010	Location: Cairns Convention Centre

Session Chair: Hyoung Seop Kim, POSTECH

#### 10:50 AM

Fue. AM

High-Pressure Torsion for Achieving Superplasticity of Mg-Li Alloy in Hot Water: Hirotaka Matsunoshita<sup>1</sup>; Shuji Honda<sup>1</sup>; Masaaki Kai<sup>1</sup>; Mitsuaki Furui<sup>2</sup>; Zenji Horita<sup>1</sup>; <sup>1</sup>Kyushu University; <sup>2</sup>University of Toyama

It is well known that high-pressure torsion can produce ultrafine grains in less ductile metallic materials such as magnesium alloys. In this study, a Mg-8mass%Li alloy was processed by HPT at room temperature for 5 revolutions under a pressure of 3 GPa. The grains were refined to an average size of ~500 nm. Tensile specimens were cut from the HPT-processed samples and pulled to failure at a selected temperature in the range of 300-473 K with an initial strain rate in the range of  $1.0x10^{-3}-5.0x10^{-2}$  s<sup>-1</sup>. Superplastic elongation of ~700% was achieved at a temperature of 373 K. Thus, tensile testing was further attempted in boiling water and the specimens were successfully deformed to more than 450%. Scanning electron microscopy revealed that the surface is roughened during the tensile deformation but this is minimized when the strain rate is faster. It was suggested that hydro-forming can be feasible with the Mg alloy when the alloy is processed by HPT.

#### 11:05 AM

**Enhancing Ductility of ECAP Processed Metals**: Núria Llorca-Isern<sup>1</sup>; *Thierry Grosdidier*<sup>2</sup>; Jose Cabrera<sup>3</sup>; <sup>1</sup>Universitat de Barcelona; <sup>2</sup>Dalian University; <sup>3</sup>Universitat Politecnica de Catalunya

Mechanical properties such as hardness, mechanical strength or fatigue resistance are by far the most successful behaviour produced by ECAP processed materials. However, the lack of ductility is the most critical negative effect associated to them. Different possibilities to enhance ductility are been studied, combination of multimodal grain size is one of the most promising solutions. These complex microstructures can be achieved by controlled thermal treatments. The aim of the present work is to understand the mechanisms by which metallic materials would be severely deformed and the evolution of the deformed grains in order to produce the multimodal grain microstructure and hence the ductility needed to build performed materials. Different characterisation techniques were used to analyse the morphology, texture and relationships between grains among them, high resolution electron microscopy and EBSD were the most useful. Associated thermal treatments and their mechanical properties influence were also investigated. US measurements were also carried out to the specimens and correlation to microstructural changes were determinated.

#### 11:20 AM

Ultrafine Grained Structure Formation in Low Carbon Steel Processed by SPD: *Jozef Zrnik*<sup>1</sup>; Sergey Dobatkin<sup>2</sup>; George Raab<sup>3</sup>; Libor Kraus<sup>4</sup>; <sup>1</sup>COMTES FHT, Inc.; <sup>2</sup>Baikov Institute of Metallurgy and Material Science, RAS; <sup>3</sup>Institute of Physics of Advanced Materials, USATU; <sup>4</sup>Comtes FHT Ltd.

The present work deals with grain refinement of low carbon steel by severe plastic deformation (SPD). The structure modification of steel was evaluated with respect to executed TM treatment prior SPD. The grain refinement was accomplished during warm angular channel pressing (ECAP) at 300°C. The evolution of microstructure during equal channel angular pressing (ECAP) was studied using SEM and TEM of thin foils. Ultrafine grained structure development is described regarding the strain applied. Employing ECAP route the progress in

structure refinement was partially modified by different initial structure of steel. At lower straining the polygonized structure dominated and subgrains structure was frequently found. The introduction of TM steel processing prior ECAP contributed to refinement of deformed structure. Due to increased deformation temperature the dynamic recovery substantially contributed to structure refinement at both structural states. The volume fraction of high angle boundaries increases with higher ECAP straining and was more frequent in TM processed steel. The deformation behaviour of UFG steel, in dependence of processing conditions, was evaluated by tensile test and related to structural characteristics. Considering the deformation behaviour of ECAP specimens, the work hardening during tensile deformation was not observed regardless any initial steel state.

#### 11:35 AM

Grain Refinement and Texture Evolution during Equal Channel Angular Extrusion of Pure Cu and Cu-Zn Alloys: Saiyi Li<sup>1</sup>; Haihong Chen<sup>2</sup>; <sup>1</sup>Central South University; <sup>2</sup>South China University of Technology

While dislocation slip is the predominant mechanism during plastic deformation at room temperature in face-centered cubic metals with high stacking fault energy (SFE), mechanical twinning also plays an important role in metals with low SFEs. In this study, equal channel angular extrusion (ECAE) experiments were conducted on as-cast pure Cu and two different Cu-Zn alloys at room temperature using a 90-degree die for up to four passes. The microstructure and texture of the processed billets were measured respectively using electron microscopy and X-ray diffraction. The results show that the three as-cast materials are all significantly refined and the average grain size decreases with decreasing SFE. The deformation textures after multiple passes depict different features and a lower SFE leads to a weaker texture. It is concluded that the SFE has significant influences on the deformation behavior and consequently on the microstructural and texture development. The role of twinning in severe plastic deformation with strain path changes by ECAE is also discussed.

#### 11:50 AM

Strain Effect on the Hardness and Structure of a Nanocrystalline Ni-Fe Alloy Processed by High-Pressure Torsion: Song Ni<sup>1</sup>; Yanbo Wang<sup>1</sup>; Xiaozhou Liao<sup>1</sup>; Hongqi Li<sup>2</sup>; Saleh N. Alhajeri<sup>3</sup>; Yonghao Zhao<sup>4</sup>; Enrique J. Lavernia<sup>4</sup>; Simon P. Ringer<sup>5</sup>; Terence G. Langdon<sup>6</sup>; Yuntian Zhu<sup>7</sup>; <sup>1</sup>School of Aerospace, Mechanical and Mechatronic Engineering, The University of Sydney; <sup>2</sup>Los Alamos National Laboratory; <sup>3</sup>Materials Research Group, School of Engineering Sciences, University of Southampton; <sup>4</sup>Department of Chemical Engineering and Materials Science, University of California at Davis; <sup>5</sup>Australian Key Centre for Microscopy and Microanalysis, The University of Sydney; <sup>6</sup>Departments of Aerospace and Mechanical Engineering and Materials Science, University of Southern California, Los Angeles; <sup>7</sup>Department of Materials Science and Engineering, North Carolina State University

A nanocrystalline Ni-Fe alloy with an initial average grain size of about 21 nm was processed using high-pressure torsion (HPT). Hardness evolution was investigated using microindentation. Strain hardening occurred at the beginning of HPT deformation followed by strain softening and strain hardening again. Microstructural investigation using X-ray diffraction (XRD) and transmission electron microscopy (TEM) revealed that the two hardening stages were associated with the increase of the dislocation density in the material while the strain softening stage was accompanied by dislocation density reduction, clearly indicating the dependence of the hardness on the dislocation and coalescence throughout the HPT process, and the average grain size reached about 53 nm at the edge of a 10-revolution disk. De-twinning also occurred during HPT. The effect of grain growth and de-twinning on the hardness evolution was not as significant as that of dislocations.

#### 12:05 PM

The Influence of Processing Conditions on Hardness Homogeneity Evolution in Commercially Pure Cast Aluminium Processed by ECAP: Sri Lathabai<sup>1</sup>; Margarita Vargas<sup>1</sup>; Matthieu Larroque<sup>1</sup>; Claude Urbani<sup>2</sup>; <sup>1</sup>CSIRO Process Science and Engineering; <sup>2</sup>CSIRO Materials Science and Engineering

Commercially pure cast aluminium was subjected to equal channel angular pressing (ECAP) at room temperature using routes A, Bc and C. Microhardness measurements were made on the longitudinal sections of the extruded billets after one, two, three and four passes for each of the processing routes. Hardness contour maps provided information on the hardness distribution. It was found that the mean hardness increased significantly already after the first pass. With subsequent passes, the rise in average hardness was smaller but the hardness distribution became narrower, indicating an increase in homogeneity. For route Bc, a slight decrease in average hardness was observed after the fourth pass. The mean hardness after four passes was the highest for the route C sample, followed by the route A and route Bc samples. To further investigate this trend for processing route Bc, additional tests involving five and eight passes were carried



out. Hardness measurements over a period of time showed that in the case of Bc samples, a slight reduction in average hardness occurred, suggesting that the microstructures generated by this route may be less stable than those produced by routes A and C.

# Symposium F: Modelling and Simulation of Microstructures and Processes: Numerical Modeling of Material Processing I

Tuesday AM	Room: D
August 3, 2010	Location: Cairns Convention Centre

Session Chairs: Qingyan Xu, Tsinghua University; Daniel Liang, CSIRO

#### 8:30 AM

2-D and 3-D Finite Element Analysis of Tubular Ovality and Stresses in Horizontal and Vertical Wells: *Tasneem Pervez*<sup>1</sup>; Sayyad Qamar<sup>1</sup>; Saif Al-Hiddabi<sup>1</sup>; Farooq Al-Jahwari<sup>1</sup>; <sup>1</sup>Sultan Qaboos University

Solid expandable tubular continues to revolutionize the design, construction and remediation of oil and gas wells. The process involves permanent diametral expansion of a tubular by passing mandrel through it. Poor understanding of mechanics of deformation and stresses developed in the tubular during down-hole expansion may result in its premature failure. Additionally, constrained tubular expansion in oval bore-holes results in non-circular cross-sections at various depths in the well. This phenomenon of tubular ovality has been previously unknown to petroleum industry. Under such circumstances, the minimum inner-diameter of expanded tubular must be larger than drift-diameter set by API standard. If the minimum inner-diameter is smaller than drift-diameter, completion equipments cannot be run successfully to complete an oil-well for production. This paper presents 2-D and 3-D finite element analysis of tubular expansion in oval bore holes. The simulation results were compared with measured ovality. Once validated, simulations were carried out for different tubular sizes used by the petroleum industry. It was found that ovality increases linearly with expansion ratio. With increase in expansion ratio, the tubular contact length and pressure with formation increases. The stresses developed during tubular expansion in oval bore-holes were also estimated and compared with those of regular boreholes.

#### 8:45 AM

#### Advances on Modelling of the Tool/Workpiece Interface during High Shear Processing: Michal Krzyzanowski<sup>1</sup>; Mark Rainforth<sup>1</sup>; <sup>1</sup>The University of Sheffield

The high shear processing during hot rolling of aluminium is effective in producing a highly deformed subsurface layer, due to asperity contact between stock and work rolls. The tribological conditions are primarily responsible. Another example associated with the tool/workpiece interface during high shear processing is heat generation and flow during friction stir welding (FSW). Understanding and prediction of physical phenomena in different scales at the same time, which are taking place at the tool/workpiece interface during high shear processing, is done in different ways combining the latest FE and discrete element (DE) analysis technology. The FE analysis is used for macro-scale simulation while the DE method is applied to simulate meso-scale phenomena taking place in the thin, sometimes a few micron thickness, surface layer. The potential of FE tools and techniques merged with DE based transient dynamics have been highlighted by the authors earlier. Different examples of such applications are considered in this work.

#### 9:00 AM

#### Effects of Forging Processing Parameters on Axial Effective Strain in Heavy Forgings: Y.C. Lin<sup>1</sup>; *Ming-Song Chen*<sup>1</sup>; <sup>1</sup>School of Mechanical and Electrical Engineering, Central South University, China

Heavy forgings are the essential parts of national basic technology equipments and are widely applied to the aero, shipping and nuclear equipments. Not only they should meet geometrical requirements, but also should have superior mechanical properties and fine grain distribution. In industrial forming processes, the metals and alloys are subjected to complex time, strain, strain rate, and temperature histories. Among them, the magnitude and distribution of strain in the large forgings is most important, which directly affects the geometry and microstructural evolution of the deformed blocks. In order to improve the quality of products, understandings of the relationship between forging processing parameters and strain evolution and microstructural evolution of alloys under hot deformation condition is of great importance for designers of metal forming processes. In this study, one thermo- mechanical coupled finite element model was developed. The effects of forging processing parameters, including deformation degree, tool width ratio, bank width ratio, and tools-workpiece frition, on the axial effective strain in large forgings were investigated. Results show that the forging processing parameters significantly affect the distribution of strain in large forgings, which are useful for the industry production.

#### 9:15 AM

# Finite Element Analysis of Multi-Pass Equal Channel Angular Extrusion/ Pressing Process: Krishnaiah Arkanti<sup>1</sup>; Uday Chakkingal<sup>2</sup>; <sup>1</sup>Osmania University; <sup>2</sup>Indian Institute of Technology Madras

Equal channel angular extrusion (ECAE) is a severe plastic deformation (SPD) method for obtaining bulk nanostructured materials. The ECAE die consists of two equal channels that intersect at an angle, usually between 90° and 135°. In the present study, the plastic deformation behavior of the Cu during the ECAE process with 120° die through multiple passes was investigated. Finite element modelling was included in order to analyze the deformation behavior as the material passes through the die. In order to perform the FEM simulations the properties of the commercial purity Cu have been selected.

#### 9:30 AM

The Influence of Density of Aluminium Foam Filler on Crashworthiness by FE Analysis: *Shujuan Hou*<sup>1</sup>; Qing Li<sup>2</sup>; Xu Han<sup>1</sup>; Shuyao Long<sup>1</sup>; <sup>1</sup>Hunan University; <sup>2</sup>University of Sydney

**Fue.** AM

Nowadays, aluminium foam has come to be one of most favorable filler materials in vehicle design to improve crashworthiness during impacting. Because of its high Energy-absorption and light weight, foam filler materials have widely used in the vehicle structures to meet the design requirements. Over the past two decades, substantial studies have been conducted with the rapid emergence of various new metallic foams. These studies showed that it is worth taking into account the influence of properties of foam material on its energy-absorbed capacities in the vehicle component design. In this paper, the density of aluminium foam material is studied in terms of its influence on the energy-absorbed capacity of the material.By varying the density,the crashworthiness of foam-filled tube is also changed. Here the Specific Energy Absorption (SEA) is taken as the objective function by using the Response Surface Method (RSM) on a basis of explicit Finite Element(FE)algorithm.

#### 9:45 AM

#### The Identification of Phase by Overlapping of First Derivative of Dilatation in Low Carbon Steels Multi-Phase Presenting: *Bong June Park*<sup>1</sup>; Jong Min Choi<sup>1</sup>; Sang Hwan Lee<sup>2</sup>; Kyung Sub Lee<sup>1</sup>; Kyung Jong Lee<sup>1</sup>; <sup>1</sup>Hanyang University; <sup>2</sup>Korea Institute of Industrial Technology (KITECH)

The phase transformation in steels has been widely measured by dilatometer using the lever rule. However, the concept of lever rule is no longer applied in case of multi-phase present. Furthermore, it is quite difficult to differentiate a low temperature phase from the others due to the small fraction change  $(10_3 - 10_4$  fraction of original length) and the plastic deformation during transformation. The overlapping of first derivatives of LVDT of several continuous cooling dilatations could be better way to identify and to analyze low temperature phases. In addition, the length change is simulated by considering the lattice parameter changes due to the temperature, composition and phase as well as decomposition kinetics of austenite in order to verify the method suggested. By comparing the simulated length change with the measured, the first derivative of dilatation interfered could be separated for each phase. As a result, the start, finish and peak temperature and the amount of each phase are determined. The method is also confirmed by OM and SEM.

#### 10:00 AM

# Homogenisation of Cast Microstructure: Thermodynamic Calculation and Kinetic Simulation: Zhanli Guo<sup>1</sup>; Peter Miodownik<sup>1</sup>; Rongshan Qin<sup>1</sup>; <sup>1</sup>Sente Software Ltd.

Alloy castings and ingots are usually given a homogenisation heat treatment prior to further processing in order to distribute the alloying elements as evenly as possible throughout the microstructure. Inhomogeneous solutes can adversely affect an alloy's properties and/or induce the formation of undesired phases. Homogenisation treatment is a critical step in alloy processing whether it is to be used in as-cast or wrought conditions. Conventional homogenisation treatments have frequently been designed empirically, which is both time-consuming and costly. An approach to simulate the homogenisation process of cast microstructure is proposed in this paper, which is designed to produce optimum homogenisation parameters for both binary and multi-component alloys. The essence of this approach is to assume that the solute segregation profile across a half dendrite arm spacing distance can be scaled to the solute concentration profile during solidification as generated by a Scheil calculation. The homogenisation process can then be handled as a one dimensional diffusion problem. Examples are drawn from cast nickel-based superalloys (Nimonic 105) and aluminium-copper alloys with and without secondary equilibrium phases.



#### 10:15 AM

Crystal Plasticity Finite Element Modelling of the Influence of Friction on Surface Roughening during Uniaxial Planar Compression: *Hejie Li*<sup>1</sup>; Zhengyi Jiang<sup>1</sup>; Dongbin Wei<sup>1</sup>; Jingtao Han<sup>2</sup>; Kiet Tieu<sup>1</sup>; <sup>1</sup>University of Wollongong; <sup>2</sup>University of Science and Technology Beijing

High surface quality of metal products is a key issue of metal manufacturing industries. In general, the surface roughening is generated by many factors such as the original surface roughness of the product, grain size, crystal orientation, texture distribution, friction, loading path, stress-strain state (deformation mode) and tool surface etc [1]. Among them, the friction is one of the main factors for surface roughening in metal forming. To figure out the relationship between the friction and the surface roughening, a finite element model is developed with the commercial finite element software ABAQUS to simulate the surface roughening of Al plate during uniaxial planar compression. Initial surface profiles and orientations are directly input into the model. During the compression, the contact friction coefficients between the tool and sample are changed according to the practical experiments. The calculated surface roughness is close to the measured value. The surface roughness increment in rolling direction is proportional to the friction coefficient. During the surface roughening process, the grain slip takes place in the "soft orientation". The "hard orientations" becomes the barrier of the slip. During the uniaxial planar compression, the contact friction can aggravate surface roughening of Al plate significantly.

10:30 AM Tea Break

# Symposium F: Modelling and Simulation of Microstructures and Processes: Numerical Modeling of Material Processing II

Fuesday AM	Room: D
August 3, 2010	Location: Cairns Convention Centre

Session Chairs: Sun-Keun Hwang, Inha University; Zhengyi Jiang, University of Wollongong

#### 10:50 AM

**The Material Flow Analysis in the Modified Orbital Forging Technology**: Jaroslaw Nowak<sup>1</sup>; *Lukasz Madej*<sup>1</sup>; Franciszek Grosman<sup>2</sup>; Maciej Pietrzyk<sup>1</sup>; <sup>1</sup>Akademia Gorniczo Hutnicza; <sup>2</sup>Politechnika Slaska

The main aim of this work is the computer aided design of the new orbital forging process. The finite element model was developed and used during research on possibility of modification of the classical orbital forging technology based on the Marciniak press to obtain more effective process. The idea of the modification of the orbital press is to develop an orbital process completely based on the small incremental deformations. In order to do that a load from the upper rotating conical die is transferred into the material by a series of small anvils. Various number of anvils i.e. varying between four and twenty four can be used. Obtained numerical results are compared with the experimental analysis, performed on the orbital press with the developed device. However due to the novelty of the developed approach the investigation on direction of material flow during deformation is under particular interest of this work. Direction of material flow and strain path change effect due to incremental character of deformation is analysed. Obtained results confirm good predictive capability of the FE model and are the basis for the comparison and discussion about the effectiveness of this modified incremental forming process.

#### 11:05 AM Keynote

#### **Numerical Simulation on Semi-Solid Metal Slurry Preparation by A-EMS**: *Zhang Zhifeng*<sup>1</sup>; 'General Research Institute for Non-ferrous Metals

Characterized by non-pollution, low cost, and easy process control, electromagnetic stirring has been a main method for producing Al-alloys semisolid slurry or billet. However, there still exist inhomogeneous microstructures in the semisolid billets especially large-sized ones due to the skin effect resulting from electromagnetic induction. To solve the problem, an advanced semi-solid metal slurry preparation process, e.g., the annular electromagnetic stirring (A-EMS), has been developed, but few quantitative studies have been carried out to present the interactive effects of macroscopic transport phenomena in the A-EMS process in spite of the indisputable experimental results. In this study, a two-dimensional computational model coupling electromagnetic stirring with a macroscopic heat and fluid flow in Al-alloys semisolid slurry preparation by A-EMS was developed. The dynamic evolution of the electromagnetic field, flow field and temperature field were presented successfully by commercial software ANSYS with corresponding experimental verification. A horizontally rotational electromagnetic field, and thereby a more intensive velocity field, were uniformly distributed in the stirred melt even at commercial frequency, and thus a lower temperature difference in the stirred melt and subsequent uniformly fine microstructures were obtained compared with the normal electromagnetic stirring. The simulation results were in good agreement with experiment ones.

#### 11:25 AM Keynote

# Numerical Simulation of Grain Selection Behavior of Single Crystal Ni<sub>3</sub>Al Based Superalloy Casting: Dong Pan<sup>1</sup>; *Qingyan Xu*<sup>1</sup>; Baicheng Liu<sup>1</sup>; <sup>1</sup>Tsinghua University

Ni,Al based superalloy is recently used for the single crystal gas turbine blade. The grain selection behavior in pigtail directly determines the casting's final microstructure and property. Usually it is difficult to precisely control the process parameters to get a whole single crystal casting for Ni<sub>3</sub>Al based superalloy. Numerical simulation technology can be applied to study the directional solidification process and predict the microstructure defects, and then optimize the pigtail design to avoid defects. In this paper, a mathematical model based on Modified CA-FD method was developed for the three-dimensional simulation of directional solidification process of single crystal castings. The microstructure evolution was simulated with the modified Cellular Automaton method, and a Discrete Layer-by-layer Calculation method was used to couple the macro and micro scale simulation. The grain selection process in the pigtail and final microstructure of casting were simulated. The results indicate that the stray grain is easy to nucleate at the middle of the pigtail because of the discontinuous mushy zone distribution. This agreed with former published experimental results. Several newly designed pigtails with optimized geometry were proposed to avoid stray grains, based on simulated results. Further experiments for those optimized pigtails are in plan for validation.

#### 11:45 AM Invited

Analysis of Advanced Strip Shape during Cold Rolling of Thin Strip: *Zhengyi Jiang*<sup>1</sup>; Xiaozhong Du<sup>1</sup>; Yanbing Du<sup>1</sup>; Dongbin Wei<sup>1</sup>; Matthew Hay<sup>1</sup>; <sup>1</sup>University of Wollongong

The demand of thin gauge strip with good quality such as the strip shape and surface finish is significantly increasing. Cold rolling is an essential method to manufacture the strip and foil products of metals. Strip shape control during cold rolling of thin strip is a significant challenge in metal rolling practice. In this study, finite element models of the strip shape during cold rolling of thin strip in both symmetrical and asymmetrical rolling were successfully developed, and the finite element simulation of the thin strip shape has been carried out in LS-DYNA. The effects of rolling parameters and surface contact features on the strip shape and profile such as the thickness distribution along the strip width and the strip edge drop have been obtained. The developed finite element model has been compared with the experimental value, which indicates they are in good agreement. The obtained results are applicable to control the rolled thin strip shape in rolling practice.

#### 12:00 PM

**Study on Casting Roll during Twin-Roll Casting of Thin Strip**: *Xiaoming Zhang*<sup>1</sup>; Zhengyi Jiang<sup>2</sup>; Dongbin Wei<sup>2</sup>; Xianghua Liu<sup>1</sup>; Guodong Wang<sup>1</sup>; <sup>1</sup>Northeastern University; <sup>2</sup>University of Wollongong

A thin strip with a thickness of 1.0-6.0 mm can be directly produced from the liquidus steel using twin-roll casting. Compared with the traditional thin strip casting and rolling, the twin-roll thin strip casting simplifies the production process significantly, and save up to 70% equipment investment and 30 - 40% production cost. This process can also improve the product microstructure, develop new material products, and produce metallic products which have hard formability and cannot be produced by a traditional method. In twin-roll thin strip casting, the temperature of casting roll affects the roll thermal stress, and influences the thermal deformation, the generation of roll surface cracks, the strip shape, and the service life of casting roll. In this paper, the features of the casting roll materials have been analysed, the effects of casting parameters, such as the casting speed and the cooling rate on the temperature field and thermal stress of casting roll have been simulated and discussed. The calculated roll surface temperatures are in good agreement with the measured values. The developed temperature model of casting roll is helpful in optimising the processing parameters and the design of the casting roll during twin-roll thin strip casting.

#### 12:15 PM

Numerical Characterization of Anisotropic Heat Sink Composites: *Thomas Fiedler*<sup>1</sup>; Graeme Murch<sup>1</sup>; Timo Bernthaler<sup>1</sup>; Irina Belova<sup>1</sup>; <sup>1</sup>The University of Newcastle

This work addresses the numerical analysis of anisotropic composite structures for thermal energy storage and temperature stabilization. The basic idea of heat sink composites is the combination of metallic matrices for fast energy transfer with phase change materials for thermal energy storage. Anisotropic matrices, such as lotus-type structures, allow for increased control of the thermal energy



flow, without the necessity of additional thermal insulation. As an example, thermal energy can be directed towards a surface cooled by convection and excess energy is stored in the phase-change material. Computed tomography data of copper lotus-type material is used for the generation of the numerical calculation models. Due to its particular meso-structure, this material is characterised by strongly anisotropic properties. The void space of this cellular metal is filled with the phase-change material paraffin in order to increase the energy storage capacity. A recently extended Lattice Monte Carlo method is used to evaluate the energy storage capacity and transient behavior (i.e. temperature stabilization) of these promising materials.

#### 12:30 PM

#### The Research on Controlling the Pre-Bending Deformation before Straightening and the Residual Stresses after Straightening of 100-Meter Rail: *Lin Chen*<sup>1</sup>; <sup>1</sup>Inner Mongolia University of Science and Technology

The repeated bending of rail is caused by different shrinkage or swelling capacity of different parts of rail with irregular cross-section during cooling after hot rolling. The straightness and residual stresses of the rail after straightening are affected by the bending deformation during cooling before straightening. By analyzing the heat boundary condition in the cooling process, the bend deformation is simulated by using the 3-D transient non-liner finite element method. The effect of the pre-bending deformation before straightening on the residual stresses after straightening is researched by controlling the bending deformation before straightening and the straightening deformation, drawing a conclusion that the residual stresses are affected by different chord heights at the same straightening rule, and the calculated results have a good accordance with the measured values on site.

## Symposium G: Thin Films and Surface Engineering: Characterisation and Properties of Engineered Surfaces II

Tuesday AM	Room: 8
August 3, 2010	Location: Cairns Convention Centre

Session Chairs: David Young, University of New South Wales; Julie Schoenung, University of California, Davis

#### 8:30 AM Keynote

Thermal Barrier Systems: A Challenge in Surface Engineering: Carlos Levi<sup>1</sup>; <sup>1</sup>University of California, Santa Barbara

Thermal barrier coatings (TBC) are now essential in the design of advanced gas turbines for propulsion and power generation. Zirconia with 7±1wt%Y2O3 (7YSZ) has been the standard TBC material since their commercial insertion. The demands for increased engine performance and fuel flexibility translate into higher temperatures and more aggressive operating environments for TBCs, motivating the search for alternate insulating oxides, mostly based on ZrO, with rare earth and/or or transition metal additions. Two groups emerge, one based on the non-transfomable tetragonal (t') structure and the other on zirconates. No candidate in either group meets all the requirements for the more advanced applications. Tetragonal materials are endowed with toughening mechanisms that underpin their durability. However, as the engine temperature increases they are compromised by sintering, destabilization of the t' phase, and by penetration of molten silicate deposits. In contrast, the zirconate materials are phase stable and offer improved resistance to sintering and silicate penetration, but are limited by the absence of intrinsic toughening mechanisms, thermochemical interactions with the thermally grown oxide that protects the underlying alloy, and often by processability. This presentation will discuss current understanding of these issues and the challenges in devising a design strategy to circumvent them.

#### 8:50 AM

Thermal Barrier Systems and Multi-Layered Coatings Fabricated by Spark Plasma Sintering for the Protection of Ni-Base Superalloys: *Daniel Monceau*<sup>1</sup>; Djar Oquab<sup>1</sup>; Claude Estournes<sup>2</sup>; Mathieu Boidot<sup>1</sup>; Serge Selezneff<sup>1</sup>; Nicolas Ratel<sup>1</sup>; <sup>1</sup>Institut Carnot CIRIMAT; <sup>2</sup>CNRS; Institut Carnot CIRIMAT

Aeronautic gas turbine blades, vanes and combustion chambers are protected against high temperature oxidation and corrosion by single or multilayered coatings. These include environmental coatings, generally based on Pt-modified Ni aluminides or MCrAIY coatings (where M = Ni and/or Co), thermal barrier coating (TBC) systems including a ceramic thermally insulating layer, and abradable seals. The present work shows the ability of the Spark Plasma Sintering technique to rapidly develop new coating compositions and microstructures. This technique allows combining powders and metallic foils on a superalloy

substrate in order to obtain multilayered coatings in a single short experiment. Fabrication of MCrAlY overlays with local Pt and/or Al enrichment is shown, as well as fabrication of coatings made of PtAl<sub>2</sub>, PtAl, alpha-AlNiPt<sub>2</sub>, martensitic beta-(Ni,Pt)Al or Pt-rich gamma/gamma prime phases, including their doping with reactive elements. The realization of a complete TBC system with a porous and adherent Yttria Stabilized Zirconia (YSZ) layer on a bond-coating is also demonstrated, as well as the fabrication of a CoNiCrAlY-based cermet coating for abradable seal application. Difficulties of fabrication are reviewed and discussed, such as Y segregation, risks of carburization, local over-heating, or difficulty to coat complex shaped parts. Solutions are given to overcome these difficulties.

#### 9:05 AM

# Investigation of Super-Hydrophobic Coatings with Hierarchical Structures Surface on Mild Steel: *Zhan-ping Zhang*<sup>1</sup>; Yu-hong Qi<sup>1</sup>; Gui-ke Mo<sup>1</sup>; Jing-zhao Wang<sup>1</sup>; <sup>1</sup>Dalian Maritime University

A hierarchical roughness surface with micro-and nano-structures was fabricated on mild steel by chemical etching with a FeCl<sub>3</sub>- HCl-H<sub>2</sub>O solution. The surface was modified by fluorination treating and multi-coated with fluoro-carbon paint which contained nano-TiO2 and tourmaline powders. The wettability of the modified surfaces was studied by contact angle method and Electrochemical Impedance Spectroscopy (EIS), and their morphology was investigated by Scanning Electron Microscopy (SEM). The roughness of the steel etched consisted of fine micro-steps in grains. The surface of coating consisted of nano-particles papilla on the irregular pieces. The treated surface exhibited superhydrophobic properties, respectively with water contact angle of about 152° after fluorination treating and 156° after multi-coated with fluoro-carbon paint. The effect of etching time on the hydrophobicity of the surfaces was investigated. The results indicated that the etching time played an important role in the formation of hierarchical roughness and of a high contact angle. With the increase of etching time, the hierarchical structure gradually formed on the surface and the contact angle gradually increased until a constant value was reached. The possible mechanism of the formation of hierarchical roughness was proposed. Also, the Cassie theory was used to explain the wettability of modified surface.

#### 9:20 AM

# Effect of Pre-Oxidation Treatment on the Thermal Shock Resistance of Thermal Barrier Coatings in a Combustion Gas Environment: *Hui Mei*<sup>1</sup>; <sup>1</sup>National Key Laboratory of Thermostructure Composite Materials

Thermal barrier coatings (TBCs) were deposited by an Air Plasma Spraying (APS) technique. The TBC coating comprised of 92 wt.% ZrO2 and 8 wt.% Y2O3 (YSZ); CoNiCrAIY bond coat; and MarM247 nickle base super alloy. Two types of TBC specimens were tested, after spray of YSZ one batch were firstly oxided in air for 10h at 1080°C, and the others were not. Both types of the samples were directly pushed into a combustion gas at 1150°C for 25 min and then out to the natural air for quenching. The combustion gas was produced by burning the jet fuel with high speed air in a high temperature wind tunnel, which simulates the real service conditions in an aeroengine. The results show that TBCs by the APS had good thermal shock resistance in the combustion gas. The first formation of a continuous  $Al_2O_3$  layer at the ceramic/bond coat is beneficial to improve the life of TBCs. Therefore, the pre-oxidation of the TBC had significant effect on its thermal shock life. The as-oxided specimens had the worse thermal shock resistance than the as-sprayed ones after 100 thermal shock cycles in the combustion gas.

#### 9:35 AM

Investigation on Oxidation Resistance of NiCoCrAIY Coating Irradiated by High Current Pulsed Electron Beam: *Xianxiu Mei*<sup>1</sup>; Cunxia Wang<sup>1</sup>; Wei Qu<sup>1</sup>; Ying Qin; Chuang Dong<sup>1</sup>; Younian Wang<sup>1</sup>; <sup>1</sup>Dalian University of Technology, China

NiCoCrAlY transition layer are widely applied in thermo-barrier coatings to reduce the thermal stress between the outer oxide layer and Ni-based high-temperature alloy substrates. DZ4 substrates were coated with NiCoCrAlY by plasma spray and subsequently treated by a high-current pulsed electron beam (HCPEB) for the purpose of smoothing the surface and releasing stresses. As a result of the HCPEB treatment the loose and originally rough surface structure were remelted and smoothened but craters and cracks appeared frequently. The phase content is increased. The stresses were effectively released as reflected by invisible XRD diffraction peak shifts. Oxidation tests at 900°C for 100h showed that the oxidation layers consist of abundant  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> and minor NiO, which improved the oxidation resistance of the NiCoCrAlY coatings significantly.

#### 9:50 AM

Microstructure and Wear Behaviour of Laser Induced In-Situ Formation of TiBx and TiC Titanium Composite Coatings: J. Liang<sup>1</sup>; C. X. Ren<sup>1</sup>; C. S. Liu<sup>1</sup>; S. Y. Chen<sup>1</sup>; <sup>1</sup>Northeastern University

Two kinds of mixed powders: Ti-6Al-4V/B/C and Ti-6Al-4V/B4C with different proportions which are pre-pasted on Ti-6Al-4V substrates separately

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were scanned by a 500W pulsed YAG laser to induce in situ formation of titanium composite coatings contained TiBx, and TiC ceramic reinforced phases. The influences of laser processing parameters including Pulse Frequency (PF). Pulse Width (PW), Laser Power (P) and Scanning Speed (V) together with the powder proportions on the microstructure and properties of the coatings were investigated. Microstructure, phase components and micro-hardness of the coating were analyzed by OM, SEM, TEM, XRD and micro-hardness tester respectively. The optimized processing parameters of a single path laser scanned specimen in this case are as follows: PF: 15Hz, PW: 3ms, for the Ti-6Al-4V/B4C specimens the laser line energy ~16.5J/mm, for the Ti-6Al-4V/B/C specimens the laser line energy ~11J/mm. TiB and TiC ceramic were formed evenly reinforced in the matrix of Ti-6Al-4V with the morphology of needle, tiny dendrites and disperse spherical particles. The maximum micro-hardness of multi-path layers is up to 800HV, which is twice of that of the substrate (367Hy). The wear weight loss of a multi-path laser scanned specimen formed under the optimized processing parameters decreased nearly 3 times that of the substrate.

#### 10:05 AM

#### Microscopic Study on the Interface Reaction between Ti and Al-Zn Alloy during Ultra-Fast Heat Treatment: Yue Zhao<sup>1</sup>; David Nolan<sup>1</sup>; David Wexler<sup>1</sup>; Andrew Carman<sup>1</sup>; <sup>1</sup>University of Wollongong

A new experimental routine is designed to investigate the reaction/diffusion behaviour between Ti and 55%AlZn alloy. In this method, we employ physical vapour deposition (PVD) to coat polished, pre-homogenized Al-Zn discs. The resultant sample is subsequently subjected to a high heating rate, high cooling rate heat-treatment. TEM study shows that Ti is relatively stable with AlZn, comparing with Si-AlZn system. Ti start to dissolve into AlZn after 560°C 10 sec Gleeble heat treatment. The 500 nm Ti film still exists after 600°C 30 sec heat treatment with 5 min heating up process (image c). When temperature increases to 700°C, Ti dissolves into AlZn rapidly. No separate Ti film is observed after 700°C 10 sec Gleeble heat treatment. The results are useful in process optimization in applications of AlZn coatings.

#### 10:20 AM

#### Microstructural Features and Nanoindentation of AlN Coatings Manufactured by Room Temperature Vacuum Cold Spraying: Kyeong-Ho Baik<sup>1</sup>; *Chang-Hyun Park*<sup>1</sup>; <sup>1</sup>Chungnam National University

In this study, aluminum nitride (AIN) coatings on a glass substrate using a room temperature vacuum spraying have been evaluated in terms of their film structure and mechanical properties. Submicron AlN particles were accelerated by gas flow in the nozzle up to velocity of several hundred m/s. During interaction with substrate, these particles formed a thick ( $1\sim50\mu$ m), uniform, hard and dense AlN coating. XRD and TEM analyses revealed that the AlN coating retained an initial crystal structure of wurtzite and severe particle impact formed nanocrystalline AlN grains of 20-100nm. Optimization of processing parameters including particle size, gas flow rate and nozzle-to-substrate distance produce a much dense AlN coating which had a modulus of ~150 GPa and a hardness of ~10 GPa.

#### 10:35 AM

Oxide Formation in Dew-Point Controlled High-Mn Steel: Youngwoon Kim<sup>1</sup>; Woong-Pyo Hong<sup>1</sup>; *Sung-Il Baek*<sup>1</sup>; Sung Dae Kim<sup>1</sup>; Gyo-Sung Kim<sup>2</sup>; Sun-Ho Jeon<sup>2</sup>; Kwang-Guen Chin<sup>2</sup>; Chang-Seok Oh<sup>3</sup>; <sup>1</sup>Seoul National University; <sup>2</sup>POSCO; <sup>3</sup>Korea Institute of Materials Science

Oxidation behavior was investigated by changing dew-point (DP) temperature in high-Mn steel. DP temperatures were changed from 0 °C to -60°C in the gas mixture of  $H_2/N_2$  ratio of 15:85 in volume. Morphologies and the distributions of the Mn-O oxides were dramatically changed above and below -20°C of DP. Mn depletion regions were observed with the DP above -20°C and the distribution of the surface oxide formed small grains with oxides at the grain boundaries. It is believed that the surface Mn atoms were consumed to form Mn-O oxides changing surface phase as ferrite. Surface and internal oxidation revealed that the fast diffusion path along the grain boundaries of ferrite. Thin surface oxide layers were found in the steel surface processed in the DP below -40°C.

#### 10:50 AM Tea Break

### Symposium G: Thin Films and Surface Engineering: Corrosion

Tuesday AM August 3, 2010 Room: 8 Location: Cairns Convention Centre

Session Chairs: Sung-Il Baek, Seoul National University; Thierry Grosdidier, Dalian University of Technology

#### 10:50 AM

High Temperature Corrosion of Fe-Cr, Fe-Al, Fe-Si and Fe-Si-Al Alloys in CO<sub>2</sub>-H<sub>2</sub>O Gases: Thomas Gheno<sup>1</sup>; Huan Li<sup>1</sup>; Jianqiang Zhang<sup>1</sup>; *David Young*<sup>1</sup>; <sup>1</sup>University of New South Wales

Iron and model alloys containing 2.25, 9, and 20 wt% Cr, 2, 4 and 6 wt% Al, 1, 2 and 3 wt% Si, and dilute Fe-Si-Al ternaries were reacted in dry and wet Ar-CO<sub>2</sub> gases at 800°C. External oxide scales grew on Fe according to fast, linear kinetics in dry CO<sub>2</sub>. Additions of H<sub>2</sub>O accelerated the reaction until steady-state parabolic kinetics were achieved. High Cr content alloys developed slow-growing chromium-rich oxide scales. Dry CO, mixtures produced faster rates than wet gas mixtures. Lower Cr allovs developed thicker iron oxide scales. featuring cavities, cracks and poor adherence, and sustained internal oxidation. The presence of H<sub>2</sub>O led to even higher oxidation rates. Aluminium additions to iron of up to 4 wt% provided no protection, but instead caused internal oxidation. A level of 6 wt% significantly slowed oxidation by forming a continuous alumina layer. Silicon additions had little effect, apart from promoting internal oxidation. However, simultaneous alloying with aluminium and silicon strongly depressed corrosion rates. The effectiveness of different alloy additions is discussed, along with the effects of water vapour and carbon activities, in the context of oxyfuel combustion technology.

#### 11:05 AM

Influence of Cryomilling on Microstructure, Phase Stability and Oxidation Behavior of NiCrAIY Bond Coats in Thermal Barrier Coatings: Experimentation and Mechanistic Investigation: Kaka Ma<sup>1</sup>; Julie Schoenung<sup>1</sup>; <sup>1</sup>University of California, Davis

Improved thermal cycling lifetime has been observed in thermal barrier coatings (TBCs) with cryomilled NiCrAlY bond coat. To understand this improved behavior, a robust experimental investigation is coupled with mechanistic explanations to describe the influence of cryomilling on microstructure, phase stability and oxidation behavior of the bond coat. It is found that cryomilling results in two significant changes in the NiCrAIY bond coat: unintentional Fe additions and creation of a homogeneous distribution of ultrafine oxide/ nitride dispersoids. Through extensive microstructural analysis combined with computational simulation using Thermo-Calc® software, it is determined that the presence of Fe stabilizes the high temperature  $\gamma$  and  $\beta$  phases in the NiCrAIY bond coat, corresponding to a decrease in the transformation temperature. The results are explained on the basis of the Gibbs free energy for the individual phases. Characterization of the thermally grown oxide (TGO) in TBCs after isothermal oxidation with rigorous statistical evaluation indicate that the TGOs in the TBCs with the cryomilled bond coats are more uniform in thickness and slower growing. Both behaviors are attributed to the more homogeneous distribution of oxide dispersoids, which are a direct result of the cryomilling, yet remain stable after extensive thermal exposure.

#### 11:20 AM

Corrosion Behavior of 6000 Series Aluminum Alloys Produced by Conventional and Powder Extruded Process through Electrochemical Impedance Method: Satoshi Sunada<sup>1</sup>; Norio Nunomura<sup>1</sup>; Kazuhiko Majima<sup>1</sup>; <sup>1</sup>University of Toyama

In this study, the electrochemical measurements such as the potentiodynamic polarization and the electrochemical impedance spectroscopy (EIS) tests were carried out in order to clarify the corrosion behavior of the aluminum alloys fabricated by P/M process compared with those by I/M process, using 6000 system aluminum alloys. Two kinds of aluminum specimens; one is fabricated by the conventional I/M specimen and the other is fabricated by the P/M process, were used for the electrochemical experiment in the sulfuric acid solution with 0.5kmol/m<sup>3</sup> concentration. Both of I/M and P/M specimens showed the linear relationship between the electrochemical potential (*E*) and the common logarithm of current density ( $i_{corr}$ ) in the cathodic region where Tafel law was recognized irrespective of stirring of the test solution though the  $i_{corr}$  was slightly increased by stirring for both specimens. On the other hand, in the anodic region, both of the two specimens indicated the almost the same  $i_{corr}$  irrespective of stirring. These experimental results interpret that the corrosion is controlled by the chemical



reaction. The EIS test indicated that the maximum charge transfer resistance ( $R_{\rm cl}$ ) was observed at -0.55V which is 0.11V higher potential than the corrosion potential ( $E_{\rm corr}$ ) for both of the two specimens.

#### 11:35 AM

Effect of Ar Bubbling during MAO Process of Magnesium Alloy on Corrosion Resistance: *Yonghwan Kim*<sup>1</sup>; Junghoon Lee<sup>1</sup>; Jaemin Kim<sup>1</sup>; Jungmin Han<sup>1</sup>; Wonsub Chung<sup>1</sup>; <sup>1</sup>Pusan National University

Micro-arc oxidation (MAO) is one of the representative surface treatments of magnesium alloys for improving corrosion resistance. In this work, a modified method for MAO process was tried. Here, Ar gas, as a promoter of plasma, was bubbled on specimen during the anodizing. In experiment, AZ31B alloy plate was used. Anodizing was conducted in diluted KOH electrolyte at  $5\pm3176$ ;C for 10 minutes with or without Ar gas bubbling, and the applied current density was 10ASD. The corrosion resistance was evaluated by polarization tests and electrochemical impedance spectroscopy (EIS). During the anodizing, the voltages grew up approximately 170 V in both case. From results of polarization test, it was found that anodic current density of MAO with Ar bubble is significantly lower than that of MAO with Ar bubble. EIS results also revealed that corrosion resistance of MAO with Ar bubble is higher than that of MAO without Ar bubble. Therefore, it was concluded that MAO with Ar bubble, a modified method, could improve corrosion resistance of magnesium alloys successfully.

#### 11:50 AM

Elecrochemical Response of ZrO<sub>2</sub>-Incorporated Oxide Layer on Magnesium Alloy Prepared by Plasma Electrolytic Oxidation: *Seung Namgung*<sup>1</sup>; Young Gun Ko<sup>2</sup>; Ki Ryong Shin<sup>1</sup>; Bongyoung Yoo<sup>1</sup>; Dong Hyuk Shin<sup>1</sup>; <sup>1</sup>Department of Metallurgy and Materials Science, Hanyang University; <sup>2</sup>School of Materials Science and Engineering, Yeungnam University

In current automobile and electronic industries, the use of magnesium alloys where energy and weight saving are attainable is increasing. Despite their light weight, there has been an inherent drawback arising from the surface vulnerable to be oxidized with ease, specifically under corrosive environments. To protect magnesium alloys from corrosion, the present work deals with the electrochemical response of the oxide layer on magnesium alloy specimen prepared by plasma electrolytic oxidation (PEO) method in an electrolyte containing zirconia powder. Surface observation evidences that a number of zirconia particles are effectively incorporated into oxide layer, resulting in higher oxidation resistance. This is a result of micro-arc during PEO treatment in spite of no polarization of zirconia. From the results of potentio-dynamic tests in 3.5% NaCl solution whose pH is 7.8, corrosion behavior of the PEO-treated magnesium alloy is also discussed in relation to Stern-Geary equation.

#### 12:05 PM

Electrochemical Characteristics of Sintered Duplex Ferritic-Austenitic Stainless Steels Produced by Powder Metallurgy Process: Satoshi Sunada<sup>1</sup>; *Keisuke Arai*<sup>1</sup>; Masahisa Miyahara<sup>2</sup>; Katsuhiko Mori<sup>2</sup>; Kazuhiko Majima<sup>1</sup>; <sup>1</sup>University of Toyama; <sup>2</sup>DIAMET Co., Ltd.

The sintered stainless steel produced by the powder metallurgy process (P/M) has attracted a growing interest because it has the advantage of better formability to fabricate complex shape products without machining and welding. The four sintered stainless steel samples: i.e., the mono-phase SUS304L SS P/M sample (hereafter denoted as 304L), the mono-phase SUS316L SS P/M sample (hereafter denoted as 316L), the duplex-phase SUS316L and SUS434L SS P/M sample (hereafter denoted as 316L+SUS434L), and the duplex-phase SUS316L and SUS434L SS P/M sample with copper (hereafter denoted as 316L+434L+Cu) were used in this experiment, and their corrosion behavior was investigated through the electrochemical procedure. It was confirmed from the potentiodynamic polarization test that their corrosion behavior was clearly classified into two groups. The one is for the mono-phase stainless steel group and the other is for the duplex-phase stainless steel group. Both corrosion current density  $(I_{ror})$  and passivation current density  $(I_{r})$  for the latter group were smaller than those for the former group, and especially the duplex-phase 316L + 434L SS sample with copper (316L+434L+Cu) showed the lowest value. This implies that the duplex-phase 316L + 434L SS sample with copper (316L+434L+Cu) has the highest corrosion resistance.

#### 12:20 PM

#### Electrochemical Corrosion Behaviors Using Double Glow Forming TiN Multi-Permeation Layer: Yan Ping Liu<sup>1</sup>; 'Taiyuan University of Technology

A new process technique that TiN permeation layer consisted of diffusion and deposition layer was synthesized on the surface of mild carbon steel has been firstly introduced, mainly according to plasma point discharge, hollow cathode effect and reactive vapor deposition technique. The surface appearance of this layer formed was uniform, compact and fine TiN cellular structure; a diffusion transitional region existed between the deposition layer and substrate; the surface texture was TiN deposition layer + TiN + Ti solid solution diffusion layer; from

surface to inner Ti and N elements appeared graded distribution. X-ray diffraction results showed that the surface of multi-permeation layer was pure TiN layer and the diffraction peak of (200) crystal face was the strongest possessing obvious preferred orientation. This paper also mainly investigated the electrochemical corrosion behaviors of this multilayer. The polarization curves of specimens in  $10\% H_2SO_4$  corrosive media were measured. The eroded surface morphologies were also surveyed by SEM. The results indicated that the erosion resistance of multi-permeation layer was increased many times than PVDTiN and a mild steel substrate, and almost equaled to that of compound process TiN layers.

#### 12:35 PM

#### Hot Corrosion Behavior of a Novel Enamel-Based Thermal Barrier Coating: Mingli Shen<sup>1</sup>; *Shenglong Zhu*<sup>1</sup>; Li Xin<sup>1</sup>; Fuhui Wang<sup>1</sup>; <sup>1</sup>Institute of Metal Research

The coating was prepared by two steps. Firstly, a 20 µm bond coat was deposited on a Ni-based superalloy K444 using magnetron sputtering. The chemical composition of the bond coat was same as the substrate. Secondly, the specimens were deposited with enamel-zirconia powder mixture by air spray, then fired in air at 1000°C. The hot corrosion tests were carried out at 900°C for 100 h. The salt used in the tests was 25% NaCl + 75% Na<sub>2</sub>SO<sub>4</sub>. The uncoated K444 specimens kept losing weight while the coated specimens got a little mass gain during tests. Spalltion of oxide scales on the uncoated specimens was observed. XRD analysis indicated the coatings on the coated specimens after hot corrosion test were t-ZrO, and NaAlSi<sub>3</sub>O<sub>6</sub>, while the oxide scales formed on the uncoated K444 were  $Cr_2O_3$ , TiO<sub>2</sub>, NiCr<sub>2</sub>O<sub>4</sub> and Na<sub>2</sub>Cr<sub>2</sub>Ti<sub>6</sub>O<sub>16</sub>. Further studies showed that a thin layer of mixture oxides of Al, Ti and Cr formed at the interface between the bond coat and substrate of the coated specimens, while thick multilavered oxide scales formed on the uncoated specimens in accordance with the XRD analysis results. Besides, deep internal oxidation zone was observed on the uncoated specimens.

# Tue. AM

# Symposium I: Biomaterials, Smart Materials and Structures: Ti-Based Biomaterials II

Tuesday AM	Room: 3	
August 3, 2010	Location:	Cairns Convention Centre

Session Chairs: Takao Hanawa, Tokyo Medical and Dental University; Taehyun Nam, Gyeongsang National University

#### 8:30 AM Keynote

Nanotube and Micropore of Ti Alloy Systems for Biocompatibility: Han-Cheol Choe<sup>1</sup>; <sup>1</sup>School of Dentistry, Chosun University

The surface modification techniques for nanopore and micropore have been used to increase the roughness of the implant surfaces for improving bonetissue integration. Cell adhesion and proliferation depend on surface roughness and metal ion dissolution. Micropore and nanotube formation on the Ti oxide are important to improve the cell adhesion and proliferation in clinical use. In this study, micropore and nanotube formation of Ti ternary alloy have been investigated using various condition of micropore and nanotube formation by femtosecond laser and anodization methods. Ternary alloys were prepared by using high purity sponge Ti Ta, Zr and Nb sphere (99.95% wt.%). Micropore formation was performed by femtosecond laser(FS). For micropore texturing on the surface, an amplified Ti: sapphire laser system was used for generating 184 femtosecond laser pulses with the pulse energy over 20 mJ/s at a 1 kHz repetition rate with a central wavelength of 800 nm. Nanotube formation was carried out using potentiostat with three electrodes. Experiments were performed in 1M H3PO4 with small additions of NaF(0.1-0.8wt%). The surface characteristics of nanotube formed and femtosecond laser textured was investigated by FE-SEM, EDX, XRD, XPS, AFM, corrosion test, wettability test and cell proliferation test.

#### 8:50 AM Keynote

# Nanostructured Titanium Biomaterials: Understanding and Applications: *Tae-hyun Nam*<sup>1</sup>; Wong-woo Khang<sup>1</sup>; Yeon-min Lim<sup>1</sup>; <sup>1</sup>Gyeongsang National University

Nanostructured implant materials are considered as promising future biomaterials. Specifically, titanium based nanomaterial is the most widely used in orthopedic, dental and vascular surgeries. Due to the advantage of nanoscale features, treatment with nano porous and nano bump surface features have shown enhanced biocompatibilities, such as adhesion, proliferation and differentiation for bone and vascular cells. In addition, nanotoxicity issue with immune cells (macrophages) is currently paramount interest for determining subsequent tissue cell response on implanted biomaterials. In this study, we demonstrated altered cellular interaction of bone, vascular and immune cells on nanostructured titanium



based alloys/materials through systematic controlling of nanoscale surface features, such as porosity and nanobumps. Furthermore, signal pathway and gen expressions are also introduced for understanding given cellular behavior. All this knowledge will be beneficial for both understanding and designing nanostructured biomaterials for increasing biocompatibility, thus, all these endeavors will lead increment of functionality of biomaterials and will eventually prolong the life time of implanted biomaterials.

#### 9:10 AM

Fabrication of Ti-Based Biodegradable Material Composites Prepared by Spark Plasma Sintering Method: *Eri Miura-Fujiwara*<sup>1</sup>; Takeshi Teramoto<sup>1</sup>; Hisashi Sato<sup>1</sup>; Equo Kobayashi<sup>2</sup>; Yoshimi Watanabe<sup>1</sup>; <sup>1</sup>Nagoya Institute of Technology; <sup>2</sup>Tokyo Institute of Technology

Tue. AM

This study aims at producing porous Ti filled with biodegradable materials for biomedical implants by means of spark plasma sintering method (SPS). To improve bone fixation and to obtain appropriate Young's modulus as a medical implant material, we applied  $\beta$ -tri calcium phosphate ( $\beta$ -TCP) to the Ti-based composite. 50vol%. Ti/ $\beta$ -TCP powder mixtures were sintered by SPS under applied stress of 45MPa with various temperature and holding time. Vickers hardness (Hv) of obtained composite was increased with increasing the holding time up to 10 min, and saturated hardness was approximately Hv = 750, which is extremely higher than bulk Ti. Hv also increased as sintering temperature increased up to 1473 K. From the results of microstructure observations by scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS), O and P containing Ti surrounded around Ti particle, and O diffused into Ti particle to a certain extent. X-ray diffraction results indicated several kinds of Ti-O and/or Ti-P-O formed in the specimen. Results indicated that brittle reaction phases formed during sintering increased the hardness.

#### 9:25 AM

Micromechanical Prediction of Fracture Toughness in Titanium Foams for Biomedical Applications: *Sadaf Kashef*<sup>1</sup>; Peter D. Hodgson<sup>1</sup>; Alireza Asgari<sup>2</sup>; <sup>1</sup>Institute for Technology, Research and Innovation. Deakin University; <sup>2</sup>School of Engineering, Deakin University

A micromechanical model for prediction of fracture toughness in Titanium foam is presented. The Mode I, Mode II and mixed mode fracture toughness are predicted by simulating the crack propagation in the foam. The maximum stress criterion is used to break the strut ahead of the crack tip in the micromechanical model made with finite element method. A semi-empirical formula is derived to predict the Mode I fracture toughness of the Titanium foam. The results for Mode I are verified and validated by comparison with experimental fracture tests of standard CT specimens. The relation between relative density of the foam and effective forces in the crack tip strut are also established. The results show that continuum fracture mechanics concepts can be used to predict the fracture of Titanium foams in biomedical applications.

#### 9:40 AM

Improvement of Mechanical Performance and Biocompatibility of Spinal Implant Rod Made of Beta-Type Ti-Nb-Ta-Zr Alloy: *Mitsuo Niinomi*<sup>1</sup>; Kengo Narita<sup>1</sup>; Masaaki Nakai<sup>1</sup>; Toshikazu Akahori<sup>1</sup>; Harumi Tsutsumi<sup>1</sup>; Kazuya Oribe<sup>2</sup>; <sup>1</sup>Tohoku University; <sup>2</sup>Showa Ika Kohgyo Co., Ltd.

Mechanical properties of beta-type biomedical Ti-29Nb-13Ta-4.6Zr (mass %) (TNTZ), which exhibits non-toxicity and low modulus similar to that of cortical bone, is improved by thermomechanical treatments including sever cold swaging. Simultaneously the biocompatibility of the spinal rod made of TNTZ with living tissue is evaluated using ovine.

#### 9:55 AM

#### MAO-Prepared Hydroxyapatite Coating on Ultrafine-Grained Titanium: Min Qi<sup>1</sup>; Yan Li<sup>2</sup>; <sup>1</sup>Dalian University of Technology; <sup>2</sup>Beihang University

Ultrafine-grained titanium was prepared through the equal channel angular pressing (ECAP). And Ca/P-containing porous titania coating were synthesized on the above ultrafine-grained Ti as well as the common coarse-grained Ti by micro-arc oxidation (MAO) in Ca/P based solution. The amounts of Ca, P and Ca/P ratio of the MAO coating formed on ultrafine-grained Ti were higher than those for coarse-grained Ti samples obviously. Hydroxyapatite and a-Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> phases appeared in MAO coating formed on ECAP-treated Ti with reaction time of 20 min (E20). The following phases can recognized from interface to superficial layer in turn: A dense TiO<sub>2</sub> diffusion layer (about 100 nm); Oxide layer with an average grain size of about 500 nm and small pores in grain boundaries and a mixture layer (about 1 - 2 µm). Bone-like apatite was completely formed on the surface when the above samples when emerged in simulated body fluid within 2 days. In summary, MAO coating formed on ECAP-treated Ti has the optimal combination of mechanical properties and bioactivity.

#### 10:10 AM Break

Symposium I: Biomaterials, Smart Materials and Structures: Ti-Based Biomaterials III

Tuesday AM	Room
August 3, 2010	Locat

n: 3 tion: Cairns Convention Centre

Session Chairs: Hideki Hosoda, Tokyo Institute of Technology; Han-Cheol Choe, School of Dentistry, Chosun University

#### 10:50 AM

Electrodeposition of Collagen to Titanium to Improve Tissue Compatibility: *Takao Hanawa*<sup>1</sup>; Hideki Kamata<sup>1</sup>; Jiyeon Choi<sup>1</sup>; Kei Oya<sup>1</sup>; Yusuke Tsutsumi<sup>1</sup>; Hisashi Doi<sup>1</sup>; Naoyuki Nomura<sup>1</sup>; Keiji Moriyama<sup>1</sup>; <sup>1</sup>Tokyo Medical and Dental University

We have attempted the electrodeposition of collagen to Ti surface to add hard tissue compatibility and soft tissue compatibility. Type I collagen was dissolved into 0.9mass%NaCl solution with a concentration of 10 mg mL-1. The pH was adjusted to 5. The cathodic or anodic potential was charged from open circuit potential to -1.0 V or +0.1 vs. SCE, respectively, at 25°C and maintained at this potential for 30 and 1800 s (DC-cathod or DC-anode). On the other hand, alternating current between -1 and +1 V vs. SCE with 1 Hz was charged for 30 s and 1800 s (AC). After electrodeposition, specimens were ultrasonically rinsed in water for 15 min. The surface was characterized using SPM and XPS. The thickness of the immobilized collagen layer was determined with ellipsometry. The specimens were shacked in water for 72 h to evaluate the durability of the immobilization. Cell culture test using MC3T3-E1 and HaCAT cells were performed for hard and soft tissue compatibilities, respectively. AC electrodeposition is the most effective to immobilize collagen to Ti surface and the collagen forms a network-like layer. The immobilized layer gives both hard and soft tissue compatibilities to Ti.

#### 11:05 AM

Pack Cementation Treatment of Titanium Using Tetracalcium Phosphate Powder for Biomedical Applications: *Kyosuke Ueda*<sup>1</sup>; Hajime Suto<sup>1</sup>; Kaori Nakaie<sup>1</sup>; Takayuki Narushima<sup>1</sup>; <sup>1</sup>Tohoku University

The surface modification of commercially pure titanium by pack cementation treatment using tetracalcium phosphate (Ca4(PO4)2O, TTCP) powder was investigated under various treatment conditions. Pores containing small hydroxyapatite (Ca<sub>10</sub>(PO<sub>4</sub>)<sub>6</sub>OH)<sub>2</sub>, HAp) particles were observed on the surface of the titanium substrate after pack cementation treatment at temperatures of 973 K and 1073 K in air. TTCP powder decomposed to HAp and CaO during the pack cementation treatment. The pores were considered to be formed by the reaction between TiO, and CaO. The TiO, and CaO were formed by oxidation of the titanium substrate and decomposition of TTCP powder, respectively. The decomposition of TTCP is related to H2O in air. The formation of HAp on the surface of titanium substrates was promoted when TTCP slurry or humid air was employed in the pack cementation process. Apatite crystallites with a network pattern were formed on the pack-cementation-treated titanium substrates after the substrates were immersed in Kokubo solution for 43.2 ks; such rapid apatite formation suggests that pack cementation treatment improves the biocompatibility of titanium.

#### 11:20 AM

Phase Stability and Mechanical Properties of Ti-Cr Based Alloys with Low Young's Modulus: *Yonosuke Murayama*<sup>1</sup>; Shuichi Sasaki<sup>1</sup>; Hisamichi Kimura<sup>2</sup>; Akihiko Chiba<sup>2</sup>; <sup>1</sup>Niigata Institute of Technology; <sup>2</sup>Institute for Materials Research, Tohoku University

The Young's modulus of human bone is from 10GPa to 30GPa that is much lower than the metallic material. The large difference of the Young's modulus between the human bone and the metallic implant material may cause the damage. Therefore, beta titanium alloys with low Young's modulus are attractive for biomedical application. This work examines the mechanical properties of Ti-Cr based alloys and focuses on the effect of the varying alloy composition on the microstructure, the Young's modulus, the deformation mechanism and the deformation behavior. The addition of Sn, Al and Zr to the Ti-Cr based alloys has the effect to decrease the Young's modulus very much. These elements suppress the athermal omega phase that forms from the competition between the metastable beta phase and omega phase during quenching. The composition range of the alloys with low Young's modulus is narrow for Cr but wide for Zr. The alloys with the composition where the quenched microstructure transits from martensite to meta-stable beta show low Young's modulus. Moreover, the alloys show the two-step yielding due to the stress-induced transformation.



#### 11:35 AM

Texture and Pseudoelasticity of Ti-Nb-Ge Alloys: *Han-Sol Kim*<sup>1</sup>; Won-Yong Kim<sup>1</sup>; Akihiko Chiba<sup>2</sup>; <sup>1</sup>Korea Institute of Industrial Technology; <sup>2</sup>Tohoku University

In this study, the effects of two types of cold rolling methods on texture formation and pseudoelasticity of Ti-Nb-Ge alloys were investigated in order to find suitable texture component for satisfiable pseudoelastic property since deformation processing of many metallic materials gives rise to vary crystallographic anisotropy. Ternary Ti-Nb-Ge alloy buttons were prepared by vacuum arc melting technique. The alloy buttons were then rapidly quenched in iced water after solution treated at 1273K for 30min. Conventional rolling and differential-speed cold rolling methods were employed. And then they the cold rolled sheets were annealed at 1123K for 1.8ks, 3.6ks, 7.2ks and 14.4ks, respectively. Cyclic tensile test was carried out to display a pseudoelastic behavior at room temperature. The major texture components of conventional rolling, differential-speed rolling are characterized by {001}<110> and <001>// ND fibre texture components, respectively. With increasing annealing time, it is found that {114}<221> or {110}<211> texture components were newly formed in differential-speed rolled sample while <110>//RD fibre continuously developed in conventional rolled sample. It is suggested that development of <110>//RD a-fiber texture component is favour to increase the martensitic recovery strain resulting in better pseudoelstic property.

#### 11:50 AM

Phase Constitution and Mechanical Properties of Ti-(Cr, Mn)-Sn Biomedical Alloys: Yasuhiro Kusano<sup>1</sup>; Tomonari Inamura<sup>1</sup>; Hideki Hosoda<sup>1</sup>; Shuichi Miyazaki<sup>2</sup>; <sup>1</sup>Tokyo Institute of Technology; <sup>2</sup>University of Tsukuba

In order to produce new  $\beta$ (bcc) Ti alloys for medical applications, systematic investigation has been done for the Ti-Nb, Ti-Mo and Ti-Sn alloys containing 3dtransition-metal-elements to evaluate the effects of the 3d additional elements on the phase constitution and mechanical properties. For example, it was revealed that the addition of these 3d-transition metal elements stabilizes  $\beta$  phase, and that 3mol% Mn addition enhances the ductility and strength of Ti-Cr-Sn alloys. Then, Ti-Cr-Sn alloys containing Mn were judged to be one of hopeful alloy systems. In this study, effects of Mn substitution for Cr on room-temperature phaseconstitution and mechanical properties of Ti-Cr-Sn alloys were investigated. Ti-7mol%(Cr, Mn)-3mol%Sn alloys were selected and prepared by Ar arc melting method followed by a set of thermomechanical treatments. It was found that these alloys exhibited sufficient cold workability. X-ray diffraction analysis showed that Ti-7Cr-3Sn consisted of the  $\beta$  phase while Ti-7Mn-3Sn was two phases of  $\alpha$ ' (hcp) and  $\beta$ . Mechanical properties obtained by tensile tests are also presented.

#### 12:05 PM

Microstructure Characterization of Laser Welded Near-ß Titanium Alloy 'TLM' under Different Process Conditions: Alex Buddery<sup>1</sup>; Gui Wang<sup>2</sup>; Zhentao Yu<sup>3</sup>; Matt Dargusch<sup>2</sup>; Samih Nabulsi<sup>4</sup>; <sup>1</sup>CAST CRC, School of Mechanical and Mining Engineering, The University of Queensland; <sup>2</sup>Defence Materials Technology Centre and CAST CRC, School of Mechanical and Mining Engineering, The University of Queensland; <sup>3</sup>Northwest Institute for Nonferrous Metal Research; <sup>4</sup>Cook Medical

The biomedical titanium alloy TLM (Ti-25Nb-3Zr-3Mo-2Sn) was recently developed using non-toxic alloying elements to achieve low elastic modulus, good strength and good processability making it an ideal candidate for hard tissue replacement. In addition to these properties the alloy has also been found to exhibit psuedoelastic properties due to a stress induced ß to a" martensitic transformation. Further research into the psuedoelastic properties of TLM has show that it would also be a good candidate for vascular stents due to the high strain requirement. A desirable manufacturing route for vascular stents involves laser welding however this can have a detrimental effect on the microstructure and properties of the component. In this paper the microstructure formed during welding TLM sheets under various conditions is reported. The effect on deformation behaviour with respect to the psuedoelasticity of the alloy is also assessed. The TLM alloy appears to show good resilience to laser welding which makes it an even more promising candidate for use in vascular stents.

### Symposium J: Materials Characterisation and Evaluation: Mechanical Properties II

Room
Locat

m: 1 ation: Cairns Convention Centre

Session Chairs: Mark Hoffman, The University of New South Wales; Oliver Kraft, KIT

#### 8:30 AM Keynote

Characterization of Crack-Tip Dislocations and Their Effects on Materials Fracture: Kenji Higashida<sup>1</sup>; Masaki Tanaka<sup>1</sup>; Sunao Sadamatsu<sup>1</sup>; <sup>1</sup>Kyushu University

Strong dependence of dislocation mobility on temperature or a strain rate induces a brittle-to-ductile transition (BDT). Pioneering works, using silicon single crystals as a model crystal, showed that the BDT is strongly affected by dislocation structures developed around a crack tip. It is essential to reveal its three dimensional structure in order to understand the mechanism behind the BDT. We have revealed the structure of crack tip dislocations, using twodimensional observation, although it was restricted to the case with low dislocation density. In the present study, therefore, three-dimensional observations of crack tip dislocations in high density were performed by combining high voltage electron microscopy and electron tomography. (001) silicon single crystals were employed. <110> cracks were introduced by a Vickers tester. The sample was heated at high temperature for one hour in order to introduce dislocations at the crack tip. The tilting series of bright field image reveals 3D structure of the crack tip dislocations such as cross slips. The determination of Burgers vector indicated dislocation reactions at the crack tip. The cross slips and reactions enhance the increase in the number of dislocations, which plays an important role on the onset of the BDT.

## 8:50 AM

**Formation and Mechanical Properties of Intermetallic Compounds in Sn-Cu High-Temperature Lead-Free Solder Joints**: *Dekui Mu*<sup>1</sup>; Hideaki Tsukamoto<sup>1</sup>; Han Huang<sup>1</sup>; Kazuhiro Nogita<sup>1</sup>; <sup>1</sup>School of Mechanical and Mining Engineering, The University of Queensland

High-temperature lead-free solders are important materials for future electrical and electronic devices due to the increasingly more environmental concern. For the successful use of such solder materials, a comprehensive understanding of the formation and mechanical properties of intermetallic compounds (IMCs) in high-temperature lead free solders is essential. In this work, the effect of nickel addition on the formation and mechanical properties of IMCs in Sn-3/4/7wt%Cu high-temperature solders dipped on copper substrate was investigated using nanoindentation and electron microscopy. The nanoindentation results demonstrated that the nickel addition can improve the elastic modulus and hardness of the formed (Cu, Ni)<sub>6</sub>Sn<sub>5</sub> in the dipped specimens. For comparison, the elastic modulus and hardness of the bulk Cu<sub>6-x</sub>Ni<sub>x</sub>Sn<sub>5</sub>(x = 0, 0.5, 1, 1.5, 2) were measured too. A relationship between the nickel content and the mechanical properties of the formed IMCs was thus established. The effect of nickel addition on the formation and layer growth of IMCs was also discussed based on the SEM and EDS analyses.

#### 9:05 AM

Study on Stress Distribution near Crack Tip in Beryllium Compact Tension Specimen: *Ping Dong*<sup>1</sup>; Ruiwen Li<sup>1</sup>; <sup>1</sup>China Academy of Engineering Physics

In order to evaluate the fracture characteristic near the crack tip of beryllium specimen, beryllium compact tension specimen with plane strain state is designed. The stress distribution near the crack tip is measured at different loading level by X3000 stress analyser. Moreover, a finite element model for calculated the stress and strain fields in beryllium compact tension specimen has also been set up. As a result, the stress and strain distribution near the crack tip at different loading has been calculated by this model. The results show that they are basically identical, except that the calculated stress is larger than the measured stress near the crack tip. According to the critical tension loading of beryllium specimen, the maximum plastic strain and the radius of plastic zone near the crack tip is about  $0.018\mu$ c and the maximum radius of plastic zone is about 0.3mm. Altogether, the fracture toughness of beryllium is obtained, which is about 19.1MPa·m<sup>1/2</sup>.





#### 9:20 AM

Tool Failure Criteria while Drilling Titanium Alloys: *Suresh Palanisamy*<sup>1</sup>; Luo Cong<sup>2</sup>; Viktor Verijenko<sup>3</sup>; Stuart McDonald<sup>1</sup>; Robert Owen<sup>4</sup>; Matthew Dargusch<sup>3</sup>; <sup>1</sup>CAST & DMTC; <sup>2</sup>University of Queensland; <sup>3</sup>Defence Materials Technology Centre; <sup>4</sup>Millatec Engineering Pty. Ltd.

This paper presents a feasible machining test to measure, compare and predict the machinability of different titanium alloys. A drilling test was developed and investigated on the two most common grades of titanium, commercial purity and Ti<sub>6</sub>Al<sub>4</sub>V. The experiments and analysis revealed that tool wear followed a characteristic pattern for all machining conditions investigated. When machining Ti<sub>6</sub>Al<sub>4</sub>V, tool life was shorter and cutting forces higher compared with commercial purity Ti. Paradoxically, despite the more difficult machining, Ti<sub>6</sub>Al<sub>4</sub>V samples had better surface integrity than commercial purity samples. A procedure was developed that could be incorporated into a real-time process monitoring device to warn of imminent tool failure.

#### 9:35 AM

# A Study of Toughness Degradation in CA6NM Stainless Steel: Yoshitaka Iwabuchi<sup>1</sup>; Isao Kobayashi<sup>1</sup>; <sup>1</sup>Kushiro National College of Technology

Tue. AM

The mechanism of toughness degradation during slow cooling in the austenite range was studied in CA,NM stainless steel, 13% Cr-4% Ni soft martensitic stainless steel. The variation of toughness, fracture mode and microstructural features were examined by means of cooling rate and isothermal heating in the austenite range together with chemical composition. Toughness degradation was referred to as the increases of FATT and intergranular fracture when those steels were cooled slowly after austenitizing and isothermally heated in the austenite range. The embrittlement was found to be related the intergranular fracture and the precipitation of carbide along prior austenite grain boundaries. Its fracture surface was characterized by mosaic-like markings when the carbide precipitation got to increase. The precipitation of intergranular carbides was resulted from the decrease of carbon solubility in the austenite. Reducing carbon, silicon and phosphorus and increasing molybdenum improve the toughness degradation as the result of retarding the precipitation of Cr<sub>23</sub>C<sub>6</sub> type carbide along grain boundary. Phosphorus was presumed to facilitate the nucleation of intergranular carbide and promote the carbide precipitation and then cause the grain boundary brittleness

#### 9:50 AM

Effects of Aging Parameters on the Mechanical Properties in Corson Alloy System with High Contents of Ni and Si: *Genjiro Hagino*<sup>1</sup>; Hayao Eguchi<sup>1</sup>; Yoshimasa Takayama<sup>2</sup>; Hajime Kato<sup>2</sup>; <sup>1</sup>Miyoshi Gokin Kogyo Co., Ltd.; <sup>2</sup>Utsunomiya University

The total concentrations of Ni and Si in Cu-Ni-Si alloys, which are called Corson alloy system, are generally less than 5.0 mass% because of the requirements of formability for applications of electric industry. On the other hand, some kinds of machine parts demand higher concentrations of Ni and Si leading to higher hardness and tensile strength. In the present study, the influences of solution treatments, prior cold drawings and aging treatments on mechanical properties in Corson alloy with high concentrations more than 8.0 mass% of Ni and Si have been investigated. As a result of the optimization of conditions, the maximum hardness and tensile strength reached 335HV and 968MPa, respectively. Effects of temperatures of solution treatment and aging on the mechanical properties were analyzed based on solid solute contents in matrix phase, which were estimated by using the linear analysis and calculated from the electrical conductivity. The solute contents were also measured directly by SEM-EDS. Consequently, the precipitation hardening of the alloys was governed mainly by the solute content of Ni and Si in matrix phase in solution treatment. The hardness depended on not only the solute content but also the excess of Ni and Si in the alloys.

### 10:05 AM Invited

Controlling Factors for Sliding Wear in Plain-Carbon and Alloy Steels: Jong Chul Kim<sup>1</sup>; Narae Yoon<sup>1</sup>; Hyouck-Woo Kwon<sup>1</sup>; *Yong-Suk Kim*<sup>1</sup>; <sup>1</sup>Kookmin University

Controlling factors for sliding wear in plain-carbon and low-alloy steels were investigated. The low-alloy steels contained various amount of C, Mn, Cr, Mo, Ti, and B. Some of the steels were heat treated under different conditions to vary their microstructure. Effects of initial mechanical properties and microstructure on the sliding wear of the steels were examined to find out the controlling factors for the wear. Dry sliding wear tests of the steels were carried out using a pinon-disk wear tester at a fixed load of 100 N at room temperature. AISI 52100 bearing steel and alumina balls were employed as a counterpart. Tensile tests and hardness measurement of the steels were carried out, and phase analysis of the steels' microstructure was performed for the investigation. Neither the initial hardness nor the strength (both yield strength and tensile strength) were successful in characterizing the wear of the steel. Correlation between microstructure (morphology and volume fraction of constituting phases) and the wear rate were explored in connection with strain-hardening at the wearing surface and deformation beneath the surface (subsurface deformation-layer formation).

#### 10:20 AM

Wear Resistance and Microstructural Study of Diamond Coated WC Tools: James Boland<sup>1</sup>; Xing Li<sup>1</sup>; Roger Rassool<sup>2</sup>; Colin MacRae<sup>1</sup>; N.C. Wilson<sup>3</sup>; Stefanie Elbracht<sup>2</sup>; Vladimir Luzin<sup>4</sup>; Paolo Imperia<sup>4</sup>; Bryn Sobott<sup>2</sup>; <sup>1</sup>CSIRO; <sup>2</sup>University of Melbourne; <sup>3</sup>CSIRO Process Science and Engineering; <sup>4</sup>ANSTO

Diamond composite materials are classified as superhard and exhibit exceptional abrasive resistance. Cemented tungsten carbide tools with a thick coating of diamond composite material are finding increased usage in materials cutting operations in manufacturing, mining, minerals, gas and petroleum exploration and civil construction industries. Two major advantages derived from these coated tools are: (a) increased wear resistance and hence increased life-span of these tools with their concomitant greater productivity and (b) their proven ability to handle "difficult-to-machine" materials as well as high-strength, extremely abrasive materials such as quartz-rich rocks, granites and basalts so often encountered in rock cutting and drilling operations. In this research, the variability of the wear resistance of diamond composite coated tungsten carbide cutting elements is correlated with microstructural variations. A detailed study of the microstructure and distribution of phases was performed using x-ray and cathodoluminescence imaging on an EPMA, direct x-ray imaging, XRD and Raman spectroscopy as well as residual stress measurements using neutron diffraction.

10:35 AM Tea Break

### Symposium J: Materials Characterisation and Evaluation: Fatigue and Fracture

Tuesday AM	Room: 1
August 3, 2010	Location: Cairns Convention Centre

Session Chairs: Kenji Higashida, Kyushu University; James Boland, CSIRO

#### 10:50 AM Keynote

The Critical Role of Microstucture in the Very High Cycle Fatigue Behavior of Advanced Structural Materials: J. Wayne Jones<sup>1</sup>; Jiashi Miao<sup>1</sup>; Christopher Szczepanski<sup>2</sup>; Anish Kumar<sup>1</sup>; Tresa Pollock<sup>1</sup>; <sup>1</sup>University of Michigan; <sup>2</sup>Universal Technology Corporation

Very High Cycle Fatigue (VHCF), with fatigue lifetimes in the gigacycle regime, is dominated by fatigue crack initiation, especially in advanced structural alloys where porosity, inclusions and other processing related features are minimized. At the low cyclic stresses associated with VHCF, microstructure heterogeneities such as variability in grain size and spatial distribution and local texture variations can play a dominant role in strain localization and subsequent fatigue crack initiation. We examine the role of microstructure variability on VHCF behavior using ultrasonic fatigue. The critical role of grain size distribution, grain orientation and grain clusters, as well as the presence of twins on fatigue crack initiation is examined in the polycrystalline nickel base superalloy Rene 88DT. The role of microtexture on the variability in fatigue behavior in the titanium alloy Ti-6%Al-2%Sn-4%Zr-6%Mo is also described. Finally, the recent development of a new methodology for in-situ characterization of fatigue damage accumulation in the VHCF regime using nonlinear ultrasonic measurements via analysis of the feedback signal of a closed-loop ultrasonic fatigue system is described. The use of this technique in identifying the initiation lifetimes will be discussed.

#### 11:10 AM Keynote

In Situ SEM Investigation of Deformation and Fracture at Small Scales: *Oliver Krafi*<sup>1</sup>; Reiner Monig<sup>1</sup>; Andreas Sedlmayr<sup>1</sup>; <sup>1</sup>Karlsruhe Institute of Technology

At micron scale, the study of mechanical properties is not straightforward due to issues related to sample preparation and handling. On the other hand, it is important to study the mechanical properties at the relevant scale since they are subject of strong size effects on strength and plasticity of metallic materials. In the last few years, micro-compression tests on sub-micron single-crystalline metallic pillars have gained quite some attention as the observed strong size effect in a nominally uni-axial loading situation has led to a debate about the underlying deformation mechanisms. In this paper, we will report recent results of ex and in situ micro-compression tests. In addition, we have made an effort to conduct in situ tensile experiments on metallic nanowires. The tests are conducted in a dual-beam scanning electron microscope and focused ion beam, where specimen manipulation, transfer, and alignment are performed using a manipulator. Results



shown will include tests on single-crystalline Cu and Au nanowires. Typically, fracture of the nanowires occurs without much homogeneous ductile deformation. Preliminary results indicate that the measured strengths exhibit a size effect, which will be discussed in the context of a statistical analysis with respect to the specimen size.

#### 11:30 AM Keynote

#### Evaluation of the Reliability of Advanced Interconnect Materials through Electrical Testing: *Robert Keller*<sup>1</sup>; David Read<sup>1</sup>; Mark Strus<sup>1</sup>; <sup>1</sup>NIST

We describe the use of rapid thermal cycling, as induced by precisely controlled AC electric current, to evaluate electrical, mechanical, and thermal damage in advanced interconnect systems. Controlled joule heating will be shown to provide a means for inducing plasticity and diffusive damage in damascene copper, similar to that found during thermal fatigue and stress voiding tests. This method is attractive due to its ability to test buried structures of virtually any geometry and dimensions, given electrical access. We will show how different damage modes can be induced by varying test conditions such as current density or frequency. A particular strength of this method is that it can not only readily assess interactions between different primary stressing conditions, such as effects of stress voids on electromigration, but it can distinguish different forms of damage, such as stress voids and electromigration voids. We will also present preliminary results using this test method to evaluate damage formation in carbon nanotube interconnect systems, with emphasis on the role of carbon/metal interfaces on conductor lifetime.

#### 11:50 AM

Microstructural Damage in Near-Electrode Regions in Lead Zirconate Titanate (PZT) and BNT-BT Induced by Electric Fatigue: Mark Hoffman<sup>1</sup>; Zhenhua Luo<sup>1</sup>; <sup>1</sup>The University of New South Wales

Cyclic electric loading on Lead Zirconate Titanate (PZT) leads fatigue of the material and degradation of piezoelectric performance. Microstructural changes in the near-electrode region of PZT during the course of electric fatigue are investigated. It is found that those regions contribute significantly to the loss of ferroelectricity after electric fatigue. By removing near electrode regions, the loss of polarization can be restored. A profile is made on the change of ferroelectric properties against the depth of removal regions. The characteristics of those regions are observed to be changed significantly compared to the bulk, including generation of microcrack and loss of domain structure. Those changes of microscopic properties in near electrode regions affect the macroscopic behaviours of PZT materials, such as decrease of polarization and increase of coercive field. The damaged near electrode regions are believed to cause most of the polarization loss during electric fatigue, as the effective electric field applied on the PZT bulk is reduced by the screening from damaged regions. The results indicate the importance of near electrode region attributed to the ferroelectric behaviours of PZT, and further improve the understanding of the causes in electric fatigue of PZT.

#### 12:05 PM

Fatigue Fracture Behavior of ARB Processed Aluminum: *Hiromoto Kitahara*<sup>1</sup>; Takuya Horike<sup>1</sup>; Masayuki Tsushida<sup>1</sup>; Shinji Ando<sup>1</sup>; Nobuhiro Tsuji<sup>2</sup>; <sup>1</sup>Kumamoto University; <sup>2</sup>Kyoto University

Fatigue fracture behavior of the ultrafine grained Al fabricated by the accumulative roll bonding (ARB) of 6-cycle was investigated. The ARB processed sheet had elongated grains, and the mean spacing of the elongated grain was 182 nm. Fatigue strengths of the ARB processed sheet were higher than those of the starting sheet. The improvement of fatigue strength resulted from that of the 0.2% proof stress. On the other hand, at the stress amplitude of 80MPa, fatigue life also significantly increased after the 6-cycle of the ARB. It is known that the fatigue crack propagation process mainly occupy the fatigue life. The crack propagation behavior was investigated by the fatigue crack growth test. As a result, the fatigue crack in the ARB processed sheet starts to propagate at the low load, but its rate is slow. The slow fatigue crack growth rate is also one of the effects on the long fatigue life.

#### 12:20 PM

# An Evaluation of Thermal Fatigue Resistance of Tool Materials: Stefan Gulizia<sup>1</sup>; Darryl Jones<sup>1</sup>; Mahnaz Jahedi<sup>1</sup>; <sup>1</sup>CSIRO

Thermal fatigue cracking is one of the main failure mechanisms of tool failure during high pressure die casting (HPDC) of aluminium alloys. A network of cracks form on the surface of the tool as a direct result of the temperature gradient, temperature cycling, and mechanical loading, resulting in the deterioration of tool surfaces and cast products. This research is conducted to study the thermal fatigue resistance of different hot work tool steels and clad material deposited by GTA welding using a new thermal fatigue test rig based on cyclic induction heating and external cooling. The specimens were periodically analysed and the microstructure, hardness profile and the surface cracks are analysed. The results showed significant differences in thermal fatigue resistance of tested materials and the resistance of H13 tool steel is slightly superior to that of maraging steel weld.

#### 12:35 PM

Thermo-Mechanical Fatigue Evaluation of a Ferritic Stainless Steel by Using the Miniature and Standard Samples: *Won Jon Yang*<sup>1</sup>; Jong Hoon Lee<sup>1</sup>; Keyong Sik Cho<sup>1</sup>; Dongyi Seo<sup>2</sup>; Scott Yandt<sup>2</sup>; Yong Jun Oh<sup>3</sup>; <sup>1</sup>Korea Institute of Materials Sciences; <sup>2</sup>National Research Council; <sup>3</sup>Hanbat University

The thermomechanical fatigue (TMF) behaviour of a ferritic automotive grade stainless steel was investigated using two different sample geometries and testing techniques. Miniature samples were tested in an advanced electro-thermomechanical test machine that was developed recently for elevated temperature testing, and standard-sized samples were tested in a universal TMF testing machine. The TMF experiments were conducted using identical test conditions (300-800°C and constraint ratios between -0.4 to -1.0) so that results from both test techniques could be compared. The results show that the cyclic deformation behavior obtained from miniature samples was similar to that obtained using standard-sized test samples and conventional TMF testing techniques. This suggests that miniature samples could be used to characterize the TMF behaviour of thin structures tubes that are used commonly in industrial applications.

### 12:50 PM

A Study on Low Cycle Fatigue of IN738LC Super Alloy at Elevated Temperature: *Kwon Tae Hwang*<sup>1</sup>; Keun Bong Yoo<sup>2</sup>; Han Sang Lee<sup>2</sup>; Jae Hoon Kim<sup>1</sup>; <sup>1</sup>Chungnam National University; <sup>2</sup>Korea Electric Power Research Institute

High strength nickel-base super alloys have been used in turbine blades for many years because of their superior performance at high temperatures. The prediction of fatigue life for super alloy is important for improving the efficiency. In this study, low cycle fatigue tests are performed on the IN738LC super alloy at elevated temperatures. The relations between strain energy density and number of cycles to failure are examined in order to predict the low cycle fatigue life of IN738LC super alloy. The lives predicted by strain energy methods are found to coincide with experimental data and results obtained from the Coffin-Manson method. And, the cyclic behavior of IN738LC super alloy is characterized by a region of small cyclic hardening at the higher strain amplitudes or softening at the lower strain amplitudes.

# Symposium L: Energy Generation, Harvesting and Storage Materials: Membrane and Thermoelectric

Tuesday AM	Room: 7	
August 3, 2010	Location:	Cairns Convention Centre

Session Chairs: Huanting Wang, Monash University; Suk Won Cha, Seoul National University

#### 8:30 AM Keynote

Alternatively Chitosan Sulfate Blending Membrane as Methanol-Blocking Polymer Electrolyte Membrane for Direct Methanol Fuel Cell: Yan Xiang<sup>1</sup>; Hong Guo<sup>1</sup>; <sup>1</sup>Beihang University

A series of blending chitosan sulfate membranes have been developed by grafting the chitosan monomers with sulfonic groups, then cross-linking the polymers from the bonds reaction between the sulfonic groups in the chitosan sulfate and the amido groups in the pure chitosan monomers. Mechanical characterizations demonstrated that the dimensional swelling as well as the methanol crossover of the chitosan membranes were suppressed successfully by the polymer blending, with area swelling value decreased from 55.1% to 39.3% and methanol diffusion coefficient decreased from  $1.0 \times 10^{-6}$  cm<sup>2</sup> s<sup>-1</sup> of pure chitosan to  $4.7 \times 10^{-7}$  cm<sup>2</sup> s<sup>-1</sup> of the membrane with chitosan sulfate content of ~9.1wt% (CCSM 110). The thermal analysis indicated that the blending chitosan sulfate membranes were structure stable below  $100^{\circ}$ C. The blending membrane showed the best conductivity (0.03 S/cm at  $80^{\circ}$ C). The methanol permeability of CCSM 110 was much lower compared with that of Nafion 112 ( $1.9 \times 10^{-6}$  cm<sup>2</sup> s<sup>-1</sup>).



#### 8:50 AM

High Water Diffusivity in Low Hydration Plasma-Polymerised Proton Exchange Membranes: Vanessa Peterson<sup>1</sup>; Cormac Corr<sup>2</sup>; Gordon Kearley<sup>1</sup>; Roderick Boswell<sup>2</sup>; Zunbeltz Izaola<sup>3</sup>; <sup>1</sup>ANSTO; <sup>2</sup>The Australian National University; <sup>3</sup>Helmholtz Zentrum Berlin für Materialien und Energie

A comparison of proton diffusion through plasma-polymerised protonexchange membranes (PEMs) produced using traditional wet-chemical methods (Nafion®) and those produced using a plasma-polymerisation method at the Space Plasma, Power and Propulsion (SP3) Laboratory, The Australian National University, is made using quasielastic neutron scattering (QENS). We find that the diffusion-rate of protons in the plasma-polymerized material and Nafion® is the same (within 1 standard error), a noteworthy result as the plasma-polymerised membrane has 80 % less water than the Nafion®, even though the ion exchange capacities are comparable. We attribute this result to the highly cross-linked structure of the plasma-polymerised membrane. We compare the two membranes by applying a simple model of proton motion describing long-range translational motions and a local region of confinement. There were no significant differences in the proton motions between the plasma-produced and Nafion® membranes using this description, indicating a similar mechanism of proton transport in both membranes.

#### 9:05 AM

Hydrogen Permeation of Pd-Free V-Based Metallic Membranes for Hydrogen Separation and Purification: Cheol-Young Kim<sup>1</sup>; Hong-Seok Chin<sup>1</sup>; Gu Yoo<sup>1</sup>; Kyoung-Won Park<sup>1</sup>; *Eric Fleury*<sup>1</sup>; <sup>1</sup>KIST, Center for High Temperature Energy Materials

The depletion of natural resources accompanied with the simultaneous increase of energy consumption and sensibility towards pollution and global warning, necessitate exploring alternative sustainable energies. Among the various sources of energy, hydrogen is one of the most promising. However hydrogen does not exist in its free form and has to be separated from gas or other chemical compounds. Hence, effective production of hydrogen is a prerequisite. Pd metal and its alloys emerged as the best candidate among metallic materials in term of performance however its cost and scarcity in the Earth's crust encourage the development of new metallic alloys. Based on the hydrogen permeability, V-, Nb- and Ta-based alloys are among the best alternative candidates. However hydrogen embrittlement and the poor of resistance of these alloys to contaminant gases have limited their development. This study was performed with the aim of acquiring a better understanding of the hydrogen embrittlement resistance of a variety of V-Al-(Co,Ta), Nb-Ti-Ni and Ta-Ti-Ni alloys. It will be shown that the highest value of hydrogen permeability (~3 10-7 mol/m.s.Pa1/2 at 400°C) was achieved for monolithic V-Al-(Co,Ta) alloys. The mechanism of hydrogen embrittlement is proposed from the correlation of observed changes in structure particularly the lattice dilatation.

#### 9:20 AM

A New Concept for Alloy Design of Nb-Based Hydrogen Permeable Alloys with High Hydrogen Permeability and Strong Resistance to Hydrogen Embrittlement: *Hiroshi Yukawa*<sup>1</sup>; Masahiko Morinaga<sup>1</sup>; Tomonori Nambu<sup>1</sup>; Yoshihisa Matsumoto<sup>1</sup>; <sup>1</sup>Nagoya University

A concept for alloy design of Nb-based hydrogen permeable alloys is proposed in order to satisfy both high hydrogen permeability and strong resistance to hydrogen embrittlement. In order to improve the resistance to hydrogen embrittlement, it is necessary to reduce the hydrogen content by reducing the heat of hydrogen dissolution into Nb. In other words, the pressure-compositionisotherms (PCT) curve of Nb should be controlled and shifted toward left and upper side in some way, for example, by alloying. On the other hand, the hydrogen flux J through the membrane with a thickness of d is expressed as,  $J = -cB\Delta\mu/d$ , where c is the hydrogen concentration, B is the mobility,  $\Delta\mu$  is the difference of hydrogen chemical potential between inlet and outlet sides of the membrane. Following this concept, Nb-based alloys have been designed and developed that possess both excellent hydrogen permeability and strong resistance to hydrogen embrittlement.

#### 9:35 AM

Thermoelectric Performance of Half-Heusler TiNiSn Alloys Fabricated by Solid-Liquid Reaction Sintering: *Yoshisato Kimura*<sup>1</sup>; Chihiro Asami<sup>1</sup>; Yaw-Wang Chai<sup>1</sup>; Yoshinao Mishima<sup>1</sup>; <sup>1</sup>Tokyo Institute of Technology

Half-Heusler compound TiNiSn is one of the most promising candidate thermoelectric materials that can be used at high temperatures around 1000 K to directly convert the waste heat into clean electric energy. Not only excellent transport properties but also eco-friendly nontoxic constituent elements are attractive advantages of TiNiSn as a thermoelectric material, while relatively high thermal conductivity is a drawback which should be improved through material design. In the present work, aiming to establish the basis of TiNiSn thermoelectric material design together with ecological fabrication process, we have evaluated thermoelectric properties of TiNiSn nearly single-phase alloys which can be fabricated by means of solid-liquid sintering using gas atomized TiNi compound powder and Sn powder. We have evaluated that the present TiNiSn nearly single-phase alloy has large electrical power factor of about  $3.5 \text{ mWm}^{-1}\text{K}^{-2}$  at around 700 K. The lattice thermal conductivity can effectively be reduced by microstructural factors such as interfaces. Consequently, the maximum value of the dimensionless figure of merit, ZT = 0.67, is achieved at around 700 K even without any tuning through alloying elements.

#### 9:50 AM

# Point Defects and Their Role on Thermoelectricity of Na<sub>x</sub>CoO<sub>2</sub> by First Principles Calculation: *Masato Yoshiya*<sup>1</sup>; Takahiro Okabayashi<sup>1</sup>; Masahiro Tada<sup>1</sup>; <sup>1</sup>Osaka University

Attention has been paid to Na<sub>2</sub>CoO<sub>2</sub>, a p-type thermoelectric layered oxide, due to its remarkable power factor, and efforts have been made to reveal underlying physics that governs the heat-to-electricity energy conversion. Although the dependence of efficiency on Na content has been extensively studied, little attention has been paid to Na vacancies which are inevitably created upon the decrease in Na content. In order to reveal any role of the Na vacancy for realization of thermoelectricity, first principles calculations have been carried out, at first, to evaluate energetics of point defect formations and then to quantitatively analyze the role of the point defects on the change in electronic structure in these series of material. It is found that Na vacancy is easily created upon sublimation while energy penalty for formation of Co and O vacancies is high. Analyses on the change in charge distribution upon Na vacancy formation revealed that the Na vacancy is not mere an empty region but plays important roles as a reservoir of electrons, minor carriers, and as a supplier of electron holes, major carrier, to CoO<sub>2</sub> conducting layer, which is presumably one of the origins of thermoelectricity in these materials.

#### 10:05 AM

Correlations between Cation Layers in Layered Thermoelectric Materials and Their Phonon Thermal Conductivity by Molecular Dynamics: Masahiro Tada<sup>1</sup>: Masato Yoshiva<sup>1</sup>: Hidevuki Yasuda<sup>1</sup>: <sup>1</sup>Osaka University

In order to reveal mechanisms of phonon thermal conduction in layered oxide thermoelectric materials, theoretical calculation has been carried out. Thermal conductivity is one of three factors to determine a figure of merit of a thermoelectric material. In our previous study, it was found that cation vacancies are responsible for the decreasing of phonon thermal conductivities of Na CoO, with affecting vibrations of Co-O layers. In order to obtain more detailed fundamental knowledge about this, we have compared Na<sub>x</sub>CoO<sub>2</sub> with Li<sub>x</sub>CoO<sub>2</sub> and K CoO, to reveal what property of cations dominates the decrease in phonon thermal conductivity. When cation vacancies were introduced, phonon thermal conductivity was decreased in all the three cobaltites. However, magnitude of the decrease was different among them. We prepared imaginary models with which individual effects of differences of their masses and in-plane or out-of-plane lattice constants of supercells between each cobaltite can be analyzed. It is found that the effect of the difference of out-of plane lattice constants was the most remarkable. This indicates that the distance between a cation layer and a Co-O layer, in other words, two-dimensionality of each layer, affects the magnitude of the decrease in thermal conductivity.

#### 10:20 AM

Thermoelectric Response in Strongly Correlated Cobaltites: Yang Wang<sup>1</sup>; Hongjin Fan<sup>1</sup>; <sup>1</sup>Nanyang Technological University

Environment-friendly thermoelectric materials have been widely studied for a long time because they hold great promise for clean energy generation. We report the thermoelectric response of Ca-site Ag, Y doped and Co-site Fe, Mn, Cu doped misfit-layered Ca<sub>2</sub>Co<sub>4</sub>O<sub>6</sub>, and La-site Ca, Sr, Ce doped perovskite LaCoO3, fabricated by solid-state reaction. The ion doping efficiently improves the thermoelectric performance of these cobaltites. Combining specific heat, transport, and magnetic properties measurements, it is revealed that the thermoelectric properties depend not only on the substitution element but also the ion site that is replaced, and the structure distortions have remarkable effects on the thermoelectric properties. Comparing the thermoelectric characteristics of the two cobaltite systems, we find the electron strong correlation and the large configurational entropy of Co ions are intrinsic in cobaltites, which underlie the unusually large thermoelectric response in the Co-oxides. The obtained highest merit of figure ZT in these doped cobaltites can reach 0.1 at room temperature (La<sub>0.9</sub>Sr<sub>0.1</sub>CoO<sub>3</sub>) and 0.4 at 1000K (Ca<sub>3</sub>Co<sub>3.9</sub>Fe<sub>0.1</sub>O<sub>9</sub>), suggesting their promising thermoelectric applications. Based on the results, some strategies for searching new high-performance thermoelectric oxides in strongly correlated systems are proposed.



### 10:35 AM

**Thermoelectric Properties of Self-Doped LnGd**<sub>1+x</sub>S<sub>3</sub> (Ln: La, Sm) Prepared by CS<sub>2</sub> Sulfurization: *Michihiro Ohta*<sup>1</sup>; Shinji Hirai<sup>2</sup>; Toshihiro Kuzuya<sup>2</sup>; <sup>1</sup>National Institute of Advanced Industrial Science and Technology (AIST); <sup>2</sup>Muroran Institute of Technology

Ternary rare-earth sulfides LnGd<sub>1+x</sub>S<sub>3</sub> (Ln: La, Sm) were prepared by CS<sub>2</sub> sulfurization. The sulfurization of binary rare-earth oxide Gd<sub>2</sub>O<sub>3</sub> and Ln<sub>2</sub>O<sub>4</sub> powders with CS<sub>2</sub> gas afforded the binary rare-earth sulfide Gd<sub>2</sub>S<sub>2</sub> and Ln<sub>2</sub>S<sub>3</sub> powders. Then mixtures of  $Gd_2S_3$ ,  $Ln_2S_3$  and  $GdH_3$  powders were reacted to prepare the self-doped LnGd<sub>1+r</sub>S<sub>3</sub> powders. The powders obtained were consolidated by pressure-assisted sintering to achieve full density. The sintered samples crystallize in the cubic defect Th<sub>3</sub>P<sub>4</sub>-type structure. The measurement of the Seebeck coefficient, electrical resistivity, and thermal conductivity was carried out on the sintered samples over the temperature range of 300 K to 950 K. All the sintered samples exhibit a negative Seebeck coefficient. The magnitude of the Seebeck coefficient and the electrical resistivity decrease systematically with increasing Gd content. Therefore, the thermoelectric power factor was improved by self-doping. The thermal conductivity of the sintered samples is low. The high thermoelectric power factor and low thermal conductivity in the self-doped LnGd<sub>1+x</sub>S<sub>3</sub> result in the high thermoelectric figure of merit (ZT) at high temperature.

## Symposium L: Energy Generation, Harvesting and Storage Materials: Hydrogen Storage I

Tuesday AM August 3, 2010 Room: 7 Location: Cairns Convention Centre

Session Chairs: Li Lu, National University of Singapore; Deryn Chu, U. S. Army Research Laboratory

#### 10:50 AM Keynote

Improving the Hydrogen Storage Properties of Mg Based Alloys by Manipulating Their Microstructure: *Min Zhu*<sup>1</sup>; <sup>1</sup>South China University of Technology

Mg based alloys have high theoretical hydrogen storage capacity which makes them promising hydrogen storage materials. However, the poor hydrogen absorption/desorption kinetics and high thermodynamic stability of their hydrides baffle the practical applications. In recent years, hydrogen storage performances of Mg based alloys have been substantially improved by microstructure manipulation. In the present work, physical vapor deposition, mechanical alloying and in-situ nano-composite formation by disproportionation reaction have been applied to obtain different microstructure. These microstructure manipulations result in substantial improvement of hydrogen storage properties. For instances, Mg/MmM<sub>5</sub> multi-layer film can reversibly absorb about 5 mass% hydrogen at 473 K. The Mg-Re(rare earth metals) based alloys with nano-composite structure induced by disproportionation reaction can reversibly absorb about 4 mass% hydrogen at 533 K and maintain 80% of this capacity after 500 hydrogenation/ dehydrogenation cycles. Both theoretical and experimental studies have revealed that surface and interfaces (including grain boundary and interphase boundary) are important factors for manipulating the thermodynamics and kinetics of hydriding/ dehydriding reaction. The interaction between different phases, which can induce and/or catalyze hydrogenation/dehydrogenation also plays an important role. In addition to the reversible hydrogen storage performance, the Mg-Re alloys with nano-composite structure can deliver hydrogen in a fast kinetics via hydrolysis.

#### 11:10 AM Keynote

Improved Hydrogenation-Dehydrogenation of the Nanostructured Melt-Spun Mg-Ni-Mm Alloys: *Ying Wu*<sup>1</sup>; S.X. Zhou<sup>2</sup>; <sup>1</sup>China Iron and Steel Research Institute Group, Advanced Technology and Materials Co., Ltd. - and - Institute for Energy Technology; <sup>2</sup>China Iron and Steel Research Institute Group, Advanced Technology and Materials Co., Ltd.

Rapid solidification (RS) technique is an efficient process in improving the hydrogen storage properties of Mg-based alloys. Microcrystalline, nanocrystalline and amorphous microstructures of melt-spun Mg-10Ni-2Mm (at.%) ribbons were synthesised by applying copper wheel surface velocities of 300, 1000 and 2000 rpm, respectively. From TEM studies we have found that  $MmMg_{12}$  intermetallic nucleates at the grain boundaries of Mg and Mg<sub>2</sub>Ni, thus, providing paths for H exchange. The kinetics of H-absorption/desorption is improved in the refined microstructures due to the fast hydrogen diffusion in the nanograins. TEM studies showed (a) stability of the nano-sized grains in the samples that underwent

cycling of hydrogen desorption and absorption during their heating to  $350^{\circ}$ C; (b) formation of MmH<sub>3-x</sub> hydride from MmMg<sub>12</sub> and its preferential location at grain boundaries of MgH<sub>2</sub>. Clearly, MmH<sub>3-x</sub> and Mg<sub>2</sub>NiH<sub>4</sub> act as nucleation centers to initiate the formation of MgH<sub>2</sub>; this, in turn, promotes hydrogen absorption by the Mg alloys. Temperature range of hydrogen thermal desorption from the hydrogenated amorphous microstructure sample is much narrower than that for the microcrystalline one that can be explained by the increase of uniformity of particle size distribution in the hydrides with the increase of solidification rate.

#### 11:30 AM

Characterisation of Hydrogen Release Behaviour in Cast Mg-Ni Alloys by Synchrotron XRD and XAFS: *Kazuhiro Nogita*<sup>1</sup>; Marc Jenke<sup>2</sup>; David Wood<sup>2</sup>; Andrew Duguid<sup>2</sup>; Stuart McDonald<sup>1</sup>; <sup>1</sup>The University of Queensland; <sup>2</sup>Hydrexia Pty. Ltd.

Hypo-eutectic Mg-Mg<sub>2</sub>Ni system can be modified by trace elemental additions which change the microstructure and the functional properties of the alloy. The modified microstructure results in improvements to the hydrogen storage properties. In alloys of optimal composition, it has been shown that the reversible storage of 6.5-7wt% H<sub>2</sub> is possible at a rate of reaction that is realistic for industrial applications. This paper discusses the mechanism of hydrogen release under air and atmospheric pressure with in-situ crystallographic phase transformation analysis obtained by synchrotron radiation X-ray diffraction (XRD) with Rietveld analysis and X-ray absorption fine structure (XAFS). The synchrotron experiments were performed at the Powder Diffraction and XAS lines in the Australian Synchrotron (Project ID: AS091/PD975, AS093/PD1726 and AS093/XAS\_QLD), and BL12 and BL15 in the SAGA Light Source (Project ID: 080624N, 081150N, 090303N).

# Tue. AM

#### 11:45 AM

Catalysing Effect of Intermetallic Compounds on Hydrogen Desorption Kinetics in Cast Magnesium Alloys: Younghee Cho<sup>1</sup>; Arne Dahle<sup>1</sup>; <sup>1</sup>The University of Queensland

Magnesium based hydrogen storage materials were prepared by a conventional technique of melting and casting. Characterisation of microstructure and hydrogen sorption properties of the alloys was carried out. Addition of Al, Cu and Ni leads to the formation of eutectic mixtures, Mg-Mg<sub>17</sub>Al<sub>12</sub>, Mg-Mg<sub>2</sub>Cu and Mg-Mg<sub>2</sub>Ni respectively with an inter-lamellar spacing of several hundred nanometers. Despite similar eutectic networks where hydrogen atom efficiently diffuses along interphase boundary, catalytic effect of Mg<sub>2</sub>Ni in the eutectic on hydrogen desorption rate was found to be far stronger than that of Mg<sub>2</sub>Cu and Mg<sub>17</sub>Al<sub>12</sub>. 3d and 4d transition metals were also added to Mg-Ni alloys and found to form intermetallic compounds with Ni during solidification. The transition metal rich intermetallics homogeneously dispersed in the alloys show a catalytic role in increasing hydrogen desorption kinetics remarkably. A systematic microstructure study is presented to understand the catalytic mechanism of intermetallic compounds in magnesium based cast alloys. On the basis of the mechanism, hydrogen migration process in bulk magnesium alloys is further discussed.

#### 12:00 PM

#### Study of Li-Based Hydrogen Storage Materials for Improvement of Thermodynamic Property: *Hiroyuki Takeshita*<sup>1</sup>; Naoki Ito<sup>1</sup>; Seito Niwa<sup>1</sup>; Tatsuya Higuchi<sup>1</sup>; <sup>1</sup>Kansai University

LiBH<sub>4</sub> has high gravimetric H storage capacity but is too stable to decompose to generate H<sub>2</sub> gas under moderate conditions. Formation of relatively stable dehydrogenated products can contribute to the decomposition at lower temperature. For example, LiBH<sub>4</sub> and MgH<sub>2</sub> with both very high hydrogen desorption enthalpies decompose in the manner of  $2\text{LiBH}_4=2\text{LiH}+2\text{B}+3\text{H}_2$  and MgH<sub>2</sub>=Mg+H<sub>2</sub>, respectively, whereas LiBH<sub>4</sub> mixed with MgH<sub>2</sub> shows H<sub>2</sub> gas desorption in the manner of  $2\text{LiBH}_4=MgH_2=MgB_2+2\text{LiH}+4H_2$ , of which the enthalpy of the reaction can be improved compared with individual dehydrogenation of LiBH<sub>4</sub> and MgH<sub>2</sub>, by the formation enthalpy of MgB<sub>2</sub> according to Mg+2B=MgB<sub>2</sub>. We have been studying about the MgAlB<sub>4</sub>-LiH-H<sub>2</sub> and Li<sub>3</sub>BN<sub>2</sub>-H<sub>2</sub> system based on the above-mentioned concept and will report about the improvement of thermodynamic properties as well as kinetic ones.

#### 12:15 PM

Hydrogen Storage Properties of Mg-Ni Alloy Catalysed by Multi-Walled Carbon Nanotubes: Sima Aminorroaya<sup>1</sup>; Hua Liu<sup>1</sup>; <sup>1</sup>University of Wollongong

The hydrogen storage performance of ball-milled sample of cast Mg-6 wt% Ni alloy was investigated. Morphology and microstructure of the cast sample and achieved powders were evaluated by high-resolution scanning electron microscopy. The activation characteristics of ball-milled alloy is compared with that of the materials obtained by ball-milling of 5 wt% Multi-walled carbon nanotubes (MWCNTs) for 0.5, 1, 2, 5 and 10 hours. MWCNTs enhanced the absorption kinetics considerably in all cases. The hydrogen content of modified powder by MWCNTs reached to the maximum hydrogen capacity within two minutes of exposure to hydrogen at 370°C and 2MPa pressure. X-ray diffraction



analysis evidenced that no new phase is formed during milling. Although, milling with MWCNTs reduced the crystallite size, even if the milling carried out for an hour. The rate-controlling steps of hydriding reactions at different milling times were determined by fitting the respective kinetic equations. The evidence is provided that nucleation and growth of hydrides accelerate drastically by homogenously distribution of MWCNTs on the surface of ball-milled powders

#### 12:30 PM

PRICM

Hydrogen Exchange Effect in MgH<sub>2</sub>-LiBH<sub>4</sub> System: *Liang Zeng*<sup>1</sup>; Hiroki Miyaoka<sup>1</sup>; Takayuki Ichikawa<sup>1</sup>; Yoshitsugu Kojima<sup>1</sup>; <sup>1</sup>Hiroshima University

Lithium borohydride has been considered as one of the most promising candidates for hydrogen storage due to its high hydrogen content of 18.5mass%. In the MgH<sub>2</sub>-LiBH<sub>4</sub> system, it was found that there was a mutual interaction between these two hydrides, but its mechanism has not been clarified yet. In the present work, we found out there was an "H-D" exchange happened during heating for the composite of MgD<sub>2</sub> and LiBH<sub>4</sub> (molar ratio 1:2). In-situ FT-IR was used to detect the variation of "B-D" vibration modes in the composites by heating, and the result revealed the peak of "B-D" vibration started to appear at around 200°C, which means this isotopic exchange proceeded in solid phase. The ball-milled and hand-milled composites of Nb<sub>2</sub>O<sub>5</sub> (1mol%)-doped MgH<sub>2</sub> and LiBH<sub>4</sub> (molar ratio 1:2) were investigated by TG-MS. It showed that the decomposition temperature of the first step was over 300°C, in spite of Nb<sub>2</sub>O<sub>5</sub> (1mol%)-doped MgH<sub>2</sub> itself can be completely decomposed at around 200°C individually. This result might suggest the decomposition of catalytic MgH<sub>2</sub> was somehow suppressed by the exchange effect we found out.

#### 12:45 PM

Thermodynamic Characterization on Hydrogen Absorption and Desorption Reactions of Lithium – Silicon Alloy: *Koichi Doi*<sup>1</sup>; Satoshi Hino<sup>1</sup>; Hiroki Miyaoka<sup>1</sup>; Takayuki Ichikawa<sup>1</sup>; Yoshitsugu Kojima<sup>1</sup>; <sup>1</sup>Hiroshima University

Lithium hydride LiH is one of the attractive hydrogen H, storage materials, because it stores 12.7 mass% of H2. However, H2 desorption reaction occurs over 600°C due to the large enthalpy change of H, desorption  $\Delta H^{\circ} = 181 \text{ kJ/}$ mol H.. The purpose of this work is to control the enthalpy change of LiH to much lower value by a mechanical alloying with Silicon Si, where the Li-Si alloy is thermodynamically more stable than Li. The alloy was synthesized from Li granule and Si powder by a mechanical ball-milling method. The H. ab/desorption properties were characterized by High-Pressure Differential Scanning Calorimetry and Thermogravimetry - Differential Thermal Analysis - Mass Spectroscopy. Pressure - Composition - Isotherm (PCI) measurements were performed at 400. 450, and 500°C to estimate the enthalpy change. It was confirmed that reversible H<sub>2</sub> ab/desorption reactions of the Li-Si alloy were expressed as  $7\text{LiH} + 3\text{Si} \leftrightarrow$ (3/7)Li<sub>12</sub>Si<sub>7</sub> + (13/7)LiH + (18/7)H<sub>2</sub>  $\leftrightarrow$  Li<sub>7</sub>Si<sub>3</sub> + (7/2)H<sub>2</sub> (theoretically 5.0 mass% H<sub>2</sub>) at 400°C. From van't Hoff plot obtained by the results of PCI measurements, the enthalpy change of the former reaction was estimated to be  $\Delta H^{\circ} = 103 \text{ kJ/mol}$ H<sub>2</sub>, which is lower than that of LiH.

# Symposium A: Advanced Steels and Processing: Recrystallisation Behaviour

Tuesday PMRoom: A/BAugust 3, 2010Location: Cairns Convention Centre

Session Chair: Sung-Joon Kim, Korea Institute of Materials Science

#### 2:00 PM Keynote

Effect of Nb Content on Hot Flow Stress, Dynamic Recrystallization and Strain Accumulation Behaviors in Low Carbon Bainitic Steel: Chengliang Miao<sup>1</sup>; Guodong Zhang<sup>1</sup>; *Chengjia Shang<sup>1</sup>*; <sup>1</sup>University of Science and Technology Beijing

The objective of present study was the effect of niobium content on the flow stress, dynamic recrystallization (DRX) and strain accumulation to clarify metallurgical mechanism during hot rolling. Compressive deformation behaviors of low carbon steels with different Nb content were investigated at the temperature from 900°C to 1150°C and strain rates from  $0.1s^{-1}$  to  $2s^{-1}$  by single-pass deformation. Multipasses processes also were carried out to examine strain accumulation. In absence of DRX, higher stress is obtained in high Nb steel (0.1wt%) compared with low Nb steel (0.06, 0.012wt%), but the discrepance will be minished as decreasing of deformation temperature. The difference of stress level between high and low Nb steel will be enlarged at low strain rate because of high mobility of dislocation at large strain rate and limited drag effect of solute Nb. In single pass deformation, occurrence of DRX is easier to be observed in low strain rate, due to lower critical stress for DRX. In presence of DRX, high Nb steel exhibits higher critical stress value, which was calculated to determine its DRX kinetics. However, there is an opposite result in the case of multi-passes rolling, because high Nb steel has larger strain accumulation between passes intervals.

### 2:20 PM Invited

# **Evolution of Different Recrystallization Textures in Steels Having {111}<112> Rolling Texture:** *Dong Nyung Lee*<sup>1</sup>; Se-Jong Kim<sup>1</sup>; Heung Nam Han<sup>1</sup>; <sup>1</sup>Seoul National University

The Goss {110}<001> orientation, which is not stable with respect to plane strain rolling, rotates toward the {111}<112> orientation forming a strong maximum. The {111}<112> rolling component returns to the Goss orientation after recrystallization. The density of the Goss component after recrystallization increases with increasing density of the {111}<112> component. On the other hand, the {111}<112> nucleation texture is strongly formed in 65% cold rolled iron electrodeposit with a weak {111}<112> texture, and results in the {111}<112> recrystallization texture, whereas a {111}<110> nucleation texture is formed in 80% cold rolled electrodeposit having a strong {111}<112> texture, resulting in the {111}<110> recrystallization texture. That is, the {110}<001>, {111}<112>, and {111}<110> recrystallization textures evolve in bcc steels having the {111}<112> rolling textures. The results have been discussed by the strainenergy-release-maximization theory. In the model the recrystallization texture is determined such that the absolute maximum stress direction due to dislocations formed during rolling can be parallel to the minimum Young's modulus direction in the recrystallized phase, whereby the strain energy release can be maximized. According to the concept, the evolution of the different recrystallization textures results from different active slip systems and their activities leading to the same deformation texture.

### 2:35 PM

Cold Rolling and Annealing Microstructures and Textures of Low Carbon Steels: Marwan Almojil<sup>1</sup>; Pete Bate<sup>1</sup>; <sup>1</sup>University of Manchester

The annealing behaviour, including studies of recrystallization kinetics and development of crystallographic texture, of IF and HSLA steels after 20, 50, 70 and 90% cold rolling reductions have been investigated using optical metallography and Electron Backscattered Diffraction (EBSD). The HSLA steel was initially processed to give a volume fraction of about 0.2 of fine pearlite colonies, which acted as mechanically hard particles. The presence of these particles on the HSLA steel significantly reduced the temperature needed for recrystallization by enhancing the recrystallization and acting as nucleation sites by the Particle Stimulated Nucleation (PSN) mechanism. The evolution of texture for the different reductions in both steels during the process of recrystallization was observed. Both cold rolling and recrystallization textures are shown to be largely dependent on the rolling reduction. With increasing rolling reduction, the texture show gradual intensification of  $\alpha$  and  $\gamma$  fibre components. Despite the dominance of PSN in HSLA steel, the recrystallization textures were similar to that of the rolling textures and, although the texture intensity was lower, to recrystallization textures in IF steel.

#### 2:50 PM

#### Influence of Phosphorous and Boron on Recrystallization, Grain Growth and Mechanical Properties of 3% Si Steel: *Seil Lee*<sup>1</sup>; B. C. De Cooman<sup>1</sup>; <sup>1</sup>POSTECH

A new approach to obtain high strength 3% Si steel by addition of phosphorus is proposed. P is known as the most effective solid solution strengthening in ferritic steels (680MPa/mass%). P-additions decrease the mechanical formability significantly due to the P grain boundary segregation. B-additions, with the aim of suppressing grain boundary P segregation, strengthening the grain boundary cohesion and enhancing the P solid solution hardening is proposed in the present study. Two 3% Si steels, a B-free 0.1%P steel and a 20 ppm B-added 0.1%P steel were analyzed. The microstructures were studied in detail by EBSD. The B-addition resulted in a pronounced rotated cube component, {100}<011>, after a hot-band annealing treatment. A y-fiber texture was developed in the B-free steel. The B-addition caused a retardation of the recrystallization, allowing for the growth of grains with a lower stored energy, such as rotated cube oriented grains. The steels were further cold rolled and recrystallization annealed to observe a similar effect after large cold reductions. The contribution will focus on the potential of this concept to obtain high strength 3% Si steels with low core losses

#### 3:05 PM

Hot Deformation Behavior of V-Microalloyed Steel: Ren Anchao<sup>1</sup>; <sup>1</sup>WISCO

Through the expansion curve of continuous cooling transformation at different cooling rates measured by THERMECMASTOR- Z thermal simulator for U75V rail steel, the continuous cooling transformation curve was obtained. Then a study was done to the influence on steel microstructure and hardness at different cooling rates. The softening behavior of isothermal deforming at austenite area 850~1000°C within interval of passes was also studied by double-pass compression



test. The results show that the product of austenite transformation was pearlite when the cooling rate was lower than 10°C. When the cooling rate was in the range of 10~ 50°C•s-1, only martensite was received. The hardness of the test steel is increasing along with the higher cooling rate. Results also indicated that 30% deformation and 3S-1 deforming rate as deformation temperature = 1000°C relaxation time for performing recrystallization was =100s and as deformation temperature = 880°C recrystallization of steel was difficult to form even if the relaxation time is extended.

#### 3:20 PM

Influence of Cu Contents in Hot Shortness of Cu Bearing EAF Steels: Young Sup Lee<sup>1</sup>; Choonho Jung<sup>1</sup>; Joong-Hwan Jun<sup>2</sup>; Yinghua Jiang<sup>1</sup>; Jae-Taek Im<sup>1</sup>; Young Ho Lee<sup>1</sup>; Sam Kyu Chang<sup>1</sup>; Man Rae Kim<sup>1</sup>; <sup>1</sup>Dongbu Steel Co., Ltd.; <sup>2</sup>Korea Institute of Industrial Technology

The surface cracking behaviours of copper-containing steels under conditions similar to those in the direct hot charging processes are investigated. The surface cracking phenomenon so called "Hot Shortness" is caused by a molten copper phase that forms due to copper enrichment as selective iron oxidizes during hot working such as continuous casting, direct hot charging/reheating and hot rolling. In order to examine the surface cracking behaviors, several specimens with differing copper contents, from 0.11 to 0.38% (by weight), were prepared, oxidation was performed in three different atmospheres at 1100, 1150 and 1200°C and hot compression test were conducted at 1050°C using a Gleeble 3500. The Cu-rich phase above critical level the surface cracking increased gradually. The specimens with high copper content exhibited critical Cu-rich phase at thinner scale thickness, while for the specimens with low copper content the critical Curich phase occurred at thicker scale thickness. In general, the oxidation rates of copper containing steels decreased with increasing Cu content. Also, oxidation potential are affected many factors such as temperature, time, atmosphere and other elements. It is suggested that overriding factor governing the surface cracking is scale thickness.

#### 3:35 PM

# The Technology of Minimize the Surface Cracks of AHSS Steels in Kwangyang Works: Yu Sung Jong<sup>1</sup>; <sup>1</sup>POSCO

The Kwangyang Works is an iron and steel making works which produces strategically automotive sheet steels of 6.5 million tones. The operation technology for continuous casting was recently developed to minimize the surface cracks causing silver-type defects on hot rolled coils. This operation technique was found to be more efficient when the continuous cast slabs have surface cracks including longitudinal crack, corner cracks, near slab edges. In pursuit with improvement of the solidification characteristics, a new type of mold flux was used and exhibited 20% slow-cooing effect by the basicity and optimized solidification temperature. The solidification characteristic of high manganese steels at a bottom exit of the mold was also improved by increasing the mold taper of 10%. Furthermore, a new type method in mold oscillation was introduced to decrease deformation stress being operative on solidifying shells, especially for steels containing 0.8~1.0% carbon, niobium and boron. The secondary cooling patterns were designed in order to decrease occurrence rate of corner cracks on cast slabs whenever the new types of steel are developed and manufactured in real lines. Through these several efforts, the Kwangyang Works is safely producing various sheet steels by stablishing casting conditions adaptive to unique characteristics of AHSS steels.

#### 3:50 PM

Effect of Hot-Rolled Processing on Microstructures in Nb Microalloying Dual-Phase Steels: *Xuehui Zhang*<sup>1</sup>; Guohui Zhu<sup>2</sup>; Weimin Mao<sup>3</sup>; <sup>1</sup>WISCO; <sup>2</sup>Anhui University of Technology; <sup>3</sup>University of Science and Technology Beijing

Cold rolled dual phase steels with low C and Si addition was microalloyed by Nb in order to refine ferrite grain size. Effect of re-heating and hot rolling processing on microstructural evolution was investigated by simulation in Gleeble. The dissolution of NbC at high temperature in re-heating was very important on refining grain size of final products. In order to promote the effect of NbC on refining grain size, the re-heating temperature has to be over 1100°C based on the modeling and experimental results. The peak strain for dynamic recrystallization of austenite was increased as the dissolution of NbC in reheating due to NbC re-precipitation induced by strain in hot rolling. It resulted in finer grain size of austenite. Experimental results illustrated that the grain size in as-hot rolled sheet would influence the grain size of final product even after cold rolling and intercritical annealing. Nb microalloying enhanced the strength of dual phase sheets by refine grain size rather than to increase the volume fraction of martensite, which is different from other alloying elements such as Cr, Mo and Si.

#### 4:05 PM Tea Break

### Symposium A: Advanced Steels and Processing: Special Session on Multiscale Design of Advanced Steels

Tuesday PM August 3, 2010 Room: A/B Location: Cairns Convention Centre

Session Chairs: Greg Olson, Northwestern University; Kaneaki Tsuzaki, National Institute for Materials Science

#### 4:30 PM Keynote

Advanced Steel Design by Multiscale Modeling: Bruno De Cooman<sup>1</sup>; H.K.D.H. Bhadeshia<sup>1</sup>; F. Barlat<sup>1</sup>; <sup>1</sup>POSTECH

The Graduate Institute of Ferrous Technology is developing a considerable expertise in the areas of steel products technologies, processing and process simulation, with a special emphasis on hot rolling, cold rolling, continuous annealing, coatings and novel application technologies. In addition, computational materials science, materials testing, physical properties determination, and steel micro-characterization using advanced techniques are also active research domains of GIFT. Research within GIFT has a pronounced focus on technology and technical innovations through steel research. Whereas most materials engineering research tends to be midway between the fundamental and the purely technical, true innovation, with the potential for a large impact on future development, is generated when the research emphasizes either the fundamentals or the advanced technologies. The GIFT approach to steel products research, development and innovation therefore is driven by a strong emphasis on long term, fundamental steel research using advanced multi-scale computational methods to support steel design: Ab-initio calculations, Molecular Dynamics, Crystal plasticity modeling, etc. These methods are being used to improve the fundamental understanding required to develop new steel grades and the contribution will highlight some of the recent results obtained at GIFT using multi-scale computation.

#### 4:50 PM Invited

Grain Boundary/Interfacial Energy Database Bridging Atomic Scale and Mesoscale Steel Properties: *Byeong-Joo Lee*<sup>1</sup>; <sup>1</sup>Pohang University of Science and Technology

Due to the decisive role of microstructures on materials properties, many simulation studies have been carried out to predict, understand and control the microstructural evolution. Even though the microstructural evolution is highly dependent on the anisotropy of grain boundary properties such as grain boundary energy and mobility, grain growth simulations that assign specific crystallographic orientations to individual grains and consider anisotropic properties of all possible grain boundaries have never been performed. One should notice here that there can be infinite number of different grain boundaries depending on the orientation relationship between the two grains that form the grain boundary. However, it has been impossible to identify individual grain boundaries among the infinite number of possible grain boundaries with various misorientation relationships. This was the main reason why grain growth simulations, based on realistic crystallographic orientations of individual grains and realistic grain boundary energy anisotropy, have been impossible. Here, we provide a systematic scheme to uniquely define individual grain boundaries among all possible grain boundaries in polycrystalline materials. This allows us to construct a grain boundary energy database in a suitable form for implementation on grain growth simulations.

#### 5:05 PM Keynote

A Systematic Study on Iron Carbides from First-Principles: *In-Gee Kim*<sup>1</sup>; G. Rahman<sup>1</sup>; J.H. Jang<sup>1</sup>; Y.Y. Song<sup>1</sup>; S.-W. Seo<sup>1</sup>; H.K.D.H. Bhadeshia<sup>2</sup>; A.J. Freeman<sup>3</sup>; Greg Olson<sup>4</sup>; <sup>1</sup>Graduate Institute of Ferrous Technology, Pohang University of Science and Technology; <sup>2</sup>Graduate Institute of Ferrous Technology, Pohang University of Science and Technology - and - Department of Materials Science and Metallurgy, University of Cambridge; <sup>3</sup>Department of Physics and Astronomy, Northwestern University; <sup>4</sup>Department of Materials Science and Engineering, Northwestern University

In modern steel design, the primary required information is phase stabilities based on thermodynamic principles, supported by computer aided thermodynamic approaches (CALPHAD methods). These are based on databases which are commonly derived from empirical data, but increasingly exploit first-principles methods which can also supply other fundamental parameters (e.g. mobilities, surface thermodynamics). The field of multiscale computational power, but there is also a need to develop theories for the linking of different modelling technologies. We propose a new approach which is a combination of first-principles methods and a Monte Carlo approach to overcome these barriers. Any macroscopic scale



thermodynamic variables can be obtained from the corresponding partition function which can be solved by a Monte Carlo method with a model Hamiltonian where the necessary parameters are obtained from first-principles calculations. The process will be illustrated using an example of  $\kappa$ -carbide formation, and will be compared with several conventional first-principles examples in iron alloys and carbides.

#### 5:25 PM Invited

**Topology of Spheroidized Pearlite**: *Yoshitaka Adachi*<sup>1</sup>; W.T. Wang<sup>1</sup>; <sup>1</sup>National Institute for Materials Science

Microstructures have been characterized by length, area or volume in general. However topological features such as connectivity or intricate degree also seem to be important. The topological features are quantified in terms of genus, Gaussian curvature and Euler characteristics which are obtained from three-dimensional (3D) images. Computer-aided 3D visualization is becoming powerful tool to shed new light on microstructural evolution. This study examines spheroidization mechanism of lamellar pearlite by topology-based 3D characterization.

#### 5:40 PM Invited

Scale-Bridging Analysis of Stress Partitioning in High Nitorgen Steel: *Mayumi Ojima*<sup>1</sup>; Yoshitaka Adachi<sup>1</sup>; Yo Tomota<sup>2</sup>; <sup>1</sup>National Institute for Materials Science; <sup>2</sup>Ibaraki University

To understand the reason of high work hardening in high nitrogen steel, a scale-bridging analysis consisting of in situ neutron diffraction, in situ electron backscattering diffraction measurement during tensile deformation and weak beam transmission electron microscopy was performed with particular attention to stress partitioning. This study demonstrates the contribution of stress partitioning, back stress due to dislocation pile up at grain boundaries and dislocation interaction to work hardening at each strain level.

#### 5:55 PM Invited

# Multiresolution Continuum Mechanics for Fracture Process: Wing Liu<sup>1</sup>; <sup>1</sup>Northwestern University

A multiresolution continuum theory is presented to predict material response when spatial microstructure evolution gives rise to severely inhomogeneous deformation at multiple scales. The proposed theory is applied by concurrently homogenizing the microstructure at each characteristic length scale associated with the inhomogeneous response. A continuum-microstructure work rate equivalence approach is used to develop a set of continuum partial differential governing equations, in terms of multiresolution microstresses (and couple microstresses). Constitutive models relating to each microstress are determined from numerical microstructure models. The multiresolution governing equations can be solved with a conventional finite element approach.

#### 6:10 PM Invited

**Quantum Engineering of Interfaces in Steels**: *Oleg Kontsevoi*<sup>1</sup>; Arthur Freeman<sup>1</sup>; Gregory Olson<sup>1</sup>; <sup>1</sup>Northwestern University

The mechanical properties of high performance steels are often controlled by the cohesion of crystal grain boundaries and matrix/precipitate interfaces. We present an overview of our efforts to establish underlying mechanisms of interfacial embrittlement based on quantum mechanical calculations. We establish a quantitative theory of embrittling potency of segregated impurities and show its robustness in predicting and controlling grain boundary embrittlement in steels. Further, it has been demonstrated that a marked increase of fracture toughness can be achieved in steels with controlled dispersions of transition metal carbides and nitrides. To understand the interface adhesion between Fe matrix and precipitates at the atomic level, we investigate the theoretical cohesive strength of Fe/M[C,N] (M=Ti,V,Nb,Mo) interfaces using first principles calculations. We further establish the effect of coherency dislocations, primary lattice dislocations at semicoherent interfaces, and interface steps on interface strength. The origins of strong interfacial bonding are identified based on electronic structure and charge density analysis. The insights provided by these predictive capabilities have already contributed to a new generation of "quantum steels" in which desired electronic structures are specifically tailored for enhanced mechanical properties

#### 6:25 PM Keynote

# Multiscale Systems Design of Advanced Steels: Greg Olson<sup>1</sup>; <sup>1</sup>Northwestern University

Over the past quarter century, a multi-institutional consortium has pioneered the computational design of materials using high-performance steels as a test case exploiting the unique depth of scientific understanding available for steels. Thermodynamics-based parametric materials design integrating materials science, applied mechanics and quantum physics within a systems engineering framework has brought a first generation of designer "cybersteels" that have now entered successful commercial applications, and a new enterprise of commercial materials design services has steadily grown over the past decade. The DARPA-AIM initiative broadened computational materials engineering to address acceleration of the full materials development and qualification cycle, demonstrating both accelerated thermal process optimization at the component level and the effective forecast of manufacturing variation with efficient fusion of minimal datasets. The recent flight qualification of the Ferrium S53 corrosion-resistant aircraft landing gear steel represents the first demonstration of the fully integrated computational design and AIM qualification process. A new level of science-based modeling accuracy has now been achieved under the ONR/DARPA "D3D" Digital Structure consortium using a suite of advanced 3D tomographic characterization tools to calibrate and validate a set of high fidelity explicit 3D microstructural simulation tools spanning the full hierarchy of microstructural scales.

### Symposium A: Advanced Steels and Processing: Stainless Steels

Tuesday PMRoom: 5August 3, 2010Location: Cairns Convention Centre

Session Chairs: Chengjia Shang, University of Science and Technology Beijing; Dong Nyung Lee, Seoul National University

#### 2:00 PM Keynote

Design and Characterization of Fe-Cr-Mn High-Interstitial-Alloyed Austenitic Stainless Steels: Sung-Joon Kim<sup>1</sup>; <sup>1</sup>Korea Institute of Materials Science

Research activities on high-interstitial-alloyed (HIA) stainless steels being carried out in KIMS are presented: alloy development, deformation behavior, corrosion resistance, and precipitation characteristics. The austenitic Fe-Cr-Mn stainless steel sheets with various nitrogen and carbon contents were designed based on thermodynamic calculation, and laboratory heats were fabricated by vacuum induction melting under nitrogen atmosphere. The solution annealed hot-rolled HIA steel sheets exhibited high tensile strength (>900MPa) with good elongation (>50%). As total amount of interstitial elements increased, yield and tensile strengths increased and their deformation mode gradually changed from strain-induced martensitic transformation to deformation twin. The stacking fault energies of the selected alloys, playing an important role in transition of deformation mode, were evaluated by the neutron diffraction analysis, and almost linear relationship between SFE and interstitial element contents was obtained. The simultaneous alloying of nitrogen and carbon enhanced pitting corrosion resistance remarkably. Upon isothermal aging, the precipitation of M23C6 was dominant and sequentially occurred in the forms of grain-boundary, cellular, and intragranular precipitation. The results obtained in HIA stainless steels will be discussed in comparison with our previous works on high nitrogen steels.

#### 2:20 PM

Ductile-to-Brittle Transition Behavior of High-Nitrogen 18Cr-10Mn-0.35N Austenitic Steels Containing Ni and Cu: *Byoungchul Hwang*<sup>1</sup>; Tae-Ho Lee<sup>1</sup>; Seong-Jun Park<sup>1</sup>; Chang-Seok Oh<sup>1</sup>; Sung-Joon Kim<sup>1</sup>; <sup>1</sup>Korea Institute of Materials Science

Influence of Ni and Cu on the ductile-to-brittle transition behavior of highnitrogen austenitic steels was investigated in terms of Charpy impact tests and fracture surface analysis. Five kinds of high-nitrogen Fe-18Cr-10Mn-0.4N austenitic steels with different contents of Ni and Cu were fabricated by melting in a pressurized induction furnace and solution heat-treatments. The fracture mode of the Charpy impact specimens tested at -196°C was cleavagelike brittle fracture with facets along the crystallographic planes {111}, i.e. active slip planes, and thus ductile-to-brittle transition occurred. Although the ductile-to-brittle transition temperature (DBTT) is known to be a function of nitrogen content in Ni-free high-nitrogen Cr-Mn austenitic steels, the DBTT of the steels investigated was varied in accordance with the Ni and Cu contents which may strongly affect the austenite stability associated with deformationinduced martensite transformation (DIMT). In order to interpret DBTT in terms of austenite stability and related DIMT, electron back-scattered diffraction and transmission electron microscopy analyses were performed on the Charpy impact specimens tested at various temperatures.

#### 2:35 PM

Effect of Alloy Addition on Hydrogen Degradation Behavior of 18Mn-0.6C Steels: *Young Soo Chun*<sup>1</sup>; Ji Soo Kim<sup>2</sup>; Hyoungseop Kim<sup>1</sup>; Chong Soo Lee<sup>1</sup>; <sup>1</sup>POSTECH; <sup>2</sup>POSCO

Present study was performed to investigate the hydrogen absorption behavior of 18Mn-0.6C TWIP steels (high Mn steels) with the variation of Al and Si



composition. Three alloys having different alloy compositions (18Mn-0.6C, 18Mn-0.6C-Al and 18Mn-0.6C-Si) were prepared and deformed to various strain level by using high pressure torsion (HPT) equipment. Hydrogen charging was carried out by electrochemical method and the amounts of internally trapped hydrogen was analyzed by thermal desorption spectrometry (TDS). The results showed that with the increase of strain, the trapping sites for diffusible hydrogen such as dislocation,  $\gamma/\epsilon$  interphase boundaries and twins were increased in all three steels. However, Al-added TWIP steels represented much less amount of diffusible hydrogen than the other two steels, due to the less  $\gamma/\epsilon$  interphase boundaries. Accordingly, Al-added steels revealed higher hydrogen delayed fracture resistance.

#### 2:50 PM

Phase Transformation and Annealing Behavior of SUS 304 Austenitic Steel Deformed by High Pressure Torsion: Innocent Shuro<sup>1</sup>; *Minoru Umemoto*<sup>1</sup>; Yoshikazu Todaka<sup>1</sup>; <sup>1</sup>Toyohashi University of Technology

The effects of severe plastic deformation and subsequent annealing on phase transformations, microstructure and mechanical properties of SUS 304 austenitic stainless steel were investigated. Phase evolution, the variation of hardness and strength as a function of annealing temperature and time were determined. Discs were subjected to severe plastic deformation by high pressure torsion (HPT). At low strain (0.2 rpm), nearly 100%  $\alpha$ '-phase was observed but at higher strain rate (5rpm) about 50%  $\gamma$  phase was observed. For the sample deformed at 5 rpm peak hardening occurred after annealing at 673 K for 1 hour with the annealed samples having a higher hardness of 6.6 GPa as compared to the hardness of 5.4 GPa after HPT. Peak hardening was also accompanied by an increase in the volume fraction of martensite ( $\alpha$ ') phase. The phase transformation  $\gamma/\alpha$  was probably assisted by the residual stress in the material. Tensile tests showed that both strength and ductility are enhanced by annealing. Higher annealing temperatures favored  $\alpha'/\gamma$  transformation resulting in a decrease in hardness.

#### 3:05 PM

Strain Rate Dependence of TRIP Behaviour and Deformation Structure in Metastable Austenitic Stainless Steel: *Yoshinori Takagi*<sup>1</sup>; Tatsuya Hukuyama<sup>1</sup>; Takashi Nakamura<sup>1</sup>; Rintaro Ueji<sup>1</sup>; Noriyuki Tsuchida<sup>2</sup>; Takashi Mizuguchi<sup>1</sup>; <sup>1</sup>Japan/Kagawa University; <sup>2</sup>University of Hyogo

The effect of tensile strain rate on transformation induced plasticity (TRIP) behavior in metastable austenitic stainless steel was studied systematically at the wide range of strain rate from  $10^{-3}$ /s to  $10^{3}$ /s. The strength increases and the elongation decreases with increase of a strain rate when the strain rate is slower than  $10^{0}$  /s. At a strain rate larger than  $10^{0/5}$ , both strength and elongation do not change largely by strain rate change. The volume fraction of  $\alpha$ '-martensite by TRIP decreases with the increase of the strain rate and, at a strain rate higher than  $10^{0/5}$ , the  $\alpha$ '-martensite is difficult to be detected by X-ray diffraction even in the sample after fracture. Transmission election microscope observation clarified that the existence of deformation twins in the sample deformed at high speed deformation. The mechanism of the inhibition of TRIP phenomenon will be discussed with the comparison between the deformation structure obtained at high speed deformation and that evolved at the warm temperature deformation ( $100^{\circ}$ C) in which TRIP is suppressed as well.

#### 3:20 PM

**High Velocity Compaction of 316L Stainless Powder**: *Sancai Deng*<sup>1</sup>; Zhiyu Xiao<sup>1</sup>; <sup>1</sup>South China University of Technology

High velocity compaction technology was used to press 316L stainless powders. Effects of impact times on stress wave, green density and ejection force were analyzed. It was found that under the same total impact energy, the first loading time and the actuation duration of the second impact in double impact process were longer when compared with single impact process, while the first delay time was shorter. Furthermore, the green density of compacts prepared by double impact was greater than that prepared by single impact, but no obvious variation in maximum ejection force can be observed between single impact and double impact process.

#### 3:35 PM

A Compatibility Evaluation for Application of Lean Duplex Stainless Steels to Seawater Systems of Nuclear Power Plants: *Hyun Young Chang*<sup>1</sup>; Heung Bae Park<sup>1</sup>; Young Sik Kim<sup>2</sup>; Yoon Young Chang<sup>3</sup>; <sup>1</sup>Korea Power Engineering Company; <sup>2</sup>Andong National University; <sup>3</sup>ANSCO

Lean duplex stainless steels have been developed in Korea for the purpose of being used in the seawater system of industry. There are also many import seawater systems in nuclear power plants. These systems supply seawater to cooling water condenser tubes, heat exchanger tubes, related pipes and chlorine injection systems. The flow velocity of some part of seawater system in nuclear power plants is high and damages of components from corrosion are severe. The considered lean duplex stainless steels are STS329LD(20.3Cr-2.2Ni-1.4Mo) and STS329J3L(22.4Cr-5.7Ni-3Mo) and PRES of them are 24.8 and

32.5 each. Physical, mechanical and microstructurla properties are evaluated, and electrochemcal corrosion resistance are measured quantitatively in NaCl solution. CPTs are measured on these alloys and pit depths are evaluated using lazer microscope. From the data of RRENs and CPTs of these alloys, the time of pitting initiation and life expectancies for the replacement are estimated in the evironment of condensers and heat exchangers of nuclear power plants. Long period field test on these alloys are now being performed, and some results are going to be presented.

3:50 PM Tea Break

### Symposium C: Light Metals and Alloys: Microstructures of Aluminium Alloys I

Tuesday PMRoom: 6August 3, 2010Location: Cairns Convention Centre

Session Chair: Michael Mills, The Ohio State University

#### 2:00 PM Keynote

**Precipitation Sequence in an Al-Si-Mg Foundry Alloy**: Barbara Rinderer<sup>1</sup>; *Malcolm Couper*<sup>2</sup>; Xiangyuan Xiong<sup>3</sup>; Sam Gao<sup>4</sup>; Jian-Feng Nie<sup>4</sup>; <sup>1</sup>Consulting in Partnership Pty Ltd; <sup>2</sup>Rio Tinto Alcan; <sup>3</sup>Monash Centre for Electron Microscopy; <sup>4</sup>Monash University

It is often assumed that the precipitation sequence and phases are similar in wrought 6xxx Al-Mg-Si alloys, such as 6063 and Al-Si-Mg foundry alloys, such as A356, which have very high levels of silicon. The foundry alloys have been less extensively studied due to added difficulties in sample preparation for TEM, resulting from the network of coarse silicon particles. However, recent work has been successful in studying the foundry alloys. The work highlights the differences and similarites bewteen wrought and foundry alloy precipitation and discusses the implications for alloy design and heat-treatment.

# Tue. PM

#### 2:20 PM

Microstructural Evolution in Various Regions of Stir Zone during Friction Stir Processing of Al-7Si-0.3Mg Cast Alloy: *Zhan Chen*<sup>1</sup>; Song Cui<sup>1</sup>; Wei Gao<sup>2</sup>; Tianping Zhu<sup>2</sup>; <sup>1</sup>AUT University; <sup>2</sup>The University of Auckland

Conventional Al-7Si-0.3Mg castings may be used more as structural components if friction stir (FS) processing could be used to eliminate defects and refine microstructure in critical locations. However, a FS zone is not necessarily refined satisfactorily and uniformly. To considerably enhance the properties, the whole FS volume featured with refined microstructure needs to be achieved. For this, we aim to understand the microstructure development of the cast alloy during FS. Experiments were conducted to evaluate how rotation and forward speeds affect material flow and the formation of various microstructural regions. It will be shown that the deforming Al dendrites and eutectic Al-Si as they approach the pin result in a complete mix of a fully recrystallized Al grains and fine Si particles - as RSM. Material merely dragged into FS zone remains microstructurally segregated - as DFM. RSM/DFM ratio affected by rotation and forward speeds will also be presented and the mechanism, the effect of the thread on RSM, for this will be explained. Finally the strain values estimated, taking the deforming Al dendrites as markers, that are consistent with the textures determined using EBSD analysis will be presented for a better understanding of the dynamic recrystallization during FS.

#### 2:35 PM

Alloy Design for Enhancing the Fracture Resistance of Heat Treated High Pressure Die-Castings: *Roger Lumley*<sup>1</sup>; Maya Gershenzon<sup>1</sup>; Dayalan Gunasegaram<sup>1</sup>; <sup>1</sup>CSIRO Light Metals Flagship

Recently, heat treatment technologies have been developed by the CSIRO Light Metals Flagship in Australia that allow the 0.2% proof stress of conventional aluminum alloy high pressure diecastings (HPDC's) to be more than doubled without encountering problems with blistering or dimensional instability. A range of other properties may also be improved such as fatigue resistance, thermal conductivity and fracture resistance. However, the current HPDC Al-Si-Cu alloys have not been developed to exploit heat treatment or to optimize specific mechanical properties. One potential limitation of heat treating such high pressure diecastings is that fracture resistance may be reduced as strength is increased. In the current paper, we present the outcomes of a program to develop highly castable, secondary Al-Si-Cu HPDC alloys which display significantly enhanced ductility and fracture resistance in both the as-cast and heat treated conditions. Kahn-type tear tests were conducted to compare the fracture resistance of the conventional A380 alloy with a selection of the newly developed compositions.



A comparison is also made to current permanent mold cast aluminium alloys, and it is shown that the new HPDC compositions typically display both higher levels of tensile properties and fracture resistance.

#### 2:50 PM

Study on Quench-Induced Precipitation Behavior in an Al-7.5Zn-1.7Mg-1.4Cu-0.12Zr Alloy: *Baiqing Xiong*<sup>1</sup>; Xiwu Li<sup>1</sup>; Yongan Zhang<sup>1</sup>; Zhihui Li<sup>1</sup>; Baohong Zhu<sup>1</sup>; Feng Wang<sup>1</sup>; Hongwei Liu<sup>1</sup>; <sup>1</sup>General Research Institute for Nonferrous Metals

Quench sensitivity of Al-Zn-Mg-(Cu) alloys is a major concern in the aerospace industry, where the growing tendency towards the ultra-thick section products. Quench-induced precipitation behavior in an Al-7.5Zn-1.7Mg-1.4Cu-0.12Zr alloy is investigated by using Jominy end quench method and TEM analysis, compared with that of traditional AA 7B04 and AA7150. The results indicate that when quenching cooling rate decreases, quench-induced precipitation phenomena occurs at the grain boundary and subgrain boundary by preference, and then  $\eta$  precipitates nucleate attached to the second-phase particles, such as Al3Zr dispersoids, chromium or manganese-containing dispersoids. On the comparison of the conductivity curves in quenching condition, the T6 temper hardness change curves and quench-induced precipitation features among these three kinds of aluminum alloys, AA 7B04 exhibits the highest quench sensitivity followed by AA7150, and the alloy show the lowest levels.

#### 3:05 PM

Effect of Pre-Ageing on the Artificial Ageing Response of Al-Mg-Si(-Cu) Alloys: *Lingfei Cao*<sup>1</sup>; Paul Rometsch<sup>1</sup>; Hao Zhong<sup>1</sup>; Barry Muddle<sup>1</sup>; <sup>1</sup>Monash University

The effect of different pre-ageing treatments on the subsequent artificial ageing response of Al-Mg-Si(-Cu) alloys has been investigated using hardness, tensile and electrical conductivity testing. The microstructural evolution was characterised by transmission electron microscopy (TEM) and 3-dimensional atom probe (3DAP) analysis. Pre-ageing treatments were carried out either at low temperatures of 65-120°C for relatively long holding times or at higher temperatures of 200-250°C for shorter times. Results show that the early stage artificial ageing response after 30 minutes at 170°C is strongly influenced by the pre-ageing and natural ageing conditions. A pre-ageing treatment performed for a short time at a high temperature and within a short delay after solution treatment was found to give a promising hardening response during subsequent artificial ageing. The mechanisms of pre-ageing to reduce the detrimental effect of natural ageing on the artificial ageing response will be discussed in relation to the formation and distribution of clusters, GP zones and/or precipitates.

#### 3:20 PM

Quantitative Analysis of Precipitate Compositions in an Al-Li-Mg-Cu Alloy Using Atom Probe Tomography: *Xiangyuan Xiong*<sup>1</sup>; Stavroula Moutsos<sup>1</sup>; Russell King<sup>1</sup>; Barry Muddle<sup>1</sup>; <sup>1</sup>Monash University

The mechanical properties and microstructures of Al-Li-Mg-Cu alloys have been studied extensively, due to the low density and high stiffness of alloys with the potential to replace conventional aluminium alloys used in aircraft structures. The microstructure and local composition distributions are important for optimizing the mechanical properties and designing new alloys. Previous studies of alloys of the type AA8090 by transmission electron microscopy have shown a microstructure comprising homogeneously distributed coherent spherical d' (Al<sub>3</sub>Li) precipitates and semi-coherent phases, S' (rod or lath-like) and T1 (platelike), after an isothermal ageing treatment at 200°C. Because these precipitates are very small, ~20 nm in diameter for the d' precipitates, accurate compositions of the precipitates in this alloy have not yet been determined. The 3-dimensional atom probe (3DAP) offers a unique technique for determining the local composition distribution on the nanometre scale. However, for the Al-Li-Mg-Cu alloys, due to the large differences in the evaporation field of the component elements, the measured composition is affected by the experimental conditions. In this work, the effects of various experimental conditions will be examined and discussed, and a set of optimum experimental conditions will be demonstrated to yield correct compositions of the precipitates using a fast 3DAP.

#### 3:35 PM

**Dispersoid Phases in 6xxx Series Aluminium Alloys**: *Katharina Strobel*<sup>1</sup>; Mark Easton<sup>1</sup>; Elizabeth Sweet<sup>1</sup>; Jain-Feng Nie<sup>1</sup>; Malcolm Couper<sup>2</sup>; <sup>1</sup>CAST CRC - Monash University; <sup>2</sup>Rio Tinto Alcan

In high strength 6xxx series aluminium alloys additions of transition metals such as Mn and Cr lead to formation of dispersoid phases of  $Al_{15}(Fe,Mn)_3Si_2$ ,  $Al_{15}(Fe,Cr)_3Si_2$  and  $Al_{13}Cr_4Si_4$  that can improve fracture toughness and inhibit recrystallization and non-uniform grain growth during extrusion. However, these dispersoids can also act as heterogeneous nucleation sites for non-hardening Mg<sub>2</sub>Si phases if the quench rate after extrusion is too low. This leads to a reduction in mechanical properties as these alloys are predominantly strengthened by Mg-Si precipitates. The composition and volume fraction of the

strengthening precipitates is strongly dependent on the amount of Mg and Si in solid solution, i.e. the maximum achievable strength is dependent on quench rate. The removal of solute Mg and Si by nucleation on the intermetallic dispersoids leads to reduction in mechanical properties - a phenomenon known as quench sensitivity. There have been a number of studies focussing on dispersoid phases, their formation and effect on the mechanical properties of the alloys. This work will give an overview of the state of current understanding of quench sensitivity and will identify the key factors that affect the severity of this phenomenon.

3:50 PM Tea Break

## Symposium C: Light Metals and Alloys: Microstructures of Aluminium Alloys II

Tuesday PMRoom: 6August 3, 2010Location: Cairns Convention Centre

Session Chair: Malcolm Couper, Rio Tinto Alcan

#### 4:30 PM Keynote

High Resolution STEM Analysis of Metastable Phases in High Strength Al-Mg-Cu and Al-Zn-Mg-Cu Alloys: *Michael Mills*<sup>1</sup>; Libor Kovarik<sup>1</sup>; Yi-Yun Li<sup>1</sup>; <sup>1</sup>The Ohio State University

The microstructure and chemistry of the metastable phases in Al alloys remain controversial despite many previous efforts devoted to their understanding. With the advent of probe-corrected STEM, it is now possible to resolve the lattice spacings in the aluminum matrix along several zone axes. High angle annular dark field is a particularly useful imaging mode for characterizing the structure of metastable phases at the atomic scale since it is quite sensitive to the net atomic number of the atomic columns. In this presentation, examples of the analysis of metastable phases in both Al-Mg-Cu alloys and Al-Zn-Mg-Cu alloys will be highlighted. With respect to the former, the structure of GPB zones and their relationship to the rapid hardening response exhibited by these alloys will be discussed. With respect to the latter, the structure of GP zones relative to the subsequent sequence of precipitation will be presented. The results shed light on the important effect of Cu on the precipitation sequence, precipitate coarsening, morphology and hardening rate. These results will also be discussed in the context of previous work in the literature.

#### 4:50 PM

Microstructural Characteristics of Electron Beam Processed Al-2Sc: Dacian Tomus<sup>1</sup>; Ma Qian<sup>2</sup>; Craig Brice<sup>3</sup>; Colleen J. Bettles<sup>1</sup>; Barry Muddle<sup>1</sup>; Peng Yu<sup>2</sup>; <sup>1</sup>Monash University; <sup>2</sup>The University of Queensland; <sup>3</sup>Lockheed Martin Aeronautic Company

Scandium is a light (2.99 g cm -3) but most potent hardening element for aluminium on a per atom basis. However, the amount of Sc that can be precipitated for hardening by a normal solution treatment is rather limited because of its limited maximum solubility in  $\alpha$ (Al) (0.35 wt%). Electron beam direct manufacturing is a novel layer additive manufacturing technique developed since 2002. Apart from near net shape forming, the process also offers a promising route for novel alloy design because of the fast cooling rate involved. Plate samples of Al-2wt%Sc alloy were processed using a 50kV EB gun at 10 to 30 mA and 0.6 m/min. They were then aged at 288°C for 8 hr to assess the hardening effect. It is shown that after EB processing, the primary Al<sub>3</sub>Sc phase disappeared and the average composition of the  $\alpha$ (Al)-Al<sub>2</sub>Sc eutectic colony increased from 0.77 to 1.89 (wt.%). The Vickers hardness increased from 45 HV of the aged base alloy to 80 HV of the EB as-processed and further to 110 HV after ageing. Electron microscopic examination confirmed a dense distribution of fine Al, Sc precipitates in the supersaturated eutectic  $\alpha$ (Al). EB processing enables significant hardening by Sc.

#### 5:05 PM

**TEM Observation of Metastable Phases in Aged Al-Mg-Ge Alloys**: *Kenji Matsuda*<sup>1</sup>; Junya Nakamura<sup>1</sup>; Keisuke Yamamoto<sup>1</sup>; Tokimasa Kawabata<sup>1</sup>; Yasuhiro Uetani<sup>2</sup>; Susumu Ikeno<sup>1</sup>; <sup>1</sup>University of Toyama; <sup>2</sup>Toyama Prefectural University

Al-Mg-Ge alloys have been reported that it has the similar precipitation sequence to that in Al-Mg-Si alloy, because Ge is homologous element as Si in the periodic table. The purpose of study is to investigate aging behavior, crystal structures of metastable phase and relative frequency of metastable phases in aged Al-Mg-Ge and Al-Mg-Ge with Si alloy using high resolution transmission electron microscope (HRTEM), energy dispersive X-ray spectroscopy (EDS) and electron energy-loss spectroscopy (EELS). Microvickers hardness measurement



and TEM observation was performed in Al- 0.52at%Mg-0.24at%Ge (balanced), Al - 0.55at.% Mg - 0.18at.% Ge (Mg-rich 1), Al- 0.95at.%Mg-0.22 at.%Ge (Mg-rich2), and Al- 0.49 at.%Mg -0.36 at.%Ge (Ge-rich) alloys. Also, alloys including Si were prepared as Al- 0.49at%Mg-0.19 at%Ge -0.38at%Si and Al - 0.55at.% Mg - 0.09at.% Ge - 0.10at.%Si alloy aged at 523K. Every alloy included rod-shaped precipitate which is the same as the typical metastable pahse,  $\beta$ ', in Al-Mg-Si alloy. Except to Mg-rich 1 and 2 alloys, the Type-A precipitates, which is a typical metastable phase in the excess Si type Al-Mg-Si alloys and popular at over aged condition, were confirmed as a large rod-shaped precipitates in alloys. This behavior is probably depends on the ratio of Mg/Ge.

#### 5:20 PM

Study of Residual Stress Relief by Cryogenic Treatment in the Al 6061: *Kijung Park*<sup>1</sup>; Hoi-Bong Kim<sup>1</sup>; Bin Huang<sup>1</sup>; Young-Rae Cho<sup>1</sup>; <sup>1</sup>Pusan National University

Aluminum alloys have long been of interest to the military community due to their modest specific strength. Processes such as heat treatment and machining may induce residual stresses in Al alloys. This residual stresses in Al alloys may generate distortion which causes some serious problems in shape. The cryogenic process, a thermal process in which an object is cooled to -196°C with liquid nitrogen (LN<sub>2</sub>), has been applied as a method to relieve residual stresses in the Al alloys. In this study, a cryogenic treatment was tried to relieve the residual stresses of the Al 6061. The tested cylindrical samples have the dimension of 20(diameter)×24(height) mm, which contained 97.42 Al, 0.23 Cu, 1.08 Mg, and 0.74 Si (wt. %). The samples were heated at 530°C for a period of 30 minutes and quenched immediately in different quenching mediums as water and LN<sub>2</sub>. Then, the samples were reheated at 175°C for a period of 0, 3, 6, 9, 12 hours, respectively. The samples were etched in Kellers. Residual stresses were measured by XRD analysis and the precipitates were observed by TEM analysis. The results showed that the cryogenic treatment (LN<sub>2</sub>) significantly reduced the residual stresses by 71.5% in the Al 6061.

#### 5:35 PM

Influence of Solution Treatment on Microstructure and Quench Cracking in a Water-Quenched Aluminium Alloy 7150: *Daokui Xu*<sup>1</sup>; Paul Rometsch<sup>1</sup>; Hua Chen<sup>1</sup>; Barry Muddle<sup>1</sup>; <sup>1</sup>Monash University

Multi-step solution treatments with progressively increasing temperatures have been carried out on an as-rolled 7150 aluminium alloy. The influence of these solution treatments on the constituent particle dissolution, overheating and associated quench cracking behaviour have been investigated after quenching in room temperature water. For comparison, the microstructure and quench cracking behaviour of single-step solution treated samples that were water quenched from a temperature range of 475-505°C were also investigated. By measuring the volume fraction of remaining constituents in differently solution treated samples, the effective dissolution capability of multi-step solution treatment procedures has been demonstrated. Based on the optical microscopy of etched microstructures of differently quenched samples, the critical conditions for quench crack occurrence in 7150 Al alloy have been investigated and the influence of overheating of constituents on quench crack initiation will be discussed. To understand and compare the quench cracking mechanisms of differently solution treated samples, the as-quenched samples were fractured along existing quench cracks and fracture surfaces were analysed with scanning electron microscopy (SEM) and EDS analysis.

#### 5:50 PM

#### Effect of Zr Addition on Microstructure and Warm Formability of Al-Mg Sheet Alloys: *Hanliang Zhu*<sup>1</sup>; M. S. Dargusch<sup>1</sup>; <sup>1</sup>University of Queensland

The trace element Zr was added to an Al-Mg alloy to investigate its effects on microstructure and warm formability of aluminium sheets. Microstructural examination showed that the Zr addition resulted in an elongated grain structure due to inhibited recovery and recrystallization by Al<sub>3</sub>Zr particles during thermomechanical treatments. In order to evaluate the warm formability, warm tensile tests were carried out at temperatures ranging from 275 to 350°C, and at strain rates ranging from 0.015 to 1.5 s-1. The results showed that the addition of Zr increased the flow stress but decreased the ductility at most test conditions. At a low strain rate of 0.015 s-1, the addition of Zr resulted in low ductility at 350°C but high ductility at 275°C. The effect of Zr additions on warm formability is discussed on the basis of the microstructural changes.

#### 6:05 PM

Multiphase Light Alloys and Nanocomposites through Consolidation of Particles by Severe Plastic Deformation: *Edward Lui*<sup>1</sup>; Wei Xu<sup>1</sup>; Xiaolin Wu<sup>1</sup>; Shouqie Goussous<sup>1</sup>; Yi Sun Wu<sup>1</sup>; Kenong Xia<sup>1</sup>; <sup>1</sup>Department of Mechanical Engineering and ARC Centre of Excellence for Design in Light Metals, The University of Melbourne

Consolidation of particles via severe plastic deformation (SPD) based techniques such as high pressure torsion (HPT) and equal channel angular

pressing (ECAP) offers several advantages over conventional high temperature sintering, including lower processing temperatures, shorter processing times, and refined and more homogeneous microstructures. SPD consolidation is particularly suitable for multiphase materials with two or more distinct structures. The lower processing temperature used in back pressure ECAP (BP-ECAP) allow unique and often desirable microstructures of the starting powder to remain unaltered after consolidation. For example, ball-milled elemental Ti-47at.%Al powder mixture with refined grains was successfully consolidated at 350°C without any reactions between the Ti and Al particles. The resulting high yield strength and good ductility of the material can be attributed to the fine grains and finely distributed Ti and Al phases. Supersaturated solid solution Al(Ti) powders formed during mechanical alloying were also consolidated using BP-ECAP, forming intermetallic phases at higher temperatures. In another instance, Al reinforced with nano-scale carbon or alumina particles were obtained using BP-ECAP consolidation, resulting in significant increase in strength through dispersion hardening and grain refinement. Finally, nano-sized Al particles were consolidated through BP-ECAP, resulting in a multiphase material of nanograined Al, alumina, and amorphous structure.

#### 6:20 PM

The Microstructure Characteristics and Wear Resistance of Rapidly Solidified Hypereutectic Al-Si Alloys: *Li Jiwen*<sup>1</sup>; Wang Aiqin<sup>1</sup>; Xie Jingpei<sup>1</sup>; Wang Wenyan; <sup>1</sup>Henan University of Science and Technology

In this paper, rapidly solidified hypereutectic Al-21Si and Al-30Si alloys are prepared by the single roller melt-spinning technique, and the microstructure and wear resistance are studied. It found that the grains are refined and the micro-nano composite structureds are formed under rapid solidification. The microstructures of the Al-21Si alloys are composed of primary micro-nanostructured a-Al phase and feather-needles-like a-Al+B-Si eutectic. The nucleation and growth of primary silicon are suppressed and primary silicon can not be precipitated. The a-Al phase is the leading phase. The microstructures are shown the typical hypoeutectic microstructure characteristics. For the Al-30Si alloy, the microstructures are the typically hypereutectic microstructure. The primary Si phase is precipitated and refined. The substructures of the primary Si phase shows the typical twin morphologies, and form the mirror symmetry orientation relations along with the twin surface. The wear resistance of the rapidly solidified Al-21Si alloys are improved five times than that of the the conventional alloys. Moreover, with the increase of the Si content, the wearability is improved. The wearability of the Al-30Si alloy is 1.7 times than that of the Al-21Si alloy. The wear mechanism of the rapidly solidified alloy is plastic deformation wear and abrasive wear.

## Symposium C: Light Metals and Alloys: Texture in Magnesium Alloys

Tuesday PMRoom: CAugust 3, 2010Location: Cairns Convention Centre

Session Chairs: Jian-Feng Nie, Monash University; Fusheng Pan, Chongqing University

#### 2:00 PM Keynote

Development of Microstructure and Texture in RE containing Magnesium Alloys during Hot Rolling: *Karl Kainer*<sup>1</sup>; Joachim Wendt<sup>2</sup>; Kerstin Hantzsche<sup>1</sup>; Jan Bohlen<sup>1</sup>; Sangbong Yi<sup>1</sup>; Dietmar Letzig<sup>1</sup>; <sup>1</sup>GKSS Research Centre Geesthacht; <sup>2</sup>Hamburg University of Technology

Despite their great potential magnesium sheets have only played a limited role in lightweight structural applications. A major technical reason is the fact that commercial magnesium sheets like AZ31 are limited in their ductility and formability especially at room temperature. Due to the hexagonal close packed lattice structure of magnesium only a limited number of deformation mechanisms are available at room temperature, which leads to a limitation of formability and to an intrinsic plastic anisotropy as well. Especially during massive deformation such as rolling, strong crystallographic textures appear. Whilst the texture evolution during recrystallisation of magnesium alloys such as AZ31 is negligible, a randomisation of the texture is possible in allovs with rare earth elements in solid solution. The exact mechanism, however, is not yet fully understood. In order to achieve a better understanding of the effect of RE-elements a detailed study of microstructure and texture development was performed with Al-free RE-containing wrought magnesium alloys based on Zn during hot rolling. The paper will address the effects of deformation and recrystallisation on texture development. Special focus will be put on the broadening of texture during all rolling passes from the initial as cast state to final gauge.



#### 2:20 PM Keynote

Effects of Twin on Deformation Behavior of Magnesium Single Crystals: Ming Zhe Bian<sup>1</sup>; Nam Kyoung Kwon<sup>1</sup>; Hwa Chul Jung<sup>1</sup>; *Kwang Seon Shin*<sup>1</sup>; <sup>1</sup>Seoul National University

The mechanical behavior of HCP metals is strongly influenced by their inherent anisotropic characteristics that originate from the crystal structure. The resolved shear stresses that activate the prismatic, pyramidal and <c+a> slip modes are much greater than those required to initiate the basal slip and tensile twin in magnesium at room temperature. Therefore, the predominant deformation modes of magnesium at room temperature are the basal slip and tensile twin. While the slip behavior of magnesium alloys has been extensively investigated, the effects of the twin on the deformation behavior of magnesium single crystals has not been thoroughly examined yet, even though twinning is an important mode of deformation in HCP metals. The interactions between the dislocations and twin boundaries are important because the deformation-induced twins could act as obstacles to dislocation motion. In this study, the effects of the twin on the deformation of magnesium single crystals were systematically investigated using scanning electron microscopy and electron backscattering diffraction (EBSD) analyses.

#### 2:40 PM Keynote

Microstructure and Texture Evolution of Twin-Roll Cast Magnesium Alloys during Thermo-Mechanical Treatments: Kyung-hun Kim<sup>1</sup>; Byeong-Chan Suh<sup>1</sup>; Jun Ho Bae<sup>1</sup>; Myeong-Shik Shim<sup>1</sup>; Nack J. Kim<sup>1</sup>; <sup>1</sup>POSTECH

Mg alloys have the great potential for high performance automotive applications due to their low density and high specific strength. For the successful application of Mg allov sheet products, thermo-mechanical treatment (TMT) such as hot/warm rolling is needed to modify the microstructure so that an optimum combination of mechanical properties can be obtained. Basal texture, however, becomes stronger during TMT, which has an adverse effect on the formability at low temperatures. Also such textured alloys usually exhibit strong tension-compression strength asymmetry. Therefore it is of utmost importance to randomize the texture of Mg alloys by various TMTs. In the present study, microstructure and texture evolution of twin-roll cast Mg-6Zn-1Mn-1Al (ZMA611) and Mg-6Zn-1Y (ZW61) allows have been investigated. It is shown that recrystallization occurs through a shear (deformation) band nucleation mechanism. Alloy composition has a large effect on the nature of the deformation bands formed during rolling, resulting in a difference in the orientation of recrystallized grains between the two alloys; double twin-related in ZMA611 and tension twin-related in ZW61. Such a contrast in microstructure results in different (002) pole figures between the two alloys.

#### 3:00 PM Invited

Texture in Magnesium Alloys: Matthew Barnett1; 1Deakin University

The extrusion of metals often involves the application of relatively large deformation strains in a single "hit". These conditions produce sharp crystallographic textures. In magnesium these textures play a significant role in determining the mechanical response. After a short summary of some key previous findings on the topic, the present paper describes three mechanisms of texture selection that we believe are active during the extrusion of magnesium alloys. Both simulation and observation are employed to make the case.

#### 3:15 PM

# Twinning-Induced Negative Strain Rate Sensitivity in Wrought Magnesium Alloy AZ31: Young Chun<sup>1</sup>; Chris Davies<sup>1</sup>; <sup>1</sup>Monash University

Measurements of strain rate sensitivity (SRS) provide a key link between dislocation-based interpretations of plastic deformation and macroscopic measurements made in mechanical tests. It is well known that plastic deformation of hcp metals is achieved not only by dislocation glide but also by twinning and that the atomic rearrangement underlying the latter mode is different from that of slip. This leads to an expectation that co-activation of twinning may affect SRS of hcp metals. This assumption was tested in the present work where strain rate jump tests in both tension and compression were conducted on highly textured AZ31 plate. It was found that the SRS of the alloy in tension decreased with strain whereas that in compression increased with strain, exhibiting negative values at low strain and positive values at higher strain. Microstructure analyses revealed that the strain regimes where negative SRS or decreasing trend in SRS with strain was observed correspond to extensive twinning, implying a negative SRS of twinning. It is concluded that dislocation model alone cannot explain the strain rate dependence of flow stress in the metals whose deformation is assisted by twinning.

### 3:30 PM

Effects of Alloying Elements and Processing Conditions on Textures and Mechanical Properties of Magnesium Alloys: Jung Woo Choi<sup>1</sup>; Cheol-Seung Hyun<sup>1</sup>; Ji Hoon Hwang<sup>1</sup>; *Kwang Seon Shin*<sup>1</sup>; <sup>1</sup>Seoul National University

In recent years, there have been attempts to improve the formability of wrought Mg alloys by controlling their textures with the addition of rare earth elements such as Y, Ce, La and Gd, and Zn as alloying elements. There have also been limited efforts to examine the effects of processing conditions such as extrusion temperature, reduction ratio and strain rate on the textures of wrought Mg alloys. In the present study, the changes in the deformation texture were examined in Mg alloys with various compositions that were extruded at different temperatures. The effects of the alloying elements and extrusion temperatures on the textures of the extruded Mg alloys were examined systematically using X-ray diffraction and electron backscattering diffraction (EBSD) analyses. The development of the microstructure and texture during the extrusion of Mg alloys was also examined. It was found that the textures and mechanical properties of extruded Mg alloys were strongly affected by the alloying elements and processing conditions.

#### 3:45 PM

# Annealed Microstructure of Gum Metal Sheets after Cold Working: Yu Ohta<sup>1</sup>; Goroh Itoh<sup>1</sup>; Yoshinobu Motohashi<sup>1</sup>; <sup>1</sup>Ibaraki University

Gum Metal is a newly developed beta titanium alloy, which has a low elastic modulus, high strength, highly super-elastic characteristics, super-plastic characteristics, Inver-like and Elinvar-like thermal dependence of expansion coefficient and elastic modulus. Some of these special characteristics have been attributed to its plastic deformation manner based on non-dislocation mechanism. The present study deals with the microstructural change in Gum Metal sheets that are cold-worked by widely ranging reductions and subsequently annealed, focusing on whether strain-induced grain boundary migration (SIBM) occurs in the Gum Metal which is a popular annealing process in most metallic materials containing medium density of dislocations. Microstructures in L-ST sections (perpendicular to the transverse direction) of the Gum Metal were observed by using optical microscope. The results obtained were compared with those for a Ti-15V-3Cr-3Sn-3AI alloy, typical beta titanium alloy. It was confirmed that the number of traces of SIBM was smaller in the Gum Metal than in the Ti-15V-3Cr-3Sn-3AI.

4:00 PM Tea Break

### Symposium C: Light Metals and Alloys: Texture and TMP of Magnesium Alloys

Fuesday PM	Room: C
August 3, 2010	Location: Cairns Convention Centre

Session Chairs: Nack J. Kim, POSTECH; Matthew Barnett, Deakin University

#### 4:30 PM

Influence of Grain Boundary on Activation of Slip Systems in Magnesium: Crystal Plasticity Analysis: *Tsuyoshi Mayama*<sup>1</sup>; Tetsuya Ohashi<sup>2</sup>; Kenji Higashida<sup>3</sup>; <sup>1</sup>Kumamoto University; <sup>2</sup>Kitami Institute of Technology; <sup>3</sup>Kyushu University

Crystal plasticity finite element analysis method considering the accumulation of geometrically necessary (GN) dislocations was applied to monotonic loading of pure magnesium bi-crystal. The deformation mechanisms considering in the present analysis method are basal slip <a>, prismatic slip <a>, 1st order pyramidal slip <a>, 2nd order pyramidal slip <a+c> and tensile twinning <a+c>. Tensile twinning is incorporated into crystal plasticity analysis assuming that twinning plane and direction of shear by twinning are equivalent to slip plane and slip direction, respectively. Critical resolved shear stresses (CRSSs) for each slip system in the literatures were used. Analysis model is designed to investigate the influence of grain boundary on the activation of slip systems. That is, one grain consisting of bi-crystal (grain A) had the crystal orientation whose Schmid factor for prismatic slip is 0.5. The crystal orientation of the other grain (grain B) was slightly deviated from that of grain A. The result of the calculation of tensile loading of the bi-crystal showed that both grains are deformed by the multiple slip of basal slip system, which resulted in the formation of GN dislocation bands.



### 4:45 PM

Thermal Stability and Effect of Texture on Ultrahigh Damping of Nanocrystalline Mg-Matrix Composites Reinforced with MAX Phases: Shahram Amini<sup>1</sup>; José Córdoba Gallego<sup>2</sup>; Magnus Odén<sup>2</sup>; Lars Hultman<sup>2</sup>; *Michel Barsoum*<sup>1</sup>; <sup>1</sup>Drexel University; <sup>2</sup>Linkoping University

We developed a unique, simple and cost effective technique to fabricate Ti<sub>2</sub>AlC-reinforced Mg-matrix composites – wherein the Mg-grain size is in the  $\sim$ 35±15 nm range – by pressureless melt infiltration. Not only do these nanograins form spontaneously, but more importantly, they are so thermally stable that taking the composite to 50°C over the melting point of Mg three times, does not result in their coarsening. Because of their nano-size, their melting point is depressed, in some cases by 40°C. The composites exhibit exceptional damping capabilities because of the presence of the MAX phase. The presence of the Mg, allows the Ti<sub>2</sub>AlC to form larger incipient kink bands than in bulk dense Ti<sub>2</sub>AlC. It also imparts the composite with exceptional Vickers hardness (2 GPa), compressive (700±10 MPa), and tensile (380±20 MPa) strengths. The technological implications of having a readily machinable, relatively inexpensive, strong, stiff (effective moduli ~ 100 GPa) and damping solid will be discussed.

#### 5:00 PM

Microstructure and Mechanical Propeties of Extruded Mg-Zn-Ca Alloy: Mingyi Zheng<sup>1</sup>; L.B. Tong<sup>1</sup>; X.S. Hu<sup>1</sup>; K. Wu<sup>1</sup>; S.W. Xu<sup>2</sup>; S. Kamado<sup>2</sup>; <sup>1</sup>Harbin Institute of Technology; <sup>2</sup>Nagaoka University of Technology

Extrusion of the as-cast Mg-5.25 wt.% Zn-0.6 wt.% Ca alloy has been performed at different extrusion velocity (0.1mm/s, 0.3mm/s and 0.5mm/s) in the temperature range of 270-330°C. Both extrusion temperature and extrusion velocity have significant effect on the grain size, texture and mechanical properties of the Mg-Zn-Ca alloy. Upon extrusion, the as-cast coarse grains underwent pronounced grain refinement and the second phases were broken up and formed stringers in the extrusion direction. With the decreasing extrusion temperature to 270°C, the grain size of the alloy was refined to about 1.5  $\mu$ m, basal texture became stronger, which led to the higher yield strength, tensile strength and moderate elongation to failure. While with the increasing of extrusion velocity, the grain size of the alloy was increased, basal texture became weaker, accordingly, the as-extruded alloy exhibited lower yield strength, tensile strength and higher elongation to failure.

#### 5:15 PM

Deformation Behavior of Twin-Roll Cast Mg<sup>-4</sup>Zn<sup>-1</sup>Gd Alloy Sheet: *Byeongchan Suh*<sup>1</sup>; Jun Ho Bae<sup>1</sup>; Myeong-shik Shim<sup>1</sup>; Dong-wook Kim<sup>1</sup>; Nack J. Kim<sup>1</sup>; <sup>1</sup>POSTECH

Mg alloys generally have low ductility and formability at room temperature. It has been reported that the activation of mechanical twinning such as (101) and (10.2) twins can improve the ductility and formability of Mg alloys. Nevertheless, there still remain uncertainties how the mechanical twinning affects the deformation behavior of Mg alloys. In this work, tensile deformation behavior of twin-roll cast Mg-4Zn-1Gd alloy was investigated with particular emphasis on the role of mechanical twinning. In order to clarify the effect of mechanical twinning on the deformation behavior, the alloy was subjected to several steps of loading and unloading along the rolling and transverse direction to analyze the nucleation and growth of twins and their interaction with dislocations. It shows that the pre-existing twins having their c-axes parallel to loading direction rotate to the orientations of their respective parent grains by detwinning. Volume fraction of deformation twins increases with an increase in the amount of deformation and the dominant twinning mode has been found to be tension twins, which have high Schmid factors for basal slip. Such change in the local texture induced by the formation of tension twins could improve the ductility and formability of Mg alloys.

#### 5:30 PM

Warm Hydroforming Process with Non-Uniform Heating for AZ31 Magnesium Alloy Tube: Ken-ich Manabe<sup>1</sup>; *Toshiji Morishima*<sup>1</sup>; Yu Ogawa<sup>1</sup>; Kazuo Tada<sup>1</sup>; Tsutomu Murai<sup>2</sup>; Humiaki Nakagawa<sup>2</sup>; <sup>1</sup>Tokyo Metropolitan University; <sup>2</sup>Sankyo Tateyama Aluminum Industry

Nowadays, the reduction of automotive weight is required to realize lowcarbon society. In tube hydroforming process of magnesium alloy, it has been confirmed that the material temperature affect the formability of tubes, and it is highly important that the determination of the temperature conditions. In the previous report, it was showed that the non-uniform temperature distribution is effective for making hydroformed part with uniform wall thickness in T-joint THF. However, the effect of temperature distribution on wall thickness did not clarify. The objective of this study is to investigate the effect of temperature distribution on wall thickness and evaluate the effect quantitatively. For this purpose, the finite element simulations are performed to analyze the appropriate temperature with the use of various temperature conditions. The simulation code used is the dynamic explicit FEM code LS-DYNA. In this study, a coupled structural-thermal analysis is conducted. The validity of the FE model of T-joint THF is verified by comparing the FE simulation and experimental results. As a result, it is showed that the wall thickness of the T-joint hydroformed parts can be more uniform with the appropriate temperature conditions.

#### 5:45 PM

# **Deep Drawing of AM31 Magnesium Alloys**: *Jaehyung Cho*<sup>1</sup>; SukBong Kang<sup>1</sup>; <sup>1</sup>Korea Institute of Materials Science

Texture and microstructure evolutions during deep drawing of AM31 magnesium alloys were investigated at various temperatures and deformation rates. Two different sheets were originally fabricated by twin roll strip casting and conventional ingot casting. They were warm-rolled down to 0.6mm and then fully-annealed for deep drawing process. Working temperatures were 200°C to 350°C and punch rates were 30mm/min, 40mm/min, 50mm/min. Blank size and punch diameter were 74mm and 37mm, respectively, and thus overall LDR(limit drawing ratio) was 2.0. Processing maps for both sheets during deep drawing at elevated temperatures were compared. Initial textures were typical basal fibers with symmetric arrangement. Sheets from ingot casting had larger grain size than those from twin roll casting. The basal fibers were evolved into other orientations during deep drawing, which contained both compression along the circumferential direction in the flange and tension along the drawing direction in the cup wall. Most evident reorientations were found in the flange, as expected. With deformation, finer grains increased. Necking and cup-failure were usually expected in the lower wall near the bottom

#### 6:00 PM

Comparison of Mechanical Properties of Pure Mg Materials Obtained by Different Back Pressure Equal Channel Angular Processing Conditions: Jizhong Li<sup>1</sup>; *Xiaolin Wu*<sup>1</sup>; Wei Xu<sup>1</sup>; Kenong Xia<sup>1</sup>; <sup>1</sup>Department of Mechanical Engineering and ARC Centre of Excellence for Design in Light Metals, University of Melbourne

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Pure Mg shows very low yield strength and limited ductility at room temperature. It has been shown that equal channel angular pressing (ECAP) may be used successfully for grain refinement in pure Mg and Mg alloys at elevated temperatures higher than 200°C, leading to improvements in the strength and ductility of these materials. However, ECAP at a temperature lower than 200°C has turned out to be difficult, especially for pure Mg. Experiments, therefore, were conducted to improve mechanical properties of pure Mg through grain refinement using back pressure equal channel angular pressing (BP-ECAP) at as low as room temperature. Effects of processing temperature, back pressure and number of passes were studied. It is exhibited that the yield strength increases and work hardening decreases with lowering processing temperature. Ductility improves as the back pressure and/or the number of passes increases.

## Symposium D: Bulk Metallic Glasses and Nanomaterials: BMGs - Properties and Processing II

Tuesday PMRoom: 4August 3, 2010Location: Cairns Convention Centre

Session Chair: Akira Takeuchi, Tohoku University

#### 2:00 PM Keynote

The Influence of High-Density Pulsing Current on the Property and Nanocrystallization Behaviour of Glassy Alloys: *Ke-Fu Yao*<sup>1</sup>; Sheng-Bao Qiu<sup>1</sup>; <sup>1</sup>Tsinghua University

It is known that high-density pulsing current can induce metallic alloys exhibit electroplasticity. Here, high-density pulsing current has been employed to act on metallic glasses and its effects on the properties and nanocrystallization behaviours have been studied in details. It has been found that the mechanical property of bulk metallic glasses could be improved by treating these glassy alloys with high-density pulsing current under suitable condition. Such as the hardness and compressive plasticity of the glassy alloys can be significantly enhanced. In addition, nanocrystallization of glassy alloys can be promoted by treating with high-density pulsing current if the technique parameters employed are suitable. Such as for an Fe-based glassy alloy, uniformly precipitated nanocrystalline particles, with about 8 nm in size, within the glassy alloy can be obtained by treating with high-density pulsing current for less than 30 seconds. It is believed that an extra force acted on the atoms by the pulsing electron drift has played an important role in improving the mechanical properties and in nanocrystallization of glassy alloys. The present results indicate that high-density pulsing current treatment is an effective way for modifying the microstructures and the properties of glassy alloys.



#### 2:20 PM Keynote

Wear Behaviors of Cast-Iron-Based Bulk Metallic Glass Coating Layers Formed by a HVOF Process: B. T. Jang<sup>1</sup>; S. S. Kim<sup>1</sup>; S. Yi<sup>1</sup>; <sup>1</sup>Kyungpook National University

For practical applications of bulk metallic glasses, bulk metallic glasses should be produced through a commercial material process using cheap industrial raw materials that may deteriorate glass forming ability. Cast iron based bulk metallic glasses that have high glass forming ability and can be cost-effectively produced in large quantities have been developed for extensive structural and functional applications. The cast-iron based bulk metallic glasses can be prepared as fully amorphous powders through a commercial atomization process under N<sub>a</sub> atmosphere. Moreover, using the cast iron based bulk metallic glass powders, fully amorphous coating layers can be formed through a commercial high velocity oxy-fuel thermal processing method. Compared with conventional bearing steels such as AISI 521000 (Hv=840), excellent tribological and wear characteristics of the amorphous coating layers have been obtained demonstrating the cast iron base bulk metallic glass powders as viable engineering materials for practical antiwear coating applications. In this presentation, wear behaviors of the amorphous coating layers during the friction test in an alumina ball-on-disc system will be discussed based upon microstructural and thermal analysis results.

#### 2.40 PM

#### Brazing of Titanium Using a Zr-Based Amorphous Alloy Filler: Jin Kyu Lee<sup>1</sup>; <sup>1</sup>Kongiu National University

In the case of brazing of Ti and Ti alloys, low melting point of the brazing filler metal is very important. Because brazing at the high temperature results in a substantial grain coarsening, loss of strength of the base metal and corrosion at the interface. Bulk metallic glasses (BMGs) with a low critical cooling rate usually have a low melting temperature due to its deep eutectic of multicomponent composition. Since the  $Zr_{41,2}Ti_{13,8}Ni_{10,0}Cu_{12,5}Be_{22,5}$  alloy has a quite low melting temperature of 725°C as compared with general Ti-base filler metals, it is considered to braze Ti. In this study, we report the microstructure and mechanical properties of the jointed Ti by brazing of Zr-based amorphous filler metal. The Zrbased filler sheets were prepared by single roll casting. Rapidly solidified sheets were prepared by remelting alloy in crucible and ejecting through a nozzle onto a rotating Cu roll. The microstructure of the brazed joint was investigated by scanning electron microscopy (SEM) with an energy dispersive X-ray analysis (EDX). Tensile tests were conducted at room temperature and at a strain rate of 1x10<sup>-4</sup> s<sup>-1</sup>. The surface of the fractured specimen was observed by SEM.

#### 2:55 PM

Consolidation Behavior of Cu-Zr-Al Metallic Glass Powder by Spark Plasma Sintering: Guoqiang Xie1; Dmitri V. Louzguine-Luzgin1; Mikio Fukuhara1; Hisamichi Kimura1; Akihisa Inoue1; 1Tohoku University

Bulk metallic glasses (BMGs) with excellent physical and chemical properties have attracted increasing attention. However, the disadvantages such as brittleness, low electrical conductivity and limited dimensions hamper their applications. To improve ductility and conductivity of BMGs, it should be a viable approach by developing BMG composites containing high-conductive crystalline phases. To overcome the limitation of dimensions, powder metallurgy process should be a good alternative. Spark plasma sintering (SPS), as a newly developed rapid sintering technique, has a great potential for producing dense glassy specimens. In this study, using gas-atomized glassy alloy powders (Ni52 N  $b_{10}Zr_{15}Ti_{15}Pt_{7.5}$ ,  $Cu_{50}Zr_{45}Al_5$ ,  $Fe_{73}Si_7B_{17}Nb_3$ , etc.) blended with metal (Cu, W, etc.) or ceramic (SiC, Al<sub>2</sub>O<sub>3</sub>, etc.) particulates, we fabricated bulk metallic glassy alloy composites (GACs) with a diameter of 20 mm by the SPS process. The sintered alloy specimens with a relative density of above 98% were obtained at a sintering temperature near Tg with a loading pressure of 600 MPa. The fabricated GACs exhibited simultaneously high strength and enhanced plasticity, also dramatically improved electrical conductivity by adding high-conductive Cu particulates. No crystallization of glassy matrix and good bonding state between the particles are responsible for good mechanical and electrical properties of the fabricated GACs.

#### 3:10 PM

Dynamic Relaxation and Its Correlation to Quasi-Static Mechanical Properties in Bulk Metallic Glass: Hidemi Kato1; Tetsu Ichitsubo2; Akihisa Inoue1; 1Tohoku University; 2Kyoto University

Dynamic relaxations in the vicinity of the glass transition temperature (Tg) were investigated in the wave frequency range from 10<sup>-2</sup> to 10<sup>2</sup> rad/s in ductile and brittle bulk metallic glasses (BMGs). The relaxation-time distribution consists of two relaxations, i.e. the glass transition ( $\alpha$ -relaxation) and the sub-Tg relaxation ( $\beta$ relaxation or so called "wing"), and was fitted by superimposition of the stretched exponent functions based on the Maxwell element model. Thermal activation for the  $\alpha$ - and  $\beta$ -relaxations is considered to be of the Vogel-Fulcher-Tammann (VFT) with the VFT temperature T0≥Tg -250 K and of the Arrhenius type with the activation energy Q~ 1-2 eV, respectively. Thus at room temperature, the relaxation of some BMGs is considered to occur in the corresponding time scale of the usual strain rate,  $= 10^{-4} \sim 10^{-2} \text{ s}^{-1}$  for the quasi-static mechanical tests, and to act as trigger for the yielding phenomenon. In this study, we intend to explain the quasi-static compressive deformation behavior, i.e. elastic modulus, yielding (creating the Shear Transformation Zone) and plastic deformation before the final fracture associated with the glass transition by the observed characteristics of the dynamic relaxation-time distribution in the BMGs.

#### 3:25 PM

Effect of Nitridation on Magnetic Properties of Nanocrystalline Fe-Co Alloy Powders: Ya Qiong He<sup>1</sup>; Changhui Mao<sup>1</sup>; Jian Yang<sup>1</sup>; <sup>1</sup>General Research Institutes for Nonferrous Metals

Nanocrystalline Fe-Co alloy powders, which were prepared by high-energy mechanical milling, were nitrided under the mixing gas of NH<sub>3</sub>/H, in the temperature range from 380°C to 510°C. X-ray diffraction(XRD) was used to analyze the grain size and reaction during the processing. The magnetic properties of the nitrided powders were measured by Vibrating Sample Magnetometer (VSM). The results show that with the appearance of  $Fe_4N$ phase after nitride treatment, and the grain-size of FeCo phase decreases with the increase of nitridation temperature between 380°C to 450°C, which may due to the thickness reduction of FeCo grain surface by nitridation reaction. The saturation magnetization of nitrided alloy powder treated at 480°C is about 18% higher than that of the initial Fe-Co alloy powder, accompanied by the reduction of the coercivity. Transmission electron microscope (TEM) was used, attempting to further analyze the effect of Fe<sub>4</sub>N phase on microstructure and magnetic properties of the powder mixtures.

3:40 PM Tea Break

### **Symposium D: Bulk Metallic Glasses and Nanomaterials: BMGs - Properties and Processing III**

Tuesday PM August 3, 2010	Room: 4	Cairns Convention Centre
August 3, 2010	Location.	Califis Convention Centre

Session Chair: Michael Ferry, University of New South Wales

#### 4:30 PM

Characteristics of Ti-Ni-Zr Thin Film Metallic Glasses Exhibiting a Shape Memory Effect after Crystallization: Junpei Sakurai<sup>1</sup>; Yuko Aono<sup>1</sup>; Yui Ishida<sup>1</sup>; Seiichi Hata1; 1Tokyo Institute of Technology

Thin film metallic glasses (TFMGs) are expected to be suitable materials for three dimensional (3D) microelectromechnical systems (MEMS), because they exhibit viscous flow and were deformed into the 3D structure in the super cooled liquid region. Moreover, if these TFMGs are crystallized to the functional materials, we can fabricate the unique 3D MEMS such as microsensor and microactuator. In this study, in order to search for novel Ti-Ni-Zr TFMGs that exhibit the stable shape memory effect after crystallization, we investigated the various characteristics of the Ti-Ni-Zr amorphous thin film before and after annealing. As these results, we found a novel  $Ti_{42}Ni_{47}Zr_{11}$  TFMG. This sample exhibited the glass transition and glass transition temperature and crystallization temperature were 703 K and 760 K, respectively. Moreover, this sample annealed at 973 K for 3.6ks showed the martensitic phase transformation of B2 to B19'. Martensitic phase start temperature and reverse martensitic phase start temperature were 296 K and 352 K, respectively. From the results of tensile tests, this sample showed the recovery strain of 2.4%.

#### 4:45 PM

Enhancement of Glass-Forming Ability of CoFeBSiNb Bulk Glassy Alloys with Excellent Soft-Magnetic Properties and Superhigh Strength: Baolong Shen1; 1Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences

Co-based bulk glassy alloys with diameters up to 4.5 mm were formed in Co46Fe20B22+xSi6xNb6 (x=0-2) system. The increase of B to Si concentration ratio is effective in improving the glass-forming ability. In addition to high glassforming ability, the glassy alloys exhibit excellent soft-magnetic properties, i.e., high saturation magnetization of 0.63–0.69 T, low coercive force of 1.17–2.35A/ m, and high effective permeability of  $1.36-2.65 \times 10^4$  at 1 kHz under a field of 1 A/ m. The bulk glassy alloys also exhibit superhigh fracture strength of 4400 MPa.



### 5:00 PM

Fabrication and Characteristics of Fe-B-Nb-RE (Rare Earth) Bulk Metallic Glasses Having Large Glass Forming Ability and High Viscous Workability: Sangmin Lee<sup>1</sup>; Hidemi Kato<sup>1</sup>; Akihiro Makino<sup>1</sup>; Akihisa Inoue<sup>1</sup>; <sup>1</sup>Tohoku University

The effects of rare earth (RE) addition on the glass forming ability (GFA) and thermal stability of a Fe-B-Nb marginal glass former was investigated and the origin of the highly improved GFA in the multicomponent system was discussed with related to a characteristic exothermic phase transformation, chemical short range ordering (CSRO), in the supercooled liquid region due to the positive mixing enthalpy between Nb and RE elements. The separating tendency between Nb and RE elements is considered to suppress precipitation of metastable Fe<sub>23</sub>B<sub>6</sub> and bcc-Fe crystalline phases, thus to result in highly improving GFA and distinct high thermal stability against heat treatment of the alloy system. The RE addition effect on  $(Fe_{0.72}B_{0.04}Nb_{0.04})_{100-x}RE_x$  was investigated with a viewpoint of GFA and thermal stability by evaluating thermal properties, activation energy for crystallization and by observing magnetic short range ordering (MSRO). In addition, ferrous metallic glass thin film (FMGTF) of Fe<sub>71</sub>B<sub>19</sub>Nb<sub>34</sub>Nd<sub>63</sub> was prepared by electron cyclotron resonance sputtering to investigate imprintability using viscous workability and magnetic transition from soft to semi-hard magnetism for functionality. Micro- and nano-scale surgical instruments capable of cutting, gripping, drilling, etc., can be realized through the synergy effect of viscous workability and functionality developed in the present paper.

#### 5:15 PM

#### Fundamental Issues of the High Entropy Alloys and Bulk Metallic Glasses: Yong Zhang<sup>1</sup>, <sup>1</sup>University of Science and Technology Beijing

Bulk metallic glasses (BMGs) and high entropy alloys (HEAs) have unique structures at the length scale of micro- and nano-meters, and exhibit unique properties, which make them the potential materials for structural as well as functional applications. It was found that the phase changes of HEAs closely related to the atomic packing efficiency (APE) of the alloys, the phase structures with higher APE will tend to change to lower one when the Al content increases. The HEA of CoCrCuFeNi is typical FCC, it will be the BCC structure after the Cu was substituted by Al. The mechanical properties of the AlCoCrFeNiTi<sub>0.5</sub> alloy, which has a BCC structure, exhibits ultrahigh fracture strength over 3 GPa and 20% plastics strain limit, these properties are competeive to most of the BMGs. The HEAs can retain their high strength over to the high tempearture. The tensile ductility of the dendrite/BMG composites can be greatly improved by the Bridgman solidification (at least 7% tensile elongation has been achieved), the growth of the dendrite solid solution phase can be controlled by keeping the alloy at the semisolid state, and changing the withdraw velocities.

#### 5:30 PM

#### High Glass-Forming Ability and Unusual Deformation Behavior of New Zr-Cu-Fe-Al Bulk Metallic Glasses: *Q. S. Zhang*<sup>1</sup>; W Zhang<sup>1</sup>; D. V. Louzguine-Luzgin<sup>1</sup>; A Inoue<sup>1</sup>; <sup>1</sup>Tohoku University

New series of bulk metallic glasses were developed by addition of Fe into the ternary  $Zr_{60}Cu_{30}Al_{10}$  alloy. Although Fe-Cu element pair shows distinct immiscibility with large positive heat of mixing, substitution of Fe for Cu significantly improves the glass-forming ability of the ternary  $Zr_{60}Cu_{30}Al_{10}$  alloy. The critical diameter for glass-formation increases from 8 mm for  $Zr_{60}Cu_{30}Al_{10}$ alloy to 20 mm for  $Zr_{60}Cu_{25}Fe_5Al_{10}$  and  $Zr_{62.5}Cu_{22.5}Fe_5Al_{10}$  alloys. As compared with the ternary  $Zr_{60}Cu_{30}Al_{10}$  alloy, the new quaternary Zr-Cu-Fe-Al alloys show lower liquidus temperatures. The  $Zr_{60}Cu_{25}Fe_5Al_{10}$  and  $Zr_{62.5}Cu_{22.5}Fe_5Al_{10}$  alloys, the best BMG-formers in this alloy system, are found to locate very near a quaternary Zr-Cu-Fe-Al eutectic point. The new Zr-Fe-Cu-Al bulk metallic glasses exhibit high strength of about 1700 MPa. The plastic strain increases from 7.8% to 11.3% with increasing the content of Fe from 0 to 12.5%. The finding of the Ni-free Zrbased bulk glassy alloy with the extremely high glass-forming ability is expected to extend the future application of bulk metallic glasses.

#### 5:45 PM

Viscous Flow Behaviour of Supercooled Liquids and Mechanical Properties in Zr-Cu-Ni-Al Bulk Metallic Glasses: *Tohru Yamasaki*<sup>1</sup>; Masahiro Yamada<sup>1</sup>; Tsuyoshi Mori<sup>1</sup>; Takeyuki Kikuchi<sup>1</sup>; Yoshihiko Yokoyama<sup>2</sup>; Akihisa Inoue<sup>2</sup>; Do Hyang Kim<sup>3</sup>; <sup>1</sup>University of Hyogo; <sup>2</sup>Tohoku University; <sup>3</sup>Yonsei University

Viscous flow behaviours of supercooled liquids and mechanical properties in  $Zr_{55-65}Cu_{10.30}Ni_{5-15}Al_{10}$  bulk metallic glasses (BMGs) with eutectic and hypoeutectic (hypo) compositions have been examined. Viscosity has been measured by using a penetration viscometer at a high-speed heating rate of 400 K/min<sup>(1)</sup>. With increasing the Zr-content,  $T_g$  tended to decrease and  $T_x$  tended to increase, resulting the  $\Delta T_x$  (= $T_x$ - $T_g$ ) increased up to about 170 K for hypo-Zr<sub>65</sub>Cu<sub>18</sub>Ni<sub>7</sub>Al<sub>10</sub> BMG under a heating rate of 400 K/min by the DSC analysis and the viscosity lowered about one order of magnitude, e.g., minimum value of the viscosity decreased from 5x10<sup>6</sup> Pa-s for the eutectic-Zr<sub>55</sub>Cu<sub>30</sub>Ni<sub>5</sub>Al<sub>10</sub> BMG down to  $5x10^{5}$  Pa-s for the hypo- $Zr_{65}Cu_{18}Ni_{7}AI_{10}$  BMG. Compression test was carried out by using an Instron-type machine with an initial strain rate of  $1\times10^{-4}/s$  at room temperature. With increasing the Zr-content, the apparent yield stress was decreased but then the plastic strain at fracture tended to increase, e.g., 1705MPa-1.9%, 1661MPa-7.1% and 1498MPa-6.4% for those of the eutectic- $Zr_{55}Cu_{30}Ni_5AI_{10}$  the hypo- $Zr_{60}Cu_{25}Ni_5AI_{10}$  and the hypo- $Zr_{65}Cu_{18}Ni_7AI_{10}$  BMGs, respectively. Above these results have suggested that there is a strong relationship between the viscosity and the mechanical properties in these BMGs.

#### 6:00 PM

#### Phase Formation during Cooling the Melt of In-Situ Mg-Based BMG Composite: *Wanqiang Xu*<sup>1</sup>; Michael Ferry<sup>1</sup>; Rongkun Zheng<sup>1</sup>; Lalu Robin<sup>1</sup>; <sup>1</sup>University of New South Wales

The presence of crystalline phases in bulk metallic glass (BMG) is known to improve ductility by combining the high strength of monolithic BMG with the high ductility and fracture toughness of the crystalline phase(s). In-situ bulk metallic glass (BMG) composite consists of crystalline phase(s) transformed during cooling from the melt dispersed within an amorphous matrix. The phase transformations that occur during casting are important as they control the volume fraction, morphology and size of the dispersed phases, thereby influencing the properties of the BMG composites. Although a number of insitu BMG composites have been developed in recent years, the nucleation and grain growth of crystalline phase from supercooled melt during casting are not well understood. In this project, we investigate the nucleation and growth of crystalline phases in Mg-based BMG composites using 3D-FIB and TEM. Particles were found to precipitate from the melt during casting with the Mgrich plates nucleating at the surface of these particles and radiating out into the cooling liquid to generate a network microstructure. Due to the fast cooling during casting, there is composition segregation in different flakes and different part of a flake.

#### 6:15 PM

#### Preparation and Characterization of Nano-Imprinted Fe-Ga-P-C-B-Si-Cr Metallic Glass: *Kenji Amiya*<sup>1</sup>; Yasunori Saotome<sup>1</sup>; <sup>1</sup>Tohoku University

Fe-based metallic glass (MG) is useful for developing magnetic devices, so many researches on the MG have been done. However, there have been a few reports on imprinted Fe-based MG because imprinting of Fe-based MG with high glass-transition temperature (Tg) is difficult, compared with polymers or Pt-based MG. The Fe-Ga-P-C-B-Si-Cr MG with soft-magnetic properties was imprinted by Newtonian viscous flow in the super cooled liquid temperature region. Good replication ability of the Fe-Ga-P-C-B-Si-Cr MG enabled nano-imprinting of the diffraction gratings with periods of 1 µm and the dots-patterns with a dot diameter of 200 nm. The convex nano-dot structure with aspect ratio over 1 was fabricated by nano-imprint on the surface of MG ribbon with a width of 1.5 mm and a length of 3 mm. The magnetization curve in vertical magnetic field of the imprinted ribbon with smooth surface is different from that with dots-surface. The explanation of this difference lies in the presence of magnetic anisotropy due to the imprinted micro/nano-dot structure. The obtained result are summarized that imprinting process for Fe-based MG is useful for the production process of micro-structural magnetic devices with good soft-magnetic properties.

### Symposium E: Solidification, Deformation and Related Processing: Deformation Processing and Mechanical Properties II

Tuesday PM	R
August 3, 2010	Lo

Room: 2 Location: Cairns Convention Centre

Session Chair: Zenji Horita, Kyushu University

#### 2:00 PM

#### Micro Forging of CP-Ti Powder Compacts Produced by ECAP with Back-Pressure: *Rimma Lapovok*<sup>1</sup>; Colleen Bettles<sup>1</sup>; <sup>1</sup>Monash University

Our previous research has shown that use of Equal Channel Angular Pressing (ECAP) with applied back pressure for compaction of powder allows significantly decrease the temperatures of consolidation compare to those used in conventional practice. The compacts from CP-Ti powder of 99.9% theoretical density and strength of 480 MPa were achieved at ECAP temperature of 300°C. The production of parts by micro-forging cannot be simply scaled down from macro processing using similarity principles and dimensional analysis. Scaling down the dimensions of a part requires decreasing the grain size proportionally and results in a change of the material behaviour at the micro-level. ECAP is well known process to reduce the grain size of bulk material from microns to nanometres range. Therefore, the twofold gain in enhanced low temperature



compaction and simultaneous grain refinement of CP-Ti justified the utilisation of ECAP with back-pressure for production of compacts used as a forging stock for micro con-rods. The micro-forging of con-rods from CP-Ti powder compacts have been studied to optimise the technological parameters of the process. The microstructure and mechanical properties of the forged parts have been investigated and compared with those produced from the bulk rod.

#### 2:15 PM

Mechanical Properties of Aluminum Alloy Al-6061 Obtained by the Liquid Forging Process: *Chun Wei Su*<sup>1</sup>; Peng Hooi Oon<sup>1</sup>; Yong Hui Bai<sup>1</sup>; Anders Jarfors<sup>1</sup>; <sup>1</sup>Singapore Institute of Manufacturing Technology

The liquid forging process has the flexibilities of casting in forming intricate profiles and features while imparting the liquid forged components with superior mechanical strength compared to similar components obtained via casting. Additionally, liquid forging requires significantly lower machine loads compared to solid forming processes. Currently, components that are formed by liquid forging are usually casting alloys of aluminum. This paper investigates the suitability of liquid forging a wrought aluminum alloy Al-6061 and the mechanical properties after forming. The proper handling of the Al-6061 alloy in its molten state is important in minimizing oxidation of its alloying elements. By maintaining the correct alloying composition of Al-6061 after liquid forging, these Al-6061 samples can subsequently undergo a suitable heat treatment process to significantly improve their yield strengths. Results show that the yield strengths of these liquid forged Al-6061 samples can be increased from about 90MPa, when they are in the as-liquid forged state, to about 275MPa after heat treatment. This improved yield strength is comparable to that of Al-6061 samples obtained by solid forming processes. As such, the liquid forging process here has been shown to be capable of forming wrought aluminum alloy components that has the potential for structural applications.

### 2:30 PM

# **Steel-Magnesium Composite Wire Obtained by Repeated Co-Extrusion**: *Olivier Bouaziz*<sup>1</sup>; Xavier Sauvage<sup>2</sup>; <sup>1</sup>ArcelorMittal Research; <sup>2</sup>Groupe de Physique des Matériaux

In order to combine strength, stiffness and a lightweight materials papers reported investigations of composite where steel is combined with a light metal as aluminium or titanium. Surprisingly no study is available from our knowledge related to a steel based composite trough a combination with magnesium despite its very low density and its non-miscibility with iron. The last point is relevant for avoiding the formation of brittle intermetallic phase at the interface between steel and the other metal appearing during thermal or thermomechanical treatments. So this paper presents first results related to the fabrication of a composite wire by the combination of steel and magnesium. This composite have been obtained by repeated co-extrusion inducing a microstructure with different length scales. The microstructure and the mechanical properties have been characterized showing that the composite exhibits ductility controlled by annealing treatment and by decohesion of the interfaces. The strength of the interfaces seems to be controlled by the cumulative plastic strain induced by the extrusion. Finally this study suggests an opportunity for the development of original lightweight multimetallic composites with strength and ductility.

#### 2:45 PM

Effect of Prior Cold-Working on Strength and Electrical Conductivity of Cu-Ti Dilute Alloy Aged in a Hydrogen Atmosphere: Satoshi Semboshi<sup>1</sup>; Hiroshi Numakura<sup>2</sup>; Wei-Lin Gao<sup>3</sup>; Hisashi Suda<sup>3</sup>; Akira Sugawara<sup>3</sup>, <sup>1</sup>Tohoku University; <sup>2</sup>Osaka Prefecture University; <sup>3</sup>DOWA Metaltech Co.,Ltd

It has been reported that aging at 673 K in a hydrogen atmosphere rendered a good combination of the strength and electrical conductivity for Cu-Ti dilute alloys, in comparison to conventional aging in a vacuum (S. Semboshi, et al., J. JRI Cu, 2009). In this study, influences of prior deformation on mechanical and electrical properties of Cu-4.2 at.% Ti alloys aged in a hydrogen atmosphere was examined. The Vickers hardness increased from 127 in a solution-treated state to 185 by cold-rolling in a reduction rate of 0.15. The hardness of the solution-treated specimen increased to 265 h by aging at 673 K for 360 h in a hydrogen atmosphere of 0.6 MPa, while that of the deformed specimen attained to a maximum of 280 by aging for 240 h in the same atmosphere. With the prior deformation, The conductivity at the peak-hardness in the deformed specimen was 27% International Annealed Copper Standard (IACS), which exceeded that in the solution-treated specimen of 20% IACS. Thus, prior deformation assisted an improvement of the properties during aging in a hydrogen atmosphere.

#### 3:00 PM

Shear Tests on Small Size Structures: Suhash Dey<sup>1</sup>; Janine Pfetzing-Micklich<sup>1</sup>; Steffen Brinckmann<sup>1</sup>; Gunther Eggeler<sup>1</sup>; Alexander Hartmaier<sup>1</sup>; <sup>1</sup>Ruhr University

To study the mechanical integrity of small-sized materials downscaling of the existing experimental techniques are required and simultaneously the new methods are also needed to be developed. At small scales, material properties are affected by high surface to volume ratio. So the aim of the present work is to develop material testing methods for small scale systems (micron-sized) and to perform shear experiments on them and to study the deformation mechanisms which ultimately affects the material properties.

#### 3:15 PM

Effect of Si Content on Fracture Behaviour Change by Strain Rate in Si Steels: *Takashi Mizuguchi*<sup>1</sup>; Ryota Oouchi<sup>1</sup>; Rintaro Ueji<sup>1</sup>; Yasuhiro Tanaka<sup>1</sup>; Kazunari Shinagawa<sup>1</sup>; <sup>1</sup>Kagawa University

The fracture behaviour transition due to change of strain rate in the steels with various Si content ranging from 2mass% to 5% were studied. The room-temperature tensile tests were conducted at wide range of strain rate ranging from  $10^{-3}s^{-1}$  to  $10^{3}s^{-1}$ . Concerning of low Si steels (no more than 3%), the nominal stress - nominal strain curves represented both uniform and local elongations at all strain rate. On the other hand, in 4%Si steels at a strain rate higher than  $10^{1}s^{-1}$ , the tensile sample broke down without local elongation (necking). The stress at breaking was nearly equal to its work hardening rate. The strain rate at which fracture behaviour transition took place in 5%Si steel ( $10^{-1}s^{-1}$ ) was lower than that in 4%Si steel. TEM observations clarified the existence of deformation twins in the sample fractured without necking. The mechanism of the change of fracture behaviour is discussed focusing on the dislocation morphologies and the formation of deformation twins.

#### 3:30 PM

Structure and Mechanical Properties of Asymmetrically Rolled IF Steel Sheet: *Dmitry Orlov*<sup>1</sup>; Rimma Lapovok<sup>1</sup>; Laszlo Tóth<sup>2</sup>; Ilana Timokhina<sup>3</sup>; Peter Hodgson<sup>3</sup>; Debashish Bhattacharjee<sup>4</sup>; Arunansu Haldar<sup>4</sup>; <sup>1</sup>Monash University; <sup>2</sup>Universite Paul Verlaine – Metz; <sup>3</sup>Deakin University; <sup>4</sup>Tata Steel Ltd.

In the modern automobile market, interstitial-free (IF) steel is in great demand for body panels. At the same time, requirements for strength and deep drawability of IF steels are becoming more stringent due to increasingly complex shapes of car bodies. New cost-effective methods are therefore required to produce IF steel sheet with improved properties. A relatively recent and most powerful method to improve these characteristics is severe plastic deformation (SPD). In SPD, large shear strains are introduced to refine material microstructures down to ultrafineor near nano-scale. However, SPD techniques remain complicated and difficult to implement in industrial processes. An alternative way to modify microstructure and properties in sheets using SPD principles is Asymmetrical Rolling, and the simplest way to introduce asymmetry into the rolling process is to make one roll idle. As-received hot-rolled 5.6mm thick IF steel sheet was symmetrically or asymmetrically cold rolled down to 1.9mm. The asymmetrical rolling was done in monotonic and reversal modes. Optical microscopy, EBSD analysis, TEM, X-Ray texture and tensile tests were used to analyse the microstructure and mechanical property evolution. The effects of rolling conditions on the texture and strength property are discussed based on the experimental results and FEM simulation.

#### 3:45 PM Tea Break

# Symposium E: Solidification, Deformation and Related Processing: Deformation Processing and Mechanical Properties III

Tuesday PM	Room: 2	
August 3, 2010	Location:	Cairns Convention Centre

Session Chair: Kyung-Tae Park, Hanbat National University

#### 4:30 PM

Precipitates Behavior during Thermomechanical Processing of Low Mn, Ti Added Pipeline Steels: *Ali Dehghan-Manshadi*<sup>1</sup>; Rian Dippennar<sup>1</sup>; <sup>1</sup>University of Wollongong

The behavior of manganese and titanium sulfides during hot deformation of a low carbon, low manganese, titanium added steel have been studied using transmission electron microscopy (TEM), scanning electron 'microscopy (SEM) and energy dispersive spectrometry (EDS) analysis. In addition, the effect of deformation temperature and strain rate on size and distribution of precipitates



have been studied using an automatic inclusion analyses system. Then, the effect of precipitate distribution on mechanical properties was studied at different deformation conditions of temperature and strain rate. The TEM and SEM analyses revealed the presence of a wide variety of simple and/or complex precipitates in the as-cast structure. These precipitates behaved differently during hot deformation the steel. While, less elongation of precipitates were observed at higher deformation temperatures, increasing the strain rate showed direct effect on increasing their elongation.

#### 4:45 PM

# Numerical Simulation for Effects of Friction on Deformation Behaviors in a 3-Dimensional Hot Upsetting Process: Y.C. Lin<sup>1</sup>; <sup>1</sup>Central South University

In metal forming processes, friction plays a significant role in studying the formability of the work material and the quality of the finished product such as, surface finish, internal structure, and product life. Friction can increase the inhomogeneity of deformation, leading to defects in the finished products. Friction can also be used beneficially to manipulate the material flow to achieve the desired end product with a minimum effort. In this study, the compressive deformation behavior of 42CrMo steel was investigated on Gleeble-1500 thermosimulation machine. Based on experimental results, the dynamic recrystallization mathematica1 models of 42CrMo steel were derived. Then, the thermo-mechanica1 coupled finite element model was developed to investigate the effects of friction on dynamic recrystallization in a deformed 42CrMo steel during hot upsetting were investigated by integrating the thermo-mechanical coupled finite element method with the derived microstructural evolution models. The results show that the distributions of strain/stress in the deformed block are inhomogeneous, and the degree of the deformation inhomogeneity changes with the frictions between work-piece and dies. The distribution of dynamic recrystallization volume fraction and dynamic recrystallization grain size, which change with the frictions, are also inhomogeneous in the deformed work-piece.

#### 5:00 PM

# The Effect of Oxygen on the Brittle-to-Ductile Transition in Silicon Single Crystals: Youn-Jeong Hong<sup>1</sup>; Masaki Tanaka<sup>1</sup>; Kenji Higashida<sup>1</sup>; <sup>1</sup>Kyushu University

Oxygen is one of the inevitable impurities in Czochralski (CZ) grown silicon single crystals due to the usage of a silica mould at crystal growth. Oxygen dissolved in silicon as an interstitial atom strongly interacts with dislocations, which suggests that the brittle-to-ductile transition (BDT) behaviour is influenced by the solute oxygen since the BDT behaviour is controlled by dislocation activities around a cack tip. In this study, therefore, the effect of solute oxygen on the BDT in silicon single crystals was investigated. CZ silicon wafers (high solute oxygen concentration) and floating-zone (FZ) grown silicon wafers (low solute oxygen concentration) were employed. The temperature dependences of apparent fracture toughness of those crystals were measured, and it was found that the BDT temperature in CZ silicon was higher than that in FZ silicon, suggesting that solute oxygen decreases dislocation mobility. However, the activation energies obtained from the strain rate dependence of the BDT temperatures were nearly the same in both the CZ and FZ silicon crystals. The origin of the difference in the BDT temperatures is discussed.

#### 5:15 PM

#### The Microstructure and Mechanical Properties of a Friction Stir Processed Al-Zn-Mg-Cu Alloy: *Margarita Vargas*<sup>1</sup>; Sri Lathabai<sup>1</sup>; <sup>1</sup>Commowealth Scientific and Industrial Research Organisation (CSIRO)

Friction stir processing (FSP) is a novel solid-state technique which combines frictional heating and severe plastic deformation to produce ultra-fine grained metallic materials. During FSP, a rotating tool with a specially designed pin and shoulder is plunged into the surface of the work piece and traversed along a path to cover the region of interest. FSP was performed on AA 7075-T6, an age hardenable high strength Al-Zn-Mg-Cu alloy commonly used in the aerospace industry. Diverse combinations of the two main processing parameters, i.e., the rotational and the translational speeds of the tool, were studied systematically in order to determine the optimal conditions for microstructural modification. As a result, several ultra-fine grained microstructures were produced, which were characterised using optical and scanning electron microscopy. The mechanical properties such as hardness, strength and ductility of the processed zone were studied and correlated with the microstructure. Significant differences in the mechanical behaviour of the FSP zone as compared to that of the as-received material were observed.

#### 5:30 PM

#### **The Effect of Si/Mn on Surface Oxidation Rate and Scale Formation in Cold Rolling Process**: *Jingyun Park*<sup>1</sup>; Dong Joon Min<sup>1</sup>; <sup>1</sup>Yonsei University

Recently, the additive content of alloying elements such as Si and Mn are increased because of demand for increase of mechanical property and reducing weight of steel. However it is well known that these kind of alloying elements make oxides, SiO<sub>2</sub>, MnO and MnSiOx, on the surface of steel. These kinds of oxides reduce the wettability between steel and molten zinc and cause the surface defect. Thus, in this study the effect of each alloying elements on surface oxidation and scale formation were experimented by the thermal gravity method. Fe-0.3~2.3wt.%Mn and Fe-0.5~2.7wt.%Si alloy were oxidized at 973~1073K in low oxygen potential. After experiment, each sample's cross sections were polished and the morphology of scale was analyzed by SEM and EPMA. It is confirmed that the oxidation rate was decreased as increase of Si and Mn content by oxidation of Fe At high oxygen potential, while at low oxygen potential, the oxidation rate was increased as increase of Si and Mn content because of selective oxidation rate of Si and Mn. And Si rich layer was formed between external and internal scale on Fe-Si alloy, on the other hand dense external scale and internal inclusion was observed on Fe-Mn alloy.

#### 5:45 PM

#### Ni Based Metal Membranes Produced by Planar Flow Casting for Hydrogen Separation: *Daniel Vieira*<sup>1</sup>; Michael Kellam<sup>1</sup>; GuangSheng Song<sup>1</sup>; Michael Dolan<sup>2</sup>; <sup>1</sup>CSIRO Process Science and Engineering; <sup>2</sup>CSIRO Energy Technology

Low-cost and high-temperature metal membranes for hydrogen separation are critical for developing large-scale production of hydrogen from carbon-based fuels, such as coal and natural gas, with water-gas-shift catalysts. A series of high melting temperature Ni based alloys have been examined for fabricating metal membranes by planar flow casting. The production of 30mm width membranes from these high melting temperature alloys with required microstructures and surface conditions faces a number of processing difficulties, including oxidation, contamination by the atmosphere and containing materials, chaotic fluid flow, non-even heat transfer, etc, which are influenced by multiple process variables. By optimizing of the operation parameters according to the understanding of fluid flow dynamics, heat transfer and solidification that are specific to these Ni based alloys, quality Ni based alloy membranes have been obtained, which have demonstrated satisfactory permeability at 300-400°C in tests with single-gas and simulated coal-derived synthesis gas.

#### 6:00 PM

#### Synthesis of Giant Magnetostrictive Iron-Rich Sm-Fe Alloy by Unidirectional Solidification in Microgravity: *Takeshi Okutani*<sup>1</sup>; Hiromichi Ono<sup>1</sup>; Hideaki Nagai<sup>2</sup>; <sup>1</sup>Yokohama National University; <sup>2</sup>National Institute of Advanced Industrial Science and Technology

Sm-Fe magnetostrictive material was produced by unidirectional solidification of Sm-Fe alloy with atomic ratio from 1/2 to 2/17 in microgravity within  $\pm 4 \times 10^{-3}$ g for 1.43s obtained using 10-m drop tower and with fluctuating gravity between 0.1 and 0.02g obtained by parabolic flight. SmFe, and a small amount of Sm<sub>2</sub>Fe<sub>17</sub> as well as Fe were formed from unidirectional solidification in microgravity of Sm-7Fe alloy, obtained using the drop tower and parabolic flight. The structure consisted of sheet dendrites of SmFe, and Fe-rich Sm-Fe layers between the sheet dendrites having no gaps with an orientation along the solidification direction. A crystalline orientation of <111> of SmFe, along the solidification direction was found in the products formed in microgravity using the drop tower, but not in those using parabolic flight. The formation mechanisms of SmFe, sheet dendrites can be explained by microsegregation caused by the lack of convection in melt in microgravity. In contrast, Sm<sub>2</sub>Fe<sub>17</sub> and a small amount of Fe were formed in normal gravity, and the resulting structure consisted of sheet dendrites without orientation. Magnetostriction of -3328ppm at the outer magnetic fields of 0.12T was achieved on a sample synthesized by unidirectional solidification of Sm-7Fe in microgravity obtained using the drop tower.

## Symposium F: Modelling and Simulation of Microstructures and Processes: Numerical Modeling of Material Processing III

Tuesday PM	Room: D
August 3, 2010	Location: Cairns Convention Centre

Session Chairs: Zhang Zhifeng, General Research Institute for Non-ferrous Metals; Michal Krzyzanowski, The University of Sheffield

#### 2:00 PM

**Microstructure of a Paint Primer - A Data-Constrained, Modeling Analysis:** Sam Yang<sup>1</sup>; Dachao Gao<sup>1</sup>; Andrew Tulloh<sup>1</sup>; Tim Muster<sup>1</sup>; Scott Furman<sup>1</sup>; *Sherry Mayo*<sup>1</sup>; Adrian Trinchi<sup>1</sup>; <sup>1</sup>CSIRO

It is common practice that aerospace metallic parts are painted with a primer coating to improve their corrosion resistance performance. The primer generally consists of a polymer matrix with embedded corrosion inhibitor and



filler particles. The performance of the primer is determined by the microscopic distributions of the particles. Various techniques have been used to quantify such distributions, including X-ray micro-CT. However, its success is often limited by factors such as different particles may have similar X-ray CT absorption properties and their sizes may be smaller than the micro-CT resolution. In this paper, we have performed two X-ray CT measurements on a paint primer sample consisting of strontium chromate particles as corrosion inhibitor and titanium oxide particles as filler. We have used Fe and Ti targets respectively as X-ray sources with different spectrum distributions. The measured CT data sets are used as constraints for a data-constrained microstructure modeling (DCM) prediction of microscopic structures of the sample. In order to evaluate the performance of the model predictions, we have done an EDX compositional map of the sample surface. The DCM predicted surface compositional map has a reasonable agreement with the EDX map.

#### 2:15 PM

Computational Fluid Dynamic Simulation of the Ladle Cycle: Mansour Al-Harbi<sup>1</sup>; Abdullah Al-Nufiee<sup>1</sup>; Sami Al-Jarallah<sup>1</sup>; <sup>1</sup>SABIC

Towards optimization of the ladle lining life an understanding of the ladle thermal cycle is essential. Improper control of the lining temperature during the ladle cycle would dramatically reduce the lining life. Therefore, an axissymmetric CFD model was developed. This model was used to predict the lining temperature during the ladle cycle. The model was adapted with the various stages of the ladle cycle. During the ladle drying/preheating the species/combustion model was activated followed by a multiphase model (steel/air) to simulate the secondary steel refining process and casting stage. The temperature at various predefined points located on the lining and the steel shell were monitored and recorded during the cycle simulation. The wall thickness reduction during the lining life was accounted in the model to study the lining thickness influence on the steel shell temperature. The model results were validated comparing with an actual measurement of the steel shell and preheating gases temperature. The model results were in good agreement with the measured temperature. Then this model was used for further optimization of the ladle cycle schedule.

#### 2:30 PM

**Fuzzy Extraction Separation Optimized Process of Tm, Yb and Lu Enriched Oxides by Computer Simulation**: Fengli Yang<sup>1</sup>; *Shaohua Yang*<sup>1</sup>; Changren Tong<sup>1</sup>; Mingzhou Li<sup>1</sup>; Bingliang Gao<sup>1</sup>; <sup>1</sup>Jiangxi University of Science & Technology

According to characteristics of Tm, Yb and Lu enriched oxides composition and problems in its traditional extraction separation process, the computer simulation of a optimized fuzzy extraction separation process, in which three products with high purity(>99.99%) could be obtained at same time, was carried out. Distributing curves of every element in both organic and aqueous phases were worked out. Distribution rules of rare earth elements in every stage of the optimized extraction process were studied. The computation results of economical and technical index showed the consumption of the acid and base chemical reagents in the optimized process would be saved more than 30%.

#### 2:45 PM

**Modeling Corrosion of a Metal under an Aerosol Droplet**: *Murali Venkatraman*<sup>1</sup>; Ivan Cole<sup>1</sup>; Dayalan Gunasegaram<sup>1</sup>; Bosco Emmanuel<sup>2</sup>; <sup>1</sup>CSIRO; <sup>2</sup>Central Electrochemical Research Institute

On a metal surface with a moisture layer of variable thickness and shape, the dissolved oxygen may induce a spatial separation of the anodic and cathodic reactions on space-time scales characteristic of the roughness, droplet size and the local kinetics of the system. This leads to a spatio-temporal variation of species concentrations, current and potential over the metal surface and thus atmospheric corrosion. Deposition of aerosol droplets produced either by marine or industrial activity on the metallic surfaces are particularly important as their chemical constitution strongly promotes corrosion. The atmospheric corrosion caused by aerosols is a result of a complex interplay between solution chemistry, atmospheric chemistry, local electrochemistry, mass transport of dissolved oxygen and other species, relative humidity, nature of corrosion products and most importantly the shape and size of the droplet. Here a fully three dimensional model is developed addressing the corrosion of a metal under an aerosol droplet. The effect of various parameters like exchange current densities, initial concentrations, shape and size of the droplet and diffusivity of oxygen on the evolution of the anodecathode separation are investigated. Also, conditions leading to precipitation are identified.

#### 3:00 PM

**Modelling of Inter-Granular Fracture in Aluminium Alloys**: Wojciech Spychalski<sup>1</sup>; Marek Muzyk<sup>1</sup>; *Krzysztof Kurzydlowski*<sup>1</sup>; <sup>1</sup>Warsaw University of Technology

Grain boundaries in metals significantly influence their properties, among others resistance to intergranular fracture. As the fracture is highly localized process, it proceeds primarily along grain boundaries of the highest energy which, in turn, depends on the segregation of the alloying elements and impurities. The paper shows results of ab-initio modelling of the energy of grain boundaries in aluminium as a function of grain boundary segregation. The ab-initio results are subsequently used to model lowest energy fracture path in polycrystals containing variable fractions of grain boundaries prone to inter-granular fracture.

#### 3:15 PM

**Models of Ternary Slag Structure**: *Angus Gray-Weale*<sup>1</sup>; Patrick Masset<sup>2</sup>; <sup>1</sup>Monash University; <sup>2</sup>TU-Bergakademie Freiberg

Molten oxide slags present in important industrial processes usually have at least three components, for example CaO-SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>. Useful simulation of such a material requires an accurate description of all interactions, and simulation of many different compositions. The oxide's high polarisability, and the importance of its higher order deformations, complicates matters further. Any useful model needs a reasonably realistic model of the melt's network structure, and the effect of calcium and aluminium ions on this structure. We report a first study of a model of the ternary slag CaO-SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>. We demonstrate the use of an integral equation theory to obtain approximate thermodynamics over a wide range of conditions. Normally, such theories are limited to rigid ions, or to species with unphysical classical fluctuations of their internal structure. We demonstrate an integral-equation approach that gives the correct internal structure for several charged and polarisable species. This method is capable of reproducing intermediate ranged order in a model network fluid.

## 3:30 PM

#### Physical Model of Aluminium Refining Process in the Batch and Continuous Reactors: Mariola Saternus<sup>1</sup>; Jan Botor<sup>1</sup>; <sup>1</sup>Silesian Technical University

Both primary and secondary aluminium need to be refined. The most popular methods of aluminium refining is barbotage. This method is based on the introduction of refining gas bubbles into liquid metal. It can be done in batch or continuous reactors. The refining gas can be introduced to the metal by lances, ceramic porous plugs or rotary impellers. The gas bubbles generated in this way are then mixed with the liquid metal and the level of mixing depends on the processing parameters such as the flow rate of refining gas or the impeller speed. Five patterns of the refining gas dispersion in the liquid metal are known: no dispersion, minimal dispersion, intimate dispersion, uniform dispersion and overdispersion. The physical modeling is the best way to visualize these kinds of dispersion. It also helps to choose the adequate processing parameters. However, it is important to remember about keeping the dynamic and geometrical resemblances. In the paper the physical modeling of the aluminium refining process is presented. Two reactors: URO-200 batch reactor with rotary impeller and URC-7000 continuous reactor with ceramic porous plugs were taken into consideration.

#### 3:45 PM

Study on Dynamic Mathematical Model of Ion Exchange Process: Changren Tong<sup>1</sup>; Fengli Yang<sup>1</sup>; Xiaoxue Zhou<sup>1</sup>; <sup>1</sup>Jiangxi University of Science & Technology

Based on the mechanism analysis of fixed-bed ion exchange process, relationship among solution flow in column, external diffusion, inner diffusion and ion exchange reaction was considered, and dynamic mathematical model about fixed-bed ion exchange process was established with thermodynamics, Fick's law of diffusion and flow solution micro-layers theory. In the paper, verification of simulation on tungsten ion exchange process was carried out. The results showed the dynamic mathematical model was matched with practical ion exchange process. Ion exchange process in practical production could be guided and referenced by this simulation model.

#### 4:00 PM Tea Break



# 5:20 PM

First Principles Study of Formation Mechanism of Fcc-NdO<sub>x</sub> in Nd-Fe-B Sintered Magnets: Ying Chen1; Satoshi Hirosawa2; Shuichi Iwata3; 1Tohoku University ; <sup>2</sup>Hitachi Metals, Ltd.; <sup>3</sup>The University of Tokyo

The fcc-NdO<sub>x</sub> phase formed at the Nd/Nd-Fe-B interface in Nd-sputtered Nd-Fe-B sintered magnets is paid rather attention recently due to its important role in coercivity generation of surface Nd-Fe-B grains. Its crystal structures have been reported to vary with the change of the oxygen concentration, and the disorder fcc phase derived from Nd<sub>2</sub>O<sub>3</sub>-C-type structure to be the main form of existence. To understand the formation mechanism of this fcc-NdO, interfacial phase, the stability of all oxygen concentration range of Nd-O system has been investigated from the first principles. Based on LSDA+U calculations for selected ordered phases at various oxygen concentration in Nd-O, the Cluster Expansion Method (CEM) is applied to evaluate the formation energy, density of states and other properties of disorder phase. Furthermore, the formation of oxygen vacancy in Nd<sub>2</sub>O<sub>3</sub>-C-type is calculated which shows that the oxygen vacancies in Nd<sub>2</sub>O<sub>3</sub> also influence the formation of fcc-NdO<sub>x</sub> phase.

#### 5:35 PM

Mechanical Properties and Size Effects of ZnO Nanowires Studied by First-Principles Calculation: Zhanjun Gao<sup>1</sup>; Yousong Gu<sup>1</sup>; Yue Zhang<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing

The mechanical properties of ZnO nanowires are important for the applications in nano-devices and conflicting results have been obtained in previous studies. In this study, first-principles density functional calculations were performed to investigate the mechanical properties of ZnO nanowires and the size effects in order to gain some insight on this subject. In the calculation, structural optimizations were first performed, and a series of deformations were introduced to the nanowires in the axial direction and the ground state energies were calculated. The elastic moduli of ZnO nanowires were calculated from the energy versus strain curves. It is found that the elastic moduli of the ZnO nanowires with three different diameters (1.2, 1.5 and 1.8nm) are 136.3, 138.6 and 138.0GPa, respectively, and that of bulk ZnO along [0001] direction is 140.1GPa. The elastic modulus of ZnO nanowire is slightly lower than that of the bulk and it decreases as the diameter decrease. Possible mechanic are discussed in terms of changes in structure and chemical bonding of the nanowires induced by surface reconstruction.

# 5:50 PM Keynote

Phase Relationships and Structures of Inorganic Crystals by Combination of Cluster Expansion Method and First Principles Calculations: Isao Tanaka<sup>1</sup>; Atsuto Seko1; Fumiyasu Oba1; 1Kyoto University

Properties of materials are strongly dependent not only on their chemical composition but also on configurations of solute atoms and/or point defects. Quantitative knowledge of the configuration-dependent properties is therefore essential for materials design. The cluster expansion (CE) method has been widely used to describe them. Advances in computational power and numerical techniques enable us to perform a large set of systematical first principles calculations to be combined with the CE calculations. In the past such calculations were limited to alloys of close-packed structures. Recently more complex structures, such as non-close packed structures, multi-element alloys and surface segregation have been successfully investigated. We have recently investigated cationic disordering behavior in a series of spinel oxides. Structures and stabilities of a series of non-stoichiometric SnO<sub>2</sub>, compounds, which are yet unknown experimentally, have also been studied. My talk reports these works together with a few other examples.

### Symposium G: Thin Films and Surface Engineering: **Thin Films - Preparation and Properties II**

uesday PM	Room: 8	
ugust 3, 2010	Location:	Cairns Convention Centre

Session Chairs: Seiichi Miyazaki, Hiroshima University; Myung-Hwa Jung, Sogang University

#### 2:00 PM Kevnote

Application of High Power DC Arc Plasma for Mass Production of High Quality Freestanding Diamond Films and Diamond Film Coated Cutting Tools: F.X. Lu1; C.M. Li1; Y.M. Tong1; W.Z. Tang1; G.C. Chen1; J.H. Song1; L.F. Hei1; 1University of Science and Technology Beijing

As quasi-thermodynamic equilibrium plasma, DC arc plasma has the advantage of very high gas temperature and thus very high degree of activation of the precursors for diamond film deposition. The present paper reviews the

# **Symposium F: Modelling and Simulation of Microstructures and Processes: First Principles Studies**

Tuesday PM Room: D August 3, 2010 Location: Cairns Convention Centre

Session Chairs: Tetsuo Mohri, Hokkaido University: Dennis Dimiduk, Air Force Research Laboratory

#### 4:30 PM Keynote

First-Principles Studies of Materials Interfaces: M. Kohyama<sup>1</sup>; S. Tanaka<sup>1</sup>; Y. Shiihara2; S. Ishibashi1; 1National Institute of Advanced Industrial Science and Technology; <sup>2</sup>The University of Tokyo/National Institute of Advanced Industrial Science and Technology

Materials interfaces have crucial roles in various materials and devices, and ab initio calculations of such interfaces combined with electron microscopy observations are of great importance. We present our recent theoretical studies of Au/oxide catalysts and metallic grain boundaries (GBs). Au-particle/ oxide systems reveal novel catalytic activities. For Au/TiO2, results of model configurations indicate the importance of interface stoichiometry and perimeteredge structures. For Au/CeO2, electron microscopy observations revealed novel structural changes of Au particles as cyclic disappearance and recovery according to switching on and off of electron beam. This phenomenon has been theoretically analyzed from the view point of the interface stoichiometry and the behavior of O vacancies and Au atoms, depending on O chemical potentials. We have examined the bonding nature of GBs in typical fcc metals, Al and Cu, and found quite different characters for these two metals.

#### 4:50 PM

First-Principles Calculations of C2H4 Adsorption on Pd Surface Stacked on Fcc-Au: Noboru Taguchi1; Shingo Tanaka2; Tomoki Akita2; Masanori Kohyama2; Fuminobu Hori<sup>1</sup>; <sup>1</sup>Osaka Prefecture University; <sup>2</sup>National Institute of Advanced Industrial Science and Technology (AIST)

So far, we have successfully synthesized binary Au-Pd nanoparticles with various structure such as core shell nanoparticle (Au-core and Pd-shell) and solid solution alloy particles by sonochemical method. The core-shell nanoparticles have higher catalytic activity for hydrogenation of 4-pentenoic acid (CH2=CH-(CH<sub>2</sub>)<sub>2</sub>-COOH) than that for Pd monometallic nanoparticle. TEM observation reveals that lattice space of the Pd-shell are expanded as the same as that of fcc-Au. We have suggested that this lattice expansion is the origin of catalytic activities of the Au-Pd core-shell nanoparticles. In order to study the relationship between the expanded lattice structure of Pd-shell and their surface reactivity, atomic and electronic structures of clean Pd surface and ethylene (CH<sub>2</sub>=CH<sub>2</sub>) (as the simulation of adsorption of 4-pentenoic acid) adsorbed Pd surface overlayers on Au(100) substrates have been calculated by first-principles calculations. In case of thin Pd overlayers (2 layers) with clean surface on the Au substrate, dband shrinking can be seen in LDOS around Pd surface. In the analysis of PDOS around surface shows that highest occupied molecular orbital states become sharp and shift to lower energy region in the ethylene adsorbed Pd overlayers on Au.

#### 5:05 PM

First-Principles Study of Stoichiometry Effects of Au Clusters on CeO, Surfaces: Shingo Tanaka<sup>1</sup>; Tomoki Akita<sup>1</sup>; Masanori Kohyama<sup>1</sup>; Seiji Takeda<sup>2</sup>; <sup>1</sup>UBIQEN, AIST; <sup>2</sup>Osaka University

Au nanoparticles supported on CeO, substrate are well known for their high catalytic activity as a water gas-shift (WGS) reaction at low temperature and a CO oxidation. The fine structure analyses of Au/CeO, catalysts by an analytical transmission electron microscope (TEM) revealed the novel structure changes of Au particles on CeO2 surfaces, the Au particles rapidly shrunk layer by layer to Au monolayer during the observation and the particles recovered after electron beam off. In this paper, we have performed the first-principles calculations of Au clusters on CeO, surfaces using the projector augmented-wave (PAW) program code. The calculated Au-Ce interlayer distance of non-stoichiometric (partially reduced) interface is in good agreement with that obtained from a high-angle annular dark-field scanning TEM (HAADF-STEM) observation, whereas that of stoichiometric one is longer. The analyses of free energies as a function of chemical potential and partial pressure of oxygen revealed that the nonstoichiometric interface is more favourable than the stoichiometric one under the reduction condition. This work was supported by Grant-in-Aid for Specially Promoted Research, the Japan Society for the Promotion of Science Research (JSPS)





progress in R&D of the novel high power DC arc plasma jet CVD system with rotating arc and operated at gas recycling mode for large area high quality diamond film deposition, developed at the University of Science and Technology Beijing in the mid 1990s. Thanks to the continuous efforts made for technological improvement in the past 15 years, a considerable progress has been achieved in the commercialization of this high power dc arc jet CVD system, which is now capable of mass production of large area high quality freestanding diamond films for optical, thermal, and mechanical (tool) applications. The present status in the commercialization and the property level of the resultant diamond films in optical, thermal, mechanical, dielectric, oxidation resistance, sand erosion resistance, and laser damage threshold etc. are presented. Based on the same high power dc arcjet technology, a novel high current extended dc arc plasma CVD system has been developed which successfully changes the diamond film deposition mode from 2D planar deposition into 3D deposition (as confined by two hollow (virtue) columns). It is demonstrated to be advantageous for mass production of diamond thin film coated WC-Co cutting tools. Recent results in the R&D of thin diamond film coated WC-Co drills and end mills, and the results in field tests are discussed.

#### 2:20 PM Keynote

Nanocrystalline Diamond/B-SiC Composite Films: Xin Jiang<sup>1</sup>; <sup>1</sup>University of Siegen

In general, a composite film obtained from two different materials is useful for applications only when the properties of both the components are reasonably incorporated into the resultant film. However, in the case of composite films containing micron sized grains, the overall properties of the film are mostly influenced by only one component, hindering the structure control and thereby the property control. It is therefore necessary to develop a composite film that contains nanometer sized grains in such a way that the availability of large volume of grain boundaries can be controlled which in turn will help in controlling the film properties. The motivation behind synthesizing nanocrystalline diamond/β-SiC composite thin films is to obtain films those posses a whole range of combined properties of diamond and β-SiC to serve tribological, thermal barrier, electronics and biological applications. In this paper, diamond/β-SiC nanocomposite film system will be reviewed with regards to its structural, mechanical and tribological properties.

#### 2:40 PM Kevnote

Influence Mechanism and Effectiveness Evaluation of Magnetic Field Parameters on Magnetron Sputtering Ion Plating Process: Bailing Jiang<sup>1</sup>; Zheng Cao<sup>1</sup>; Yuanyuan Lu<sup>1</sup>; <sup>1</sup>Xi'an University of Technology

The ion density, electron density, and electron temperature (EED) of magnetic field Unclosed state and Closed state magnetron sputtering plasma were compared during sputtering of Cr target in an Ar atmosphere. The results showed that the Unclosed state plasma exhibited a low density; while Closed state plasma exhibited a significantly increased number of charged particles in the center of the vacuum chamber. Cr coatings were depostied using these two magnetic field configuration. The thickness and microstructure of the coatings were characterized using X-ray diffraction, scanning electron microscopy. It was found that the deposition rate of the Closed state Cr depositions was similar to the Unclosed state exhibited large columnar grains and rough surface with protuberances. On the other hand, the inerruption of the large columnar grain growth accompanied denser microstructure and a smoother surface in the Closed state coating. It was hard to achieve high degree of ionization of the sputtered material from the target by magnetic field parameters.

#### 3:00 PM

# Two-Phase nc-TiN/a-(C,CNx) Nanocomposite Films: A HRTEM and MC Simulation Study: Y.H. Lu<sup>1</sup>; X.J. Hu<sup>1</sup>; *Yaogen Shen*<sup>1</sup>; <sup>1</sup>City University of Hong Kong

The grain growth in two-phase nanocomposite Ti-Cx-Ny thin films grown by reactive unbalanced magnetron sputtering in an Ar-N<sub>2</sub> gas mixture with microstructures comprising of nanocrystalline (nc-) Ti(N,C) phase surrounded by amorphous (a-) (C,CNx) phase was investigated by a combination of highresolution transmission electron microscopy (HRTEM) and Monte Carlo (MC) simulations. The HRTEM results revealed that amorphous-free solid solution Ti(C,N) thin films exhibited polycrystallites with different sizes, orientations and irregular shapes. The grain size varied in the range between several nanometers and several decade nanometers. Further increase of C content (up to ~19 at.% C) made the amorphous phase wet nanocrystallites, which strongly hindered the growth of nanocrystallites. As a result, more regular Ti(C,N) nanocrystallites with an average size of ~5 nm were found to be separated by ~0.5-nm amorphous phases. When C content was further increased (up to ~48 at.% in this study), thicker amorphous matrices were produced and followed by the formation of smaller sized grains with lognormal distribution. Our MC analysis indicated that with increasing amorphous volume fraction (i.e. increasing C content), the transformation from nc/nc grain boundary (GB)-curvature-driven growth to a/nc GB-curvature-driven growth is directly responsible for the observed grain growth from great inhomogeneity to homogeneity process.

#### 3:15 PM

#### Highly <111>-Oriented μc-Si Thin Film Grown by Plasma Enhanced Magnetron Sputtering: Su Yuanjun<sup>1</sup>; Takahashi Eiji<sup>2</sup>; Xu Jun<sup>1</sup>; Fan Penghui<sup>2</sup>; <sup>1</sup>Dalian University of Technology; <sup>2</sup>Nissin Electric Co., Ltd.

Microcrstalline Silicon ( $\mu$ c-Si) thin film has been widely recognized as a candidate of a-Si thin film for thin film transistors (TFT) and solar cells because of its high electron mobility and low light-induced degradation. There are various techniques on the preparation of  $\mu$ c-Si thin film such as excimer laser annealing (ELA), hot-wire chemical vapor deposition (HW-CVD), plasma-ehanced CVD (PECVD) and magnetron sputtering (MS). In industry application, PECVD which using highly hydrogen diluted silane is mainly method for preparation of  $\mu$ c-Si thin film. MS method because of it not using toxic and dangerous gases such as silane, phosphine and diborane, and low equipment cost becomes a promising technique to substitute for PECVD. We have successfully deposited  $\mu$ c-Si thin film by inductive coupled plasma (ICP) enhanced magnetron sputtering in hydrogen and argon mixture gases at temperature below 300°C. The  $\mu$ c-Si thin film shows highly <111> oriented growth on both <100> single crystalline Si wafer and glass substrate. The electric characteristic of plasma around deposition area was also detected by Langmuir probe.

### 3:30 PM

**The Preparation for Cu(Sn) Films of Barrierless Interconnection**: Liyan Xu<sup>1</sup>; *Xiaona Li*<sup>1</sup>; Xin Jiang<sup>1</sup>; Chuang Dong<sup>1</sup>; <sup>1</sup>Dalian University of Technology

Cu metallization in Si devices has been widely employed by ultra-largescale integration manufacturers because of the high conductivity and greater electromigration resistance of Cu. However, Cu readily reacts with Si at relatively low temperatures of 200°C, resulting in device failures. A barrier layer is thus needed between Cu and Si to prevent detrimental reactions. In addition, as the dimension of a device decreases, the resistivity inevitably increases due to electron diffuse scattering by the barrier layer. It is therefore essential to propose a new class of Cu metallization, some people use diffuse barrier elements adding to the sputtering copper. The preparation method of Cu (Sn) films, the microstructure and electrical properties of the these films was studied in this paper. The films were deposited onto silicon (100) substrate using magnetron-sputtering, then respectively annealed at various temperatures. The atom percentage of incorporation elements in the film were measured by EPMA. The phases in the films before and after different annealing temperatures were tested by X-ray diffraction and the resistivity of the films was determined by four-point probe method. The results showed that: the Sn element has a certain effect inhibiting the diffusion between Cu to Si substrate.

#### 3:45 PM

Effect of Titania Nanoparticles on Micro-Arc Anodizing of AM60B Magnesium Alloy: Anna Da Forno<sup>1</sup>; Ke Liu<sup>1</sup>; Massimiliano Bestetti<sup>1</sup>; <sup>1</sup>Politecnico di Milano

Oxide films have been produced on AM60B magnesium alloy using microarc oxidation process in an environmentally friendly alkaline solution with and without addition of titania nanoparticles. The surface morphology and crosssection of anodized samples were analyzed by Scanning Electron Microscopy (SEM) coupled with EDS and the phase composition was investigated by X-ray Diffraction (XRD). Hardness tests were performed by means of a micro-hardness tester and the corrosion resistance was evaluated in 3.5% NaCl solution using potentiodynamic polarization tests. Addition of titania nanoparticles does not affect corrosion resistance significantly, but the oxide films produced in titaniacontaining solution are denser, thicker and have more uniform morphology. In the oxides produced at 130V-140V in presence of titania (4g/l) a spinel (MgAl<sub>2</sub>O<sub>4</sub>) was observed. Spinel phase increases the hardness up to 550 HV (10mN, 10 s). The addition of titania nanoparticles in the solution represents an interesting way to synthesize hard coatings on magnesium alloy AM60B by micro-arc oxidation in a short process time (10 minutes).

#### 4:00 PM Tea Break



### Symposium G: Thin Films and Surface Engineering: Process Chemistry and Engineering I

Tuesday PM	Room: 8	
August 3, 2010	Location:	Cairns Convention Centre

Session Chairs: F.X. Lu, University of Science and Technology Beijing; Bailing Jiang, Xi'an University of Technology

#### 4:30 PM Keynote

Characterization of La- and Mg-Diffused HfO<sub>2</sub>/SiO<sub>2</sub> Stack Structures for Next Generation Gate Dielectrics: *Seiichi Miyazaki*<sup>1</sup>; Akio Ohta<sup>1</sup>; Daisuke Kanme<sup>1</sup>; <sup>1</sup>Hiroshima University

In the implementation of Hf-based gate dielectrics, one of the major issues is to overcome the anomalous change in the effective work function of metal gate on Hf-based dielectrics with keeping required electrical thickness and dielectric reliability within allowable leakage current. Recently, it has been reported that such effective work function change of metal gate can be eliminated by incorporation of elements with different oxidation numbers into Hf-based dielectic such as Mg, La and Y atoms for nMOSFETs and Al atoms for pMOSFETs. However, the impact of such additive elements on the chemical bonding features and defect state density in the Hf-base dielectric has not been fully understood yet. In this work, we have focused on La- and Mg-diffusion into HfO<sub>2</sub>/SiO<sub>2</sub> stack structure from ultrathin capping layer by thermal anneal and made the depth analysis of the chemical bonding features using high-resolution XPS in combination with wet-chemical etching. Also, the influence of the La- and Mg- incorporation on the gap states in HfO, has also been examined by total photoelectron yield spectroscopy(PYS). As a result, the defect compensation with La- and Mgincorporation is likely to be responsible for a reduction of work function change in metal gate on HfO<sub>2</sub>.

#### 4:50 PM Keynote

# Synthesis and Possible Thermoelectric Application of Pb-Based Oxide Thin Films: *Myung-Hwa Jung*<sup>1</sup>; <sup>1</sup>Sogang University

A Pb-based oxide material of PbPdO<sub>2</sub> was proposed to be a gapless semiconductor using first principle calculations. Because of the unique gapless band structure, the bulk system of sintered PbPdO<sub>2</sub> powders showed a relatively high thermoelectric power of ~ 270 micro-V/K. It is suitable for thermoelectric application, but it may not be suitable for lack of electrical conductivity. In order to improve the electrical conductivity, we doped Co for Pd in PbPdO<sub>2</sub> and checked possible application for thermoelectric devices with the Co-doped PbPdO<sub>2</sub> thin films. Pure and 10% and 20% Co-doped PbPdO<sub>2</sub> thin films were prepared by pulsed laser deposition. In a thin-film form of PbPdO<sub>2</sub>, we could enhance the electrical conductivity, which is more improved by Co doping. In addition, the materials are ferromagnetic at low temperature due to the strong spin-orbit coupling. From these observations, we conclude that the Pb-based oxide thin films are good candidates for spintronic and thermoelectric applications.

#### 5:10 PM

#### Adaptation of Pulsation and Organic Additives to Control the Mass Transfer of Nickel Electrodeposition: *Jooyul Lee*<sup>1</sup>; Man Kim<sup>1</sup>; <sup>1</sup>Korea Institute of Materials Science

Various kinds of electromagnetic shielding materials have been available in electronic devices and display panels. Electromagnetic shielding by using metallic sheet is thought the most effective, but it has shortcoming not to emit the accumulated heat within the devices. So, mesh shaped metallic materials with high aperture are applied particularly in the high frequency electronic devices. For this purpose, it is critical to control the mesh shape in the perpendicular direction so as not to reduce optical properties of electromagnetic shielding films. In this research, The copper plating was deposited by pulse reverse current method with additives. Results are compared for different additives for pulse plating conditions. When it added in only  $CH_4N_2S$  or only  $C_{10}H_{13}NO_3S$ , the effect of surface side-growth of Cu was not different. But when it added in TU and SVH, surface side-growth of Cu decreased. Polarization curves were measured at a different scan rate and surface and cross section images were observed to investigate the effect of PR parameters on the metal nucleation and its growth.

#### 5:25 PM

# **Development of a Processing Window for the Transformation Hardening of Nickel-Aluminium-Bronze**: *Ryan Cottam*<sup>1</sup>; Milan Brandt<sup>1</sup>; <sup>1</sup>Swinburne University

Nickel-Aluminium-Bronzes (NAB) are typically used in marine applications because of their good combination of corrosion resistance and strength. Even

though these alloys exhibit good properties they do suffer from wear, corrosion, dealloying, cavitation corrosion-erosion or corrosion fatigue during service. Therefore methods of increasing this class of alloy resistance to surface sensitive damage mechanisms are desirable. Transformation hardening through laser processing offers the potential to increase the resistance of these alloys surface sensitive mechanisms of damage and increase their life. A processing window has been developed through the use of an analytical heat transfer model to determine laser processing parameters that are close to the critical temperature for surface melting. The absorption of the laser by NAB has been determined and the processing window calculated taking into account the velocity of the laser, laser spot size and type as well as laser power.

#### 5:40 PM

# Development of Infrared-Ray Curing Technology at Continuous Coil Coating Line: Kyung Park<sup>1</sup>; Dae Woo<sup>1</sup>; <sup>1</sup>Dongbu Steel

The No. 1 continuous coil coating line of Dongbu Steel produces 70,000 tons of commercial quality products annually, and the pre-coated metal(PCM) products are widely used for home appliances or building material. The lines are consisted of pre-treatment, painting, curing and tension levelling processes. The typical curing methods are injection of hot air, infrared rays, electron beam, or induction heated oven. Peak Metal Temperature(PMT) is the vital factor that determines the product quality. The curing oven of No. 1 continuous coil coating line revamped from injection of hot air to infrared rays type and operated from 2007. A PMT setup model was developed for control the strip temperature, and applied in site, and systemized. The operation results showed that the accuracy of the setup model achieved above 96% consistently.

#### 5:55 PM

Effects of Ammonia and Fluoride on Nickel Electroplating for Mg Alloy in Pyrophosphate Bath: *Junghoon Lee*<sup>1</sup>; Yonghwan Kim<sup>2</sup>; Narae Lee<sup>1</sup>; Wonsub Chung<sup>1</sup>; <sup>1</sup>Pusan National University; <sup>2</sup>BK21 Innovative Graduate Education Program for Advanced Materials of Transportation System

Nickel electroplating on Mg alloy AZ31B was conducted in potassium pyrophosphate, nickel sulfate, potassium fluoride and ammonium hydroxide electrolyte. The possibility of direct nickel electroplating in pyrophosphate nickel electrolyte by means of adding potassium fluoride was investigated using polarization methods. Ammonium hydroxide was added to the electrolyte and its effect was investigated. Due to added potassium fluoride in electrolyte, formed fluoride film inhibits the dissolution of magnesium alloy substrate in electrolyte. Added potassium fluoride affects the stability of the Mg alloy in electrolyte. Nickel-ammonia complexes formed by added ammonium hydroxide in electrolyte lead to decrease of charge transfer resistance and overpotential of nickel reduction reaction. Surface cracks on the nickel layer were eliminated by adding ammonium hydroxide. The direct nickel plated Mg alloy showed significantly improved corrosion resistance compared to an uncoated Mg alloy. Due to the addition of potassium fluoride and ammonium hydroxide, a successful and protective electroplated nickel layer was formed on the Mg alloy in pyrophosphate nickel electrolyte.

#### 6:10 PM

Effects of Pulse-Reverse Current on Purity of Deposit in Electrowinning of Cobalt: Jung Min Han<sup>1</sup>; Won Sub Chung<sup>1</sup>; Yong Hwan Kim<sup>2</sup>; Jae Min Kim<sup>1</sup>; <sup>1</sup>Pusan National University; <sup>2</sup>BK21 Innovative Graduate Education Program for Advanced Materials of Trasportation System

In the commercial electrowinning process of cobalt (Co), direct-current (DC) has been mainly used. In this work, modified current modes, pulse-reverse (PR), were applied for producing high purity Co during electrowinning. The electrolyte was based on sulphate solutions, and we selected Cu, Ni, and Fe as the main impurities. The frequency used for pulse reverse current was 100Hz, and the average current density was 6ASD. The duration time ratio, positive peak current density and reverse peak current density in PRC conditions were changed. All of the Cu content in PRC increased from 16% up to 58% compared to DC. On the other hand, the Fe content in all of the PRC case decreased from 60% up to 73% compared to DC. The Ni content in the PRC increased about 44% compared to DC. According to increasing the reverse time, however, the Ni content in the PRC was similar to the DC condition, or it even decreased about 8%.

#### 6:25 PM

# Investigation on the Aligned Surface Morphology of Pure Iridium Experienced Isothermal Oxidation at 1373 K: Zebin Bao<sup>1</sup>; Hideyuki Murakami<sup>1</sup>; Yoko Yamabe-Mitarai<sup>1</sup>; <sup>1</sup>National Institute for Materials Science

Platinum group metals (PGMs) are attracting greater attentions due to their competitive performance at elevated temperatures. Among them, the face-centered cubic (fcc) structured Ir is believed as a promising alloy base in developing next generation of structural materials because of its superior high melting point and excellence of high-temperature strength. The unavoidable challenge for its application as hot-section components is the consumptive mass loss induced by



the gaseous oxide transpiration which prevails at temperatures above 1369 K. In order to improve the feasibility of Ir-based alloy being used in an aggressive environment, this preliminary work dedicates to investigate oxidation behavior of pure Ir at a dangerous temperature of 1373 K, with a focused objective on the surface microstructure change. The mechanism of how the aligned surface morphology formed is discussed basing on experimental results plus crystalline simulations.

# Symposium H: Advanced Ceramics: Porous Ceramics

Tuesday PM August 3, 2010 Room: 3 Location: Cairns Convention Centre

Session Chairs: Katsutoshi Komeya, Yokohama National University; Kyeong-Ho Baik, Chungnam National University

#### 2:00 PM Keynote

Fabrication, Structure Control and Application of Porous Ceramics with High Porosity and High Strength: *Chang-An Wang*<sup>1</sup>; Ankun Yang<sup>1</sup>; Liangfa Hu<sup>1</sup>; Ruifeng Chen<sup>1</sup>; Yong Huang<sup>1</sup>; <sup>1</sup>Tsinghua University

Compared to water, tert-butyl alcohol (TBA) has higher saturated vapor pressure and low surface tension, which is suitable for gelcasting process as solvent. In this report, two novel processing techniques for fabrication of porous ceramics with high porosity and high strength as well as controllable pore structure will be introduced. By gelcasting process, porous ceramics with uniformly distributed pore structure were prepared with high porosity and high strength. Uniform pore structure, submicron to micron pore size and formation of sintering necks between adjacent grains are main reasons for high porosity and high strength. Combining gelcasting and freeze-drying processes, a freeze-gelcasting process was put forward to fabricate porous ceramics with one-dimensional channel pore structure and gradient pore structure, respectively. Some research progress on porous zirconia ceramics, which is for applications with light-weight, heatinsulation and load-bearing purpose, and porous piezoelectric ceramics (PZT), which is for applications in ultrasonic transducers like hydrophones and underwater transducers, will also be introduced.

#### 2:20 PM

**Development of CNT-Dispersed Si**<sub>3</sub>**N**<sub>4</sub> **Ceramics**: *Junichi Tatami*<sup>1</sup>; Sara Yoshio<sup>1</sup>; Tomohiro Yamakawa<sup>1</sup>; Toru Wakihara<sup>1</sup>; Katsutoshi Komeya<sup>1</sup>; Takeshi Meguro<sup>1</sup>; <sup>1</sup>Yokohama National University

Si<sub>3</sub>N<sub>4</sub> ceramics have been used for engineering components. Such Si<sub>3</sub>N<sub>4</sub> ceramics are insulator, so that static electricity sometimes causes problems. Carbon nanotubes (CNTs) are good candidate as fillers to give electrical conductivity. However, the reaction between CNT and Si<sub>3</sub>N<sub>4</sub> or SiO<sub>2</sub> during sintering have to be suppressed. In this study, based on in-situ measurement of sintering of Si<sub>3</sub>N<sub>4</sub> ceramics, the effect of TiO<sub>2</sub> and/or HfO<sub>2</sub> addition as sintering aids on densification behavior was investigated in order to develop CNT dispersed Si3N4 ceramics. TiO, addition lowered the onset temperature of densification by dissolution and precipitation due to phase transformation from  $\alpha$ - Si<sub>3</sub>N<sub>4</sub> to  $\beta$ - Si<sub>2</sub>N<sub>4</sub>. HfO<sub>2</sub> additive resulted in the rapid densification with grain growth of  $\beta$ - Si<sub>3</sub>N<sub>4</sub>. As a result, optimization of sintering temperature and additives leaded to the dense CNT-dispersed Si<sub>3</sub>N<sub>4</sub> ceramics. It was found that CNTs existed in the grain boundary by TEM observation. The developed CNT-dispersed Si<sub>2</sub>N<sub>4</sub> ceramics showed high strength and electric conductivity. The threshold of the percolation of CNTs for electrical conductivity were about 0.5vol %. Tribological properties were changed by dispersing CNTs in Si<sub>3</sub>N<sub>4</sub> ceramics.

#### 2:35 PM

One-Step Wet Chemical Synthesis of Solid Solution of  $SrTi_x Zr_{1,x}O_3$  Powder for High-Voltage Applications and the Thermodynamics Analysis of the Process: *Xiaolin Liu*<sup>1</sup>; Yanting Liu<sup>1</sup>; Yong Zhang<sup>2</sup>; Jianfeng Chen<sup>1</sup>; <sup>1</sup>Beijing University of Chemical Engineering; <sup>2</sup>Tsinghua University

The combination of attributes of  $SrZrO_3$  and  $SrTiO_3$  may be desirable in producing perovskite-based dielectrics with useful dielectric constants and high breakdown strengths. At present  $SrZrO_3$ - $SrTiO_3$  ceramics can be made by phase-pure powders of  $SrZrO_3$  and  $SrTiO_3$  materials which came into being ununiformity in the ceramics. In this work single phase solid solution of  $SrTi_xZr_{1,x}O_3$  (x>0.4) powders were synthesized through one-step aqueous process at low-temperature (80°C), in which strontium nitrate, zirconium oxychloride, titanium tetrachloride and sodium hydroxide were used as reactants. The series of samples were characterized by X-ray diffraction (XRD). The characteristic peaks of  $SrTi_xZr_{1,x}O_3$  with the perovskite structure moved to large angle and

the crystalline of SrTi<sub>x</sub>Zr<sub>1,x</sub>O<sub>3</sub> tended to more perfect with the increase of x from 0.4 to 1. And when x<0.4, SrTi<sub>x</sub>Zr<sub>1,x</sub>O<sub>3</sub> can be obtained by additional aging step of the sample for a period of time. We have also explained the synthesis process of SrTi<sub>x</sub>Zr<sub>1,x</sub>O<sub>3</sub> (x=1~0) solid solution powders by calculating Gibbs free energy change of SrTi<sub>x</sub>Zr<sub>1,x</sub>O<sub>3</sub> during its forming process based on thermodynamics principle. The experimental result showed that this synthesis method might also offer an effectively new way to synthesis other titanate powders with perovskite structure.

#### 2:50 PM Keynote

Synthesis of SiC Porous Bulk in Na Vapor: Takayuki Shirai<sup>1</sup>; Haruhiko Morito<sup>1</sup>; *Hisanori Yamane*<sup>1</sup>; <sup>1</sup>IMRAM, Tohoku University

SiC porous bulk was prepared by heating a mixture of Si and carbon black powder in Na vapor at 973 - 1173 K. The porosity of the bulk was about 50-60%. The shape and size of large pores in the bulk samples depended on those of the Si grains in the starting mixture. The bending strength of the porous bulk was 14 MPa. The porosity of the bulk could be increased to about 85% by using a carbon-rich starting mixture and by oxidation of the extra carbon after SiC formation.

#### 3:10 PM

**Preparation of Nano-Zeolite by Bead Milling**: *Toru Wakihara*<sup>1</sup>; Ryuma Ichikawa<sup>1</sup>; Junichi Tatami<sup>1</sup>; Katsutoshi Komeya<sup>1</sup>; Takeshi Meguro<sup>1</sup>; <sup>1</sup>Yokohama National University

Zeolites are hydrated, crystalline tectoaluminosilicate that are constructed from  $TO_4$  tetrahedra (T = tetrahedral atom, e.g. Si and Al). They contain nanometersized and well-ordered void spaces in their structures. Because of their welldefined structures and large ion-exchange capabilities, they have been used as ion exchangers, adsorbents, catalysts, molecular-sieving membranes and so on. For these uses, nano-zeolites often show better properties because of their high surface area and easy access to internal pores. Therefore, the synthesis of nanozeolite is attracted considerable attention as one of the main topics in zeolite science. In the present study, we focus our attention on the use of bead milling for the preparation of nano-zeolite A (Nano-LTA) via top-down approach. It was confirmed that nano-zeolite A with high crystallinity has successfully been prepared by bead milling. Further, ion-exchange property was improved by the milling and subsequent hydrothermal treatment of zeolite A.

#### 3:25 PM

Synthesis of Porous Metal Oxide Beads Using Alginate as a Template: Maryline Chee Kimling<sup>1</sup>; Nicholas Scales<sup>2</sup>; Rachel Caruso<sup>3</sup>; <sup>1</sup>Particulate Fluids Processing Centre, School of Chemistry, The University of Melbourne; <sup>2</sup>Institute of Materials Engineering, Australian Nuclear Science and Technology Organisation; <sup>3</sup>CSIRO, Materials Science and Engineering

Hierarchical porous materials have found extensive applications in the fields of catalysis, controlled delivery or adsorption/separation; benefiting from high surface areas and fast mass transport properties. In this study we demonstrate the feasibility of using alginate as a scaffold for producing millimetre-sized mesomacroporous inorganic beads. Sol-gel chemistry was employed in fabricating mixed titania/zirconia beads with high surface areas of up to 226 m<sup>2</sup> g<sup>-1</sup>. The encapsulating property of alginate was studied towards the incorporation of additional templates or fillers for the production of inorganic beads of varying pore architecture, surface area and tailorable porosity. For instance, submicrometer titania beads with a monomodal pore size distribution were utilised to broaden the mesopore size regime of the inorganic beads from 6 nm to 9 nm in an attempt to provide further and better access for fluids to flow through and within the porous bead matrix. The adsorption kinetics and capacity of the synthesized materials were studied using molecular probes. Such mixed titania/zirconia beads would be ideally suited as adsorbents in column chromatographic separation techniques for the treatment of contaminated water or nuclear waste.

#### 3:40 PM

# Solid-State Reaction Synthesis and Mechanism of Lithium Silicates: *Tao Tang*<sup>1</sup>; Huogen Huang<sup>1</sup>; Deli Luo<sup>1</sup>; <sup>1</sup>CAEP

Lithium-based ceramics have been recognized as promising tritium breedingmaterials for D-T fusion reactor blankets. Lithium silicates,  $Li_4SiO_4$  and  $Li_2SiO_3$ , are recommended by many ITER research teams as the first selection for the solid tritium breeder. A lot of methods have been proposed to synthesize lithiumcontening ceramics, but the solid state reaction method is yet the most important way to synthesize lithium silicates. In present study, the processes of solid-sate reaction between amorphous silica and  $Li_2CO_3$  powders was investigaed by TGA/ DSC; the lithium silicate powders were synthesized at 973~1173 K with different Li:Si molar ratio using solid-state reaction method. The as-prepared lithium silicates were characterized by XRD, SEM and other technology. The optimized synthesis temperature and the solid-state reaction mechanism were derived on the base of experimental results.



#### 3:55 PM

Characterization and Activity of La<sub>1-x</sub>Ce<sub>x</sub>CoO<sub>3</sub> (x=0, 0.2, and 0.4) Prepared by Sol-Gel Process of Metal-Nitrate Precursors as Tar Removal Catalyst: Use of Toluene as a Model Compound: Duangduen Atong<sup>1</sup>; *Kanit Soongprasit*<sup>2</sup>; <sup>1</sup>National Metal and Materials Technology Center; <sup>2</sup>Chulalongkorn University

Perovskite-type mixed oxides have been investigated for many applications especially steam reforming of volatile organic compounds. The performance of mixed oxides catalysts are generally related to porosity and surface area which strongly depends on synthesis method. In this paper, La<sub>1-x</sub>Ce<sub>x</sub>CoO<sub>3</sub> (x=0, 0.2, and 0.4) were prepared by sol-gel process using polyvinyl alcohol (PVA) as a gelating agent with metal nitrate to PVA molar ratio of 1:1. The precursor solutions were calcined at 700°C for perovskite phase transformation. After calcination, phase, morphology, and elemental dispersion were investigated by x-ray diffraction (XRD), scanning electron microscope (SEM), and energy dispersion spectroscopy (EDS), respectively. XRD diffractogram showed high crystalline structure of perovskite oxide catalyst. SEM and EDS confirmed homogeneous structure with low agglomeration. Particle size and BET specific surface area were in the range of 9.58-21.23  $\mu m$  and 3.10-11.6 m2/g, respectively. Steam reforming of volatile organic compounds was carried out in a fixed bed quartz reactor at 500-800°C. Higher temperature of reforming appeared to enhance preferred fuel gas species in terms of quantity and selectivity. However further gas upgrading may be required because low heating value and H,/CO were obtained under these conditions.

#### 4:10 PM Tea Break

# Symposium H: Advanced Ceramics: Sintering of Ceramics

Tuesday PM	Room: 3
August 3, 2010	Location: Cairns Convention Centre

Session Chairs: Yong Huang, Tsinghua University; Ik Kim, Hanseo University

#### 4:30 PM Keynote

#### Silicon Carbide Ceramics with Aluminum Nitride through Various Processes: Junichi Hojo<sup>1</sup>; <sup>1</sup>Kyushu University

Silicon carbide ceramics have been applied to high strength, wear resistance and semiconducting materials. There are two ways for controlling the sintered texture: selection of sintering additive and optimization of heating schedule. Spark plasma sintering (SPS) technique has a great advantage of rapid heating owing to an energy concentration into specimens. It has been found that aluminum nitride is effective as additive to produce nanostructured silicon carbide ceramics by the SPS process. Aluminum nitride can be dissolved into silicon carbide in a wide compositional range. The dissolution of aluminum nitride tended to induce the phase transformation of silicon carbide. Silicon carbide composites with aluminum nitride have been found to be densified without additional sintering aid by the SPS technique. A fine-grained microstructure was obtained at low temperatures and homogenized with grain growth by elevating temperature owing to formation of the solid solution. The dissolution of aluminum nitride increased the electrical resistance of silicon carbide. The silicon carbide composites were also fabricated by using polymer precursor composed of organic silicon and aluminum compounds. Silicon carbide and aluminum nitride composite powder was also produced by a carbothermal reduction nitridation process.

#### 4:50 PM

Millimeter-Wave Reactive Sintering of Neodymium-Doped Yttrium Aluminum Garnett: *M. Ashraf Imam*<sup>1</sup>; Arne Fliflet<sup>1</sup>; Steven Gold<sup>1</sup>; Ralph Bruce<sup>2</sup>; Chad Stephenson<sup>3</sup>; Jerry Feng<sup>1</sup>; <sup>1</sup>Naval Research Lab; <sup>2</sup>Bethel College; <sup>3</sup>University of Notre Dame

Millimeter-wave sintering of ceramic laser host materials has been under investigation at the Naval Research Laboratory (NRL) for high-energy laser (HEL) applications. Advantages of polycrystalline, compared to single-crystal laser host materials include lower processing temperature, higher gain from higher dopant concentration, cheaper fabrication, and larger devices. We are currently investigating the solid-state reactive sintering of neodymium-doped yttrium aluminum garnet (Nd:YAG) using a high power millimeter-wave beam as the heat source. The 83 GHz beam is generated in High Frequency Materials Processing Facility of NRL that is powered by a 15 kW, CW, 83 GHz GYCOM gyrotron. The starting powder is a mixture of commercially available alumina, yttria, and neodymia powders. Near transparency and over 99% theoretical density have been achieved with grain sizes of 5 to 10  $\mu$ m. The fluorescence lifetime of the Nd<sup>+3</sup> 1.06  $\mu$ m lasing transition was measured to be about 200  $\mu$ s, in good agreement with other work. SEM studies of the sintered microstructure

show residual porosity caused by trapped pores that must be eliminated to produce fully transparent material.

### 5:05 PM

#### Reaction Sintering Kinetics of Mullite Ceramics Prepared from High Aluminum Fly Ash: Jinhong Li<sup>1</sup>; <sup>1</sup>China University of Geosciences, (Beijing)

This research proposed a model of mullitization mechanism of fly ash and bauxite reactants couples reaction, which was termed as cenosphere mullitization to summarize the sintering process of the reactions from high aluminum fly ash. Firstly, crystallization of mullite takes place around the pristine mullite seeds in the shell of the cenosphere of fly ash with the formation of gas during combustion. And then the hollow microspheres were broken for the swelling of gas and leading to green ceramics volume shrinkage. Lastly, the mullite crystal grows as the reaction route among a-Al<sub>2</sub>O<sub>3</sub> and amorphous SiO<sub>2</sub>. It was found that mullitization reaction takes place in the order of: Al<sub>2</sub>O<sub>3</sub>(liq)+SiO<sub>2</sub>(liq)>Al  $_{2}O_{3}(s)$ + SiO<sub>2</sub>(liq) >Al<sub>2</sub>O<sub>3</sub>(liq)+SiO<sub>2</sub>(s)>Al<sub>2</sub>O<sub>3</sub>(s)+ SiO<sub>2</sub>(s) via thermo-dynamic and kinetics calculation. In fly ash-bauxite couple systems, the apparent free engery is ~100 kJ/mol lower than those of decomposition reactions of andalusite, sillimanite and kyanite, and also lower than that in the systems of  $a-Al_{a}O_{a}+quartz$ or a-Al<sub>2</sub>O<sub>3</sub>+quartz. The apparent mullitization active energy is around 151 kJ/mol at the range of 1100~1500 °C. When 4 wt% Na<sub>2</sub>O added into the initial batches of AS90(Al<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub>=0.9 in molar ratio), the apparent active energy was evaluated to decrease to 92 kJ/mol at the range of 1100~1300°C.

#### 5:20 PM Invited

Gelcasting of Ceramic Particle Stabilised Foams: George Franks<sup>1</sup>; <sup>1</sup>University of Melbourne

The microstructure of gelcast ceramic foams is an important factor which influences mechanical strength, permeability, thermal conductivity and density. The present work studies the morphology, such as the amount and average size of porosity of alumina (Al<sub>2</sub>O<sub>2</sub>) gelcast ceramic foams. The microstructure of the ceramic foams is influenced by the surfactant concentration and type which is added to the ceramic suspension to cause the particles to become hydrophobic so that they stabilise air bubbles introduced by beating. It was found that the microstructure transforms from a closed pore (bubble) morphology, at low surfactant concentration to opened pore (granular) morphology at high surfactant concentration. The change in morphology is related to the surface hydrophobicity of the particles which controls the stability of the bubbles. The fired ceramic foams contain between about 50 and 80% porosity with average pore size ranging from about 100 to 400 microns depending on the formulation. The use of polyvinyl alcohol and a temperature activated crosslinking agent as a gelcasting system minimized drying related cracking so that large and complex shaped components may be fabricated. The ceramic foams have compressive strength in the range of about 15 to 40 MPa depending on the formulation.

#### 5:35 PM

Effect of HfO<sub>2</sub> and TiO<sub>2</sub> Addition on Sintering Behavior of the Si<sub>3</sub>N<sub>4</sub>-Sinetring Aids: *Katsutoshi Komeya*<sup>1</sup>; Tomohiro Yamakawa<sup>1</sup>; Junichi Tatami<sup>1</sup>; Toru Wakihara<sup>1</sup>; Noriaki Endo<sup>2</sup>; <sup>1</sup>Yokohama National University; <sup>2</sup>JEOL

Silicon nitride(Si<sub>3</sub>N<sub>4</sub>) ceramics can be generally fabricated by liquid phase sintering with additives. To obtain more reliable materials as bearing balls, we have studied the the additives of HfO<sub>2</sub> and TiO<sub>2</sub> to the Si<sub>3</sub>N<sub>4</sub>-Y<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub>-AlN composition. It is meaningful to clarify the role of these additives from the standpoint of sintering kinetics, grain/grain boundary interaction and microstructural characterization. The objective of this work is to understand the effect of TiO<sub>2</sub> and HfO<sub>2</sub> addition on the sintering behavior and the microstructure of the sintered bodies, in which six kinds of the HfO<sub>2</sub>/TiO<sub>2</sub> ratios for the fixed basic compositions were selected as 0/0, 0/5 to 5/0(wt%/wt%). By the addition of TiO<sub>2</sub> and/or HfO<sub>2</sub>, the densification was enhanced. In these compositions, HfO<sub>2</sub>/TiO<sub>2</sub>=1.5/3.5 showed the highest densification rate, 5/0 was similar to the 0/5 composition. The densification promotion was explained by the typical liquid phase sintering accompanying with eutectic reaction in the system HfO<sub>2</sub>-Y<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> and the diffusion of Ti or Hf in the grain boundary glassy phases.

#### 5:50 PM

#### Joining of SiC by Liquid-Phase-Sintered SiC interlayer with Al<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub> Additives: *Wubian Tian*<sup>1</sup>; Hideki Kita<sup>1</sup>; Naoki Kondo<sup>1</sup>; Hideki Hyuga<sup>1</sup>; Takaaki Nagaoka<sup>1</sup>; <sup>1</sup>National Institute of Advanced Industrial Science and Technology (AIST), Japan

SiC substrates were successfully joined by powder mixtures or tapes of SiC (a- and  $\beta$ -polytype) with Al<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub> additives in the temperature range of 1700-1900°C. It was found that dense joints can be obtained at temperatures higher than 1800°C whatever the starting materials are. Elongated grains in SiC joints are frequently presented as starting from  $\beta$ -SiC joining materials due to the phase transformation of SiC polytype. Moreover, tape-like joining materials result in thinner and more uniform microstructures than powder-like ones, which subsequently leading to superior mechanical properties of SiC joints. SiC joints


fracturing within SiC substrates were produced by using optimized joining variables.

#### 6:05 PM

**Densification Behavior in Spark-Plasma-Sintering of MgAl<sub>2</sub>O<sub>4</sub> Spinel:** *Koji Morita*<sup>1</sup>; Byung-Nam Kim<sup>1</sup>; Hidehiro Yoshida<sup>1</sup>; Keijiro Hiraga<sup>1</sup>; <sup>1</sup>National Institute for Materials Science

We have recently demonstrated that, for spark-plasma-sintering (SPS) processing, slow heating rate is effective for attaining transparent oxide ceramics rather than the widely used high heating rate. For a slow heating rate of 10°C/ min, for example, transparent spinel can be fabricated for only a 20 min soak at 1300°C. Since the heating rate dependent sintering behavior can be related to densification mechanisms, the present study was performed to examine the densification mechanisms during SPS processing. As density  $\rho$  increases, that is, as effective stress  $\sigma_{eff}$  decreases, stress exponent n evaluated from  $\sigma_{eff}$  dependence of densification rate varies from  $n \ge 4$  at low  $\rho$  to  $n \approx 1$  at high  $\rho$ . The two regions may be connected by  $n \approx 2$  at the intermediate  $\rho$ . TEM observation shows that significant stacking faults caused by partial dislocations are observed at the low  $\rho$ , but limited at high  $\rho$ . By combining the analysis of the densification behavior with the microstructural observations, the densification mechanism in SPS processing can be ascribed to partial dislocation mechanism in SPS processing for the intermediate  $\rho$  and diffusion-related creep for the high  $\rho$ .

#### Symposium J: Materials Characterisation and Evaluation: 3D Tomography

ue. PN

Room: 1 Location: Cairns Convention Centre

Session Chair: Jin Zou, The University of Queensland

#### 2:00 PM Keynote

Tuesday PM

August 3, 2010

Atomic Tomography of Insulating Ceramics by Laser Assisted 3D Atom Probe: *Kazuhiro Hono*<sup>1</sup>; T. Ohkubo<sup>1</sup>; Y. M. Chen<sup>2</sup>; M. Kodzuka<sup>2</sup>; F. Li<sup>1</sup>; <sup>1</sup>National Institute for Materials Science; <sup>2</sup>University Tsukuba

Recent successful implementation of pulse lasers to assist field evaporation has expanded the application areas of the atom probe technique to a wide variety of materials including semiconductors and their thin film devices. It was believed that only electrical conductive materials can be analyzed by the atom probe technique with only a few exceptions of ultra-thin insulating oxide films. To the author's knowledge, there has been no report on successful atom probe analyses of *bulk* insulating ceramics. The main objective of this work was to demonstrate that even insulating bulk ceramics can be quantitatively analyzed by the 3DAP assisted with ultra-violent (UV) femotosecond laser pulses. A Yb:KGW femtosecond laser with a third harmonic generator ( $\lambda$ =343nm, 400 fs) operating at the pulse frequency of 2kHz was adopted to a locally built 3DAP instrument with CAMECA's fast delay line detector. As demonstration samples, we have tested Y<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub>-MgAl<sub>2</sub>O<sub>4</sub>, Al<sub>2</sub>O3, Li(Co,Ni,Mg,Al)O<sub>2</sub>, (Ce,Dy)O<sub>2</sub>, ZnO, Y<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> sintered bulk ceramics. Even from these insulators, we were able to obtain field ion microscope (FIM) images. The atom probe tomography obtained from these samples have demonstrated that phase separations, nanocomposite structures and grain boundary segregations of insulating ceramics can be accurately reconstructed by the laser assisted 3DAP.

#### 2:20 PM Keynote

#### **Dynamic 3-D Digital Structure: Program Overview**: Julie Christodoulou<sup>1</sup>; <sup>1</sup>Office of Naval Research

The Dynamic 3-D Digital Structure program aimed to develop and demonstrate the fundamental research approaches and tools necessary to provide an integrated computational materials science foundation for materials design, processing and behavior prediction. During the five-year effort, three teams led by QuestTek/Northwestern University, the Ohio State University, and the Naval Research Laboratory were supported jointly by the Defense Advanced Research Projects Agency (DARPA) and the Office of Naval Research to focus on three primary objectives. They developed characterization and computational tools necessary to translate micrographs (analog data sets) into digital data sets to more accurately and quantitatively describe salient microstructural features and their distributions. This work was tightly integrated with efforts to develop models of microstructural evolution derived at appropriate length scales and dynamically coupled to one another to better articulate the mechanisms of steel and titanium alloy responses to processing or service conditions. Critically, the teams also developed and tested a system architecture and protocol for data and knowledge exchange between experimentalists and computational scientists and between research groups. Progress made toward these goals will be discussed.

#### 2:35 PM Invited

Direct Atomic Scale Observation of the Structural and Compositional Transition across Order/Disorder Gamma Prime/Gamma Interfaces in Nickel Base Superalloys: *Rajarshi Banerjee*<sup>1</sup>; Srinivasan Rajagopalan<sup>2</sup>; Gopal Viswanathan<sup>3</sup>; Junyeon Hwang<sup>1</sup>; Soumya Nag<sup>1</sup>; Jaimie Tiley<sup>3</sup>; Hamish Fraser<sup>4</sup>; <sup>1</sup>University of North Texas; <sup>2</sup>Exxon Mobil Research and Engineering Company; <sup>3</sup>Air Force Research Laboratory; <sup>4</sup>Ohio State University

In high temperature materials, such as nickel base superalloys, the interface between the ordered gamma prime precipitate and the disordered gamma matrix plays a critical role in determining its high temperature microstructural stability, including the rate of precipitate coarsening, the strengthening mechanisms, and mechanical properties. Combining aberration-corrected high resolution scanning transmission electron microscopy (HRSTEM), carried out in an FEI TITAN microscope, with three-dimensional atom probe (3DAP) tomography, carried out in a local electrode atom probe (LEAP) system, the transition in atomic scale structure and composition across the order/disorder interface in a nickel base superalloy has been determined. These investigations clearly reveal the presence of two interface widths, one corresponds to the order-disorder transition, while the other corresponds to the compositional gradient across the interface. While, the order-disorder transition across the interface is  $\sim$  6-8 atomic layers thick, the compositional gradient across the same interface is ~ 12-14 atomic layers thick, raising fundamental questions regarding the definition of these interfaces. The variation of the interface width for different types of gamma prime precipitates (primary and secondary) has also been studied as a function of cooling rate (from a supersolvus annealing temperature) and aging time and will be discussed in this presentation.

#### 2:50 PM

**3D** Imaging of Lattice Structure and Dopant Distribution in Silicon Using Atom Probe Tomography: *Andrew Breen*<sup>1</sup>; Michael Moody<sup>1</sup>; Leigh Stephenson<sup>1</sup>; Simon Ringer<sup>1</sup>; <sup>1</sup>Australian Key Centre for Microscopy and Microanalysis, The University of Sydney

Imaging the lattice position of atoms within advanced materials is an ultimate goal of microscopy. This has been achieved in part with the use of aberration corrected transmission electron microscopy to resolve sub angstrom atomic separations along 2D crystallographic projections. Atom probe tomography (APT) is another powerful microscopy technique capable of resolving the lattice structure and chemical composition of atoms in 3D. In this study, advanced spatial distribution map (SDM) techniques have been developed to characterise the crystallographic information present in APT reconstructions of doped silicon and define the limits of spatial resolution in the analysis of semiconductor materials achievable via APT. Techniques based on the crystallography characterised via SDM have also been developed to restore the diamond cubic lattice structure of the original specimen. Once applied these lattice rectification techniques have allowed the impurity distribution within doped silicon to be presented with lattice resolution for the first time.

#### 3:05 PM

**Challenges Associated with the Characterisation of Nanocrystalline Materials Using Atom Probe Tomography**: Fengzai Tang<sup>1</sup>; Talukder Alam<sup>1</sup>; M.P. Moody<sup>1</sup>; B. Gault<sup>2</sup>; *Julie Cairney*<sup>1</sup>; <sup>1</sup>The University of Sydney; <sup>2</sup>University of Oxford

Very high strength and hardness in nanocrystalline metals have been attributed to unique deformation mechanisms. One means by which nanocrystalline Al deforms is stress-induced grain growth. Recent evidence suggests that impurities at the grain boundaries hinder this grain growth, increasing strength, but direct observation of these impurities has not been accomplished to date due to experimental limitations. In this study, atom probe tomography has been used to directly measure grain boundary O levels in nanocrystalline Al thin films for the first time. Specimens were prepared using a focused ion beam (FIB)based technique developed in-house. Results confirm decreasing grain size and increasing strength with increasing levels of O segregation. Comparison with TEM images showing evidence of stress-driven discontinuous grain growth support suggestions that a critical impurity concentration is required to sufficiently pin or immobilize grain boundaries against the coupling of applied stresses, dramatically increasing the strength, but lowering ductility. Controlled addition of O during processing may therefore offer the opportunity to tailor the mechanical properties of these materials to the needs of designers and engineers



#### 3:20 PM

The Fluctuation of In Composition in InGaN Based LED Investigated by Atom Probe Tomography: *Bo Hwa Kim*<sup>1</sup>; Gil Ho Gu<sup>1</sup>; Chan Gyung Park<sup>2</sup>; <sup>1</sup>Pohang University of Science and Technology (POSTECH); <sup>2</sup>Pohang University of Science and Technology (POSTECH) and National Center for Nanomaterials (NCNT)

It has been known that indium (In) may cause the high emission efficiency of InGaN based LED, despite of high density of misfit dislocations in GaN layer. In the present study, the relationship between the fluctuation of indium composition and internal quantum efficiency of InGaN based LEDs has been investigated. The indium composition of commercial InGaN based LEDs with and without annealing at 900°C has been analyzed by using atom probe tomography (APT). The results revealed that the decrease in indium composition caused by annealing at 900°C for 30 min. exhibited the wavelength change (approximately 6.13 nm blueshift) in the photoluminescence (PL) of the InGaN multi quantum well (MQW). The wavelength change in the PL of InGaN based LEDs will be discussed in terms of the composition and distribution of indium in InGaN/GaN.

#### 3:35 PM

High Angle Resolution Electron Channeling Electron Spectroscopy to Analyze Valence States of Transition Metal Ions in Oxide Ceramics: *Kazuyoshi Tatsumil*<sup>1</sup>; Ikuo Nishida<sup>1</sup>; Shunsuke Muto<sup>1</sup>; Jan Rusz<sup>2</sup>; <sup>1</sup>Nagoya University; <sup>2</sup>Uppsala Unversity

We have developed a site-selective EELS analysis for the crystalline materials by using electron-channeling effects. Systematic beam orientations controlled by a PC script allow us to acquire a series of EELS fine structures where site-specific core-loss (ELNES) intensities are mixed with different weights. Applying the multivariate analysis on the data, the truly site-specific ELNES are clearly distinguished (J. Phys.: Condens. Matter. 21 (2009) 104213), for example, Fe L<sub>23</sub> spectra at the tetrahedral and octahedral sites in spinel structures. Since the shape of Fe L<sub>23</sub> is mostly determined by its local coordination and valence, we can analyze how much different valence states are mixed in a specific site, by fitting the site-specific spectrum with known Fe L23 spectra of different valences but similar local coordination. For this high angle resolution electron channeling electron spectroscopy (HARECES), we calculate the dependence of the siteselectivity on the beam orientations, based on many-beam dynamical electron scattering theory. We discuss about further quantitative analyses on the sitespecific chemical states in complex crystal structures of some ceramic materials. Our calculation and experimental scheme has attained a quantitative site-by-site chemical state analysis for the first time, which is beyond the capability of the ALCHEMI method using X-ray spectroscopy.

#### 3:50 PM

The Study on the Nucleation Behaviours in IF Steel by 3-D FIB-EBSD **Tomography**: *Wanqiang Xu*<sup>1</sup>; Michael Ferry<sup>1</sup>; <sup>1</sup>University of New South Wales 3-DFIB-EBSD tomography was used to analyze the structure and crystallography of nuclei, and nucleation process in 3-D space during annealing a cold rolled IF steel. It is revealed that the structure of nucleus at its very initial formation stage can be divided into two parts: (1) nucleation core, having a dislocationcontained subgrain structure, mainly bounded by low angle grain boundary with surrounding deformation subgrain, (2) newly-grown region, having a dislocation free structure, formed from high store energy deformation structures surrounding nucleus, and mainly bounded by high angle grain boundary. It is directly approved that origin and orientation of nucleus are the nucleation core which is a subgrain in deformation structure. The nuclei prefer to grow heterogeneously into surrounding high store energy deformation structures in a way of orientation pinning, such as grain boundaries and shear bands, due to the high growth driving force. The growing fronts of nuclei do not take an half spherical but a wedge shape. The wedge penetrates into the maximum store energy region, followed by the part behind growing sideward to coarsen to an equiaxed section.

#### 4:05 PM Tea Break

#### Symposium J: Materials Characterisation and Evaluation: Synchrotron and Other Advanced Technology

Tuesday PM August 3, 2010	Room: 1	Cairps Convention Centre
August 3, 2010	Location:	Cairns Convention Centre

Session Chairs: Julie Christodoulou, Office of Naval Research ; Klaus-Dieter Liss, ANSTO

#### 4:30 PM Keynote

**3D** Characterisation of Grain Deformation under Synchrotron Radiation: *Masakazu Kobayashi*<sup>1</sup>; Hiroyuki Toda<sup>1</sup>; Kentaro Uesugi<sup>2</sup>; Akihisa Takeuchi<sup>2</sup>; Yoshio Suzuki<sup>2</sup>; <sup>1</sup>Toyohashi University of Technology; <sup>2</sup>Japan Synchrotron Radiation Research Institute

Recently, three-dimensional (3D) observation and analysis have attracted considerable attention in materials science field. By using the synchrotron radiation, the tomography makes possible high-resolution 3D observation dynamically and the diffraction analysis is available for 3D orientation mapping. In this study, grain deformation behavior in polycrystalline aluminum alloy has been characterized by 3D observation method applying the synchrotron radiation. Inhomogeneous deformation in polycrystalline metals is important matter for structural use materials, because it affects the mechanical properties such as deformability, ductility, fracture, and so on. However the deformation process in polycrystalline metals that consists of many grains have not been explained completely in spite of the knowledge that the fundamental mechanism of deformation on crystalline metals is activation of slip systems. The consecutive and non-destructive observation by tomography and a test rig on a beam line brings us every moment dynamics. Especially, the method to measure inner strain distribution by means of microstructural features tracking provides strain distribution within the sample, which we could not access before. The effect of grain orientation and its interaction during tensile deformation was discussed with the obtained strain distribution

#### 4:50 PM Keynote

Materials Characterisation at the Australian Synchrotron: Ian Gentle<sup>1</sup>; <sup>1</sup>Australian Synchrotron

The Australian Synchrotron, located in Clayton Victoria, is Australia's largest scientific user facility with over 1000 external users. The synchrotron is a source of intense light, with energy ranging across the spectrum from infrared to hard X-rays. Opened in 2007 at a cost of over \$220M, there are currently 9 world class beamlines offering a wide range of experiments taking advantage of the unique properties of synchrotron light, including crystallography (proteins, small molecules and powders), spectroscopy (infrared, soft and hard X-rays), small angle scattering and a range of sophisticated imaging techniques such as fluorescence microscopy and tomography. Applications range across nearly all areas of the physical and life sciences. Access is freely available on a competitive merit basis, with travel and accommodation costs for Australian users paid by the facility. In this presentation I will describe the applications of current beamlines of the Australian Synchrotron for materials characterisation, and preview some of the important developments that are planned over the next few years.

#### 5:10 PM Invited

X-Ray Phase-Contrast Tomography for Quantitative Characterisation of Self-Healing Polymers: *Sheridan Mayo*<sup>1</sup>; Andrew Stevenson<sup>1</sup>; Stephen Wilkins<sup>1</sup>; Dachao Gao<sup>1</sup>; Steven Mookhoek<sup>2</sup>; Sam Meure<sup>1</sup>; Tony Hughes<sup>1</sup>; James Mardel<sup>1</sup>; <sup>1</sup>CSIRO; <sup>2</sup>Delft University of Technology

X-ray phase-contrast imaging and tomography add an additional dimension to conventional x-ray methods by exploiting the x-ray refraction in addition to absorption in image formation. This greatly enhances the visibility of edges, voids and boundaries within a sample. It also makes it possible to characterise weakly x-ray absorbing samples which would produce little or no contrast in conventional x-ray imaging. This talk will outline the application of phasecontrast imaging techniques to 2D and 3D materials characterisation, including the phase-retrieval methods required to treat this kind of data. These techniques have been successfully applied in a range of areas including analysis of self-healing polymers, visualisation of fluids in porous materials, and as the starting point for data-constrained modelling. Examples of materials characterisation applications using laboratory-based x-ray phase-contrast instruments will be given, along with a brief discussion of the wider range of phase-contrast techniques available at synchrotrons.



#### 5:25 PM

Deformation Mechanisms of Twinning Induced Plasticity Steels: In Situ Synchrotron Characterization and Modelling: Kun Yan1; David Carr1; Mark Callaghan2; Klaus-Dieter Liss1; Huijun Li2; 1ANSTO; 2University of Wollongong

It has been widely accepted that, due to its low stacking fault energy, twinning is an effective means of deformation in high manganese austenitic, so-called twinning induced plasticity steel, which has been supported by transmission electron microscopy and electron backscatter diffraction, showing that the volume fraction of twins increases concomitant with plastic deformation. Nevertheless, the combination of various conventional surface microstructure analysis methods are limited in provision of bulk information about the evolving dislocation density, bulk texture, local strain, stacking faults and the deformation mechanisms operating in the total volume of the material. In present study, twinning induced plasticity steels of composition Fe-25Mn-3Si-3Al are investigated by means of in-situ synchrotron high energy X-ray diffraction and compared to self-consistent simulations. It is the first time the alternating interaction of {111} <110> slip and {111} <112> twinning have been directly observed in-situ while undergoing uniaxial tension. The deformation texture is determined mainly by dislocation gliding, while deformation twinning impedes the reinforcement of texture.

#### 5.40 PM

Three Dimensional Microstructure Characterization of an Al-Zn-Mg Alloy Foam Using Synchrotron X-Ray Microtomography: Oiang Zhang<sup>1</sup>: Hirovuki Toda1; Masakazu Kobayashi1; Yoshio Suzuki2; Kentaro Uesugi2; 1Toyohashi University of Technology; <sup>2</sup>Japan Synchrotron Radiation Research Institute (JASRI)

Synchrotron X-ray microtomography (Spring-8, Japan) has been used for the microstructure characterization in an closed cell Al-Zn-Mg alloy foam. Some sophisticated microstructure features, such as micropores and intermetallic particles inside the cell wall, were visualized and quantified three dimensionally(3D) by the high-resolution phase contrast imaging technique. By microtomographies tuned to energies above and below the Zn K-absorption edge, the 3D quantitation of Zn distribution was obtained using subtraction imaging technique. It has been clarified that the Zn distribution was inhomogeneous in the cell wall of the foam. And the agglomeration of Zn-bearing particles was confirmed to induce the brittle fracture of cell wall. The 3D distribution of Ti and Ca in the foam can also be visualized by subtraction mechod. The current tomographic techniques provide novel solutions for the 3D microstructure analysis in the highly inhomogeneous foam materials.

#### 5:55 PM

Characterization of Cyclic-Loading Effects on Superalloys Using In-situ Neutron Diffraction and Thermal Measurements: E-Wen Huang1; Bjørn Clausen2; Peter Liaw3; 1National Central University; 2Los Alamos National Laboratory; 3University of Tennessee

Cyclic loading and the subsequent lattice-strain and temperature evolutions have been investigated with the in-situ neutron-diffraction and thermal characterization for a nickel-based superalloy. The lattice strain and thermal response to the applied load are investigated as a function of fatigue cycles. Cyclic-loading effects were observed with bulk hardening, softening, and eventual saturation evident in the diffraction patterns and the thermal-evolution features. An increase in the dislocation density is responsible for hardening during the first few cycles. The transition to saturation cycles is characterized by the anisotropy of the lattice-strain evolution. Moreover, the inhomogeneity of the thermal response and irreversible compression of the lattice planes were found in the final saturation fatigue cycles. The local fatigue damages are studied by the transmission-electron microscopy. The microscopy results are presented along with the in-situ measurements. The developments of irreversible cyclic-loading effects are discussed.

#### 6:10 PM

#### Advanced X-Ray Scattering Techniques for Multi-Length Scale Materials Analysis: John Daniels1; Diego Pontoni2; Veijo Honkimäki2; 1University of New South Wales; <sup>2</sup>European Synchrotron Radiation Facility

Combined small and wide angle x-ray scattering (SAXS/WAXS) is a very powerful technique for materials studies from the atomic to nanometer length scales. While combined scattering techniques are common at regular x-ray energies, when applied at high x-ray energies (E > 50keV), three important materials studies are possible; 1) in-situ strain analysis reveals anisotropic strain behaviour in the WAXS as well as the SAXS signals, 2) the possibility of collecting WAXS to very large q for pair distribution function analysis (SAXS/ PDF), and 3) the possibility of complex sample environments is opened due to the penetrating power of the high x-ray energies. We have applied this technique for the study of deformation mechanisms in biomaterial composites, and for the study of nano-particle solutions at the atomic and nano-meter length scales.

6:25 PM

High-throughput Evaluation of Crystallization Temperature of Pd-Cu-Si System Using Integrated Thin Film Samples: Yuko Aono<sup>1</sup>; Junpei Sakurai<sup>1</sup>; Akira Shimokohbe1; Seiichi Hata1; 1Tokyo Institute of Technology

Combinatorial method, which is simultaneous synthesis and evaluation of large arrays of different materials, is an efficient searching method for new alloys. However, high-throughput evaluation techniques have achieved evaluation of only a few properties because each synthesised sample is very small and thin, and all samples are integrated on one wafer which is called a thin film library. We have tried to realize a novel evaluation method for evaluation of crystallization temperature of thin film amorphous alloys on the thin film library. Crystallization is detected as an emissivity change by a thermography. The method needs only monitoring of the thin film library surface while increasing temperature. In this report, crystallization temperature of a Pd-Cu-Si compositional graded thin film library has been measured. In all 107 amorphous samples on the thin film library, crystallization temperature can be evaluated at once. There is obvious dependency of crystallization temperature on the Si at.%. The highest crystallization area agrees with a eutectic composition of Si-Pd phase diagram. Then, several compositions on the library were re-produced and their crystallization temperatures were measured by conventional differential scanning calorimeter. The differences in the crystallization temperature between conventional method and proposed one are within 10 K.

#### **Symposium K: Composites and Hybrid Materials:** Nanocomposites

Tuesday PM August 3, 2010 Room: 5 Location: Cairns Convention Centre

Session Chairs: Ken Goto, Japan Aerospace Exploration Agency; Richard Callinan, DSTO

#### 4:30 PM Keynote

Superhydrophobic/Superhydrophilic Switchable Wettability on Hybrid Nanostructured Surfaces: Kilwon Cho1; 1Pohang University of Science and Technology (POSTECH)

We fabricated superhydrophobic/superhydrophilic switchable smart surfaces by using hybrid nanostructured surfaces consisting of microporous polyelectrolyte multilayers, inorganic nanoparticles, and external stimuliresponsive materials. By combining layer-by-layer deposition of polyelectrolytes and inorganic nanoparticles and subsequent coating of photosensitive fluorinated azobenzene molecules, we can fabricate novel smart nanostructured surface with wettability that can be reversibly switched between superhydrophobicity and superhydrophilicity with UV/visble exposure. This approach can be used to make substrates with erasable and rewritable patterns of extreme wetting properties as a result of selective UV irradiation. In the second approach, polyelectrolyte brushes comprising quaternary ammonium groups were synthesized by surface-initiated atom transfer radical polymerization on a gold textured surface. The wettability of the polyelectrolyte-modified rough surface can be reversibly switched between superhydrophobicity and superhydrophilicity by selection of the appropriate counteranion.

#### 4:50 PM

Quantification of the Interface Interactions in Polymer Nanocomposites: Qinghua Zeng<sup>1</sup>; Wen Xu<sup>2</sup>; Aibing Yu<sup>2</sup>; Donald Paul<sup>3</sup>; <sup>1</sup>School of Engineering, University of Western Sydney, Penrith Campus; 2School of Materials Science and Engineering, The University of New South Wales; 3Department of Chemical Engineering, The University of Texas at Austin

Interfaces are important for many properties and applications of multiphase materials. This is particular true for particle-reinforced polymer composites, where the interfacial characteristics between particle and polymer play a crucial role in load transfer and mechanical properties. In polymer nanocomposites, the adhesion strength between particle and polymer matrix is a major factor in determining their mechanical properties. In this work, we present our recent study towards the quantification of the interaction strength at the interface of clay-based polymer nanocomposites by molecular dynamics simulation.



#### 5:05 PM

Friction and Wear Behaviors of Solid Lubricants/Polyimide Composites in Liquid Mediums: Mu Liwen<sup>1</sup>; *Feng Xin*<sup>1</sup>; Shi Yijun<sup>2</sup>; Wang Huaiyuan<sup>3</sup>; Lu Xiaohua<sup>1</sup>; <sup>1</sup>Nanjing University of Technology; <sup>2</sup>Nanjing Forestry University; <sup>3</sup>Daqing Petroleum Institute

In the petroleum and chemical industry, where water, alkali and other media are often dispatched, special materials have to be used. Polyimide (PI) has excellent mechanical properties and good chemical resistance, which has been used in aerospace, automobile and microelectronics industry. However, high friction coefficient and wear rate of pure PI limit its wider use in friction systems. The solid lubricants with a layered crystal structure can shear easily to provide low friction and to prevent wear damage between the sliding surfaces. The tribological properties of PI composites reinforced with graphite or MoS, sliding in alkali, dry, oil and water lubricating situations were investigated on a ring-on-ring tester. It was found that the relationship of friction coefficient and wear rate of PI composites filled with graphite or MoS, were  $\mu(dry) > \mu(water) > \mu(oil) > \mu(alkali)$ and W(water)>W(dry)> W(oil) >W(alkali). Results also showed that addition of graphite was more effective than MoS2 in enhancing in the friction and wear resistance of PI composites sliding in different liquid medium. The worn surfaces and transfer film of PI composites were then examined with scanning electron microscope (SEM). The transfers onto the counterpart rings were significantly hindered when they were sliding in liquid medium.

#### 5:20 PM

**Fabrication of Magnetic Fe<sub>3</sub>O<sub>4</sub> and Mesoporous Silica Core-Shell Microspheres**: *Jian Liu*<sup>1</sup>; Zhi Gang Chen<sup>1</sup>; Frances Stahr<sup>1</sup>; Sandy Budi Hatono<sup>1</sup>; Max Lu<sup>1</sup>; Shi Zhang Qiao<sup>1</sup>; <sup>1</sup>The University of Queensland

We present the controlled synthesis of sandwich structured mesoporous magnetic silica microspheres (MMSMs) and the influence of silica coating on the magnetic properties of formed particles. The MMSMs composed of magnetic Fe<sub>3</sub>O<sub>4</sub> cores and silica shells with perpendicularly oriented channels have been prepared through a surfactant-templating method combined with a sonochemical treatment. All materials were characterised by XRD, TEM, nitrogen sorption and magnetic susceptibility measurements. The obtained microspheres have high magnetization values (20~50 emu/g), uniform accessible mesochannels, high surface area (300~900 m<sup>2</sup>/g), and large pore volume (0.2~0.5 cm<sup>3</sup>/g). They can be well-dispersible in water. A good control of the silica shell thickness (60~350 nm) and pore size (1.7~4.5 nm) on the shell has been achieved by adjusting the silane concentration, changing the surfactant or adding 1,3,5-trimethylbenzene (TMB) as additives, respectively. Because of the useful magnetic properties, unique structure and well developed porous shell, the microspheres can provide great promise for many applications such as magnetically controlled drug delivery, water treatment, catalysis, and so on.

#### 5:35 PM

Form Factor for the Design of Pultruded Structural Members Under Compression: *Jin-Woo Choi*<sup>1</sup>; Seungsik Lee<sup>2</sup>; Hyung-Joong Joo<sup>1</sup>; Young-Jong Sim<sup>3</sup>; Soon-Jong Yoon<sup>1</sup>; <sup>1</sup>Hongik University; <sup>2</sup>Korea Institute of Marine Science & Technology Promotion; <sup>3</sup>Korea Land & Housing Co.

As a new construction material, fiber reinforced polymeric plastic structural shapes are readily available. Therefore, construction and structure rehabilitation using FRP materials is ever increasing trend because of its superior chemical and mechanical properties compared with those of conventional construction materials such as steel and concrete. Pultruded fiber reinforced polymeric plastic structural members are most popular for the civil engineering applications and they are made of fiber reinforced polymer resin system. So it has relatively low modulus of elasticity and also cross-section of structural shapes is composed of plate components such as flange and web. Therefore, stability is an important issue in the design of pultruded structural shapes. For the design of pultruded structural member under compression, buckling and post-buckling strengths of plate components must be taken into account. In the structural steel design following AISC/ LRFD, this effect, in addition to the buckling strength, is incorporated with a form factor. In this research, form factor for the design of pultruded structural shapes under compression is investigated by experiment. Based on the experimental results, form factor for the design of pultruded structural shapes have been suggested.

#### 5:50 PM

High Strength and Lead-Free Machinable Brass by Powder Metallurgy Process: *Hisashi Imai*<sup>1</sup>; Shufeng Li<sup>1</sup>; Yoshiharu Kosaka<sup>2</sup>; Akimichi Kojima<sup>2</sup>; Haruhiko Atsumi<sup>3</sup>; Katsuyoshi Kondoh<sup>1</sup>; <sup>1</sup>Joining and Welding Research Institute Osaka University; <sup>2</sup>San-Etsu Metals Co., Ltd.; <sup>3</sup>Osaka University

The aim of this paper was to produce high strength and lead-free machinable brass, contented with chromium for strengthening, and dispersed with graphite particles for machinability. Cu-40mass%Zn alloy powder containing 1.0 mass% Cr was prepared by the water atomization. Graphite particles, having a mean

particle size of 5  $\mu$ m, were added to the as-atomized powders by the ball milling machine for 14.4h under 120 rpm. Spark plasma sintering process was used to consolidate the above elemental mixed powders (sintering material). Sintering material was heat-treated for the precipitation of much Cr (HT material). The machinability of Cu-40Zn brass alloys was evaluated by a drilling test using a drill tool under dry conditions. The matrix hardness of sintering material was higher than that of HT material. On the other hand, the machinability of sintering material was higher than that of HT material. There is no trade-off relationship in the matrix hardness and machinability. From SEM-EDS observation, Cr quantity dissolved in the brass matrix of sintering material and HT material was 0.42 mass% and 0.19 mass%, respectively. As the reason why machinability of HT material lowered, the precipitation of the hard Cr particle or generation of Cr-C compound seemed to inhibit the machinability.

#### 6:05 PM

## **Performance of Aluminium** /**Vinylester Particulate Composite**: *Jayantha Epaarachchi*<sup>1</sup>; <sup>1</sup>University of Southern Queensland

Particulate materials and powder fillers are becoming more popular in composite manufacturing as the weight of the massive composite structures are being reduced substantially while maintaining the required material properties, by using fillers. Further the particulate composite materials are convenient for manufacturing of complex parts. As such, performances of Aluminium/Vinylester composites were studied in detail in order to investigate its suitability for engineering applications. This study examined the suitability of atomised aluminium particles for particulate reinforcement of a vinyl ester resin. Mechanical properties were obtained for the composites of various percentages of Aluminium powder (75-150 um) and Vinvlester resin. It has been found that the inclusion of Al powder has not changed the properties of vinylester resin considerably whilst improving the ductility of the composite. The optimal performances were exhibited by 15% Al composition. The properties of the particulate composites were modelled using a few empirical models. Unfortunately a significant difference was found between experimental and predicted properties of the Al/Vinylester particulate composite. This paper intends to details the variation of mechanical properties with the change of Al volume in the composite and the performances of empirical models in prediction of the properties of particulate composites.

#### Symposium L: Energy Generation, Harvesting and Storage Materials: Hydrogen Storage II

Tuesday PM August 3, 2010 Room: 7 Location: Cairns Convention Centre

Session Chairs: Min Zhu, South China University of Technology; Ying Wu, China Iron and Steel Research Institute Group

#### 2:00 PM Keynote

#### Complex Hydrides as Advanced Energy Storage Materials: Shin-ichi Orimo<sup>1</sup>; <sup>1</sup>IMR, Tohoku University

Complex hydrides with the (BH<sub>4</sub>)- anion are expressed as M(BH<sub>4</sub>)n (n: valence of metal M), which shows ionic bonding between the Mn+ cation and the (BH<sub>4</sub>)- anion. These hydrides have been attracting great interest as potential candidates for advanced hydrogen storage materials because of their high hydrogen densities. In addition, one of the complex hydrides, Li(BH<sub>4</sub>), exhibits another novel material property, that is, lithium fast-ion conduction (more than 1×10<sup>-3</sup> S/cm over 390 K). Moreover, we report the discovery of novel lithium fast-ion conductors of complex hydrides consisting combinations of the (BH<sub>4</sub>)– and (NH<sub>2</sub>)– anions, for example, Li<sub>2</sub>(BH<sub>4</sub>)(NH<sub>2</sub>) (2×10<sup>-4</sup> S/cm at RT, which is more than four orders of magnitude higher than that of Li(BH<sub>4</sub>) and Li(NH<sub>2</sub>)). Some of the complex hydrides also for solid-electrolytes.

#### 2:20 PM

Visible-Light Water-Splitting Performance of TiO<sub>2</sub> Nanotube-Arrays Using Co-Catalysts of WO<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub>, and BiVO<sub>4</sub>: *Su-min Son*<sup>1</sup>; Se-Im Kim<sup>1</sup>; Seuing-uk Lee<sup>1</sup>; Ji-hun Hwang<sup>1</sup>; Bee Lyong Yang<sup>1</sup>; <sup>-1</sup>Kumoh National Institute of Technology

We report photocatalytic properties for composites of  $\text{TiO}_2$  nanotube/metal oxides(WO<sub>3</sub> and BiVO<sub>4</sub>) to address issues of improving the efficiency of watersplitting under visible-light irradiation. TiO<sub>2</sub> nanotube arrays with ~2µm length and ~180nm diameter were grown by anodizing Ti foils using electrolytes based on DMSO (dimethyl sulfoxide). The amorphous samples after the anodization were annealed to crystallize at 550°C in oxygen ambient for 4hr. Then after dipping TiO, nanotube arrays into metal oxide liquid solutions such as V-acetylacetonate,

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 $Cr(NO_3)_3H_2O$ ,  $H_2WO_4$  and  $Bi(NO_3)_2H_2O$ , they were annealed in  $O_2$  ambient as a function of different times and temperatures. Results of nano-structural analysis by FE-SEM, XRD and TEM for these samples show that metal oxides nano-particles of ~10nm diameter formed stably on the surface of TiO<sub>2</sub> nanotubes. Effects of the co-catalysts for TiO<sub>2</sub> nanotubes were investigated by photo-current and GC measurements under visible light and the results will be presented in terms of photo-conversion efficiency for water-splitting.

#### 2:35 PM

Microscopic Reaction Mechanism of Hydrogen Storage Materials by TEM: Shigehito Isobe<sup>1</sup>; Yongming Wang<sup>1</sup>; Akifumi Ono<sup>1</sup>; Hiroko Hirasawa<sup>1</sup>; Hao Yao<sup>1</sup>; Naoyuki Hashimoto<sup>1</sup>; Somei Ohnuki<sup>1</sup>; Takayuki Ichikawa<sup>2</sup>; Yoshitsugu Kojima<sup>2</sup>; <sup>1</sup>Hokkaido University; <sup>2</sup>Hiroshima University

For utilizing hydrogen as one of the secondary energies, it is necessary to establish high performance hydrogen storage technologies. Three hydrogen storage ways of liquid hydrogen, high-pressure gas hydrogen and absorbed hydrogen in hydrogen storage materials are considered as hydrogen storage tanks for fuel cell vehicles. Among them, hydrogen storage materials can store more hydrogen than highnpressure gas or liquid hydrogen. We have studied on microscopic reaction mechanism of hydrogen storage materials such as  $MgH_2$ ,  $MH-NH_3$ ,  $NH_3BH_3$ , and  $NAAIH_4$  by means of transmission electron microscope (TEM) with controlling temperature and gas atmosphere. By using a high voltage electron microscope (HVEM), we can observe lattice image of these materials during the reaction. For example, we have observed the hydrogen desorption process of  $MgH_2$  with  $Nb_2O_5$  catalyst at the temperature range from R.T. to  $250^{\circ}C$ . The decomposition of  $MgH_2$  occurred at the boundary between  $MgH_2$  and catalyst of  $Nb_2O_5$ . With increase of temperature, the phase of Mg is growing.

#### 2:50 PM Invited

#### Ammonia Borane for Chemical Hydrogen Storage: Effect of Nano-Confinement: Xiangdong Yao<sup>1</sup>; <sup>1</sup>Griffith University

Recently, many efforts have been devoted to chemical hydrogen storage by ammonia borane (AB), NH<sub>3</sub>BH<sub>3</sub>, due to the release of irreversibility by off-board regeneration from the U.S. Department of Energy (DOE). Ammonia borane is significantly beneficial from its extremely high hydrogen content (theoretically 19.5wt%) and moderate decomposition temperature that may satisfy the requirement of hydrogen storage for proton exchange membrane (PEM) fuel cells. However, the slow kinetics of AB decomposition to release hydrogen hinders its practical application. Many strategies have been developed in order to improve the kinetics of AB decomposition. Confined AB in nanopores of a framework is one of the effective ways to significantly enhance the hydrogen release rate from AB. There are a few publications but systematic investigations are highly desirable. Here, we will present the current research outcomes from our group in this specific topic and try to understand, to some extent, the insight of nano-confinement effect on the kinetics of AB decomposition.

#### 3:05 PM

Microscopic Observation on Hydrogen Storage Materials of Sc-M-B-H System (M = Mg, Ca) by TEM: *Ryota Inazuki*<sup>1</sup>; Shigehito Isobe<sup>1</sup>; Yongming Wang<sup>1</sup>; Naoyuki Hashimoto<sup>1</sup>; Somei Ohnuki<sup>1</sup>; Keiji Shimoda<sup>2</sup>; Tessui Nakagawa<sup>2</sup>; Takayuki Ichikawa<sup>2</sup>; Yoshitugu Kojima<sup>2</sup>; <sup>1</sup>Hokkaido University; <sup>2</sup>Hiroshima University

In this study, we investigated the microstructural properties of Sc-M-B-H system (M = Mg, Ca), which are regarded as promising hydrogen storage materials due to their high gravimetric and volumetric hydrogen densities, by using a Transmission Electron Microscopy (TEM: JEOL2010: 200keV) and a High Voltage Electron Microscopy (HVEM: JEM-ARM1300: 1250keV). Samples were prepared by performing ball-mill to the mixtures of 2ScH,+3MgB, and 2ScH<sub>2</sub>+CaB<sub>4</sub>, which were as starting materials. The condition of ball-mill was at room temperature for 80 hours under 6.0 MPa of hydrogen atmosphere. We expected generation of borohydrides such as Mg(BH4)2 and Ca(BH4)2 by ball-mill. As results of TEM and HVEM, we could observe borohydrides and ScB, as the reaction products. According to obtained high resolution image, the lattice image of  $Mg(BH_4)_2$  with around 10 nm of average crystalline size was observed. This result indicates that Mg(BH<sub>4</sub>)<sub>2</sub> was produced by using ball-mill for hydrogenation of 2ScH<sub>2</sub>+3MgB<sub>2</sub>. In the case of Ca system, we could not observe  $Ca(BH_{A})_{2}$  in the high resolution image. We can suggest two possibilities to explain this reason. One is that Ca(BH<sub>4</sub>)<sub>2</sub> could not be generated by ball-mill. The other is Ca(BH<sub>4</sub>), could be immediately decomposed by electron irradiation.

#### 3:20 PM

Visualization of Diffusive Hydrogen in Some Metallic Materials: *Takahito Watakabe*<sup>1</sup>; Goroh Itoh<sup>1</sup>; Tomohiro Tsutsumi<sup>1</sup>; Nobuhide Itoh<sup>1</sup>; <sup>1</sup>Ibaraki University

In recent years, the use of hydrogen as a clean energy has been paid attention to in terms of the prevention of global warming. Tanks composed solely of steel and cylinders consisting of aluminum liner reinforced with C-FRP in the surrounding are used to store high-pressure hydrogen gas in hydrogen stations and in fuel cell vehicles, respectively. On the other hand, hydrogen embrittlement has been known to occur in some metallic materials under several certain conditions. Also, it has been generally known that the environmental hydrogen that invades the material during services plays major role in hydrogen embrittlement. For this reason, investigations on the behavior of environmental hydrogen in metallic materials are needed. In this study, close attention has been paid to what is called diffusive hydrogen atoms that have been known to affect directly to the embrittlement; hydrogen atoms has been considered to diffuse from newly created metallic surface to the front of crack tip. The behavior of hydrogen in cathodically electric-charged chromium-molybdenum steel plate and pure aluminum plate has been investigated by visualizing the hydrogen atoms with respect to the microstructure, by means of hydrogen microprint and tritium autoradiography techniques.

#### 3:35 PM

Resistance to Hydrogen Embrittlement and Behavior of Hydrogen in 6000 Series Aluminum Alloys: Goroh Itoh<sup>1</sup>; *Masataka Watanabe*<sup>2</sup>; Kazuya Kuroyanagi<sup>2</sup>; Zhao Pizhi<sup>3</sup>; <sup>1</sup>Ibaraki University; <sup>2</sup>School of Science and Engineering, Ibaraki University; <sup>3</sup>Nippon Light Metal Co., Ltd

In recent years, the fossil fuel exhaustion and global warming have become serious problems. As a way to solve these problems, the development of the fuel cell vehicles has been paid attention to. In the fuel cell vehicles, highpressure hydrogen gas is stored in a container consisting of aluminum liner and surrounding fiber-reinforced plastic layer. To increase the mileage per filling, an aluminum alloy having higher strength than the currently used 6061 alloy is needed. It has been known that increase in the Si content in 6000 series aluminum alloy brings about increase in the strength. In this study, a 6061 aluminum alloy with relatively high Si content (6061HS), 6061 alloy with a typical composition and other 6000 series alloys with high Si content have been subjected to slow strain rate technique (SSRT) tensile tests in a moist air to assess the resistance to hydrogen embrittlement, and also to thermal desorption spectroscopy (TDS) to investigate the hydrogen behavior. The results obtained have been discussed in terms of the effect of Si content. Moreover, to investigate the effect of grain size, 6061HS alloy sheets with different grain size have been also subjected to the SSRT tensile tests and TDS

3:50 PM Tea Break

#### Symposium L: Energy Generation, Harvesting and Storage Materials: Other Functional Materials

Tuesday PM	Room: 7	
August 3, 2010	Location:	Cairns Convention Centre

Session Chairs: Shin-ichi Orimo, IMR, Tohoku University; San Ping Jiang, Nanyang Technological University

#### 4:30 PM Keynote

Superior Radiation Resistance of ODS Ferritic Steels: *Ryuta Kasada*<sup>1</sup>; Hirotatsu Kishimoto<sup>2</sup>; Hiromasa Takahashi<sup>1</sup>; Kentaro Yutani<sup>1</sup>; Jaehoon Lee<sup>1</sup>; Akihiko Kimura<sup>1</sup>; <sup>1</sup>Kyoto University; <sup>2</sup>Muroran Institute of Technology

Oxide dispersion strengthened (ODS) ferritic steels are expected to be used for the first-wall component in the fusion reactors as well as the fuel pin cladding in the Generation IV nuclear fission systems. Our previous studies revealed that the high-Cr ODS ferritic steels have excellent resistance to void swelling, irradiation hardening, and change in morphology of nano-oxide particles. Including the previous studies, dual-beam ion irradiation techniques have been stated as a powerful tool to understand the irradiation effects on the material properties of fusion reactor materials with simultaneous helium injections. The present paper unveils the potential of the ODS ferritic steel as well as non-ODS reduced-activation ferritic (RAF) steel for swelling resistance up to high dose irradiation with helium injection at elevated temperature by using DuET facility at Kyoto University. The obtained results can be concluded as follows: 1) after a high dose irradiation of 60 dpa with 900 appm He injection at 500 °C, the swelling value of ODS ferritic steels was significantly lower than that of RAF steels. 2) The nano-size oxide particles dispersed in the matrix of ODS ferritic steels probably act as trapping site for the implanted helium to suppress swelling.



#### 4:50 PM

**Manostructured and Nano-Architectural MnO<sub>2</sub> Supercapacitors**: H. Xia<sup>1</sup>; W. Xiao<sup>1</sup>; *Li Lu*<sup>1</sup>; <sup>1</sup>National University of Singapore

Supercapacitors are alternative energy storage systems which may provide high power density and long cyclic life. One of key factors in controlling capacitance of supercapacitors is surface area of electrodes. In this research several methods have been used in the fabrication of nanostructured  $MnO_2$ films, including electrodepositions, composites prepared using hydrothermal growth, and lithiation of  $MnO_2$  thin films. It is found that the morphologies of the nanostructure materials and composites, and hence surface areas are dependent on deposition current density, temperature and concentration of solvent, and lithiation cycles. Although carbon nanoture (CNT) shows the best capacitance shape, CNT-MnO<sub>2</sub> composite possesses high capacitance due to contributions from high conductivity of CNT and from high capacitance of  $MnO_2$ . This research also provides an evidence of formation of nanostructured and porous  $MnO_2$ , through lithiation of  $MnO_2$  thin film.

#### 5:05 PM

**Broad-Band Vibro-Impacting Power Harvester**: *Scott Moss*<sup>1</sup>; Ian Powlesland<sup>1</sup>; Michael Konak<sup>1</sup>; Alex Barry<sup>1</sup>; Steve Galea<sup>1</sup>; Gregory Carman<sup>2</sup>; <sup>1</sup>Defence Science and Technology Organization; <sup>2</sup>UCLA Active Materials Laboratory

The certification of retro-fitted structural health monitoring (SHM) systems for use on aircraft raises a number of challenges. One critical issue is determining the optimal means of supplying power to these systems, given that access to the existing aircraft power-system is unlikely to be permitted. Other conventional options such as primary cells can be difficult to certify and would need periodic replacement, which in an aircraft context would pose a serious maintenance issue. Previously, the DSTO has shown that a structural-strain based energy harvesting approach can be used to power a device for SHM of aircraft structure. Acceleration-based power harvesting from airframes is more demanding (than a strain based approach) since the vibration spectrum of an aircraft structure varies dynamically with flight conditions, and hence a frequency agile or (relatively) broadband device is often required to maximize the energy harvested. This paper reports on the development of a prototype vibro-impacting power harvester with a ~59 gram flying mass and two piezoelectric bimorph-stops. The harvester delivers an average of ~7.3 mW from a 400 milli-g sinusoidal excitation, over the frequency range 29-41 Hz.

#### 5:20 PM

Effect of Flow Fields and Scan Rates on Performance of Micro-DMFC: Y. Lu<sup>1</sup>; *Ramana Reddy*<sup>1</sup>; <sup>1</sup>The University of Alabama

Effect of bipolar plate floe fields on performance of micro-direct methanol fuel cells (micro-DMFCs) was experimentally investigated. Designing, manufacturing, and testing of micro-fuel cells were studied. Compared with conventional machining technology and rapid prototyping (RP) technology, microelectromechanical system (MEMS) technology was identified as a suitable method to fabricate the bipolar plates (BPs) with channels of micro-channel for the micro-DMFCs. The effect of design flow fields on performance of micro-DMFCs was electrochemically evaluated using four types flow fields. The micro-DMFCs with double-channel serpentine presented the highest maximum power density and the micro-fuel cells with mixed multi-channel serpentine with narrow channels had the lowest maximum power density.

#### 5:35 PM

Chemical Property of Ground Calcium Carbonate as Carbon Dioxide Absorbent: *Seiji Yokoyama*<sup>1</sup>; Muhd Nor Nik Hisyamudin<sup>2</sup>; Shunsuke Hirano<sup>3</sup>; <sup>1</sup>Toyohashi University of Technology; <sup>2</sup>University of Tun Hussein Onn Malaysia; <sup>3</sup>Graduate School of Toyohashi University of Technology

Carbon dioxide is well-known for one of the green house gases, and the separation and fixing of it from exhausted gases has been required. The fundamental research to use mechanically activated calcium carbonate as an adsorbent of carbon dioxide was performed in this study. The precipitated calcium carbonate was ground by a vibration rod mill. The crystal structure of the calcium carbonate was changed from calcite to aragonite by the grinding. Generally speaking, the decomposition pressure of carbon dioxide for the ground calcium carbonate at high temperature increased with the increase in the grinding time. The grinding decreased the standard enthalpy and entropy change of the thermal decomposition reaction. From the view point of kinetics, the grinding did not influence both the rate of decomposition of calcium carbonate and the rate of synthesis of calcium carbonate after decomposition. The adsorption of carbon dioxide on the ground calcium carbonate and the desorption of it occurred even at the room temperature. The maximum amount of adsorbed carbon dioxide per the volume of the ground calcium carbonate was seven tenth of that of a molecular sieve carbon.

#### 5:50 PM

Microstructural Analysis of Ball-Milled Composites of Nano-Structural Graphite and Alkali (-Earth) Metal: *Sumito Yamada*<sup>1</sup>; Yoshitsugu Kojima<sup>2</sup>; Soumei Ohnuki<sup>1</sup>; Shigehito Isobe<sup>1</sup>; Yongming Wang<sup>1</sup>; Naoyoki Hashimoto<sup>1</sup>; Toshiyuki Yamanaka<sup>2</sup>; Takayuki Ichikawa<sup>2</sup>; <sup>1</sup>Hokkaido University; <sup>2</sup>Hiroshima University

Li-C-H system, which can store about 5.0 mass% of rechargeable  $H_2$ , has been reported as promising hydrogen storage system by T. Ichikawa et al. This system was investigated from the thermodynamic and structural point of view. However, hydrogen absorption/desorption mechanism and the state of hydrogen atoms absorbed in the composite have not been clarified yet. In order to find new or better hydrogen storage system, graphite powder and nano-structural graphite ball-milled under  $H_2$  and Ar atmosphere were prepared and milled with Li and Mg under Ar atmosphere in this study. Microstructural analysis for those samples by TEM revealed that LiC<sub>6</sub> and/or LiC<sub>12</sub> were formed in Li-C-H system. On the other hand, MgC<sub>2</sub> was found in Mg-C-H system ball-milled under H<sub>2</sub> atmosphere, but not in the system ball-milled under Ar atmosphere. These results indicated that nano-structure in composites of nano-structural graphite is different from that of alkali (-earth) metal. For these reason, metal-C-H system can be recognized to be a new family of hydrogen storage materials.

#### 6:05 PM

## **Storage of CO<sub>2</sub> in Low Al<sub>2</sub>O<sub>3</sub> EAF Oxidizing Slag by Grinding with Vibration Mill**: *Nik Hisyamudin Muhd Nor*<sup>1</sup>; Yokoyama Seiji<sup>2</sup>; Umemoto Minoru<sup>2</sup>; <sup>1</sup>University Tun Hussein Onn Malaysia; <sup>2</sup>Toyohashi University of Technology

This research attempts to investigate the feasibility and the capacity of  $CO_2$  storage in the low  $Al_2O_3$  EAF oxidizing slag under wet grinding. Interest in using the slag as storage of  $CO_2$  has arisen because it is readily available and most often produced near the emision source of  $CO_2$ . The slag was wet ground in the vibration ball mill in the presence of  $CO_2$  at room temperature. The amount of  $CO_2$  absorption increased with increasing volume of water, weight of slag and initial pressure of  $CO_2$ . However, it did not increase further when too much water and slag was used. The conversion ratio of CaO to CaCO<sub>3</sub> increased when the volume of water and  $CO_2$  pressure increased, but decreased when the weight of slag increased. The  $CO_2$  had been absorbed with high capacity and high conversion ratio in the slag in comparison with that in the high  $Al_2O_3$  EAF oxidizing slag. Because of this apparatus operated by electrical power,  $CO_2$  was emitted from a power plant during the operation of the machine. The amount of  $CO_2$  absorption by this apparatus was higher than the amount of  $CO_2$  emission which was exhausted from hydraulic, thermal, atomic, wind and solar power plant.

#### 6:20 PM

**Rare Earth Element Aliovalent Doping Substitution and Electrochemical Performance of LiFe**<sub>1-x</sub>Nd<sub>x</sub>PO<sub>4</sub>: *Minshou Zhao*<sup>1</sup>; Li Zhang<sup>1</sup>; <sup>1</sup>Changchun Institute of Applied Chemistry, Chinese Academy of Sciences

LiFe<sub>1.v</sub>Nd<sub>v</sub>PO<sub>4</sub>/C are synthesized by two-step heating solid-state reaction. The structure and electrochemical performance of the sample are studied by XRD, FE-SEM, EDS, EIS and charge-discharge method. The results show that LiFe1. Nd, PO, /C sample has the same olivine structure as LiFePO, and a small amount of aliovalent Nd<sup>3+</sup>-doping substitution on Fe<sup>+2</sup> can effectively reduce the particle size of LiFePO<sub>4</sub>, and with the accession of glucose and Nd<sup>3+</sup>-ion, the charge transfer resistance decreases. When the dopant of Nd3+ ion is 6% (mole) and the precursor is calcined at 700°C for 16 h, the sample delivers the highest discharge capacity of 165.2 mAh•g-1 at 0.2 C rate, and the capacity retention rate is 92.8 % after 100 cycles, and 146.8, 125.7, 114.8 mAh·g<sup>-1</sup> at 1, 2, 5 C rates, respectively. The measured theoretical capacity is about 168.6 mAh · g<sup>-1</sup>. With Si element as internal standard correction, XRD patterns of LiFe<sub>1-x</sub>Nd<sub>x</sub>PO<sub>4</sub> are carried out, and the cell parameters of LiFe<sub>1.</sub>Nd<sub>v</sub>PO<sub>4</sub> have little change. XRD patterns of LiFe<sub>1</sub>  $_{x}M_{x}$  PO<sub>4</sub> (M = La, Ce, Pr) are performed also, and no any new peak is observed. The all results imply that rare earth element aliovalent doping substitution on Fe site is tolerant.



#### Symposium A: Advanced Steels and Processing: Low-Carbon/Pipeline Steels/TMP

Thursday AM August 5, 2010 Room: A Location: Cairns Convention Centre

Session Chairs: Mayumi Ojima, National Institute for Materials Science; Yonghua Rong, Shanghai Jiao Ttong University

#### 8:30 AM Keynote

**Radiation Tolerant Nanostructured Ferritic Alloys**: *Michael Miller*<sup>1</sup>; <sup>1</sup>ORNL The high temperature and irradiation response of a new class of nanostructural ferritic alloys have been investigated by atom probe tomography. These materials are candidate materials for use in the extreme environments that will be present in the next generation of power generating systems. Atom probe tomography has revealed that the yttria powder is forced into solid solution during the mechanical alloying process. Subsequently during the extrusion process, 2-nm-diameter Ti-, Y- and O-enriched nanoclusters are formed. These nanoclusters have been shown to be remarkably stable during isothermal annealing treatments up to 0.92 of the melting temperature and during extended high temperature creep. No significant difference in sizes, compositions and number densities of the nanoclusters was also observed between the unirradiated condition and material neutron irradiated to a dose of 3 displacements per atom.

#### 8:50 AM

**Crystallography of Ferrite Nucleation at Austenite Grain Boundary in Low Carbon Steels**: *Tadashi Furuhara*<sup>1</sup>; Hayato Saito<sup>2</sup>; Naoki Takayama<sup>1</sup>; Goro Miyamoto<sup>1</sup>; <sup>1</sup>Institute for Materials Research, Tohoku University; <sup>2</sup>JFE Steel Corporation

Proeutectoid ferrite preferentially nucleates at austenite grain boundary in low carbon steels. Crystallography of ferrite nucleation plays an important role in grain refining of ferrite structures. In the present study, variation in orientation relationships of grain boundary ferrite against its austenite matrices with transformation temperature has been examined. In general, grain boundary ferrite holds approximately the Kurdjumov-Sachs (K-S) orientation relationship with respect to at least one of the adjacent austenite grains in the isothermally transformed Fe-1.5Mn-0.2C alloy. At a higher transformation temperature, i.e., with smaller undercooling, a more fraction of ferrites has near-rational orientation relationship with respect to the opposite austenite grain. As the undercooling is increased by lowering the transformation temperature, the deviation of orientation relationship from the K-S relationship becomes smaller against the near K-S related austenite matrix whereas the deviation against the irrationally oriented austenite matrix becomes smaller. There is strong variant selection by the austenite grain boundary for ferrite nucleation even in the bainite transformation range. Alloy carbide precipitation at the austenite grain boundary has suppressed development of Widmanstatten morphology for grain boundary ferrite by slightly increasing the deviation of orientation relationship from the K-S relationship.

#### 9:05 AM

#### **New Insights into Intragranular Ferrite in Low-Carbon Low-Alloy Steels:** *Kaiming Wu*<sup>1</sup>; Gang Huang<sup>1</sup>; <sup>1</sup>Wuhan University of Science and Technology

Single and multiple nucleation events on non-metallic inclusions have been observed leading to the intragranular formation of ferrite as a function of transformation temperature. Three-dimensional shapes have also been characterized. When the ferrite forms at elevated temperatures it is in the form of isolated idiomorphs, whereas larger undercoolings are associated with the multiple nucleation of plates emanating from the inclusions. The plates grow with a fixed orientation with the parent austenite. Nanohardness tests indicate that the idiomorphs are softer when compared with the plates. The formation of intragranular ferrite laths or plates can facilitate the attainment of fine-grained microstructures.

#### 9:20 AM

#### Correlation between Deformed Austenite Grains and Subsequent Transformed Microstructure in X80 Linepipe Steel: Sha Qingyun<sup>1</sup>; <sup>1</sup>Ansteel

Correlation between deformed austenite and subsequent transformed microstructure in X80 linepipe steel has been investigated with industry hot strip. The pancaked austenite grains were clearly revealed after etching with picric solution and then transformed microstructure inside the austenite grains were also observed after modified etching. The observations demonstrate that acicular ferrite (AF) are mainly formed inside deformed austenite grain, but some AF grains growing form one side to other side of deformed austenite grain boundaries were also found when austenite grains were subject to larger deformation leading

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to a smaller short axis of deformed austenite grain. Most granular bainite ferrite (GB) grains form along with deformed austenite grain boundaries and extend to center of austenite grain. M/A constituents were dispersed among matrix showing no direct relationship with austenite grain boundaries. Contrary to AF and GB formed in deformed austenite grain, a small amount of fine polygonal ferrite (PF) grains were observed on deformed austenite grain boundaries.

#### 9:35 AM

#### Effect of Austenitizing Temperature and Cooling Rate on Boron Distribution in Low Carbon Steels: *Yang Mo Koo*<sup>1</sup>; Dong Jun Mun<sup>1</sup>; Jae Sang Lee<sup>1</sup>; <sup>1</sup>Pohang University of Science and Technology

It is well known that the addition of a small amount of boron remarkably increases the hardenability of low alloy steels. This beneficial effect is attributed to the grain boundary segregation of boron, which retards the transformation of austenite to ferrite by reducing the grain boundary energy. In order to maximize the effect of boron in steels, it is necessary to control segregation and precipitation, which usually depends on alloying elements(N, Ti, Mo) and various processing parameters such as austenitizing temperature, cooling rate, heat treating temperature, and so on. In this study, characteristics of boron distribution with variation of cooling rate and austenitizing temperature were investigated in low carbon steels. The segregation and precipitation behavior of boron in austenite phase was mainly studied by means of Neutron Autoradiography method. The effect of boron on transformation of low carbon steels was also investigated by means of dilatometry and microstructural observations.From those results, the relationship between boron distribution and boron effect was discussed.

#### 9:50 AM

Stress Dependence of Microstructural Evolution in Heat Resistant Steels during Creep: Yoshinori Murata<sup>1</sup>; Yoshihiro Saito<sup>1</sup>; Yuhki Tsukada<sup>1</sup>; Toshiyuki Koyama<sup>2</sup>; Masahiko Morinaga<sup>1</sup>; Yasutoshi Sasaki<sup>1</sup>; Yasushi Hasegawa<sup>3</sup>; <sup>1</sup>Nagoya University; <sup>2</sup>National Institute for Materials Science; <sup>3</sup>Nippon Steel

Microstructural evolution in heat resistant steels is known to be affected by the existence of stress at high temperatures. The state of the microstructure can be evaluated quantitatively by the free energy called as the system free energy, which composes mainly chemical free energy, surface energy and elastic strain energy. In this study, microstructural evolution of ferritic heat resistant steels during creep was evaluate by the system free energy and its stress dependence was expressed quantitatively by using the relaxation time in each component of the free energy. The steels used in this study were P91 (9Cr-1Mo-C-N-V-Nb) steel and P92 (9Cr-Mo-W-C-N-V-Nb-B) steel. The obtained results are as follows: (1) the relaxation time of elastic strain energy was expressed as a function of stress and temperature, (2) surface energy of P92 increased during creep due to the formation of the Laves phase, (3) the relaxation time of the chemical free energy in P92 was larger than that in P91, and (4) the system free energy in both steels was expressed as a function of time, stress and temperatures.

#### 10:05 AM

#### Effect of Niobium Content on Laminar Precipitate and High Temperature Mechanical Properties of 21-2N Valve Steel: Cheng Shichang<sup>1</sup>; Liu Zhengdong<sup>1</sup>; Lin Zhaojie<sup>1</sup>; Bao Hansheng<sup>1</sup>; <sup>1</sup>CISRI

Effect of niobium content on laminar precipitate and high temperature mechanical properties of 21-2N vavle steel has been systematically studied, using specimens contain 0.25%, 0.45%, 0.65% 0.85% and 1.05% Nb. After different solid solution treatment and 750°C aging heat treatment, experimental results show that laminar precipitate is suppressed by niobium addition, and with the increasing of niobium content, laminar precipitate content is decreased and size, distribution and morphology of laminar precipitate is ameliorated. Then creep rupture strength and fatigue strength at 725°C of testing steels are improved with increasing of niobium.

#### 10:20 AM

#### **Process Window Study for Heat Resistant Nanocoated Steel**: Changhoon Choi<sup>1</sup>; Yong-gyun Jung<sup>1</sup>; Jae-ryung Lee<sup>1</sup>; <sup>1</sup>POSCO Technical Research Laboratories

Hot press forming (HPF) is an automobile parts manufacturing process using 22MnB5 steel. Several materials are coated to prevent scale formation on steel surface during HPF process. Metal coatings such as Al, Al-Si, or Zn are mainly used for scale protection. Metals are coated in a hot bath by passing steel strip. Organic-inorganic chemical coating is a promising substitute for metal coatings on steel surface. Recently developed weldable nanocoated HPF steel has been tested for process window in this study. Performances of hot press formed steel are tested depending on heating time and atmosphere and their surface has been studied using SEM, XRD, etc.

#### 10:35 AM Tea Break



#### Symposium A: Advanced Steels and Processing: TRIP and UFG Steels

Thursday AM	Room: A	
August 5, 2010	Location:	Cairns Convention Centre

Session Chairs: Xue-Jun Jin, Shanghai Jiao Tong University; Young-Kook Lee, Yonsei University

#### 11:00 AM Keynote

The Role of Epsilon Martensite on the Hydrogen Degradation Behavior of High Mn Steels: *Chong Soo Lee*<sup>1</sup>; Young Soo Chun<sup>1</sup>; Kyung-Tae Park<sup>2</sup>; Young-Kook Lee<sup>3</sup>; <sup>1</sup>POSTECH; <sup>2</sup>Hanbat National University; <sup>3</sup>Yonsei University

High Mn steels are nowadays receiving a great interest due to its excellent combination of strength and ductility. Furthermore, the resistance to hydrogen embrittlement is also excellent owing to the high solubility of hydrogen in the austenite (gamma) matrix. However, the austenite matrix frequently transforms to hard and brittle martensite (epsilon) phase. In the severe corrosive environment, the gamma/epsilon interphase boundary is one of the susceptible sites for hydrogen embrittlement. Consequently, it is essential to study hydrogen degradation behavior of epsilon martensite phase in the context of the volume fraction of epsilon martensite. In this study, specimens consisting of different volume fraction of epsilon martensite were produced and tested for hydrogen degradation utilizing slow strain rate test. The results were compared with those of fully austenite phase. Hydrogen detrapping activation energy of each phase was also obtained by use of thermal desorption analysis.

#### 11:20 AM

Constitutive Modeling of TWIP Steel in Uni-Axial Tension: *Jinkyung Kim*<sup>1</sup>; Yuri Estrin<sup>2</sup>; Hossein Beladi<sup>3</sup>; Sungkyu Kim<sup>4</sup>; Kwanggeun Jin<sup>4</sup>; B.C. De Cooman<sup>1</sup>; <sup>1</sup>Graduate Institute of Ferrous Technology; <sup>2</sup>Monash University; <sup>3</sup>Deakin University; <sup>4</sup>Technical Research Laboratories, POSCO

Many studies have been done on modeling of the tensile behavior of TWIP steel especially for Fe22Mn0.6C TWIP steel. However, very little information is available about the contribution of DSA on the flow stress of TWIP steel even though many authors reported the occurrence of DSA in TWIP steel. Therefore, any modeling of the tensile behavior of TWIP steel must take strain localization caused by DSA into account. The purpose of this study was to determine the contribution of all the relevant deformation mechanism : slip, twinning and DSA. Constitutive modeling was carried out based on the Kubin-Estrin (KE) model. In the KE model, the densities of mobile and forest dislocations are coupled since a continuous immobilization of mobile dislocations occurs during straining. These coupled densities are also used for simulating the contribution of DSA on the flow stress. The KE model was modified to include the effect of twinning. In order to ascertain the validity of the present model, microstructure and texture evolutions were characterized in detail by using TEM and EBSD.

#### 11:35 AM

#### Effects of Chemical Compositions and Processing Conditions for Development of High Manganese TWIP Steel with 980MPa Tensile Strength: Sangho Han<sup>1</sup>; Kwang-geun Chin<sup>1</sup>; Ohpil Yong<sup>1</sup>; <sup>1</sup>POSCO

It is well known that TWIP (TWinning Induced Plasticity) steels achieve high strengths (800-1200MPa) and large uniform elongations (45~60%) in uni-axial tension test. TWIP steel is new material for automotive use, which is hardened mainly by twin in addition to dislocation, and its microstructure is high manganese and high carbon austenitic steel with varying aluminum or silicon content, resulted into the extraordinary excellent strength-elongation balance. In this study, unique properties such as stress-strain curve, n-value and HER(Hole Expansion Ratio) with Mn, Al contents was studied to apply to automobiles industry. To observe deformation mechanism, microstructure which is related to SFE(Stacking Fault Energy) was analyzed in FeMnCAl TWIP steels. Also delayed fracture and weldability were investigated to make optimum conditions for automobiles

#### 11:50 AM

Selective Oxidation and Sub-Surface Phase Transformation during Austenitic Annealing of TWIP Steels: *Yong Feng Gong*<sup>1</sup>; Hansoo Kim<sup>1</sup>; Seongkyu Kim<sup>2</sup>; B. C De Cooman<sup>1</sup>; <sup>1</sup>POSTECH; <sup>2</sup>Automotive Steel Research Group, POSCO Gwangyang Works

The selective oxidation of Al-free and Al-added Twinning Induced Plasticity (TWIP) steels during full austenitic annealing at 800°C in  $N_2$ +10%H<sub>2</sub> atmosphere at a dew point of -17°C was investigated by means of HR-TEM of FIB cross-sectional samples. For Al-free TWIP steel, a dense MnO layer was formed on

the surface. Crystalline c-xMnO.SiO<sub>2</sub>(x≥2) particles and amorphous a-xMnO. SiO<sub>2</sub>(x<0.9) particles were found at the MnO layer/steel matrix interface. In the subsurface, Mn depletion resulted in the transformation of the austenite to the ferrite structure in a narrow zone. For Al-added TWIP steel, a continuous outer MnO layer and a transition layer consisting of amorphous a-xMnO.SiO<sub>2</sub>(x<0.9) and crystalline c-MnO.Al<sub>2</sub>O<sub>3</sub>(0.8<x<1.2) were formed. The interface between the a-xMnO.SiO<sub>2</sub>(x<0.9) and c-MnO.Al<sub>2</sub>O<sub>3</sub>(0.8<x<1.2) layers had rough structure and 20~50nm diameter voids were formed at the interface. Meanwhile, a narrow Mn-depleted ferrite layer was also formed in the subsurface. The void formation is very likely related to Kirkendall effect occurring during the oxides formation, or delamination of the amorphous and crystalline oxides. The thick MnO layer and the voids constitute major challenges to the successful hot dip galvanization of TWIP steels in industrial HDG lines.

#### 12:05 PM

**Deformation Behavior of Austenite-Base High Mn Steels**: *Ki Hyuk Kwon*<sup>1</sup>; Kyoung-Hun Kim<sup>2</sup>; Chang-Hyo Seo<sup>1</sup>; Ka Young Choi<sup>1</sup>; Soon Gi Lee<sup>3</sup>; Jong-Kyo Choi<sup>3</sup>; Nack Joon Kim<sup>1</sup>; <sup>1</sup>Graduate Institute of Ferrous Technology, Pohang University of Science and Technology; <sup>2</sup>Center for Advanced Aerospace Materials, Pohang University of Science and Technology; <sup>3</sup>Plate Research Group, POSCO

Microstructure of high Mn steels can consist of austenite,  $\varepsilon$ -martensite, and  $\alpha$ '-martensite in various proportions depending on the alloying elements. Although their deformation behavior and mechanical properties can vary a lot depending on the respective volume fraction of each constituent phase, the subject has not been investigated in detail. In the present study, the deformation behavior of austenite-base high Mn steels containing various volume fractions of  $\varepsilon$ -martensite and  $\alpha$ '-martensite has been investigated with particular emphasis on the deformation induced martensitic transformation (DIMT). It shows that there occurs a multiple-martensitic transformation (austenite $\rightarrow \varepsilon$ -martensite $\rightarrow$  $\alpha$ '-martensite) with increasing the amount of deformation. In order to correlate the DIMT with the tensile deformation and work hardening behavior, electron back-scattered diffraction and transmission electron microscopy analyses were performed on the specimens subjected to interrupted tensile tests.

#### 12:20 PM

Effect of N on the Microstructure and Tensile Behavior of TWIP Steel: *Sang Won Lee*<sup>1</sup>; Jinkyung Kim<sup>1</sup>; Hansoo Kim<sup>1</sup>; Sungkyu Kim<sup>2</sup>; Kwanggeun Chin<sup>2</sup>; B. C. Decooman<sup>1</sup>; <sup>1</sup>Graduate Institute of Ferrous Technology; <sup>2</sup>Automotive Steel Application Research Group, POSCO

Present study focused on the design and testing of new 2nd generation TWIP steels to find the alternatives to the existing FeMnC alloy systems in order to reduce the Mn addition. In order to investigate the effect of nitrogen addition, 12Mn0.6C-N was examined with the 18Mn0.6C-N steel as a reference. Effects of N and Mn on microstructure and mechanical properties were investigated by using X-ray diffraction, optical microscopy, scanning electron microscopy and tensile test. The deformation microstructures of 12Mn0.6C-N were mixtures of twinned austenite grain and some of the 5~10% of e-martensite. On the other hand, only deformation twinning was observed in 18Mn0.6C-N during the deformation due to its higher stacking fault energy than 12Mn0.6C-N. 18Mn0.6C-N steel exhibited higher strength and elongation than 12Mn0.6C-N steel. The effect of strain rate on the tensile behavior of both materials was also examined by means of uniaxial tensile test, X-ray diffraction, optical microscopy and scanning electron microscopy. While 18Mn0.6C-N showed clear negative strain rate sensitivity, 12Mn0.6C-N didn't show clear relationship between flow stress and strain rate. The effect of annealing temperature on the tensile behavior and microstructure was also examined.

#### 12:35 PM

#### Study on Selective Oxidation of TRIP Steels Containing Si, Mn, and B: Suk-Kyu Lee<sup>1</sup>; <sup>1</sup>POSCO

It is well known that selective oxidation of Si, Mn and B on the surface of TRIP steels during the heat treatment. However, it is not clear that the relationship between the oxide formation and manufacturing factors such as cooling temperature, cooling rate, etc. In this study, the behavior of oxide formation was studied with different annealing conditions: dew point, cooling temperature, cooling rate, as well as wettability characteristics. The oxide was characterized by FE-SEM, EPMA, AES, and XPS.

#### 12:50 PM

**The Sheet Formability of Superbainitic TRIP Steel**: *JoongEun Jung*<sup>1</sup>; JongBae Jeon<sup>1</sup>; Young-Roc Yim<sup>2</sup>; YoungWon Chang<sup>1</sup>; <sup>1</sup>POSTECH; <sup>2</sup>POSCO Technical Research Lab.

Various high strength low alloy (HSLA) steels have been developed in the past few decades to reduce the weight of automobiles in an effort to enhance fuel efficiency and to reduce air pollution. Nano-structured steels termed as superbainitic (SB) TRIP steels have recently been developed in the above regard thru



shear transformation without rapid quenching or expensive alloying elements. These steels could provide tensile strength greater than 2.2 GPa, but insufficient tensile elongation resulting into very poor formability. There have been several studies in recent years about the effects of retained austenite stability on uniform elongation under uni-axial tensile loading. In this study, isothermal bainite transformation (IBT) treatment has thus been carried out first at several different temperatures to vary the stability of retained austenite to examine its effects on uniform elongation and also on sheet formability of SB TRIP steels. The stability of retained austenite seemed to affect the sheet formability of these steels through deformation induced martensitic transformation (DIMT).

#### Symposium B: Advanced High Temperature Structural Materials: Titanium and Titanium Aluminides

Thursday AMRoom: 7August 5, 2010Location: Cairns Convention Centre

Session Chairs: Matthew Dargusch, Defence Materials Technology Centre; Ji Zhang, China Iron and Steel Research Institute Group

#### 8:30 AM Keynote

**Emerging Robust Beta Gamma TiAl Alloys**: *Young-Won Kim*<sup>1</sup>; Sang-Lan Kim<sup>1</sup>; Chris Woodward<sup>2</sup>; <sup>1</sup>UES, Inc.; <sup>2</sup>Air Force Research Laboratory, AFRL/RX The primary technical reasons why gamma TiAl alloy sheets have not been

explored for numerous aerospace applications include: 1) large anisotropic

lamellar grains and compositional segregation that have to be converted

through multi-step forming process and 2) high brittle-ductile transition temperatures resulting in poor rolling properties leading to unconventional processing requirements. These issues significantly increase the production cost and degrade the balance of structural properties. Recently a new class of TiAl based alloy system, called beta gamma, that utilizes a beta solidification and subsequent beneficial transformation pathways has shown great promise in overcoming these barriers. Typical beta gamma alloys for sheet rolling exhibit fine cast microstructures and improved processibility with a desired three-phase distribution containing a local maximum of beta volume around a relatively low temperature. The local maximum of beta volume allows direct rolling of cast plates with no preconditioning such as hot-working. Once processed and properly heat treated, the material exhibit significantly enhanced strength levels and machinability. This presentation reviews our development process for a lowcost fabrication methodology that involves: optimization of alloy composition for desired phase distribution, direct pack-rolling to thin sheet at a temperature around a local maximum of beta volume, and low stress surface grinding.

#### 8:50 AM Keynote

Wrought Process of Hard-to-Deform High Temperature Materials: *Ji Zhang*<sup>1</sup>; Guangpu Zhao<sup>1</sup>; Zhanglin Ma<sup>1</sup>; <sup>1</sup>China Iron and Steel Research Institute Group

Gualgod Zhao', Zhangin Wa', China Hon and Steer Research Research institute Ordup Higher temperature tolerance and less weight is always the driven force to develop new structural materials for advanced aircraft engines. Increasing the creep limit usually causes a decreased deformation window because the alloys need to contain more volume fractions of strengthening phases. GH586 is one of the Ni-based hard-to-deform alloys in the Chinese system, which contains more than 30%  $\gamma'$  phase. This presentation analyses the technical issues, especially the control of deformation temperature, in the production of GH586 based on its deformation behavior and industry experience. On the other hand, using TiAI intermetallics-based alloys can significantly reduce the weight of aircraft engine but also be up against very limited hot deformability. In order to improve their flow behavior, minor additions of Ni are considered to introduce more softening mechanisms. Thereafter, the commercialization approach of TiAI alloys is discussed based on the deformation behavior comparison between TiAI and GH586 as well as the current trial of TiAI ingot break-down process by extrusion and forging.

#### 9:10 AM Keynote

Fabrication of TiAl Matrix Composite Plate by Rolling and Heat Treatment: Lin Geng<sup>1</sup>; Hong-Lin Wang<sup>1</sup>; Yi-Biao Song<sup>1</sup>; Jie Zhang<sup>1</sup>; <sup>1</sup>Harbin Institute of Technology

Silicon carbide particle reinforced aluminum matrix composite (SiCp/Al) sheet and pure-titanium plate are used for raw materials, and TiAl-matrix composite plate is prepared through the multi-layer combined rolling deformation and heat treatment. Fabrication process of TiAl-matrix composites plate is investigated. Phase composition and reaction kinetics of the reaction layer during heat treatment process are studied by SEM, TEM and XRD. SiCp/Al composite with particle volume fraction of 5% is prepared by powder metallurgy technique. The optimum temperature of hot pressing and rolling of the multi-layered plate is determined to be 400°X and 300°X, respectively, by comparing the yield strength between Ti and SiCp/Al composite. After 60% reduction in thickness by rolling, the reduction of Ti and SiCp/Al sheet is 58% and 62% respectively. After two steps of heat treatment (650°X×25h and 950×40h), the multi-layered Ti-SiCp/Al sheets have transformed to TiAl-based composite sheet, in which  $Ti_3Si_3$  and fine TiC phases are dispersed in TiAl phase.

#### 9:30 AM

Using Accumulative Roll Bonding to Process Gamma Titanium Aluminide Alloys: Peng Qu<sup>1</sup>; Rengang Zhang<sup>1</sup>; Gajanan Chaudhari<sup>1</sup>; *Viola Acoff*<sup>1</sup>; <sup>1</sup>The University of Alabama

The processing and fabrication of certain alloys, intermetallic compounds, and layered composites are inordinately expensive because of the sophisticated methodologies associated with existing processing techniques for these materials. The reactive metals including titanium are a prime example. They are difficult to process in the molten state because they react with crucible linings, as well as with oxygen and nitrogen in air, therefore vacuum systems and water-cooled crucibles must be used to produce titanium and titanium alloy ingots. This work employs accumulative roll bonding followed by reaction annealing to process Ti-48Al, Ti-50Al, and Ti-46Al-9Nb (atomic %) gamma titanium aluminide alloys from elemental titanium, aluminum, and niobium foils. The sheets were severely deformed by repeated cold rolling with interspersed folding of the sheets. Following severe plastic deformation, the specimens were subjected to a two-stage annealing treatment to promote the formation of a lamellar microstructure consisting of Ti3Al and TiAl lamella. The structural evolution of the resulting material was investigated and characterized using transmission electron microscopy (TEM), scanning electron microscopy (SEM), x-ray diffraction (XRD), and differential scanning calorimetry (DSC). Tensile testing and microhardness testing were used to evaluate the mechanical properties.

#### 9:45 AM

The Effect of Heat Treatments on Microstructure and Creep Properties of Power Metallurgy Beta Gamma Titanium Aluminide Alloys: *Trevor Sawatzky*<sup>1</sup>; Dongyi Seo<sup>2</sup>; Henry Saari<sup>1</sup>; Daniel Laurin<sup>1</sup>; D.J. Kim<sup>3</sup>; Young-Won Kim<sup>4</sup>; <sup>1</sup>Carleton University; <sup>2</sup>Structures and Materials Performance Laboratory, Institute for Aerospace Research, National Research Council Canada; <sup>3</sup>Structures and Materials Performance Laboratory, Institute for Aerospace Research, National Research Council of Canada; <sup>4</sup>Division of Materials and Process, UES

The microstructure and creep properties of two powder metallurgy 'beta gamma' titanium aluminide alloys in specific material conditions are presented. Alloy powders with nominal compositions of TiAl-4Nb-3Mn (G1) and TiAl-2Nb-2Mo (G2) were produced by gas atomization and consolidated by a two-step hot isostatic pressing (HIP) process (1250 °C/200 MPa/1 h + 1100 °C/200 MPa/3 h + slow cooling to room temperature). After HIP, the materials were given a step cooled heat treatment (SCHT) of 40 min at 1400 °C, furnace cooling to 1280 °C, and air cooling to room temperature. Selected specimens were aged for 6 or 24 h at 900 °C. The SCHT yielded similar fully lamellar microstructures for both alloys, with a lamellar spacing of 0.04  $\mu$ m, but with different grain sizes averaging 80  $\mu$ m (G1) and 40  $\mu$ m (G2). The aging treatments generated beta precipitates along lamellar grain boundaries in both alloys, but along lamellar interfaces only in alloy G2. Constant load tensile creep tests were performed at 760 °C and 276 MPa. Alloy creep properties are compared as a function of heat treatment history and microstructural features.

#### 10:00 AM

Effect of Y on Microstructure and Mechanical Properties of a Ti Alloy: *Zhiguang Liu*<sup>1</sup>; Changjiang Zhang<sup>1</sup>; Lihua Chai<sup>1</sup>; Yuyong Chen<sup>1</sup>; Kee-Do Woo<sup>1</sup>; <sup>1</sup>Harbin Institute of Technology

Near-a titanium alloys are extensively used in jet engines as compressor discs and blades due to their advantages of light weight and superior fatigue and creep properties at elevated temperatures up to 600°C. Various methods have been proposed to enhance high temperature properties of these alloys and to extend their application temperature over 600°C. In the present study, a near-a high temperature titanium alloy, Ti-6Al-2.5Sn-4Zr-0.7Mo-0.30Si, was designed and produced with various Y additions from 0 to 0.7 wt.% to study the influence of Y on its oxidation behavior. Oxidation experiments were performed in air at 600, 650 and 700°C, respectively. It was found that small amount of Y obviously changes the oxidation behavior of this Ti alloy. The surface oxides are mostly TiO<sub>2</sub> and a small amount of Al<sub>2</sub>O<sub>3</sub>. The addition of Y decreased significantly the weight gain rate of Ti-6Al-2.5Sn-4Zr-0.7Mo-0.3Si alloy. However, the influence of Y addition on the surface morphology and the grain size does not show apparent monolithic. The influence of Y mainly contributed to the reduction of the thickness of oxide layer. It is attributed to the fact that the addition of Y inhibits the diffusion of oxygen and the oxidation of Ti.



#### 10:15 AM

Formation of Protective Intermediate Phase in Ta Addition TiAl during High Temperature Oxidation: *Keizo Hashimoto*<sup>1</sup>; Kyoichi Seita<sup>1</sup>; <sup>1</sup>Teikyo University

Gamma Titanium Aluminides (TiAl) have been investigated extensively during past twenty five years. The recent technical achievements are that TiAl has been applied to a turbine component in the latest jet engine. However, the mechanical properties and the oxidation behaviors of TiAl have to be improved more to apply them in the severe conditions. It became clear that more than 4at%Ta doped TiAl demonstrated more superior oxidation resistance than the other ternary TiAl compounds, according to the weight gain results of the intermittent oxidation experiments at 1173 and 1273K. Oxidation behaviors are strongly influenced by the Ta composition in TiAl. XRD, SEM-EDS and TEM-EDS observations have been carried out to determine the microstructures and the compositions of multilayered oxides near the surface. It was revealed that a protective intermediate phase formed between the matrix TiAl and Ta rich oxides layers simultaneously. Ti53Al32Ta15 ternary compound exists as a equilibrium phase at 1373K, based on the proposed Ti-Al-Ta ternary phase diagram. This ternary compound plays an important role in protecting the penetration of oxygen from the surface. The formation mechanism of the intermediate phase has been discussed in conjunction with diffusion of Ta atom in TiAl.

#### 10:30 AM Tea Break

Symposium B: Advanced High Temperature Structural Materials: Oxidation of High-Temperature Alloys

Thursday AM	Room: 7
August 5, 2010	Location: Cairns Convention Centre

Session Chairs: David Young, University of New South Wales; Hyun Uk Hong, Korea Institute of Materials Science

#### 11:00 AM Keynote

**Materials Technology for PC-TPP in Green Economic Era**: *JeongTae Kim*<sup>1</sup>; Byeong-ook Kong<sup>1</sup>; <sup>1</sup>Doosan Heavy Industries and Con, Ltd

Fossil power generation should be amalgamated with efficiency improvement technology of electric-power production and treatment technology of greenhouse gas in green economic era. The efficiency of fossil power plant can be achieved by increment of temperature and pressure of steam, and the performance improvement of the components. The improvement of temperature and pressure of live steam can be supported by the materials with high temperature strength and the excellent resistance to oxidation. Specially, verified materials have to be used in large quantities for both repair and replacement of steam facing components. The efficiency of newly constructed PC-TPP is higher than 42% at USC conditions of  $250 \sim 300$  bar and  $600 \sim 610^{\circ}$ C. Recently, material technology has been developed to build PC-TPP of the steam condition of advanced USC with more than 46% efficiency. However, it is expected to have problems to economically manufacture in large quantities. Therefore, the paper explains the current the state of the art of materials technologies and the issues with the steam.

#### 11:20 AM Keynote

#### Kinetic Study on Surface Dissolution of Nitrogen in High Nitrogen Steel Making Process: Wan Ho Kim<sup>1</sup>; Seung Min Han<sup>2</sup>; *Dong Joon Min*<sup>1</sup>; <sup>1</sup>Yonsei University; <sup>2</sup>POSCO

Recently, nitrogen control for the steelmaking process have focused on the positive aspect of high nitrogen steel, however, nitrogen is still difficult to control the content in iron and steel. Although a wide variety of research has been carried out, there still remains unclear attributes of nitrogen control. Thus, in the present study, the effects of surface active and alloying elements on the rate constant of nitrogen dissolution in liquid steel have been investigated in order to clarify the kinetics of nitrogen dissolution by an isotope exchange technique. The effects of O. S. C. B. and Mn addition on surface reaction have been considered at 1873 K. Experimental results show that the rate determining step of nitrogen dissolution into molten Fe-O-S alloys would be dissociation reaction and the rate constant on bare surface of the liquid steel(k0) is 3.84×10<sup>-5</sup> (mol/cm<sup>2</sup>·s·atm). The adsorption coefficients for oxygen, sulfur, and boron which were applied the dissociation determining model were calculated to be KO=120, KS=65, and KB=0.9, respectively. In case of manganese addition, the rate constant can be increased with increasing the content of manganese. It seems that rate constant of bare surface of Fe-Mn alloy should be affected by addition of manganese.

#### 11:40 AM

Initial Stages of Oxidation of Ti<sub>45</sub>Al<sub>7</sub>Nb<sub>0.4</sub>Y Alloy at 900° in Air: *Junpin Lin*<sup>1</sup>; Lili Zhao<sup>1</sup>; Laiqi Zhang<sup>1</sup>; Xiping Song<sup>1</sup>; Feng Ye<sup>1</sup>; Guoliang Chen<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing

The initial stage of oxidation of  $Ti_{45}Al_7Nb_{0.4}Y$  alloy (at.%) oxidized at 900°C in air was investigated by using X-ray photo electron spectroscopy and Auger electron spectroscopy. Experimental results revealed that  $YAl_2$  segregated along the grain boundaries preferentially oxidized to  $Y_2O_3$  and  $Al_2O_3$  due to strong affinity of Y to oxygen. Oxides grew faster at the grain boundaries than in lamellar colonies. As a result, Y and Al oxides pegs protruded into the substrate which can increase the contact areas of oxide scale and substrate. Moreover, inward diffusion of oxygen more easily occurred along the grain boundaries. So it promoted the external oxidation of Al within the grains due to lower inward diffusion flux of oxygen. And coarse grained  $Y_2O_3$  blocked the cationic intergranular diffusion. Therefore, Y addition can effectively enhance the  $Al_2O_3$  layer and suppress the TiO, outmost layer.

#### 11:55 AM

The Oxidation of TiAl Alloys Coated by Fluorine Resin in SO<sub>2</sub> Contained Atmospheres: *Aleksander Gil*<sup>1</sup>; Zbigniew Zurek<sup>2</sup>; Adam Stawiarski<sup>2</sup>; <sup>1</sup>AGH University of Science and Technology; <sup>2</sup>Cracow University of Technology

There are several ways to improve the oxidation resistance of TiAl alloys. One of them is the alloying with the other elements like Nb, Ta, W, Si, Ag, Zr, Hf, etc. However, the alloying of the whole bulk alloys influences the mechanical properties. The surface treatment of TiAl alloys by the small amounts of halogens leads to the formation of the protective alumina scale (so called "halogen effect"). The halogens can be apply by ion technique (ion implantation, plasma immersion implantation), as well as by spraying or dipping with halogen containing inorganic and organic compounds. The deposition of the fluorine containing resin on the surface of TiAl alloys is the easiest way for achieve the best results. The presence of SO<sub>2</sub> impurity in the air influences the oxidation behavior of the alloys. In this work the results of the oxidation of Ti-48Al-2Cr alloy coated by fluorine containing resin in the synthetic air, air contained 1% SO<sub>2</sub> and in pure SO<sub>2</sub> were presented. The oxidation were carried out in the temperature range 800-1000°C.

#### 12:10 PM

Isothermal Oxidation Behavior of a New Directionally Solidified Ni-Base Superalloy in Air at Different Temperature: *Lei Wang*<sup>1</sup>; Yan Huang<sup>1</sup>; Zhuo Zhao<sup>1</sup>; Yang Liu<sup>1</sup>; Jiantao Wu<sup>2</sup>; Ping Yan<sup>2</sup>; <sup>1</sup>Northeastern University; <sup>2</sup>Central Iron and Steel Research Institute

The isothermal oxidation behaviour under static atmosphere of a new directionally solidified Ni-base superalloy used for the gas turbine blades was investigated. The results showed that the oxidation kinetics curves of the alloy follows parabolic law in a temperature range of  $750~950^{\circ}$ C during isothermal oxidation. The diffusion activation energy *Q* is to be about 244.86 kJ•mol<sup>-1</sup> and the oxidation within this temperature range is mainly controlled by Cr<sup>3+</sup> diffusion among Cr<sub>2</sub>O<sub>3</sub> oxidation film. The oxidation resistance grade of the alloy is perfect anti-oxidation within 750~850°C, and anti-oxidation within 900~950°C. The oxidation film can be divided into several layers in the order of TiO<sub>2</sub>, Cr<sub>2</sub>O<sub>3</sub>+TiTaO<sub>4</sub>+NiCr<sub>2</sub>O<sub>4</sub>, Al<sub>2</sub>O<sub>3</sub> and TiN, from the surface to inside of the alloy. During the oxidation, priority oxidations of the carbides, grain boundaries and eutectics of the surface and the hypo-surface occur easily. This kind of priority oxidation and the inside oxidation of Al is considered with the oxidation of Ti and the depletion of Cr, and the steady growth of the oxidation films will be influenced and it leads the decreasing of oxidation resistance.

#### 12:25 PM

Effect of Cerium Addition on the Oxidation Behaivor of CoNiCrAlY Alloy: *Yu-Duo Zhang*<sup>1</sup>; Zhi-Gang Yang<sup>1</sup>; Hao Lan<sup>1</sup>; Chi Zhang<sup>1</sup>; <sup>1</sup>State Key Laboratory of New Ceramics and Fine Processing, Department of Materials Science and Engineering, Tsinghua University

The effect of rare-earth-element cerium on the isothermal oxidation behaviour of Co-32Ni-21Cr-8Al-0.6Y alloy at 800 and 1000 centigrade in static air has been investigated. The scale compositions and morphology were analysed by SEM, EDS and XRD. The results revealed that the alloys with and without the addition of 0.2 wt% Ce showed similar oxidation rates at the present exposure of 800 centigrade, while addition of Ce reduced the oxidation rate in the stable oxidation stage by about a factor of two at exposure of 1000 centigrade. The addition of Ce decreased the time of establishing the protective Al<sub>2</sub>O<sub>3</sub> scale due to promoting the transformation from metastable transition Al<sub>2</sub>O<sub>3</sub> to more stable alpha-Al<sub>2</sub>O<sub>3</sub> at elevated temperature. The oxide scale of Ce-containing alloy at 1000 centigrade rendered a continuous and compact alumina layer coupled with thin layer of chromia on the top surface. At 1000 centigrade, Ce addition led to some nitrides formation at scale/substrate interface in Al-depleted zone.



#### CANCELLED

Oxidation Resistance of Innovative Carbon-Based Materials - Results of Extremat: Rainer Moormann<sup>1</sup>; Hans-Klemens Hinssen<sup>1</sup>; Baerbel Schloegl<sup>1</sup>; <sup>1</sup>FZJ

Within of the EU research project ExtreMat C-based materials were developed for application in fusion, fission, aerospace and chemical technology. Whereas C-based materials show many advantageous high-temperature properties, their oxidation resistance may become a crucial item. For that detailed examinations of high-temperature oxidation resistance in oxygen and in steam were performed. Experimental methods applied will be described and the theoretical background is outlined. Materials examined are improved graphites with and without coatings, carbon fiber composites and composites containing a major fraction of SiC. Results of these oxidation measurements indicate that TiC coatings do not improve the oxidation resistance, probably because the Ti-oxide layer formed is not glass-like (as SiO<sub>2</sub>) but remains porous and thus penetrable for oxidizing gases. Similar holds for ZrC, too. Promising oxidation resistance was revealed by double coatings, containing a rare oxide layer. SiC/C-composites show a pronounced oxidation resistance, if containing more than 30% of SiC. Main influence parameters on oxidation rates of pure carbons are outlined. Limits of presently applied oxidation models are discussed.

#### 12:40 PM

**High-Temperature Sulfurization of Crofer 22APU in H**<sub>2</sub>/H<sub>2</sub>S Atmosphere: *Zbigniew Zurek*<sup>1</sup>; Artur Jaron<sup>1</sup>; Adam Stawiarski<sup>1</sup>; Aleksander Gil<sup>1</sup>; <sup>1</sup>Cracow University of Technology

The Crofer steel is usually used as an interconnector at SOFC on the cathode and anode atmospheres. When the industrial gases (H<sub>2</sub> or CH<sub>4</sub>) are used in the cathode atmosphere they can be impure by water vapor and sulphur compounds. One of the commonly meeting pollutant is sulfur in H<sub>2</sub>S and it can have a significantly effect on corrosion of interconnector. In this paper authors present results of sulfidation of Crofer steel in H2/H2S. The high temperature corrosion behavior of Crofer 22APU and preoxidized Crofer steel were studied in the temperatures range 600 - 900°C in H<sub>2</sub>/H<sub>2</sub>S atmospheres. Sulphur partial pressures (pS<sub>2</sub>) was in the range 10<sup>-7</sup> to 10<sup>-1</sup> Pa. The kinetics show that the sulfidation process of preoxidized samples was going slower. Morphology of the scales have been observed by SEM. Phase and chemical composition have been studied by EDX and XRD methods. It was found that scale formed on Crofer steel was built with porous sulphides and in some case  $(pS_2, temperature)$  the internal sulfidation of steel was observed. The phase analysis of the scale reveled that it is composed mainly of sulphides of iron and chromium but in low sulfur partial pressure only chromium sulfide was detected.

#### Symposium C: Light Metals and Alloys: Casting of Aluminium Alloys

Thursday AM August 5, 2010 Room: 6 Location: Cairns Convention Centre

Session Chair: Zhan Chen, AUT University

#### 8:30 AM

In Line Rolling of Aluminum Alloy Strip Cast by a High Speed Twin Roll Caster: *Toshio Haga*<sup>1</sup>; <sup>1</sup>Osaka Institute of Technology

In line rolling was operated on the strip cast by a high speed twin roll caster. AA5182 aluminum alloy and AA6022 aluminum alloy were used in this study. The high speed twin roll caster could cast the aluminum alloy strip at speed of 60m/min. The temperature of the as-cast strip was higher than 400°C. The as-cast strip was immediately hot rolled at in line after the roll casting. The reduction of rolling was about 20%. The roll cast AA5182 strip had heavy ripple mark on the surface and the porosity at inside. The surface of the strip became sound by in line rolling. The ripple mark on the surface was erased. The porosity, which existed at center line of the thickness direction, was improved, too. The alligator crack occurred at the AA6022 strip cast by the high speed twin roll caster. The alligator crack was bonded by the in line rolling. The effect of the in line rolling on the high speed roll casting was shown in this study.

#### 8:45 AM

Roll Casting of Al-SiCp Strip: Toshio Haga<sup>1</sup>; *Hisaki Watari*<sup>2</sup>; <sup>1</sup>Osaka Institute of Technology; <sup>2</sup>Gunma University

Al-SiCp aluminum alloy has some useful advantages i.e., low thermal expansion, better thermal conductivity and wear registrant. Recently, the plate of Al-SiCp, which thickness is thinner than 1mm, is demanded. The Al-SiCp is hard and brittle. Therefore, only the many times of hot rolling was useful process

to make thin plate. In the present study, the roll casting of Al-SiCp strip was tried by a vertical type high speed twin roll caster from the point of energy sayving. The Al-SiCp strip, which thickness was about 3mm, was cast directly from the molten metal. The particles of SiC were dispersed uniformly. The as-cast Al-SiCp could be coiled. The roll cast Al-SiCp had superior ductility. This reason was that the eutectic Si of matrix aluminum alloy (Al-Si alloy) became fine and globular. The cold rolling could be operated after 1-pass of hot rolling. The 1mmthick Al-SiCp plate could be made by one-pass of hot rolling and 3-pass of cold rolling/annealing from as-cast strip. The process saving was attained. The width of Al-SiCp as-cast strip could be increased up to 600mm.

#### 9:00 AM

**Refinement of TiB<sub>2</sub> in Al-Ti-B Grain Refiner Alloys by Ultrasound and the Effect on Al Grain Size**: Da Shu<sup>1</sup>; BaoDe Sun Sun<sup>1</sup>; *Jiawei Mi*<sup>2</sup>; Patrick Grant<sup>2</sup>; <sup>1</sup>Shanghai Jiao Tong University; <sup>2</sup>University of Oxford

The use of Al-Ti-B as an extrinsic grain refiner to Al alloy ingots and castings is a standard industrial practice in metal industry. However, for a very long time, quantitative assessment of the chemical and particle size distribution effects on grain refining performance of Al-Ti-B has received little attention, despite its commercial importance. In this paper, we describe an investigation of the application of high-intensity ultrasound during the reaction between halide salts and molten Al at different Ti/B ratios with the aim of speeding up the reaction and providing a means to control TiB<sub>2</sub> particle size. The subsequent grain refining performance in Al alloys was also experimentally evaluated and results compared with the free-growth model developed by Greer et al. Ultrasonic vibration significantly refined both the blocky TiAl<sub>3</sub> particles and polygonal TiB<sub>2</sub> particles. The mean size of TiB<sub>2</sub> particles was reduced to 646.9 nm, with a narrow size distribution ranging from 0.1-1.5 µm in a Al-5Ti-1B alloy. Because of the narrow size distribution of the TiB<sub>2</sub> particles, the grain refining performance of Al-Ti-B on Al alloys was greatly enhanced.

#### 9:15 AM

Effect of Electromagnetic Field on Horizontal Direct Chill Casting of 7075 Aluminum Alloy: *Qingfeng Zhu*<sup>1</sup>; Zhihao Zhao<sup>1</sup>; Xiangjie Wang<sup>1</sup>; Jiangzhong Cui<sup>1</sup>; <sup>1</sup>Key Laboratory of Electromagnetic Processing of Materials, Ministry of Education, Northeastern University

The influence of different electromagnetic fields on the horizontal direct chill (HDC) casting of aluminum alloy is studied. 7075 aluminum alloy with 100-mm in diameter is produced by HDC casting process; single electromagnetic field located before or around mold and combination electromagnetic field (CEMF) are applied in the HDC casting process. The effect of different electromagnetic fields on the HDC casting solidification behavior and as-cast structure is investigated. The electromagnetic field can effectively make uniform thermal distribution in the molten pool, amend the sump shape and refine the as-cast structure. The CEMF is more efficient than the single electromagnetic field in improving the as-cast structure of HDC casting ingots.

#### 9:30 AM

Modelling of the Metal/Mould Interactions in the DC Casting Process for Aluminium and Magnesium: Arvind Prasad<sup>1</sup>; Ian Bainbridge<sup>1</sup>; <sup>1</sup>University of Queensland

The process of direct chill (DC) casting of aluminium and magnesium alloys is regarded as a mature technology. The thrust of more recent work to understand and upgrade the technology has been centred on developing models of the process, the most advanced of which (e.g., Alsim and Calcasoft) have been used to examine what may be considered macro-features of the process (macrosegregation, hot cracking, etc.). These models, by being macroscopic in nature, rarely elaborate on the role of mould-wall heat transfer in the DC casting process. As part of the work on DC casting being conducted at CAST, for the investigation of small scale features of the process (e.g. heat extraction through the mould wall), a 2D, Finite Difference model of the process near the mould-wall region has been developed. The basic features of the model are described and initial results outlined. In particular, the effect of mould-wall heat transfer on the solid shell formed during the steady state regime of DC casting will be presented.

#### 9:45 AM

Assessing of Bendability of Aluminum Alloy Sheets for Autobodies: Kazuhiro Masuda<sup>1</sup>; Goroh Itoh<sup>2</sup>; Nobuhide Itoh<sup>2</sup>; <sup>1</sup>Graduate Student, School of Science and Engineering, Ibaraki University; <sup>2</sup>Mechanical Engineering, Ibaraki University

In late years, reducing carbon dioxide is being demanded in terms of preventing global warming. Lightening with Al-Mg-Si alloy is effective. Although the Al-Mg-Si alloys have the advantage that they have hardenability during paint baking and do not cause stretcher strain patterns, poor formability is a crucial drawback. Bendability is one of the most important properties related to formability. However, there has been no assessing method having both speediness and quantitative reproducibility. In this study, we have developed a assessing method based on the electric resistance decrease arising from the decrease in conducting



section when cracks are formed by bending. Two 6016 alloy specimens with different iron contents were prepared in forms of cold-rolled sheets with a thickness of 1mm in T4-temper. Test pieces for bending were cut from the sheets in longitudinal and transverse directions (bending direction was parallel to rolling and transverse directions, respectively), resulting in four kinds of test pieces to be examined. Bendability was assessed by electrical resistance change as well as by crack density on the tension surface measured with an SEM. It was found that the new method have far greater speediness with the same quantitative reproducibility than the crack measurement method.

#### 10:00 AM

#### Solidification Structure and Sound Absorbability of A356 Alloy Foams: Yanxiang Li<sup>1</sup>; <sup>1</sup>Tsinghua University

Metallic foams of A356 alloy with a uniform porosity of 80~85% were produced with the melt foaming process. The microstructure inside the cell walls of the metallic foams has been studied. It is found that the microstructure in the cell wall of the metallic foam is greatly different from the cast structure of the base alloy. It is believed that the thickening process with calcium, the adding of foaming agent and the mixing process during the foaming process all play important roles on the solidification process and microstructure of the foam walls. The morphology of primary aphase, the morphology and distribution and eutectic silicon, and the compound formed by reaction of calcium with elements in the base alloy were studied. The sound absorb coefficient of the alloy foams was experimentally studied. According to the analysis on the mechanism of sound absorbing of metallic foams, two methods of small hole drilling and pressing to the foam have been developed to improve the sound absorbability of the alloy foam in low frequency field.

#### 10:15 AM

#### Ultrasonic-assisted Fabrication of SiC Nanoparticles Reinforced Aluminum Matrix Composites: Wenzhen Li<sup>1</sup>; <sup>1</sup>Tsinghua University

SiC nanoparticles reinforced ADC12 aluminium alloy nanocomposites were synthesized by mechanical stirring and high-intensity ultrasonic dispersion processing. Ambient temperature tensile tests revealed significant improvement in UTS and elastic modulus of ADC12 with the progressive addition of SiC nanoparticles. As compared to ADC12 aluminium alloy matrix, the tensile strength, elastic modulus and elongation of the 2.0wt.%SiC /ADC12 nanocomposite were improved by 22.9%, 43.1% and 155.9% respectively in permanent mold casting. The strength and ductility of the nanocomposites were improved simultaneously. Microstructural studies conducted on the composites specimens shown uniform reinforcement distribution. It is clear that SiC nanoparticles were dispersed and distributed very well throughout the aluminium alloy matrix. TEM study of the interface between SiC nanoparticles and matrix showed that SiC nanoparticles were well-bonded with matrix without forming an intermediate phase.

#### 10:30 AM

## **Preparation Aluminum-Strontium Alloy by Molten Salt Electrolysis Method**: *Sh Yang*<sup>1</sup>; Fengli Yang<sup>1</sup>; Qingsheng Liu<sup>1</sup>; Lizhi Fang<sup>1</sup>; <sup>1</sup>Jiangxi University of Science and Technology

Aluminum-strontium alloy was prepared by molten salt electrolysis method with 38(w)%BaF2-43(w)%NaF-19(w)%LiF as electrolyte. Electrolytic temperature was 820°C, and SrCl<sub>2</sub> was taken as raw materials. The results showed that content of strontium in alloy could be higher than 8(w)%. With the increase of current density and electrolysis time, current efficiency was increased sharply, and then decreased gradually. The highest current efficiency was 86.4%, and the lowest was 61.4%. Variation of cell voltage was in 0.5V during electrolytic process.

10:45 AM Tea Break

#### Symposium C: Light Metals and Alloys: Magnesium Casting Alloys

Thursda	ay	AM	
August	5,	201	0

Room: 6 Location: Cairns Convention Centre

Session Chairs: Bong Sun You, Korea Institute of Materials Science; Jianzhong Cui, Northeastern University

#### 11:00 AM

The Influence of Eutectic Morphology on the Impact Properties of High Pressure Die Cast Mg-Rare Earth Alloys: Mark Easton<sup>1</sup>; Katharina Strobel<sup>1</sup>; Suming Zhu<sup>1</sup>; Mark Gibson<sup>1</sup>; Jian-Feng Nie<sup>1</sup>; <sup>1</sup>CAST CRC

This paper continues an investigation into the role of microstructure in the properties of Mg-RE based alloys. Previously it has been shown that room

temperature strength and ductility are related to the type and volume fraction of intermetallics. Creep was also shown to improve with the volume fraction of intermetallics, but was more dependent upon the amount of solute in the a-Mg which facilitates dynamic precipitation. Another feature of the microstructure that varied between the rare earths was the morphology of the eutectic. The Mg-La alloys have a lamellar structure with Mg<sub>12</sub>La as the intermetallic phase, Mg-Ce alloys had rod shaped a-Mg contained within a continuous Mg<sub>12</sub>Ce intermetallic, and the Mg-Nd alloys had a lamellar structure with Mg<sub>3</sub>Nd as the intermetallic phase. Interestingly, the alloys with a more lamellar eutectic had higher elongations in elevated temperature tests and absorbed more energy during impact testing than the room temperature test results would indicate. It appears that, at both higher temperatures and at higher deformation rates, fracture tended to pass through the eutectic, rather than just along the grain boundaries, and that a lamellar structure provides more resistance to fracture than those where the intermetallic phase is more continuous.

#### 11:15 AM

Development of Heat-Resistance and Creep-Resistance Magnesium Alloy of Die-Casting Mg-4Al-4La and/or Ce: *Jian Meng*<sup>1</sup>; Jing Huai Zhang<sup>2</sup>; De Ping Zhang<sup>1</sup>; Zheng Tian<sup>1</sup>; Wei Sun<sup>1</sup>; Hua Yi Lu<sup>1</sup>; Ding Xing Tang<sup>1</sup>; <sup>1</sup>Changchun Institute of Applied Chemistry, Chinese Academy of Sciences; <sup>2</sup>Harbin Engineering University

A new alloy named AE44 developed by hydro magnesium has more excellent high temperature creep and strength performance and has been successfully used for producing large structural magnesium castings, which comprises of Ce, La, Nd and Pr. However, due to the rising price of Nd and Pr, it results quickly in cost increase of AE44. The mixture of Ce and La is very abundant and its price is much lower in China. In this paper, several Mg-4Al-4La/Ce alloys were developed and their microstructure and mechanical properties were evaluated. The phase compositions of Mg-4Al-4La alloy consist of a-Mg and Al, La, phases. While two binary Al-RE phases, Al<sub>11</sub>RE<sub>3</sub> and Al<sub>2</sub>RE (RE = Ce/La), are formed in Mg-4Al-4Ce/La alloy, and Al<sub>11</sub>Ce, and Al<sub>2</sub>Ce are formed in Mg-4Al-4Ce alloy. The optimal tensile properties are obtained in Mg-4Al-4Ce/La alloy, in which the UTS, YS and d are 250, 149.2 MPa and 12.24% at room temperature, and 157.8. 116 MPa and 27% at 150°C, and 99.1. 118.2 MPa and 23.2% at 250°C. respectively. The mechanics properties of Mg-4Al-4Ce/La alloy are nearly same with commercial AE44. Mg-4Al-4Ce/La alloy also has the best corrosion resistance. The compression creep is more lower than that AJ62 alloy.

#### 11:30 AM

The Skin Effect in a Mg-Re High Pressure Die Cast Alloy: Kun Yang<sup>1</sup>; Nagasekhar Venkata Anumalasetty<sup>1</sup>; Carlos Caceres<sup>1</sup>; Mark Easton<sup>2</sup>; <sup>1</sup>ARC Centre of Excellence for Design in Light Metals, The University of Queensland; <sup>2</sup>CAST Co-operative Research Centre, Monash University

Cross-sectional microhardness maps of cast-to-shape flat specimens with thickness 3mm have been obtained for three binary alloys of compositions Mg-2.87%Ce, Mg-3.44%La and Mg-3.53%Nd. Higher microhardness numbers were generally found near the casting surface, at the corners and parts of the segregation band for all three alloys. The higher hardness values can be ascribed to the finer solidification microstructure near the surface and the positive macro segregation at the segregation band. The majority of lower hardness numbers were found at the core region due to the coarser grain size, the rest of which were accounted for by dispersed microporosity and large externally solidified dendritic grains. The non uniformity of the harder surface layer in both depth and hardness can be accounted for by local homogeneities of the grain size distribution.

#### 11:45 AM

Microstructural Evolution of Rheo-Diecast AZ91D Magnesium Alloy with Gadolinium Addition: Jinling Zhang<sup>1</sup>; Xitao Wang<sup>1</sup>; Yong He<sup>1</sup>; Yanli Wang<sup>1</sup>; Yang Zhang<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing

The rheo-diecasting (RDC) process, a novel semi-solid processing technology, was used to produce cast components with high integrity, fine and uniform microstructure, and therefore enhanced performance. Experiments were carried out to investigate the effects of the intensity of forced convection, shearing time, and shearing temperature on the microstructure evolution during the RDC processes. It was found that under intensive forced convection, the primary phase produced inside the twins-crew slurry maker has fine particle size, spherical morphology and uniform distribution throughout the samples. The  $Mg_{17}AI_{12}$  β-phase produced inside the die cavity formed a continuous network. The β-phase was a brittle intermetallic compound, which was the main reason for weak thermal properties of the alloy at rather high temperatures. In order to improve thermal fatigue behaviors, the RE was added. This improvement was attributed to the consummation of aluminum in melt by precipitation of the  $AI_{11}RE_3$  phases. This process was attributed to the reduction of  $Mg_{17}AI_{12}$  phase volume fraction and consequent decrease of the brittle  $Mg/Mg_{17}AI_{12}$  interface.



#### 12:00 PM

Inoculation during Metal Castings: Beyond Grain Refining: Dong Qiu<sup>1</sup>; Mingxing Zhang<sup>1</sup>; <sup>1</sup>The University of Queensland

Inoculation is the most common grain refining technique during metal/alloy castings. However, only a small fraction, typically 1~2%, of inoculants serve as nucleation sites while most of them do not participate in the nucleation event and hence they are termed 'inactive' inoculants. But 'inactive' does not mean 'useless'. Our recent studies showed some extra merits of the inoculated particles. In Mg-10wt%Y cast alloy, addition of 1wt%Al leads to in-situ formation of Al,Y particles. Small fraction of the particles acts as effective nucleation sites and refines the grains by 5 times. The majority of Al<sub>2</sub>Y particles segregate along the grain boundaries (GBs). They inhibit the GB migration so that the grain size still keeps constant after solution treatment at 550°C for 2 days. In contrast, addition of 0.5wt%Y in Mg-9wt%Al results coarsening of Mg grains by 3 times. The reduction of GB volume reduces its creep strain rate by 5 times at 150°C. However, its room temperature strength is still well maintained despite of grain coarsening. This is attributed to the dispersion strengthening of the intermetallic particles in-situ formed during castings. The current research represents a new approach to microstructure design through manipulation of inoculated particles during castings.

#### 12:15 PM

Aluminium-Rich Coring Structures in Mg-Al Alloys Inoculated by Carbon: *Yuanding Huang*<sup>1</sup>; Xiuhua Zheng<sup>1</sup>; Gyu Seok Kim<sup>1</sup>; Karl Ulrich Kainer<sup>1</sup>; Norbert Hort<sup>1</sup>; <sup>1</sup>GKSS Research Center

A homogeneous microstructure of as-cast magnesium alloys is desired to improve the formability during their subsequent thermomechanical processing. In Al-containing magnesium alloys, the grain refinement by carbon inoculation was considered to be the best approach until now. However, the mechanism of grain refinement was unclear. The present work investigates the coring microstructure in Mg-Al alloys with carbon inoculations using FIB, SEM and TEM techniques. In each grain there exist one or more "hillocks" with carbon, manganese and aluminium etc. enriched, which is possibly related to the inhomogeneous nucleation of alpha-magnesium. The precipitates in these "hillocks" are always surrounded by the aluminium-rich zones. This characteristics of microstructure observed in Mg-Al alloys with carbon inoculations are compared with that observed in Al-free magnesium alloys inoculated by zirconium. The similarities between them are discussed. A novel mechanism is suggested to explain the grain refinement in Mg-Al alloys inoculated by carbon.

#### 12:30 PM

On the Influence of Vibration Frequency on the Solidification of AZ61 Magnesium Alloys during Electromagnetic Vibration: *Mingjun Li*<sup>1</sup>; Takuya Tamura<sup>1</sup>; Naoki Omura<sup>1</sup>; Kenji Miwa<sup>1</sup>; <sup>1</sup>National Institute of Advanced Industrial Science and Technology, Materials Research Institute for Sustainable Development

We solidified the AZ61 alloy using an electromagnetic vibration technique and investigated the microstructure development as a function of vibration frequency. The microstructure evolution was quantitatively examined in terms of the total average grain size and the individual grain size distribution and the texture was profiled. The origin of the microstructure formation was clarified when considering a significant difference in electrical resistivity between a primary solid and its surrounding liquid in the mushy zone, which makes the solid move faster than the liquid and thus generating uncoupled motion. The influence of the non-synchronous motion on microstructure formation is discussed as a function of vibration frequency during EMV. Moreover, the resultant structure and texture can be well elucidated when considering the competition between the Lorentz force due to EMV and the magnetization torque due to the static magnetic field during solidification.

#### 12:45 PM

Microstructures and High Temperature Mechanical Behavior of Mg-Al-Ca-Zn Alloys: Seung Hwa Choi<sup>1</sup>; *Hyun-wook Han*<sup>1</sup>; Byoung-Gi Moon<sup>2</sup>; KyungHyun Kim<sup>2</sup>; Keun Yong Sohn<sup>1</sup>; <sup>1</sup>Inje University; <sup>2</sup>Advanced Materials Research Division, Korea Institute of Materials Science

Magnesium alloys have been used to fabricate many automobile components because they offer excellent strength-to-weight ratio with good castability. However, their applications to those components that are exposed to elevated temperatures above 150°C have been limited because of relatively poor creep resistance. It has been known that Mg-Al-Ca alloys constitute a pseudo-binary eutectic between magnesium and Al<sub>2</sub>Ca intermetallic compound. In this work, we have investigated the effect of relative volume fraction of the pseudobinary eutectic constituent on the high temperature strength and creep resistance of the alloys. In addition, the effect of zinc addition to the pseudobinary system has been studied to identify any changes in constituting phases. Zinc has been reported to strengthen magnesium matrix by precipitation hardening. The effect

of aging for Mg-Al-Ca-Zn alloys at 175°C up to for 278 hours on their mechanical properties has been investigated as well. Results showed that the addition of zinc did not significantly change the composition of constituting phases and effectively improved creep resistance of Mg-Al-Ca alloys during artificial aging. The strength and elevated temperature behavior of Mg-Al-Ca-Zn alloys will be discussed in terms of microstructural changes based on transmission electron microscopy as well.

#### Symposium C: Light Metals and Alloys: Microstructures and Properties of Mg-Y-Zn Alloys

Thursday AM	Room: C
August 5, 2010	Location: Cairns Convention Centre

Session Chairs: Joseph Robson, University of Manchester; Sangmok Lee, Korea Institute of Industrial Technology (KITECH)

#### 8:30 AM

Effect of Volume Fraction of LPSO Phase on Mechanical Properties in Extruded Mg-Zn-Y Alloys: Yoshihito Kawamura<sup>1</sup>; <sup>1</sup>Kumamoto University

Recently we have found that some Mg-TM-RE (RE is limited to rare-earth metals of Y, Gd, Dy, Ho, Er, Tb and Tm, and TM is limited to transition metals of Co, Ni, Cu and Zn) cast ingots formed a long period stacking ordered (LPSO) structure as well as rapidly solidified alloys. The LPSO alloys are essentially duplex, which are composed of LPSO and  $\alpha$ -Mg phases. Hot plastic deformation improves the mechanical properties of the LPSO alloys drastically. The improvement of the mechanical properties in the duplex alloys seems to be due to the formation of kink bands in LPSO phase and the grain refinement of  $\alpha$ -Mg phase through dynamic recrystallization. In this study, I will report the effect of volume fraction of LPSO phase on mechanical properties in extruded Mg-Zn-Y alloys. Tensile strength increased with increasing volume fraction of LPSO phase. The wrough LPSO Mg-Zn-Y alloys having LPSO phase volume fraction of 40 ~ 60 % exhibited a good ductility above 5 % in tensile elongation.

#### 8:45 AM

Characterisation of Microstructures in Mg-Y-Zn Alloys: Yuman Zhu<sup>1</sup>; Allan Morton<sup>2</sup>; Jian-Feng Nie<sup>1</sup>; <sup>1</sup>Monash University; <sup>2</sup>CSIRO

Ternary Mg–Y–Zn alloys have received considerable attention in the past 10 years due to their excellent mechanical properties and unique microstructures. The microstructure of these alloys usually contains a range of long-period stacking ordered (LPSO) structures, with 18R and 14H being the most commonly observed structures. The 18R and 14H structures are also observed in other magnesium alloys such as those based on the Mg–Gd–Zn, Mg–Gd–Y–Zn, Mg–Dy–Zn, Mg–Ho–Zn, Mg–Er–Zn and Mg–Y–Cu systems. While these two LPSO structures are commonly observed, their detailed atomic structures and structural relationships remain to be unambiguously established. The present study involves detailed characterisation of the crystal structures of 18R and 14H and their structural relationships in Mg-Y-Zn alloys using atomic-resolution high-angle annular dark-field scanning transmission electron microscopy. The stacking faults that formed in these alloys are also characterised and computed in detail. These results will be presented and compared with those reported previously by other researchers.

#### 9:00 AM

**Formation of a Novel X Phase in Mg–Gd–Zn–Zr Alloy**: Yujuan Wu<sup>1</sup>; *Liming Peng*<sup>1</sup>; Xiaoqin Zeng<sup>1</sup>; Dongliang Lin<sup>1</sup>; Wenjiang Ding<sup>1</sup>; <sup>1</sup>Shanghai Jiao Tong University

The coherent fine-lamellae consisting of the 2H-Mg and the 14H-type long period stacking ordered (LPSO) structure within  $\alpha$ '-Mg matrix have been observed in an as-cast Mg–Gd–Zn–Zr alloy. During subsequent solid solution heat treatment at 773K, in addition to the lamellae within matrix, a novel lamellar X phase(Mg–8.37±1.0at.%Zn–11.32±1.0at.%Gd) with the 14H-type LPSO structure was transformed from the dendritical  $\beta$  phase. The 14H-type LPSO structure existing in Mg–Gd–Zn–Zr alloys derives from two variant ways: the formation of LPSO structure within  $\alpha$ '-Mg matrix and the transformation of the dendritical  $\beta$  phase to a lamellar X phase with the LPSO structure.

#### 9:15 AM

Multimodal Microstructure Evolution in Wrought Mg-Zn-Y Alloy with High Strength and Increased Ductility: *Michiaki Yamasaki*<sup>1</sup>; Kenji Hashimoto<sup>1</sup>; Koji Hagihara<sup>2</sup>; Yoshihito Kawamura<sup>1</sup>; <sup>1</sup>Kumamoto University; <sup>2</sup>Osaka Univesity

A high-strength Mg-Zn-Y alloy was developed, with increased ductility and a multimodal microstructure. The microstructure of the extruded Mg-Zn-Y alloy



consisted of three regions: a dynamically recrystallized alpha-Mg fine-grain region with random orientation, a hot-worked alpha-Mg coarse-grain region with strong basal texture, and a long-period stacking ordered (LPSO) phase region with kink deformation bands. Bimodal microstructure evolution in the alpha-Mg matrix was influenced by the morphology of the LPSO phase in the as-cast state, so we investigated the effect of LPSO phase morphology in the cast state on the microstructure evolution and mechanical properties of the extruded Mg-Zn-Y alloy. An increase in dynamically recrystallized alpha-Mg grains having random crystallographic orientation improved ductility; the effective dispersion of the hot-worked alpha-Mg grains with a strong basal texture and a kink-deformed LPSO phase brought about strengthening of the alloy.

#### 9:30 AM

Preparation and Mechanical Property of Mg-Zn-Y Alloy with a Long Period Ordered Phase: *Takaomi Itoi*<sup>1</sup>; Yasuki Kuroda<sup>1</sup>; Yoshinito Kawamura<sup>1</sup>; Mitsuji Hirohashi<sup>1</sup>; <sup>1</sup>Japan

Magnesium alloys have several distinct advantages as structural materials because of their low density and high specific strength. In present study, in order to develop high strength Mg alloy, we have prepared several compositions of the Mg-Zn-Y alloys with LPO (Long Period Ordered) phase. Mg-Zn-Y cast alloys with binary phases (Mg and LPO phases) exhibited high-strength with comparative elongation. The 0.2% proof strength (s0.2) was increased with increasing of Zn and Y elements in the alloy system. On the other hand, elongation (d) was decreased in Zn and Y rich region in the alloy system. Also, we have prepared Mg-Zn-Y alloy sheets by hot rolling using these cast alloys, and investigated these microstructure and mechanical properties. The Mg<sub>94</sub>Zn<sub>3</sub>Y<sub>3</sub>(at%) alloy sheet prepared by hot rolling exhibited s0.2 of 379MPa, ultimate tensile strength (sB) of 403MPa, and d of 8% at room temperature. After annealed state (773K for 10 min) of the sheet exhibited s0.2 of 291MPa, sB of 370MPa, d of 14% at room temperature.

#### 9:45 AM

Temperature Dependence of Compressive Deformation Behavior of Mg<sub>89</sub>Zn<sub>4</sub>Y<sub>7</sub>Extruded LPSO-Phase Alloys: *Koji Hagihara*<sup>1</sup>; Akihito Kinoshita<sup>1</sup>; Yuya Sugino<sup>1</sup>; Michiaki Yamasaki<sup>2</sup>; Yoshihito Kawamura<sup>2</sup>; Hiroyuki Yasuda<sup>1</sup>; Yukichi Umakoshi<sup>3</sup>; <sup>1</sup>Osaka University; <sup>2</sup>Kumamoto University; <sup>3</sup>National Institute for Materials Science

Mg alloys containing long period stacking ordered (LPSO) phases were very focused owing to their excellent mechanical properties. In order to clarify the strengthening mechanism by the LPSO-phase, the deformation behavior of Mg<sub>89</sub>Zn<sub>4</sub>Y<sub>7</sub>(at.%) extruded alloy mostly composed of LPSO-phase was investigated, focusing on its temperature dependence. Several heat-treatments were conducted for the extruded alloy, and the correlation between the microstructure and the mechanical properties were also examined. The yield stress of as-extruded alloy showed extremely high value of ~480MPa at RT, and the deformation occurred accompanied by the formation of deformation kinks and small amounts of non-basal slips. The high yield strength was maintained even at 200°C, but the yield stress was rapidly decreased at 300°C. The microstructure was highly stable against the heat-treatment, but the yield stress of the specimen annealed above 475°C was largely decreased due to the disturbance of texture, and the (0001) basal slip governed the plastic behavior. The yield stress of the annealed specimens could be estimated by the Hall-Petch relationship by regarding the length of long-axis of plate-like grains as a grain size. As similar to that in the as-extruded alloy, the yield stress of the annealed specimens showed a large decrease at 300°C.

#### 10:00 AM

Thermal Stability and Mechanical Properties of Mg-Zn-Y Alloys with Long-Period Stacking Order Phase: *Masafumi Noda*<sup>1</sup>; Yoshihito Kawamura<sup>2</sup>; <sup>1</sup>Kumamoto technology and industry fundation; <sup>2</sup>Kumamoto University

Magnesium alloys are very attractive in such applications as automotive and aerospace. However, the mechanical properties of Mg alloys are inferior to those of Al alloys, and this limits their range of applications. In the present study, effects of annealing on the mechanical properties and the thermal stability of the microstructure in LPSO Mg-Zn-Y extruded alloys were investigated. Even if elongation showed 10% with having maintained high strength to annealing temperature at 573 K for 3.6 ks, and this alloy extended annealing time for 1.0 kh, LPSO phase did bent and was able to confirm kink bands. On the other hand,  $\alpha$ -Mg phase was fine structure without pronounced grain growth. In elevated temperature deformation of as extruded and annealed materials, LPSO phase did fine dispersed to  $\alpha$ -Mg phase by grain boundary sliding of  $\alpha$ -Mg phase, but fracture did not occurred in the interface between LPSO and  $\alpha$ -Mg phases. Kink bands introduced in LPSO phase during plastic deformation and fine-grained  $\alpha$ -Mg phase were important to obtained of high strength and ductility.

#### 10:15 AM

The Microstructure, Tensile Properties and Creep Behavior of Mg–Zn–Y– RE Alloys: *Jonghyun Kim*<sup>1</sup>; Yoshihito Kawamura<sup>2</sup>; <sup>1</sup>Kumamoto Technology & Industrial Foundation; <sup>2</sup>Kumamoto University

Several wrought magnesium alloys such as AM- and AZ-series are structural materials which are suitable for use in the computers, mobile phone and automobile industries mainly because of their low densities and high specific resistance. However, the number of commercially available Mg alloys is still limited especially for application at room and elevated temperatures. It has been demonstrated that rare earth metals (RE) are the most effective elements to improve the creep and strength properties of magnesium especially at elevated temperatures. Recently, Kawamura et al. have developed the Mg-Zn-Y alloys with excellent mechanical properties at room and elevated temperatures. The Mg-Zn-Y alloy consisted of fine  $\alpha$ -Mg and LPSO phase and the improvement of mechanical properties in this alloy is consider to the originated in the high dispersion of the bent LPSO structure and the refinement of those structures and  $\alpha$ -Mg grains. This paper focuses on the microstructure, mechanical properties and creep behavior of the extruded Mg-Zn-YRE alloys.

10:30 AM Tea Break

#### Symposium C: Light Metals and Alloys: Mechanical Properties of Magnesium Alloys

Thursday AM August 5, 2010 Room: C Location: Cairns Convention Centre

Session Chairs: Yoshihito Kawamura, Kumamoto University; Koji Hagihara, Osaka University

#### 11:00 AM

Crystallographic Orientation Dependence of Fatigue Crack Propagation in Rolled AZ31B Magnesium Alloy: *Shigeki Morita*<sup>1</sup>; Nobuyoshi Ohno<sup>1</sup>; Fujio Tamai<sup>2</sup>; Yuji Kawakami<sup>2</sup>; <sup>1</sup>Saga University; <sup>2</sup>Industrial Technology Center of SAGA

Wrought magnesium alloys have HCP (hexagonal close-packed) structure and strong textures have been formed by extrusion and rolling processes. Owing to the microstructures, wrought magnesium alloys show unique cyclic deformation behavoir such as mechanical anisotropy, pseudoelasticity in loadingunloading and asymmetricity of hysteresis loops in fatigue tests, etc. However, the effect of texture on fatigue crack propagation behavior is still insufficiently understood. In the present study, fatigue crack propagation behavior of rolled AZ31B magnesium alloy was investigated. Two specimens were machined from the loading axis parallel to rolling direction; fatigue crack propagation direction is parallel to the c-axis (L-S specimen), and vertical to the c-axis (L-T specimen). Fatigue crack propagation tests were performed on an Electro-hydraulic testing machine (capacity: 9.8kN) with stress ratio R=0.1 and frequency of 10Hz at room temperature in air. Crack propagation rate of L-T specimen was approximately 10 times higher than that of L-S specimen. SEM-EBSD analysis showed the caxis direction is unfavorable for the fatigue crack propagation in polycrystalline magnesium alloy.

#### 11:15 AM

Fatigue Behavior of an Mg-Zn-Y-Zr Alloy in Vacuum at Room and Cryogenic Temperatures: Shangli Dong<sup>1</sup>; Yi Zhang<sup>1</sup>; Gang Lu<sup>1</sup>; Shiyu He<sup>1</sup>; Enhou Han<sup>2</sup>; <sup>1</sup>Harbin Insitute of Technology; <sup>2</sup>Institute of Metal Research, Chinese Academy of Sciences

Fatigue behavior of an as-forged Mg-Zn-Y-Zr alloy has been studied in vacuum at room and cryogenic temperature, as well as in air at room temperature for comparison, by use of a fatigue testing machine equipped with an environmental chamber. The fatigue properties of the examined alloy were evaluated, while the fracture surfaces of the fatigued specimens and microstructure in the region adjacent to fracture surface were examined by scanning election microscope and optical microscope, respectively. It is shown that the fatigue strength of the asforged decreased in the order of in vacuum at cryogenic temperature (-190°), in vacuum at room temperature and in air at room temperature. A longest fatigue life of the allov could be obtained in vacuum at cryogenic temperature, while a shortest one in air at room temperature. Crack initiation site, crack prorogation path and rapid failure zone were distinguishable on the fractured surfaces of the specimens fatigued either in air or in vacuum at room and cryogenic temperatures. Fatigue cracks appeared to generally initiate at surface of the specimens, and many dimples could be found on the rapid failure zone. Dislocation slip was found to be the main deformation mode under the three testing conditions.



#### 11:30 AM

Inhomogeneous Deformation Behaviors and Their Effects on Mechanical Performance in a Warm-Extruded Magnesium Alloy with LPSO Phase: *Tatsuya Morikawa*<sup>1</sup>; Yuuki Mitani<sup>1</sup>; Jun Hirotani<sup>1</sup>; Kenji Higashida<sup>1</sup>; <sup>1</sup>Kyushu University

Inhomogeneous deformation in a dual-phase magnesium allov has been investigated using high-precision markers drawn by electron beam lithography. Mg alloys containing Zn and rare earth elements such as Y have a characteristic microstructure consisting of the LPSO phase and the usual hcp matrix phase. In the as-cast condition, the mechanical performance of this alloy is not so unique, but the process of warm-extrusion around the temperature of 623K causes a remarkable enhancement of its strength without losing ductility. Such superior mechanical properties of this Mg alloy should be closely related to the characteristic of microstructural inhomogeneity induced by plastic deformation. The microstructure developed by the warm extrusion consists of elongated grains with fine-lamellae of LPSO phase and fine-grained matrix of hcp phase, which must enhance microstructural inhomogeneity by the constraint along their grain boundaries. However, the details of inhomogeneous deformation in this alloy have not been clarified yet. In the present work, we employed a high-precision marking method using electron beam lithography in order to measure the local displacement due to tensile deformation. The influence of the characteristic of deformation inhomogeneity on the mechanical property such as strength or ductility of the Mg alloy is discussed.

#### 11:45 AM

Effect of Loading Direction on the High-Cycle Fatigue Behavior of Rolled AZ31 Magnesium Alloy: *Sung Hyuk Park*<sup>1</sup>; Seong-Gu Hong<sup>2</sup>; Chong Soo Lee<sup>1</sup>; <sup>1</sup>Pohang University of Science & Technology; <sup>2</sup>Korea Research Institute of Standards and Science

Rolled AZ31 magnesium (Mg) alloy has a strong basal texture so that its

deformation behavior would be significantly influenced by the loading direction applied. This is because the angle relationship between the crystallographic lattice orientation and the applied load governs the activation of twinning, which can accommodate plastic deformation with significantly lowered stress. These facts imply that the fatigue behavior of the alloy may be dominated by the alternation of the twinning and detwinning during each cycle depending on the applied loading direction. In this study, the effect of loading direction on the high-cycle fatigue behavior of rolled AZ31 Mg alloy was investigated by performing the fatigue test along the rolling direction and the normal direction. The results showed that the cyclic hardening characteristic played a key role on the fatigue deformation because it directly influenced the amount of plastic strain accommodated by twinning. The cyclic hardening of the rolling direction was more severe than that of the normal direction so that larger plastic strain was developed along the normal direction and the reduction in the developed plastic strain with increasing cycles was much greater in the rolling direction.

#### 12:00 PM

Grain Refinement by Combined ECAE/Extrusion and Dieless Drawing Processes for AZ31 Magnesium Alloy Tubes: *Tsuyoshi Furushima*<sup>1</sup>; Tetsuhide Shimizu<sup>1</sup>; Ken-ichi Manabe<sup>1</sup>; <sup>1</sup>Tokyo Metropolitan University

Grain refinement processing by severe deformation, combined Equal-Channel Angular Extrusion (ECAE) processing and conventional tube extrusion, was applied to AZ31 magnesium alloy. By a combination of ECAE processing and tube extrusion, a fabricated tube, with outer and inner diameters of 2 mm and 1 mm, respectively, had fine, homogeneous, and equiaxed grain structure with an average grain size of 1.5 micro\_m. Tensile test results indicate that the fine-grained tubes exhibited a superplasticity potential m value = 0.55. The maximum elongation (688%) was obtained at a temperature of 673K. In addition, compared with tubes only processed by extrusion, ECAE-extruded tubes showed remarkable ductility with an elongation of 245% at a high strain rate. Furthermore, the tubes fabricated by combined ECAE/extrusion process is applied to dieless drawing process without using any tool and die. As a results, dieless drawing limit is enhanced by high strain sensitivity. From these results, the effectiveness of new grain refinement processing for fabricating fine-grained tubes and its application for dieless drawing process was demonstrated experimentally.

#### 12:15 PM

Grain Refinement of Magnesiun Alloy AZ31 under Torsion Extrusion with a Square-Hole Die: *Susumu Mizunuma*<sup>1</sup>; Takamichi Iizuka<sup>1</sup>; Kazuhiro Mitsui<sup>1</sup>; Hidehito Okumura<sup>1</sup>; Masahide Kohzu<sup>2</sup>; <sup>1</sup>Kanagawa Institute of Technology; <sup>2</sup>Osaka Prefecture University

The grain refinement behavior and crystal orientation of Mg alloy AZ31 under torsion extrusion are investigated using a square-hole die. Torsion extrusion, in which the die is rotated during extrusion, is a large strain process used to produce very fine grained materials. In principle, materials can be subjected to limitless strain through a single pass reduction during this process, due to both the high hydrostatic pressure of extrusion and the size immutability of torsion. Optimum conditions were clarified for both the working temperature and the ratio of the die rotation speed to the extrusion speed, resulting in uniformly distributed grains of submicron over the entire cross section of the worked specimen. The crystal orientation of the specimen was revealed by electron backscatter diffraction (EBSD) analysis and compared with a conventional extrusion specimen. In the case of the torsion extrusion, a very strong [0001] texture was observed along the extrusion axis, especially in the central region of the cross section; however, with conventional extrusion, the [0001] direction of many grains tended to become perpendicular to the extrusion axis.

#### 12:30 PM

Sound Insulation Study on Mg Sheet and Mg Honeycomb Panels: Gao Feng Quan<sup>1</sup>; Zhaoming Liu<sup>1</sup>; Xiue Gu<sup>1</sup>; Feng Yan<sup>1</sup>; <sup>1</sup>Dalian Jiaotong University

The sound insulation experiments were conducted to magnesium alloy sheet and magnesium honeycomb panels. It is found that the sound insulation capacity (SIC) of honeycomb panel is higher than that of the sheet in full sound frequency spectra, and the SIC spectra of both single sheet and honeycomb panel is similar with the sound frequency, thus the SIC is increased obviously with the frequency. Comparably, the SIC of honeycomb panels is always higher than that of the sheets, especially at middle range of prequencies 6000~8000 Hz, the sheet has a SIC of 27 to 31dB, and the honeycomb panel has 34-39 dB. The analysis of sound insulation of honeycomb panel is carried out.

#### Symposium E: Solidification, Deformation and Related Processing: Various Aspects of Solidification and Deformation

Thursday AM	
August 5, 2010	

Session Chairs: Wanqi Jie, Northwestern Polytechnical University; Chun Wei Su, Singapore Institute of Manufacturing Technology

Location: Cairns Convention Centre

Room: 2

#### 8:30 AM

Severe Plastic Deformation by Cold Spray: Peter King<sup>1</sup>; Mahnaz Jahedi<sup>1</sup>; <sup>1</sup>CSIRO

Cold Spray is an emerging technology for the solid-state deposition of materials. There are numerous applications for cold spray protective coatings, refurbishment of worn parts, and in the direct manufacture of components. The processes of microstructural evolution during the high-speed impact of cold spray particles were investigated. Particles underwent plastic deformation at very high strain rates, resulting in (a) extensive grain refinement and (b) large inhomogeneities in microstructure. Localization of deformation near the particle interfaces leads to the development of adiabatic shear instabilities, which facilitate interparticle bonding. Cold spray may be compared with other techniques for severe plastic deformation of materials, with the unique property that a wide variety of thermomechanical conditions exist.

#### 8:45 AM

Controlled Synthesis of Iron Compounds Complex Particles by Pulsed Laser Irradiation in Liquids: Zaneta Swiatkowska-Warkocka<sup>1</sup>; Kenji Kawaguchi<sup>1</sup>; Hongqiang Wang<sup>1</sup>; Yukiko Katou<sup>1</sup>; Naoto Koshizaki<sup>1</sup>; <sup>1</sup>AIST

In recent years, there is increasing attention on the morphology and size control of materials synthesized on the micro- and nanoscale due to the fact that morphology and size play very important roles in determining chemical and physical properties of materials. The laser light can serve as a convenient tool to control the size and shape distribution of often inhomogeneous nanoparticles samples. Here we demonstrate a novel method for successive preparation of submicron iron compounds complex spherical particles with controlled sized and tunable phase composition by pulsed laser irradiation in liquids. Source magnetite and hematite colloid solutions were irradiated in various organic solvents by Nd: YAG laser with a 532 nm wave length, 30 Hz repetition and 20~100 mJ/pulse pulse energy. The third harmonic (355 nm) and fundamental (1064 nm) laser lights were also examined. The structure, phase composition, size, morphology and magnetic properties were confirmed by XRD, TEM, SAED, EDS, SEM and SQUID. In our work we investigated how structure and magnetic properties of iron compound nanoparticles evolve according to changing irradiation conditions. It was found that the size, composition and magnetic properties of the obtained particles can be tuned in a good controllable manner.



#### 9:00 AM

**Development of a Magnesium Alloy Rotor with a Pin-Point Gate Mold**: *Young Cheol Lee*<sup>1</sup>; Hyung Ho Cho<sup>1</sup>; In Deok Park<sup>2</sup>; <sup>1</sup>Korea Institute of Industrial Technology; <sup>2</sup>Busan Metropolitan City Hall

Rotor is a key element to determine the performance of a compressor and many attempts have been made to improve the efficiency of compressor by changing the design of rotors. Rotor is usually made of several layers of steel sheets with thin cavities through the steel sheets and aluminium alloys are used to combine the steel sheets through the cavities by high pressure die casting process. Because of its high fluidity and good damping ability, magnesium alloys can be a good alternative for a high efficiency rotor. In this study, magnesium alloys were used for manufacturing rotor by high pressure die casting process using pin-point gate mold. By adopting a pin-point gate system, additional machining was eliminated and casting defects were reduced due to good castability of magnesium alloys

#### 9:15 AM

**Grain Refinement Performance of Al Cast Using Machining Chips**: *Yoshimi Watanabe*<sup>1</sup>; Kenichi Tabushi<sup>1</sup>; Hisashi Sato<sup>1</sup>; Eri Fujiwara<sup>1</sup>; <sup>1</sup>Nagoya Institute of Technology

Microstructure of as-cast Al is often refined by using grain refiner such as Al-Ti, Al-Ti-B and Al-Ti-C alloys. In this study, grain refinement performance of as-cast Al using machining chip of Al in stead of the grain refiner is investigated. At first, the machining chips of pure Al are place in metallic mould. Then, pure Al melt is inserted into the mould with the machining chips. From the microstructure of the as-cast Al using the machining chips, it is found that this machining chip in mould can induce grain refinement of as-cast Al. The increment of the Al chips enhances the grain refinement of the as-cast Al. Moreover, it is shown that preheating the mould can reduce the pore inside as-cast Al using the machining chips. This grain refinement effect by the machining chips would come from the enhancement of cooling rate and the role of the nucleation site. Therefore, it is concluded that the machining chips of Al can enhance the grain refinement of as-cast Al.

#### 9:30 AM

#### Microstructure and Properties Tailoring in Kinetic Spraying: Nickel Coating onto Mild Steel: *Gyuyeol Bae*<sup>1</sup>; Kicheol Kang<sup>1</sup>; Sangmin Ha<sup>1</sup>; Changhee Lee<sup>1</sup>; <sup>1</sup>Hanyang University

A commercially pure nickel (CP-Ni) powder (mean size of ~35 µm) was sprayed onto mild steel substrates using a kinetic spraying system combined with a powder preheating system under different impact conditions (i.e. particle velocities and temperatures). At relatively low velocity (~560 m/s) with temperatures from 773 to 873 K, considerably dense and thick coating (~1150  $\mu m)$  which has ~90% deposition efficiency (DE) could be obtained, while from 298 to 673 K, the DE of the coating was found to be quite low (<25%) and detachment of the coating from the substrate and interparticle cracks were observed. Also, the DE of this thick coating was much higher than that of the coating (~20%) formed at higher velocity (~685 m/s) without powder preheating. The microhardness of the tailored coating (~275 Hv0.1) was ~1.5 times higher than that of the powder (~174 Hv0.1) due mainly to the effect of strain hardening. The local nanoindentation hardness values (2-5.5 GPa) in a severely deformed particle proved the presence of ultra fine grains (~150 nm) confirmed by observations using a transmission electron microscope. The deposition characteristics and coating properties of CP-Ni shown here are coupled to the results of finite element modeling.

#### 9:45 AM

**Observation of Hot Tear Formation by Acceleration Sensor**: *Hisao Esaka*<sup>1</sup>; Yoshiaki Naitoh<sup>1</sup>; Daisuke Uotani<sup>1</sup>; Kei Shinozuka<sup>1</sup>; <sup>1</sup>National Defense Academy

Hot tear is the one of the biggest problems of cast products. There have been many researches in order to understand the formation mechanism of hot tear. However, it is still uncertain when a hot tear initiates and how it propagates. Thus, the preliminary research has been carried out to observe the vibration of permanent mold which correspond to the initiation or propagation of hot tear. Al-2.0 wt% Cu alloy was used for test alloy. The signals of acceleration sensor were recorded at an interval of 5 ms. After casting, the vibration, which lasted scores of milliseconds, were observed intermittently. This may indicate that hot tear initiates or propagates intermittently and the strain due to contraction of solidification or cooling is relaxed by hot tear.

#### 10:00 AM

**Crack Initiation in Bending Deformation in a Corson Series Copper Alloy Related to the Microstructure**: *Tomonori Nakanome*<sup>1</sup>; Goroh Itoh<sup>2</sup>; Yoshiki Yamamoto<sup>3</sup>; Nobuhide Itoh<sup>2</sup>; <sup>1</sup>Graduate student, School of Science and Engineering, Ibaraki University; <sup>2</sup>Department of Mechanical Engineering, Ibaraki University; <sup>3</sup>Hitachi Cable, Ltd.

In this study, crack initiation in bending deformation in a Corson series copper alloy related to its microstructure was investigated. Cold-rolled sheets were solution-treated, water-quenched and finally aged. Test pieces were cut from the sheets in the longitudinal (G.W.: Good Way) and transverse (B.W.: Bad Way) directions. Then, microscopic crystal orientation was characterized by EBSD and subsequently bend deformation was conducted. Exactly the same area where the EBSD characterization was made was closely observed with an SEM. Microscopic strain of individual grains was measured from the pair of micrographs before and after bending deformation, and it was confirmed that the measured strain of the grains increased with increasing the maximum Schmidt factor and with decreasing Taylor factor values. The two grains adjacent to the cracked grain boundary have a larger difference between each other in the measured microscopic strain, maximum Schmidt factor and Taylor factor values than the grains adjacent to uncracked boundaries. From these results, it was deduced that a crack initiates around a grain that is hard to be deformed, i.e., has low maximum Schmidt factor and high Taylor factor values, and is surrounded by grains that are easy to be deformed.

#### 10:15 AM Tea Break

#### Symposium E: Solidification, Deformation and Related Processing: Solidification I

Thursday AM	Room: 2
August 5, 2010	Location: Cairns Convention Centre

Session Chair: Hur Bo Young, Gyeongsang National University

#### 11:00 AM Invited

Heterogeneous Nucleation and Grain Formation on Spherical and Flat Substrates: *Ma Qian*<sup>1</sup>; <sup>1</sup>The University of Queensland

This work provides an overview of the recent understanding of the classical models of heterogeneous nucleation in terms of the effect of the substrate morphology and size. Turnbull was the first to realise that the linear dimension of a flat substrate (d) in Volmer's classical model must be greater than 2r\* (r\*: critical embryo radius) for effective grain formation. The recently developed free growth model has reached the same criterion, which now appears to apply to grain formation on both potent and non-potent substrates. This offers a different perspective on assessing the applicability of the classical models to nucleation on potent substrates. Fletcher's spherical substrate model extends perspectives on Volmer's model and revealed the negative size effect of spherical substrates on nucleation. Significant similarities exist between the two models when assessed using a novel thermodynamic approach, which gives new insights into nucleation. An analysis of the physical implications of Fletcher's model suggests that the sizes of effective nucleants should be chosen to be  $> 10r^*$  and close to  $40r^*$  but there is no need to be >  $40r^*$ . The implications of these developments are discussed in the context of cloud seeding, freezing of water and solidification of liquid metals.

#### 11:15 AM

Analysis of an Equiaxed Dendrite Growth Model with Comparisons to In-Situ Results of Equiaxed Dendritic Growth in an Al-Ge Alloy: *Shaun McFadden*<sup>1</sup>; Paul Schaffer<sup>2</sup>; David Browne<sup>3</sup>; Ragnvald Mathiesen<sup>4</sup>; <sup>1</sup>Dublin Institute of Technology; <sup>2</sup>Hydro Sunndalsøra; <sup>3</sup>University College Dublin; <sup>4</sup>NTNU

The Lipton Glicksman Kurz (LGK) growth model is commonly used to predict growth rates for equiaxed dendrites in solidifying mushy zones. However, the original LGK method treats an isolated dendrite growing in an infinite volume of liquid. In an equiaxed mushy zone, with multiple nucleation events, thermal and solutal interactions take place between the equiaxed dendrites. An extended version of the LGK model was developed that allows for solute build-up ahead of the dendrites. To investigate the validity of the model, comparisons were made with results obtained from in-situ synchrotron X-ray videomicroscopy of solidification in a Bridgman furnace of an Al-12wt.%Ge alloy inoculated with Al-Ti-B grain refiner. The resulting structure was a mixture of directional and equiaxed dendrites. In the early stages of the solidification sequence, two neighbouring equiaxed dendrites nucleated, grew, and ultimately impinged upon each other. The centre-to-centre distance between the two equiaxed dendrites was maintained at 700 µm. In the early stages of growth the dendrites were solutally isolated from each other; however, due to the gradually diminishing tip interspacing, the dendrites showed increasing solutal interaction in the final stages of growth. Comparisons between the original LGK and extended LGK models will be presented for discussion.





#### 11:30 AM

The Influence of External Mechanical Stresses on Agglomeration and Bending of Solidifying Crystals: Somboon Otarawanna<sup>1</sup>; Christopher Gourlay<sup>2</sup>; Hans Laukli<sup>3</sup>; Arne Dahle<sup>4</sup>; <sup>1</sup>National Metal and Materials Technology Center (MTEC); <sup>2</sup>Imperial College London; <sup>3</sup>Hydro Aluminium; <sup>4</sup>The University of Queensland

Agglomeration and bending of primary crystals during solidification are important phenomena influencing many aspects of casting processes and often affect the microstructure of the as-cast component. In this work, agglomeration and bending of equiaxed crystals have been studied by microstructural characterization of hypoeutectic Al cast specimens produced by near-static cooling, gravity die casting and high-pressure die casting (HPDC), where the solidifying crystals experience different levels of mechanical stresses. Grain misorientation data, which is linked to crystal agglomeration and bending behavior during solidification, was acquired by electron back-scatter diffraction (EBSD) technique. The number fraction of low-energy grain boundaries in HPDC samples was substantially higher than in gravity die-cast and "statically cooled" samples. This is attributed to the high amount of shear applied on the solidifying alloy, which promotes crystal collisions and agglomeration. In-grain misorientations were found to be significant only in branched dendritic crystals which were subjected to significant shear stresses. This is related to the increased bending moment acting on long, protruding dendrite arms compared to more compact crystal morphologies.

#### 11:45 AM

The Effect of Solute Distribution in Cu-Ag Alloy during Directional Solidification with Different Growth Velocity: *Woo-Hyun Lee*<sup>1</sup>; Bok-Hyun Kang<sup>1</sup>; Ki-Young Kim<sup>1</sup>; Hoon Cho<sup>2</sup>; <sup>1</sup>Korea University of Technology and Education; <sup>2</sup>Korea Institute of Industrial Technology

Cu-Ag allov is well known as a suitable combination of high strength and high conductivity. The second phase appears as an intercell, interdendrite and Cu-Ag eutectic phase in this alloy. The distribution of Ag phase in the Cu matrix affects the electrical properies. Since the presence of the Ag phase in the Cu matrix disrupts the electrical conductivity, less Ag in the Cu matrix is better in terms of electrical properties. The control of temperature gradient ahead of the solid-liquid interface and growth velocity is important parameters in the solute distribution during solidification. In this study, the effect of the solute distribution for investigated electrical conductivity during directional solidification for the Cu-8wt%.Ag was studied. The Cu-8wt.% Ag was melted in the bridgeman apparatus with super kanthal and grown with different growth velocities. The growth velocity during directional solidification are 1µm/s to 200µm/s. As the growth velocity was increased, Ag contents in the Cu matrix was decreased. The tendency of electrical conductivity decreased with increasing the growth velocity. The maximum electrical conductivity occurred at approximately was about 87% IACS with directional solidification in Cu-8Ag alloy. The effect of solute distribution with different growth rate on the electrical conductivity results was discussed.

#### 12:00 PM

Undercooling Behavior and Solidification Microstructure Evolution of Sn-Cu-Ni Solders Modified by Minute Amount of Mixed Rare Earth La-Ce: Minbo Zhou<sup>1</sup>; Xiao Ma<sup>1</sup>; Xin-Ping Zhang<sup>1</sup>; <sup>1</sup>South China University of Technology

Sn-Cu-Ni (SCN) alloy solders have attracted considerable attention from electronic packaging manufacturers and suppliers owing to silver- and lead-free feature and low-cost advantage of the solders. However, there is still a lack of indepth understanding on composition optimization and microstructure control of the solders. In the present study, the influences of the addition of minute amount of mixed rare earth (MRE) La-Ce, in the range of 0.05-0.50wt%, on melting characteristics, undercooling behavior, solidification microstructure evolution and wettability of Sn-0.7wt%Cu-0.05wt%Ni-xMRE (x=0.05, 0.10, 0.25, 0.50 wt%) alloy solders were investigated. The results show that the addition of rareearth La-Ce has brought about an obvious decrease of the undercooling for Sn-Cu-Ni series of solders, consequently the growth of primary solidification phase of Cu(Ni)-Sn type intermetallic compound (IMC) has been suppressed and the β-Sn phase has exhibited a microstructural transition from dendritic grain to equiaxed grain with increasing the amount of MRE La-Ce added to the solder. The results also manifest that the solder of adding 0.05wt% of MRE La-Ce shows superior wettability on Cu substrate, and thereafter a larger addition amount of MRE La-Ce can result in deterioration of the solder's wettability.

#### 12:15 PM

## DC Casting of Mg Alloy under Combined Electromagnetic Field with Ultrasonic Field: *Cui Jianzhong*<sup>1</sup>; <sup>1</sup>Northeastern University

The effects of low frequency electromagnetic field(LFEC) and ultrasonic field(US) on the microstructures, macrosegregation of alloying elements and the

mechanical properties of AZ80 alloy DC cast were studied in this paper. Results shows that both LFEC and US fields can refine the grains of the ingots, which resulted in increase in mechanical properties and uniform of alloying element distribution. The effective refinement takes place in the edge of ingots when applied LFEC. However, the effective refinement takes place in the center of ingots when applied US. Combined the characteristics of LFEC and US fields a new process for DC casting of Mg- ECUS casting, was developed by application of combining LFEC with US, by which the grain size was refined significantly in the whole ingots everywhere and more uniform, and the mechanical properties of the ingots were improved.

#### 12:30 PM

#### Improving the Microstructures and Mechanical Properties of Hypereutectic AI-Si Alloys by Spray Forming Technique: Hua Cui<sup>1</sup>; Longgang Hou<sup>1</sup>; Jishan Zhane<sup>1</sup>: <sup>1</sup>USTB

The hypereutectic Al-Si alloys containing Fe element with different Cr addition (0-2.0 wt.%) had been prepared by spray forming technique. With adding Cr element into Al25Si5Fe3Cu (wt.%, signed as 3C) alloy, the long needlelike d-Al<sub>4</sub>FeSi<sub>2</sub> phases in 3C alloy were almost substituted by skeletal  $\alpha$ -Al(Fe,Cr)Si phase. But in as-deposited preforms, the short-rod  $\beta$ -Al<sub>5</sub>FeSi phase (< 10  $\mu$ m), as the dominant Fe-bearing phase in spray-formed 3C alloy besides refined Al matrix and primary Si phase, became the granular  $\alpha$ -Al(Fe,Cr)Si phase with sizes less than 3-5 µm in Cr-added hypereutectic Al-Si alloys, which could be beneficial to the enhancement of mechanical properties. Some plate-like ß-Al<sub>s</sub>(Fe,Cr)Si phases (~20 µm) in as-deposited C20 alloy are disappeared after hot extrusion. The results of different isothermal treatments show that the granular  $\alpha$ -Al(Fe,Cr)Si phases in spray-formed Cr-added alloys possesses excellent thermal stability without any observable coarsening or growth, contributing to obtain good high temperature properties. The microhardness, yield strength and ultimate tensile strength of the spray-formed and hot-extruded alloys are increased to 173.1 MPa (increased by 47.1 %), 309.76 MPa (increased by 115.1 %) and 364.39MPa (increased by 60.6 %), respectively, accompanied by the reduction of elongation from 0.8 % to 0.33 %

#### Symposium F: Modelling and Simulation of Microstructures and Processes: Phase Field and Related Modelling Techniques

Thursday AM August 5, 2010 Room: 5 Location: Cairns Convention Centre

Session Chairs: Yves Brechet, Grenoble-INP; M. Kohyama, National Institute of Advanced Industrial Science and Technology

#### 11:00 AM Keynote

Modeling Displacive-Diffusional Coupling in Phase Transformation and Plastic Deformation: Yunzhi Wang<sup>1</sup>; <sup>1</sup>Ohio State University

Microstructural evolution in materials often involves coupled displacive and diffusional processes as a rule rather than as an exception. For example, structural phase transformations often involve coupled lattice shear, shuffle, and diffusion. Dislocation shearing of ordered precipitates can couple strongly to chemical reordering during plastic deformation. In these processes it is the coupling that governs the rate of transformation and deformation. On the other hand, dislocation plasticity will change precipitate microstructure, which in turn will alter dislocation dynamics. Mechanistic studies of these mechanochemically coupled processes require modeling capabilities at micrometer, nanometer and even atomistic length scales but diffusional time scales. In this presentation, recent efforts in developing multi-scale modeling approaches by integrating ab initio calculations and atomistic simulations with continuum phase field method at all length scales will be reviewed. Examples will be given to demonstrate quantitative aspects of the approaches in (a) predicting defect size and energy, and thermally activated processes of defect nucleation and migration, utilizing directly ab initio information as model input, and (b) identifying and incorporating transformation and deformation mechanisms in combination with advanced experimental characterizations.

#### 11:20 AM Keynote

Application of Phase Field Modeling to Phase Transformations in Steel: S. G. Kim<sup>1</sup>; P-R. Cha<sup>2</sup>; W. T. Kim<sup>3</sup>; J. K. Lee<sup>4</sup>; <sup>1</sup>Kunsan National University; <sup>2</sup>Kookmin University; <sup>3</sup>Cheongju University; <sup>4</sup>POSCO

A multi-phase field model for diffusional transformation was developed and applied to phase transformation in steel i.e. primary austenite  $\rightarrow$  ferrite transformation and following eutectoid reaction. The primary diffusional



transformation took place through two different processes. Firstly, ferrite grains nucleated heterogeneously from prior austenite grain corners or grain boundaries grow preferentially along austenite grain boundaries, forming grain boundary(g. b.) allotriomorph ferrite. Secondly, the g.b. ferrite grow inward toward the center of austenite grains. Morphology change from g.b. allotriomorph to cellular ferrite took place due to the occurrence of interface instability induced by C partitioning. The effects prior austenite grain size, cooling rate, contact angle at triple junction, nucleation rate, interface mobility on microstructural evolution and transformation kinetics will be presented. Also the effect of stress on ferrite morphology and transformation kinetics will be discussed.

#### 11:40 AM Keynote

Mechanisms for the Formation of Self-Organized Egg-Type Microstructure by Phase Field Modeling: Rongpei Shi<sup>1</sup>; Cuiping Wang<sup>1</sup>; *Xingjun Liu*<sup>1</sup>; Yunzhi Wang<sup>2</sup>; <sup>1</sup>Xiamen University; <sup>2</sup>The Ohio State University

Recently self-organized egg-type composite powers, where one alloy encase another, were produced successfully [1] by conventional gas atomization from a variety of alloys that have a large miscibility gap in the liquid state. Due to the extremely fast process, a quantitative understanding of the formation mechanism of the egg-type microstructure is still lacking. In this work, computer simulations using the phase field method were carried out to study microstructural evolution during gas atomization of liquid immiscible Fe-Cu alloys. The model takes into account simultaneously phase separation via liquid spinodal decomposition, fluid flow, and Marangoni motion, and the effect of the free surface. The simulation results well document the entire formation process. The relative contributions from each of these processes are investigated as function of droplet size. Results from systematic parametric study of the effects of major material and processing parameters (including alloy composition, droplet size, temperature gradient and surface energy difference between two liquid phases) on the morphology and formation kinetics of the egg-type microstructure will be presented. This work provides an important theoretical basis for the design and development of the self-organized egg-type composite materials.

#### 12:00 PM

#### A New Algorithm of Phase-Field Approach to Polycrystalline Dendritic Solidification in Two Dimensions and Three Dimensions: *Tao Jing*<sup>1</sup>, <sup>1</sup>Tsinghua University

A new algorithm of phase-field model is developed to simulate polycrystalline dendritic solidification growth in undercooled melts. The algorithm adopts a single phase-field order parameter model incorporated with the anisotropy of solid-liquid interfacial energy and mobility. To describe space crystallographic misorientations of any grain-to-grain, the algorithm via artificial orientation-field in whole growth region allow the simulations of 2-D or 3-D polycrystalline dendrites with arbitrary orientations in a straightforward way. The model validation is performed by comparing the simulations with the theory analytical results and experimental information for both single and multi-grain dendritic growth, which demonstrates the quantitative capabilities of the proposed algorithm. The simulations qualitatively reproduce realistic 2-D and 3-D polycrystalline dendrites of equiaxed and columnar pattern with various orientations and well-developed side branches.

#### 12:15 PM

Microscopic Phase-Field Simulation of Strain Induced Pre-Precipitation Phase in Ni-Cr-Al Alloy: Yan-Li Lu<sup>1</sup>; Zheng Chen<sup>1</sup>; Jing Zhang<sup>1</sup>; Yong-Xin Wang<sup>1</sup>; <sup>1</sup>Northwestern Polytechnical University

Based on the microscopic phase-field field dynamic model, the effect of elastic strain energy on the pre-precipitation phase in Ni<sub>75</sub>Cr<sub>14.5</sub>Al<sub>10.5</sub> alloy is studied. The simulation results show that when the strain energy is zero, there is no pre-precipitation phase formed, the L1<sub>2</sub> phase is firstly precipitated from the disordered matrix, whereafter, the DO<sub>22</sub> phase is precipitated. When the strain energy is not zero, although the precipitation sequence remained unchanged, i.e., L1<sub>2</sub> phase is precipitated first, and DO<sub>22</sub> phase comes next, the island pre-precipitation phase with L1<sub>0</sub> structure is formed before L1<sub>2</sub> phase. The larger the strain energy is, the more obvious the characteristics of pre-precipitated L1<sub>0</sub> phase appear. At the same time, the evolution of microstructure, volume fraction and order parameter is mainly analyzed.

#### 12:30 PM

#### **Phase Field Modelling of Dendrite Fragmentation during Thermal Shock**: *Zhipeng Guo*<sup>1</sup>; Jiawei Mi<sup>1</sup>; Patrick Grant<sup>1</sup>; <sup>1</sup>University of Oxford

Dendritic grain growth and its break-up under conditions of thermal shock have been investigated using numerical modelling and experiments using a low temperature transparent organic alloys as analogues of metallic binary systems. The dendritic grain growth, fragmentation and evolution of a succinonitrile transparent alloy during solidification subject to an ultrasonic pulse to agitate the liquid in the vicinity of the dendrite front or a sudden thermal shock due melt convection have been investigated by using high speed digital video (~8000 frames per second) and simulated using a 2D phase field model including the coupled effects of heat flow and solute redistribution.Modelling and experimental investigations indicated that thermal shock and ultrasonic pulse enhance the fragmentation of dendritic grains, and their flow away from the dendrite, during which they undergo rapid changes in size and morphology. The dendrite fragments are known technologically to play an important role in providing nucleation embryos for the growth of equiaxed grains, which are generally preferred in castings. The enhancement of fragmentation refines the solidification microstructures and provides a potent intrinsic grain refinement effect. The underlying physics controlling fragmentation are discussed and future directions to enhance further these important effects are presented.

#### 12:45 PM

**Phase Field Simulation of Ni**<sub>4</sub>**Ti**<sub>3</sub> **Precipitation in Porous NiTi Shape Memory Alloys under Applied Stresses**: *Changbo Ke*<sup>1</sup>; Xiao Ma<sup>1</sup>; Xin-Ping Zhang<sup>1</sup>; <sup>1</sup>South China University of Technology

Porous NiTi shape memory alloys have drawn a great deal of attention in recent years owing to the combined virtue of shape memory effect, superelasticity and adjustable mechanical properties. Generally, porous NiTi alloys may undergo thermomechnical treatment which brings about precipitation of Ni4Ti3 particles, consequently martensitic transformation occurs in complicated paths and thus superelasticity of the alloys may change. It is now imperative to study precipitation behavior of Ni4 Ti3 in porous NiTi alloys and deepen our understanding of the effect of Ni Ti, precipitates on martensitic transformation and supereleasticity. This study presents simulation of evolution of multiple Ni4Ti3 variants during stress assisted aging of NiTi alloys containing nano-scale pores with different sizes, by using phase field approach. The simulation shows that under a high applied stress more Ni4Ti, particles precipitated around pores than that under a low stress regardless of pore size; also the larger pores can "capture" more precipitates while less particles precipitated around smaller pores. Moreover, the precipitation of Ni4Ti, particles exhibits different regional preferences near pores. The applied [100]B, stress can cause most particles to precipitate in near-pore region along [010]B<sub>2</sub>, while [010]B<sub>2</sub> stress along [100]B<sub>2</sub>. The uniaxial compressive stress can result in inhomogeneous Ni<sub>4</sub>Ti<sub>3</sub> precipitation around pores.

#### Symposium G: Thin Films and Surface Engineering: Process Chemistry and Engineering II

Thursday AM August 5, 2010 Room: 8 Location: Cairns Convention Centre

Session Chairs: Kyosuke Yoshimi, Tohoku University; Mingxing Zhang, The University of Queensland

#### 8:30 AM Keynote

Crater Eruption Induced by High Current Pulsed Electron Beam (HCPEB) Treatment: *Thierry Grosdidier*<sup>1</sup>; XiangDong Zhang<sup>2</sup>; Jiang Wu<sup>1</sup>; JianXing Zou<sup>2</sup>; Kemin Zhang<sup>2</sup>; Ying Qin<sup>1</sup>; Shengzhi Hao<sup>1</sup>; Chuang Dong<sup>1</sup>; <sup>1</sup>Dalian University of Technology; <sup>2</sup>University of Metz

High current pulsed electron beam (HCPEB) is a fairly new technique for surface modifications that can improve surface properties of metals. One of the negative effects induced by HCPEB is the formation of craters. The consequences of crater formation are an increase in surface roughness and the formation in the near-surface layer of local regions with highly nonuniform strain-stress states. These changes can impair the corrosion-resistance by promoting pitting. Recent investigations have shown that under proper treatment, crater formation can be used to remove the impurities on the surface layer via a so-called "selective purification" effect and that improved corrosion resistance could be obtained when the density of carter was controlled. The aim of the present study is to determine the effect of processing parameter on the nucleation and growth of craters in different materials including steels, bulk metallic galsses and single crystals of different orientation. The effects of structural defects (precipitates, grain boundary) and the energy imparted to the material will be emphasis.

#### 8:50 AM

**CVD and PVD Techniques and their Applications in Thin Film Solar Cells**: *Lishi Wen*<sup>1</sup>; Aimin Wu<sup>1</sup>; Guifeng Zhang<sup>1</sup>; Fuwen Qin<sup>1</sup>; Xiaona Li<sup>1</sup>; Yuanjun Su<sup>1</sup>; Penghui Fan<sup>1</sup>; Chuang Dong<sup>1</sup>; <sup>1</sup>Dalian University of Technology

Silicon-wafer-based solar cells have been the most successful in the photovoltaic market. To meet the demand from sustaining growth of 20-25% up to 2020, as envisioned by the road map for the US industries, thin film depositions of amorphous or crystalloid silicon on low-cost large area substrates at low temperature are important alternatives. Many varieties of thin film solar cells



have been developed using a-Si:H, or a-Si:H/c-Si:H to increase the efficiency and to lower the cost. Chemical vapor deposition (CVD) techniques are the most widely used for making silicon thin film solar cells, and physical vapor deposition (PVD) techniques are just emerging. In this work, we will introduce several CVD and PVD techniques and their applications in silicon thin film solar cells. The general situation in China is also summarized. Research works in the Key Laboratory of Materials Modification are specially focused covering the use of PECVD, Hot-wire CVD, ECR-PECVD, and non-equilibrium magnetron sputtering in the preparations of silicon thin films.

#### 9:05 AM

Enhancement of Photocatalytic Reaction of Titanium Dioxide Film by Surface Texturing: Jun Shimizu<sup>1</sup>; Libo Zhou<sup>1</sup>; Kaoru Takamori<sup>1</sup>; Hirotaka Ojima<sup>1</sup>; Takeyuki Yamamoto<sup>1</sup>; Han Huang<sup>2</sup>; <sup>1</sup>Ibaraki University; <sup>2</sup>The University of Queensland

This study aims to clarify the influence of surface topography on the photocatalytic reaction of TiO<sub>2</sub> film surface. In this report, a textured TiO<sub>2</sub> film surface was produced by conducting anodic oxidation on a titanium plate with microcutting grooves, and its wettability was evaluated. Micro grooves were generated by the cutting on a 3-axis NC control precision machine tool using a single point diamond tool with a tip radius of several hundred nanometers. Anodic oxidation experiments were conducted on the titanium substrates with a large number of microcutting grooves by using self-developed equipment under several tens volts with diluted acetic acid as the electrolyte. It was found that the wettability of the TiO<sub>2</sub> film surface textured by the microcutting was superior to that of the polished and oxidized surface under the irradiation environment of ultraviolet rays. The results thus demonstrated that the wettability of photocatalytic film surfaces can be improved by increasing both the surface roughness and actual surface area. The effectiveness of various manufacturing techniques for texturing titanium dioxide film surfaces was also evaluated in this paper.

#### 9:20 AM

**Preparation and Characterization on Cellulose Nanofiber Film**: *Liyuan Zhang*<sup>1</sup>; Takuya Tsuzuki<sup>1</sup>; Xungai Wang<sup>1</sup>; <sup>1</sup>Deakin University

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Cellulose is the most abundant natural polymer on earth and also a kind of sustainable and renewable resource. In nature, cellulose polymers, which are the major constituent of plant cell walls and are also produced by some bacteria. have diameters less than 100 nanometres. These cellulose nanofibres have many outstanding properties such as a large surface-to-volume ratio, a high Young's modulus, high strength, good transparency and a very low coefficient of thermal expansion. In this study, cellulose nanofibers from wood were obtained using chemo-mechanical and freeze drying techniques and thin films made of these wet and dry cellulose nanofibers have been produced. Micro and nano morphology was carried out by optical microscope and scanning electron microscopy (SEM) separately. SEM image analysis revealed that thin films were constituent of the cellulose nanofibers with the average diameter around 52nm. Other prosperities were also characterized, including the degree of crystalline by X-ray diffraction, mechanical properties by tensile tests, dynamic mechanical properties by dynamic mechanical analyser (DMA), regular light transmittances, thermal properties using differential scanning calorimetry (DSC). This foldable, strong, low-CTE and optically translucent cellulose nanofiber film has many potential applications such as micro/nano electronic devices, biosensors and filtration media, etc.

#### 9:35 AM

Surface Treatment of 316 Stainless Steel by High Current Pulsed Electron Beam: *Shengzhi Hao*<sup>1</sup>; Yang Xu<sup>1</sup>; Mincai Li<sup>1</sup>; Chuang Dong<sup>1</sup>; <sup>1</sup>Dalian University of Technology

High current pulsed electron beam (HCPEB) is now developing as a useful tool for surface treatment of materials. When concentrated electron flux transferring its energy into the surface layer of target material within a short pulse time, the coupled thermal and stress processes would lead to the formation of metastable microstructures with improved properties. In the present work, the HCPEB treatment of YG8 hard alloy was carried out and the microstructural changes in modified surface layer were characterized with optical microscopy, X-ray diffractometry and secondary electron microscopy (SEM) techniques. The mechanical properties of modified surface were measured by microhardness and friction wear testing machines. The evolution regularities of surface microstructure occurring in HCPEB treatments were discussed combining with their influence on mechanical performance.

#### 9:50 AM

The Addition of Silica Nanoparticles with Different Sizes for a Silica Film on Stainless Steel without Crack Formation: *Hohyeong Kim*<sup>1</sup>; Gyuntak Kim<sup>1</sup>; HeungYeol Lee<sup>1</sup>; Taejin Hwang<sup>1</sup>; <sup>1</sup>KITECH

Pure inorganic protective silica film was tried on the surface of stainless steel using a sol-gel chemical route. Coating sol was prepared through the hydrolysis and polycondensation of tetraethoxysilane (TEOS) and methyltriethoxysilane (MTES) in an ethanol solution followed by an addition of two different sets of silica nanoparticles. Then the film was deposited on the stainless steel substrate by a dip coating. It was intended that a denser and thicker film would be obtained even after a lower heat treatment at as low as 200°C by mixing the very small and the larger particles at the same time. The sufficient consolidation of the film was possible because of the high surface activity of the smaller nanoparticles and the film thickening was also possible because of the filling effect of the larger particles. The prepared film showed enhanced adhesion when compared with a silica film without particles addition. The film also showed improved protectability against corrosion. For the characterization of the film scanning electron microscopy (SEM), scratch test, ellipsometry and electrochemical impedance analysis were performed.

#### 10:05 AM

Preparation and Property of Fe-Doped DLC Multilayer by Ion Sources: Ni Ren<sup>1</sup>; Z.J. Ma<sup>1</sup>; Dongcai Zhao<sup>1</sup>; G.J. Xiao<sup>1</sup>; S.H. Wu<sup>1</sup>; <sup>1</sup>Lanzhou Institute of Physics, National Key Lab of Science and Technology on Surface Engineering

A novel technique has been successfully developed to grow Fe ion doped diamond-like corbon (DLC) multilayer, with the modified linear ion sources(LIS), on Si(100)substrates. The Fe ions, generated in the specially designed ion source and accompanying the argon ions were implanted into one DLC layer previously grown by pulsed arc ion plating during argon ions sputtering and etching. The Fe-DLC multilayer can be grown by alternating growth of DLC layer and Fe ion implantation. The microstructures and mechanical properties of Fe-DLC films were characterized with X-ray photoelectron spectroscopy(XPS)and Raman spectroscopy. The results show that the Fe ion implanted multilayer significantly improves its mechanical properties. As compared with the conventional DLC films, its stress drops to 3.9 GPa from 4.5 GPa, its friction coefficient is down to 0.1 from 0.14, and its hardness remains unchanged.

#### 10:20 AM

Study on the Determination of Deleterious Element Pb, Cd and Hg in Coating-Plating Surface of Coating-Plating Material: Yu Yuanjun<sup>1</sup>; <sup>1</sup>Ansteel

The method principle and application of determination on deleterious element Pb, Cd and Hg with the inductively coupled plasma mass spectrometry was introduced in the article. The efficient distillation method was studied through the leaching experiment. The fast and accurate method of determination on trace deleterious element Pb, Cd and Hg in coating-plating material surface with the inductively coupled plasma mass spectrometry technology was established through the establishment of ICP-MS analysis mode, the obviation of the polyatomic ions interference and the research on the matrix effect and instrumentation drift calibration method. The detection lower limit can up to ppb level. The pricision is lower than 5.0%, and the recoveries are between 95.0%~105.0%. The method can provide the guideline on the evaluation of the coating-plating material, increase the product additive value, and improve the international trade of the metallurgy coating-plating product.

10:35 AM Tea Break

#### Symposium G: Thin Films and Surface Engineering: Surface Engineering and Coatings

Thursday AM August 5, 2010 Room: 8 Location: Cairns Convention Centre

Session Chair: Carlos Levi, University of California, Santa Barbara

#### 11:00 AM Keynote

Near-Surface Engineering of Silicon Using Nanoindentation: James Williams<sup>1</sup>; <sup>1</sup>Research School of Physics and Engineering

Nanoindentation with a sharp diamond tip can induce phase transformations in silicon at room temperature. Following indentation the end phases can either be crystalline (a mixture of high pressure phases, so-called Si-III and Si-XII) or amorphous (a-silicon) depending on the pressure release rate. In this paper we review this field: in particular our experimental work that is aimed at understanding and exploiting this phase transformation behavior. We describe indentation experiments that lead to the production of nanoscale regions having very different (insulating and/or conducting) electrical properties to that of the surrounding matrix. We furthermore demonstrate that the Si-XII phase is a semiconductor that can exhibit a range of intriguing properties after indentation, including electrical activation with boron and phosphorus. The high pressure phases are also found to transform to conducting polycrystalline Si-I at temperatures as low as 200°C but that the a-Si phase remains amorphous and electrically insulating



at such temperatures. In-situ and ex-situ electrical measurements, atomic force microscopy, micro-Raman spectroscopy and transmission electron microscopy have been used to determine the structure, phase composition and electrical properties of the phase transformed zones. Using this method, we demonstrate the ability to 'write' conducting and insulating patterns in Si at room temperature.

#### 11:20 AM

#### Triangular Dislocation Loop Model for Surface Displacement in Indented Thin Films: *Shinji Muraishi*<sup>1</sup>; <sup>1</sup>Tokyo Institute of Technology

Indentation displacement is important factor for derivation of Young's modulus and Hardness measurement, which is influenced by residual stress due to the size of plastic domain enclosed by dislocation arrays. In present study, indentation displacement by Berkovich pyramid has been calculated by triangular dislocation loop model, which constructed from L-shape angular dislocations in semiinfinite half space. Contour of the displacement largely influenced by superposed triangular loops, where elastic recovery at the center of pyramidal tip amounts to 47% of indentation plastic displacement. The AFM and TEM observation reveal that continues surface pile-up in Al film / Si substrate is almost consistent with the theoretical recovery ratio, whereas intensive local shear displacement in Al<sub>2</sub>Cu on Si yields 2 times larger than the present calculation. The elastic recovery and hardness behavior in film/substrate composite has been discussed from dislocation image force in two phase material.

#### 11:35 AM

Patterning of Gold Nanoparticles on Silicon and Silicon-Dioxide by Nanoindentation: Simon Ruffell<sup>1</sup>; Dinesh Venkatachalam<sup>1</sup>; Avi Shalav<sup>1</sup>; Robert Elliman<sup>1</sup>; <sup>1</sup>Australian National University

Controlled growth of metal nanoparticles on semiconductor or oxide surfaces is a research topic of current interest for nanoscale opto-electronic device applications. In particular, Au nanoparticles attract great attention due to their catalytic properties and are extensively used for the synthesis of semiconductor and oxide nanowires as well as components of future plasmonic devices Recently, a range of lithographic techniques have been developed and utilized for nanoscale patterning of such particles. In this study, we demonstrate a novel pre-patterning process based on nanoindentation which allows selective nucleation of size-controlled Au nanoparticles on Si and SiO<sub>2</sub> surfaces. This is a simple, maskless, one-step process where the topography of the surface is modified to nanometre resolution by nanoindentation. Sub-micron ordered arrays of Au nanoparticles with narrow particle size distributions of average size ~1  $\mu$ m to ~20 nm are achievable using this process. Au nanoparticles deposited by physical vapour deposition techniques decorate the indented Si surface upon high temperature treatment. Patterned Au nanoparticles have also been formed on a sub 100 nm thermal SiO<sub>2</sub> film on Si(Au) by performing indentation such that the residual impression penetrates through the oxide thus exposing bare Si and thus Au in specific locations.

#### 11:50 AM Invited

**Surface Nanocrystallization and Surface Alloying of Al Alloys**: *Mingxing Zhang*<sup>1</sup>; Haiwei Chang<sup>1</sup>; Yinong Shi<sup>1</sup>; <sup>1</sup>The University of Queensland

Surface mechanical attrition treatment (SMAT) is one of the most effective technologies to activate the surface of pure Al and Al alloys through surface nanocrystallization. Experimental results showed that diffusion rate of Zn in Al alloys can be significantly increased due to the nanostructured grains generated by SMAT in the surface layers. Therefore, the surface alloying treatment temperature can be reduced down to 160°C. The thermal stability of the nanostructured grains generated by SMAT depends on the grain boundary structure of the nanometre scaled grains. For high angle grain boundaries, the nanostructure is relative stable up to temperature of 350°C. However, nanostructured grains with low angle grain boundaries tend to coarsening below 200°C. In addition, the thermal stability of nanostructured grains is also affected by the secondary hard particles segregated along the grain boundaries. It has been observed that, nanometre scaled grains in Al-Si alloy is more stable than those in pure Al. The actual influence of Si on the mechanism of surface nanocrystallization by SMAT in Al alloys has also been discussed.

#### 12:05 PM

Superior Multifunctional Coatings Using Modulated Pulse Power Sputtering: Brajendra Mishra<sup>1</sup>; John Moore<sup>1</sup>; Jinliang Lin<sup>1</sup>; <sup>1</sup>Colorado School of Mines

The importance of nanostructured and nanocomposite coatings research have in recent years been considerably extended by the realization of the possibilities of synthesizing multifunctional materials with a combination of mechanical, physical, and chemical properties to meet various severe corrosion, oxidation, and wear environmental conditions. With the recent development of High Power Pulsed Magnetron Sputtering (HPPMS) and Modulated Pulse Power (MPP) Sputtering, major advances in surface engineering have been made. In the present study, nanocomposite binary (TiC-a:C), quaternary (Ti–B–C–N) and quinternary (Ti–Si–B–C–N) multicomponent coatings were deposited using pulsed closed unbalanced magnetron sputtering (P-CFUBMS) from both elemental and composite targets. The microstructure and composition of the nanocomposite coatings were characterized using x-ray diffraction (XRD), x-ray photoelectron spectroscopy (XPS), and transmission electron microscopy (TEM).

#### 12:20 PM

## Surface-Hardening of SUS316L by Para-Equilibrium Solid-Carburizing with C60: Kyosuke Yoshimi<sup>1</sup>; Yuya Miyazaki<sup>1</sup>; Kouichi Maruyama<sup>1</sup>; <sup>1</sup>Tohoku University

Fullerenes as represented by C60 are the third carbon allotrope, and have attracted attention as carbon source for metallurgy and materials processing because of their unique physical and chemical properties. In order to study the solid-state reactivity of fullerenes with metallic materials, the surface-hardening behavior of the SUS316L austenite stainless steel is investigated through solid-carburizing using C60 in this study. Carburizing was conducted using 98 wt.% C60 in a vacuum of  $5 \times 10^{-3}$  Pa at 475 or 500°C, which is under the paraequilibrium condition of SUS316L. The surface hardness of SUS316L that was approx. 150 Hv before carburizing was increased with increasing carburizing time and reached 450 Hv at 500°C for 100 h. XRD profiles exhibited no carbide formation but the increase in the lattice parameter of the austenite matrix, indicating that the carburizing proceeded under the para-equilibrium condition and thus supersaturated carbon atoms induced the expansion of the austenite fcc lattice. It was found that C60 not only behaves as carbon source but also exerts reducing ability against the passive film of Cr2O3 for SUS316L. The results obtained in this study demonstrate that fullerenes have high potential for a novel solid-carburizing technique.

#### 12:35 PM

Surface Modification for Enhanced Corrosion Resistance Using Fluid Bed Reactor Chemical Vapour Deposition (FBR-CVD): Kevin Ralston<sup>1</sup>; Dan Fabijanic<sup>2</sup>; Nick Birbilis<sup>1</sup>; <sup>1</sup>Monash University; <sup>2</sup>Deakin University

The use of materials with otherwise desirable mechanical properties is often problematic in practice as a result of corrosion. Susceptibility may arise for a number of reasons, including an electrochemically heterogeneous surface or destabilisation of a passive film. These shortcomings have historically been overcome through the use of various coatings, claddings, or conversion coatings. However, a more robust surface layer with enhanced corrosion resistance could possibly be produced via local surface alloying using a fluidised bed. A fluidised bed treatment allows a surface to be alloyed (albeit with some limitations), producing a distinct concentration graded layer up to tens of microns thick. Surface alloying additions can be selected on the basis of whether they are known or suspected to enhance the corrosion resistance of a particular material, at a minimum alloying likely provides a more electrochemically homogeneous surface. Electrochemical evaluations using potentiodynamic polarisations in NaCl electrolytes have shown chromised plain carbon and stainless steel surfaces have decreased rates of corrosion, decreased passive current densities, and ennobled pitting potentials relative to untreated specimens. Current work is focused on the use of fluidised bed technology to alloy the surface of light metals as well as surface characterisation of alloyed layers using XPS.

#### 12:50 PM

#### In Situ TiB<sub>2</sub> Particles Reinforced Copper Matrix Composite Coating on Mould Steel by Laser Cladding: Xue Liu<sup>1</sup>; Sen Yang<sup>1</sup>; <sup>1</sup>Nanjing University of Science and Technology

To extend the mould cycle duration and to reduce cost, mould repair and modification is becoming increasingly important. Laser cladding, as a viable, insitu and cost-effective technique, has been widely used to improve the surface properties of many kinds of alloys in the past three decades and still attracts a great of attention. The aim of the present investigation is to synthesize insitu TiB, particles reinforced copper matrix composite coating on mould steel substrate by laser cladding to improve cooling and, hence, to increase the mould cycle duration. The experimetal results show that TiB, particles of various shapes and sizes embedded in copper based alloy are in-situ synthesized during laser processing. An excellent bonding between the coating and the mould steel substrate is obtained. The microstructure of the coating is mainly composed of  $\alpha$ -Cu dendrites and dispersed TiB, particles. Much more and larger TiB, partices are formed in the top surface of the coating, which leads to a gradient profile of the microhardness from top surface to the bottom of the coating. The maximum microhardness of the coating was about HV<sub>01</sub>750. The effects of the laser processing parameters on the microstructures and properties of coating were also investigated.



#### Symposium I: Biomaterials, Smart Materials and Structures: Shape Memory Alloys

Thursday AM	Room: 3	
August 5, 2010	Location:	Cairns Convention Centre

Session Chairs: Tae-hyun Nam, Gyeongsang National University; Allan Morton, CSIRO

#### 8:30 AM Keynote

Magnetic-Field-Induced Phase Transformation and Multifunctions NiMnGaCu Magnetic Shape Memory Alloys: Chengbao Jiang<sup>1</sup>; Jingmin Wang<sup>1</sup>; Panpan Li<sup>1</sup>; <sup>1</sup>Beihang University

Since the large magnetic-field-induced strain (MFIS) was reported in Ni2MnGa, ferromagnetic shape memory alloys (FSMAs) have attracted considerable attentions. In this paper, search for a transformation from paramagnetic martensite to ferromagnetic austenite is performed in NiMnGaCu alloys. The composition dependence of the martensitic transformation temperature TM, the magnetic transition temperatures TCA of the austenite and TCM of the martensite was systematically investigated. The sequence of the martensitic transformation and magnetic transition was determined. The phase diagram on the structural and magnetic transition in a specific system Ni46Mn25+xGa25+xCu4 was outlined, in which a transformation from paramagnetic martensite to ferromagnetic austenite is predicted, exhibiting TCM < TM < TCA. Such a transformation was then experimentally achieved in Ni<sub>46</sub>Mn<sub>33</sub>Ga<sub>17</sub>Cu<sub>4</sub> alloy. Further, the effect of magnetic field on the phase transformation was studied in Ni<sub>46</sub>Mn<sub>33</sub>Ga<sub>12</sub>Cu<sub>4</sub> alloy. The transformation temperature was lowered by 7 K under a field of 9 T. Magnetic fieldinduced reverse martensitic transformation was realized. The switching field for the MFIRMT was discussed in terms of free energy change  $\Delta G$  of the martensite. Based on the MFIRMT, the magnetic-field-induced shape recovery (shape memory), magnetoresistance and magnetocaloric effects are achieved in NiMnGaCu alloys.

#### 8:50 AM

Development of Polyimide/SMA Thin-Film Actuator: Akira Ishida<sup>1</sup>; Morio Sato<sup>2</sup>; <sup>1</sup>National Institute for Materials Science ; <sup>2</sup>National Institute for Materials Science

Ti-Ni-Cu shape-memory alloy (SMA) thin films were sputter-deposited on heated polyimide substrates. Films deposited at a substrate temperature of 543K or higher were found to be crystalline. Especially, a Ti<sub>48</sub>Ni<sub>29</sub>Cu<sub>23</sub> film deposited at 583K exhibited a high martensitic transformation temperature above room temperature and a narrow temperature hysteresis, which enable the film to be used at room temperature. Double-beam cantilevers made of an 8µm thick Ti48Ni20Cu22 film deposited on a 25µm thick polyimide substrate displayed a repeatable shapememory effect by a battery of 1.5V and moved 0.18g wings of a toy dragonfly up and down. A simple increase in the thickness of the polyimide film increased the force of the actuator. The same Ti-Ni-Cu film deposited on a 125um thick polyimide film was capable of lifting a 15g weight. Additional deposition of a copper film on the Ti-Ni-Cu film enabled direct soldering. A large reduction in power consumption was also achieved by leaving the copper film on parts other than the actuator. Since polyimide/SMA thin films can be cut out any arbitrary shape for use and are easily driven by a battery, they are expected as a convenient actuator for small parts.

#### 9:05 AM

Shape Memory Properties of Equal Channel Angular Extruded Ti-35 Wt.%Nb Alloy: *R. Arockiakumar*<sup>1</sup>; S.M. Kim<sup>1</sup>; Joong-Keun Park<sup>1</sup>; <sup>1</sup>Korea Advanced Institute of Science and Technology

Because of its excellent biocompatibility, Ti-Nb alloy has been considered as a potential candidate for biomedical shape memory application. However, the alloy has a problem to exhibit a poor shape memory property as compared to a widely used Ni-Ti alloy. In an effort to improve its shape memory property, the present research was to study the equal channel angular extrusion (ECAE) processing of Ti-35 wt.%Nb alloy. The as-received (AR) alloys were ECAE processed at 400°C up to 4 passes via three different deformation routes A, Bc and C. They were subsequently annealed at 600°C for 30 min to produce an ultrafine grain structure of 0.3µm. Shape memory properties were measured by loading-unloading tensile tests. The stress for slip increased from initial 400 up to 580 MPa due to the grain refinement by ECAE processing. The stress for reorienting martensite was varied from 25 to 90 MPa with the deformation route. The transformation strain, depending on the deformation route, showed a maximum 3.5% (in route C), which was close to a theoretical maximum. The X-ray pole figure analysis indicated that the variation of transformation strain could be interpreted in terms of texture variation with the deformation route.

Effect of Rolling Reduction on Mechanical Property in Cast Ti-Rich Ti-Ni Shape Memory Alloy: *Kazuhiro Kitamura*<sup>1</sup>; Yukiharu Yoshimi<sup>2</sup>; <sup>1</sup>Aichi University of Education; <sup>2</sup>Yoshimi Inc.

The rolling reduction of a cast Ti-rich Ti-Ni shape memory alloy (SMA) plate from self-propagating high temperature synthesis (SHS) ingot was investigated. DSC and Tensile test specimens were cast by lost-wax process from SHS ingot. The composition of the ingot was Ti-49.8at%Ni. Specimens were cold rolled by rolling machine. The heat treatment conditions were as cast, 400°C, 500°C, 600°C- 60min. for differential scanning calorimetry (DSC) and tensile test specimen. Transformation temperatures were measured by DSC. Mechanical properties were measured by a tensile test at 17°C. From DSC measurement, in 400°C and 500°C specimens, R-Phase was observed by cold rolling. In 600°C specimen, transformation temperature was decrease by cold rolling. From tension test, in cold rolling specimen, the S-S curve similar to the bulk material was obtained. Shape memory characteristic of casting specimen was improved by cold rolling.

#### 9:35 AM

Effect of the Addition of Gd on Ni<sub>53</sub>Mn<sub>22</sub>Co<sub>6</sub>Ga<sub>19</sub> High-Temperature Shape-Memory Alloy: *Yunqing Ma*<sup>1</sup>; Shuiyuan Yang<sup>1</sup>; Sanli Lai<sup>1</sup>; Shiwen Tian<sup>1</sup>; Cuiping Wang<sup>1</sup>; Xingjun Liu<sup>1</sup>; <sup>1</sup>Xiamen University

In our previous investigation, the ductility of polycrystalline Ni<sub>ss</sub>Mn<sub>21</sub>Co<sub>4</sub>Ga<sub>10</sub> allow has been greatly improved due to the introduction of ductile  $\gamma$  phase by Co addition. However, the shape memory effect decreases since the reorientation of martensitic variants is hampered by  $\gamma$  phase. Meanwhile, many reports revealed that the improvements of the mechanical properties of Ni-Mn-Ga alloy could be also obtained by the refinement of grain size by adding rare earth elements, and the negative effect of  $\gamma$  phase particles on shape memory effect could be greatly reduced when  $\boldsymbol{\gamma}$  phase particles were dispersed on grain boundaries rather than among martensitic variants. The size, shape, volume fraction and distribution of  $\gamma$  phase affect the mechanical properties and the shape memory effects of Ni-Mn-Ga alloys greatly. So in this study, rare earth elements Y, Gd, Dy are added into Ni<sub>53</sub>Mn<sub>22</sub>Co<sub>6</sub>Ga<sub>19</sub> alloy to adjust the grain size, the shape and distribution of  $\gamma$  phase, so as to improved the ductility and shape memory effect simultaneously. Their compositions, microstructures, martensitic transformation behaviours, mechanical properties, as well as shape memory characteristics were investigated. The obtained results may provide important information for the development of Ni-Mn-Ga-based alloys as practical high temperature shape memory alloys.

#### 9:50 AM

Hysteretic Behavior of Concrete Cylinders Confined by Active Confining SMA Wire Jackets: *Eunsoo Choi*<sup>1</sup>; Hacksoo Lee<sup>2</sup>; Joonam Park<sup>3</sup>; Bak-Soon Choi<sup>4</sup>; <sup>1</sup>Hongik University; <sup>2</sup>Hannam University; <sup>3</sup>Korea Railroad Research Institute; <sup>4</sup>Inje University

Shape memory alloy (SMA) wire jackets are distinct from conventional jackets for concrete members since they can provide active confinement easily. The applications of SMAs in civil engineering are on the increase since SMAs show unique properties of shape memory effect and superelastic behavior. Especially, the seismic application of SMAs for confining concrete becomes a hot issues in recent. Thus, this study conducts cyclic compressive tests of concrete cylinders jacketed by shape memory effect. Monotonic compressive tests provide only the peak strength and the ultimate strain for confined concrete. However, confined concrete is exposed to the repeating of loading and unloading during an earthquake. Also, this study performs the same tests for concrete cylinders jacketed by steel jackets which provide only passive confinement and the results are compared to those of SMA wire jackets. This study provides the basic information of concrete behavior confined by active or passive external jackets.

#### 10:05 AM

Shape Memory Effect in Biopolymers: Zbigniew Stachurski<sup>1</sup>; Subbu Venkatraman<sup>2</sup>; <sup>1</sup>Australian National University; <sup>2</sup>Nanyang Technological University

For biomedical devices using the concept of thermally induced shape-memory effort, the shape memory polymer will typically undergo a thermomechanical loading-unloading cycle. The polymer is first deformed from its initial shape to a temporary shape by applying a mechanical force at drawing temperature. Subsequently, the temporary shape of the material can be maintained by lowering the temperature to below glass transition temperature and removing the external load. The polymer can largely maintain this shape as long as the temperature remains unchanged. The shape recovery or device deployment is activated by raising the temperature to above glass transition where the desired initial shape is recovered. From the practical viewpoint, it is highly desirable that the thermomechanical behavior of polymer can be predicted. In an attempt to capture these effects on the thermomechanical behavior of polymer, a one-dimensional



thermomechanical constitutive model was developed for amorphous polymer and it serves as the foundations for the development of model for semi-crystalline and bi-layer polymeric system.

#### 10:20 AM

## The High Temperature Connected Strength and Thermal Stability of Ti, Nb, SMA Pipe Coupling: Wei Jin<sup>1</sup>; <sup>1</sup>Institute of Metal Research, CAS

The creeping properties of  $Ti_{44}Ni_{47}Nb_9$  shape memory alloys were studied at 300°C. The connecting strength, deformation and thermal stability of the connected units of shape memory alloy pipe couplings and 16×1.5mm 1Cr18Ni9Ti tubes were investigated under constant load and high temperature. The results indicated that  $Ti_{44}Ni_{47}Nb_9$  alloys had excellent creeping properties at 400MPa stress and 300°C; the connected units of SMA pipe couplings and 1Cr18Ni9Ti tubes had good connected properties and thermal stabilities at small axial tensile load and 400°C; the essence of high temperature relaxation of the connected units of SMA pipe couplings and 1Cr18Ni9Ti tubes was that the strength of SMA pipe couplings and connected tubes reduced with increasing temperature, and pipe couplings had relative displace to connected tubes under axial tensile load.

#### 10:35 AM Tea Break

#### Symposium I: Biomaterials, Smart Materials and Structures: Smart Materials

Thursday AM	Room: 3	
August 5, 2010	Location:	Cairns Convention Centre

Session Chairs: Ze Zhang, Zhejiang University; Erich Kisi, The University of Newcastle

#### 11:00 AM Keynote

## **Ordered Mesoporous Materials and Their Applications**: *Dongyuan Zhao*<sup>1</sup>; <sup>1</sup>Fudan University and Monash University

Here we demonstrate facile approaches to synthesize ordered mesoporous molecular sieves with large pores (2 ~ 45 nm) and extra-large mesotunnels or entrance sizes (up to 27 nm); rich structure types; controlled morphologies and designable compositions. We will focus on an amphiphilic surfactant-templating approach to synthesize ordered mesoporous polymers and carbon frameworks. A family of ordered mesoporous organic polymers and carbon solids are simply achieved by using resols as precursors, followed by a thermopolymerization process, then carbonization. The mesoporous polymers have a large uniform mesopore, high surface areas (2500 m<sup>2</sup>/g) and large pore volume (~ 2.0 cm<sup>3</sup>/g). The scale-up synthesis can be carried out, kilogrammes of ordered mesoporous carbons are easily obtained for applications in catalysis and electrochemical supercapacitors. We will also show some recent results about the applications of mesoporous molecular sieves on catalysis, bio- immobilization, water purification and electronic energy storage.

#### 11:20 AM Keynote

#### Cellular Materials in Nature: Lorna Gibson<sup>1</sup>; <sup>1</sup>MIT

Cellular materials are widespread in nature. Wood and cork have a honeycomblike structure with cells that are roughly hexagonal prisms. Trabecular bone, plant parenchyma, adipose tissue, coral and sponge all have a foam-like structure, with polyhedral cells. Natural structures often have a cellular component: skulls and leaves of monocotyledon plants are sandwich structures, with dense outer skins separated by a foam-like core; animal quills and plant stems are nearly fully dense cylindrical shells supported by a foam-like core; and palm and bamboo stems are cylinders with radial density gradients. This talk provides an overview of cellular materials in nature and illustrates how the cellular structure gives rise to increased mechanical performance.

#### 11:40 AM Keynote

## **Bio-Inspired, Smart, Multiscale Interfacial Materials**: *Lei Jiang*<sup>1</sup>; <sup>1</sup>Chinese Academy of Sciences

Bio-inspired smart materials should be a "live" material with various functions like organism in Nature, they must have three essential elements as sense, drive and control. The studies on lotus and rice leaves reveal that a super-hydrophobic surface with both a large CA and small sliding angle needs the cooperation of micro- and nanostructures. Considering the arrangement of the micro-and nanostructures, the surface structures of the water-strider's legs were studied in detail. The cooperation between surface micro- and nanostructures and surface modification of poly (N-isopropylacrylamide) gave reversible switching. Besides organic surfaces, a series of inorganic switchers were also made.

#### 12:00 PM Invited

**Regioselectively Controlled Synthesis of Colloidal Button-Mushroom-Nanostructures and Their Hollow Derivatives**: *An-Hui Lu*<sup>1</sup>; <sup>1</sup>State Key Laboratory of Fine Chemicals

In this study, a facile synthetic rout for the fabrication of dissymmetrical, buttonmushroom nanostructures (FexOy@PSD&SiO<sub>2</sub>) and their hollow derivatives has been established, i.e., by consecutive immobilization of FexOy (Fe<sub>3</sub>O<sub>4</sub> or Fe<sub>2</sub>O<sub>3</sub>) with polymer spheres and then positioning controlled anchoring silica hemisphere steps. It was found that the surface accessible FexOy nanoparticles on the Janus type FexOy@PSD nanospheres are the keys for directing the growth of the silica hemisphere on the FexOy@PSD nanospheres. The size and the porosity of the silica hemispheres in the button-mushroom nanostructures are tunable by adjusting the amount of TEOS used and adding of proper surfactant in a Stöber process. After leaching out the iron oxide cores with concentrated HCl, button-mushroom nanostructures with hollow interiors were obtained, where the morphology of the hollow interior faithfully replicated the shape of the iron oxide core. This synthetic strategy might provide an easy and controllable method for large scale preparation of dissymmetrical colloidal nanostructures which would serve as building block for assembly of new type nanostructures.

#### 12:15 PM

## Synthesis of Multi-Shelled Mesoporous Silica Hollow Nanospheres and their Application in Controlled Release of Drug: Jian Liu<sup>1</sup>; *Shizhang Qiao*<sup>1</sup>; <sup>1</sup>The University of Queensland

Highly dispersed hollow nanospheres with uniform particle size have great potential to be used as carriers of diagnostic agent and medical therapeutics as well as nanovessels for the confined reactions. In this work, multi-shelled mesoporous silica hollow nanospheres (MMSHNs) with uniform size distribution (~150 nm) and tuneable shell thickness and pore size have been successfully synthesized through a facile vesicle template approach drove by surfactants FC<sub>4</sub> [C<sub>3</sub>F<sub>7</sub>O(CFC F<sub>3</sub>CP<sub>2</sub>O)<sub>2</sub>CFCF<sub>3</sub>CONH(CH<sub>2</sub>)<sub>3</sub>N+(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>CH<sub>3</sub>I-] and F127 (EO106PO70EO106). The formation of these hollow nanospheres was monitored by the dynamic light scattering. A vesicle templated mechanism was proposed to explain the molecular build-up of these silica hollow nanospheres. The materials were characterized by transmission electron microscopy, scanning electron microscopy, nitrogen sorption analysis. Furthermore, CdTe quantum dots (QDs) can be in-situ encapsulated into the hollow nanospheres, which have great potential in fluorescence biological probes and bio-imaging. We further demonstrate that these hollow nanospheres can be used for the loading and subsequent release of various drugs molecules.

#### 12:30 PM Keynote

Structural Determination of Ordered Porous Materials by Electron Tomography: *Jin Zou*<sup>1</sup>; Jin Zou<sup>1</sup>; Chengzhong Yu<sup>2</sup>; Graeme Auchterlonie<sup>1</sup>; John Drennan<sup>1</sup>; Pei Yuan<sup>3</sup>; <sup>1</sup>The University of Queensland; <sup>2</sup>Fudan University; <sup>3</sup>The University of Queensland and Fudan University

Ordered macro-/nano- porous materials with ultrahigh surface area and pore volume have become a globally focused topic because of their potential applications in separations, biosensors, and drug delivery. Comprehensive determination of their internal structure and external morphology is vital for understanding the formation mechanism and controlling the novel synthesis, which is of scientific importance and technological necessity. Although TEM is useful to investigate the fine structure of many materials, the conventional TEM only provides twodimensional projections of three-dimensional structures. Therefore, for materials with fine structural features, TEM may mislead the information. On the other hand, electron tomography (ET) is a newly developed technique to obtain the reconstructed complex three-dimensional structures from tilt series of TEM images. In this study, we employed ET to determine various three-dimensional structures of ordered macro-/nano-porous materials. Through detailed structural characterisation using ET, we have uniquely determined packing structures for several macro-/nano-porous materials. It has been shown that ET is a powerful tool in determining the complex ordered porous materials.

#### 12:50 PM

#### Australian Defence Applications of Advanced Smart Materials Research: *Christine Scala*<sup>1</sup>; Matthew Ibrahim<sup>1</sup>; Alan Wilson<sup>1</sup>; Darren Edwards<sup>1</sup>; Tan Truong<sup>1</sup>; <sup>1</sup>DSTO

This paper will give an overview of recent S&T innovations in smart materials at the Australian Defence Science and Technology Organisation (DSTO). The program is aimed at developing and transitioning innovative smart materials research across the maritime, air and land domains. Its aims are to increase the safety of Defence assets, improve their structural integrity, and enhance their availability and maintainability. Specific examples of Defence smart materialsrelated research to be discussed include: (i) Innovative application of carbon nanotubes/conducting polymers as artificial muscles for low-power propulsion and control of small autonomous underwater systems; (ii) Advances in smart sensing for prognostics-based platform management; (iii) Advanced nano-



coatings for increased wear-resistance and reduced corrosion of naval assets; and (iv) Fabrication of nanostructured and ultrafine grained materials through topdown severe plastic deformation processing of bulk materials.Future program directions in smart materials and structures under the Initiative will be outlined, particularly in the area of multi-functional materials.

#### Symposium J: Materials Characterisation and Evaluation: Steels

Thursday AM	Room: 1
August 5, 2010	Location: Cairns Convention Centre

Session Chair: George Gray, Los Alamos National Laboratory

#### 8:30 AM

**Unique Microstructure of Transition in Shock-Compressed Iron**: Shujuan Wang<sup>1</sup>; Qiuhong Lu<sup>1</sup>; Yongtao Chen<sup>2</sup>; Qingzhong Li<sup>2</sup>; Yongbo Xu<sup>1</sup>; Haibo Hu<sup>2</sup>; *Manling Sui*<sup>3</sup>; <sup>1</sup>Institute of Metal Research, Chinese Academy of Sciences; <sup>2</sup>Institute of Fluid Physics, China Academy of Engineering Physics; <sup>3</sup>Beijing University of Technology

A unique twin lamellar microstructure, of which three {112} twin planes presented threefold symmetry, was found in shock-compressed iron samples for the first time by TEM investigations. Compared with two {112} planes with an angle of 70.5° or 109.5° in a bcc grain, three {112} planes with an exact angle of 120° no longer belonge to one grains. Considering that the body centre cubic (bcc) alpha-phase might transform to the hexagonal-closed packed (hcp) epsilon-phase under shock loading above 13 GPa. The formation of the threefold symmetric microstructure was attributed to the martensitic transformation from hcp epsilonphase to bcc alpha-phase after shock loading. This work not only experimentally conformed the occurrence of epsilon to alpha phase transformations under shock loading, but also revealed the mechanisms of the alpha-epsilon-alpha transitions.

#### 8:45 AM

#### Comparison of Dislocation Substructures in 304L Forgings for Four Forging Techniques: John Smugeresky<sup>1</sup>; <sup>1</sup>Sandia National Laboratories

A preferred forging technique for hydrogen storage applications is High Energy Rate Forging (HERF) because of the compatibility of the resultant microstructure. It is the presence of fine subgrains and dislocation substructures in a dynamically recrystallized or recovered state that is believed the key feature for the hydrogen compatibility. However, HERF has become a sunset technology and as such is becoming either too expensive or no longer available. Three alternative forging techniques use Mechanical, Hydraulic, and Screw Presses to achieve shape and microstructure. This evaluation examined the TEM microstructures of samples processed by the four techniques by commercial forging facilities for two forging temperatures to attempt to determine which of the alternate techniques could come closest to producing the same subgrain structures and catalog the unique features HERF substructures. A correlation of the TEM results with DSC measurements will be presented.

#### 9:00 AM

Investigation of Austenite Stability in Different Austenite Formation Sites in TRIP Steels: *Eunji Yu*<sup>1</sup>; Jongryoul Kim<sup>1</sup>; <sup>1</sup>Hanyang University

Transformation induced plasticity (TRIP)-assisted multiphase steels have been attracted much attention to the automotive applications due to their excellent mechanical balance of high strength and elongation. It was reported that the elongation was strongly affected by the austenite stability which was related with the amount of solute carbon in retained austenite. Also, it was found that more retained austenite in inner grains remained compared with austenite in grain boundaries after plastic deformation. In this paper, the precious lattice parameters of local austenite phases (grain boundary/inner grain) in TRIP steels was calculated using the convergent beam electron diffraction (CBED) mode in transmission electron microscopy (TEM) in order to observe the austenite stability. The changes of carbon contents in the local phases were determined by the measured lattice parameters.

#### 9:15 AM

Effect of Ordering Behaviors on Magnetic Properties of 6.5 Wt% GO Silicon Steels: *Heejong Jung*<sup>1</sup>; Sang-Beom Kim<sup>2</sup>; Jongryoul Kim<sup>1</sup>; <sup>1</sup>Hanyang University; <sup>2</sup>Korea Electric Power Research Institute

In this paper, the effect of ordering behaviors on magnetic properties was studied using TEM. The specimens were fabricated through 3 step processes: fabrication of GO silicon steel, diffusion into GO silicon steel and then, homogenizing treatment. A cold-rolled silicon steel with a thickness of 0.3 mm was prepared through a conventional vacuum induction melting, hot rolling, and followed cold rolling processes. After annealing treatment in vacuum at 1200°C for 12 hours, all the strip samples consisted of (110) grains with a perfectly oriented <001> direction. Each annealed strip inserted between SiO2 textiles was heat-treated at 1200°C under 6N hydrogen atmosphere in a quartz tube. Through diffusion process of Si that generated from SiO2 decomposition, silicon content was controlled to fix 6.5 wt% silicon steels by annealing time. The fabricated 6.5 wt% silicon steels were treated by homogenizing treatment at 1200°C in order to investigate the relationship between ordering and magnetic properties. As a result, a reduction of iron loss might be caused by uniform distribution of the finely ordered phases.

#### 9:30 AM

**Multiple-Beam Irradiation Effects in Reduced Activation Ferritic Steels**: Naoyuki Hashimoto<sup>1</sup>; Hiroshi Kinoshita<sup>1</sup>; Somei Ohnuki<sup>1</sup>; *Hiroshi Oka*<sup>1</sup>; <sup>1</sup>Hokkaido University

Reduced-activation ferritic steels (RAF) and Fe-Cr model alloys were irradiated using a High Voltage Electron Microscope (HVEM) as the experimental evaluation of the multiple-scale modeling and simulations. Growth rate and saturated number density of dislocation loops in Fe-Cr model alloys and RAF under electron and ion irradiation with and without helium and hydrogen were measured and Arrhenius plotted to calculate defect activation energies such as vacancy and interstitial migration energies Electron irradiation experiment indicated that net migration energy of vacancy in the welded RAF tended to be lower compared to that in base metal, which could be relating to difference of carbon concentration in matrix between welded and base metal. In both cases of single beam (electron) and dual beam (electron and helium) irradiation, net migration energies of vacancy were slightly higher than that of interstitial. Furthermore, as helium implantation ratio increases, vacancy migration energies become higher, while there are little differences between interstitial migration energies. This result indicates that vacancy would be trapped by implanted helium due to their strong interaction and appeared to have higher migration energy.

#### 9:45 AM

## Grain Boundary Engineering and Alterations in Anisotropy of Interfacial Properties: *Pavel Lejcek*<sup>1</sup>; Aleš Jäger<sup>1</sup>; Viera Gärtnerová<sup>1</sup>; <sup>1</sup>Institute of Physics, AS CR

Analysis of the data on grain boundary properties which are frequently published in literature show surprisingly in some cases a reversed course of the structural dependence than is expected or directly prove its qualitative change with changing temperature. The examples of this "reversed anisotropy" found for grain boundary segregation, diffusion and migration are demonstrated. We propose to explain this reversed anisotropy of grain boundary properties on basis of so called "compensation effect". Because the structural dependence of grain boundary properties is frequently represented by concentrations, triple product or mobility, the altered character of such representation of the anisotropy evokes principal doubts about classification of the grain boundaries and thus they can have serious consequences for grain boundary engineering. On the other hand, it is shown that the anisotropy of thermodynamic characteristics of the grain boundary properties is independent of temperature. Therefore, it is suggested to classify the grain boundaries to special and general exclusively on the basis of the well-defined thermodynamic parameters.

#### 10:00 AM

Studies on Estimating Methods of Polarization Performance for Coated Steel in Seawater: *Yuhong Qi*<sup>1</sup>; Zhanping Zhang<sup>1</sup>; Mei Miao<sup>1</sup>; Xiazhen Zhang<sup>1</sup>; <sup>1</sup>Dalian Maritime University

To reduce corrosion, marine structures are commonly protected by designing coating system and cathodic protection (CP) system. Estimating the overall current demand for cathodic protection in seawater requires accurate polarization data for the marine structure materials involved. Computer models that predict cathodic protection system also requires accurate polarization data. Polarization curves for most marine structure materials in seawater are frequently found in some standards or measured by potentiostat. But it is difficulty to measure polarization data for the structure materials covered by organic coating and coating deteriorate with time. This has lead to one approach which ignores the existence of coating in the design of CP system. The CP system is designed to provide corrosion protection in the event of complete coating failure which result in an expensive over-design. In this paper estimating methods of polarization performance for coated steel in seawater are studied by measuring polarization data of steel covered organic coating with the different degree of man-made damage using electrochemical workstation IM6ex with Coating & Laminate Tester (COLT) and from these data analyzing CP system using BEASY CP corrosion simulation software.

#### 10:15 AM Tea Break



#### Symposium J: Materials Characterisation and Evaluation: Surface Engineering

Thursday AM	Room: 1	
August 5, 2010	Location: Cairns Convention Centre	

Session Chairs: Naoyuki Hashimoto, Hokkaido University; John Smugeresky, Sandia National Laboratories, CA

#### 11:00 AM Keynote

Surface Oxides Characterization of Materials in High Temperature Pressurized Water: *En-Hou Han*<sup>1</sup>; <sup>1</sup>Institute of Metal Research, Chinese Academy of Sciences

Nuclear power plants in the world have been rapidly developed since it is clean energy against global warming. Although lots of fundamental research has been done in advanced countries, there still many incidents or accidnets exist. The accidents indicate that there need more research work especially on the corrosion mechanisms of the NPPs' materials in high temperature pressurized water. The corrosion experiments of Alloy 625 in boric buffer solutions with different  $pH300^\circ C$  in the temperature range of  $25^\circ C\text{-}300^\circ C$  were conducted. The effects of temperature on passive/oxide film property were studied by electrochemical impedance spectra technique and X-ray photoelectron spectroscopy analysis. Mott-Schottky curves were also measured and analyzed to understand the semi-conduction properties. The property of the passive/oxide was analyzed by combining the electrochemistry results and Mott-Schottky analysis. The temperature was found to have an obvious effect on the structure and composition, as well as protective property of the passive/oxide film. This may be mainly due to the temperature-induced variation of the property of the barrier layer in the passive/oxide film. Some results obtained from 304L stainless steel and alloy 690 were also included for comparison purpose.

#### 11:20 AM

Hydrogen Evolution Behaviour during Tensile Deformation in Austenitic Stainless Steels Exposed to High Compressed Hydrogen Atmospheres: *Keitaro Horikawa*<sup>1</sup>; Hidetoshi Kobayashi<sup>1</sup>; Motohiro Kanno<sup>2</sup>; <sup>1</sup>Osaka University; <sup>2</sup>Chiba Institute of Technology

Hydrogen embrittlement sensitivity of austenitic stainless steels, SUS316L and SUS310S exposed to high compressed hydrogen gas atmospheres (35MPa) was evaluated by means of a slow strain rate testing (SSRT) in air. Hydrogen evolution behaviour during tensile deformation and fracture was also investigated by using a testing machine equipped with a quadrupole mass spectrometer installed in an ultrahigh vacuum chamber. When the SUS 316L specimen with hydrogen gas charging were deformed at a slow cross head speed of 1.67×10<sup>-</sup> <sup>9</sup>m/s, a slight decrease of the ductility was identified as compared to the specimen without hydrogen gas charging. On the other hand, no decrease of the ductility was observed in the SUS310S specimen with hydrogen gas charging even in the SSRT. In the hydrogen charged SUS316L specimen, the amount of continuous hydrogen evolution was much higher than that in the specimen without hydrogen gas charging throughout deformation. In addition, sudden hydrogen evolutions were sometimes identified in the SUS316L specimen with hydrogen gas charging during the deformation. At the moment of fracture, a large amount of hydrogen gas was evolved in the SUS316L specimen with hydrogen gas charging.

#### 11:35 AM

Low Cycle Fatigue Behavior of Ni-Base Superalloy IN738LC at Elevated Temperature: Keun Bong Yoo<sup>1</sup>; Han Sang Lee<sup>1</sup>; *Jaehoon Kim*<sup>2</sup>; Kwontae Hwang<sup>2</sup>; <sup>1</sup>Korea Electric Power Research Institute; <sup>2</sup>Chungnam National University

High strength nickel-base super alloys have been used in turbine blades for many years because of their superior performance at high temperature. The prediction of fatigue life for superalloy is important for improving the efficiency. In this study, low cycle fatigue tests are performed as the variables of total strain range and room and elevated temperature. The relations between plastic and total strain energy densities and number of cycles to failure are examined in order to predict the low cycle fatigue life of IN738LC super alloy. The fatigue live is evaluated Coffin-Manson equation, also the predicted lives by plastic and total strain energy density are compared with experimental results.

#### 11:50 AM

Stress Corrosion Cracking of Alloy 690tt in High Temperature High Pressure Water: *Jianqiu Wang*<sup>1</sup>; En-Hou Han<sup>1</sup>; Wei Ke<sup>1</sup>; <sup>1</sup>Institute of Metal Research, Chinese Academy of Sciences

This paper aims to investigate stress corrosion cracking of Alloy 690TT in high temperature high pressure water. The Alloy 690TT was as-received, mechanical

ground and electro polished respectively and immersed in simulated primary water at DO = 2 ppm and DH = 2.5ppm respectively. The microstructure of surface and the compositions and morphology of the surface film on Alloy 690TT after immersion test were studied using scanning electron microscopy (SEM), transmission electron microscopy (TEM), Auger electron spectroscopy (AES) and focused ion beam (FIB). The results showed that surface finish affected corrosion and stress corrosion of Alloy 690TT.

#### 12:05 PM

Study on the Invalidation of High Temperature Oxidation under Tensile Load of Cr<sub>5</sub>Mo Alloy: Chen Shunqing<sup>1</sup>; <sup>1</sup>Shanghai Institute of Technology

The damage process of high temperature oxidation under tensile load had been experimentally researched on Cr. Mo alloy. The surface visages and reaction rates of high temperature oxidation under different stress also had been researched in this paper. With comparing the experiment results of oxidation process under tensile load or without tensile load on Cr<sub>s</sub>Mo alloy, the exactly research conclusion about the law of high temperature oxidation invalidation under tensile load had been gotten on Cr<sub>s</sub>Mo alloy. Follow experimental results have been gotten. The layer growth rate contain obvious influence under the condition of the outside tensile load. The function of the influence is most obviously at the. start stage of the high temperature oxidization of the alloy steel material. Under the general condition to the 12CrMoV alloy material, the high temperature oxidization layer growth rate is also increase with the tensile load increase. For the individual circumstance, after pulling to attain certain degree in response to the alloy, the influence will be bluntness to the high temperature oxidization layer growth rate, enlarge again to pull namely and have already can't influence to oxidize a layer growth rate obviously in response to the alloy.

#### 12:20 PM

Characterisation of the Densification Mechanism of Plasma Sprayed Cordierite: *Núria Llorca-Isern*<sup>1</sup>; Gemma Bertran-Vidal<sup>1</sup>; <sup>1</sup>Universitat De Barcelona

Free-standing samples of plasma sprayed cordierite with a high level of porosity were heat treated at different temperatures and for different holding times in order to study the densification process. Optimisation of a new characterisation methodology for the interconnected defects network in the specimens of plasma spraved cordierite based on fluorescence confocal scanning laser microscopy (CSLM) information was the main objective of the present work. Optical, electron and confocal microscopes were use to determine percentage, distribution and morphology of the defects in the specimens. Fractal geometry combined to the CSLM images was useful for evaluating structural characteristics, complexity and level of connectivity of the defects network of these samples. All this information allowed studying the level of densification accomplished by the samples under the different thermal treatments conditions. After the evaluation of the results produced, this methodology can be validated for other applications or processes such as conventional sintering, etc. Results showed low percentage of porosity with dispersion the latter related to the degree of anisotropy of the samples. However an increase of porosity with the crystallisation was also observed.

#### 12:35 PM

**Micro-Scratching of UHMW Polyethylene Surfaces**: Heiko Timmers<sup>1</sup>; *Yanyan Liu*<sup>1</sup>; Laura Gladkis<sup>1</sup>; <sup>1</sup>UNSW

Polymers are used for a variety of macroscopic applications. Recently polymers have been applied as one of the preferred materials towards the miniaturization of functionality in the form of micro- and nano-scale devices. The tribological performance under such circumstances, where loads are small and where surface properties tend to be more important than bulk properties, is expected to be different than in macroscopic applications. Nevertheless, it has been suggested that the amount of wear debris generated by a small number of micro-scratches correlates linearly with the macroscopic abrasive wear performance, as it may be measured in conventional pin-on-disk wear tests. Taking advantage of the fact, that silicon fractures along crystal planes with nano-scale definition, silicon micro-asperities have been made and characterized with electron microscopy. The micro-scratching of UHMWPE by these asperities is being studied to understand how debris production depends on actuation. Micro-scratch grooves and debris generated by the scratch have been characterized and quantified using scanning electron and scanning probe microscopy. The three-dimensional spatial sensitivity of scanning probe microscopy allows for detailed volumetric measurements of scratch grooves and of debris particles previously not achieved with electron microscopy. Progress will be reported and an outlook given.



#### Symposium K: Composites and Hybrid Materials: Ceramic Based Composite

Thursday AM August 5, 2010 Room: 4 Location: Cairns Convention Centre

*Session Chairs:* Soon Hyung Hong, Korea Advanced Institute of Science and Technology; Kilwon Cho, Pohang University of Science and Technology (POSTECH)

#### 8:30 AM Keynote

Fiber Reinforced Ceramic Composites for Space Propulsion System, Nozzle and Combustion Chamber Application in Japan: *Ken Goto*<sup>1</sup>; <sup>1</sup>Japan Aerospace Exploration Agency

Ceramic composites, SiC fiber and carbon fiber reinforced composites, are strong and tough heat resistant material. Advantages of these ceramic composites to use in nozzles and combustion chambers are not only their heat resistivity but also their small densities and high toughness. Author is now involved in two development activities of future space propulsion systems in Japan Aerospace Exploration Agency those require high temperature materials to use nozzle and combustion chamber without cooling by cryogenic fuels, one is an apogee engine or a post boost stage of solid propellant rocket, and the other is an air breezing engine. To meet with their demand, SiC fiber reinforced SiC composites is chosen for apogee engines because of its lower gas permeability and partially siliconized carbon fiber reinforced carbon composites for an air breezing engines for its higher heat resistance. Development studies of nozzle and combustion chamber made by these ceramic composites were carried out by ground level firing tests of test engines and the results demonstrated feasibility of these two ceramic composites for space propulsion system.

#### 8:50 AM

Crystallization of Amorphous SiCN Matrix of C/SiCN Composite under Conditions of Vacuum and Tension-Tension Fatigue: Yi Xia<sup>1</sup>; *Qiao Shengru*<sup>1</sup>; Chao Yan<sup>2</sup>; Chengyu Zhang<sup>1</sup>; <sup>1</sup>Northwestern Polytechnical University; <sup>2</sup>Northwestern Polytechnical University - and - Chengdu Aircraft Industrial (Group) Co. Ltd

Carbon fiber reinforced SiCN matrix composite (C/SiCN) was used for present investigation, its amorphous SiCN matrix was derived from the hexamethyldisilazane by chemical-liquid and vapor-infiltration into the carbon fiber weaving preform. The tension-tension fatigue was conducted at 1300°C in vaccum both for the as-received and 1500°C-annealed C/SiCN with 60Hz and 41MPa. The phase and microstructure evolution of SiCN during fatigue were examined by X-Ray diffraction and transmission electron microscopy (TEM), respectively. The results indicate that pronounced crystallization takes place in the as-received C/SiCN during fatigue, and only B-SiC crystallites are emerging within amorphous SiCN matrix, Si<sub>3</sub>N<sub>4</sub> can't be detected. By contrast, literatures reported that there isn't exists crystallization when C/SiCN is annealed at 1300°C under nitrogen or argon atmosphere. As for1500°C-annealed C/SiCN, there isn't exists crystallization during fatigue, and it can be attributed to phase stabilization on the SiCN matrix after annealing. Both vacuum and fatigue stress promote the crystallization course because they accelerate the decomposition of amorphous SiCN and atomic diffusion. Degradation - crystallization mechanism is used to explain the crystallization behavior of amorphous SiCN under condition of vacuum and tension-tension fatigue.

#### 9:05 AM

Effect of Humidity and Thermal Cycling on Carbon-Epoxy Skin/Aramid Honeycomb Structure: *Eudora Yeo*'; John Wang<sup>1</sup>; Leo Mirabella<sup>1</sup>; Andrew Rider<sup>1</sup>; <sup>1</sup>Defence Science and Technology Organisation

Many modern military aircraft are constructed from composite and bonded structure. Operation of these platforms in Australian and global conditions will subject the structure to potentially high levels of humidity and extremes in temperature. Aircraft deployed in maritime operations will be subject to salt spray conditions. The Tiger and MRH90 helicopters have skins made from thin carbon-epoxy laminate bonded to Kevlar® honeycomb. The thin composite laminate is likely to rapidly absorb moisture in a humid environment and enable permeation of moisture into the adhesive and core. In addition to the chemical influence of moisture on the composite structure, the moisture trapped in the honeycomb structure may freeze and expand with changes in altitude during operations or simply due to daily temperature fluctuations at the resident airbase. The combination of moisture ingress in the honeycomb structure and thermal cycling may lead to deteriorated strength of the honeycomb panels over time that would not be observed for long term humid exposure alone. Long term salt water

absorbtion may also have an adverse effect on composites structures. This study investigates the effects of humid environments, thermal cycling and salt spray conditions on the mechanical properties of composite and honeycomb structures. **9:20 AM** 

#### 9:20 AN

Fabrication of Carbon Nano-Fiber / Aluminum Composites by Low-Pressure Infiltration Method: *Gen Sasaki*<sup>1</sup>; Yoshimasa Hara<sup>1</sup>; Zhefeng Xu<sup>1</sup>; Kenji Sugio<sup>1</sup>; Yong Bum Choi<sup>1</sup>; Kazuhiro Matsugi<sup>1</sup>; <sup>1</sup>Hiroshima University

Recently, carbon containing aluminum matrix composites have been grate watched for heat sink and heat exchanger, and good electrical and thermal conductivity with good mechanical properties are required for the composites. In this study, the fabrication of carbon containing aluminum composites was attempted by using low-pressure infiltration method. At first, porous perform containing carbon nano-fiber and pure aluminum powder was fabricated by spark plasma sintering (SPS) method. Carbon nano-fiber used is vapor grown nano-fiber (VGCF) fabricated by Showa Denko Co. Porosity in perform was controlled by changing applied pressure during spark sintering. Consequently, porous perform with 40-50vol% in porosity was obtained, which has enough compression strength for low-pressure infiltration. Then molten pure aluminum infiltrated to porous preform with 0.4MPa in applied pressure at 1023K, and consequently we can obtain the composite with 90% over in density. In dense region in composites, the electrical conductivity was almost equal to that of the monolithic aluminum block.

#### 9:35 AM

Fabrication of Stainless Steel-Tungsten Carbide Composites by Powder Injection Moulding: Nutthita Chuankrerkkul<sup>1</sup>; <sup>1</sup>Chulalongkorn University

Composites of stainless steel particulate-reinforced with tungsten carbide were fabricated by powder injection moulding technique. An environmentally friendly binder system was employed. The binder contains a major fraction of polyethylene glycol (PEG) and a minor fraction of a very finely dispersed polymethyl methacrylate (PMMA). PEG can be removed by water immersion method, creating open-pore channels that let the remaining binder, PMMA, to be removed by pyrolysis during ramping up to the sintering temperature. Selections of processing parameters, i.e. particle size, binder composition, sintering treatment, were carried out. The relationship of these parameters on microstructure and properties of the composites were investigated and will be presented in this manuscript.

#### 9:50 AM

SiC/SiC Composite Thruster for Upper Stage Liquid Rocket Engines: Ken Goto<sup>1</sup>; Yuuichi Yoshida<sup>2</sup>; Shinichiro Tokudome<sup>1</sup>; Hiroshi Hatta<sup>1</sup>; Toshiharu Fukunaga<sup>2</sup>; <sup>1</sup>Japan Aerospace Exploration Agency; <sup>2</sup>Kyoto University

SiC fiber reinforced SiC composite thrust chamber was tested for the non-toxic liquid fuel (N<sub>2</sub>O/Ethanol) rocket engine (under development in ISAX/JAXA). A SiC/SiC composite thrust chamber was made by the lamination of three axes braiding layers, and the Tyrano ZMI SiC fibers were used as reinforcements and the SiC matrix was made by the combination of the chemical vapor infiltration process and polymer infiltration and pyrolysis process. The plane woven structured laminate type Tyrano ZMI fiber SiC/SiC composite plates made via same infiltration process were also manufactured to measure mechanical properties and gas permeability property of the material. To guarantee all the processes were successfully done, the trust chamber was pressurized by water with using a rubber bag. The mechanical response of the SiC/SiC chamber was monitored by strain gages. The maximum pressure was set to 2.5 times higher than its operating pressure. The chamber was successfully pressurized and measured strains at the maximum pressure were consistent with the design values. The Firing tests were conducted for several times with changing the engine conditions. The firing test of SiC/SiC thrust chamber was successfully conducted for 30 s with maximum temperature of ~ 1630 K.

#### 10:05 AM

Microstructure and Toughening Mechanism of Abalone Nacre: *Hideki* Kakisawa<sup>1</sup>; <sup>1</sup>National Institute for Materials Science

Natural inorganic/organic composites such as bone, tooth and nacre have an excellent "strength-toughness balance" due to their well-organized microstructures. Among them, nacre, the inner layer of mollusk shells, is composed of about 95% inorganic aragonite (a mineral form of CaCO<sub>3</sub>) layer (thickness ~500 nm), with only a few percent of organic biopolymer layer (thickness ~50 nm) and achieves a 1000-fold increase in toughness (work of fracture) over the monolithic mineral aragonite, without trading off its strength. Previous studies have revealed that the submicron-order laminar structure and the energy-dissipative organic matrix are keys for its mechanical properties. Besides, a nano-structure within the aragonite layer has recently been reported and its contribution to the toughness has been suggested. In the paper the toughening mechanisms of nacre brought by its hierarchical structure are introduced and the possibilities of applying them to artificial composites are discussed.



#### 10:20 AM

An Experimental Investigation on the Structural Behavior of FRP-Concrete Composite Compression Members: *Joon-Seok Park*<sup>1</sup>; In-Kyu Kang<sup>2</sup>; Jong-Hwa Park<sup>3</sup>; Hong-Taek Kim<sup>1</sup>; Soon-Jong Yoon<sup>1</sup>; <sup>1</sup>Hongik University; <sup>2</sup>Vniel Consultant Co., Ltd.; <sup>3</sup>Hyundai Engineering & Construction Co., Ltd.

In construction industries, new construction materials are needed to overcome some problems associated with the use of conventional construction materials due to the change of environmental and social requirements. Accordingly, the requirements to be satisfied in the design of civil engineering structures are diversified. As a new construction material in the civil engineering industries, fiber reinforced polymeric plastic (FRP) has a superior corrosion resistance, high specific strength/stiffness, etc. Therefore, such properties can be used to mitigate the problems associated with the use of conventional construction materials. Nowadays new type of bridge piers and marine piles are being studied for new construction. They are made of concrete filled fiber reinforced polymeric plastic tubes (CFFT). In this paper, a new type of FRP-concrete composite piles which are composed of pultruded sections wrapped by filament winding layer are proposed to improve compressive strength as well as flexural strength of a CFFT. The load carrying capacity of proposed CFFT is discussed based on the result of experimental investigation.

#### 10:35 AM Tea Break

Symposium K: Composites and Hybrid Materials: Modeling and Properties of Composites

Thursday AM August 5, 2010

Room: 4 Location: Cairns Convention Centre

Session Chair: Xungai Wang, Deakin University

#### 11:00 AM Keynote

Computational Analysis of the Structural Integrity of Self-Healing Composites: Chun Wang<sup>1</sup>; Feng Zhao<sup>1</sup>; Adrian Mouritz<sup>1</sup>; <sup>1</sup>RMIT University

One class of self-healing composites incorporate hollow fibres for storing the required healing agent or resin. The purpose of this work is to investigate the influence of the micro-vessels, which are potential sites of structural weakness, on the structural integrity and damage tolerance of composites. A fundamental understanding of the effect of the relationships between the shape, size and volume fraction of the micro-vessels and the mechanical properties of aerospace composites is essential to the optimisation of self-healing systems. Experimental studies have revealed that the interlaminar strength and fracture toughness can vary considerably depending on the size, shape and orientation of hollow fibres. To quantify the influence of hollow fibres on the stress distributions and fracture behaviour of composites, finite element models have been developed to represent the induced changes in local geometrical and material distributions around an embedded hollow fibre. This paper presents the results of the stress distribution and the growth mechanism of interlaminar crack

#### 11:20 AM

Preliminary Review of Physically Based Methodologies for Predicting the Strength of Visibly Damaged Composite Laminates: *Alex Harman*<sup>1</sup>; Andrew Litchfield<sup>2</sup>; Rodney Thomson<sup>2</sup>; <sup>1</sup>DSTO; <sup>2</sup>CRC-ACS

Approaches to detect, assess, monitor and repair damage in critical aircraft components fabricated from composite materials are essential for safe and cost effective operation. In metallic aircraft structures, it is common to leave some fractures in situ for a prescribed period until it is convenient to repair, provided strict inspection and verification processes are in place. Under current military aircraft structural management guidelines, visible damage to critical composite components requires either immediate repair or replacement. Much has been learnt about the behaviour of damaged composite structures, but further investigation is required to develop validated residual strength and life prediction tools. A preliminary review of an early physically based, residual strength prediction method was conducted. The accuracy of this method for use in predicting the strength of composite following a complex damage was tested by comparing the results with compression-after-impact test data for a composite laminate representative of F/A-18 fracture critical structure. Gaps in understanding were identified and opportunities for future development proposed.

#### 11:35 AM

**Deformation Behavior for Hybrid CFRP Observed by In-situ FE-SEM**: *Yoshihisa Tanaka*<sup>1</sup>; Kimiyoshi Naito<sup>1</sup>; Satoshi Kishimoto<sup>1</sup>; Yutaka Kagawa<sup>2</sup>; <sup>1</sup>National Institute for Materials Science; <sup>2</sup>The University of Tokyo

The deformation behavior of hybrid CFRP during tensile loading was investigated by in-situ FE-SEM observation coupled with multi-scale pattern. The composite material used was ultrahigh strength PAN-based (IM600) and ultrahigh modulus pitch-based (K13D) hybrid carbon fiber reinforced epoxy matrix composites. The deformation mechanism of the hybrid CFRP at different scale such as fiber bundles and fibers play an important rule in determining the composite strength and deformability. Prior to in-situ tensile loading, the multiscale pattern combined with a grid and random dots was fabricated by electron beam lithography technique on the polished side surface to facilitate direct observation of multi-scale deformation. The electron moiré method was applied to measure the strain distribution in the deformed specimens at fiber bundle order scale and digital images correlation method was applied to measure the localized deformation such as the interface between the fiber and the matrix at a micron meter scale acquired before and after loading. The multi-scale deformation behavior around the reinforcement and the fiber bundle based on in-situ FE-SEM observation will be discussed.

#### 11:50 AM

Residual Strength of a Helicopter Composite Structure Subjected to Small Arms Fire: *Richard Callinan*<sup>1</sup>; John Wang<sup>1</sup>; Caleb White<sup>1</sup>; <sup>1</sup>DSTO

As a result of high strength/weight ratio, high stiffness and good fatigue life, carbon fibre composites are being used in the structure of the Tiger helicopter in order to reduce weight. The Tiger helicopter is in service with the Australian army as an armed reconnaissance vehicle, and as a result of that role is vulnerable to small arms fire. As a result a series of ballistic tests were carried out on sandwich and laminate test specimens using 7.62mm and 12.7mm small arms fire. Residual tension testing was carried out on pristine, ballistically damaged and machine damaged laminate specimens. For the range of damage considered design curves were proposed to predict failure using the point stress method. An equivalent crack length concept proposed to represent damage to specimens was verified. Residual compression tests were carried out on honevcomb specimens. These specimens consist of pristine and damaged specimens. For a range of damage considered design curves were proposed and the same point stress parameter from tension testing was found to apply. A more advanced criteria is also considered for ultimate failure based on the last ply failure, and is found to agree well with the experimental data.

#### 12:05 PM

Development of High Modulus/High Strength Carbon Fiber Reinforced Nanoparticle Filled Polyimide Based Multiscale Hybrid Composites: *Kimiyoshi Naito*<sup>1</sup>; Jenn-Ming Yang<sup>2</sup>; Yutaka Kagawa<sup>3</sup>, <sup>1</sup>National Institute for Materials Science; <sup>2</sup>University of California, Los Angeles; <sup>3</sup>The University of Tokyo

The polyacrylonitrile(PAN)-based and pitch-based carbon fiber-reinforced nanoparticle filled polyimide matrix multiscale hybrid composites have been fabricated using vacuum assisted resin transfer molding (VaRTM) and autoclave curing. The carbon fibers used in this study were high tensile strength PANbased (T1000GB) and high modulus pitch-based (K13D) carbon fibers. Fiber orientations of the T1000GB/K13D hybrid composites were set to  $[0_{(T1000GB)}]$  $0_{(K13D)}]_{28}$ . The fiber volume fraction was 50 vol% (T1000GB: 24.9 vol%, K13D): 25.1 vol%). Polyimide used in this study was a commercially available polyimide precursor solution (Skybond 703). Four different types of nanoparticle (25nm-C. 20-30nm-β-SiC, 130nm-β-SiC and 80nm-SiO<sub>2</sub>) and particle volume fraction was 5.0 vol% used for the inclusion. The tensile properties and fracture behavior of T1000GB/K13D nanoparticle filled and unfilled hybrid composites have been investigated. For 25nm-C, 20-30nm-\beta-SiC and 80nm-SiO, nanoparticle filled and unfilled hybrid composites, the tensile stress-strain curves show a complicated shape. By the high modulus pitch-based carbon fiber, the hybrid composites show the high modulus in the initial stage of loading. Subsequently, when the high modulus carbon fiber begin to fail, the high strength fiber would hold the load (strength) and the material continues to endure high load without instantaneous failure.

#### 12:20 PM

**Strength Evaluation of Bolted Connection in PFRP Structural Member**: *Young- Geun Lee*<sup>1</sup>; Seungsik Lee<sup>2</sup>; Jeong-Hun Nam<sup>1</sup>; Hong-Teak Kim<sup>1</sup>; Soon-Jong Yoon<sup>1</sup>; <sup>1</sup>Hongik University; <sup>2</sup>Korea Institute of Marine Science and Technology Promotion

Fiber reinforced plastic structural shapes are readily available in civil engineering applications. Especially, pultruded fiber reinforced plastic is an attractive construction material for the structural applications because it can be produced with mass production, and it has good mechanical and chemical



properties compared with existing conventional structural materials. To be used in the construction field, connection of the pultruded structural member is unavoidable. Bolted connections may be most suitable for civil engineering applications compared with bonded connection. But bolted connection has disadvantages such as reduction of strength due to bolt hole in the connection. Experimental and analytical studies on the bolted connection of PERP plated member have been carried out. Four different types of connection distinguished by number and arrangement of bolts is investigated. Geometrical test parameters are edge distance, width, longitudinal and transverse spacing. The effects of the parameters are evaluated and quantified based on the observations, such as failure load and failure mode, obtained in the experiment. In addition to the experimental investigation, analytical study is also conducted to predict the failure load of the member with bolted connection.

#### 12:35 PM

Modelling of Reinforced Asphalt Products for Road Applications: Binh Vuong1; Sing-ki Choi2; Zahid Hoque3; 1Swinburne University of Technology/ ARRB Group; <sup>2</sup>Commonwealth Scientific and Industrial Research Organisation; <sup>3</sup>Roads and Traffic Authority of New South Wales

The rehabilitation of cracked pavements with asphalt overlays has been the common solution adopted in Australia and world wide. However, unless highperformance designs and materials are used, cracks propagate in most cases through the new overlay during the first few years of service. New technology of reinforcement materials in asphalt overlays can offer a potentially cost effective solution for treating cracked pavements. To date, however, there is no design process allowing the use of available reinforcement products in road applications in Australia; whereas overseas design procedures are not readily available. A new 3D finite element (FE) analysis procedure, which is an extension of Austroads new FE pavement design procedure, was considered where reinforced asphalt layer is modelled as a thin 3D compound non-homogeneous layer which is then converted into a thin 3D orthotropic layer. This procedure was used to analyse performance of three reinforcing products for road applications in Australia and results indicated that it is possible to predict and compare "retard reflection cracking" performance of various reinforced products and, hence, select the most effective overlay design solution for the road applications concerned.

#### 12.50 PM

Corrosion and Wear Resistances of Ni-P-Al<sub>2</sub>O<sub>3</sub> Composite Film Prepared by Electroless Plating: Rongguang Wang<sup>1</sup>; Hiroki Sawada<sup>1</sup>; Mitsuo Kido<sup>1</sup>; <sup>1</sup>Hiroshima Institute of Technology

The composite film of Ni-P-Al<sub>2</sub>O<sub>3</sub> was prepared on a mild steel of SS400 by using an electroless plating method. In this process, the Al<sub>2</sub>O<sub>2</sub> particles with mean diameter of 50nm were added into the Ni-P plating solution and the plating solution was stirred aiming at uniformly dispersing of the particles. After then, the polarization test on the prepared specimens was carried out in 1.0% NaCl aqueous solution and the wear resistant was measured by a ball-on-disk method with vertical load of 1.96N. As a result, the thickness of the composite film increases with the plating time, however, it becomes thinner when Al<sub>2</sub>O<sub>3</sub> particles were added into the plating solution. Especially the film is thinner and rougher when the stirring is not carried out. The mixed amount of Al<sub>2</sub>O<sub>3</sub> particles into the film is about 1.0mass% in this work. The particles evenly dispersed in the film when the plating solution was stirred. It is found that both the corrosion resistance and the wear resistance of the composite film with stirring is better than that without Al<sub>2</sub>O<sub>3</sub> or without stirring.

#### **Symposium M: IOMMMS Global Materials Forum:** Session I

Thursday AM	Room: 5
August 5, 2010	Location: Cairns Convention Centre

Session Chair: Robert Shull, National Institute of Standards and Technology

#### 8:30 AM

Energy Saving Magnetics: Robert Shull1; 1National Institute of Standards and Technology

Huge amounts of energy are lost worldwide each day in operating electrical equipment, like computers, electric vehicles, motors, transformers, and generators. Much of this loss is due to inherent losses in the magnetic materials used in those devices. All electronic devices contain permanent magnets for transforming voltages to those needed by the device. If the input power is an alternating voltage, then the magnetic field applied to those magnets is also alternating and the magnet is subjected to a hysteresis cycle and it loses the energy required to cycle the magnetization of that magnet. If the AC voltage is at 60 Hz, as is common in the US, then the hysteresis loss occurs 60 times per second times the amount of time the device is turned on. Similarly, energy is lost in electric vehicles because of the weight of the magnets in the electric motor.

#### 8:45 AM

#### PV Industry and Technology in China: Yafang Han<sup>1</sup>; <sup>1</sup>C-MRS

Energy demand has increased by one billion tons of coal between 2001 and 2006 and the energy demand related to automobiles and housing is a new phenomenon. This has led to emerging environmental problems in the form of coal smoke and greenhouse gases. Energy security and the protection of the environment present competing pressures. Presently, China relies too much on fossil fuels. China's basic strategy in developing renewable energy consists of government support (compulsory market regulation and economic incentive policy), legal guarantees (use overseas experience, compel power companies to purvey or purchase renewable energy power through legislative provisions), introducing competition, technological progress, and international cooperation. China aims to raise the share of renewable energy in total primary energy consumption to 10% by 2010, and to raise this share to 15% by 2020. China has great potential for photovoltaic generation.

#### 9:00 AM Invited

Development of Materials for Green Energy Production in Korea: Ohjoon Kwon1; Soon Young Hwang1; 1RIST

The Korean government has recently announced a very aggressive goal of the CO, emission. To achieve the goal, the green society policy is widely practiced and various green technologies are now under active development to reduce the emission. In this presentation, various new material developments in renewable energy will be presented such as photovoltaics, wind turbines, high capacity secondary batteries, and fuel cells. For the photovoltaics, poly-silicon is the major material, and several new plants have recently been constructed employing the Siemens process. However, new efforts are now made to develop innovative technologies which are more economic than the Siemens process. In case of the wind turbines, new light weight, high performance and low cost material developments are under way for towers, platforms, and blades. With regard to the high capacity secondary batteries, new commencement is made concerning lithium batteries, redox flow batteries and NAS batteries. Fuel cells are another area of dynamic R&D. Solid fuel oxide cell (SOFC) stack development up to 150KW capacity is now under way at RIST. Commercialization trials of molten carbonate fuel cells (MCFC) and polymer membrane fuel cells (PEMFC) will also be presented.

#### 9:15 AM Invited

Corrosion Issues in Pipeline Steels for Ethanol-Fuel Blend Transport: Brajendra Mishra1; David Olson1; 1Colorado School of Mines

The interest in the use of bio-fuels as a source for renewable energy has resulted in the need to transport these fuels safely through pipelines. In addition, the integrity of the existing pipelines for oil and gas transport has become critically important. The need for highly reliable and accurate corrosion inspection techniques for pipelines has led to the widespread adoption of magnetic flux leakage pigging tools. Magnetic flux leakage applies a saturating magnetic field and measures the induced magnetic field for areas of higher magnetic field that reveal defects and anomalies in the pipeline. A very significant effect has been measured in the laboratory on the hydrogen content in steel after electrochemical hydrogen charging with and without a two Tesla applied magnetic field, as well as a strong increase in hydrogen-induced cracking and pitting. The electrochemical study has been performed to characterize the environment during microbiologically influenced corrosion (MIC) of carbon line-pipe steels and assess the damage caused during ethanol blend transport. The role of microbiologically influenced corrosion of line-pipe steels was correlated with electrochemical analyses and interpretation to offer a better insight into microbiologically influenced corrosion behavior.

#### 9:30 AM Invited

Research and Development of Nano-Composite Materials for Hydrogen Storage: Yoshitsugu Kojima1; 1Hiroshima University

Hydrides with light elements such as MgH2, LiH, NH2 and NH2BH2 are known as high hydrogen containing materials. However, the high work temperature and the slow reaction rate limit the practical application of hydride systems. Those properties can be improved by the nano-composite materials. The nanocomposite materials for hydrogen storage encompass a catalyst and composite hydrides at the nanometer scale. The catalyst increases reaction rate. The thermodynamic stability of the nano-composite materials can be controlled by the composite hydrides. In addition, the hydrogen absorption kinetics is accelerated by the nano-size materials and they may change the thermodynamic stability of the materials. In this study, we reviewed our experimental results on hydrogen storage properties of light weight nano-composite materials, i.e. Mg-based nano-



composite material, metal-nitrogen-hydrogen (M-N-H) system and hydride ammonia (MH-NH<sub>3</sub>) system. The Mg-based nano-composite material with Nb<sub>2</sub>O<sub>5</sub> showed excellent kinetics as compared with that of Mg. The Li-Mg-N-H system absorbed and desorbed above 5.5 mass % of H<sub>2</sub> at 423K (8LiH + 3Mg(NH<sub>2</sub>)<sub>2</sub> ↔  $3Li_{2.667}$ MgN<sub>2</sub>H<sub>1.333</sub> + 8H<sub>2</sub>). We found that the H<sub>2</sub> absorption and desorption of the MH-NH<sub>3</sub> (M: Li, Na, K) system takes the following reaction path, MH + NH<sub>3</sub> ↔ MNH<sub>2</sub> + H<sub>2</sub>.

#### 9:45 AM

#### **Development of High Performance Pipeline Steels for Energy Projects**: *Chengjia Shang*<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing

In recent years, lots of large pipeline projects have been putting in practice in China. Experimental and industrial research works aimed for high performance and low cost caused great progresses in the metallurgical technology of high grade pipeline steel. In this presentation, the development of high performance, pipeline steel is focused from rolling process to welding performance, therein, some new technology and ideas will be exhibited, which include the full refinement and slow coarsening of austenite grains through static recrystallization and intense solute drag effect, higher strain accumulation and the suppression of partial dynamic recrystallization during strip rolling, the uniformity of effective grain sizes in pancaken austenite grain, and the weldability of pipeline steel. The comprehensive investigation results illuminated a novel industrial route for the production and application of high performance pipeline steels.

#### 10:00 AM Invited

Conservation of Energy through Life Extension of Nickel Superalloy Turbine Blades Used in Aero-Engine and Power-Generation Turbines: *Mahesh Chaturvedi*<sup>1</sup>; <sup>1</sup>University of Manitoba

Nickel based superalloys are extensively used in aero-engines and power generation turbines due to their excellent high temperature properties. The turbine components experience hostile environment during service that results in cracking and material loss. Extensive amount of energy and mineral resources are consumed in 1) the production of superalloys and 2) in fabrication of turbine blades from them. Therefore, to conserve energy and natural resources, and environment it is necessary to extend life of turbine components by repairing them rather than replacing them. A commonly used repair method is by overlay to build-up the lost material, and by welding of cracks. However, most  $\gamma'$ precipitation hardened superalloys crack in heat-affected-zones during welding, which is often prevented/minimized by using dissimilar low strength and ductile filler alloys. However, in most instances these filler alloys are not age-hardenable, and produce welds that are relatively crack-free but are significantly weaker than the base metal. Therefore, a research program was initiated to examine the causes of weld cracking in superalloys and ways to prevent it to extend the life of turbine blades made of these superalloys. In this presentation an overview of repair-weld cracking and ways to prevent/minimize it will be presented

10:15 AM Tea Break

#### Symposium A: Advanced Steels and Processing: Hot Rolling and Coating

Thursday PM August 5, 2010 Room: A Location: Cairns Convention Centre

Session Chairs: Byoung Ho Lee, Pohang Steel Company; Chris Killmore, BlueScope Steel

#### 4:30 PM

Aluminide Coating Formation on Hot Press Forming Steels: *Dong Wei Fan*<sup>1</sup>; Han S. Kim<sup>1</sup>; Rho Bum Park<sup>2</sup>; B. C. De Cooman<sup>1</sup>; <sup>1</sup>Graduate Institute of Ferrous Technology; <sup>2</sup>POSCO

In the present work, the formation of an aluminide coating prior to Hot Press Forming (HPF) was investigated. It was found that the formation of FeAl and Fe<sub>3</sub>Al phases could suppress coating degradation during the HPF process. This new method was studied as a way to simultaneously improve the coating ductility and achieve a good hot corrosion resistance. In this new method, the conventional type1 aluminized coating was transformed into an aluminide coating. This aluminide coating protects the steel from high temperature oxidation and enables its plastic deformation at high temperature. The effect of this new solution on the mechanical properties of HPF steel will be reviewed in detail.

#### 4:45 PM

Nitriding of a Nb-Microalloyed Thin Cast Strip Steel at 525 Degrees: Yuxuan Kelvin Xie<sup>1</sup>; Chen Zhu<sup>1</sup>; Julie Cairney<sup>1</sup>; Simon Ringer<sup>1</sup>; Chris R. Killmore<sup>2</sup>; Frank J. Barbaro<sup>2</sup>; James G. Williams<sup>2</sup>; <sup>1</sup>The University of Sydney; <sup>2</sup>Metallurgical Technology, BlueScope Steel

This study investigates the effect of nitrogen diffusion on a Nb microallyed CASTRIP® steels at 525 degrees in a sodium/potassium nitrate salt-bath using transmission electron microscopy (TEM) and atom probe tomography (APT). It was observed that nitriding treatment up to four hours increases the yield strength of the steel by ~50% without sacrificing ductility. Six hours nitriding causes brittle fracture of the specimen. Dispersion of ultrafine NbN precipitates present in nitrided steel was compared to the aged steel without nitrogen diffusion, where both precipitate size and number density were studied statistically.

#### 5:00 PM

Enhanced Hot-Dip Gavalizability of 590 TRIP Steel Using Oxidation-Reduction Scheme: *Sung-Hwan Kim*<sup>1</sup>; Jun-Mo Im<sup>1</sup>; Joo-Youl Huh<sup>1</sup>; Rho-Bum Park<sup>2</sup>; Jong-Sang Kim<sup>2</sup>; <sup>1</sup>Korea University; <sup>2</sup>POSCO

Transformation-induced-plasticity (TRIP) steels possess desirable properties such as high strength and excellent formability for automotive applications. To be successfully used in automotive applications, TRIP steels need corrosion protection, most effectively by means of a continuous galvanizing process. However, it has long been known that the segregation and selective oxidation of Si and Mn on the sheet surface during intercritical annealing prior to hop-dip galvanizing deteriorate the galvanizability of TRIP steels, leading to bare spots on the coating surface. This study examined the hot-dip galvanizability of 590 MPa TRIP steel, which contains 1.6 wt.% Mn and 1.5 wt.% Si, using an oxidation/ reduction scheme. The 590 TRIP steel sheets were first pre-oxidized isothermally at various temperatures ranging from 400°C to 700°C in a direct fired furnace and then the pre-oxidized sheets were annealed at 800°C in  $\mathrm{N_2}+\mathrm{H_2}$  ambience with different H, fractions and dew points in a hot-dip galvanizing simulator. Our primary focus was on the internal oxidation behavior of Si and Mn during the pre-oxidation and intercritical annealing processes. In this presentation, we will discuss how the hop-dip galavanizability of pre-oxidized 590 TRIP steel is influenced by the pre-oxidation temperature and annealing atmosphere.

#### 5:15 PM

Analysis of Advanced Strip Shape during Cold Rolling of Thin Strip: *Zhengyi Jiang*<sup>1</sup>; Xiaozhong Du<sup>1</sup>; Yanbing Du<sup>1</sup>; Dongbin Wei<sup>1</sup>; Matthew Hay<sup>1</sup>; <sup>1</sup>University of Wollongong

The demand of thin gauge strip with good quality such as the strip shape and surface finish is significantly increasing. Cold rolling is an essential method to manufacture the strip and foil products of metals. Strip shape control during cold rolling of thin strip is a significant challenge in metal rolling practice. In this study, finite element models of the strip shape during cold rolling of thin strip in both symmetrical and asymmetrical rolling were successfully developed, and the finite element simulation of the thin strip shape has been carried out in LS-DYNA. The effects of rolling parameters and surface contact features on the strip shape and profile such as the thickness distribution along the strip width and the strip edge drop have been obtained. The developed finite element model has been verified with the experimental value, which indicates they are in good agreement. The obtained results are applicable to control the rolled thin strip shape in rolling practice.

#### 5:30 PM

Analysis of Edge Crack of Thin Strip during Cold Rolling: Haibo Xie<sup>1</sup>; Zhengyi Jiang<sup>1</sup>; <sup>1</sup>University of Wollongong

Cracks in metal product decrease the strength, rigidity, toughness, plasticity and residual life, which affect the thin strip quality and productivity significantly. In this paper the experiment and mechanics research on edge crack defection of thin strip during cold rolling was investigated, and a globe analysis is applied to the problem of free edge of thin strip. The effective stress intensity factor ranges are important because they represent the major physical cause of crack growth. An investigation was carried out on the mechanism of crack initiation on thin strip edge during cold rolling. Moreover the effects of rolling parameters on crack closure are examined with data from experimental observations. The present study provides insights in the mechanics of edge crack growth that has been frequently observed in thin strip rolling.

#### 5:45 PM

**Development of Thin Cold-Rolled Sheet with High Strength and Good Formability**: *Byoung Ho Lee*<sup>1</sup>; Jeong Bong Yoon<sup>1</sup>; Jai-Ik Kim<sup>1</sup>; Ki Soo Kim<sup>1</sup>; <sup>1</sup>Pohang Steel Company

In this study, development of high strength steel sheet (Y.S. > 650 MPa) with good formability (no crack after r=0 bending) was introduced (Thickness < 0.4mm t). To develop the steel sheet with high strength and good forability, three kind of processing concepts (1) recovery heat treatment after cold rolling,



2) full recrystallization and following 2nd rolling of cold rolled sheet, 3) phase transformation just after cold rolling) were simulated for various alloying steels (Ultra low carbon, low carbon, medium carbon steel). The results shows that purposed mechanical properties can be achieved by adopting second rolling of full recrystallized cold rolled steel with newly designed alloying steel (solid solution hardening of P addition). And another possibility of thin steel sheet development with superior strength and formability by using phase transformation (strengthening by adopting hard phase) during continuous annealing line was also introduced.

#### 6:00 PM

## Research on Multi-Plate Rolling Technology of Plate Mill: Yuchuan Miao<sup>1</sup>; <sup>1</sup>Baosteel

Control rolling is widely used during plate rolling, but control rolling process has much influence on mill production. By using multi-plate rolling technology, the influence ,to some extent ,can be solved. This paper first introduce the system of control rolling, then the different mode of multi-plate rolling with one stand or double stand was given, the advantage and disadvantage of each mode were also analysed. Finally ,some actual data of multi-plate rolling technology of baosteel plate mill were given.

#### 6:15 PM

## Study on New Model of Tension Process in Continuous Rolling: *Shenbai Zheng*<sup>1</sup>; Jinhong Ma<sup>1</sup>; Zengfeng Li<sup>1</sup>; <sup>1</sup>Hebei Polytechnic University

It has been proved that tension peak occurred in the beginning of tension process, and then the tension peak changed into a stable value through the continuous rolling test on the two stands. This biting shock and steady tension dropped after impacting was direct proportional to the difference of velocity, and the tension decreased by the slip of bar in deformation zone and the changing of motor speed. Monotonously increasing original integral equation of tension could not represent the actual rolling process. Based on the speed difference, motor rigidity, changing of forward and backward slip, considering the difference of tension action about fast and slow, the dynamic equation of the kinematical mechanics was built. After tension balancing, the exit speed in front stand equaled to the speed of behind stand under the tension, and the dynamic equation translated into algebra equation, then the stable tension value attained. The simulation results of the tension beginning accorded with the experimental results. The steady tension calculation was used for the design of revolution to the draft scheme, the calculation of shock peek provided with the important reference for the technology of continuous rolling.

#### 6:30 PM

## Hot Ductility of a High Carbon Steel Produced by CSP: Gang Huang<sup>1</sup>; Kaiming Wu<sup>1</sup>; *Dezhi Wen*<sup>2</sup>; <sup>1</sup>Wuhan University of Science and Technology; <sup>2</sup>Lianyuan Steel Works, Hunan Valin Group

Hot ductility of a high carbon steel 65Mn produced by CSP (Compact Strip Production) was tested on Gleeble-3500 simulator. Results show that the great reduction of hot ductility occurred between 700°C-900°C. This is resulted from the formation of ferrite allotriomorphs along prior austenite grain boundaries and aluminum nitride precipitation along and near austenite grain boundaries. In order to prevent from surface cracking on thin slabs, it is suggested that the unbending temperature should be controlled above 950°C.

#### Symposium A: Advanced Steels and Processing: TRIP

Thursday PM August 5, 2010 Room: A Location: Cairns Convention Centre

Session Chair: Elena Pereloma, University of Wollongong

#### 2:00 PM Keynote

Microstructures and Tensile Properties of Annealed Medium Mn TRIP Steels: Young-Kook Lee<sup>1</sup>; Byoung-Jo Kwak<sup>1</sup>; Jae-Eun Jin<sup>1</sup>; <sup>1</sup>Yonsei University

Recently advanced high strength Mn steels have been paid attention to especially automobile industry because of their superior tensile strength and ductility to current automotive steels. To improve the mechanical properties of conventional TRIP steel having less than 3%Mn and approximately 10% retained austenite, the amount of retained austenite should be increased, which changes to strain-induced martensite during plastic deformation. In that viewpoint, medium Mn TRIP steels are suitable to secure more retained austenite by controlling annealing conditions such as continuous annealing and batch annealing. The effects of Mn concentration and annealing temperature and time Ms, and tensile properties were systematically investigated. Phase identification, phase fraction measurement, and partitioning of alloying elements like C and Mn between different phases were also examined using XRD, SEM-EBSD and TEM-EDS. Finally, the tensile properties of both batch- and continuous annealed medium Mn TRIP steels were compared based on microstructure and austenite stability. **2:20 PM** 

on the microstructural evolution, critical temperatures such as Ac1, Ac3, and

## **Development of a Robust TRIP800 Concept for GI/GA Automotive Applications**: *Youryeol Lee*<sup>1</sup>; Jaehyun Kwak<sup>2</sup>; Jongsang Kim<sup>2</sup>; B. C. De Cooman<sup>1</sup>; <sup>1</sup>Graduate Institute of Ferrous Technology; <sup>2</sup>POSCO

A robust TRIP 800MPa tensile strength concept was developed for automotive applications. Using extensive testing, an optimal TRIP steel composition containing 0.3 mass-% Si and 1.0 mass-% Al was identified. Galvannealing tests revealed that this TRIP steel had an ideal surface structure prior to hot dipping. Galvannealing could be achieved successfully in normal operating conditions. The presentation will give an in depth overview of the development of this new TRIP800 concept, with a special focus on achieving the optimum properties in various CGL line configurations.

#### 2:35 PM

Effect of Austenite Grain Refinement on the Deformation Induced Martensitic Transformation and High Cycle Fatigue Properties in Fe-17Mn Alloy: *Soon Gi Lee*<sup>1</sup>; Woo Kil Jang<sup>1</sup>; Hak Cheol Lee<sup>1</sup>; Kyung-Keun Um<sup>1</sup>; Jong-Kyo Choi<sup>1</sup>; <sup>1</sup>POSCO Technical Research Lab.

The High cycle fatigue properties of Fe-17Mn binary alloy with the microstructure of austenite and epsilon martensite have been studied for the first time in this alloy and the effective way of improving these properties has been addressed by grain refining. It has been known that a significant stress concentration takes place at phase and/or grain boundaries on which deformation induced epsilon martensite plates impinge at the very early stage of plastic deformation, which results in the onset of a quasi-cleavage fracture leading to the detrimental effect on the fatigue properties. With increasing austenite grains as large as a few hundreds micron, very thick and several variants of epsilon martensite plates form and impinge at the boundaries under plastic deformation which is particularly liable to fatigue crack initiation, when refined to size of a few micron, however, those types of epsilon martensite are found to be suppressed to a large extent and therefore the fatigue properties are improved.

#### 2:50 PM

Effect of Heat Treatment on Microstructure and Mechanical Properties of Low-Carbon TRIP Steel Tube: *Zicheng Zhang*<sup>1</sup>; Fuxian Zhu<sup>2</sup>; Hongshuang Di<sup>2</sup>; Yanmei Li<sup>2</sup>; Ken-ichi Manabe<sup>3</sup>; <sup>1</sup>Northeastern University - and - Department of Mechanical Engineering, Tokyo Metropolitan University; <sup>2</sup>State Key Laboratory of Rolling and Automation, Northeastern University; <sup>3</sup>Department of Mechanical Engineering, Tokyo Metropolitan University

In this study, a low-carbon transformation induced plasticity (TRIP) steel tube (Fe-0.15C-1.34Si- 1.45Mn-0.029Nb-0.024Ti wt.%) which is expected to be used in the hydroforming process was successfully fabricated using piercing, cold-drawing and two-stage heat treatment process. The optimal heat-treatment conditions (intercritical annealing "IA" and isothermal bainite treatment "IBT") were investigated to maximize the volume fraction and stability of retained austenite. The effects of temperature and holding time for IA and IBT on the microstructure of the TRIP steel tube were studied via optical microscopy. SEM, TEM and XRD. Its mechanical properties in the axial direction and hydroformability were also evaluated by conventional tensile test and flaring test, respectively. Two-stage heat-treatment carried out in a salt-bath furnace revealed that the volume fraction of retained austenite (RA) increased at first then decreased with increasing IBT holding time for a particular set of IA temperature, IA holding time and IBT temperature. It was also observed that high tensile strength of 618MPa, total elongation of 35% or above, n-value 0.23 and better hydroformability could be successfully produced in this TRIP steel tube at IA temperature of 800°C, holding for 10 min, and IBT of 410°C for 4 min holding time

#### 3:05 PM

Mechanical Properties of Micro-Alloyed TRIP Steel: *Jaehyuk Jung*<sup>1</sup>; Jagendra Singh<sup>2</sup>; Hansoo Kim<sup>2</sup>; Sungil Kim<sup>3</sup>; Jongsang Kim<sup>3</sup>; B. C. De Cooman<sup>2</sup>; <sup>1</sup>Graduate Institute of Ferrous Technology ; <sup>2</sup>Graduate Institute of Ferrous Technology; <sup>3</sup>Technical Research Lab, POSCO

In the present study, micro-alloying concept in Transformation-induced plasticity (TRIP) steels was investigated in order to improve the strength level without the deterioration of elongation and weldability. Nb and Ti were selected as micro-alloying elements. These elements are well known to contribute to grain refinement and precipitation strengthening. The addition of micro-alloying elements resulted in complex interactions with the mechanical properties of TRIP



steels: (i) loss of interstitial C in the ferrite matrix due to precipitate formation, (ii) reduction of the elongation due to incoherency between precipitates and matrix, (iii) deterioration of strain hardening resulting from a smaller grain size. In order to evaluate the effect of micro-alloying on the strain hardening, the relationship between micro-alloying and the stability of the retained austenite was examined. Several analytical methods were used to have a clear understanding of precipitation behavior including carbon replica TEM, precipitation extraction analysis and modeling. These results were correlated with the mechanical properties.

#### 3:20 PM

#### **New Pearlitic Transformation in an Ultrafine Grained High Carbon Steel:** *Yongning Liu*<sup>1</sup>; Tao He<sup>1</sup>; Fuliang Lian<sup>1</sup>; <sup>1</sup>Xi'an Jiaotong University

Pearlite and martensite are two basic and very important constituents in steels for both hypo- and hypereutectoid compositions. Pearlite is formed when steels are heated to austenite temperature, and then cooled in the furnace or in air. The pearlite is made up of lamellar cementite Fe3C and ferrite, and it is formed in a eutectoid transformation, which is a kind of diffusive transformation. Is it still true if we refine the grain size to the micron or nanometre scale? We designed an experiment to study the solid phase transformation with an ultrafine-grained pure hypereutectoid Fe-C alloy. When the grain size was reduced to the scale of  $3 \sim 5 \mu m$ , normal pearlite could not be obtained when the steel was cooled in air; instead, ferrite and ultrafine granular carbides which are  $10 \sim 50$  nm in diameter were formed in the eutectoid transformation.

#### 3:35 PM

**Ultra Fine-Grained 6wt% Manganese TRIP Steel**: *Seawoong Lee*<sup>1</sup>; Kyooyoung Lee<sup>1</sup>; B. C. De Cooman<sup>1</sup>; <sup>1</sup>Graduate Institute of Ferrous Technology

Ultra-fine grained TRIP steels (UFG-TRIP) containing 6wt%Mn were produced by intercritically annealing. An Ultra-fine grained microstructure with a grain size less than 1um was obtained. The formation mechanism of the high volume fraction of retained austenite was investigated by dilatometry, XRD and EBSD. The fraction of retained austenite was strongly dependent on the annealing temperature. The tensile properties were also found to be strongly dependent on the annealing temperature with poorer mechanical properties being observed at higher annealing temperatures. It was found that the stabilization of the retained austenite was a size-effect, made possible by the grain refinement due to the reverse martensite transformation.

#### 3:50 PM

Ultrafine Grained High Strength Low Alloy Steel with High Strength and High Ductility: *Jie Shi*<sup>1</sup>; Wenquan Cao<sup>1</sup>; Han Dong<sup>1</sup>; <sup>1</sup>National Engineering Research Center of Advanced Steel Technology, Central Iron & Steel Research Institute

In this study a C-Mn High Strength Low Alloy steel(HSLAs) was processed by quenching and martempering (QMT). An ultrafine grained duplex microstructure was obtained by means of reverse transformation of martensite, which was characterized by scanning electron microscopy equipped with electron back scattered diffraction, transmission electron microscopy and x-rays diffraction(SEM/EBSD, TEM and XRD). Mechanical properties of this processed steel were measured by uniaxial tensile testing. Microstructural observation revealed that the full hard martensitic microstucture gradually transformed into ultrafine grained dualphase (ferrite and austenite) structure with austenite volume fraction up to 30% at specific martempering conditions. The mechanical property measurements demonstrated that an excellent combination of strength (Rm~1GPa) and total elongation (A5~40%) at 30% metastable austenite condition was obtained in HSLAs. This substantially improved strength and ductility were attributed to the strain induced phase transformation of retained austenite dispersed throughout the ultrafine grained microstructure. At last it is proposed that the QMT is a promising way to produce high strength and high ductility steel products.

#### 4:05 PM Tea Break

#### Symposium B: Advanced High Temperature Structural Materials: Co- and Nb-Based Alloys

Thursday PM August 5, 2010	Room: 7
August 5, 2010	Location: Cairns Convention Centre

Session Chairs: Yuansheng Yang, Institute of Metal Research, Chinese Academy of Sciences; Yuhki Tsukada, Nagoya University

#### 4:30 PM

**Mo Effect on the Microstructure in Co-Al-W-Based Superalloys**: Fei Xue<sup>1</sup>; Zhiqiang Li<sup>1</sup>; Yanhui Chen<sup>1</sup>; *Qiang Feng*<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing

The newly developed Co-Al-W-base superalloys with stable L1<sub>2</sub>-structured  $\gamma^2$ precipitates show greater high-temperature strength than those of traditional Cobase and Ni-base superalloys. However, the  $\gamma/\gamma$ ' two-phase region of this ternary system is extremely limited according to the previous report and our investigation. In this study, the microstructure and compressive flow stress of Co-9Al-10Wbase alloys with various levels of Mo additions were investigated at 900°C after solution treatment. The results revealed that Mo promoted the formation of  $(\mu + \gamma)$ eutectic in the as-cast alloys and  $\boldsymbol{\mu}$  phase after solution treatment. After aging treatment at 900°C for 50 h, an extensive network of DO<sub>10</sub> plates was observed besides  $\gamma$  and  $\gamma'$  primary phases. Meanwhile, solid transformation from  $\mu$  phase to  $\mathrm{DO}_{19}$  phase occurred and long-term aging enhanced the formation of  $\mathrm{B}_2$  phase. In addition, higher level of Mo addition promoted the precipitation of these secondary phases significantly. The compression tests have been carrying out and the effects of Mo additions on the microstructure and compressive flow stress will be discussed. This study will provide the fundamental knowledge of Mo on phase diagrams and mechanical properties of Co-base superalloys, and optimize the design for new classes of high temperature alloys.

#### 4:45 PM

Cobalt-Based Alloys for High Temperature Applications: Rabindra Mahapatra<sup>1</sup>; *M. Ashraf Imam*<sup>2</sup>; <sup>1</sup>Naval Air Systems Command; <sup>2</sup>Naval Research Lab

The temperature capability of Ni-base superalloys has been improved by more than 300°C over the last 50 years and is approaching 1100°C. In spite of these efforts, however, a further improvement in their temperature capability is becoming more difficult due to low melting point of Ni. Considering the increasing demands on materials with higher temperature capabilities for gas turbines with higher efficiency, it is important to search for new alloys. Consequently, we are investigating a new generation of Co-base alloys through chemistry modifications utilizing phase relations and their beneficial effect of high temperature strengths as well as significant improvements in oxidation resistance at higher temperature. These attractive features, along with relatively lower density, make these alloys attractive for high temperature structural application. The oxidation behavior of these alloys was investigated up to a period of 312 hour in air from 1000 to 1300°C, and compared with the oxidation behavior of conventional Ni-base superalloy (Inconel713). Analyses of the microstructure, morphology, and composition of scales formed after oxidation will be discussed. The phase stability of the alloys after extended periods of exposure in air from 1000 to 1300°C was examined using Transmission Electron Microscope and will be presented.

#### 5:00 PM

Alloy Design of Nb-Si Based High Temperature Alloys by Phase Stability Control: *Seiji Miura*<sup>1</sup>; Tatsuichi Tanahashi<sup>1</sup>; Yoshinao Mishima<sup>2</sup>; Tetsuo Mohri<sup>1</sup>; <sup>1</sup>Hokkaido University; <sup>2</sup>Tokyo Institute of Technology

In order to spheroidize Nb<sub>s</sub>Si<sub>3</sub> strengthening phase embedded in Nb matrix for attaining good room temperature toughness of Nb-Si alloy, the authors have proposed a microstructure-control technique by combining eutectic and eutectoid reactions. Nb<sub>3</sub>Si intermetallic compound formed during solidification is a key phase for the microstructure control, but its stability is very sensitive to the alloying elements. Nb<sub>3</sub>Si disappears by adding W and Mo as small as 3 at%, while these elements are very effective for the solid solution strengthening of Nb phase. For a further alloy development, establishment of an alloy design concept based on the control of phase stability of Nb<sub>3</sub>Si is needed. Similarly to ferrous allovs such as stainless steels where Cr and Ni are added to control the stability of bcc phase and fcc phase, two alloying elements (one is stabilizing element and the other is destabilizing element for Nb<sub>3</sub>Si phase) are added to a Nb-Si binary master alloy and their microstructure is investigated using SEM. The stabilizing element is found to enlarge the composition area where Nb,Si exists even with the destabilizing element, and it is confirmed that the phase stability concept is useful for designing Nb-Si based alloys.



#### 5:15 PM

Improvement in the Mechanical Properties of an Nb-Nb Silicide Based Ultrahigh Temperature Alloy by Integrally Directional Solidification: *Xiping Guo*<sup>1</sup>; Haisheng Guo<sup>1</sup>; Yong Wang<sup>1</sup>; Ping Guan<sup>1</sup>; <sup>1</sup>Northwestern Polytechnical University

Nb-Nb silicide based ultrahigh temperature alloys have been developed as structural materials that can be employed in the temperature beyond the enduring limit of Ni-based single crystal superalloys. The integrally directional solidification was conducted with the use of special crucibles in the melt temperatures of 2000-2100°C at a withdrawing rate varying from 2.5 to 100  $\mu$ m/s. The results show that the integrally directionally solidified microstructure consists of well-aligned and couple-grown Nbss/(Nb,X)5Si3 (hereafter Nbss denotes Nb based solid solution, and X represents Ti, Hf and Cr elements) eutectic cells and primary (Nb,X)<sub>5</sub>Si<sub>2</sub> columns along the longitudinal axis of the specimens. With increase in the withdrawing rate, both diameter and lamellar spacing of the eutectic cells and the cross-sectional size of the primary (Nb,X)<sub>s</sub>Si<sub>3</sub> columns decrease, and the S/L interface undergoes a change from a coarse cellular, to cellular dendrites and finally a fine cellular morphology. Both the room temperature fracture toughness and tensile stress rupture life at 1200°C/70 MPa have been improved by integrally directional solidification significantly. The microstructure evolution during directional solidification and its effects on the deformation and rupture behaviors have been elucidated.

#### 5:30 PM

High Temperature Strength of Ir/Ir<sub>2</sub>Y Two Phase Alloys in the Ir-Pt-Y System: *Nobuaki Sekido*<sup>1</sup>; Yoko Yamabe-Mitarai<sup>1</sup>; <sup>1</sup>National Research Institute for Materials Science

Potential of C15 type Laves phases as a strengthening phase against an FCC matrix has been examined in the Ir-Pt-Y ternary system. Two phase alloys comprised of Ir solid solution (A1) and Ir<sub>2</sub>Y (C15) phases are focused in this study. Although A1 and C15 are not in equilibrium in the Ir-Y binary system, this equilibrium is attained by small Pt additions to the binary system. High temperature strength of A1/C15 mono-variant eutectic alloys was investigated by compression tests under vacuum at temperature up to 1773 K. High temperature strength of the present alloys was much lower than that of Ir based  $\gamma/\gamma$  type alloys previously reported. One reason for the low strength is the lack in solution hardening effect within the A1 matrix; for Y hardly dissolves in Ir. This solubility behavior appears to be a challenge to designing for Laves phase strengthened heat resistant alloys.

#### 5:45 PM

#### Key Factors in Quality Control of a High Cr Content Cast Ni-Base Superalloy K4648: *Liang Zheng*<sup>1</sup>; Chengbo Xiao<sup>1</sup>; Guoqing Zhang<sup>1</sup>; Guohong Gu<sup>1</sup>; Dingzhong Tang<sup>1</sup>; <sup>1</sup>Beijing Institute of Aeronautical Materials

K4648 possesses the highest Cr content in applied cast Ni-base superalloys and has been selected to manufacture the integral castings of advanced gas turbines in China. However, some problems currently arising in the application of this alloy, for instance the large blocky primary  $\alpha$ -(Cr,Ni) phase had detrimental effect on the impact ductility, and the alloy severely reacted with the silica-based ceramic core during the solidification process. The investigation result indicated that the primary a phase precipitated in the late solidification at 1190°C. The Vickers microhardness value of α-(Cr,Ni) is 6.3 GPa relative to 1.9 GPa of the  $\gamma$  matrix at the load of 0.2N. The primary a phases transformed to much brittle M<sub>23</sub>C<sub>6</sub> carbides after solid solution treatment in the range of 1180~1220°C. The cracked a or transformed M23C6 can be observed at the fracture surface and longitudinal section of the impact specimens. Moreover, the severe reaction existed in the metal/ceramic interface. The reaction products consisted mainly of the oxides which elements came from both alloy and ceramic core. For high Cr content superalloys, the solidified parameters should be controlled properly to inhibit primary a phase precipitation and more stable alumina-based core was recommended in the casting production.

#### 6:00 PM

High Temperature Alloys from Nb-Cr-Si System with Other Minor Additions: Benedict Portillo<sup>1</sup>; David Alvarez<sup>1</sup>; Alma Vazquez<sup>1</sup>; *Shailendra Varma*<sup>1</sup>; <sup>1</sup>The University of Texas at El Paso

Several Nb based alloys (Nb-20Mo-15Si-25Cr, Nb-20Mo-15Si-25Cr-5B, Nb-20Si-20cr-5Al, Nb-20Si-20Cr-5Al-5B and Nb-20Si-20Cr-(5,10)Hf) have been prepared to evaluate the oxidation resistance from 700 to 1400°C in air. The phase identification was determined by calculating the isothermal sections in this temperature range using Pandat software. The experiments involve static heating for 24 hours (short term oxidation, STO) or 7 cycles of 24 hour heating (long term oxidation, LTO). Weight gain per unit area as a function of either temperature (STO)or time (LTO) have been used to determine their oxidation resistance. However, SEM, EDS on SEM, x-ray mapping, XRD and TEM have been used to evaluate the oxide scale characterization and the influence of various

microconstituent effects have been determined. It appears that B addition may be beneficial while Al is advantageous in comparison to Hf addition. The problem of pesting, typically, in a range of temperature from 900 to 1100°C, needs to be controlled through minor additions since the alloys exhibit fairly good resistance at lower and higher temperatures up to almost 1400°C. This presentation will summarize the results of this study.

#### 6:15 PM

Effect of Second Phase on Grain Growth of Spray Formed Superalloy GH742y: *Wenyong Xu*<sup>1</sup>; <sup>1</sup>Beijing Institute of Aeronautical Materials

The influence of solid solution treatment on grain size of spray forming GH742y have been investigated, and pinning effects of the second phase on grain growth have also been studied. The results show that the limiting ultimate grain size is about 40 $\mu$ m. The equilibrium  $\gamma$ ' solvus temperature has determined to be about 1130°C by means of SEM and DSC. Carbonitride has good thermal-stability and hardly change in its amount, size and morphology at 1250°C solid solution treatment. It inhibits the movement of grain-boundary and grain growth, by pinning the grain-boundary.

#### Symposium B: Advanced High Temperature Structural Materials: Superalloys

Thursday PM	
August 5, 2010	

Room: 7 Location: Cairns Convention Centre

Session Chairs: Yoshinao Mishima, Tokyo Institute of Technology; Qiang Feng, University of Science and Technology Beijing

#### 2:00 PM

Grain Refinement and Nuclei Formation Mechanism of Ni-Based Superalloy K417: *Yuansheng Yang*<sup>1</sup>; Xiaoping Ma<sup>1</sup>; Yingju Li<sup>1</sup>; <sup>1</sup>Institute of Metal Research, Chinese Academy of Sciences

Low Voltage Pulsed Magnetic Casting (LVPMC) is developed for grain refinement castings in recent years. This paper investigates the grain refinement effect of LVPMC on superalloy K417 and deals with the grain refinement mechanism. The experimental results show that the grains in the alloy are equiaxed and refined to 60  $\mu$ m averagely. The melt flow and heat transfer during solidification are modeled and simulated to reveals the grain refinement mechanism. It is considered that the melt vibration and convection caused by the pulsed magnetic field, as well as mold temperature and undercooling contribute to the refinement of solidified grains.

#### 2:15 PM

Effect of Growth Rates on the Microstructure and Properties of Single Crystal (sc) Superalloys during High-Gradient Directional Solidification: *Taiwen Huang*<sup>1</sup>; Lin Liu<sup>1</sup>; Jun Zhang<sup>1</sup>; Shuangmin Li<sup>1</sup>; Hengzhi Fu<sup>1</sup>; <sup>1</sup>Northwestern Polytechnical University

In directional solidification, a higher thermal gradient allows higher growth rates imposed on the production of single crystal superalloys. However, the effect of higher growth rates on the microstructure and mechanical properties are seldom investigated owing to the lack of the high thermal gradient directional apparatus. In this paper, we employed a high thermal gradient up to 250-300K/cm in the experiments. Cylindrical bars of SC superalloys DD3 were directionally solidified at various growth rates (v=100 $\mu$ m/s,300 $\mu$ m/s and 600 $\mu$ m/s , respectively). The obtained microstructures showed that the primary dendrite arm spacing were decreased from 130 $\mu$ m to 50 $\mu$ m with increasing the growth rate. Moreover, the sizes of  $\gamma$ ' and the microsegregation were both significantly reduced. The best mechanical properties involving the stress rupture at 1040°C and 177MPa had been obtained in the tested bars at 300 $\mu$ m/s, rather than 600 $\mu$ m/s, since a flock of subgrain boundaries was observed at 600 $\mu$ m/s that was likely to damage the high temperature stress rupture life.

#### 2:30 PM

Partition of Alloying Elements in Co-Al-W-Base Systems: *Toshihiro Omori*<sup>1</sup>; Jun Sato<sup>1</sup>; Katsunari Oikawa<sup>1</sup>; Ikuo Ohnuma<sup>1</sup>; Ryosuke Kainuma<sup>1</sup>; Kiyohito Ishida<sup>1</sup>; <sup>1</sup>Tohoku University

Recently, we have found a new ternary compound  $Co_3(AI,W)$  [1], and the Co-Al-W-based alloys having the  $\gamma/\gamma$ ' two-phase structure show high strength at elevated temperatures [2], which is expected as constituting high-temperature materials. The phase diagram is one of the most important sources of information for development of a new type of precipitation strengthened Co-based superalloy. In this study, the effect of alloying elements on the phase equilibria of Co-Al-W system was investigated. Co-Al-W-2at%X alloys were annealed at 1173 K or



1273 K, and their equilibrium compositions were determined by EPMA. DSC measurements were also conducted. The phase equilibria between the  $\gamma$ ,  $\gamma'$ ,  $D0_{19}$  and B2 phases were determined, and it was found that Ti, V, Nb, Ta and Mo tend to concentrate in the  $\gamma'$  or  $D0_{19}$  phase in the  $\gamma/\gamma'$  or  $\gamma/D0_{19}$  equilibria, respectively, while Cr, Mn and Fe tend to be distributed in the  $\gamma$  phase. A correlation between the partition and the  $\gamma'$  solvus temperature was found.[1] J. Sato et al., Science 312 (2006) 90. [2] A. Suzuki, T. M. Pollock, Acta Mater. 56 (2008) 1288.

#### 2:45 PM

## Microstructure and Mechanical Properties of Al Added Ni<sub>3</sub>(Si,Ti) Intermetallic Thin Sheets: *Yasuyuki Kaneno*<sup>1</sup>; Yasunori Fujimoto<sup>1</sup>; Takayuki Takasugi<sup>1</sup>; <sup>1</sup>Osaka Prefecture University

The effect of Al addition on microstructures and tensile properties of coldrolled Ni<sub>3</sub>(Si,Ti) intermetallic alloys with L1<sub>2</sub> ordered structure, which were fabricated through thermomechanical processing from arc-melted ingots, were investigated. Addition of 4 and 8 at.% Al to Ni<sub>3</sub>(Si,Ti) was conducted in two ways that Al substituted for Ti site and both for Ni and Ti sites, respectively. The alloys made by the former way showed two-phase microstructure consisting of disordered fcc Ni solid solution dispersions in the L1<sub>2</sub> matrix, irrespective of Al contents, while the only 4 at.% Al alloy made by the latter way exhibited an L1<sub>2</sub> single-phase microstructure. These alloys were successfully cold-rolled to thin sheets with a thickness of 200 µm except the 8 at.% Al alloys, high-temperature yield stress was higher in the alloy made by the latter way than in the alloy made by the former one, suggesting that the single-phase microstructure consisting of whole L1<sub>2</sub> ordered structure is favorable for high-temperature tensile property.

#### 3:00 PM

**Coarsening Kinetics of Gamma Prime Precipitates in the Commercial Nickel Base Superalloy Rene' 88 DT**: *Jaimie Tiley*<sup>1</sup>; Gopal Viswanathan<sup>1</sup>; Antariksh Singh<sup>2</sup>; Soumya Nag<sup>2</sup>; Dennis Dimiduk<sup>1</sup>; Rajarshi Banerjee<sup>2</sup>; Hamish Fraser<sup>3</sup>; <sup>1</sup>Air Force Research Laboratory; <sup>2</sup>University of North Texas; <sup>3</sup>Ohio State University

Rene' 88 DT samples were subjected to different cooling rates after a supersolvus treatment, and aged for varying periods of time from 25 to 200 hours at 7600C. Primary and secondary  $\gamma'$  precipitate sizes were measured after each heat treatment. Coarsening rate constants were calculated and reported from the measured values of precipitate sizes. When describing the change in radius (r) as a function of time (t), fits between the experimental results and analysis were investigated for two types of functional relationships, r3 vs. t and r2 vs. t. The experimental rate constants derived from this analysis were compared with analytical values deduced from two different models – volume diffusion and bulk diffusion through the interface. The applicability of the two mechanisms for  $\gamma'$  coarsening has been discussed based upon the comparison between the analytically derived and experimentally observed values, of these rate constants.

#### 3:15 PM

The Effect of Alloying Elements on Microstructure and Strength Property of Dual Two-Phase Intermetallic Alloys Based on Ni<sub>3</sub>Al-Ni<sub>3</sub>V Pseudo-Binary Alloy System: Kouji Kawahara<sup>1</sup>; Taku Moronaga<sup>1</sup>; Yasuyuki Kaneno<sup>1</sup>; *Takayuki Takasugi*<sup>1</sup>; <sup>1</sup>Osaka Orefecture University

Dual two-phase intermetallic alloys that have alloy compositions of  $Ni_{75}Al_xV_{25x}$  and are composed of geometrically close packed (GCP)  $Ni_3Al(L1_2)$  and  $Ni_3V$  (D0<sub>22</sub>) phases containing Nb and Ti were studied, focusing on the effect of alloying element on microstructure and high-temperature hardness. The two-phase microstructures defined by primary  $Ni_3Al$  precipitates and eutectoid (i.e., channel) region (consisting of  $Ni_3Al$  and  $Ni_3V$  phases) were characterized in terms of volume fraction of primary  $Ni_3Al$  precipitates and interfacial area between primary  $Ni_3Al$  precipitates and channel. The high-temperature hardness was evaluated as a function of primary  $Ni_3Al$  phase precipitates, and interfacial area between primary  $Ni_3Al$  precipitates and channel region were found to be important factors affecting the hardness of the dual two-phase intermetallic alloys. Possible mechanisms responsible for the observed extra hardening were discussed, taking the role of interfaces among the constituent phases and solid solution hardening due to Nb and Ti into consideration.

#### 3:30 PM

Microstructure Evolution and Creep Behavior of a Single Crystal Nickel-Base Superalloy with [011] Orientation: *Tian Gui*<sup>1</sup>; Zhang Shu<sup>1</sup>; Xiao Li<sup>1</sup>; <sup>1</sup>Shenyang University of Technology

By means of the measurement of creep curves and microstructure observation, an investigation has been made into the creep behaviors of [110] oriented single crystal nickel-base superalloy. Results show that after full heat treated, the cubic r'phase is cohenertly embedded in the  $\gamma r$  matrix and regularly arranged along <100> orientation. During tensile creep, the cubic  $\gamma r$ 'phase is transformed into the strip-like rafted structure along the direction parallel to [001] orientation.

The change of the strain energy density in the different interfacec of the cubic r'/r phases is thought to be the driving force of the elements diffusion and the directional coarsening of r'phase. In the later stage of creep, the distortion of the rafted r'phase occurs in the region near the fracture. The deformation feature of the alloy during creep is that the slipping of the dislocations are activated on the r matrix channels and the dislocations shear in the rafted r'phase. Thereinto, after the rafted  $\gamma r$ 'phase is formed, the maximum of the shearing stress is applied along the rymatrix channels, making easily the dislocations slipping along the  $\gamma r$  matrix channel, which is thought to be the main reason of the alloy having higher strain rate and shorter creep lifetime.

#### 3:45 PM

**Phase Equilibira and Microstructure on** γ' **Phase in Co-Ni-Al-W-Cr System**: *Kazuya Shinagawa*<sup>1</sup>; Toshihiro Omori<sup>1</sup>; Katsunari Oikawa<sup>1</sup>; Ikuo Ohnuma<sup>1</sup>; Ryosuke Kainuma<sup>1</sup>; Kiyohito Ishida<sup>1</sup>; <sup>1</sup>Tohoku University

Recently, our group discovered a new  $\gamma'$  (Co<sub>3</sub> (Al, W)) phase with the L1<sub>2</sub> structure having good coherency with the fcc  $\gamma$  matrix and the  $\gamma$ - $\gamma'$  two phase microstructure is similar to that of the Ni-base superalloys strengthened by the  $\gamma'$  (Ni<sub>3</sub>Al) phase precipitation. Moreover, the Co-Al-W-base alloys exhibit good high-temperature strength. However, additional properties such as high phase stability and oxidation and corrosion resistance are also required for superalloys, therefore Ni and Cr are considered to be necessary for high temperature applications. In this study, the phase equilibria in the Co-Al-W-Ni and Co-Al-W-Ni-Cr systems and their microstructures were investigated. In the Co-Ni-Al-W system, the  $\gamma$ - $\gamma'$  two phase region and the  $\gamma'$  solvus temperature increase with increasing Ni content, while the addition of Cr decreases the stability of the  $\gamma'$  phase. The morphology of the  $\gamma'$  precipitate changes from cuboidal to spherical by the addition of Ni and was also affected by Cr, which is due to the partition change of each element between the  $\gamma$  and  $\gamma'$  phases in the Co-Al-W-Ni and Co-Al-W-Ni-Cr alloys.

#### 4:00 PM

Competitive Bi-Crystal Growth in High Thermal Gradient Directional Solidification of Single Crystal Superalloys: *Lin Liu*<sup>1</sup>; Xinbao Zhao<sup>1</sup>; Jun Zhang<sup>1</sup>; Hengzhi Fu<sup>1</sup>; <sup>1</sup>Northwestern Polytechnical University

Stray grains or misoriented grains in nickel based single superalloys could cause new grain boundaries, which have greatly depressed the mechanical properties. It is known normal dendritic crystal growth is along <001> for nickel based single superalloys. In order to explore the mechanism of the competitive growth of stray garins, bi-crystal growth was performed on a high thermal gradient directional solidification furnace. Here, differently oriented seeds were used for the directional solidification at a same solidification rate. In particular, constantly oriented bi-crystals were directional solidified at increased solidification rate. It is founded that the grains with the smaller misorientation from the cylindrical axis had the senior growth advantage. However, if the inclined dendrites of adjacent grain blocked the well-developed dendrites, the unfavorably oriented grain would overgrow the favorably oriented grain. At the same solidification rate, the larger mismatch of the orientation between the two grains, the faster the grain boundaries moved, which is irrelative of the relationship of two grains are converging or diverging. Furthermore, the larger solidification rate would lead faster overgrowth.

#### 4:15 PM Tea Break

#### Symposium C: Light Metals and Alloys: Microstructures of Magnesium Alloys

Thursday PM	Room: C
August 5, 2010	Location: Cairns Convention Centre

Session Chair: Mark Easton, CAST CRC

#### 2:00 PM

Microstructural Evolution during Friction Stir Processing of Magnesium Alloy Castings: Observation and Modelling: *Joseph Robson*<sup>1</sup>; Zhan Chen<sup>2</sup>; Song Cui<sup>2</sup>; <sup>1</sup>University of Manchester; <sup>2</sup>AUT University

Friction stir processing (FSP) has been shown to be highly effective in modifying the microstructure of cast magnesium alloys, and hence improving mechanical properties. FSP is reported to lead to a homogenized microstructure, with dissolution of eutectic intermetallics. This study investigates the mechanism by which this homogenization occurs and the influence of process parameters such as tool travel speed. Die cast plates of alloy AM50 with a fine initial microstructure and large cast ingots of AZ91 with a coarse initial microstructure have been



investigated. A model has been developed to predict the microstructural evolution of second phases and the homogenization effect of FSP. This uses a process model developed for aluminium alloys and adapted to magnesium castings to predict the thermal and flow history during FSP. It is shown that whilst the mechanical action of FSP always leads to microstructural refinement by fracturing coarse particles, dissolution and uniform distribution of solute depends critically on initial microstructure and process conditions. It is also shown that phases that are insoluble during FSP (such as the manganese containing intermetallics) are often not uniformly distributed after the process, but are concentrated in bands, as observed widely in other particle containing materials after FSP.

#### 2:15 PM

Microstructure of Laser Treated ZE41A-T5 Magnesium Alloy: *Yvonne Durandet*<sup>1</sup>; Shoujin Sun<sup>1</sup>; Milan Brandt<sup>2</sup>; <sup>1</sup>CAST CRC / Swinburne University of Technology: <sup>2</sup>CAST CRC

Modification of the microstructure of ZE41-T5 magnesium alloy substrates was investigated by laser surface re-melting and solidification using a 2.5kW Nd: YAG laser. The effects of laser power, scan rate and beam profile were examined. The microstructure of laser treated ZE41 consisted of small precipitates dispersed in a fine dendritic alpha magnesium matrix at high scan rates, and of a cellular structure plus precipitates at low scan rate. The redistribution of chemical elements depended mainly on the dwell time in the liquid stage. At high scan rates, long dwell times were achieved by splitting the laser beam in two spots trailing in the scan direction which resulted in a more homogenous distribution of Mg, Zn and Zr. Cracking due to thermal shrinkage during solidification was prevented by reducing the temperature of the melt pool. This was achieved by lowering the laser power, increasing the scan rate and laser spot size. Increasing the laser spot size in the scan direction was conducive to producing homogeneous microstructure without cracks.

#### 2:30 PM

Solute Segregation at  $\Sigma$ 11(113)[110] Grain Boundary and Effect of the Segregation on Grain Boundary Cohesion in Aluminum from First Principles: *Tokuteru Uesugi*<sup>1</sup>; Kenji Higashi<sup>1</sup>; <sup>1</sup>Osaka Prefecture University

The fracture mechanism due to the segregation of harmful impurities to grain boundary has been a well established mechanism. In aluminum alloys, segregation of impurities, such as alkali metals, at grain boundaries are sometimes discounted as a cause of grain boundary embrittlement. In this work, we investigate the energies of segregation of various solutes including Na and Ca at symmetric tilt  $\Sigma$ 11(113)[110] grain boundary in aluminum from the first principles calculations. As energy of segregation of Na and Ca is negative, these alkali elements tend to segregate at the grain boundary. Furthermore, on basis of the Rice and Wang model, we study the effect of the segregation of these alkali metals on the grain boundary embrittlement of aluminum. Our first principles calculations of energies of segregation at grain boundary and free surface show that these alkali metals behave as embrittler. The decreasing charge density at the gain boundary also demonstrates that the Na and Ca atoms form weaker metallic bonds with neighboring Al atoms in the grain boundary region.

#### 2:45 PM

#### Structure, Energetics, and Mechanical Stability of Mg-Li bcc Alloys from Ab Initio Calculations: *Zhe Liu*<sup>1</sup>; Voon Han Wong<sup>1</sup>; <sup>1</sup>Monash University

Ab initio calculation has becoming increasingly useful for material scientists and engineers in designing new alloys. In this paper, atomic volumes, mixing energies, and the elastic properties of bcc MgLi solid solutions are studied by ab initio computational methods based on the cluster expansion framework. In contrast to the phase separation shown in the composition-temperature phase diagram, we obtain a set of ground state structures, suggesting an ordering tendency of this bcc alloy instead. The concentration-dependent elastic moduli in disordered solid solutions are calculated and the obtained results are found in good agreement with available measurements. While the bulk modulus B and C44 are positive for all concentrations, tetragonal shear modulus C' is predicted to be negative for Li concentration less than 7 atomic %. Our results thus indicate that the bcc Mg-Li alloy can be stabilized with as little as 7 at.% Li. Engineering parameters such as the bulk modulus over shear modulus B/G and the Young's modulus over density Y/rho are also obtained. Analysis of B/G and Y/rho shows that bcc Mg-Li alloys with 20-50 at.% Li offer the most potential as a lightweight structural material.

#### 3:00 PM

Effect of Minor Additions of Al and Si on the Mechanical Properties of Cast Mg-3Sn-2Ca Alloys in Low Temperature Range: *Pitcheswara Kamineni*<sup>1</sup>; Suresh Kalidass<sup>1</sup>; Norbert Hort<sup>2</sup>; Karl Kainer<sup>2</sup>; <sup>1</sup>City University of Hong Kong; <sup>2</sup>GKSS Research Centre

Mg-Sn-Ca alloys have shown superior creep properties compared to the creep resistant alloy AE42. In the present study, the effects of small amounts of Al and Si additions on the mechanical properties have been investigated on Mg-3Sn-

2Ca (TX32) alloy. The Al content in the selected alloys was 0.4 wt% and the Si content was varied from 0–0.8 wt% in steps of 0.2 wt.%. The alloys were cast in pre-heated permanent molds to obtain cylindrical billets of 100 mm diameter and 350 mm length. Cylindrical specimens of 10 mm diameter and 15 mm height were machined from the cast billets. Compression tests were conducted on these specimens in the low temperature range of 25–250°C and at a strain rate of 0.0001 s<sup>-1</sup>. The alloy with only 0.4 wt% Al has shown increased strength up to a temperature of 175°C compared with TX32 base alloy. This is attributed to the solid solution strengthening of Al in Mg. The alloy with 0.4% Al and 0.2% Si has shown compressive strengths that are similar to TX32 alloy. However, with increased additions of Si (from 0.4–0.8 wt%), the alloys have exhibited reduction in strength, in particular with increase in temperature.

#### 3:15 PM

## Study of Microstructure and Fracture Behaviour in Magnesium Alloy WE54 Subjected to Multiple-Stage Ageing Treatments: *Zhou Xu*<sup>1</sup>; Jian-Feng Nie<sup>1</sup>; <sup>1</sup>Monash University

Among structural engineering alloys, magnesium alloys are by far the lightest which makes them favourable for aerospace and automotive applications. WE54 alloy is one of the most successful commercial magnesium alloys because of its excellent combination of strength and creep resistance. However, it has been reported that long-term exposure of this alloy to temperatures around 150°C can incur a ductility drop. And this embrittlement problem can affect the high temperature applications of this alloy. In the present study, the microstructures of samples subjected to secondary ageing treatments have been examined by transmission electron microscope and high-angle annular dark field - scanning transmission electron microscope. The nano-scale secondary precipitates, which are believed to have played an important role in the embrittlement problem, have been characterized in detail. A re-ageing process at 250°C was then performed on the embrittled WE54 samples, and it leads to full retrieve of the ductility, with the tensile strength being reduced to the level of the T6 condition. Tensile fracture surfaces of samples prepared under different heat treatments will also be reported.

#### 3:30 PM

#### Study on Mechanical Properties and Microstructures of Mg-5Zn-2Gd-0.6Zr New Sand-Casting Magnesium Alloy: *Guangyu Yang*<sup>1</sup>; Jiehua Li<sup>1</sup>; Wanqi Jie<sup>1</sup>; Zhong Yu<sup>1</sup>; 'Northwestern Polytechnical University

A new composition sand-casting magnesium alloy, Mg-5Zn-2Gd-0.6Zr, was developed. It was found that the room temperature mechanical properties of the alloy were superior:  $\sigma$  b=270MPa,  $\sigma$  0.2=175MPa,  $\delta$ =8%. Meanwhile, the elevated-temperature strength and creep resistance were significantly improved, comparing with Mg-5Zn-0.6Zr (ZA51A) commercial magnesium alloy. The as-cast microstructure of the alloy was mainly consisted of  $\alpha$ -Mg matrix, ( $\alpha$ +Mg<sub>3</sub>Gd2Zn<sub>3</sub>) eutectic, and little Mg<sub>3</sub>Gd phase. The secondary phases were distributed along the  $\alpha$ -Mg grain boundary with coarse netted shape. After solid-solution treatment and subsequently aging treatment for the alloy, the secondary phases re-precipitated as fine discontinues semi-netted or short rod-like precipitations, also including some fine spherical precipitations in the  $\alpha$  matrix, which were responsible for the properties improvement of the alloy.

#### 3:45 PM

Effects of Trace Cu Addition on the Microstructure and Tensile Properties of ZK60 Alloy: *Hongmei Zhu*<sup>1</sup>; Chengping Luo<sup>1</sup>; Jiangwen Liu<sup>1</sup>; Zongwen Liu<sup>2</sup>; Simon P. Ringer<sup>2</sup>; <sup>1</sup>South China University of Technology; <sup>2</sup>The University of Sydney

ZK60 alloy is one of the most important wrought magnesium alloys commercially. However, it suffers from several deficiencies like severe hot crack tendency and relatively low mechanical properties as compared to aluminum alloys. In this discussion, the microstructure and room temperature tensile properties of a ZK60-(0, 0.5 Cu, wt.%) alloy at different heat treatment states were examined using OM, XRD, SEM, TEM and tensile test. The results indicate that trace Cu addition could dramatically improve the casting properties and tensile performance of the ZK60 alloy. In the peak-aged condition, for example, the ultimate tensile strength and relative elongation were 261.4 MPa and 17.51% for the current ZK60-0.5Cu alloy, in contrast to 222.9 MPa and 5.97% for the ZK60 alloy, respectively. This could be mainly attributed to the elevated number density and refinement of the dominant strengthening phase MgZn, together with the presence of Mg<sub>2</sub>Cu and C15 Laves phase MgZnCu formed in the ZK60-0.5Cu alloy. In addition, no appreciable change in yield strength was observed. Fracture analysis showed that the added Cu significantly narrowed the cleavage facets and promoted the formation of river patter and dimple characteristics of a ductile fracture.

#### 4:00 PM Tea Break



#### Symposium C: Light Metals and Alloys: Chemical and Mechanical Behaviors of Light Alloys

Thursday PM	Room: C
August 5, 2010	Location: Cairns Convention Centre
, lagaet e, 2010	

Session Chairs: Young-Min Kim, Korea Institute of Materials Science; Tokuteru Uesugi, Osaka Prefecture University

#### 4:30 PM

**High Strength Non-Combustible Magnesium Alloys**: *Young-Min Kim*<sup>1</sup>; Han-Won Lee<sup>1</sup>; Bong Sun You<sup>1</sup>; <sup>1</sup>Korea Institute of Materials Science

Magnesium is a lightest structural material, with excellent physical and mechanical properties. In particular, a high strength to weight ratio makes these alloys ideal materials for automotive and aerospace applications. Despite such properties, however, the use of magnesium alloys is still relatively limited and untrustworthy aspect such as ignition restricts their practical applications. While the researches on improvement in strength and formability of magnesium alloys have been extensively carried out, ignition or high temperature oxidation has received little attention so far. Improvement in ignition resistance of magnesium alloys can be achieved by either formation of protective oxide layer on the melt or change in the morphology and structure of oxide by alloying. In this study, therefore, research on development of magnesium alloys with both high strength and excellent high temperature oxidation resistance has been carried out. The effects of alloving elements on high temperature oxidation behavior of cast magnesium alloys were investigated and mechanical properties of rolled/extruded magnesium alloys were evaluated as well in this study. The results indicate that Mg-Al and Mg-Zn based alloys show excellent combination of strength and ignition resistance but Mg-Sn based alloys have poor ignition resistance.

#### 4:45 PM

Microstructure and Corrosion Behavior in Al-Mg Based Alloys Subjected to Continuous Cyclic Bending and Annealing: Yoshimasa Takayama<sup>1</sup>; Yuki Kido<sup>1</sup>; Hajime Kato<sup>1</sup>; Hideo Watanabe<sup>1</sup>; <sup>1</sup>Utsunomiya University

It is well known that Al-Mg based alloys have a good corrosion resistance. However, corrosion sensitivity may rise after a kind of heat treatment, and then, dependence of microstructure on corrosion behavior is interesting to make high performance alloys. In this study, coarsening of grain structure in surface layers in 5454 and 5083 Al-Mg based alloy sheets has been made by continuous cyclic bending (CCB), which is a useful straining technique to produce the high strain on the surface layers and the lower strain in the central layer of the sheet, and subsequent annealing. The microstructure on cross-section of the samples was analyzed by SEM/EBSD technique. For the samples before and after CCB/Annealing corrosion behaviors in NaCl or HCl solution were investigated. As a result, the samples subjected to CCB/Annealing showed higher corrosion resistance on coarse-grained surface layers. Less change in the corrosive solutions was observed on the grains parallel to {111} plane.

#### 5:00 PM

Understanding the Directional Dependence of Intergranular Corrosion in Aluminium Alloys: *Steven Knight*<sup>1</sup>; Graham Clark<sup>1</sup>; Alison Davenport<sup>2</sup>; Antony Trueman<sup>3</sup>; <sup>1</sup>RMIT University; <sup>2</sup>University of Birmingham; <sup>3</sup>Defence Science and Technology Organisation

Intergranular corrosion can lead to significant problems such as failures, and increased maintenance, as a result of sub-critical crack growth or loss in section strength. This type of corrosion is found in most types of aluminium alloys, but is a particularly significant problem in aerospace aluminium alloys. The form of intergranular corrosion is not always the same and may depend on alloy composition, product form, environmental conditions and the presence or otherwise of local or global stresses. Most notable, is the occurrence of intergranular corrosion due to atmospheric corrosion, in which salts and deposits deliquesce on the surface forming discrete corrosion cells.Intergranular corrosion of aluminium alloys is usually most rapid in the rolling or extrusion direction of wrought alloy. The reasons for this are not fully understood, and may include texture effects that produce highly susceptible grain boundaries, the varying distribution of noble constituent particles, and stresses acting at a microscale. This paper will discuss the evidence for and against for the different effects mentioned.

#### 5:15 PM

Improvement of Corrosion Resistance of Extruded Mg-Zn-Y Mg/LPSO Two-Phase Alloys by Fourth Element Addition: *Shogo Izumi*<sup>1</sup>; Michiaki Yamasaki<sup>1</sup>; Yoshihito Kawamura<sup>1</sup>; <sup>1</sup>Kumamoto University

Magnesium alloys have attracted a great deal of attention as structural materials because of their light weight and high specific strength. For Mg alloys to find wider application in various fields of industry, they will need to have higher strength, ductility and corrosion resistance. Especially, the improvement of corrosion resistance is a common task we have to tackle when developing Mg alloys. Recently, high strength heat-resistant Mg-Zn-rare earth (RE) alloys were developed. The Mg-Zn-Y alloy consists of an alpha-Mg matrix and a long period stacking ordered (LPSO) structure. The LPSO-phase in Mg/LPSO two-phase alloys plays as strengthening-component in mechanical property. However, from viewpoint of corrosion science, LPSO phase is a secondary phase that may cause the potential difference to the matrix. The main reason for the low corrosion resistance of Mg alloys is galvanic attack due to impurities, alloying elements and secondary phases. The technique for improving corrosion resistance of Mg/LPSO two-phase alloys is required. This paper provides a solution for improvement of corrosion property of Mg-Zn-Y Mg/LPSO alloys by addition of fourth elements such as Al and lanthanoids. Role of added element for corrosion property will be investigated using electrochemical techniques.

#### 5:30 PM

The Effect of Cd Addition on NDE of As-Cast Mg-Cd Alloy in 0.1M NaCl Solution: Junhua Dong<sup>1</sup>; <sup>1</sup>Institute of Metal Research, CAS

The effect of Cd addition on NDE of as-cast Mg-Cd alloy in 0.1M NaCl solution was investigated using gas collection method, potentiostatic current decay test and in-situ Laser Raman Spectrum. The experimental results showed that, in the cathodic region the addition of Cd reduced the hydrogen evolution and the magnesium hydride formation, while in the anodic region the addition of Cd restrained the NDE but slightly increased the anodic dissolution.

#### 5:45 PM

Crystallographic Orientation Relationship between Discontinuous Precipitates and the Matrix in Commercial Mg Alloys: *Takumi Gonoji*<sup>1</sup>; Kenji Matsuda<sup>1</sup>; Kaname Fujii<sup>2</sup>; Tokimasa Kawabata<sup>1</sup>; Yasuhiro Uetani<sup>3</sup>; Susumu Ikeno<sup>1</sup>; Katsumi Watanabe<sup>1</sup>; <sup>1</sup>University of Toyama; <sup>2</sup>Industrial Research Institute of Ishikawa; <sup>3</sup>Toyama Prefectural University

AZ91 is the most popular Mg alloys because of its superior castability, mechanical strength and ductility. The Mg17Al12 intermetallic compound is the only precipitate formed during ageing after the solution heat treatment. Discontinuous precipitates exist in grain boundary randomly, and continuous precipitates exist in transgranular. The past report mainly aimed at the continuous precipitates and orientation relationship in the matrix. However, the study on the discontinuous precipitates and its orientation relationship in the matrix has not been reported. In this study, there specimen of commercial cast AZ91 magnesium alloy and rolling before the solution heat treatment in this alloy were prepared. The two types of specimens were solution heat-treated and then guenched into water. Cross section discontinuous precipitates of TEM samples were prepared using the focused ion beam (FIB). TEM observations were performed to investigate discontinuous precipitation and crystallographic orientation in matrix. The cross section of discontinuous precipitation in AZ91 magnesium alloy was investigated, and it showed the same orientation relationship as the Burgers. Potter and Crawley orientation relationship. However, longitudinal direction of discontinuous precipitation was deviated from regular direction. All longitudinal direction plotted in pole figure. It had the zone axis of [1 1 1] direction of the precipitates.

#### 6:00 PM

**Experimental Investigation of the Dynamic Behavior of Aluminum Foams**: *Shanqing Xu*<sup>1</sup>; Dong Ruan<sup>1</sup>; John Beynon<sup>1</sup>; Guoxing Lu<sup>2</sup>; <sup>1</sup>Swinburne University of Technology; <sup>2</sup>Nanyang Technological University

The potential application of metallic foams in vehicle safety components depends on a thorough understanding of the material properties under dynamic loading conditions that are representative of crashes and other impacts. The compressive behaviour of two closed cell aluminium foams (Alporas and Cymat) were extensively investigated over a wide range of strain rates, 0.001 to 1000/s. Quasi-static, intermediate strain rate and high strain rate compressive tests were conducted on MTS material testing system, Instron high rate test machine and split Hopkinson pressure bar (SHPB), respectively, with an emphasis on the intermediate strain rates (1 to 200/s). High speed camera videos show that localised deformation was observed in Alporas foam specimens, while for Cymat foams, cells collapsed in the middle region. The stress-strain curves for Alporas foams are comparatively smooth and regular, while for Cymat foams, those curves oscillated dramatically. The relative density plays the primary role on the dynamic behaviour of aluminium foams. Alporas foams exhibit strain rate



sensitivity, i.e. with an increase in strain rate, the normalized plateau stress and energy absorption capacity increase. However, Cymat foams are not sensitive to strain rates, i.e. no significant increase in energy absorption is observed.

#### 6:15 PM

Structural Behavior of FRP Lighting Pole System: *Sun Hee Kim*<sup>1</sup>; Gi-Nam Kim<sup>1</sup>; Soon-Jung Hong<sup>1</sup>; Chang-Won Kim<sup>1</sup>; Soon-Jong Yoon<sup>1</sup>; Won-sup Jang<sup>2</sup>; <sup>1</sup>Hongik University; <sup>2</sup>Naekyung Engineering Co., Ltd.

A street lighting system is an essential structure for the safety of traffic. Most of the lighting pole is made with steel and it is vulnerable to corrosion due to its hazardous surrounding environment. To mitigate such corrosion problems, stainless steel and iron steel are usually adopted, but they are also usually manufactured by hand, so it is not economical. Therefore, to overcome such problems new type FRP lighting pole is proposed. In recent years, FRP materials in the construction market are popularly utilized as an alternative against conventional construction materials. FRP material has many advantages such as high chemical resistance, electro-magnetic transparency, high specific strength and stiffness, etc. In the study, structural behavior of FRP lighting pole is compared with that of conventional lighting pole structures and we proposed the cross-section configuration of the FRP lighting pole with details on the manufacturing procedure.

#### Symposium E: Solidification, Deformation and Related Processing: Solidification II

Thursday PMRoom: 2August 5, 2010Location: Cairns Convention Centre

Session Chair: Ma Qian, The University of Queensland

#### 2:00 PM Keynote

Hypereutectic Al<sub>2</sub>O<sub>3</sub>/YAG/ZrO<sub>2</sub> In Situ Composite Prepared by Laser Zone Melting: Kan Song<sup>1</sup>; *Jun Zhang*<sup>1</sup>; Haijun Su<sup>1</sup>; Lin Liu<sup>1</sup>; Hengzhi Fu<sup>1</sup>; <sup>1</sup>Northwestern Polytechnical University

 $Al_2O_3/YAG/ZrO_2$  eutectic ceramic in situ composite has now been regarded as new generation of high temperature structural material due to its excellent performance even close to its melting point. In this paper, hypereutectic  $Al_2O_3/YAG/ZrO_2$  in situ composite is prepared by the horizontal laser zone melting technique. The relationship between the solidification microstructure and the processing parameter is studied. Pore free samples with smooth surface are obtained. The minimum eutectic spacing is only 0.20µm when the laser scanning rate is 48mm/min. Compared with hypoeutectic and eutectic  $Al_2O_3/YAG/ZrO_2$ , hypereutectic  $Al_2O_3/YAG/ZrO_2$  shows more regular and fine microstructure at the same solidification condition. It is considered that the weakly faceted  $ZrO_2$  with increasing quantity counteracts the typically faceted growth of  $Al_2O_3$  and YAG, which results in the relatively regular eutectic morphology. Meanwhile, it is found that the lamellar spacing remains almost as constant at very high scanning rate, which has never been reported in previous research.

#### 2:20 PM

**Directional Solidification and Characterization and of** Al<sub>2</sub>O<sub>3</sub>/Er<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> **Eutectic In Situ Composite by Laser Zone Remelting**: *Haijun Su*<sup>1</sup>; Jun Zhang<sup>1</sup>; Yangfang Deng<sup>1</sup>; Kan Song<sup>1</sup>; Lin Liu<sup>1</sup>; Hengzhi Fu<sup>1</sup>; <sup>1</sup>State Key Laboratory of Solidification Processing

Directionally solidified (DS) oxide eutectic in situ composites are attracting increasing attention because of their unique properties and potential applications to high temperature structural materials, optical or electronic devices. Among the alumina-based eutectic composites, DS Al<sub>2</sub>O<sub>3</sub>/Er<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>(EAG) eutectic is considered to be promising candiate for use as selective emitter at high temperature. In this work, eutectic Al<sub>2</sub>O<sub>3</sub>/EAG rods having smooth surface and full density are successfully prepared by directional solidification using the laser zone remelting method, aiming to investigate the growth characteristic of this novel binary eurectic under high temperature gradient. The microstructure is investigated by scanning electron microscopy (SEM), energy disperse spectroscopy (EDS) and X-ray diffraction (XRD). The Al<sub>2</sub>O<sub>3</sub>/EAG eutectic presents a fine irregular network structure consisting of only a-Al<sub>2</sub>O<sub>3</sub> and Er<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> phases without grain boundaries and amorphous phases between interfaces. The eutectic interphase spacing is strongly dependent on the laser scaning rate, decreasing at the submicron levels for the samples grown at high rate. Furthermore, the microstructural formation and evolution of the composite are analysed.

#### 2:35 PM

#### Fabrication of Porous Metals with Directional Pores through Unidirectional Solidification of Gas-Dissolved Melt: *Hideo Nakajima*<sup>1</sup>; <sup>1</sup>Osaka University

Porous metals with long cylindrical pores aligned in one direction are fabricated by unidirectional solidification using pressurized gas method (PGM) and thermal decomposition method (TDM). The pores are evolved from insoluble gas when the molten metal dissolving the gas is solidified. In the conventional PGM, hydrogen pressurized in a high-pressure chamber is used to dissolve hydrogen in the melt. However, the use of high-pressure hydrogen is not desirable because of inflammable and explosive gas, in particular, for scaling up to mass production of lotus/Gasar metals. In order to overcome this shortcoming, the thermal decomposition method was developed as an alternative simple fabrication method. Gas-forming compounds such as hydrides were added into the molten metal to fabricate lotus/Gasar metals. The porosity and pore size were controlled by the amount of gas-forming compounds, solidification rate, atmospheric pressure, etc. It is significant that in TDM fine clusters of metallic compound formed after dissolving the gas-forming compound can serve as an nucleation sites of the gas evolution so that the uniformity of pore size is improved compared with PGM. The TDM method is applied to three fabrication techniques of the mold casting technique, continuous zone melting technique and continuous casting technique.

#### 2:50 PM

Microstructure Evolution and Compression Properties of a Directionally Solidified Ni-24.8%Nb Hypereutectic Alloy: *Shuangming Li*<sup>1</sup>; Binglun Jiang<sup>1</sup>; Hengzhi Fu<sup>1</sup>; <sup>1</sup>Northwestern Polytechnical University

At normal solidification conditions, in-situ composites of Ni-24.8%Nb hypereutectic alloy could be produced at the growth rate below  $5\mu$ m/s with a thermal gradient of 180K/cm and the low productivity remarkably restricts the application of this kind of in-situ composites. In this paper, we proposed an approach that employed an abrupt growth rate to make the in-situ composites grow stably out of the coupled zone. In-situ composites of Ni-24.8%Nb hypereutectic alloy were obtained at the growth rate of 100µm/s and the productivity was greatly improved. This value is the same order magnitude imposed on the single crystal superalloys. The compression properties including tensile strength and plasticity were investigated based on the different microstructures involving the coupled eutectics and non-coupled eutectics. The results showed that the cracks distribution and extension mainly focused on the primary dendrites of Ni3Nb phase in the non-coupled eutectics and in-situ composites with the entirely coupled eutectics have improved mechanical properties and differently deformed behaviors.

#### 3:05 PM

Effect of Growth Rate and Zr Addition on the Microsegregation during Directional Solidification of Cu-Ag-Zr Alloy: *Bok-Hyun Kang*<sup>1</sup>; Woo-Hyun Lee<sup>1</sup>; Ki-Young Kim<sup>1</sup>; Hoon Cho<sup>2</sup>; <sup>1</sup>Korea University of Technology and Education; <sup>2</sup>Korea Institute of Industrial Technology

Although Cu-Ag alloys possess good electrical conductivity with satisfactory strength, the actual solubility of Ag in Cu matrix is normally higher than its equilibrium solubility. This non-equilibrium high solubility of Ag can deteriorate the mechanical and electrical properties. In order to enhance the mechanical properties and the electrical conductivity further along with the slight increase of precipitation, addition of elements such as Zr and Sr for reducing the solubility of Ag is of interest recently. The effect of growth rate and Zr addition on the microsegregation during directional solidification of Cu-Ag-Zr alloy was investigated. Cu-2wt.%Ag-2wt.%Zr alloy was directionally solidified under argon atmosphere, applying different growth rates (V=1-200 um/s) in a Bridgeman furnace. The influence of growth rate was investigated by observing the microstructure and measuring the solute compositions within the Cu-matrix and dendrite boundary. Addition of Zr pushed out the Ag solute to dendrite boundary from Cu matrix which was investigated by comparing with the previous results of Cu-2wt.%Ag binary alloy. Experimental results showed that with increasing growth rate, both the primary and secondary arm spacing decreased and micro hardness increased. Zr solute was not observed in the Cu-matrix. Ag solute at dendrite boundary increased with increased growth rate.

#### 3:20 PM

Non-Equilibrium Solidification in Cu-Mg-Sn Alloys for Tribological Applications: Agustín Bravo<sup>1</sup>; *Rafael Schouwenaars*<sup>1</sup>; Victor Jacobo<sup>1</sup>; Armando Ortiz<sup>1</sup>; <sup>1</sup>Universidad Nacional Autonoma de Mexico

Cu-Pb alloys are the standard alloy in the journal bearings of heavy combustion engines. Pb provides tribological compatibility by preventing adhesion between the bearing surface and the shaft. Elements with low chemical affinity for Fe promote compatibility, but most such elements are either environmentally harmful or expensive. Only Sn and Mg seem to provide a possibility to develop a copperbased alloy capable of substituting the old Cu-Pb alloys. This work describes the casting of such alloys in the form of thin ingots to produce cold rolled strip, as is



done for commercial Al-Sn-based ductile triboalloys. Casting of Cu-Mg alloys is notoriously difficult, but sound ingots could be produced with simple laboratory equipment, yielding slabs of Cu<sub>1</sub>Mg<sub>1</sub>Sn, Cu<sub>1</sub>Mg<sub>2</sub>Sn, Cu<sub>3</sub>Mg<sub>1</sub>Sn and Cu<sub>3</sub>Mg<sub>5</sub>Sn. Cooling curves were monitored by embedded thermocouples. Invariant points could be identified after appropriate filtering of the signal but did not correspond to the ternary equilibrium. Segregation and non-equilibrium phases were confirmed by metallography. The as-cast material is superior in strength, ductility and wear characteristics compared to other Cu-based triboalloys, but its hardness is probably too high for applications against non-surface treated shafts and the high-Mg compositions may lack the workability to allow rolling to thin strips for journal bearings.

#### 3:35 PM Tea Break

#### Symposium E: Solidification, Deformation and Related Processing: Solidification III

Thursday PM	Room: 2
August 5, 2010	Location: Cairns Convention Centre

Session Chair: Dayalan Gunasegaram, CSIRO Light Metals Flagship

#### 4:30 PM

In-Situ Observation of Transformation from the  $\delta$  Phase to the  $\gamma$  Phase during Solidification of Carbon Steels: *Hideyuki Yasuda*<sup>1</sup>; Tomoya Nagira<sup>1</sup>; Masato Yoshiya<sup>1</sup>; Noriaki Nakatsuka<sup>1</sup>; Akira Suigiyama<sup>2</sup>; Kentaro Uesugi<sup>3</sup>; Keiji Umetani<sup>3</sup>; <sup>1</sup>Osaka University; <sup>2</sup>Osaka Sangyo University; <sup>3</sup>JASRI

Time-resolved X-ray imaging has been developed for observing solidification phenomena of metallic alloys. A significant advantage is to observe solidification in-situ for the alloy system of interest. Since, in conventional carbon steels, the volume change due to the transformation from the  $\delta$  phase to the  $\gamma$  phase can cause deformation of solidifying shell, it is important to understand the transformation manner. However, it is unclear how the the  $\gamma$  phase forms during solidification. This paper shows the time-resolved imaging of the transformation from the  $\delta$  phase to the  $\gamma$  phase during solidification of carbon steels. The observation was performed at a beam lime BL20B2 of SPring-8. In the Fe-0.44mass%C alloys, there were two different transformation modes. One is that the  $\gamma$  phase was produced through the the peritectic reaction. This mode was selected at the relatively low cooling rates (<10K/min). The other is that the  $\gamma$  phase was produced in the solid state after the solidification with the  $\delta$  phase completed. This mode was often observed at the higher cooling rates. The observed modes will provide valuable knowledge to evaluate stress and strain, which are induced by the transformation, in the solidifying shell.

#### 4:45 PM

#### Investigation of the Solidification Mechanisms in the Sn-Cu(-Ni) Lead-Free Solder System: *Tina Ventura*<sup>1</sup>; Younghee Cho<sup>1</sup>; Arne Dahle<sup>1</sup>; <sup>1</sup>The University of Queensland

Pb-free solders based on near-eutectic Sn-0.7Cu-xNi alloys provide excellent solderability during wave soldering with cost advantages compared to Agcontaining alternatives. However, there is only limited knowledge about the solidification mechanisms in this alloy system and, furthermore, the ternary Sn-Cu-Ni phase diagram is not yet fully established. In this study, unidirectional solidification has been conducted in a Bridgman furnace using both binary alloys from the Sn-Cu6Sn5 system and ternary Sn-rich Sn-Cu-Ni alloys. The influence of Ni additions on the solidification mechanisms is assessed by comparing the microstructures of the ternary and binary alloys. Furthermore the crystallographic properties of the various intermetallics present and its influence on morphology have been studied using TEM. The results are used to discuss the contrasting Sn-Cu-Ni phase diagrams reported in the literature. The results demonstrate the complex phase relations in the Sn-Cu alloy system, and the important role of a few ppm of various solute elements.

#### 5:00 PM

Modelling and Experiments Concerning Dendrite Re-Melting and Its Role in Microstructural Evolution in Spray Formed Ni Superalloys: *Yong Zhang*<sup>1</sup>; Zhipeng Guo<sup>2</sup>; Guoqing Zhang<sup>1</sup>; Jiawei Mi<sup>2</sup>; Patrick Grant<sup>2</sup>; <sup>1</sup>Beijing Institute of Aeronautical Materials; <sup>2</sup>University of Oxford

It has long been known that an equiaxed microstructure forms in almost any alloy system spray formed under sensible conditions. It has been accepted generally that thermal and/or mechanical shock of the solid component within the atomised droplets experienced at deposition causes endemic fragmentation of the dendrites comprising this solid/semisolid component, and that these fragmented dendrite arms then act as embryonic grains to promote a grain multiplication effect, resulting in the final uniform equiaxed microstructure. However, neither the fragmentation process nor the accompanying partial re-melting that the solid component must experience at deposition have been studied in any detail. In this paper, we use a Gleeble 3500 physical simulator apparatus to create controlled thermal and mechanical shock conditions in solid-liquid mixtures generated from Ni superalloy IN718 atomised powder, which simulated the environment of droplet deposition during twin-atomiser spray forming in IN718 at BIAM. The experiments were complemented by phase field modelling studies of these phenomena at Oxford. A critical droplet diameter and thermal condition (heating rate and temperature) for promoting dendrite fragmentation were indentified and processing strategies for enhancing equiaxed grain refinement in twin-atomiser spray forming are proposed.

#### 5:15 PM

Monte Carlo Simulation of Thermophysical Properties for Al-Ce Liquid Alloys: Wen-Jing Yao<sup>1</sup>; Nan Wang<sup>1</sup>; <sup>1</sup>Northwestern Polytechnical University

Amorphous phase can be obtained in Al-RE alloys by melting spinning technique whereas it does form in other Al-Based alloys such as Al-Cu etc. Considering the larger atomic size of RE elements than Cu, the effect of diffusion coefficient should play a key role. Due to the lack of the experimental data, the diffusion coefficient and other thermophysical properties of liquid and undercooled liquid Al-RE alloys should be investigated by simulation work. The Monte Carlo method with EAM potential is applied to simulate the liquid Al-Ce binary alloy system and the thermophysical properties including surface tension ( $\sigma$ ), viscosity ( $\eta$ ) and diffusion coefficient (D) of Al-4, 8, and 10 at%Ce liquids are determined. The simulated  $\sigma$  values decrease with the increasing of temperature. Based on the relationship between  $\sigma,\eta$  and D, the various viscosity and diffusion coefficient of liquid Al-Ce alloys under different temperatures were determined. The comparison of the simulated results with some experimental measurements is discussed, indicating that the simulation method and EAM parameters in simulation are acceptable. The dependence of viscosity and diffusion coefficient of liquid Al-Ce alloys on the composition and temperature are established to be helpful in further investigation of Al-Ce amorphous.

#### 5:30 PM Keynote

**Optimization of the Cooling Process of a Heavy Hydraulic Turbine Runner Band Casting in Heat Treatment**: *Jinwu Kang*<sup>1</sup>; Tianjiao Wang<sup>1</sup>; <sup>1</sup>Tsinghua University

Heat treatment contributes to the deformation of castings as well as casting process. It is necessary to study the deformation behavior of casting during heat treatment and the effect of processing parameters on deformation. Hydraulic turbine lower band castings, of the matensitic stainless steel (Cr13%,Ni5%,Mo1%), is susceptible to deformation in heat treatment process. The coupling analysis of forced air flow, heat transfer, and thermal stress in heat treatment process of a turbine band casting with outer diameter of 8000mm is carried out by using ANSYS software. The air flow fields, temperature fields and stress fields are obtained. And the deformation of the band is predicted. The band undergoes significant deformation to be eclipse shaped in the cooling process of normalizing. It is found that the forced air flow pattern is a key factor which influences the cooling evenness and efficiency. It is optimized by adjusting the cooling fans' orientation relative to the casting, and consequently, the cooling efficiency and evenness are improved and deformation is significantly reduced.

#### 5:50 PM

Application of a Criterion for Cold Cracking to Casting High Strength Aluminium Alloys: *Mehdi Lalpoor*<sup>1</sup>; D.G. Eskin<sup>1</sup>; Hallvard G. Fjær<sup>2</sup>; Andreas Ten Cate<sup>3</sup>; Nick Ontiji<sup>3</sup>; Laurens Katgerman<sup>4</sup>; <sup>1</sup>Materials Innovation Institute; <sup>2</sup>Institute for Energy Technology; <sup>3</sup>Corus RD&T; <sup>4</sup>Delft University of Technology

Direct Chill (DC) casting of high strength 7xxx series aluminium alloys is difficult mainly due to solidification cracking (hot cracks) and solid state cracking (cold cracks). Poor thermal properties along with extreme brittleness in the ascast condition make DC-casting of such alloys a challenging process. Therefore, a criterion that can predict the catastrophic failure and cold cracking of the ingots would be highly beneficial to the aluminium industry. The already established criteria are dealing with the maximum principal stress component in the ingot and the plane strain fracture toughness (KIc) of the alloy under discussion. In this research work such a criterion was applied to a typical 7xxx series allow which is highly prone to cold cracking. The mechanical properties, constitutive parameters, as well as the KIc values of the alloy were determined experimentally in the genuine as-cast condition and used as input data for the finite element package ALSIM5. Thermomechanical simulations were run for billets of various diameters and the state of residual thermal stresses was determined. Following the contour maps of the critical crack size gained from the model, the casting conditions were optimized to produce a crack-free billet.


#### 6:05 PM

Effect of Oxygen on the β-Grain Size of Cast Titanium: Michael Bermingham<sup>1</sup>; Stuart McDonald<sup>2</sup>; Matthew Dargusch<sup>1</sup>; David StJohn<sup>2</sup>; <sup>1</sup>Defence Materials Technology Centre; <sup>2</sup>CAST CRC

Grain refinement of titanium alloys during solidification is believed to have many benefits for processing and properties. Recent work has emphasized the importance of solute elements in grain refining cast titanium and it was demonstrated that the growth restriction factor is useful for predicting the grain refining effectiveness of solute elements in titanium. Despite oxygen being the major impurity element present in titanium alloys and having been previously identified as a theoretical growth restricting solute, its effect as a ß-grain refiner is still unexplored. This paper investigates the effect of oxygen on the grain size in cast titanium alloys.

#### Symposium F: Modelling and Simulation of Microstructures and Processes: Materials Modelling II

Thursday PM	Room: 5	
August 5, 2010	Location:	Cairns Convention Centre

Session Chairs: Yunzhi Wang, Ohio State University; Mojmir Sob, Masaryk University, Faculty of Science

#### 2:00 PM Keynote

Monte-Carlo Modeling of Recrystallization Kinetics of Cold-Rolled Titanium: Young Bum Chun<sup>1</sup>; Lee Semiatin<sup>1</sup>; *Sun-Keun Hwang*<sup>2</sup>; <sup>1</sup>ARC Centre of Excellence for Design in Light Metals, Department of Materials Engineering, Monash University; <sup>2</sup>Inha University

The recrystallization behavior of cold-rolled, commercial-purity titanium was studied experimentally and with Monte-Carlo (MC) modeling. Utilization of EBSD-OIM as input for MC modeling resulted in realistic predictions of recrystallization kinetics, microstructure and texture, which were in good agreement with experimental results. MC modeling of recrystallization kinetics predicted that the non-uniform stored energy distribution, heterogeneous nucleation of recrystallization and recovery in combination leads to a negative deviation from linear JMAK kinetics. It was found that concurrent recovery that takes place during recrystallization is an important process that controls both the overall recrystallization kinetics and the deviation of linear JMAK kinetics. On the other hand, the non-uniformly distributed stored energy itself has little effect on the negative deviation from JMAK kinetics but intensifies the deviation when heterogeneous nucleation is combined. MC Modeling results also revealed that heterogeneous nucleation of recrystallized grains and their early impingement in local areas of high deformation are essential for producing a lognormal distribution of grain size and a typical recrystallization texture of rolled titanium.

#### 2:20 PM Keynote

**Solving Complex Thermal and Mass Transport Problems with the Lattice Monte Carlo Method**: Thomas Fiedler<sup>1</sup>; *Graeme Murch*<sup>1</sup>; Irina Belova<sup>1</sup>; <sup>1</sup>The University of Newcastle

The widespread availability of inexpensive computational power has led to renewed interest in developing Monte Carlo methods in science and technology. The Lattice Monte Carlo (LMC) method developed by the authors over the last few years is an unusually powerful method in which a given phenomenological thermal or mass transport problem is mapped onto a fine-grained lattice which is then analyzed with discrete random walk methods. In this paper, by way of a number of case studies, we describe some of the recent progress with the LMC method. For thermal transport examples we describe LMC applications to the calculation of effective thermal conductivities in multiphase materials, temperature profiles in phase change materials and porous metals, including an example where CT scans of a real material are used directly in the calculation. For mass transport examples, we describe LMC applications of the delineation of grain boundary diffusion regimes in solids, the calculation of effective ionic conductivities in composite electrolytes and the calculation of concentration profiles for diffusion with reaction and interface sharpening by interdiffusion. Extensions to thermal conduction combined with convective transport and thermal conduction combined with mass transport (Soret effect) will also be described.

#### 2:40 PM

Advanced Material-Technological Modeling of Complex Dynamic Thermomechanical Processes: *Bohuslav Masek*<sup>1</sup>; Hana Jirkova<sup>1</sup>; Jiri Malina<sup>1</sup>; Stepan Jenicek<sup>1</sup>; <sup>1</sup>University of West Bohemia in Pilsen, Research Centre of Forming Technology

Material-technological modelling has made great progress over the last years thanks to the new possibilities opened up by developments in sensor technology, and especially in new methods of control supported by innovative electronic elements and electronic circuits. One such device is the thermomechanical simulator which was built in the laboratories of the Research Centre of Forming Technologies (FORTECH), in Pilsen, in the Czech Republic. Thanks to new knowledge and technical equipment the majority of technological processes or even technological chains can be modelled. The most considerable and most important innovation in the material-technological modelling process. The present options even allow modelling of highly dynamic processes, such as rolling wire. This paper presents the broad possibilities of modern material-technological modelling. The process of detecting technical and manufacturing problems during rolling and the possibilities of failure elimination are introduced on a practical example.

#### 2:55 PM

Understanding Phase Stability of FeCr Alloys through Synergistic Modelling and Experiment: *Maria Samaras*<sup>1</sup>; Camelia Borca<sup>1</sup>; Anne-Christine Uldry<sup>1</sup>; Andi Idhil<sup>1</sup>; Maximo Victoria<sup>1</sup>; <sup>1</sup>Paul Scherrer Institute

Defects, clustering and segregation are issues which alter the structural and mechanical properties of a material. In ferritic steels these issues are of great importance in understanding the lifetime of the materials. Indeed, depending on the Cr content, the material can be in a state of short-range order (below approx. 10%Cr) or contain clusters of Cr atoms above this concentration. Multiscale materials modelling is emerging as a next step in research and development programs internationally. Multiscale modelling promises the ability to go beyond the life-time assessment methods used today, to reduce the time and costs of experimental programs, bring forth a fundamental understanding of the issue at hand and in the long term provide the ability to perform materials design. Electronic structure calculations will be shown that enable an understanding of the structural configurations of the Fe-Cr alloy. To validate such calculations, which are at the fundamental building block of multiscale modeling schemes, model validation of these results are vital. Synchrotron based X-ray microanalysis techniques are used to characterise the material by analysis of forces on the local structural environment. These results will be discussed in the framework of modelling Fe-Cr alloys

#### 3:10 PM

## Evaluation of Plastic Anisotropy in Rolled Ferritic Stainless Steel: Sungyeun Won<sup>1</sup>; <sup>1</sup>POSCO

Plastic anisotropy evolution in ferritic stainless steel sheets was investigated experimentally and theoretically. As-received rolled sheets were rolled in three directions to get different types of platic anisotropy. The anisotropy was evaluated by flow stress and Lankford coefficient of tensile tests in various directions in the sheets. Crystallographic textures were measured by X-ray diffraction, from which crystal orientations were determined and then incorporated into the finite element polycrystal model. The anisotropy developed by the numerical simulation of rolling (plane strain compression) showed good agreement with the experiment.

#### 3:25 PM

Multiscale Particle-in-Cell Analysis of Solids with Heterogeneous Microstructures: *Alireza Asgari*<sup>1</sup>; Chunhui Yang<sup>1</sup>; Peter D. Hodgson<sup>2</sup>; Bernard F. Rolfe<sup>1</sup>; <sup>1</sup>School of Engineering, Deakin University; <sup>2</sup>Institute for Technology, Research and Innovation. Deakin University

A multiscale analysis framework for solids with heterogeneous microstructure is presented. The proposed framework is based on computational homogenization technique with Particle-In-Cell (PIC) method at both macro and micro levels. The method is used to model metallic materials with complex and realistic microstructural data. The simplified circular, ellipsoidal or spherical representative volume elements are compared with these complex microstructural models. In addition, the results from PIC method are compared with Finite Element method and analytical calculations. The pros and cons of each method in terms of accuracy, speed, predictive power of the method and ease of implementation are presented. The results of these comparisons are used to define areas of application of the multiscale particle-in-cell method.



#### 3:40 PM

Molecular Dynamics Based Observations of Grain Boundaries and Lattice Defects Functions in Fine Grained Metal: *Toshihiro Kameda*<sup>1</sup>; Baorong Zhang<sup>1</sup>; <sup>1</sup>University of Tsukuba

In order to study the characteristics of fine grained polycrystalline metals, it is important to recognize the function of grain boundaries (GB), crystal defects such as dislocation and/or nanoscale voids, since the fraction of GB increases as grain sizes decreases, the deformation process of these metals could be different from those in larger size grains. In this study, we first evaluate the hypothesis that GB behaves as dislocation source and sink during the deformation of fine grained metal, then compare the behavior between GB and a tiny defect from the view point of dislocation source and sink phenomena. Since continuous dislocation supplies could be considered as the key issue to improve the toughness of fine grained metals, this concept could be helpful to design next generation polycrystalline metals.

#### 3:55 PM

Deformation Mechanism of Au Single-Crystalline Nanowires: A Molecular Dynamics Approach: *Na-Young Park*<sup>1</sup>; Ho-Seok Nam<sup>1</sup>; Pil-Ryung Cha<sup>1</sup>; Seung-Cheol Lee<sup>2</sup>; <sup>1</sup>Kookmin University; <sup>2</sup>Korea Institute of Science and Technology (KIST)

We performed molecular dynamics simulations to study the deformation behavior of Au nanowires subjected to tensile loading. Au nanowires have FCC structures with a <110>-crystallographic orientation along tensile loading direction and with four {111} lateral surfaces, and the ratio of length to width is nine. In order to investigate the effect of nanowire dimension and different empirical potentials on the deformation behavior, nanowires with the widths ranging from 4nm to 20nm were considered and three different empirical embedded atom method potentials were considered. All nanowires showed the plastic deformation by twin formation and the migration of twin boundaries which induces the formation of <100>-oriented grain along tensile direction and its growth. The yield stress was observed to increase with decreasing nanowire dimension and the change of deformation mechanism was observed between 4nm and 10nm widths. During tensile loading, nanowires with the width larger than 4nm showed the full re-orientation from <110>-oriented wire to <100>-oriented one by the propagation of twin boundaries while nanowire with 4nm width broke down in <100>-oriented region during the migration of twin boundaries. The deformation behavior of nanowires also shows strong dependence on the empirical potentials, which will be also presented in this study.

4:10 PM Tea Break

#### Symposium F: Modelling and Simulation of Microstructures and Processes: Microstructure Modelling and Analysis

Thursday PM August 5, 2010

Room: 5 Location: Cairns Convention Centre

Session Chairs: Hamish Fraser, The Ohio State University; W. T. Kim, Cheongju University

#### 4:30 PM Keynote

**Microstructure–Property–Design Relationships in the Simulation Era**: *Dennis Dimiduk*<sup>1</sup>; Michael Groeber<sup>1</sup>; Andrew Rosenberger<sup>1</sup>; Yoon-Suk Choi<sup>2</sup>; Triplicane Parthasarathy<sup>2</sup>; Chris Woodward<sup>1</sup>; <sup>1</sup>Air Force Research Laboratory; <sup>2</sup>UES, Inc.

Computational methods are affecting a paradigm change for using microstructure-property relationships within materials and structures engineering. This talk examines the emergent development of quantitative computational methods and new experimental tools for microstructure-property-design relationships, primarily for structural alloys. The driving forces motivating changes to the design-materials paradigm are selectively highlighted. Three general phases of design-materials interactions are described as a historical 'serial paradigm,' current 'integrated computational materials engineering' and, future 'virtual materials systems' that are emerging from advances in multiscale materials modeling. The latter two phases bring unique demands for integrating microstructure representations, constitutive descriptions, numerical codes and experimental methods. Importantly, these approaches are forcing a fundamental restructuring of materials data for structural engineering wherein data centers on a hierarchy of model parameterizations and validations, rather than the current application-specific design limits. Examining aspects of current research on microstructure-sensitive design tools for single-crystal turbine blades provides

an accessible glimpse into future microstructure-property tools and their data requirements.

#### 4:50 PM Keynote

Recent Progress in Atomistic Simulations for Structural Materials: Byeong-Joo Lee<sup>1</sup>; <sup>1</sup>Pohang University of Science and Technology

Atomistic simulations such as molecular statics (MS), molecular dynamics (MD) and Monte Carlo (MC) simulations are used to understand the materials behaviour in more fundamental level, e.g. the atomic level. MD simulations have been performed to investigate dynamic behaviour of materials during phase transformations or deformations. However, their applicability has been limited only to pure elements or simple alloy systems. This was because the empirical interatomic potentials necessary for the simulations were available only for a limited range of materials systems. Further, the materials phenomena that could be investigated using the MD simulations were highly limited because of the short simulation time. In the present talk, it will be emphasized that recent progresses in interatomic potential modelling extended the materials systems into a wider range including multicomponent carbide and nitride systems. A MS+MC or MS+MC+MD hybrid simulation that overcame the time limitation of MD simulations and enabled a correct examination of the effect of alloying elements on materials behaviour will be introduced. A grain boundary or interfacial energy database constructed by an atomistic computation and can be implemented on mesoscale simulations for prediction of microstructure evolution based on realistic grain orientations will also be introduced.

#### 5:10 PM Keynote

Microstructural Analysis by Orientation Image-Based Micromechanical Simulation in Steels: *Heung Nam Han*<sup>1</sup>; Se-Jong Kim<sup>1</sup>; Do Hyun Kim<sup>1</sup>; Kyu-Hwan Oh<sup>1</sup>; A.D. Rollett<sup>2</sup>; R. A. Lebensohn<sup>3</sup>; <sup>1</sup>Seoul National University; <sup>2</sup>Carnegie Mellon University; <sup>3</sup>Los Alamos National Laboratory

A fast Fourier transform (FFT) based full-field formulation for viscoplastic polycrystals was applied to the analysis of microstructual evolution including the deformation behavior in steels during various deformation processes. An image measured by electron back-scattering diffraction (EBSD)-based orientation imaging microscopy (OIM) was used as an input microstructure. In order to simulate the more general deformation process with the FFT method, an elasto-plastic finite element method (FEM) was newly formulated by utilizing the viscoplastic solution in a statistical volume element obtained by the FFT method for each finite element. The microstructures including the local misorientations of grains predicted by the FFT-based simulations were compared with the experimental data measured by the EBSD-based OIM with some devices specially designed.

#### 5:30 PM Keynote

Physically-Based Models for Coupled Phenomena in Microstructural Evolutions: Examples of Simultaneous Recrystallisation and Phase Transformations: Yves Brechet<sup>1</sup>; Christopher Hutchinson<sup>2</sup>; Hatem Zurob<sup>3</sup>; <sup>1</sup>Grenoble-INP; <sup>2</sup>Monash University; <sup>3</sup>McMasterUniversity

Physically-based modelling of microstructural evolution presents the advantage over a direct phenomenological approach, that the robustness of the model with respect to the variation of experimental process parameters (temperature, strain, strain-rate) is increased, sometimes at the expense of a decrease in the numerical accuracy of the description. Such approaches have been developed over the years for phase transformations, precipitation and recrystallisation. In many realistic thermo-mechanical processes, the situation is one of coupling between elementary phenomena occurring simultaneously: coupling between recovery and recrystallisation, coupling between deformation and recrystallisation, coupling between precipitation and recrystallisation or coupling between deformation and phase transformations. The elementary phenomena are often strongly interacting, either in a competing manner or in a catalysing way so that a simple superposition of models is bound to fail. These situations make the "physically-based approach" even more necessary if the ultimate goal is efficient microstructural design. The situation becomes rapidly inextricable, unless simple models are proposed. The aim of this paper is to exemplify in these "coupling situations" the value of simple analytical models based upon physical mechanisms.

#### 5:50 PM Keynote

The Effect of Magnetism on Strength and Structural Stability in Metals and Intermetallics: *Mojmir Sob*<sup>1</sup>; Martin Zeleny<sup>2</sup>; Martin Friak<sup>3</sup>; <sup>1</sup>Masaryk University, Faculty of Science; <sup>2</sup>Academy of Sciences of the Czech Republic; <sup>3</sup>Max-Planck-Institute for Iron Research

Magnetic solids constitute a basis of many technologically important materials, however, very little is known how their magnetic behavior changes when a highstrain deformation is applied (as it is, for example, in heavily deformed regions of extended defects, such as grain boundaries, dislocation cores, crack tips etc.). Here we report on the effect of magnetism on ideal tensile strength and structural stability of iron, cobalt and nickel and of selected magnetic intermetallic





lattice parameters and magnetic states of iron, cobalt and nickel overlayers on various (001) and (111) substrates. Importance of magnetic ordering on the theoretical tensile strength of these overlayers is also revealed. Whereas magnetism does not play an important role in stabilization of the ground-state structure of nickel and Ni-intermetallics, the magnetic effects in iron and Fe-intermetallics are vital.

#### 6:10 PM

#### Application of Continuous Displacement Cluster Variation Method to Study Phase Equilibria: *Tetsuo Mohri*<sup>1</sup>; <sup>1</sup>Hokkaido University

Cluster Variation Method (CVM) has been widely recognized as one of the most reliable theoretical tools to study phase equilibria in metallic alloy systems. The conventional CVM, however, does not allow atomic local displacements and, therefore, calculated results often encounter various inconveniences such as the overestimation of transition temperatures. Continuous Displacement Cluster Variation Method (CDCVM) was proposed to circumvent such deficiencies of the conventional CVM. Preliminary studies on an order-disorder phase diagram based on CDCVM indicate that the transition temperature is shifted downward reproducing experimental tendencies. In the present study, CDCVM calculations based on various approximations are attempted and compared.

#### 6:25 PM

Empirical Model of Prior Austenite Grain-Size Prediction in Low Carbon Ti-Nb Microalloyed Steels: *Kijung Park*<sup>1</sup>; Bin Huang<sup>1</sup>; Hoi-Bong Kim<sup>1</sup>; Young-Rae Cho<sup>1</sup>; <sup>1</sup>Pusan National University

Empirical model equation for prior austenite grain size (PAGS) in Ti-Nb microalloyed steels has been investigated. Ti and Nb are the most commonly used alloying elements to induce grain-size refinement in low carbon steels. Prediction of PAGS is an important factor to control the final microstructure and mechanical properties of low carbon steels. In this paper, the proposed empirical model equation is based on Beck's and Sellars' results. The microalloyed steel, with the dimension of 10×5×6 mm, contained 0.104 C, 1.01 Mn, 0.108 Ti, 0.007 Nb: 0.111 C, 0.993 Mn, 0.094 Ti and 0.054 Nb (wt. %). The samples were heated to austenitizing temperature, in a range of 950-1250°C, for 5 min and 30 min in an argon atmosphere and finally quenched immediately in ice water. The quenched samples were sectioned and prepared for metallographic observation. The mean grain-size was measured by using the Image Pro Plus program. The modified  $(8.314 \times (T+273))$ ). The observed grain size was between 10 and 144  $\mu$ m in Ti-Nb microalloyed steels. Quantitative predictions of austenite grain growth by this model fitted well with the experimental grain-growth data.

#### Symposium G: Thin Films and Surface Engineering: Thin Films - Preparation and Properties III

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Room: 8 Location: Cairns Convention Centre

*Session Chairs:* Shinji Muraishi, Tokyo Institute of Technology; Brajendra Mishra, Colorado School of Mines

#### 2:00 PM Keynote

Thursday PM

August 5, 2010

#### A Fabrication of Polyimide Thin Films Containing High Density Oxide or Metallic Nanoparticles: Young-Ho Kim<sup>1</sup>; Dong Joo Choi<sup>1</sup>; <sup>1</sup>Hanyang University

We developed a process to fabricate the polyimide (PI) thin films containing oxide or metallic nanoparticles. First, a thin layer of metal film is deposited onto substrates. Then, the polyamic acid (PAA), a precursor of PI, is spin-coated onto the metal film. The PAA reacts with the metal film. Finally, thermal curing is conducted to imidize PAA to PI after soft baking to evaporate the solvent. During curing, high density nanoparticles form in the PI film. Transmission electron microscopy showed that highly dense nanoparticles such as Cu, Cu<sub>2</sub>O, and ZnO formed in a polyimide film. The size of nanoparticles varied from 2-4 nm to larger than 10 nm. The particle size, density, and distribution depend on the curing temperature, the curing time, and the curing environment. We can fabricate a monolayer of nanoparticles or randomly dispersed nanoparticles in the PI film by controlling the reaction of PAA with a metal film and the curing environment. The properties and applications of polyimide films containing nanoparticles will be discussed.

#### 2:20 PM Keynote

**MgO-Based Magnetic Tunnel Junctions for STT-RAM**: *Kyung-Ho Shin*<sup>1</sup>; <sup>1</sup>Korea Institute of Science and Technology

STT-RAM is expected to be the most promising candidate among the next generation memory thanks to the realization of 'spin-transfer-torque(STT)' phenomena and the success in the MgO barrier fabrication. Two major prerequisites for STT-RAM to be commercialized are (1) the reduction of a critical current density for switching a magnetic bit in STT-RAM to the order of 1 MA/cm<sup>2</sup> and (2) the attainment of magnetoresistance larger than 200% at RA of 10 Oµm<sup>2</sup> or smaller. A magnetic tunnel junction (MTJ) structure with layers having an out-of-plane anisotropy or with a synthetic free layer has turned out to provide potential solutions for the reduction of the switching current density. We have fabricated MgO-based MTJs with various synthetic free layers and those with layers having out-of-plane anisotropy. A high magnetoresistance and large exchange-bias field can be obtained simultaneously by adopting an in-situ annealing during the deposition of multilayers. The paper will report how their tunnel magenetoresistance (TMR) and critical switching current density (Jc) are influenced by the structure and materials of multilayers and the fabrication processes.

#### 2:40 PM

Direct Measurement for Electric Resistance of Ferromagnetic Metal-Nano-Contacts in Oxide Layer: *Masashi Sahashi*<sup>1</sup>; Yoshiyuki Watanabe<sup>1</sup>; Shohei Kawasaki<sup>1</sup>; Kousaku Miyake<sup>1</sup>; <sup>1</sup>Tohoku University

Nano-Contacts Magneto-Resistive(NCMR)devices using NOL(Nano-Oxide Layer) as the spacer layer of Spin-Valve type thin film have been reported to show a new type MR effect by our group. At each of NCs domain wall is geometrically confined, at which electron spins are scattered because both of domain width and length is only 1-2nm in size. In this system Ferromagnetic Metal-Nano-Contacts are fabricated in NOL by self-organized process during Surface Oxidation. So MR performance such as MR ratio and Resistance Area product(RA)is correlated with the purity of each NC. Lower resistivity of NCs composed of a ferromagnetic metal leads higher MR performance as higher MR ratio and lower RA. In general, the resistance of NC is given by the sum of the diffusive resistance and the sharvin resistance. If the resistivity of NC nears to the bulk's value without limit the NC resistance will be almost controlled by only sharvin resistance, where highest DWMR ratio could be obtained. In this study we constructed a current measurement circuit for direct measurement of NC resistance with in-situ conducting AFM and tried to evaluate the resistance of the single NC on the NOL surface to make it possible to evaluate quantitatively the conductive Nano-Contact channels.

#### 2:55 PM

Fabrication of Numerous Ferromagnetic Metal Nanowires UsingElectrodeposition Technique:Takeshi Ohgai<sup>1</sup>; Takafumi Fujimaru<sup>1</sup>; KeizoTakao<sup>1</sup>; Akio Kagawa<sup>1</sup>; <sup>1</sup>Nagasaki University

To synthesize an array of numerical ferromagnetic metal nanowires, iron-group metals such as Ni, Co, Fe and Ni-Fe alloys were electrodeposited from aqueous solution into a nanoporous template with numerical cylindrical nanochannels. The shape of nanowires was precisely transferred from the nanochannel template and the aspect ratio reached to around 150. Magnetic hysteresis loops revealed that Ni, Co and Fe nanowires were spontaneously magnetized to the long axis direction. Coercive force of the nanowires with 6000 nm in length was increased in decreasing the pore-diameter. The coercive force of Co nanowires with 40 nm in diameter has increased up to 1084 Oe.

#### 3:10 PM

#### Growth and Characterization of Ge<sub>1-x</sub>Sn<sub>x</sub> Layers for High Mobility Tensile-Strained Ge Channels of CMOS Devices: *Osamu Nakatsuka*<sup>1</sup>; Yosuke Shimura<sup>1</sup>; Shotaro Takeuchi<sup>1</sup>; Norimasa Tsutsui<sup>1</sup>; Shigeaki Zaima<sup>1</sup>; <sup>1</sup>Nagoya University

We have investigated the growth and characteristics of GeSn and tensilestrained Ge heteroepitaxial layers on Si substrates. Tensile-strained Ge and GeSn layers are attractive for high mobility channel materials of future CMOS devices, because both electron and hole mobilities of tensile-strained Ge and GeSn are expected to be higher than strained-Si. We succeeded the growth of strain-relaxed GeSn layers with a Sn content over 9% by controlling the dislocation structure on Si substrates. The low temperature growth and the large misfit strain between GeSn and Si lead to the high density of defects such as vacancy in GeSn layers. They effectively enhance the propagation of misfit dislocations and the strain relaxation with suppressing the precipitation of Sn atoms from GeSn layers. As a result, a tensile-strained Ge layer can be grown on a GeSn layer and a strainvalue of 0.71% was achieved. We also characterized the Hall mobility of GeSn layers and found that the incorporation of Sn into Ge effectively reduced the concentration of holes related with vacancy defects and improved on the hole mobility. In our presentation, we will also report the crystalline and electrical characteristics of GeSn layers formed on III-V substrates.



#### 3:25 PM

Research on Influence of Ion-Assisted Deposition to the Optical and Mechanic Properties of SiO<sub>2</sub> Film: *Wang Duoshu*<sup>1</sup>; Luo Chongtai<sup>1</sup>; Chen Tao<sup>1</sup>; Xiong Yuqing<sup>1</sup>; Wang Jizhou<sup>1</sup>; <sup>1</sup>Lanzhou Institute of Physics

SiO<sub>2</sub> film has been widely applied in surface engineering area for its good characters of chemical stability, abrasion resistance and wide transparent spectrum range. For example, it can be used in multi-layer optical film applied in ultraviolet, visible and infrared spectrum range, and also used as protection film in order to resist abrasion, deliquescence and erosion. Several methods are usually adopted to prepare SiO<sub>2</sub> film, such as PECVD (Plasma Enhanced Chemical Vapor Deposition), ion-assisted e-beam evaporation (IAEE), sol-gel method and so on. In the paper, ion-assisted e-beam evaporation method was used to deposit SiO<sub>2</sub> film on calcium aluminate glass substrate with good abrasion resistance and high optical transmission in spectrum range of  $3.0 \sim 5.0 \mu m$ , and the technology was studied. The result shown that ion power, substrate temperature and evaporating rate were key factors which affected the abrasion resistance and transmission of SiO<sub>2</sub> film. At the end, we succeeded in preparation of good abrasion resistance and high transmission SiO, film on the special glass substrate.

#### 3:40 PM

Synthesis and Its Characteristic of Silicon Nitride Film Deposited by ECR-PECVD at Low Temperature: *Aimin Wu*<sup>1</sup>; Hongyun Yue<sup>1</sup>; Xueyu Zhang<sup>1</sup>; Fuwen Qin<sup>1</sup>; Tingju Li<sup>1</sup>; Xin Jiang<sup>2</sup>; <sup>1</sup>Key Laboratory of Materials Modification, MOE, Dalian University of Technology; <sup>2</sup>Institute of Materials Engineering, Siegen University

Silicon nitride films were widely used in the semiconductor device industry as passivation layers, diffusion barriers, gate dielectrics and isolation material. Recently, hydrogenated amorphous silicon nitride(a-SiNx:H, SiN for short) film as efficient antireflection coating(ARC) for thin film silicon solar cells has attracted more attention. In this work, the silicon nitride films have been deposited by Electron Cyclotron Resonance(ECR) -PECVD method at low temperature. The optimum deposition parameters of SiN films for photovoltaic application as an efficient AR coating have been investigated. The actual composition of the films will be varied with the deposition conditions, such as gas flow rate ratio( $N_2/SiH_4$ ), substrate temperature, and microwave power. The effect of deposition parameters on the optical performance of SiN films was determined by Ellipsometry. The Si-N and N-H stretching characteristic peaks of SiN films have been observed by FTIR spectroscopy. Results shown that uniform silicon nitride films with low hydrogen content can be deposited at high deposition rate(10.7nm/min), and the refractive index increased with the increasing of substrate temperature and microwave power. The film shows good optical properties (refractive index is 2.0 or so) and satisfied surface quality (average roughness is 1.45nm) when the deposition parameter is 350°C and microwave power is 650W.

#### 3:55 PM

## Huge Gain for Transmission Spectra in 1D Photonic Crystal with Complex Dielectric: Gao Yingjun<sup>1</sup>; <sup>1</sup>Guangxi University

Using the method of optical transfer matrix, the character of photonic forbidden band, gain and absorption of transmission spectra of 1D dual-periodical photonic crystal with complex dielectric layer are studied. The investigation results show that photonic band gap (PBG) of this structure is wide and many transmission resonance peaks appearing in PBG of transmission spectra get a large gain if the imaginary part of dielectric constant is negative. With increasing of the absolute value of the imaginary part, the transmission gain increases firstly and then gets to its apex. The imaginary parts of dielectric constant corresponding to transmission gain apex are different according to wavelength. However, the transmission ratio of resonance peaks is less than 1 if the imaginary part of dielectric constant is positive. These properties might be used to design multi-narrow-channel band filter and optical amplification device synchronously.

#### 4:10 PM Tea Break

#### Symposium G: Thin Films and Surface Engineering: Thin Films - Preparation and Properties IV

Thursday PM	Room: 8
August 5, 2010	Location: Cairns Convention Centre

Session Chairs: Young-Ho Kim, Hanyang University; Kyung-Ho Shin, Korea Institute of Science and Technology

#### 4:30 PM

The Effects of Substrate Temperature on the Composition and IR Transmission Properties of Germanium Carbon Films Deposited by Reactive RF Magnetron Sputtering: *Yangping Li*<sup>1</sup>; Zhengtang Liu<sup>1</sup>; Feng Yan<sup>1</sup>; Wenting Liu<sup>1</sup>; <sup>1</sup>Northwestern Polytechnical University

Germanium Carbon (GeC) films were prepared on ZnS substrates by reactive RF magnetron sputtering of a Ge target in Ar and CH4 mixtures. IR transmittance spectra of the specimens were measured by Fourier transform infrared (FT-IR) spectroscopy, with which the H content in the GeC films was investigated qualitatively. The C and Ge content and atomic binding state were studied by X-ray photoelectron spectroscopy (XPS). The results show that H atoms involved in the films combine mainly with C at low substrate temperatures. IR absorption peaks caused by the C-H bonds decrease with increasing substrate temperature, indicating the reduction of the C-H bonds content in the GeC film. At relatively low substrate temperature, the reduction of the C-H bonds content is mainly caused by decomposition of the adsorbed CHx and CH4, hence the GeC film is high in C content and low in refractive index. At high substrate temperature, the reduction of the C-H bonds content is mainly caused by desorption of the adsorbed CHx and CH4, hence the GeC film is low in C content and high in refractive index. Both C-Ge and C-C bonds exist in the GeC film and O impurity combine mainly with Ge.

#### 4:45 PM

Effect of Ultra-Thin Pt Layer on the Preferred Orientation of AlN Films: *Takashi Harumoto*<sup>1</sup>; Shinji Muraishi<sup>1</sup>; Ji Shi<sup>1</sup>; Yoshio Nakamura<sup>1</sup>; <sup>1</sup>Tokyo Institute of Technology

C-axis orientated aluminum nitride (AIN) films have been prepared on the bulk substrates coated with the ultra-thin Pt layer. AIN films were deposited by reactive direct current (DC) magnetron sputtering in the argon-nitrogen mixed gas. The preferred orientation of AIN films were analyzed using X-ray diffraction (XRD) and transmission electron microscopy (TEM). The AIN film without the ultra-thin Pt layer shows almost no preferred orientation, while the AIN film with the Pt layer exhibits c-axis preferred orientation. Since the Pt layer demonstrates (111) preferred orientation, the c-axis texture of the AIN film may relate to 6-fold rotational symmetry of Pt (111) surface and hexagonal structure of AIN. The stresses of AIN films are measured using XRD and discussed related with the preferred orientation. According to TEM observations and selected area electron diffraction (SAED), the Pt layer has strong effect on the preferred orientation of AIN films and the AIN films have the columnar structure.

#### 5:00 PM

#### The Preparation and Thermal Stability of TiNbON Solar Selective Absorbing Coatings: Yu Liu<sup>1</sup>; Cong Wang<sup>1</sup>; Yafei Xue<sup>1</sup>; <sup>1</sup>Beihang University

Recently, transition metal nitrides/nitro-oxides have been chosen in solar energy spectral selective absorbing coatings for solar photo-thermal conversion. The coatings are required to have high absorbance in the wavelength of 300 nm-2500 nm, low thermal emittance during 2500 nm to 50000 nm and thermal stability at high temperature. In this paper, a series of Cu/TiNbN/TiNbON/SiON multilayer films were prepared by magnetron sputtering. The film layers are, respectively, a high metal volume fraction cermet solar absorptive layer of TiNbN, a low metal volume fraction cermet solar absorptive layer of TiNbON, and an anti-reflection layer of SiON, on Cu substrates, here Cu substrates works as IR reflector. Heat treatments in vacuum were carried out at different temperatures and times so that the thermal stability was evaluated.Coatings exhibited a high absorptance of 0.95 and low emittance of 0.07 at room temperature. No significant change in the absorptance and emittance values was observed until the heat treatment temperature is over 600°C. The variation of the microstructure, diffusion of the component and the influences on the spectral performance were investigated. It is revealed that diffusion between Cu substrates and absorptive layer results in the degradation of performance.



#### 5:15 PM

Magnetoresistive Effect in Co-Ti-O Films with Oxygen-Concentration Modulation: *Ji Shi*<sup>1</sup>; Tomio Ohtsuki<sup>1</sup>; Osamu Sasaki<sup>1</sup>; Yoshio Nakamura<sup>1</sup>; <sup>1</sup>Tokyo Institute of Technology

Metal-insulator granular films have been found to exhibit magnetoresistance. Since the magnetoresistance in such films results from spin-dependent tunneling between the magnetic metal granules, it depends strongly on the property of the insulating matrix. Highly insulating, not incorporating magnetic metal atoms, forming sharp interface with magnetic metal granules are required of the matrix material. In this sense, TiO2 is a promising material for matrix because of the strong bonding between Ti and O. In this work the magnetoresistive effect in Co-Ti-O system has been studied. The Co-Ti-O films were sputter deposited onto fused quartz substrates using a two-facing-target magnetron sputter apparatus. Pure Co and Ti plates were used as targets, and an Ar and O<sub>2</sub> gas mixture was used as the sputtering gas. Oxygen flow rate was controlled during deposition to modulate oxygen concentration in the film. The thickness of the O-rich layer was varied from 1 to 5 nm, and the thickness of O-deficient layer was varied from 4 to 10 nm. Generally, totally fifteen to twenty periods were deposited. It is fournd that for a film with a proper thickness ratio of O-rich to O-deficient layer, it exhibit 10% of magnetoresistance at room temperature.

#### 5:30 PM

Structural and Magnetic Properties of Ferromagnetic Nanowires and Nanotubes: *Naeem Ahmad*<sup>1</sup>; Shamaila Shehzadi<sup>1</sup>; Rehana Sharif<sup>1</sup>; Chen Jun-Yang<sup>1</sup>; Han Xiufeng<sup>1</sup>; <sup>1</sup>Institute of Physics

Magnetic nanowires and nanotubes have received a considerable interest due to their potential applications in the field of patterned recording media, magnetoresisitive nanosensors and racetrack memory. Nanowires and Nanotubes have been fabricated by elctrodeposition due to its cost-effectivenss, simplicity and versatility into anodized aluminium oxide (AAO) and track-etched Polycarbonante membranes. We have investigated an extensive study of single element as Ni, Fe, Co and their alloys as NiFe, CoFe, CoFeB, CoPt, CoPd, CoCrPt Nanowires and Nanotubes with variation in composition, length and diameter. A crossover of easy axis from parallel to perpendicular direction in case of nanowires has been found for a critical length, diameter and temperature. Magnetization reversal mechanism by curling is the dominant process in nanowires and nanotubes. Also a transition of magnetization reversal from curling to coherent is observed for a critical thickness and angle between NT axis and applied field. Above study shows that shape anisotropy is dominant in nanowires and surface effects are dominant in case of nanotubes. Magnetic field annealing causes a remarkable improvement in the magnetic properties due to diffusional pair ordering and stress relief among grains of nanowires. This study will help for applications in ultrahigh density recording media.

#### 5:45 PM

Transport Properties in LSMO/AIO/CoFeB Magnetic Tunnel Junction: Role of Minority-Spin States at the Half-Metal/Insulator Interface: Syed Rizwan<sup>1</sup>; S. M. Guo<sup>2</sup>; Y. Wang<sup>3</sup>; Z. C. Wen<sup>1</sup>; Y. G. Zhao<sup>2</sup>; J. Zou<sup>3</sup>; X. F. Han<sup>1</sup>; <sup>1</sup>State Key Laboratory of Magnetism, Institute of Physics, Chinese Academy of Sciences; <sup>2</sup>Department of Physics, Tsinghua University; <sup>3</sup>Materials Engineering and Center for Microscopy and Microanalysis, The University of Queensland

We have observed the transport properties in magnetic tunnel junction with multilayered structure:  $La_{0.67}Sr_{0.33}MnO_3$  (50)/Al (1)-O/CoFeB (5)/Ta (10)/Ru (10), all thicknesses are measured in nm. The maximum inverse tunneling magnetoresistance (TMR) ratio was found to be -3% at a lower temperature of 5 K and the maximum normal TMR ratio was observed to be 12.4% at a higher temperature of 100 K. The inverse TMR effect decreases with increase in temperature until it reaches a critical temperature of 50 K whereby, it switches to the normal TMR behavior. The appearance of inverse TMR only at low temperatures is attributed to the presence of minority-spin density of states (DOS) of the half-metallic LSMO present at the half-metal/insulator interface. The vanishing of inverse TMR and increase of normal TMR at elevated temperatures is due to reduced contribution from the minority-spin DOS in the transport process. Moreover, a small TMR ratio of only 0.5% is observed at room temperature because of the surface magnons.

#### 6:00 PM

**Employment of PS Template in the Surface Modification and Performance Improvement of Wide Bandgap Semiconductor Film**: *Junying Zhang*<sup>1</sup>; Feng Pan<sup>1</sup>; Hailing Zhu<sup>1</sup>; Chunzhi Li<sup>1</sup>; Liugang Wang<sup>1</sup>; Tianmin Wang<sup>1</sup>; <sup>1</sup>Beihang University

In order to improve the performance of wide bandgap semiconductor such as  $TiO_2$  or ZnO, a variety of micro-grids were deposited on the surface of the semiconductor film using PS template in our research group. Metal micro-grid such as Ag, Au and Cu were endowed high electric conductivity, enhancing the separation of photo-generated electrons and holes, and thus improved

the photocatalytic activity of the semiconductor film. Besides, the excellent transparency of the metal micro-gird made it a good candidate as front electrode for sandwich type photodetector. Another semiconductor micro-grid, e.g.  $Cu_2O$ ,  $Bi_2O_3$  and  $WO_3$ , was prepared on the surface of  $TiO_2$  or ZnO film. Since the top layer has different energy band structure with the bottom layer, the absorption spectra were widened and the photo-generated charge carrier utilization was increased. Furthermore, the two semiconductors have similar opportunity to be exposed to the adsorbates. As a result, the composited semiconductor film. Using PS template, heterojunction with special interface can be manufactured. If the two semiconductor layers exhibit curved interface, the interface area will be enlarged, promising more efficient charge carrier transfer.

#### 6:15 PM

## Interface Engineered Highly Epitaxial Thin Films with Anomalous Physical Phenomena: Chonglin Chen<sup>1</sup>; <sup>1</sup>University of Texas at San Antonio

Interface engineered material has attracted more and more attention in the multifunctional materials research and active device fabrication. It plays a key role to control the physical properties of advanced nanomaterials and results in the discovery of various new physical phenomena with excellent opportunity for developing new metamaterials for active devices and engineered nanosystems. We have focused on the systematical studies on the formations and the characterizations of various highly epitaxial oxide thin films and multilayered layered structures to understand the nature of interface induced anomalous physical phenomena. In this talk, several new results will be discussed such as our recent achievements on a giant dielectric tunability of 80% from highly epitaxial ferroelectric Mn:(Ba,Sr)TiO<sub>3</sub> thin films from the interface controlled nano domain structures, a new record of magnetoresistance ratio of 1010 (four order higher than the previous record) from the artificial interface domain structured (La,Ca)MnO, epitaxial thin films: an anomalous domain locked ferroelectric phenomena from the multilayered BaTiO<sub>2</sub>/SrTiO<sub>2</sub> superlattices, and the strong anisotropic phenomena in highly epitaxial (Pb,Sr)TiO, thin films, etc. On the other hand, a series of models were developed to understand these interface phenomena. Details will be presented in the talk.

#### Symposium H: Advanced Ceramics: Functional Ceramics

Thursday PM August 5, 2010 Room: 6 Location: Cairns Convention Centre

Session Chairs: Hideki Kita, National Institute of Advanced Industrial Science and Technology(AIST); George Franks, University of Melbourne

#### 2:00 PM Keynote

### **Control of Defect Energy Level in Ta<sub>2</sub>O<sub>5</sub> and Defective Modification of TiO<sub>2</sub> Nano-Particles for Their Application to Photo-Catalysis**: *Sang-Yeup Park*<sup>1</sup>; <sup>1</sup>Gangneung National University

Defect energy levels in Ta<sub>2</sub>O<sub>5</sub> and nitrogen-doped Ta<sub>2</sub>O<sub>5</sub> have been investigated experimentally. Thermal treatment of Ta<sub>2</sub>O<sub>5</sub> in reducing atmosphere increases photoluminescence (PL) intensity of the deep energy levels in band gap as compared to the shallow ones. Nitrogen doping creates no new defect energy levels to even deeper states. The doped nitrogen is interpreted to fill the oxygen vacancy at "in-plant" lattice sites when it is doped to TiO, octahedron. A novel process has been devised to prepare a series of TiO, nano-particles that possess different degree of O/Ti ratios. The prepared series of nano-particles have been characterized using X-ray diffraction, X-tray photoelectron spectroscopy, ultraviolet-visible absorption spectroscopy, transmission electron microscopy, and Fourier-transformed infrared spectroscopy. By controlling the O/Ti ratio via appropriate processing variables, optical absorption edge of the defective TiO<sub>2</sub> nano-particles was extended significantly into the visible light regime. This observation indicates that the nano-particles can successfully absorb visible light from the sun as compared to normal TiO<sub>2</sub> particles which can absorb only ultraviolet portion of the sun light. The capability of the prepared defective TiO, nano-particles in photo-catalysis of organic matter is first presented herein, before applying the nano-particles to photolysis of water.

#### 2:20 PM Keynote

Relaxor Ferroelectricity and Electrostrictive Behaviour of KNN-ST Ceramics: *Huiqing Fan*<sup>1</sup>; <sup>1</sup>Northwestern Polytechnical University

The new electrostrictive ceramics have been produced from the (1-x)  $K_{0.5}Na_{0.5}NbO_{3.x}SrTiO_3$  (KNN-STO, x=0.1-0.50) system by conventional mixedoxide methods. Sintered temperature raised (1190°C-1300°C) with increasing



SrTiO<sub>3</sub> content(x=0.1-0.50) in this system, moreover, extremely narrow sintered temperature range to each composite were found. The x-ray diffraction patterns showed that KNN ceramics with the SrTiO<sub>3</sub> exhibited a single perovskite structure with the co-existence of the orthorhombic and cubic phase. With SrTiO<sub>3</sub> increasing, KNN-STO crystal structure trend to the pseduo-cubic structure, no extra phase appeared. The elastic strain of the KNN-STO ceramics induced by applied electric fields have been investigated, the strain response is similarly as in the classical PMN (lead magnesium niobate), but lower (order of the 10<sup>-5</sup>) than PMN (order of the 10<sup>-3</sup>). The relaxor ferroelectric property of the strain are influenced with the SrTiO<sub>3</sub> content changed were also discussed in this paper. Electrostrictive coefficients are shown to be constant over a broad temperature range and similar to the dielectric properties, independent of electric-field cycles. Furthermore, this system show translucent, high dielectric constant, thus suggests possible applications in electric-optic devices, electromechanical transducer applications.

#### 2:40 PM

#### **Subtle Interplay between Hydrogen and Magnetism in Co Doped ZnO:** *Yuebin Zhang*<sup>1</sup>; M.H.N. Assadi<sup>1</sup>; S. Li<sup>1</sup>; <sup>1</sup>The University of New South Wales

Distribution of Co ions and its effect on magnetic properties of Co doped ZnO (ZnO:Co) in the presence of hydrogen, either interstitial ( $H_1$ ) or substitutional ( $H_0$ ), have been investigated using first-principles density functional calculations. The study provides a fundamental theoretical understanding on the correlation between magnetism and the distribution of magnetic ions and the native point defect in the semiconducting host. Results show that Co ions have a strong tendency toward aggregation in the presence of hydrogen, which mainly contributes to the room temperature ferromagnetism observed experimentally in ZnO:Co. Furthermore, in ZnO:Co the formation of  $H_0$  with four-fold hydrogenic bonds is favored over  $H_1$  by 0.4 eV.

#### 2:55 PM

**Core/Shell Nanophosphors for LED Lighting**: Jinkyu Han<sup>1</sup>; Gustavo Hirata<sup>2</sup>; Jan Talbot<sup>1</sup>; *Joanna McKittrick*<sup>1</sup>; <sup>1</sup>UC San Diego; <sup>2</sup>Center for Nanoscience and Nanotechnology

Luminescent nanopowders (nanophosphors) have generated widespread attention due to the unusual luminescence properties and the applicability for a variety of applications. Wide band-gap nanophosphors have poor quantum efficiencies, due to surface defects. One way to mitigate this effect is to coat an inert shell on the nanophosphors. We report on the luminescence properties of  $Y_2O_3$ :Eu<sub>3+</sub> and  $Y_2SiO_5$ :Tb,Ce core nanoparticles synthesized by a Pechini-type sol-gel process, and SiO<sub>2</sub> shells deposited by the Stöber process. The morphology and particle size of the core/shell particles studied by scanning electron microscopy, show that core/shell particles to be well-dispersed and have a highly uniform shape. The photoluminescence intensity of core/shell particles increases with thinner SiO<sub>2</sub> shells and the luminescent intensity of all shell thicknesses was enhanced over the bare phosphor particles.

#### 3:10 PM

Structure and Diffused Ferroelectric Properties in Ba(Ti<sub>0.8</sub>Sn<sub>0.2</sub>)O<sub>3</sub> Ceramics: Dong-Yun Gui<sup>1</sup>; Hua Hao<sup>1</sup>; Yue Sun<sup>1</sup>; Ming-He Cao<sup>1</sup>; Zhi-Yong Yu<sup>1</sup>; Li-Hui Xue<sup>1</sup>; *Han-xing Liu*<sup>1</sup>; <sup>1</sup>Wuhan University of Technology

Temperature-dependent Raman scattering, dielectric properties, and Impedance spectra have been carried out on Ba(Ti<sub>0.8</sub>Sn<sub>0.2</sub>)O<sub>3</sub> ceramic sample. The number of modes observed for the compositions of cubic symmetry is more than that predicted by group theory, and the A1(TO1) and E(TO2) modes become Raman active below the critical temperature (around 300K) in the lower frequency region. This gives evidence that there are some deviations of the local structure from the average global symmetry. Moreover, the characteristic of A1(TO3) mode is confirmed be associated with the diffused dielectric properties. Impedance/ modulus analyses indicate that a third resistance-capacitance (RC) response is present in the sample, in addition to the grain and grain-boundary RC elements above 573K. Thus, it probably inferred that the existence of inter- and/or intragranular surface layers associate with the abnormal dielectric properties, which is different from the normal ferroelectrics. Variation of bulk ac conductivity as a function of temperature shows that Ba(Ti<sub>0.8</sub>Sn<sub>0.2</sub>)O<sub>3</sub> follows the classical Arrhenius relation. The results show that the activation energy of grains and grain boundaries are 0.61 and ~1.08 eV, respectively.

#### 3:25 PM Keynote

Impact Indentation Properties and Interface Strength of Ceramic Coatings: *Yiwang Bao*<sup>1</sup>; <sup>1</sup>China Building Materials Academy

Spherical impact indentation method with different impact angles  $(90^\circ, 60^\circ, 45^\circ, and 30^\circ)$  was used to understand the impact resistance and interfacial adhesion of zirconia (ZrO2) ceramic coating on steel and aluminum substrates, respectively. A linear rail with an adjustable angle was used to guide the slipping impact indenter to impact the specimen. The impact load and the impulse were recorded by the test system automatically. It is shown that impulse increased with

increasing impact load. Based on the morphology of the impacted specimen, it is found that the peak impact load decreases but the contact indentation becomes longer with decreasing impact angle. Under the same impact load, the smaller the impact angle, the higher the impulse. The experimental results indicate that the  $ZrO_2$  coating on steel substrate has higher impact resistance than that on the aluminum substrate. The fact that the impact with smaller angle results in greater damage indicates that the combination of compressive stress and shear stress may cause serious coating failure. It is concluded that the interfacial strength for  $ZrO_2$  coating on steel substrate is higher that on aluminum substrate. This method is feasible for evaluating the interfacial strength of ceramic coatings.

#### 3:45 PM

High Performance of Sub-Micro-Layered Ti<sub>3</sub>C<sub>2</sub>/(Cu-Al) Cermets Prepared by In-Situ Hot-Extruding Method: *Zhenying Huang*<sup>1</sup>; Hongxiang Zhai<sup>1</sup>; Mengqi Li<sup>1</sup>; Wenjuan Wang<sup>1</sup>; <sup>1</sup>Beijing Jiaotong University

A series of new sub-micro-layered Ti<sub>3</sub>C<sub>2</sub>-Cu(Al) cermets were prepared by in-situ hot-extruding a mixture of Ti,AlC, and Cu powders, and some properties of these materials were tested. The Al of Ti<sub>3</sub>AlC<sub>2</sub> is dissolved and diffused into the liquid Cu at the high temperature of 1150°C, forming the Ti<sub>3</sub>C<sub>2</sub>-Cu(Al) cermet consisting of Ti<sub>3</sub>C<sub>2</sub> phase and Cu(Al) alloy phase. These cermets have quite high fracture strength and electric conductivity, due to the strong combination between Ti<sub>3</sub>C<sub>2</sub> and Cu(Al), and a special network microstructure formed by the Cu(Al) phase surrounding the sub-micro-sheet layered Ti<sub>3</sub>C<sub>2</sub> phase.The in-situ hotextruding after pressless sintering can effectively eliminate pores contained in Cu(Al) phase, and accelerate the diffusing of Cu towards the interlayer between Ti<sub>3</sub>C, layers, so the fracture strength and electric conductivity are increased. For the Cu content of 50% in volume, the strength is 915.8 MPa and the electric conductivity is about  $4.0/\mu\Omega$ .m in case of the pressless sintering; the strength and the conductivity can be further heightened over 1200 MPa and  $4.35/\mu\Omega m$ , respectively, in case of the hot-extruding. With increasing the content of ceramic phase, the strength of the cermets can be further heightened while the ductility is reduced.

#### 4:00 PM

**Early Stages of Phase Evolution in Aged Thermal Barrier Coatings**: *Jessica Krogstad*<sup>1</sup>; Stephan Krämer<sup>1</sup>; Don Lipkin<sup>2</sup>; Carlos Levi<sup>1</sup>; <sup>1</sup>University of California, Santa Barbara; <sup>2</sup>General Electric Global Research Center

The temperature capability of state-of-the-art thermal barrier coatings is intimately coupled to the rate at which the "non-transformable" t'-phase evolves into a depleted tetragonal phase predisposed to the monoclinic phase transformation upon cooling. Recent work has shown that the t'-phase decomposes rapidly into a modulated microstructure consisting of coherent yttria-rich and yttria-lean lamellae. These lamellae have compositions close to those expected from equilibrium considerations for the cubic and tetragonal phases. The interleaving cubic lamellae appear to constrain the yttria-lean tetragonal lamellae from transforming to monoclinic. Coherency is expected to influence the rate of coarsening and hence the onset of tetragonal-to-monoclinic transformation, and can in turn be influenced by the chemical composition of the oxide through its effects on the lattice parameters of the respective phases.

#### 4:15 PM Tea Break

#### Symposium H: Advanced Ceramics: Properties of Ceramics

Room: 6 Location: Cairns Convention Centre

Session Chairs: Joanna McKittrick, University of California, San Diego; Junichi Hojo, Kyushu University

#### 4:30 PM Keynote

Thursday PM

August 5, 2010

## Al<sub>2</sub>TiO<sub>5</sub> Ceramics for New Diesel Particulate Filter and High Temperature Applications: *Ik Kim*<sup>1</sup>; <sup>1</sup>Hanseo University

The use of  $Al_2 TiO_5$  ceramics in diesel engine exhaust systems has been limited by the problem of thermal stress and thermal instability caused by the anisotropic thermal expansion between different materials. In order to avoid this problem, one of the choices is to develop low or zero-level thermal expansion materials that can be used, for example in diesel particulate filters (DPF), in which the original dimensions of the material are maintained, without being affected by thermal shock at high temperatures. For such applications,  $Al_2 TiO_5$  ceramic particulate filters must also have chemical inertness, thermal durability, high filtration efficiency, low pressure drop-, and adequate strength. In particular, DPF



ideally combine low thermal expansion, low pressure drop for engine efficiency, high filtration efficiency, high strength, and low production cost. This study examined the publications and patents for thermal stability of  $Al_2 TiO_5$  ceramics with different inorganic materials having a low thermal expansion and high thermal shock resistance, suitable porosity and a narrow pore size distribution suitable for diesel exhaust filtration applications.

#### 4:50 PM

Ferroelectric and Photocatalytical Properties of Ta-Based and Nb-Based Oxide Ceramics and Powders from Environmentally Friendly Water-Soluble Tantalum and Niobium Precursors: *Ai-Dong Li*<sup>1</sup>; Hai-Fa Zhai<sup>1</sup>; Ji-Zhou Kong<sup>1</sup>; Di Wu<sup>1</sup>; <sup>1</sup>Nanjing University

Due to the great potentials of tantalite and niobate materials in ferroelectric and photocatalytic applications, development of proper tantalum or niobium precursors is urgently need. In wet chemical synthesis of Ta-based or Nbbased oxide, alkoxides of tantalum and niobium are most frequently used as starting sources. However, certain inherent problems are associated with Ta or Nb alkoxides. Recently a simple polymerizable complex (PC) route, has been developed to solve these problems. In this work, a simple novel route to synthesize environmentally friendly aqueous tantalum and niobium precursors has been developed using cheap and stable Nb<sub>2</sub>O<sub>5</sub> or Ta<sub>2</sub>O<sub>5</sub> as starting source. Using home-made Ta and Nb precursors, several photocatalytic nanopowders such as Ta-doped ZnO and BiNbO4, and ferroelectric ceramics such as 0.65Pb(Mg<sub>1/3</sub>Nb<sub>2/3</sub>)O<sub>3</sub>-0.35PbTiO<sub>3</sub> (PMN-PT), have been prepared by a modified PC method. These powders have pure crystalline phases with uniform sizes of 20-100nm and larger specific surface area in the range of 9-30 m<sub>2</sub>/g, compared to conventional solid phase reaction. The photocatalytical and ferroelectric properties indicate that this is an attractive and flexible approach for fabrication of tantalate and nibonate functional materials.

#### 5:05 PM

**Optical Properties of Transparent MgO-Doped Alumina Fabricated by Spark Plasma Sintering**: *Byung-Nam Kim*<sup>1</sup>; Keijiro Hiraga<sup>1</sup>; Koji Morita<sup>1</sup>; Hidehiro Yoshida<sup>1</sup>; <sup>1</sup>National Institute for Materials Science

We fabricated fully-dense transparent MgO-doped alumina by spark plasma sintering at 1100-1600°C and at a pressure of 80 MPa. Fast densification occurred for pure and MgO-doped alumina powders without any pre-treatment. The MgO doping reduced the final grain size and enhanced the transparency. For the 0.03% MgO-doped alumina sintered at 1300°C for 20 min, the average grain size is 0.7  $\mu$ m and the total forward transmission is 70% for the red wavelength (640nm). In a range of 1150-1350°C, the total forward transmission increased with increasing sintering temperature. The transparency is sensitive to the porosity, but less sensitive to the grain size; the increasing sintering time yields higher transparency. We also examined the hardness, strength and other mechanical properties of the alumina.

#### 5:20 PM

Optimization of Dielectric Properties of Glass Added Ba<sub>x</sub>Sr<sub>1-x</sub>TiO<sub>3</sub> Ceramics for Pulsed Power Applications: *Qingmeng Zhang*<sup>1</sup>; Lei Wang<sup>1</sup>; Jun Luo<sup>1</sup>; Qun Tang<sup>1</sup>; Jun Du<sup>1</sup>; <sup>1</sup>General Research Institute for Nonferrous Metals

Fri. AM

The development of solid dielectrics with high dielectric constant and high breakdown strength (Eb) has been one of the key breakthroughs in realization of solid state pulse forming line (SSPFL) for compact pulsed power applications. Currently, ferroelectric ceramics have been widely investigated as potential candidates, since they generally behave in high dielectric constant and relatively low dielectric loss. However, their relatively low Eb would limit their energy density. Thus the improvement of Eb becomes a key for their future applications in SSPFL. In this work, Ba<sub>x</sub>Sr<sub>1-x</sub>TiO<sub>3</sub> (0.2=x=0.5) ceramics with 10 vol% BaO-SiO<sub>2</sub>-B<sub>2</sub>O<sub>2</sub> glass addition were prepared. The microstructure and dielectric properties were investigated by XRD, SEM, ferroelectric tester and impedance analyzer. The results show that with the decrease of Ba content, the pulse breakdown strength of the samples is increased, and the dielectric constant and dielectric loss is decreased gradually. In the frequency from 10 kHz to 20 MHz. the dielectric constant of all the samples shows good frequency stability. For x=0.2, the samples exist high breakdown strength of 37.7 kV/mm, moderate dielectric constant of 311 and low dielectric loss of 5×10<sup>-4</sup>, which is a promising dielectric for SSPFL.

#### 5:35 PM Keynote

Environmental Impact Evaluation and Rationalization of Ceramics Process on the Basis of Exergy Analysis: *Hideki Kita*<sup>1</sup>; Hideki Hyuga<sup>1</sup>; Takaaki Nagaoka<sup>1</sup>; Naoki Kondo<sup>1</sup>; <sup>1</sup>National Institute of Advanced Industrial Science and Technology(AIST)

Although structural ceramics parts have high potential for the contribution to sustainable manufacturing, these are not in widespread use because the costs are very expensive. However, in addition to the economic costs, it is also important to consider the value of reducing the impact of a product in terms of natural resource consumption and environmental impacts throughout its life cycle. In order to achieve this, we need a method for quantitative evaluation of natural resource consumption similar to measurements of economic cost. Based on these considerations, we carried out an exergy analysis for ceramics in order to clarify the value of environmental impact reduction throughout their life cycles. In this paper, we estimated the environmental impact in the production and usage stages when ceramic heat-tubes were used, Additionally, the relationship between the input exergy in sintering step and the sintering temperature, holding temperature as well as the total time is analyzed by regression method, which gives a guideline for the rationalization of manufacturing process.

#### 5:55 PM

### Effect of Ti Content on Microstructure and Mechanical Properties of $Si_3N_4$ $Si_3N_4$ Joints Brazed with Ag-Cu-Ti+Mo Composite Filler: *Zhang Jie*<sup>1</sup>; He Yanming<sup>1</sup>; <sup>1</sup>Harbin Institute of Technology

Mo particles have been introduced into Ag-Cu-Ti brazing alloy for the joining of Si<sub>3</sub>N<sub>4</sub> ceramic. Effects of Ti content on microstructure and mechanical properties of the joints were investigated. The results present that a compact reaction layer which is composed of TiN and Ti<sub>5</sub>Si<sub>3</sub> was formed at Si<sub>3</sub>N<sub>4</sub>/brazing alloy interface. The central part of the joint was composed of Ag based solid solution, Cu based solid solution and Mo particles together with some Cu-Ti intermetallic compounds. By increasing Ti content in the composite filler, both the thickness of reaction layer at the Si<sub>3</sub>N<sub>4</sub>/brazing alloy interface and the amount of Cu-Ti intermetallic compounds in the brazing seam increased, which is beneficial for the bending strength of the joints. However, the reaction between Ti and Si<sub>3</sub>N<sub>4</sub> ceramic proceeded more excessively. Simultaneously, more Cu-Ti intermetallic compounds were precipitated while elevating the Ti content to 6wt.%, leading to deterioration of the bonding strength. The maximum bending strength (429.4MPa) was obtained at 900°C for 5 minutes when the Ti content is 4wt.% in the composite filler.

#### 6:10 PM

Microstructural Evolution and Electrical Conductivity of Ca-Doped LaCr<sub>2</sub>O<sub>3</sub> SOFC Interconnect in Atmospheric Plasma Spraying: *Kyeong-Ho Baik*<sup>1</sup>; Tae-Hwan Jeong<sup>1</sup>; <sup>1</sup>Chungnam National University

Ca-doped LaCr<sub>2</sub>O<sub>3</sub> was deposited onto a NiO-YSZ anode by atmospheric plasma spraying for SOFC interconnector. The as-sprayed Ca-doped LaCr<sub>2</sub>O<sub>3</sub> interconnector layer had defects such as microcracks and intersplat pores that were formed by a large thermal contraction of splat during cooling, and subsequently reduced the gas tightness. Controlling feedstock powder characteristics allows the minimization of micro-defects in the sprayed coating, which led to better electrical performance and gas tightness. Subsequent heat treatment at elevated temperatures effectively eliminated the intersplat pores and transformed from layered splat structure to equiaxed grain structure. The electrical conductivity of Ca-doped LaCr<sub>2</sub>O<sub>3</sub> interconnect coating reached up to ~50 S/cm in air environment and ~2.0 S/cm in H, reduction environment at 800°C.

#### 6:25 PM

Crack Propagation and Fracture Induced by Rapid Cooling of Fabrication Ceramic Prosthesis: *Zhongpu Zhang*<sup>1</sup>; Shiwei Zhou<sup>1</sup>; Qing Li<sup>1</sup>; Wei Li<sup>1</sup>; Michael Swain<sup>2</sup>; <sup>1</sup>School of Aerospace, Mechanical and Mechatronic Engineering, University of Sydney; <sup>2</sup>Faculty of Dentistry, University of Sydney

Fabrication of multilayered ceramics signifies an important topic of research in many advanced applications. In prosthetic dentistry, more patients are tending to receive all-ceramic restorations for their outstanding aesthetics and excellent biocompatibility. However, there have been a substantial number of reports showing that some zirconia- or alumina-based ceramic devices exhibited considerable failure rates or defects during fabrication. The quality and longlasting success of dental restoration has been the foremost concern of dentists and patients. This study aims at characterising crack growth and fracture by adopting extended finite element method (X-FEM). The numerical model is generated in evaluating the bi-layered dental ceramics first under a controlled cooling rate from high temperature to room temperature. The results are verified through comparing with experimental tests. It is found that results are more sensitive to the layer thickness, cooling rates and mismatches in temperature-dependent material properties. In addition, dental crown model is created to simulate the crack initiation and propagation path under rapid cooling. The numerical analyses provide us with new understanding of multilayered ceramic fracture behaviours. Such findings show significance to design of ceramic components undergone functional and fabrication thermal loading.



#### Symposium I: Biomaterials, Smart Materials and Structures: Other Functional Materials

Thursday PM	Room: 3
August 5, 2010	Location: Cairns Convention Centre

Session Chair: Dong Yang Wu, The Boeing Company

#### 2:00 PM

**Engineered Microcavities for Device Manufacture**: *Erich Kisi*<sup>1</sup>; Jennifer Forrester<sup>2</sup>; <sup>1</sup>The University of Newcastle; <sup>2</sup>University of Florida

A new method for making engineered internal microcavities 100-500 microns in diameter and up to several cm long in alumina ceramics is presented. Based upon the Kirkendall effect, the method combines mechanically activated powders and refractory metal templates and has the potential to make cavities of virtually any shape. Depending upon the sintering temperature and time, the microstructure within the diffusion zone surrounding the cavities ranges from a complex 5-zone assembly to a single ternary phase. Cavities can be tailored to have smooth or highly textured internal walls which may be useful for micro-fluidic mixing. Cavities can also be combined with inert materials such as Pt foils which may prove useful as diaphragms or one-way valves in the manufacture of microdevices. This process and the use of alumina as the matrix material gives much greater flexibility of microcavity shape, and requires no subsequent assembly and sealing; all advantageous compared with cavities built up using surface deposition on silicon or similar substrates. Results will be presented concerning the shape, size, orientation, surface roughness and surrounding microstructure of selected cavities thought to be potentially useful for the manufacture of micro-devices. Current limitations of the process will also be discussed.

#### 2:15 PM

#### Effect of Grain Boundary Segregated Dopant on Phase Stability in Tetragonal Zirconia Polycrystal: *Yorinobu Takigawa*<sup>1</sup>; Takahisa Yamamoto<sup>2</sup>; Kenji Higashi<sup>1</sup>; <sup>1</sup>Osaka Prefecture University; <sup>2</sup>The University of Tokyo

The effect of grain boundary segregated dopant on phase stability of tetragonal zirconia polycrystal (TZP) is examined by accelerated exposure tests ageing in hot water. The materials used in this study are 3 mol%Y $_{2}O_{3}$  stabilized TZP (3Y-TZP) and some oxide-doped 3Y-TZP. Accelerated exposure tests in an autoclave reveal that the tetragonal phase stability of 3Y-TZP in water is highly affected by the kind of grain boundary segregated dopant and the grain size. When the grain size of TZP is about 0.55µm, the change in phase transformation behavior with dopant is explained from the change in grain boundary diffusivity of hydroxyl ion. Grain boundary diffusion of hydroxyl ion must be blocked by the presence of some segregated ion which reduces the effective area of grain boundary diffusion. On the other hand, when the grain size is about 0.35µm, the phase transformation behavior seems to be controlled by the grain boundary stress. Increased grain boundary stress by the segregation of some dopant must retard the phase transformation of 3Y-TZP.

#### 2:30 PM

## Lattice Contraction and Resistivity Jump in Antiperovskite Mn<sub>3</sub>XN Compounds: Cong Wang<sup>1</sup>; <sup>1</sup>Beihang University

Some intermetallic compounds with antiperovskite structure exhibit negative thermal expansion (NTE) behavior and abnormal electronic transport in certain temperature range when they have a magnetic transition, As in perovskite oxides ABO3, the antiperovskite nitrides Mn3XN have displayed many interesting physical properties, which shows that the charge, spin and lattice are closely correlated. The NTE behaviors and abnormal electronic transport properties were induced by possible orbital and charge ordering during a magnetic transition. The Mn<sub>3</sub>XN (X= Ni, Zn, Sn, etc.) compounds and their solid solutions with antiperovskite structure were prepared by solid state reaction. Their magnetic transition and simultaneous abnormal thermal expansion behaviors were studied by SQUID, PPMS, VSM, variable temperature X-ray and neutron diffraction. The near zero dp/dT and large isotropic lattice contraction in Mn,NiN compound were first reported. On the other hand, A phase separation with different magnetic structures and lattice parameters was observed in Mn<sub>3</sub>ZnN. The mechanism has been discussed, combined with the different element doping effects. In summary, the materials with antiperovskite structure display abundant and interesting physical phenomena and mechanisms, as like perovskite oxides, which are worthy of deep exploration

#### 2:45 PM

Heat Treatment of ASTM F75 Co-Cr-Mo-C-Si-Mn Alloys: *Alfi Rano*<sup>1</sup>; Shingo Mineta<sup>1</sup>; Shigenobu Namba<sup>2</sup>; Takashi Yoneda<sup>3</sup>; Kyosuke Ueda<sup>1</sup>; Takayuki Narushima<sup>1</sup>; <sup>1</sup>Tohoku University; <sup>2</sup>Kobe Steel, Ltd.; <sup>3</sup>Yoneda Advance Casting Co., Ltd.

The carbide precipitation behavior of biomedical ASTM F75 Co-Cr-Mo-C-Si-Mn alloys were investigated during solution treatment and aging. In this study, Si and Mn were selected as the alloying elements listed in ASTM F75. The chemical compositions of alloys were Co-28Cr-6Mo-0.25C-1Si, Co-28Cr-6Mo-0.25C-1Mn and Co-28Cr-6Mo-0.25C-1Si-1Mn (mass%). The alloys were solution treated and aged at temperatures of 1448-1548 K and 873-1473 K, respectively, for a holding time of 1.8-43.2 ks. The precipitates in the as-cast alloys were M23C6 type carbide,  $\eta$ -phase (M<sub>6</sub>C-M<sub>12</sub>C-type carbide) and  $\pi$ -phase (M<sub>7</sub>T<sub>3</sub>X type carbide with a B-manganese structure). Addition of the alloving elements markedly affected the dissolution behavior of precipitates during the solution treatment. The alloy with Si addition required a longer solution treatment time for complete precipitate dissolution as compared to the alloy with Mn addition. The phase and morphology of the precipitates observed during the solution treatment depended on the heat-treatment temperature and holding time and the alloy composition. The addition of Mn also decreased the precipitation area in the time-temperatureprecipitation (TTP) diagram as compared to Si addition.

#### 3:00 PM

Effects of Phase Constitution of Zr-Mo Alloys on Their Magnetic Susceptibilities to Prevent Artifacts in MRI: *Naoyuki Nomura*<sup>1</sup>; Suyalatu<sup>1</sup>; Ryota Kondo<sup>1</sup>; Yusuke Tsutsumi<sup>1</sup>; Hisashi Doi<sup>1</sup>; Takao Hanawa<sup>1</sup>; <sup>1</sup>Tokyo Medical and Dental University

The magnetic susceptibilities and microstructures of Zr-Mo alloys were investigated to develop a Zr alloy with a low magnetic susceptibility for magnetic resonance imaging (MRI). The magnetic susceptibility was measured with a magnetic susceptibility balance, and the microstructure was evaluated with an X-ray diffractometer (XRD), an optical microscope (OM), and a transmission electron microscope (TEM). Zr-Mo alloys as-cast showed a minimum value of magnetic susceptibility at 3 mass% Mo, and the value abruptly increased up to 10 mass% Mo, followed by a gradual increase with the increase of the Mo content. XRD, OM, and TEM revealed that the minimum value of the susceptibility was closely related to the appearance of the athermal omega phase in the beta phase. Since the magnetic susceptibility of Zr-(0.5-1)Mo consisting of an alpha phase was higher than that of Zr-3Mo consisting of the omega and beta phases, that of the omega phase was lower than that of the alpha and beta phases. The magnetic susceptibilities of the Zr alloys as-cast were almost one-third that of Ti-6Al-4V, which is commonly used for medical implant devices. The effect of heat treatment on the magnetic susceptibility was also discussed.

#### 3:15 PM

#### On the Effective Mechanical Properties of Fluid-Saturated Composites: A Homogenization Approach: *Chunhui Yang*<sup>1</sup>; Lianhua Ma<sup>1</sup>; Bernard Rolfe<sup>1</sup>; Qingsheng Yang<sup>2</sup>; <sup>1</sup>Deakin University; <sup>2</sup>Beijing University of Technology

The composites containing saturated fluid are widely distributed in nature, such as saturated rocks, colloidal materials and biological cells. In this study, to determine effective mechanical properties of fluid-saturated composites, a micromechanical model and a multi-scale homogenization-based model are both developed. In the micromechanical model, the internal fluid pressure is generated by applying eigenstrains in the domain occupied by the fluid inclusion, and the explicit expressions of effective bulk modulus and shear modulus are then obtained using the Eshelby-Mori-Tanaka equivalent inclusion method. On the other hand, the multi-scale homogenization theory is employed to develop the homogenization-based model, which is numerically solved to determine effective properties at the small scale as a unit cell level with periodic boundary conditions. The numerical results are compared with those from analytic formula, and a very good agreement is achieved. Applying two proposed approaches, the effects of internal pressure of hydrostatic fluid on effective properties are further investigated. The results show the overall effective bulk modulus depends on the volume fraction and internal pressure of the fluid inclusion. The internal pressure can strengthen the overall effective bulk modulus, but it has no contribution to the overall effective shear modulus of fluid-saturated composites.

#### 3:30 PM Tea Break



#### Symposium I: Biomaterials, Smart Materials and Structures: Implants and Self-Healing Materials

5
Cairns Convention Centre

Session Chair: Marcus Zipper, CSIRO

#### 4:30 PM Keynote

Latest Developments and Applications of Self-Healing Polymeric Materials: Dong Yang Wu<sup>1</sup>; <sup>1</sup>The Boeing Company

Polymeric materials are used in a broad range of applications i.e. transport vehicles (cars, aircrafts, ships, and spacecrafts), sporting goods, civil engineering, and electronics, etc. However, these materials are susceptible to damage induced by mechanical, chemical, thermal, UV radiation, or a combination of these factors, resulting in formation of microcracks deep within the structure where detection and external intervention are difficult or impossible. In an attempt to address these challenges, the concept of self-healing polymeric materials was proposed in the 1980's to enable healing of invisible microcracks for extending the working life and safety of the polymeric components. This is an emerging and fast expanding area of research. This presentation will cover the latest developments on self-healing polymers including novel method for preparing self-healing microcapsules, new healing agents, delivery systems for self-healing materials, and the potential use of the self-healing materials in coating, electronic, packaging, and aerospace applications.

#### 4:50 PM

## Behavior of Corrosion Prevention in Self-Healing Polymer Coatings: Soo Hyun Cho<sup>1</sup>; Sangmin Jeon<sup>2</sup>; <sup>1</sup>POSCO; <sup>2</sup>POSTECH

Self-healing polymer coatings, at a very simplistic level, mimic the selfhealing nature of skin in living systems. This study demonstrates self-healing polymer coatings which provide very good corrosion resistance to metal substrates. Polymer coatings are commonly applied to metal substrates to prevent corrosion in aggressive environments. However, once the polymer coating has been breached, for example due to cracking or scratches, it loses its effectiveness, and corrosion can rapidly propagate across the substrate. The self-healing system we will describe prevents corrosion by healing the damage through a healing reaction triggered by the actual damage event. The anti-corrosion properties of the self-healing polymer on metal substrates are investigated by corrosion resistance and electrochemical tests as well as microscopic observation. Even after scratch damage completely through the substrate, the coating is able to heal, while control samples which do not include all the necessary healing components reveal rapid corrosion propagation. This self-healing coating solution can be easily applied to most substrate materials, and is compatible with most common polymer matrices. Self-healing coatings have the potential to extend the life time of coatings and can dramatically reduce the frequency of repair.

#### 5:05 PM Invited

## **Mathematical Modeling of Polymer Biodegradation and Erosion**: Yuhang Chen<sup>1</sup>; *Qing Li*<sup>1</sup>; <sup>1</sup>University of Sydney

Fri. AM

The biodegradable polymers are widely used in therapeutic surgery and pharmaceutics, in which the degradation process has drawn significant attention in recent years. In this paper, we propose a mathematical model to predict the polymer degradation in tissue engineering applications. A stochastic model is introduced to characterize the hydrolysis reaction in an elemental fashion and the mass transport is also performed to investigate the diffusive mechanisms of polymer erosion. Two representative polymeric plates in different configurations are studied. It is found that for biodegradable systems, mass transport plays an important role in controlling the erosion pathway, in which the matrix configuration could be one of the key factors that determine the characteristics of erosion and drug release rates. The proposed model makes a useful benefit to the design optimization of biodegradable polymers, i.e. the tissue scaffold and drug delivery system.

#### 5:20 PM

**Biodegradable Ocular Implants**: *Subbu Venkatraman*<sup>1</sup>; Peng Yan<sup>1</sup>; Zbigniew Stachurski<sup>2</sup>; Tina Wong<sup>3</sup>; <sup>1</sup>Nanyang Technological University; <sup>2</sup>Australian National University; <sup>3</sup>Singapore National Eye Centre

For treatment of post-surgical scarring, drug-eluting film implants are a novel approach. Specifically, we have developed biodegradable thin films that can elute anti-inflammatory agents over weeks. In order to maintain drainage for glaucoma patients, such implants are made to remember a shape (shape memory). The process used to incorporate such shape memory is described in detail, along with issues encountered for specific polymers. The influence of the "setting temperature" and the recovery temperature on the shape memory effect are also described. Results show that the important structural features that influence shape memory are crystallinity and the magnitude of the glass transiton temperature relative to the recovery temperature. We have also implanted these films into rabbit eyes, and monitored their degradation over time. Results show that the PLGA(n=9) and the PCL copolymer (n=9) microfilms degraded steadily within the subconjunctival pocket and remained in-situ without migration. Clinical monitoring showed that both types of microfilm degraded gradually without inciting any inflammation or toxicity in all test eyes. In vitro studies show that such films can incorporate anti-inflammatories and release them in a sustained fashion over 2 months. Films show Fickian diffusion kinetics.

#### 5:35 PM

## Changes of Bone Quality and Quantity in rhM-CSF-Treated Osteopetrotic (op/op) Mice: Jee-Wook Lee<sup>1</sup>; Takayoshi Nakano<sup>1</sup>; <sup>1</sup>Osaka University

Bone mineral density (BMD) can not perfectly predict bone fracture risks in some clinical cases, while BMD is a useful index for diagnosis of the bone condition. Recently, various parameters relating to bone soundness have been investigated. Among the various factors, we have been focusing on the preferential orientation of the biological apatite (BAp) crystallites as the bone quality parameter, analyzed by micro-XRD which is a powerful tool to analyze BAp crystallites in bones. BAp which is a main component of bone is an ionic crystal with hexagonal lattice accompanied with the anisotropic properties. In this study, we investigated a formation mechanism for BAp orientation during endochondral or membranous ossification using administration model of M-CSF in the osteopetrotic (op/op) mice which the M-CSF is deficient. op/op mice treated by intraperitoneal injections of 5µg rhM-CSF, the first injection was the 14th day after birth. In the treated op/op mice, the bone marrow cavities were expanded significantly, associated with increased numbers of osteoclast. Moreover, BAp orientation along longitudinal direction exhibited high degree compared with the non-administration group. These results suggest that M-CSF is a controlling parameter for the microstructural formation of preferential alignment of BAp caxis.

#### 5:50 PM

## Effect of Penetration Rate on Insertion Force in Trabecular Bone Biopsy: *Renae Mulligan*<sup>1</sup>; Ling Yin<sup>1</sup>; Anthony Lamont<sup>1</sup>; Zongxiao Peng<sup>1</sup>; Mark Forwood<sup>1</sup>; Swee Hin Teoh<sup>1</sup>; <sup>1</sup>James Cook University

Bone biopsy is a common procedure in bone disease diagnoses, therapies and research. In this procedure, bone biopsy needles are operated to insert into bone tissues. Although needle insertion of bone is often essential for the diagnosis of bone diseases, the hard tissue-needle interactions are not quantitatively understood. In this paper, we describe a quantitative assessment of forces involved in insertion of healthy trabecular bone using clinically applied Jamshidi CrownTM bone biopsy needles of gauge 8 (4-mm diameter). The forces were measured to be related to the insertion depths up to 25 mm and insertion rates of 1 mm/s to 5 mm/s. At the initial insertion stage, a clear linear force-depth relation was measured. With the increase of the insertion depth, the forces increased nonlinearly. In the final stage of insertion, the forces increased much more quickly at the lower insertion rate than that at the higher insertion rate. The maximum insertion force can reach approximately 1000 N when the insertion depth reached 25 mm at the insertion rate of 1 mm/s.

#### 6:05 PM

## Study of Electroless-Deposited Nano-Silver on Medical Polyurethane Catheter: Ying Liu<sup>1</sup>; Xiaobing Li<sup>1</sup>; <sup>1</sup>Nanchang University

Polyurethane catheter is widely used to medical devices and implants. The main objective to study electroless-deposited silver on the surface of medical polyurethane is for improving the anti-bacterium properties. In this study, the sodium hypophosphite, dextrose and potassium sodium tartrate were selected as reducers for electroless-deposited nano-silver. The influences of technological parameters, such as concentration of silver nitrate, mixture ratio of silver nitrate and reducer, reaction temperature and reaction time, on electroless-deposited nano-silver were investigated. The experimental results showed that with sodium hypophosphite as reducer, the optimal parameters are silver nitrate concentration of 0.25mol/L, mixture ratio of silver nitrate and reducer of 2:1, reaction temperature of 50°C, reaction time of 30min, and the average diameter of nanosilver is 25nm. With dextrose as reducer, the optimal parameters are silver nitrate concentration of 10g/L and reaction time of 30s, and the average diameter of nanosilver is 40nm. With potassium sodium tartrate as reducer, the optimal parameters are silver nitrate concentration of 16g/L, reaction time of 3min, and the average diameter of nano-silver is 50nm. In addition, adding the polymer protect agents and dispersing agents will help prevent nano-silver particles to continue growing and improve the dispersion uniformity of nano-silver particles.



#### Symposium J: Materials Characterisation and Evaluation: Processing and Modelling

Thursday PM	Room: 1	
August 5, 2010	Location:	Cairns Convention Centre

Session Chair: Zhe Liu, Monash University

#### 2:00 PM Keynote

Recent Progress on First-Principles Calculations and Experimental Results of Single-Crystalline Magnetic Tunnel Junctions with MgO Barriers: Jia Zhang<sup>1</sup>; Yan Wang<sup>1</sup>; Xiao-Guang Zhang<sup>2</sup>; Xiu-Feng Han<sup>1</sup>; <sup>1</sup>Chinese Academy of Science; <sup>2</sup>Oak Ridge National Laboratory

Since the theoretical predictions and experimental observations of giant tunneling magnetoresistance (TMR) effect at room temperature in magnetic tunnel junctions (MTJs) with single-crystalline MgO(001) barrier, MgO-based MTJs have been extensively studied due to their broad potential applications in spintronics devices. In this work, recent progress on theoretical calculations and experimental results in MgO-based MTJs is reported. Spin-dependent electronic structure and transport properties of MgO-based MTJs, including structures of Fe(001)/MgO/Fe/MgO/Fe, Fe(001)/Co/MgO/Co/Fe, Fe(001)/Mg/MgO/Fe, and Fe(001)/Mg/MgO/Mg/Fe, have been studied using the Layer-KKR method. The quantitative result not only provides a better way to understand the electronic structures and spin-dependent transport properties of MgO-based MTJs, but also shows a direction to exploit new kinds of spintronics materials with high room-temperature TMR ratio.

#### 2:20 PM

## Numerical Simulation of Double Specimens in Split Hopkinson Pressure Bar Testing: *Muhammad A. Kariem*<sup>1</sup>; John H. Beynon<sup>1</sup>; Dong Ruan<sup>1</sup>; <sup>1</sup>Swinburne University of Technology

The split Hopkinson pressure bar (SHPB) is the most commonly used technique to characterize the dynamic behaviour of materials at very high strain rates. However, a classic single specimen testing only generates single stress-strain behaviour at the average strain rate of the test. This paper proposes three arrangements on the use of double specimens in SHPB compression testing. All waves propagating along the bars have been used to analyse the dynamic behaviour of the specimens. A method on how to construct a strain profile from a final length of samples measurement was also proposed. To simulate the test and predict its dynamic performance, an axisymmetric finite element analysis using LS-DYNA was conducted for the experiment using 13 mm bar diameter. Validity of the simulations was checked with the experimental data from a classic SHPB testing. Based on the simulations, the modified techniques are achievable and at least two stress-strain behaviours of materials are possible to be extracted without violating the requirement of a valid SHPB testing.

#### 2:35 PM

Carrier Recombination Activity and Potential Barrier at Grain Boundaries in Polycrystalline Silicon: *Sadahiro Tsurekawa*<sup>1</sup>; Hiroaki Takahashi<sup>1</sup>; Yumi Nishibe<sup>2</sup>; <sup>1</sup>Kumamoto University; <sup>2</sup>Tohoku University

Importance of polycrystalline silicon has been recognized in the electronic device technology. The interfacial states in the band-gap and potential barrier associated with grain boundaries in polycrystalline silicon can exert their detrimental influence on electrical conductivity and then on device performance. However, grain boundaries are not always at the origin of electrical activity because individual grain boundaries have their own character depending on the orientation relation between two adjoining grains. We apply the electronbeam-induced current technique and the Kelvin probe force microscopy to observe the carrier recombination intensity and the potential barrier height, respectively, at well-characterized grain boundaries in polycrystalline silicon. The observed barrier height of grain boundaries varied in the range from 10 to 100 meV depending on the grain boundary character. The potential barrier height was found to be approximately twice higher at random boundaries than at coincidence boundaries. In addition, we found that impurity contamination (Fe, Cu) increased grain boundary potential barrier height by 3 - 5 times with respect to the non-contaminated sample. The influence of impurity contamination on the barrier height was more significant at random boundaries than at  $\Sigma$  3 and  $\Sigma$  9 CSL boundaries.

#### 2:50 PM

Using Chemiluminescence to Study the Photodegradation of Materials: *Keith Millington*<sup>1</sup>; Michael Jones<sup>1</sup>; Siti Farhana Zakaria<sup>2</sup>; George Maurdev<sup>1</sup>; <sup>1</sup>CSIRO Materials Science and Engineering; <sup>2</sup>School of Fashion and Textiles, RMIT University

Conventional techniques for studying free radicals, either directly by electron spin resonance (ESR) or indirectly via separation and fingerprinting of free radical oxidation products, are complex, time consuming and the necessary hardware is expensive. Chemiluminescence (CL) is a simple alternative method for studying free radical reactions in organic materials, which usually emit weak CL when they undergo oxidative degradation. This CL originates from side reactions of peroxy radical and hydroperoxide intermediates formed during the autoxidation chain reaction. CL techniques have been widely used to study the thermal degradation of polymers, but their application to photodegradation of materials has been very limited. Here we discuss a simple modification to an existing commercial CL instrument (Lumipol 3) and an effective experimental protocol to study photoinduced chemiluminescence (PICL) from materials. Application of PICL to polymer films and coatings, fibrous webs, such as textile fabrics or paper, and powdered samples is described, together with the effects of additives such as UV absorbers, antioxidants, dyes and pigments on PICL emission.

#### 3:05 PM

Design of Pre-Weakening and Evaluation of Structural Safety for Explosive Demolition of Concrete Silo: *So-Young Park*<sup>1</sup>; Hoon Choi<sup>1</sup>; Seung-Cheol Baek<sup>2</sup>; Hyo-Jin Kim<sup>3</sup>; Soon-Jong Yoon<sup>1</sup>; <sup>1</sup>Hongik University; <sup>2</sup>Andong National University; <sup>3</sup>Korea Land & Housing Corporation

Recently the demand of demolition for the unnecessary cylindrical silo structure is increasing due to the deterioration and malfunctional conditions and the issue of demolition is becoming a major highlight. To minimize environmental hazards created during the process of demolition, the explosive demolition method has been applied increasingly. The concrete silo structure is often built by the slipform method which is the continuous concrete casting operation. As a result, the structure does not have expansion joints or other structural weaknesses that can be used to assist in the demolition process. This study presents the preweakening of explosive demolition of the cylindrical concrete silo structure by overturning method. Pre-weakening for the explosive demolition of the structure is usually conducted by the field experience without technical guideline for the pre-weakening procedure. Hence, there always exist safety-related concerns. To demolish the structure by blast effectively and safely, rational approach for the pre-weakening and evaluation method of the load carrying capacity of the pre-weakened structure should be established. For this, mechanical properties of concrete are estimated by experiment. Based on the result of inuestigation, design or operation methods of pre-weakening for the blast demolition are discussed.

#### 3:20 PM

#### Processing Temperature and Oxide versus Non-Oxide Filler Effects on Low Temperature Cured Aluminosilicate Ceramic Properties: Damian Fullston<sup>1</sup>; Kwesi Sagoe-Crentsil<sup>1</sup>; <sup>1</sup>CSIRO Materials Science & Engineering

The effects of processing temperature on oxide and non-oxide filled geopolymer materials properties have been studied. Both processing temperature, from ambient to 60°C, and filler type are shown to significantly influence the materials properties. At the elevated curing temperatures, early strength gain is enhanced with oxide fillers of Alumina and Titania. Non-oxide filler material, boron nitride, however exhibits reduced strength at all processing temperatures investigated. Thermogravimetric and differential thermal analysis indicated minor alterations to thermal stability of samples cured under the different processing temperatures and with the different filler materials. Abrasion resistance was affected more by filler type than cure temperature. Microstructural characterizations revealed the importance of interfacial reactions between filler and matrix in determining the final material properties. This paper explores the basis for the enhanced properties of oxide additives to these geopolymer systems and the potential of improved interfacial bonding between aluminosilicate matrix and filler particles.

#### 3:35 PM

#### The Bonding Structure of the Various High Purity or Binderless Polycrystalline Cubic Boron Nitride Compacts Sintered at 5 to 7GPa and 1600 to 2000°C: Akhmadi Eko<sup>1</sup>; Itsuro Tajima<sup>1</sup>; Minoru Akaishi<sup>1</sup>; <sup>1</sup>Mitsubishi Materials Corporation

The method for sintering the high purity or binderless polycrystalline cubic Boron Nitride (cBN) compact using the high pressure high temperature technology has been widely known. In this paper we review the cBN-cBN direct bonding structure of some cBN compacts made by the method of the direct conversion of the pyrolytic-BN and wurtzite-BN to cubic-BN at the pressure of 7GPa and temperature 2000°C and the cBN compact made by the method of sintering the cBN powder along with polyvinylidene chloride (PVDC) at the condition of 5.5GPa and 1600°C. The PVDC became a supercritical fluid that will act as a



cBN crystal surface cleaner in advancing the direct bonding between the cBN crystals. The XRD, SEM, TEM analysis and the wear resistance on the ductile cast iron machining has been investigated. While the XRD analysis of the above cBN samples showed that all samples consist only of cBN, and the SEM observation showed that they all have the cBN-cBN direct bonding structure, A TEM analysis showed the existence of the impurities at the cBN crystal boundary. The starting materials and the sintering method affect the degree of these impurities.

#### 3:50 PM

**Rheological Properties of a Particulate-Filled Polymeric Composite through Fused Deposition Process**: *Mostafa Nikzad*<sup>1</sup>; Syed Masood<sup>1</sup>; Igor Sbarski<sup>1</sup>; Andrew Groth<sup>2</sup>; <sup>1</sup>Swinburne University of Technology; <sup>2</sup>CSIRO

This paper presents an investigation on rheological and mechanical properties of a new ABS (acrilonitile-butadeine-styrene)-Iron composite for application in Fused Deposition Modelling prototyping process. Test samples of ABS-Iron composites have been made by controlled centrifugal mixing, thermally compounding through a single-screw extruder and compression moulding. Rheological characterization was conducted using a capillary rheometer by measuring pressure drop under varying extrusion speeds. Apparent shear rate and shear stress as well as viscosity of the melts were calculated. Computer numerical analysis was used taking into account a very high shear viscosity due to volume fraction content of up to 30% of metallic filler in order to substantiae existing models of viscosity for such composites. Modulated differential-scanning calorimetry techniques were used in order to characterize viscoelastic properties of these newly developed composites materials for use in fused deposition modelling process. Non-Newtonian behaviour of the composite melt was shown to follow a cross model of shear thinning characteristics by calculating Rabinowitsch-Mooney corrected values of shear viscosity and shear rate. Dynamic mechanical analysis conducted on a multi-frequency-dual cantilever showed a substantial increase of storage modulus and a shift in glass transition temperature comparing to that of a non-filled matrix material.

4:05 PM Tea Break

#### Symposium J: Materials Characterisation and Evaluation: Other Advanced Technology

Thursday PMRoom: 1August 5, 2010Location: Cairns Convention Centre

Session Chairs: Ian Gentle, Australian Synchrotron; Sadahiro Tsurekawa, Kumamoto University

#### 4:30 PM Keynote

First Scientific Results from the New OPAL Research Reactor in Sydney, Australia: Robert Robinson<sup>1</sup>; <sup>1</sup>ANSTO

Australian science is entering a new "golden age", with the recent startup of bright new neutron and photon sources in Sydney and Melbourne, respectively. The OPAL reactor and the Australian Synchrotron can together be considered the greatest single investment in scientific infrastructure in Australia's history. Fuel was loaded into the OPAL reactor in August 2006, and full power (20MW) achieved in November 2006. The formal user commenced in 2007, and fully analysed data sets have now been taken on all seven of the initial suite of instruments. 3 further instruments are in various states of construction, and substantial additional investment is also being made in sample-environment, extra instrumental options and polarised-neutron technology. An update will be given on the status of OPAL, the performance of its thermal and cold neutron sources and instruments, a selection of the first scientific results and future plans, with particular reference to Advanced Materials and Processing.

#### 4:50 PM

#### Modern Diffraction Methods for the Investigation of Thermo Mechanical Processes: *Klaus-Dieter Liss*<sup>1</sup>; Kun Yan<sup>1</sup>; Lewis Ryan<sup>1</sup>; Ian Watson<sup>1</sup>; Ulf Garbe<sup>1</sup>; <sup>1</sup>ANSTO

Well collimated, high energy X-rays of 90 keV from synchrotron sources have been used to study metals undergoing phase transformations and plastic deformation in-situ, in real time and in the bulk of the materials. The spottiness and intensity distribution around the Debye-Scherrer rings, showing reflections from individual crystallites is analyzed to obtain grain statistics, mosaic spread and orientation. Upon cold deformation, coarse grained materials show fingerprints of sub-grain formation, grain rotation, grain refinement and the evolution from a single grain into the asymptotic texture. Heating of metals under continuous load drives the observation through the regimes of phase transformation and grain orientation relationships therein, grain coarsening, dynamic recovery and dynamic recrystallization. Complementary in-situ neutron diffraction reveals further insight into atomic order and disorder, phase transformations and the thermo-physical properties of large bulk materials. The paper points out these different phenomena which have been observed without precedence.

#### 5:05 PM Keynote

**Defect and Damage Evolution Quantification in Dynamically Deformed Metals Using Orientation-Imaging Microscopy**: *George Gray*<sup>1</sup>; Veronica Livescu<sup>1</sup>; Ellen Cerreta<sup>1</sup>; <sup>1</sup>Los Alamos National Laboratory

Orientation-imaging microscopy offers unique capabilities to quantify the defects and damage evolution occurring in metals following dynamic and shock loading. Examples of the quantification of deformation twin type activation, volume fraction, and damage evolution as a function of one-dimensional shock loading, dynamic shear localization, and sweeping-wave shock loading will be presented. Examples of damage evolution in Cu, Ta, Zr, and Fe will be detailed. In addition, the limitations and caveats involved in using velocimetry and single-pass radiography to elucidate the 3-D aspects of defect generation, storage, and recovery will be examined in detail. Examples of how both "real-time" and post-mortem experimental approaches are needed to quantify dislocation / defect generation, shock-induced phase transitions, and damage evolution as a function of stress state will be discussed.

#### 5:25 PM

Investigation of Structural Stability of Monolayer MnO<sub>2</sub> Sheet under Electron Beam Irradiation: *Yong Wang*<sup>1</sup>; Chenghua Sun<sup>1</sup>; Jin Zou<sup>1</sup>; Lianzhou Wang<sup>1</sup>; Sean Smith<sup>1</sup>; Gaoqing Lu<sup>1</sup>; David Cockayne<sup>2</sup>; <sup>1</sup>The University of Queensland; <sup>2</sup>Oxford University

Atomic structures determine the properties (such as physical, chemical and mechanical) of materials, especially, when materials go from 3 dimensional (3D) down to 2D, 1D or even to several atomic layers [e.g., nanowires, naonotubes and nanosheets]. Obviously, knowledge from the dimensional stability of these nanomaterials will play a vital role in their potential applications. For example, will irradiation on these nanomaterials cause new structure or phase transformation? In the last few years, much attention has been paid on related investigation of 1D nanowires and nanotubes. Recently, people reported unexpected finding of free-standing graphene and related novel properties, which opens a door to 2D monolayer sheets. However, within this atomic level, whether these sheets with many dangling bonds are stable or atomic reconstructions will happen is still unclear and remains a great of challenge. Here we present interesting findings on the structural stability of monolayer MnO2 sheets under electron-beam irradiation. Our study revealed that the monolayer MnO, sheets are not stable under irradiation and two new-ordered atomic reconstructions were observed. The observed atomic reconstructions of monolayer MnO, sheets are expected to prominently broaden our knowledge on the structure of 2D ultrathin sheets.

#### 5:40 PM

High-Speed Fracture Phenomena of Glass Bottle by Underwater Shock Wave: *Hidetoshi Sakamoto*<sup>1</sup>; Shinjirou Kawabe<sup>2</sup>; Yoshifumi Ohbuchi<sup>1</sup>; Shigeru Itoh<sup>1</sup>; <sup>1</sup>Kumamoto University; <sup>2</sup>Kumamoto Prefecture Technical Collage

In this study, I pay attention to the recycling process by smashing the glass bottle, and I will discuss the smashing technique of glass bottle by underwater shock wave in order to make the 'cullet' (small fragments of glass) effectively. The high-speed fracture mechanism of glass bottle is clarified by observing with a high-speed camera and propose the practical technique of this new efficient recycling method of the glass bottle. This propose method have a lot of advantages in comparison with a conventional large-scale smash machine such as 1) the smashing equipment cost is low, 2) the smashing fragments of glass are without scattering on the outside and is able to collect completely because the smashing work is done in water(or washing liquid), 3) the washing are unnecessary before the smashing and the smashing fragments are able to wash completely (this method is applicable for the medicine glass ), 4) the smashing and washing operation are able to do continuously.

#### 5:55 PM

#### **Properties of Screen Printed Ceramic Green Films Determined by Optical** Laser Profilometry: *Robert Muecke*<sup>1</sup>; Norbert Menzler<sup>1</sup>; Hans Peter Buchkremer<sup>1</sup>; Detlev Stöver<sup>1</sup>; <sup>1</sup>Forschungszentrum Juelich GmbH

The green density, roughness, and tensile stress of green ceramic layers determine their mechanical and micro structural properties after final sintering. These properties can be measured precisely by laser profilometry. The green density of thin layers (20-50  $\mu$ m) could quickly be determined as accurately as 0.5% theoretical density. The influence of paste parameters (powder conditioning, solid content, binder, and dispersing agent) on the green density was studied systematically for electrolyte pastes (8 mol.-% yttria stabilized zirconia, d<sub>s0</sub> = 0.15 ... 0.4  $\mu$ m) typically used in solid oxide fuel cell applications. It could be shown that a minimal binder content is required to achieve acceptable green



densities. Pre-calcination of the powder yielded also significantly higher film densities. Dispersant agents contributed to a smoother surface in any case, however a significant effect on the packing density was only observed for the fine, non-calcined powder. The measurement of the pastes' viscosities and flow limits together with roughness data and stress calculations allowed the physical interpretation of the observed dependencies.

#### 6:10 PM

**Texture Refinement Using a Reitveld Strategy**: *Saurabh Kabra*<sup>1</sup>; Kun Yan<sup>2</sup>; Klaus-Dieter Liss<sup>1</sup>; <sup>1</sup>ANSTO; <sup>2</sup>University of Wollongong

Texture refinement techniques have come of age in recent years. Depending on the limitation of the experiment, it is not always possible to measure a full orientation map in order to determine the texture. It is desirable to extract the most out of the available data in these cases. The advances in computing power, better optimization algorithms and the development of better area detectors have made it possible to extract ODFs/texture from limited x-ray and neutron diffraction datasets. The Rietveld technique which has proved its usefulness in multiple areas of diffraction is also proving to be effective in texture computation. Rietveld combined with advanced texture models like EWIMV (by Luterotti and Wenk) have made texture computation possible under a number of limiting conditions and situations. In this study, we have used this recent technique to refine data obtained at the newly built diffractometer WOMBAT at ANSTO to show its application and effectiveness.

#### 6:25 PM

TEM and STEM Study of the Au Nano-Particles Supported on Cerium Oxides: *Tomoki Akita*<sup>1</sup>; Shingo Tanaka<sup>1</sup>; Koji Tanaka<sup>1</sup>; Masanori Kohyama<sup>1</sup>; <sup>1</sup>National Institute of Advanced Industrial Science and Technology (AIST)

It is well-known that the Au nano-particles supported on metal oxides exhibits high catalytic activity. It was also reported that the catalytic properties of Au nano-particles are sensitive to the size of Au particles and the interface structure between Au and metal oxide support. Some experimental results indicate that the perimeter of Au particle and metal oxide interface performes key role for the low temperature CO oxidation, but the details are not undrestood. Thus, it is important to elucidate the structure of Au nano-particles and metal oxides interface in atomic scale by electron microscopy. In this study, the structures of Au particles on CeO, were observed by an analytical transmission electron microscopy (TEM) equipped with annular dark field scanning transmission electron microscopy (HAADF-STEM) systems. The Au/CeO, model catalysts were prepared by using the substrates of CeO<sub>2</sub> poly-crystal. The Au particles of 2-5 nm in diameter were deposited on the substrates by controlling the amount of Au. The orientation relationship of (111)[1-10]Au// (111)[1-10]CeO, was frequently observed in cross sectional HRTEM images. High resolution HAADF-STEM images were also obtained for Au-CeO<sub>2</sub> interface. The position of atomic columns of Au and Ce at Au-CeO, interface is directly investigated from HAADF-STEM images.

#### Symposium K: Composites and Hybrid Materials: Polymer and Other Composites

Thursday PM	Room: 4
August 5, 2010	Location: Cairns Convention Centre

Session Chair: Chun Wang, RMIT University

#### 2:00 PM Keynote

Protein Fibre Powders and Their Applications in Functional Composite Materials: *Xungai Wang*<sup>1</sup>; Weilin Xu<sup>2</sup>; <sup>1</sup>Deakin University; <sup>2</sup>Wuhan University of Science and Engineering

This talk is focused on the latest research in new composite and hybrid materials containing ultra-fine protein fibre powders. The production of natural organic powders from protein fibres is introduced. Ultra-fine powders from wool and silk fibres have been produced using a combination of different milling techniques. The applications of these fine powders in functional composite materials are discussed. These applications include hybrid fibres combining the advantages of natural and synthetic polymer fibres, tissue engineering scaffolds with enhanced mechanical properties, permeable membranes, and strong and biocompatible small-diameter vascular grafts. In each of the application areas, incorporation of the natural powders has significantly enhanced the properties and functionalities of the resultant composite materials. For instance, the powders improved the moisture uptake and comfort attributes of the hybrid fibres, increased the compression modulus of the silk scaffolds, and enhanced the permeability of powder-containing membranes and the biocompatibility of the vascular grafts.

#### 2:20 PM Invited

**Porous Coordination Polymer Composite Membranes for Gas Separation**: *Bradley Ladewig*<sup>1</sup>; Matthew Hill<sup>2</sup>; Aaron Thornton<sup>2</sup>; <sup>1</sup>Monash University; <sup>2</sup>CSIRO Materials Science and Engineering

Widespread concern over climate change is driving the development of membrane technologies which can cost-effectively separate carbon dioxide from flue gas streams, so that it can be sequestered in deep geological formations. Our recent work in this field has examined the use of porous coordination polymers, which have some particularly exciting capabilities to separate gas molecules of very similar size by molecular sieving. Molecular dynamic simulations on recently characterized Zeolitic Imidazolate Frameworks (ZIFs) show that very high selectivities may be possible. However the preparation of useful membranes from such materials is extremely challenging, and as such we propose a novel composite membrane architecture that moves beyond the typical mixed-matrix type membranes, to produce flexible, manufactuable composite membranes. This novel architecture should allow the porous coordination polymer to sieve the gas molecules, while being bound into a sealable, gas-tight structure which is flexible due to a polymer binding agent. Preliminary membrane characterisation and permeation results will be presented.

#### 2:35 PM

Effect of TEOS Addition on Adhesion and Electrochromic Properties of Silica-Polyaniline Core-Shell Composite Films on ITO Glass: *Gyuntak Kim*<sup>1</sup>; Hohyeong Kim<sup>1</sup>; Taejin Hwang<sup>1</sup>; Heung Yeol Lee<sup>1</sup>; <sup>1</sup>KITECH

The adhesion enhancement effect of tetraethoxysilane (TEOS) addition to a polyaniline-silica composite film on ITO glass substrate was studied. The composite film was prepared with the polyaniline-silica composite nanoparticles which were prior synthesized by a chemical polymerization of polyaniline in a colloidal solution of silica. A small amount of TEOS and acidic water was added to the dispersion solution of polyaniline-silica nanoparticles. The addition of TEOS was intended to increase the adhesion of electrochromic film to substrate and hence to improve the electrochromic properties such as response time and cycle life. The composite film with TEOS showed a stronger adhesion to the ITO-coated glass substrate compared with the film deposited without TEOS. The adhesion and characterization of the film were performed by tape test, scratch test, scanning electron microscopy (SEM) and Fourier transformedinfrared spectroscopy (FT-IR). The cyclic voltammetry and the optical response results showed that the strong adhesion of the film enhanced the electrochromic properties.

#### 2:50 PM

Extended-Chain Crystals of High-Pressure Crystallized Poly (Ethylene Terephthalate) Oligomer/Bisphenol-A Polycarbonate Blends: Jun Lu<sup>1</sup>; Rui Huang<sup>2</sup>; <sup>1</sup>Southwest Jiaotong University; <sup>2</sup>Sichuan University

With a combination of three-dimensional crystal ordering and long-chain molecular orientational ordering, the extended-chain crystals of polymers are an ideal system for the studies on low-dimensional physics and have potential applications as functional components. In this study, the crystallization behaviors of the poly (ethylene terephthalate) oligomer/bisphenol-A polycarbonate (PETO/ BAPC) blends were investigated under high pressure, and the recovered PETO/ BAPC samples were characterized using XRD, DSC, SEM and AFM. The results showed that high-quality extended-chain single crystals with large C-axis thickness were formed in the multi-phase system within a relative short time, and the inner defects of the disclosed crystals could be removed by a self-healing process through high-temperature and high-pressure annealing. Morphologies of extended-chain crystals with different characteristics were also revealed with SEM and AFM. Wedge-shaped and bent extended-chain morphologies suggested that the sliding diffusion and the transesterification occurred simultaneously in the system, and the crystallization of the large crystals was a self-assembling process induced by chemical reactions at high pressure. This process provides a new route to grow large polymer extended-chain crystals and suggests selfassembly under high pressure is a promising method to create materials with new structures and properties.

#### 3:05 PM

Improvement of Mechanical Properties of Injection Molded Wood/ Polypropylene Composites Parts with Ultrasonic Oscillation Assistant: Lei Xie<sup>1</sup>; Timo Gruenerberg<sup>2</sup>; Leif Steuernagel<sup>1</sup>; Gerhard Ziegmann<sup>1</sup>; Holger Militz<sup>2</sup>; <sup>1</sup>Clausthal University of Technology; <sup>2</sup>University of Goettingen

Due to the water absorption of wood fillers and poor adhesion between wood fillers and polymer matrix, the loosen material structure always appears in wood/ polymer composites after injection molding process, which results in reduced composites mechanical properties. In this study, two kinds of wood particles with different sizes and properties were compounded with Polypropylene (PP) in highly filled level (by 50% and 60% weight concentration). The experimental tensile test samples were prepared by one double-gate injection mould integrated



an ultrasonic generator unit. The experiments were carried out for studying how the ultrasonic output power and the oscillation inducing time affect the injection molded wood/PP composites mechanical properties. 3 output power levels (400W, 600W and 800W) and 2 inducing mode were set (Mode1. the oscillation is induced from injecting moment to ejection moment; Mode2. the oscillation is induced from injecting moment to packing procedure finishing). The results show that the E module, tensile strength and density of the test parts are obviously changed with various ultrasonic output power and inducing time. Comparing the mechanical properties of parts with and without ultrasonic assistant indicates that the ultrasonic oscillation is a practical method to improve mechanical properties of injection molded wood/PP composites parts.

#### 3:20 PM

In-Plane Compressive Properties of Hybrid Dyneema®/Carbon Fiber Reinforced Polymer Matrix Composites: *Shahram Amini*<sup>1</sup>; John Shaw<sup>1</sup>; Michael Rossol<sup>1</sup>; Frank Zok<sup>1</sup>; <sup>1</sup>University of California, Santa Barbara

Fiber reinforced polymer (FRP) matrix composites have emerged as structural materials used in a diverse range of applications such as aircrafts, helicopters, satellites, ships, automobiles, sporting goods and civil infrastructure among many others. The characteristic microstructure of these composites is a two-dimensional (2D) laminated structure, with no fibers in the through-thickness (or z-) direction, resulting in low in-plane mechanical properties. The principal objective of the present study is to investigate the potential performance benefits derived from the addition of high performance polyethylene fibers to carbon fiber reinforced polymer (CFRP) composites. The study focuses specifically on 3D orthogonal weaves with carbon employed for the warp and weft yarns and Dyneema® for the z-yarns. Experiments on a series of composite panels with various volume fractions of z-yarns demonstrate that the retained in-plane compressive strength following impact is indeed enhanced by the presence of the z-yarns. The benefits derive from a reduced propensity for delamination during impact and buckling of the in-plane fibers during subsequent compressive loading. Analogous trends are obtained for open-hole compression. Insights into the failure mechanisms are gleaned from microstructural examinations of impacted specimens as well as insitu full-field strain measurements during compression testing.

#### 3:35 PM

Polymer Based Composite and Hybrid Materials for Wind Power Generation: Nikoloz Chikhradze<sup>1</sup>; Fernand Marquis<sup>2</sup>; Levan Japaridze<sup>3</sup>; Guram Abashidze<sup>1</sup>; Levan Okujava<sup>4</sup>; <sup>1</sup>Mining Institute of Georgia/Georgian Technical University; <sup>2</sup>Naval Postgraduate School, Wayne Meyer Institute of Systems Engineering; <sup>3</sup>Mining Institute of Georgia; <sup>4</sup>Institute of Building Mechanics

In recent years considerable attention has been dedicated to renewal power sources, such as wind power. This work was carried out in order to develop a small wind turbine with 1-10kW power generation capability. This wind turbine is designed to be energetically more efficient by 30-50% and having a lesser specific cost (by 25-30%). This work focused on the development of composite materials for application on the blades in the wind generator. In this paper we present the results of the research work done on the development of flexible technology for the fabrication matrix-epoxy resin based hybrid composites, reinforced with carbon, basalt and glass fibers. These new composite and hybrid materials were fabricated using epoxy matrixes. These matrices were reinforced with basalt and carbon fibers of different content and strengthened by mullite-like crystals. The basalt fibers for this composite reinforcing were prepared from raw materials, with chemical composition: SiO<sub>2</sub>-47.4%; Al<sub>2</sub>O<sub>3</sub>-15.3%; CaO-10.8%; Na<sub>2</sub>O-4.2%; MgO-8.8%; Fe<sub>2</sub>O<sub>3</sub>-12.1%; MnO-0.7%; TiO<sub>2</sub>-0.7%. The properties new composites developed depend on content/and position of reinforcing components and are: tensile strength- (0.012-1.590)GPa; compression strength (0.078-0.656)GPa; modulus of elasticity: (8.4-162.9)GPa; Poisson ratio: (0.015-0.559). The variation of strength and elastic characteristics under tension/and compression of the new composites will be presented.

#### 3:50 PM

## **Extrusion of Hybrid Lightweight Profiles**: *Soeren Mueller*<sup>1</sup>; Jérôme Muehlhause<sup>1</sup>; <sup>1</sup>TU Berlin

The latest efforts in reducing the  $CO_2$  emissions and with that the increasing interest of the automotive and transportation industries in light-weight constructions have caused a new era for magnesium alloys as a construction material. But as of today the use of magnesium alloys in high-volume production cars is very limited. Besides some other influences this is also due to the poor corrosion resistance of most wrought magnesium alloys. So in order to increase the use of magnesium profiles the corrosion resistance needs to be improved. This can be achieved by coating the profiles after the extrusion. Unfortunately this would result in an extra production step and thus additional costs and time. A coating in the same step as the extrusion could overcome this problem. Therefore, the idea is to use the commonly used light alloy aluminum as a "coating" for magnesium profiles. The profiles are manufactured by direct and indirect

extrusion from specially prepared hybrid Mg/ Al billets. Hence light magnesium profiles with a corrosion resistant aluminum coating can be produced in a single production step.

#### 4:05 PM Tea Break

#### Symposium K: Composites and Hybrid Materials: Other Composites

Thursday PMRoom: 4August 5, 2010Location: Cairns Convention Centre

Session Chair: Bradley Ladewig, Monash University

#### 4:30 PM

**Mechanical Reinforcement of Three-Dimensional Spacer Fabric Composites**: *Shaokai Wang*<sup>1</sup>; Min Li<sup>1</sup>; Yizhuo Gu<sup>1</sup>; Zuoguang Zhang<sup>1</sup>; Boming Wu<sup>2</sup>; <sup>1</sup>Beihang University; <sup>2</sup>Changzhou Bolong Three Dimensional Composites Co., Ltd

Three-dimensional (3-D) spacer fabric composite is a novel lightweight sandwich structure, the reinforcement of which is integrally woven with two facesheets connected by continuous fibers (named piles) in the core. Usually the 3-D spacer fabric composite without extra reinforcement is called monospacer fabric composite, which provides outstanding facesheet/core debonding resistance. However, its mechanical properties cannot meet the demand of structure application because of the thin facesheet and low load-bearing capacity of high piles. Hence, two reinforcement methods were developed by laminating additional weaves at the facesheet and filling foam materials in the core to strengthen the facesheet and piles, respectively. This paper aims to investigate the influences of reinforcement methods on the mechanical behaviours and damage modes of 3-D spacer fabric composites under flatwise compressive, shear, edgewise compressive and three-point flexural loads, by comparing with mono-spacer fabric composites. The results indicate that additional weaves reinforcement can enhance edgewise compressive and flexural properties effectively. Foam filling is one of the best options to improve the flatwise compressive and shear properties, and especially, there are synergy effects between piles and foam under flatwise compressive load. Besides, the failure modes of reinforced and mono-spacer fabric composites are different.

#### 4:45 PM

**The Australian Carbon Fibre Research Facility**: *Bronwyn Fox*<sup>1</sup>; Brad Dunstan<sup>2</sup>; <sup>1</sup>Deakin University; <sup>2</sup>VCAMM Limited

The Australian Carbon Fibre Research Facility forms part of the recently announced new Australian Future Fibre Research and Innovation Centre (AFFRIC) and will deliver the world's first pilot scale research plant capable of producing significant quantities of aerospace quality carbon fibre. The fibre will be used for research purposes as well as enabling research into the chemical, mechanical and nanoscale characteristics of new carbon fibre products. By leveraging the combined capabilities and resources of the partners, Deakin University, CSIRO Materials Science and Engineering and VCAMM, the facility will offer a university based, industrial-scale carbon fibre research facility unique in the world. This paper will outline the research themes which will be the focus of the new facility in addition to describing highlights from existing composite research projects.

#### 5:00 PM

Localised Contact and Impact Effects on Aluminium Foams and Laminates: Mark Hoffman<sup>1</sup>; *Tania Vodenitcherova*<sup>1</sup>; Kaveh Kabir<sup>1</sup>; Maizlinda Idris<sup>1</sup>; <sup>1</sup>The University of New South Wales

Firstly, the deformation of aluminium foam under uniaxial, flat and spherical punch contact is studied. Distinct deformation modes are determined are associated with compression of the foam cells and tearing of the cell walls. The relative significance of each mode in terms of the deformation energy is significant and varies with the nature of the contact. Secondly, the effect of the mechanical properties of the skin in determining the nature of the spherical contact and impact deformation is considered. The skin is high- and low-yield strength aluminium which is thin compared to the panel thickness. Having ascertained the nature of foam and sandwich composite deformation, the panels are loaded in bending to determine the effect of localised damage upon the ultimate strength of the system and compared with undamaged panels. A threshold level of contact damage is found to exist, above which the sandwich system demonstrates no strength degradation but above which strength reduction is significant. Importantly, the value of this threshold is also found to depend upon the foam panel thickness. Energy-based analytical models have been developed to describe



the experimentally-observed behaviour and provide predictive capabilities in regards to optimum panel architecture and material selection.

#### 5:15 PM

The Indentation Behaviour of Carbon Fibre Composite Tubes – Experiments and Modelling: *Ranjani Sudharsan*<sup>1</sup>; Bernard Rolfe<sup>1</sup>; Peter Hodgson<sup>1</sup>; <sup>1</sup>Deakin University

Metallic tubes have been extensively studied for their crashworthiness as they closely resemble automotive crash rails. Recently, the demand to produce light weight yet safer vehicles has led to the need to understand the crash behaviour of novel materials such as fibre reinforced polymer composites, metallic foams and sandwich structures. This paper discusses the static indentation response of carbon fibre reinforced polymer (CFRP) tubes. The side impact on a CFRP tube involves various failure mechanisms. This paper highlights these mechanisms and compares the energy absorption of CFRP tubes with similar Aluminium tubes. The indentation response of the CFRP tubes was modelled in ABAQUS finite element software using a composite fabric material model. The material inputs were given based on standard tension and compression test results and the in-plane damage was defined based on cyclic shear tests. The failure modes and energy absorption observed during the tests were well represented by the finite element model.

#### 5:30 PM

Role of Different Flyashes on the Formation of Geopolymers: *Pre de Silva*<sup>1</sup>; Kwesi Sagoe-Crentsil<sup>2</sup>; <sup>1</sup>Australian Catholic University; <sup>2</sup>CSIRO

Chemically, geopolymers are a group of inorganic polymers obtained by lowtemperature alkali activation of alumina- (Al<sub>2</sub>O<sub>3</sub>) and silica- (SiO<sub>2</sub>) containing materials. The chemical and physical properties of the geopolymers can vary depending on the ratio (i.e. degree of polymerisation) of Si:Al. The polymeric character of geopolymers increases with increasing Si:Al ratio. The type, chemical composition and reactivity of the raw materials and the curing conditions play a major part in controlling the chemistry of the resulting geopolymers, and hence the properties. Silica and alumina are major chemical constituents of fly ash. Therefore fly ash can be considered as a good source for geopolymer formation. Depending on the original coal, the chemical composition and reactivity of fly ash can be very different. This work examines the behaviour of different fly ashes (obtained from different power stations in Australia) with respect to alkali activation and geopolymer formation. Chemical and microstructural development of solid phases and the physical properties of the flyash-based geopolymer products are discussed with respect to the reactive alumina and silica amounts in the original flyashes.

#### 5:45 PM

Strengthening Behavior of Nanocrystalline Aluminum 2024 Alloy Based Composite Reinforced with Carbon Nanotubes: *Jaehyuck Shin*<sup>1</sup>; Hyun-joo Choi<sup>1</sup>; Byung-ho Min<sup>1</sup>; Dong-hyun Bae<sup>1</sup>; <sup>1</sup>Yonsei University

This study investigates strengthening effects of grain refinement, precipitation, and dispersion of multi-walled carbon nanotubes (MWNTs) in aluminum alloy. Al 2024 alloy powders are produced by chipping out of solution-treated alloy rods and the composites are produced by hot-rolling of the ball-milled mixture of Al 2024 alloy powders and MWNTs. During a milling process, MWNTs are gradually dispersed and embedded within the Al 2024 alloy powders. Simultaneously, grain sizes of the Al 2024 alloy matrix are effectively reduced down to 100 nm. Based on the specific process, composite containing 3 vol. % of MWNTs with grain size of 100 nm exhibits a tensile strength of ~800 MPa with a 2% plastic elongation to failure.

#### 6:00 PM

A Method to Extract Materials Properties from Multilayer Material Systems: Charles Moy<sup>1</sup>; Massimiliano Bocciarelli<sup>2</sup>; Simon Ringer<sup>1</sup>; Gianluca Ranzi<sup>1</sup>; <sup>1</sup>The University of Sydney; <sup>2</sup>Politecnico di Milano

This article presents an inverse analysis method based on an instrumented indenter to extract materials properties from multilayer material systems. In this case, a 12-layers multilayer system comprising of two alternate materials is considered. Each layer is 1  $\mu$ m thick. The alternate layers material selected are within the range of common commercial aluminium alloys. The yield stress and strain hardening exponent of the two layers were identified based on a power law type equation to define the stress-strain relationship. The use of finite element analyses was substituted with a fast and equally accurate approach for the iterative procedure. Thus, the computation time was considerably reduced. The robustness is tested using pseudo-experimental results with added random noises of 2.5% and 5%. The indentation curve and imprint on the material were utilized in the approximation. In addition, it was found that employing two indentation depths starting with the shallower one, helped improve the accuracy to determine the material parameters. The results are very promising with a maximum of 5% error with respect to the true material parameter values for the 5% added noise.

The technique can provide a valuable solution to multilayer coatings and small devices in order to obtain their properties.

#### 6:15 PM

Mechanical Properties of Metallic Closed Cellular Materials Containing Polymer Fabricated by Polymer Penetration: *Satoshi Kishimoto*<sup>1</sup>; Toro Shimizu<sup>2</sup>; Fuxing Yin<sup>1</sup>; Kimiyoshi Naito<sup>1</sup>; Yoshihisa Tanaka<sup>1</sup>; <sup>1</sup>National Institute for Materials Science; <sup>2</sup>National Institute of Advanced Industrial Science and Technology

Cellular materials have unique thermal, acoustic, damping and energy absorbing properties that can be combined with their structural efficiency. Therefore, many kinds of cellular materials have been developed and fabricated as structural, energy absorbing and damping materials. Particularly, closed cellular materials are thought to have many favorable properties and applications. In this study, metallic closed cellular materials containing polymer were fabricated by the penetrating polymer into metal foam. The aluminum and stainless steel foams were selected for the metal foam and epoxy resin and polyurethane resin were selected for the penetrated polymer. The many kinds of mechanical properties of this material were measured. The results of the compressive tests show that these materials have different stress-strain curves among the specimens that include different materials in the cells, higher compressive strength and higher Young's modules than those of closed cellular materials without any polymer. Also, other results show that this material has high-energy absorption, higher the internal friction than that of closed cellular material without any polymer.



#### Symposium B: Advanced High Temperature Structural Materials: Welding and Creep

Friday AM	Room: 7	
August 6, 2010	Location:	Cairns Convention Centre

Session Chairs: Yvonne Durandet, Swinburne University of Technology; Xiping Guo, Northwestern Polytechnical University

#### 8:30 AM Keynote

Welding Soundness of a Thick-Walled 9Cr-1Mo Steel: *Woo-Seog Ryu*<sup>1</sup>; Sung-Ho Kim<sup>1</sup>; Dae-Whan Kim<sup>1</sup>; <sup>1</sup>Korea Atomic Energy Research Institute

High Cr steels are a candidate material for the reactor pressure vessel of a very high temperature gas cooled reactor (VHTR). The thickness of reactor pressure vessel of VHTR may be above 100 mm. So it is very important to select the welding method for the sound weld of thick-wall vessel. Among many welding method, narrow gap welding has an attention for the thick-wall RPV material. The 9Cr-1Mo steel was welded by narrow gap SAW method. The welding integrity was checked by optical microscopy and hardness test. The hardness of base metal, weldment and HAZ also was measured by Vickers micro hardness tester. Impact energy and lateral expansion were increased slightly with the depth of weldment, but the difference was not so much. Fracture surfaces after impact test were shown as a normal appearance. Yield stress and tensile stress showed little difference with welding depth. Elongation was also almost same with depth. The fracture surface after tensile test was appeared as a normal. Thus, it concluded that the soundness of the narrow gap welding of a thick-wall 9Cr-1Mo steel was confirmed and the difference of impact and tensile properties according to weldment depth were negligible.

#### 8:50 AM Invited

Fundamentals of Spark-Plasma Sintering: Applications to Net-Shaping of High Strength Temperature Resistant Components: Eugene Olevsky<sup>1</sup>; Evan Khaleghi<sup>1</sup>; Cristina Garcia<sup>1</sup>; William Bradbury<sup>1</sup>; <sup>1</sup>San Diego State University

Spark-plasma sintering is an emerging powder consolidating technique which provides significant advantages to the processing of high temperature materials with poor deformability into configurations previously unattainable. Net-shaping capabilities of spark-plasma sintering are analyzed both theoretically and experimentally. Modeling and experimentation are conducted for cylindrical, prismatic, and complex powder specimen shapes. The impact of the "shape factor" on the non-uniformity of electric current density, temperature, relative density, and grain size spatial distributions is analyzed. The influence of various deformation schematics (pressing in rigid dies, forging, extrusion) on the outcomes of spark-plasma sintering of high temperature materials is considered. The modeling includes the novel constitutive concept of spark plasma sintering and the finite-element analysis with coupled electrical, thermal, and mechanical boundary conditions. The validity of conventional hot pressing models for the description of spark-plasma sintering is investigated. The modeling results are compared to the experimentally obtained data on the spark plasma sintering of high strength temperature resistant powder-based (TaC, SiC, and carbonnanotube-containing composites) specimens of various shapes. The conducted research indicates the promising capabilities and addresses the challenges of spark-plasma sintering of complex-shape parts.

#### 9:05 AM

The Potential of HAZ Property Improvement through Control of Grain Boundary Character in a Wrought Ni-Based Superalloy: *Hyun Uk Hong*<sup>1</sup>; In Soo Kim<sup>1</sup>; Baig Gyu Choi<sup>1</sup>; Hi Won Jeong<sup>1</sup>; Young Soo Yoo<sup>1</sup>; Chang Yong Jo<sup>1</sup>; <sup>1</sup>Korea Institute of Materials Science

The effects of grain boundary serration on grain coarsening and liquation behaviour in simulated weld heat-affected-zone (HAZ) of a wrought Ni-based superalloy Alloy 263 have been investigated. Recently, the present authors have found that grain boundary serration occurs in the absence of adjacent coarse  $\gamma'$ particles or M223C6 carbides when a specimen is direct-aged with a combination of slow cooling from solution treatment temperature to aging temperature. This serration leads to a change in grain boundary character as special boundary based on the crystallographic analysis demonstrating that the grain boundaries tend to serrate to have specific segments approaching to one {111} low-index plane at a boundary. The present study was initiated to determine the interdependence of the serration and HAZ property with a consideration of this serration as a potential for the use of a damage tolerant microstructure. It was found that the serrated grain boundaries suppress effectively grain coarsening, and are highly resistant to liquation cracking in HAZ due to their lower tendency to be wetted and penetrated by the liquid phase. These results reflect closely a significant decrease in interfacial energy as well as grain boundary configuration by the serration.

#### 9:20 AM

#### Characterisation of a Ni Alloy Fabricated by Using Selective Laser Melting: Xinhua Wu<sup>1</sup>; Fude Wang<sup>1</sup>; *Junfa Mei*<sup>1</sup>; <sup>1</sup>The University of Birmingham

Selective Laser Melting using laser powder bed has been used to manufacture 3D components from their CAD files and off-shelf commercial powders. This presentation highlights results of a systematic investigation with respect to the influence of laser processing parameters on dimensional accuracy, surface roughness, number of cracks, top surface concavity, micro and macrostructure and mechanical properties of samples of Hastelloy X manufactured using a laser powder bed facility. It has been found that the nominal laser power density is the dominant factor but the influence of scan spacing and scan speed can sometimes be significant. Density of >99.5% can be obtained using most conditions. Cracks are observed at corners of the samples. An optimised process window can be derived from the above systematic analysis under which the component can be built smoothly, with good surface finish and dimensional accuracy, consistent mechanical properties and the properties are comparable with that of the forged.

#### 9:35 AM Keynote

Novel Design Concept for Advanced Austenitic Heat Resistant Steels: Masao Takeyama<sup>1</sup>; <sup>1</sup>Tokyo Institute of Technology

Why there is no austenitic heat resistant steels strengthened by intermetallic phases? This is because of the formation of Fe,M Laves phase, since the Laves phase formed in heat resistant steels are in general believed to degrade the creep properties. However, this is not true and the Laves phases are rather promising for long-term creep rupture strength. In this talk, a novel design concept for development of a new class of austenitic heat resistant steels strengthened by Laves phases applicable for advanced ultra-super critical (A-USC) power plants is presented, based on the fundamental studies on phase equilibria between g-Fe and the Laves phases, precipitation kinetics, crystallography. A model steel of carbon free Fe-20Cr-30Ni-2Nb (at%) exhibits prolonged creep rupture strength superior to any of the currently existing commercial steels. This superior property is caused by precipitation of Fe,Nb Laves phase at the grain boundaries. The higher the grain-boundary area fraction of the Laves phases, the lower the creep rate, leading to longer rupture life. This strengthening method, named "Grainboundary precipitation strengthening", is very effective, particularly under low stress levels. The details of the morphology control of the Laves phase as well as the strengthening mechanism will be also given.

#### 9:55 AM

**Improving Creep Properties through Grain Boundary Engineering**: *Milo Kral*<sup>1</sup>; Dan Drabble<sup>1</sup>; <sup>1</sup>University of Canterbury

Grain boundary engineering is a promising methodology to improve the properties of many FCC metals and alloys. The aim of the present work was to investigate the feasibility of using grain boundary engineering to improve the high-temperature properties of an austenitic nickel-iron-chromium alloy. Samples of 800H (32%Ni-21%Cr-44%Fe with Al, Ti additions) were 'grain boundary engineered' using a range strain/anneal conditions and then characterized using Electron Backscatter Diffraction (EBSD) analysis. The various processing conditions provided samples with a range of grain size, grain boundary character and grain boundary connectivity. This work has culminated in correlations between the microstructural characteristics and high-temperature material properties such as steady-state creep rate and strength/ductility under typical service conditions.

#### 10:10 AM

High Temperature Mechanical Behavior of Fe-Base ODS Alloys: *Jinsung Jang*<sup>1</sup>; Sung-Soo Kim<sup>1</sup>; Chang Hee Han<sup>1</sup>; Song Nan Yin<sup>1</sup>; Woo-Gon Kim<sup>1</sup>; <sup>1</sup>Korea Atomic Energy Research Institute

Oxide dispersion strengthened (ODS) alloy is a good candidate alloy for the application to in-core components of advanced nuclear reactors due to its good resistance to neutron radiation and its excellent high temperature mechanical properties. 9 Cr and 12 Cr Fe-base ODS alloys are prepared by mechanical alloying of raw metal powders with yttrium oxide particles, hot isostatic pressing, and hot rolling. Besides impact and tensile tests, ODS alloy specimens were creep rupture tested at 700°C with various loads from 70 to 100 MPa. Impact absorption energy of 9 Cr ODS alloy at room temperature showed more or less isotropic behavior; absorption energy in transverse direction showed about 0.8 of that in longitudinal one. In case of 12 Cr ODS alloy specimen, however, the absorption energy in the transverse direction showed about one quarter of that in the longitudinal direction, indicating a severe anisotropic feature. Fracture surfaces and microstructures are investigated with respect to the mechanical behavior in the different directions, and the grain boundaries and dispered particles in the creep ruptured specimens are also examined with respect to the creep crack propagation.

#### 10:25 AM Tea Break



#### Symposium B: Advanced High Temperature Structural Materials: Thermal Stabilty and Creep

Friday AM	Room: 7
August 6, 2010	Location: Cairns Convention Centre

Session Chairs: Milo Kral, University of Canterbury; Jaimie Tiley, US Air Force

#### 11:00 AM

Phase-Field Simulation on Phase Transformation during Creep Deformation in Type 304 Steel: *Yuhki Tsukada*<sup>1</sup>; Atsuhiro Shiraki<sup>2</sup>; Yoshinori Murata<sup>1</sup>; Shigeru Takaya<sup>3</sup>; Toshiyuki Koyama<sup>4</sup>; Masahiko Morinaga<sup>1</sup>; <sup>1</sup>Nagoya University; <sup>2</sup>Kobe Steel, Ltd.; <sup>3</sup>Japan Atomic Energy Agency; <sup>4</sup>National Institute for Materials Science

Type 304 steel exhibits paramagnetism at room temperature as a metastable state. In creep tests at high temperatures, however, it was reported that magnetization corresponding to the ferromagnetism was detected due to the phase transformation from the fcc phase ( $\gamma$ ) to the bcc phase ( $\alpha$ ). This phenomenon is peculiar to gauge (stress induced) portion, and it implies that the magnetization is related to the creep damage. In this study, the mechanism of the phase transformation during creep was examined based on the TEM observations of the crept samples. And also, defect energies near  $M_{22}C_c$  carbide were estimated based on the micromechanics, and it was suggested that dislocation energy introduced during creep could be the main driving force for the precipitation of the ferromagnetic  $\alpha$  phase. On the basis of the experimental results mentioned above, the correlation of the dislocation energy density with the formation of the  $\alpha$  phase was examined by the phase-field method. In this simulation, simultaneous nucleation and growth of both  $M_{23}C_6$  carbide and the  $\alpha$  phase were reproduced. It was found that the small difference in the dislocation density led to the significant change in mole fraction of the  $\alpha$  phase formed in the steel.

#### 11:15 AM

Creep Behaviour and Microstructural Changes of Advanced Creep Resistant Steels after Long-Term Isothermal Ageing: Vaclav Sklenicka<sup>1</sup>; Kveta Kucharova<sup>1</sup>; Milan Svoboda<sup>1</sup>; Ales Kroupa<sup>1</sup>; J. Cmakal<sup>1</sup>; <sup>1</sup>Institute of Physics of Materials, Academy of Sciences of the Czech Republic

Creep behaviour and degradation of properties of creep resistant materials are phenomena of major practical relevance, often limiting the lives of components and structures designed to operate for long periods under stress at elevated and/or high temperatures. In this paper we will try to further clarify the creep-strength degradation of selected advanced creep resistant steels. In order to accelerate some microstructural changes and thus to simulate degradation processes in long-term service, isothermal ageing at 650°C for 10 000 h was applied to P91, P92 and T23 steels in their as-received states. The accelerated tensile creep tests were performed at temperature 600°C both in their as-received states and after long-term isothermal ageing, in an effort to obtain a more complete description of the role of microstructural stability of these steels. Creep tests were followed by microstructural investigations by means of electron microscopy and by the thermodynamic calculations. The applicability of the accelerated creep tests was verified by the theoretical modelling of the phase equilibria and basic analysis of diffusion kinetics at different temperatures. It was found that under restricted oxidation microstructural instability is the main detrimental process in the longterm degradation of the creep rupture strength of these steels.

#### 11:30 AM

Microstructure and High Temperature Strength in Fe<sub>3</sub>Al Base Alloys Containing Fine Carbide Particles: *Satoru Kobayashi*<sup>1</sup>; Ryo Makihara<sup>2</sup>; Takayuki Takasugi<sup>2</sup>; <sup>1</sup>Tohoku University; <sup>2</sup>Osaka Prefecture University

The precipitation of carbide particles and high temperature strength in Fe<sub>3</sub>Al base alloys containing Cr, Mo and C were investigated. Fe-27Al-0.6C-2.0Cr-Mo (at.%) were arc melted, warm rolled, heat treated at 1200°C in the alpha(A2) single-phase field and followed by annealing at temperatures between 1000°C and 700°C for 10 min - 10 h. Three types of carbide phases were precipitated during annealing: kappa-Fe<sub>3</sub>AlC (E21), M<sub>2</sub>C (B81) and M<sub>5</sub>C (Orthrhombic). The kappa phase was precipitated in grain interior (GI) as a needle shape and grain boundaries (GB) as a film-like morphology at a certain annealing condition. The M<sub>2</sub>C phase was finely distributed within GI and the formation kinetics was faster as Mo content of the alloy increased. The M<sub>3</sub>C phase was formed at slower rate compared with the other carbide phases. High temperature tensile properties will be tested to evaluate the role of GB precipitates and fine GI particles.

#### 11:45 AM

**Evaluation of Creep-Fatigue Crack Growth for Grade 91 Steel Wide Plate:** *Hyeong-Yeon Lee*<sup>1</sup>; Jong-Bum Kim<sup>1</sup>; Jae-Han Lee<sup>1</sup>; <sup>1</sup>Korea Atomic Energy Research Institute

An assessment of a creep-fatigue crack initiation and growth for Mod. 9Cr-1Mo steel wide plates with and without weldments have been carried out based on an extended French high temperature design code, RCC-MR A16 guide. The defect assessment guide of the A16 provides assessment procedures for a creep-fatigue crack initiation and growth for an austenitic stainless steel, but no guidelines are available yet for a Mod. 9Cr-1Mo steel. In this study, a  $\sigma d$  approach was extended to evaluate creep-fatigue crack initiations, and assessments of a creep-fatigue crack growth at defects of wide plates with and without welded joint have been extended based on the A16 guide for austenitic stainless steel. The crack behaviours at wide plates with and without welded joints are to be compared. The assessment results are to be compared as well with those of the structural test with the same specimens used in the assessments at the creep-fatigue test with one hour of hold time at 550°C.

#### 12:00 PM

## Effects of Long Term Aging on Creep Properties of HP Reformer Tubes: Karl Buchanan<sup>1</sup>; Milo Kral<sup>1</sup>; <sup>1</sup>University of Canterbury

The centrifugally cast HP series has become the dominant reformer tube material for the petrochemical industry. HP alloys with small additions of niobium and titanium are reported to have superior creep properties over standard HP alloys in accelerated creep testing. However, tubes removed after 3-5 years service typically exhibit lower remaining life than expected. The present work studies the effects of long-term laboratory aging on the creep performance of these alloys. HP-Niobium and HP-Micro alloys were aged un-stressed at 1000-1100°C for 500-10,000 hours. Both as-cast and aged samples were subjected to accelerated creep testing. Detailed characterization of the as-cast and aged samples was carried out using high resolution SEM and high resolution TEM. Attention was paid to the size and distribution of the niobium-titanium rich phases in each alloy.

#### 12:15 PM

Creep Crack Growth Rates on the Base and Welded Metals of Modified 9Cr-1Mo Steel: *Woo-Gon Kim*<sup>1</sup>; Jae-Young Park<sup>2</sup>; Song-Nan Yin<sup>1</sup>; Yong-Wan Kim<sup>1</sup>; Seon-Jin Kim<sup>2</sup>; <sup>1</sup>Korea Atomic Energy Research Institute; <sup>2</sup>Pukyong National University

This paper deals with the creep crack growth rates on the base and welded metals of modified 9Cr-1Mo steel, which are regarded as promising candidates for structural materials for Gen-IV reactors. Since their structures are designed for a lifetime of 60 years, their creep crack growth behavior should be considered for the weld region as well as the base region. To obtain mechanical properties concerning the base and welded metals, a series of creep and tensile tests were conducted at 600°C, and creep crack growth tests were also performed under different applied loads using 1/2" CT specimens at 600°C. Welded specimens were prepared by the Shielded Metal Arc Weld (SMAW) method. Their creep crack growth rates were calculated using the empirical equation of the da/dt vs. C\* parameter and compared. It appeared that, for a given value of C\*, the rate of creep propagation was about 2.0 times faster in the welded metal than the base metal. This reason is because the creep strain rate in the welded metal was fast when compared with that in the base metal. This result can be utilized for assessing the creep propagation on the base and welded metals for the modified 9Cr-1Mo steel.

#### 12:30 PM

Effects of Ferrite Content on the Tensile Strength and Impact Toughness of 2.25Cr-1Mo-0.25V Steels: *Tae Kyu Kim*<sup>1</sup>; Chang Hee Han<sup>1</sup>; Sung Ho Kim<sup>1</sup>; Hee Kyung Kwon<sup>2</sup>; Dong Jin Kim<sup>2</sup>; <sup>1</sup>Korea Atomic Energy Research Institute; <sup>2</sup>Doosan Heavy Industries & Construction Co. Ltd.

A 2.25Cr-1Mo steel is considered as a material for a heat-resistance pressurized vessel of IGCC plant in the future. In order to evaluate the effects of ferrite content on the tensile strength and impact toughness of this steel, several samples with a different area fraction of ferrite in the range from 0 to 80% were prepared by a control of cooling rate from an austenitization heat treatment at 930°C. The samples were then tempered at 690°C, followed by a heat treatment at 705°C for a simulation of post-weld heat treatments. The results of microstructural observation indicated that the ferrite was uniformly distributed in the banitic matrix. The results of tensile and impact toughness at -29°C were continuously degraded with increasing ferrite content. On the basis of these results, the dependence of mechanical properties on the ferrite content is discussed.



#### Symposium E: Solidification, Deformation and Related Processing: Solidification IV

Friday AMRoom: 2August 6, 2010Location: Cairns Convention Centre

Session Chair: Jishan Zhang, USTB

#### 8:30 AM Keynote

**Research on Foamability and Deformation of Al-Alloy Metallic Foam**: *Hur Bo Young*<sup>1</sup>; Jeong Seung Reung<sup>1</sup>; Li Yuxuan<sup>1</sup>; <sup>1</sup>I-Cube Center, ReCAPT, Gyeongsang National University

Aluminum alloy foams are well known as materials for absorbing energy such as sound, vibration and impact. Normally aluminum alloy foam is a cellular structure material and its representative characteristic is a very high porosity. It means that even if aluminum alloy foams are applied to industries component, the weight increase is a few. However, systematic research and further development are necessary to satisfy fully needs to safety of high-performance structure. Firstly, aluminum alloy foams were fabricated by casting method and then the pore structures such as cell size, cell wall thickness and porosity were investigated by using microscopy and image analyzer. Pore sizes were 1.5mm ~ 3.0mm and the porosities of the foams were 85~92%. Investigation of the aluminum alloy foam was conducted through the bending test and the properties which also including aluminum alloy foams were investigated through the compressive test. The values of the bending tests were increased and energy absorbing areas were also increased after applied aluminum alloy foams. The values of compressive tests were increased strength and also plateau areas were increased. This means that the amounts of absorbing energies were increased after applied aluminum alloy foams.

#### 8:50 AM

Study on Powder Metallurgical Preparation of NiCoMnIn Alloy Foam: *Gang Wang*<sup>1</sup>; XiaoMing Xiong<sup>1</sup>; Yandong Liu<sup>1</sup>; Chunyan Wang<sup>1</sup>; Yandong Wang<sup>1</sup>; Liang Zuo<sup>1</sup>; <sup>1</sup>Northeastern University

The magnetic shape-memory alloy NiCoMnIn shows, in monocrystalline form, a large reversible magnetic-field-induced strain (MFIS). But it is difficult to achieve the properties in polycrystalline NiCoMnIn alloys. The technique of powder metallurgical preparation of NiCoMnIn foam was studied to improve the properties of polycrystalline NiCoMnIn alloys in the present paper. We introduced a processing route including choosing appropriate space-holding fillers, sintering NiCoMnIn alloy and the filling agent with appropriate grain size. The sintering temperature and time and the optimum ratio of alloy to the filling agent were determined by analysis of the structure of sintered bulk foams. The yield strength of the alloy foam was lower by about an order of magnitude than the original alloy, which could influence very much on the stress-induced matensitic transformation or magnetic-field-induced martensitic transformation of NiCoMnIn alloy.

#### 9:05 AM

The Contribution of Diffusion Coefficient to the Eutectic Instability and Amorphous Phase Formation: *Nan Wang*<sup>1</sup>; Xiao Wang<sup>1</sup>; Wenjing Yao<sup>1</sup>; <sup>1</sup>Department of Applied Physics, Northwestern Polytechnical University

The diffusion coefficient D decides the diffusion length of solute boundary and plays a key role in the microstructure selection. As the growth velocity increases, the interface temperature becomes lower that reduces the diffusion coefficient, which then significantly decreases the diffusion length. When the length becomes the order of atomic distance, non-equilibrium effects become important, leading to either partitionless solidification or amorphorization. This paper examines quantitatively the contribution of diffusion coefficient to the eutectic instability and amorphorization ability. The maximum growth velocity  $V_{\text{max}}$  and the maximum undercooling  $\Delta T_{\text{max}}$  as functions of activation energy Q in strong liquids and those of fragility parameter B in fragile glass forming systems are deduced theoretically based on eutectic growth model by separating Q or B from D. It reveals that the larger the Q or B, the smaller the  $\Delta T_{max}$  and  $V_{max}$ , which shows the same tendency as experimental values in some Al-based alloys and glass formers. This indicates that it is the sluggish movement of atoms that makes the transition from eutectic to others structural morphologies, even to amorphous phase, occur at smaller interface growth velocity or undercooling, which is the main contribution of the diffusion coefficient to the amorphorization ability.

#### 9:20 AM

The Correlation between the Liquid Structure and the Solidification Microstructure of Sn-Cu Lead-Free Solders: *Xuemin Pan*<sup>1</sup>; <sup>1</sup>Dalian University of Technology

Eutectic Sn-Cu solder has been recommended as a promising lead-free solder, and has been widely used in wave soldering and flip chip applications During soldering process, the molten solder will react with the Cu substrate and surface finishes, and then intermetallic compounds (IMCs) will be formed at the interface. The rate of such interfacial reaction and the thickness of the interfacial IMC layer play a very important role on the performance of the packaging and on the reliability of the component in service. As the interfacial reaction occurs between molten solder and solid substrate, so the liquid structure of solder must has significant effect on the reaction and the formation of interfacial product. However, the work about liquid structure of molten solder hasn't been carried out yet. In this paper, for molten solder, the Ashcroft–Langreth structure factor S(Q) was gotten from the scattering intensity and the viscosity was measured using a torsional oscillation viscometer. The correlation-ship between the liquid structure and the viscosity was then discussed. The interfacial reaction between solders and Cu substrates was also analyzed.

#### 9:35 AM

The Character of Melt of A357 Alloy and Its Influences on the Solidification Microstructures: *Rong Zhang*<sup>1</sup>; Limin Zhang<sup>1</sup>; Zhihuai Yang<sup>2</sup>; <sup>1</sup>School of Science, Northwestern Polytechnical University; <sup>2</sup>Department of Physics, Baoji University Arts and Sciences

The relativity between the alloy melt state and solidification microstructure has been received great interesting recently. Some previous studies proved that control the state of alloy melt can improve the microstructure and properties. In this paper, the character of melt of A357 alloy between 700°C-1150°C was studied by using DSC. The result indicates that there are exothermic reactions over liquidus which correspond to dissolution of crystalline particle or cluster in the melt. In order to investigate the effects of character of melt on the microstructure, the alloys were first superheated to 720°C, 820°C and 920°C respectively with different character of melt, held for 20min, then the melts cooled to 720°C before quenching in Ga-In-Sn melt. The microstructure of samples was analyzed. The results show that with the increase of superheating temperature, the secondary dendrite arm spacing (SDAS) of a-Al is reduced, especially as the temperature increase from 820°C to 920°C, the SDAS decrease steeply. Meanwhile the superheating temperature also influences the eutectic silicon both in size and shape. When the temperature rises, eutectic silicon becomes finer and more uniformly dispersed instead of coarse platelike.

#### 9:50 AM

**Formation of Primary Intermetallic Compounds in Sn-Ag-Cu Alloys**: *Yoshiko Takamatsu*<sup>1</sup>; Hisao Esaka<sup>1</sup>; Kei Shinozuka<sup>1</sup>; <sup>1</sup>National Defense Academy

Sn-Ag-Cu alloys are considered one of the most favorable lead-free solder systems. In slowly-cooled eutectic Sn-Ag-Cu alloys, sometimes large primary Ag,Sn or Cu,Sn, intermetallic compounds (IMCs) form. These IMCs may affect the mechanical properties of solders. However, explanations for the formation of these IMCs are still not clear. This study deals with interrupted tests in order to clarify the nucleation of IMCs in the liquid phase. In this study, Sn-4.5Ag-0.8Cu and Sn-3.5Ag-1.5Cu alloys were prepared. According to thermodynamic calculation, Pandat, the equilibrium solidification paths are described as follows. Sn-4.5Ag-0.8Cu :L  $\rightarrow$  primary Ag<sub>3</sub>Sn  $\rightarrow$  binary eutectic (Ag<sub>3</sub>Sn + Sn)  $\rightarrow$  ternary eutectic.  $\cdot$  Sn-3.5Ag-1.5Cu :L  $\rightarrow$  primary Cu<sub>6</sub>Sn<sub>5</sub>  $\rightarrow$  binary eutectic (Cu<sub>6</sub>Sn<sub>5</sub> +Sn)→ ternary eutectic. The actual solidification process was different from the estimation from equilibrium phase diagram. In the case of Sn-4.5Ag-0.8Cu, only Ag<sub>3</sub>Sn grew as a primary phase in the liquid, while in the case of Sn-3.5Ag-1.5Cu, not only primary Cu<sub>6</sub>Sn<sub>5</sub> but also psudo-primary Ag<sub>3</sub>Sn grew in the liquid. Ag<sub>3</sub>Sn may nucleate easily in the liquid phase, but Cu<sub>6</sub>Sn<sub>5</sub> would not nucleate in the liquid.

#### 10:05 AM Tea Break



#### Symposium E: Solidification, Deformation and Related Processing: Solidification V

Friday AM	Room: 2	
August 6, 2010	Location:	Cairns Convention Centre

Session Chair: Arne Dahle, The University of Queensland

#### 11:00 AM

Researches on the Nucleation Behaviors of NH<sub>4</sub>Cl Crystal on Coarse Aluminum Surfaces: *Meng Wang*<sup>1</sup>; Ying Zhang<sup>1</sup>; Senfeng Zhu<sup>1</sup>; Xin Lin<sup>1</sup>; Weidong Huang<sup>1</sup>; <sup>1</sup>Northwestern Polytechnical University

Surfaces with different coarseness were prepared on aluminum sheets and the surfaces were exposed in the atmosphere for different time intervals. The wettability of NH4Cl-70wt%water solution on the surfaces was investigated, and the sheets were immerged into NH<sub>2</sub>Cl-70wt%water solution and undercooed to trigger the nucleation of NH4Cl crystals. Experimental results indicate that the surface wettability of aluminum surface which has a step-like structure shows a transformation from strong wettability to non-wettability during its exposure in the atmosphere, in the same time, nucleation density on the strongly wetting surface is significantly higher than that of the non-wetting surface. A hypothesis is proposed to explain the relationship between the wettability and the nucleation density on the coarse surfaces, which can be expressed as follows: the capillary effect helps the solution infiltrate into the rough structures of the surface with strong wettability and drive the air out of the surface structures, so nucleation is effectively promoted by the sharp corners and hollows of the coarse surface; conversely, the air bubbles trapped in the non-wetting surface structures decrease the number of the effective nucleation sites, and leads to a higher nucleation energy barrier, thus the nucleation density is decreased on the non-wetting surfaces.

#### 11:15 AM

#### Semisolid Sinter-Forging of Hyper-Eutectic Al-Si Alloy Using Rapid Resistance Heating: *Seijiro Maki*<sup>1</sup>; Masayuki Noda<sup>1</sup>; Eitoku Nakanishi<sup>1</sup>; <sup>1</sup>Mie University

In forming of hyper-eutectic Al-Si base alloys by ordinary casting, primary Si grains grow, which deteriorates the mechanical properties of the formed parts. This problem can be removed by application of sinter-forging. As for the sinter-forging, a good performance is expected by generation of liquid phase. Then, semisolid sinter-forging using rapid resistance heating is taken up. In this study, a green powder compact of hyper-eutectic Al-25 mass% Si alloy is rapidly resistance-heated into a semisolid state and then forged, and the performance is examined especially from the viewpoint of the conditions of the primary Si grains in the sinter-forgings in the relation to the liquid phase ratio in the processing. Through the experiments, the following features are revealed. (1) In the resistance heating of the green powder compact, the eutectic liquid first arises and, therefore, the primary Si grains is resultantly suppressed. (3) The liquid phase generated in the forging.

#### 11:30 AM

## Shear Deformation and Reynold's Dilatancy of Light Alloys during Solidification: *Bastian Meylan*<sup>1</sup>; Arne Dahle<sup>1</sup>; Christopher Gourlay<sup>2</sup>; <sup>1</sup>University of Queensland; <sup>2</sup>Department of Materials, Imperial College London

The deformation of partially solid alloys can lead to a variety of casting defects including hot tearing, microshrinkage porosity and many forms of macrosegregation. With the increased requirements of cast light metal components, there is a need for an improved understanding of the phenomena occurring during solidification. Recent work has shown that AZ91 behaves as a granular material at a solid fraction shortly after dendrite coherency and exhibits Reynold's dilatancy and strain localisation. This study shows the influence of grain size and dendritic morphology on the dilatancy of several aluminium alloys. The results are compared with the literature on granular materials mechanics where only a few studies have reported on the characteristics of the rheological behaviour of granular materials containing solid with complex shapes similar to the morphologies found in solidifying metallic suspensions.

#### 11:45 AM

The Effect of Constricting the Melt Flow on the Mechanical Properties of High Pressure Die Castings: *Dayalan Gunasegaram*<sup>1</sup>; Robert O'Donnell<sup>1</sup>; Michel Givord<sup>1</sup>; Barrie Finnin<sup>2</sup>; <sup>1</sup>CSIRO Light Metals Flagship; <sup>2</sup>CSIRO Materials Sciene & Engineering

The addition of a constriction in the melt flow path of high pressure die castings is discussed in terms of its influence on modifications to mechanical properties. It is shown through experimentation that, for the different constriction configurations and process parameters investigated, the ultimate tensile strength and elongation to fracture of as-cast tensile specimens increased whilst their 0.2% proof strength remained largely unchanged. Refinement of defect-forming inclusions and their more homogeneous dispersion are shown to be the reasons for the improved fracture properties. Mechanisms responsible for the refinement are proposed as (a) increased shear rates engineered by the constriction and (b) enhanced levels of turbulence resulting from melt accelerating through the constriction. These proposals are backed up with results from computational fluid dynamics simulations.

#### 12:00 PM

#### Solidification Behavior of Mg Alloys during Twin-Roll Casting: Jun Ho Bae<sup>1</sup>; D.-W Kim<sup>1</sup>; B.-C. Suh<sup>1</sup>; M.-S. Shim<sup>1</sup>; Nack J. Kim<sup>1</sup>; <sup>1</sup>POSTECH

The development of wrought Mg alloys, particularly in the sheet form, is essential to support the growing needs of the automotive industry for lightweight components. Recently, it has been shown that twin-roll casting (TRC) can produce low-cost, high-quality Mg alloy sheets that have comparable mechanical properties to those of conventional ingot cast Mg alloy sheets. However, only a few alloy systems have been fabricated by TRC so far. The difficulties come from the inherent characteristics of Mg alloys such as low specific heat and large freezing range, which can result in the formation of several casting defects, e.g., centerline and inverse segregations. Such defects in as-cast structure can cause serious problems in the mechanical properties of final products. In the present study, several Mg alloys were subjected to TRC and their solidification behavior has been investigated. The main processing variables are roll gap, roll velocity and melt temperature. In order to find the optimum casting conditions which reduce the segregation, solidification behavior during TRC is simulated and the results are compared with actual TRC. Effects of alloying elements and thermomechanical treatment on the microstructure and mechanical properties of the rolled sheets will also be discussed.

#### 12:15 PM

Mechanical Properties and Metallurgical Qualities of High Aluminum Content Magnesium Alloys Fabricated by Twin-Roll Casting: *Hisaki Watari*<sup>1</sup>; Yoshimasa Nishio<sup>1</sup>; Mayumi Suzuki<sup>2</sup>; Toshio Haga<sup>3</sup>; Nobuhiro Koga<sup>4</sup>; <sup>1</sup>Gunma University; <sup>2</sup>Tohoku University; <sup>3</sup>Osaka Institute of Technology; <sup>4</sup>Nippon Institute of Technology

This paper describes the twin-roll casting technology of magnesium alloys that contain relatively high weight ratios of aluminum, such as AZ81, AZ91, AZ101 and AZ111. The magnesium alloy sheets were cast by a twin roll caster to manufacture relatively high-strength Mg alloys with high aluminum content. The influences of such process parameters as roll materials, casting temperature, and roll speed were ascertained. A simple method of predicting the convection heat transfer coefficient between casting rolls and molten metal is introduced. The microstructures of cast magnesium allov sheets are microscopically observed to investigate the effects of roll-casting conditions on crystal growth in the cast products. It was found that Mg alloys with high aluminum content can be fabricated at a roll speed of 15m/ min with a horizontal-roll caster. The grain size of the manufactured wrought magnesium alloy sheet was about 30 micrometers. It was also demonstrated that high aluminium content Mg alloys such as AZ81, AZ91, AZ101 could be hot rolled when in choosing an appropriate hot rolling temperature. A warm deep drawing test of the cast magnesium sheets after being hot rolled was performed to demonstrate the formability of the magnesium alloy sheets produced by a roll strip casting process.

#### 12:30 PM

# Strength and Conductivity of Deformed Cu-Fe Composites after Solidified with a Horizontal Magnetic Field: *Xiaowei Zuo*<sup>1</sup>; Engang Wang<sup>1</sup>; Lei Qu<sup>1</sup>; Lin Zhang<sup>1</sup>; Jicheng He<sup>1</sup>; <sup>1</sup>Key Laboratory for Electromagnetic Processing of Materials, Ministry of Education, Northeastern University, China

In this paper, the Cu-Fe alloys are fabricated with and without a 1.0 Tesla horizontal magnetic field and they are drawn to composite wires under different drawing ratios, then their strength and conductivity are investigated. The results show that, when the drawing ratio is small, the strength of the drawn Cu-Fe composites pre-solidified under a horizontal magnetic field is lower than that without a magnetic field, which might be resulted by the coarsened solidification microstructure induced by the injected magnetic field. However, the strength of the drawn Cu-Fe composite with a 1.0 Tesla horizontal magnetic field is quickly



increased with the increasing of the Fe content and the drawing ratio in compared with the case without the magnetic field. A relationship is fitted to predict the influence of the magnetic field on the strength of the drawn Cu-Fe composites wires. The conductivity of the drawn Cu-Fe composite wires is decreased with the increasing of the Fe content. However, the difference of the conductivity between with and without the magnetic field is small. Therefore, by way of a larger deformation of the solidified Cu-Fe alloy with a magnetic field, higher strength of Cu-Fe composites with high Fe contents can be fabricated.

#### Symposium I: Biomaterials, Smart Materials and Structures: Fabrication and Testing

riday AM	Room: 3	
ugust 6, 2010	Location:	Cairns Convention Centre

Session Chairs: Qing Li, University of Sydney; Ho Yeon Song, Soonchunhyang University

#### 8:30 AM Keynote

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Failure of Protein Materials in Extreme Conditions and Disease: Markus Buehler<sup>1</sup>; <sup>1</sup>Massachusetts Institute of Technology

Biological protein materials feature hierarchical structures, ranging through the atomistic, molecular to macroscopic scales, forming functional biological tissues as diverse as spider silk, tendon, bone, skin, hair or cells. Here I will present computational studies, focused on how protein materials deform and fail due to extreme mechanical conditions, disease and injuries. Based on a multiscale atomistic simulation approach that explicitly considers the architecture of proteins including at the chemistry level, we developed predictive models of protein materials, validated through quantitative comparison with experimental results. This bottom-up approach enables us to extract fundamental physical concepts that control the properties of protein materials. I will present studies of several classes of protein materials, including cellular alpha-helical protein networks, beta-sheet structures as found in spider silk and amyloids, and collagenous tissues found in bone. Materials failure in the context of genetic diseases will be discussed.

#### 8:50 AM Keynote

**Evaluation and Control of Crystallographic Alignment of Biological Apatite Crystallites in Bones**: *Takayoshi Nakano*<sup>1</sup>; Takuya Ishimoto<sup>1</sup>; Jee-Wook Lee<sup>1</sup>; Sayaka Miyabe<sup>1</sup>; Naoko Ikeo<sup>1</sup>; Hidetsugu Fukuda<sup>1</sup>; <sup>1</sup>Osaka University

Our group is focusing on preferential degree of biological apatite c-axis, which is an impotant bone quality parameter in intact, pathological and regenerated bones1, 2). Preferential degree of BAp c-axis strongly depends on the bone position, bone growth, degree of pathology or regeneration, bone turnover rate, in vivo stress distribution, activity of bone cells, gene defect, etc. We are also trying to challenge clarification of formation mechanism of the BAp preferential alignment and finally to control the degree of BAp orientation based on both self organaization in vivo and artifical technique in vitro. The BAp orientation was finally concluded to be one of the most important indices to evaluate bone microstructure and to control the related mechanical function in bone. 1) T. Nakano, K. Kaibara, Y. Tabata et al.: Unique alignment and texture of biological apatite crystallites in typical calcified tissues analyzed by microbeam X-ray diffractometer system.

#### 9:10 AM

Investigation of Biomimetic Apatite Growth on DLC-ZrO<sub>2</sub> Thin Films Prepared by MOCVD: *Md Sadequl Amin*<sup>1</sup>; Lakshman Randeniya<sup>1</sup>; Avi Bendavid<sup>1</sup>; Phil Martin<sup>1</sup>; Edward Preston<sup>1</sup>; <sup>1</sup>CSIRO Materials Science & Engineering

Thin films of diamond-like carbon (DLC) containing zirconium dioxide (DLC-ZrO<sub>2</sub>) have been deposited onto conducting (100) silicon wafer substrates using a pulsed direct-current metal-organic chemical vapour deposition (MOCVD) technique. Zirconium t-butoxide and methane were used as precursors. DLC-ZrO2 thin films were immersed in simulated body fluid (SBF), which has ion concentrations nearly equal to human blood plasma at 37°C. The formation of apatite as a function of time was determined using a number of characterization techniques including XPS, FTIR, XRD, SEM and EDX. The XPS results confirmed the presence of calcium and phosphorus on the DLC-ZrO, film surfaces after immersion in SBF at 37°C. FTIR and XRD results confirmed that biomimetic apatite was formed on DLC-ZrO, as an amorphous film. SEM micrographs showed that although only a very thin apatite film was formed on the DLC-ZrO2 film surface after 4 weeks, a much larger growth of biomimetic apatite has occurred after 12 weeks of immersion. EDX results also confirmed that Ca and P were deposited on the film surface. The results suggest that these materials have the potential to be used as surface coatings for orthopaedic implants.

#### 9:25 AM

Fabrication of Bio-Mimetic Artificial Bone by Electrospun PMMA/HAp-Collagen Tubular in Bone Cement Wrapping: *Yang-hee Kim*<sup>1</sup>; Swapan Kumar Sarkar<sup>1</sup>; Hae-in Lee<sup>1</sup>; Ho-yeon Song<sup>2</sup>; Byong-Taek Lee<sup>2</sup>; <sup>1</sup>Department of Biomedical Engineering and Materials, School of Medicine, Soonchunhyang University; <sup>2</sup>Department of Immunology, School of Medicine, Soonchunhyang University

Collagen is a natural extracellular matrix (ECM) component of many tissues such as bone and other connective tissues. Calcium phosphate such as HAp, BCP are similar with human nature bone. To fabrication of small artificial bone, HAp-collagen and BCP was used. Channeled porous BCP cylindrical perform with a characteristic 7 cell morphology was fabricated by multi-pass extrusion process. The 1.5 mm diameter cylinders were arranged together to form a boney perform and was joined together by electrospinning of collagen solution. Electrospinning technique can easily fabricate nano- and micro fibrous structure. The fabricated collagen bound BCP channeled body mimics like a small bone with similar boney architecture. For improvement of mechanical properties, HAp was contained in the collagen solutions. Depending on amount of HAp, mechanical properties of electrospun was evaluated. The mechanical properties of the fabricated bone along with the detailed microstructure were investigated. In-vitro investigation was performed to evaluate the biocompatibility of the artificial bone with fibroblast–like cells (L-929) and osteoblast-like cells (MG-63).

#### 9:40 AM

Fabrication of Bone Substitutes by Sponge Replica Method: *Minsung Kim*<sup>1</sup>; Dong-Woo Jang<sup>1</sup>; Young-Ki Min<sup>1</sup>; Hun-Mo Yang<sup>1</sup>; Ho-Yeon Song<sup>1</sup>; Byong-Taek Lee<sup>1</sup>; <sup>1</sup>SoonChunHyang University

Calcium phosphate bone substitutes are widely used for providing support to in-growth of hard tissue in dental, orthopedic, and various medical applications. Recently, researches of bone substitutes, which have interconnected open pore structure, have been focused on improving their mechanical properties and modifying their surface with proteins such as collagen or bone morphogenetic protein for early bone formation. Especially, it is highly required to develop functional gradient structured bone substitute which is available for controlling the bioresoption rate. Porous BCP scaffold was fabricated by the sponge replica method using PU sponge. After 3 times of dip coating and the subsequent oven drying, burning out and microwave sintering was carried out. Several approaches were attempted to fabricate functional gradient scaffold. HAp was synthesized using sol-gel process, and infiltrated into the hollow space which formed after burning out of PU sponge. Polycaprolactone was coated on the sintered HAp-BCP scaffold. X-ray diffraction analysis was performed to characterize the phase identification of the BCP scaffold. Microstructures of the composites were observed using scanning electron microscopy. Compressive strength was compared depending on the number of layer.

#### 9:55 AM

**Evaluation of Mechanical Properties of Regenerated Bone by Nanoindentation Technique**: *Takuya Ishimoto*<sup>1</sup>; Takayoshi Nakano<sup>1</sup>; <sup>1</sup>Osaka University

To evaluate intrinsic material properties of regenerated bone is important to clarify the mechanical performance in the regenerated portion. Generally, the regenerated bone tissue is inhomogeneous in its size and morphology, so it is difficult to precisely elucidate material properties by conducting conventional mechanical tests such as compressive test, bending test, etc. Nanoindentation technique has been used to evaluate material properties of small or microstructured materials because it is not necessarily to require a large and welldesigned specimen. Thus, nanoindentation technique may be a powerful tool for evaluation of material properties in the regenerated bone. In this study, nanoindentation technique was applied for assessment of Young's modulus, hardness and viscoelasticity on intact and newly-regenerated rabbit long bones. Both intact and regenerated long bones exhibited the remarkable viscoelasticity as a creep deformation during the nanoindentation procedure, and the degree of creep deformation was significantly high in the regenerated bone. Moreover, the regenerated bone remarkably exhibited lower Young's modulus and hardness than the intact bone. The regenerated long bone possesses the impaired mechanical properties, which may be due to the difference in nano-organization of the collagen fibers and mineral crystals which are the main ingredients of bone tissue, from the intact bone.

#### 10:10 AM

#### Fabrication of Bio-mimetic Fibrillar Adhesives Using Porous Template Method: Aravindaraj G Kannan<sup>1</sup>; Hugh Brown<sup>1</sup>; <sup>1</sup>University of Wollongong

The fibrillar adhesive system on a Gecko's foot consists of a soft elastic layer made up of fibers in a hierarchical manner, which conforms to the surface roughness resulting in high adhesion by surface forces. Such dry adhesives have been of great interest due to their ability to repeatedly attach to a wide range of surfaces and their ability to repel dirt and self-clean. Fabrication of hierarchical



synthetic fibrillar adhesives to mimic this behavior involves careful optimization of several structural features such as fibrillar size, aspect ratio, tilt angle, tip shape and hierarchy. So far, the fabrication process has mainly involved complex lithographic techniques, and nanomolding techniques, which have limited possibilities in their areal size coverage as well as requiring an expensive and sophisticated process. In this work, we have used simple dissolvable porous templates for geometric confinement of micro-phase separated block copolymers via capillary action, followed by ultraviolet (UV)-ozone etching for hierarchical fiber fabrication. The formed hierarchical structure has been characterized using scanning electron microscopy (SEM) and atomic force microscopy (AFM). Also, the adhesion measurement of the fabricated structures using micro-mechanical adhesion techniques will be presented.

#### 10:25 AM

#### Fabrication of Artificial Bone by the Combination of Electrospinning, Extrusion and Slurry Processes: *Hiep Nguyen*<sup>1</sup>; Byong-Taek Lee<sup>1</sup>; <sup>1</sup>Soonchunhyang University

The aim of this work, we focus on manufacture of artificial metacarpus for medical replacement base on combination of individual advantage properties of ceramic and polymer. Here, periosteum was fabricated by HAp-ZrO<sub>2</sub> frame while compact bone was fabricating from multi-layer PEEK/collagen electro-spun loaded BCP. Spongy bone is formed from PCL/PLGA spongy coated collagen and hyapluronic acid. Biocompatibility of periosteum and compact bone layer are evaluated base on osteoblast cells through spreading, proliferation (SEM (Scanning Electron Microscope) observation and confocal image), cytotoxicity and cell viability (MTT assay) while ESCs (Embryonic Stem Cells) responded to spongy bone-like. Mechanical properties were evaluated by pending strength, fracture surface, stiffness.

#### 10:40 AM Break

#### Symposium I: Biomaterials, Smart Materials and Structures: Biological Test of Biomaterials

Friday AM	Room: 3	
August 6, 2010	Location:	Cairns Convention Centre

Session Chairs: Markus Buehler, Massachusetts Institute of Technology; Takayoshi Nakano, Osaka University

#### 11:00 AM Keynote

In Vitro and In Vivo Evaluation of Calcium Phosphate Bone Graft Substitutes: *Ho Yeon Song*<sup>1</sup>; Young-Hee Kim<sup>1</sup>; Jyoti M. Anirban<sup>1</sup>; In-Seon Byun<sup>1</sup>; Kyung-A Kwak<sup>1</sup>; Byong-Taek Lee<sup>1</sup>; <sup>1</sup>Soonchunhyang University

Calcium phosphate ceramics such as ß-tricalcium phosphate, bicalcium phosphate and hydroxyapatite have been used as a bone graft biomaterial because of their good biocompatibility and similarity of chemical composition to natural bones. To increase the mechanical and osteo-inductive properties, the granule and spongy type porous bone graft substitutes were prepared by fibrous monolithic process and polyurethane foam replica method, respectively. The pore sizes obtained using this approach ranged between 250-300 and 100-600  $\mu m,$ respectively, which is ideal for cellular proliferation. The cytotoxicity, cellular proliferation, differentiation and ECM deposition on the bone graft substitutes were observed by SEM and confocal microscopy. Moreover, the scaffolds were implanted in the rabbit femur. New bone formation and biodegradation of bone graft were observed through follow-up X-ray, micro-CT analysis and histological findings. Six months after implantation, we observed that new bone formation induced by bone graft substitute was more than two times active than control group. From the results, the granule and spongy type of porous calcium phosphate bone graft substitutes showed excellent biocompatibility and osteoinduction.

#### 11:20 AM

Mechanics in Multi–Length–Scale Bone Structure – An Investigation Using High-Energy X-Rays: Mark Hoffman<sup>1</sup>; *Ruiping Hoo*<sup>1</sup>; John Daniels<sup>1</sup>; John Dunlop<sup>2</sup>; Peter Fratzl<sup>2</sup>; <sup>1</sup>The University of New South Wales; <sup>2</sup>Max–Planck Institute of Colloids and Interfaces

Cortical bone has a unique multi–length–scale structure. The assembly of two basic components: the mineral crystals and the collagen fibrils at the nano–scale, a layered lamellar structure, and the Haversian system at the micro–scale each act to give a stiff and tough structure at the macro length-scale. High-energy X-rays coupling wide-angle X-ray diffraction (WAXD) and small-angle X-ray scattering (SAXS) techniques, together with in-situ tensile testing were used to investigate the deformation and failure mechanisms of the multi-scale bone structure. Strains at three material levels, at the bone tissue level and at the nanometer-level (the collagen fibrils and mineral crystal lattice) were measured using digital image correlation method, SAXS and WAXD, respectively. These methods have high strain resolution, while giving texture information. The X-ray exposure time is short which allows continuous monitoring of the entire deformation process. Our experiment results show that at the nanometre–length-scale, the staggered mineralized fibril bone structure is dominant in the elastic regime, analogous to an iso–strain composite model. At the micro–scale, collagen–fibril based bridging, rough fracture surface, and non–linear crack paths highlight the role of microstructure in toughening the macroscopic structure in the post–yielding regime of bone.

#### 11:35 AM

Tunable Antibacterial Coatings that Support Mammalian Cell Growth Pacific: Krasimir Vasilev<sup>1</sup>; <sup>1</sup>University of South Australia

Bacterial infections present an enormous problem causing human suffering and cost burdens to healthcare systems worldwide. This work presents novel tunable antibacterial coatings which completely inhibit bacterial colonization by both gram positive and gram negative bacteria, but allow normal adhesion and spreading of osteoblastic cells. The coatings are based on amine plasma polymer films loaded with silver nanoparticles via silver ion in-diffusion followed by reduction. The process of plasma polymerization makes the coatings directly applicable to many biomedical devices. The procedure for loading silver nanoparticles allows flexible control over the amount of loaded silver nanoparticles. The release of silver ions from the coatings is efficiently controlled by applying an additional plasma polymer film of predetermined thickness. This work demonstrates that via careful material design it is possible to generate surfaces inhibiting bacterial colonization but still allowing attachment of mammalian cells. The coating presented in this work may open new horizons for the design of a next generation of antibacterial coatings.

#### 11:50 AM

Kinetic and Microstructural Features of Demineralized and Deproteinated Bone: Po-Yu Chen<sup>1</sup>; Ekaterina Novitskaya<sup>1</sup>; Ana Castro-Cesena<sup>2</sup>; Gustavo Hirata<sup>3</sup>; *Joanna McKittrick*<sup>1</sup>; <sup>1</sup>UC San Diego; <sup>2</sup>Centro de Investigacion Científica y de Educación Superior de Ensenada; <sup>3</sup>Centro de Investigación Científica y de Educación Superior de Ensenada

Bone is a hierarchically structured composite consisting of a protein phase (type-I collagen) and a mineral phase (carbonated hydroxyapatite). Mineralized collagen fibrils comprised of nano-sized collagen molecules and mineral platelets (~ 2-4 nm in thickness) are arranged in osteons in compact bone and a lamellar structure in the cancellous bone. We investigated the structural and mechanical properties of the mineral and protein phases in compact and cancellous bone by demineralization and deproteination. Structural features of demineralized, deproteinated, and untreated samples at different hierarchical levels were characterized by micro-computed tomography (CT), x-ray diffraction, optical microscopy, SEM, TEM and TEM tomography. Both the deminerlized and deproteinated bone samples appeared identical at macro-scaleCompression tests were performed on treated and untreated bone. Kinetic parameters of demineralization were determined. This research is supported by the National Science Foundation grant DMR 0510138.

#### 12:05 PM

Creating Biomaterials Inspired by the Microstructure of Cuttlebone: Joseph Cadman<sup>1</sup>; Yuhang Chen<sup>1</sup>; Shiwei Zhou<sup>1</sup>; Qing Li<sup>1</sup>; <sup>1</sup>University of Sydney

Cuttlebone is a natural material possessing both high compressive strength and high porosity – a combination holding significant potential in the materials industry. A method for designing biomaterials mimicking the properties of cuttlebone is developed. The microstructure of cuttlebone is investigated using Scanning Electron Microscopy (SEM). A graded aspect ratio of the base cells between layers is evident in some samples. A method for designing graded biomaterials mimicking this cuttlebone microstructure is developed. Simplified 3D biomaterial samples are created using CAD software. These biomaterials are fabricated using a stereolithographic apparatus (SLA). The homogenisation technique is used to evaluate the mechanical properties of the original cuttlebone sample and the fabricated biomaterial sample. Good agreement is found between the Young's moduli of corresponding layers. However, it is inconclusive whether the Young's moduli have a proportional relationship to the aspect ratio of the base cell at this stage of the study.

#### 12:20 PM

Biomimic Artificial Cortical Bone with Aligned Microstructure by the Combination of Multi-Extrusion and Rolling Processes: *Dong-Woo Jang*<sup>1</sup>; Swapan Sakar<sup>1</sup>; Minsung Kim<sup>1</sup>; Yong-Ki Min<sup>1</sup>; Ho-Yeon Song<sup>1</sup>; Byong-Taek Lee<sup>1</sup>; <sup>1</sup>SoonChunHyang University

As the increasing of human life time as well as increasing of industrial and traffic accidents, the hand tissues like artificial bone substitutes have been widely

# Fri. AM



used in the clinical surgery. Recently, calcium phosphate based bioceramics have become interesting materials for biomedical applications due to their excellent biocompatibility and bioactivity. However, Ca/P based bioceramics have lower fracture toughness than human bone. In this work, artificial cortical which had osteon mimic structure bone was fabricated to improve fracture toughness using the combination of fibrous monolithic and rolling processes. BCP and graphite powders were mixed with ethylene vinyl acetate and stearic acid using shear mixer and then, extruded to make filaments by the extrusion process, separately. BCP sheet was prepared by rolling process. BCP filaments and carbon filaments were arranged one by one on the BCP sheet and then, subsequently rolled to make cortical bone structure. Burning out and sintering processes were performed to remove the organic binder and graphite as well as densification. Material properties such as relative density, bending strength, hardness, and elastic modulus were measured. X-ray diffraction analysis was performed to characterize the phase identification of the composite. Microstructures of the composites were observed using scanning electron microscopy.

#### 12:35 PM

Quantity and Quality of Regenerated Bone in Grooves Aligned at Different Angles from the Implant Surface: *Yoshihiro Noyama*<sup>1</sup>; Takuya Ishimoto<sup>1</sup>; Koichi Kuramoto<sup>2</sup>; Takashi Sakai<sup>3</sup>; Hideki Yoshikawa<sup>3</sup>; Takayoshi Nakano<sup>1</sup>; <sup>1</sup>Division of Materials and Manufacturing Science, Graduate School of Engineering, Osaka University; <sup>2</sup>Nakashima Medical Co., Ltd.; <sup>3</sup>Department of Orthopedic Surgery, Osaka University Medical School

Quantity and quality of regenerated bone depend strongly on the principal stress direction in vivo, so in vivo stress distribution near bone implants should be optimized based on the shape of interface between implant and bone tissue. Since preferential alignment of the biological apatite (BAp) c-axis closely related to the stress distribution, direction in principal stress should be matched toward the grooved direction on the implant surface. Hip implants with grooves introduced by different angles from the surface were prepared and inserted to optimize the grooved angle to the implant surface by using 2 year-old beagles. As a result, bone formation and degree of preferential alignment of biological apatite c-axis as a bone quality parameter in grooves on the implant surface strongly depends on the grooved angle to the principal stress vector and the grooves parallel to the principal stress direction and the preferred direction of the apatite c-axis was approximately parallel to the grooved direction in the groove where the new bone was preferentially produced.

#### Symposium J: Materials Characterisation and Evaluation: Nanomaterials and Nanotechnology

Friday AM August 6, 2010 Room: 1 Location: Cairns Convention Centre

Session Chair: Julie Cairney, The University of Sydney

#### 8:30 AM Keynote

Dynamic Atomic Mechanisms of Plasticity of Metallic Nanowires and Nano Crystalline Ultra-Thin Films: Xiaodong Han<sup>1</sup>; Ze Zhang<sup>1</sup>; <sup>1</sup>Beijing University of Technology

The strength of metallic materials can be aggressively enhanced by reducing the grain size until it reaches nano-scale. Recent computer simulations revealed novel plastic deformation mechanisms when the extreme high stress exceeded the elastic limits of some nanowires. However, these new plastic deformation mechanisms have rarely been clearly demonstrated in experiments. Here, we use our recently-developed in situ transmission-electron-microscopy techniques, reveal that the FCC structured Ni nanowires possess ultra-high strength post a super-elastic deformation process. Full and partial dislocations can be emitted from not only the twinning boundaries, but also inside grains. These dislocation activities are functions of twinning thickness from 2 to 10 nm. Low and high angle grain boundaries were formed through bending force by both of tensile and compressive stresses and the process were captured in situ at atomic scale. Our results provide a novel and direct atomic scenario of high angle grain boundary formation process in nano metallic materials. Though these results were discovered in Ni nanowires, we speculate that it should be in general applicable to the metallic alloys with high stacking fault energy such as Ni with nano-scale deformation regions. We also expand the deformation techniques to several FCCstructured metallic nano thin films.

#### 8:50 AM Keynote

**Transmission Electron Microscopy of Aluminum Materials**: *Jianghua Chen*<sup>1</sup>; <sup>1</sup>Hunan University

It has been known for more than 50 years that AlMgSi(Cu) alloys are mostly strengthened when small needle-like precipitates are formed. These nanoprecipitates are called in the literature either as GP(I) and GP(II) zones, or as pre-B" and B" phases, or even as Si/Mg co-clusters and GP zones in the early stages. The ambiguity in naming reflects that accurate knowledge about these hardening precipitates has not yet been established. Here we use through-focus exit-wavefunction reconstruction to image the atomic structure of the growing early-stage needles. From a recorded through-focus series of 20 HRTEM images, we retrieved the electron wavefunction at the exit-plane of the specimen. The phase of the exit-wavefunction is then displayed as an atomic-resolution image of the specimen. The obtained atomic-resolution image of a nanoprecipitate clearly demonstrates its monoclinic lattice. All the atomic columns are clearly resolved as bright dots. The present work explains how the hardening nanoprecipitates in AlMgSi alloys are different and why they show the same needle-like morphology and the same monoclinic lattice. It is also shown that the Cu-containing particles, as the precursors of the so-called Q phase can be formed in the early-stage precipitation process of AlMgSi(Cu) alloys.

#### 9:10 AM Invited

Nanoindentation of 1D and 2D Nanostructural Materials: Han Huang<sup>1</sup>; <sup>1</sup>The University of Queensland

The characterization of mechanical properties of 1D and 2D nanostructural materials is extremely challenging due to their small feature sizes. This paper reports the nanoindentation of nanowire (1D) and thin film (2D) materials. The nanoindentation of tungsten microwhiskers showed that the average hardness of 1D single crystal tungsten is considerably higher than that of the bulk single crystal tungsten. The significant increase in hardness could be due to the lacking of dislocation avalanche that is the typical yielding characteristic of the bulk tungsten. The elastic modulus measured is much lower than that reported for the bulk tungsten. Surface roughness of the specimen and its resin support might be responsible for this discrepancy. The nanoindentation of 2D materials focused on the mechanical property testing of silicon nitride thin films. Simple equations are proposed for determining elastic modulus and hardness of thin films on substrates from nanoindentation experiments. An empirical formulation relates the modulus E and hardness H of the film/substrate bilayer to corresponding material properties of the constituent materials via a power-law relation. Geometrical dependence of E and H is wholly contained in the power-law exponents, expressed as sigmoidal functions of indenter penetration relative to film thickness.

#### 9:25 AM

**Preparation of Highly Crystalline Mesoporous TiO**<sub>2</sub> **by Using Carbon to Protect the Pore Structure at Elevated Temperature**: Yulan Ding<sup>1</sup>; *Xiaohua Lu*<sup>1</sup>; Wei Zhuang<sup>1</sup>; Linghong Lu<sup>1</sup>; <sup>1</sup>State Key Laboratory of Materials-Oriented Chemical Engineering, Nanjing University of Technology

Mesoporous TiO<sub>2</sub> with highly crystalline pore wall has an enormous potential for applications in photocatalysis, catalyst support, drug delivery, etc. Previous studies in our group have developed a series of mesoporous TiO, from a potassium dititanate whisker precursor by ion exchange and thermal treatment. However, with the sintering temperature increasing, the crystallinity of the pore wall increases while the mesopore structure tends to collapse. In this work, we presented a novel strategy for creating highly crystalline mesoporous TiO, by using carbon to protect the pore structure at elevated temperature. In the preparation, furfuryl alcohol was used as the carbon source to be filled into the mesopore of initial TiO<sub>2</sub> that calcined at 500°C, and then the composite was calcined at 800°C under N<sub>2</sub> atmosphere. The highly crystalline mesoporous TiO<sub>2</sub> (MT800) was finally obtained by removing carbon. The structural characterizations indicated that MT800 has highly crystalline anatase phase and processes Brunauer-Emmett-Teller (BET) surface area of 50 m<sup>2</sup>/g. The photocatalysis performance was evaluated by organic degradation and the result showed that MT800 has superior photocatalysis activity than the TiO<sub>2</sub> (T800) calcined at 800°C without carbon, because of the synergies of crystallinity and mesostructure.

#### 9:40 AM

**Influence of Metal Ions on the Synthesis of Silver Nanoparticles**: *Xuchuan Jiang*<sup>1</sup>; Aibing Yu<sup>1</sup>; <sup>1</sup>University of New South Wales

Silver nanoparticles have shown considerable interest because of their specific/ unique optical properties and potential applications in many areas such as optical probes, and surface enhanced Raman Spectrum. This study presents the synthesis of silver nanoplates through a synergetic hydrochemical reduction process using 3 reducing agents (NaBH<sub>4</sub>, citric acid, and L-ascorbic acid) at room temperature; as well as the influence of metal ions on the formation and growth of silver nanoparticles. The particle characteristics including shape, size, distribution, and structure have been characterized by various experimental techniques (e.g.,



TEM, AFM, and UV-vis). The possible surface-adsorption or lattice modification mechanism of the effect of metal ions on silver nanoparticles is discussed. The findings will be useful for shape-controlled synthesis of silver and other metal nanostructures for desired optical properties.

#### 9:55 AM

Nanoindentation Characterization of Intermetallics Formed at the Lead-Free Solder/Cu Substrate Interface: *Hideaki Tsukamoto*<sup>1</sup>; Zhigang Dong<sup>1</sup>; Han Huang<sup>1</sup>; Tetsuro Nishimura<sup>2</sup>; Kazuhiro Nogita<sup>1</sup>; <sup>1</sup>The University of Queensland; <sup>2</sup>Nihon Superior Co. Ltd.

The intermetallics of  $Cu_6Sn_5$  are formed at the Sn-based solder/ Cu substrate interface, which play a significant role in solder joint reliability. The evaluation of the mechanical properties of the interface  $Cu_6Sn_5$ , which normally have a thickness of several micrometers, is essential to understand the mechanical performance and structural integrity of the solder joints. In this study, the interface  $Cu_6Sn_5$  and  $(Cu,Ni)_6Sn_5$  formed in Sn-Cu and Sn-Cu-Ni ball grid array (BGA) joints were investigated using nanoindentation. The results demonstrated that the elastic modulus and hardness of the  $(Cu,Ni)_6Sn_5$  were slightly higher than the respective values of the  $Cu_6Sn_5$  in all the reflowed samples. The strain rate sensitivity and the activation volume of these intermetallics were affected by the reflow times and load conditions. The effect of the Ni content in  $(Cu,Ni)_6Sn_5$  on the mechanical properties was also addressed. The creep behaviour of the intermetallics was discussed in detail based on the indentation load-displacement curves.

#### 10:10 AM

**Post-Hydrogen Permeation Characterization of V-Based Crystalline Alloy Membranes**: *Guang Sheng Song*<sup>1</sup>; Michael Dolan<sup>1</sup>; Daniel Liang<sup>1</sup>; Michael Kellam<sup>1</sup>; <sup>1</sup>CSIRO

Hydrogen-selective membranes have the potential to facilitate the generation of pure hydrogen for use in fuel cells. Vanadium (V) alloys are attractive membrane materials due to their low cost and high hydrogen permeability as compared to Palladium(Pd) alloys. However, surface preparation, and in particular hydrogen embrittlement, are research challenges facing the practical application of these vanadium allovs. These two issues are addressed by surface characterization and hydrogen induced cracking (HIC) analysis in order to produce an improved Vbased alloy membrane with optimum surface characteristics and strong resistance to hydrogen embrittlement. Composition and phase analysis have been carried out on the surface of the V-alloy membranes to identify the true causes leading to Pd-coating delamination and poor permeability performance. Metallographic and fractographic studies and X-ray radiographic examinations have been carried out on the failed membrane samples caused by HIC. In all the failed membrane samples, hydrogen induced cracks were found to occur on transgranular cleavage planes. Major cracks were observed to go through the voids in the propagation direction, while minor cracks were found to start from the voids. In light of these results obtained for the failed membranes, a new failure mechanism has been proposed for hydrogen embrittlement.

10:25 AM Tea Break

#### Symposium J: Materials Characterisation and Evaluation: Other Novel Characterization Methods

Friday AMRoom: 1August 6, 2010Location:

Location: Cairns Convention Centre

Session Chairs: Xiaodong Han, Beijing University of Technology; Jianghua Chen, Hunan University

#### 11:00 AM

Effect of Cr Content on Annealing Twin Formation of Cold-Rolled Ni-Cr Alloys: *Hyo-Min Kim*<sup>1</sup>; Han-Sol Kim<sup>1</sup>; Won-Yong Kim<sup>1</sup>; <sup>1</sup>Korea Institute of Industrial Technology

Ni-Cr alloy sputtering targets have been widely used for bond layer in the FCCL (Flexible Copper Clad Laminate). Recently, however, the general trend of downsizing in electronic devices demand improved properties of deposition and etching processes. In this study, we have investigated the texture and microstructure of Ni-Cr alloys with scanning electron microscope (SEM) and electron back scatter diffraction (EBSD) according to production process parameters, such as cold rolling and annealing processes. The Ni-Cr ingots were cast by vacuum induction casting method, the cast ingots were cold rolled at room temperature with a total reduction of 90% in thickness. And then the cold rolled sheets were annealed at 873K and 1073K. The cube texture {100}<100>

equiaxed grain structures appeared and the grain size increased due to grain growth with increasing annealing temperature and time. With increasing Cr content, a fraction of twin boundary increased and  $\{223\}<113>$  texture component rotated close to the direction of  $\{122\}<112>$  for samples annealed at 1073K for 120min. Details will be discussed in correlation with texture and deposition properties.

#### 11:15 AM

## Preparation of SOG-Si by Electrochemical Reduction of SiO<sub>2</sub> Pellet in Molten Salt: *Ki-Hwan Kwon*<sup>1</sup>; Jae-Soo Noh<sup>1</sup>; Ki-Young Kim<sup>1</sup>; <sup>1</sup>Korea University of Technology and Education

The high production cost of solar grade silicon in conventional processes limits the wide spread use of Si. Thus, there is a strong demand to find a new cost effective process to produce SOG-Si at low cost. Recently, electrochemical reduction is also being considered as one of the promising developments. To gain reduce contamination and lower experimental temperature is main issues demanded recently in electrochemical reduction. In this research, the electrochemical reduction of SiO, pellet using LiCl - Li<sub>2</sub>O molten salt as an electrolyte was investigated. Since SiO, powder is difficult to rinse with in HCl, SiO, powder was pelletized by CIP. The  $\mathrm{SiO}_{\mathrm{2}}$  pellet(Working electrode) was immersed in molten salt after winding molybdenum wire. Glassy carbon was used as a Counter/Reference electrode. The reduction of the pellet was conducted in an argon atmosphere at 923K by potentiostatic electrolysis. Sample was washed in distilled water and analyzed by XRD, SEM, EDX after separation of the Si-pellet from molybdenum wire. The reduced Si-pellets were melted under vacuum at 1773K for 10 minutes by using a high frequency induction furnace and chemical composition of the sample was analyzed by ICP.

#### 11:30 AM

Structural Characteristics of Pultruded FRP Composite Experienced Freezing and Thawing Cyclic Temperature: *Jae-Wook Kim*<sup>1</sup>; Kwang-Yeoul Shin<sup>1</sup>; Dong-Min Ok<sup>2</sup>; Dong-Jun An<sup>3</sup>; Soon-Jong Yoon<sup>1</sup>; <sup>1</sup>Hongik University; <sup>2</sup>Daewoo Engineering & Construction; <sup>3</sup>Woojoo Engineering Co., Ltd.

Due to the advantages of FRP composite such as corrosion resistance, light weight, high specific strenth and stiffness, flexibility, etc., the use of FRP composite in construction sites is increasing steadily. Especially, corrosion resistance is very strong point of FRP composite. Although FRP composite has many advantages, however, the material properties of FRP composite under various environmental situation, to be used in the construction sites, are not well investigated. In this paper, we present the results of experimental investigations of FRP composite experienced the extremely low cyclic temperature. In this investigation, we performed experimental studies to find the stress versus strain characteristics of FRP composite. In the experimental program, strength and stiffness of the pultruded FRP composite specimen under uniaxial tension affected by the freezing and thawing mechanism are evaluated and the results are discussed.

#### 11:45 AM

Adsorption of Lead and Cadmium onto Natural and Modified Diatomite: *Pusit Pookmanee*<sup>1</sup>; Pongthep Jansanthea<sup>1</sup>; Sukon Phanichphant<sup>2</sup>; <sup>1</sup>Maejo University; <sup>2</sup>NANOTEC Center Excellence at Chiang Mai University

The adsorption of lead and cadmium standard solutions onto natural and modified diatomite was studied. Natural diatomite was modified with hydrochloric acid by a low temperature hydrothermal method. Chemical composition of natural and modified diatomite was characterized by X-ray fluorescence spectroscopy (XRF). The morphology of natural and modified diatomite was studied by scanning electron microscopy (SEM). The particle size of natural and modified diatomite was examined by the particle size distribution analysis. The surface area was investigated by specific surface area analysis (BET). The adsorption of lead and cadmium standard solutions was determined by atomic absorption spectroscopy (AAS).

#### 12:00 PM

Influence of Vibration at High Temperature on Lead-Free Solder Joint Reliability: Yoonki Sa<sup>1</sup>; Sehoon Yoo<sup>1</sup>; Junki Kim<sup>1</sup>; Yeong Kim<sup>2</sup>; *Chang-Woo Lee*<sup>1</sup>; <sup>1</sup>KITECH; <sup>2</sup>Korea Aerospace University

In this study, vibration on the reliability of solder joints at high temperature was analyzed. Sn-3.5Ag, Sn-0.7Cu and Sn-5.0Sb solder balls were used to fabricate BGA test chips. The BGA test chips were assembled on the daisy chain circuit board to prepare the test modules. The modules were secured in a temperature controllable chamber to apply random vibration. The frequency range of the vibration was between 10 and 1000 Hz under constant acceleration of 29.4 m/s2. Under the vibration, the temperature was chosen to be 150°C. The resistances of the tests modules were monitored in-situ to examine the failure time during the tests. The original shear strengths and microstructure with respect to IMCs morphologies and thickness of the solder joints were recorded and analyzed and those were compared after the tests to identify the failures. A numerical simulation to obtain the natural frequency and corresponding modes of the module were also performed to estimate the frequency range effect of the random vibration on the



test module failures. It was found that IMCs growth of Sn-0.7Cu solder joint was comparatively inhibited, which seems to exhibit superior mechanical properties among the investigated solder materials in the present test method.

#### 12:15 PM

Vibration Fracture and Microstructural Behavior with Respect to Pb-Free Solders: Sanghun Jin<sup>1</sup>; Namhyun Kang<sup>1</sup>; Changwoo Lee<sup>2</sup>; Sehoon Yoo<sup>2</sup>; Wonsik Hong<sup>3</sup>; Dae-Geun Nam<sup>2</sup>; <sup>1</sup>Pusan National University; <sup>2</sup>Korea Institute of Industrial Technology; <sup>3</sup>Korea Electronics Technology Institute

Environmental and health concerns over the lead have led to investigation of the alternative Pb-free solders to replace commonly used Pb-Sn solders in microelectronic packaging application. Most leading candidates for leadfree solder alloys are near eutectic Sn-Ag-Cu alloys. Solder interconnections in automobile applications require the improvement of vibration resistance significantly as compared with the near eutectic Sn-Ag-Cu alloys. In modern times, however, rapid rise of Ag price demands solder compositions of low Ag content. Solder compositions of low Ag content have been reported to enhance vibration reliability. The study investigated the effect of alloying elements (Bi,In and Ag) on the vibration fracture and microstructural behavior. Prior to the vibration test, the SACX1205 including the element 'In' indicated the lowest shear and pull strength. However, the vibration resistance was improved by adding the In and Bi alloying elements to SACX1205 and SACX0307, respectively. The microstructure of the as-solded cross-section indicated the IMC (Cu<sub>6</sub>Sn<sub>5</sub>, Ag<sub>3</sub>Sn and Cu,Sn). The IMC was not changed post to the vibration test. And, there was no great difference of IMC thickness as a result of vibration test for 20 hrs. The effect of the alloying elements on the vibration fracture will be discussed in the study.

#### Symposium K: Composites and Hybrid Materials: Metal-Based Composites I

Friday AMRoom: 4August 6, 2010Location: Cairns Convention Centre

Session Chair: Shinhoo Kang, Seoul National University

#### 8:30 AM Keynote

Thermo-Physical Properties of Ti-Coated Diamond/Al Composites Prepared by Pressure Infiltration: Yang Zhang<sup>1</sup>; *Xitao Wang*<sup>1</sup>; Jianhua Wu<sup>1</sup>; Senbao Jiang<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing

Thermo-physical properties of diamond reinforced Al composites were investigated. Volume fraction of diamond particles was up to 55%. In order to improve the interfacial bonding between diamond and aluminium, diamond particles were pre-coated with titanium using molten salt method. XRD and SEM observation showed that the Ti coating on diamond consists of carbide layer and metal layer which mainly depend on temperature and time. The influences of the Ti coating on interfacial characteristic and the thermal-physical properties of the composites are studied. The interfacial characterization and thermal diffusivity measurements indicate that Ti coating on diamond results in an increase of thermal conductivity of the composites, from 200 to 430 W/mK along with a coefficient of thermal expansion of  $6.40 \times 10^{-6}$ K.

#### 8:50 AM

#### Effects of Cu Powder Size on the Microstructure of TiB<sub>2</sub>/Cu Composites Fabricated by Reactive Infiltration Process: *Kato Shinji*<sup>1</sup>; Kobashi Makoto<sup>1</sup>; Kanetake Naoyuki<sup>1</sup>; <sup>1</sup>Nagoya University

Recently, the technology for both improving thermal conductivity and controlling the coefficient of thermal expansion of heat sink materials is getting very important because of the downsizing movement of electronic devices. We have been investigating the innovative processing method for  $\mathrm{TiB}_{\mathrm{2}}$  dispersed Cu matrix composite by reactive infiltration process in which the combustion reaction of elemental powders (Ti+2B+Cu  $\rightarrow$  TiB<sub>2</sub>+Cu) and pressureless infiltration of molten Cu into porous reaction product (TiB<sub>2</sub>/Cu composite) are combined. By this process, fine TiB, particles (2~3µm) can be dispersed in Cu matrix homogeneously. However, for better thermal conductivity and reduced thermal expansion, 3-dimentionally continuous inter-penetrating structure of TiB, and Cu phases is suitable. In this study we research the effects of Cu powder size and content in Ti,B,Cu green powder compact on the microstructure of the combustion synthesized TiB<sub>2</sub>/Cu composite. When Cu powders smaller than 45µm were used, TiB, particles were uniformly dispersed in Cu matrix. However, when Cu powders larger than 150µm were used, monolithic Cu area without TiB, dispersion was formed. The monolithic Cu area tended to be connected each other by increasing the amount of Cu powders, which resulted in the formation of 3dimensionally continuous inter-penetrating TiB<sub>2</sub>/Cu microstructure.

#### 9:05 AM

**Cast Bonding of Cast Irons to Ferritic Stainless Steel**: *Celal Cingi*<sup>1</sup>; Veijo Rauta<sup>1</sup>; Eero Niini<sup>1</sup>; Juhani Orkas<sup>1</sup>; <sup>1</sup>Aalto University School of Science and Technology

Composite metal products consisting of two different alloys can be prepared by a few methods. Cast bonding is one of these methods. The bond between the two materials forms primarily in the solid state by diffusion, after casting of a cladding alloy on to the preheated surface of a substrate. In this work, a ferritic stainless steel was used as the substrate, and, gray iron or nodular iron as the cast alloy. It was found that these two alloys can be successfully joined, and under specific casting parameters, a very strong bond develops between the two alloys. Bond strength was found to be greater than that of gray iron. Microstructural zones on both sides of the bond were studied. It was found that diffusion of chromium into iron and diffusion of carbon into steel is significant in bonding. Chemical composition changes due to diffusion was studied by EDS. Fe-Cr-Mn carbides were formed at the bond during the casting. These carbides were largely eliminated by a subsequent high temperature heat treatment.

#### 9:20 AM

Effect of Soak Time on the Microstructural Evolution at the Interface of Kiln Cast White Iron/Steel Composites: *Timothy Lucey*<sup>1</sup>; Paul Huggett<sup>2</sup>; Richard Wuhrer<sup>1</sup>; Wing Yeung<sup>1</sup>; <sup>1</sup>University of Technology, Sydney; <sup>2</sup>Materials Solutions Pty Ltd

A novel kiln casting process is used to join steel and white iron in manufacture of composites for usage in the mining industry. Steel is used extensively in the construction of wear parts of many mining applications. The use of composites incorporating white cast iron and steel permits wear resistant materials with a reasonably soft core structure to be used in applications where moderate impact conditions are encountered. The kiln cast process allows the production of complex shaped products with significantly improved wear resistance and impact strength for high performance mining applications. The present study is to develop a fundamental understanding of the effects of the process variables such as alloy compositions, process temperatures and atmosphere on the microstructure and phase developments in the interfacial areas of the composite materials. The effects of the soak time of the process on the microstructural evolution of the white iron/steel composites will be reported in this article. It was found that as the soak time increased, the thickness of the interfacial area continued to increase with a significant formation of carbides. The relation of the interface development and carbide formation was studied and analysed.

#### 9:35 AM

**Thermal Stability of Fe Filaments in Deformed In Situ Cu-Fe Composites:** *Engang Wang*<sup>1</sup>; Lei Qu<sup>1</sup>; Xiaowei Zuo<sup>1</sup>; Lin Zhang<sup>1</sup>; Jicheng He<sup>1</sup>; <sup>1</sup>Key Laboratory for Electromagnetic Processing of Materials, Ministry of Education, Northeastern University

In this paper, the Cu-12.8wt.%Fe alloys are fabricated and then drawn to Cu-Fe composite wires with the drawing ratio of 8.2. The microstructural stability of Fe  $filaments in the deformed \,Cu\mbox{-}12.8 wt.\% Fe \,composite \,wires \,under \,different \,elevated$ temperature is investigated. The results show that the Fe fibrous morphology gradually takes place the longitudinal splitting, cylinderization, spheroidization and coarsening with the increasing of the elevated temperature. The longitudinal boundary splitting is determined by the greater cross sectional aspect ratio (width/thickness, w/t) and the ratio of boundary to interfacial energy and thus the splitting Fe filaments subsequently evolve into the cylinders. The instability of the cylindrical Fe filaments is controlled by the Rayleigh perturbation instability mode and two dimensional Ostwald coarsening mode. The simulations of the Fe cylinders via the two modes indicate that the perturbation breakup firstly occurs at the smaller cylindrical Fe filaments in diameter. The breakup time decreases with increasing elevated temperature. The diameters of cylindrical Fe filaments increase in linear proportion as the holding time. The smaller is the diameter of cylindrical Fe filaments, the larger is the coarsening rate.

#### 9:50 AM

Ballistic Performance of Zr-Based Bulk Metallic Glass/Ti Surface Composites Fabricated by High-Energy Electron-Beam Irradiation: *Jeonghyeon Do*<sup>1</sup>; Changwoo Jeon<sup>1</sup>; Duk-Hyun Nam<sup>2</sup>; Choongnyun Paul Kim<sup>1</sup>; Young Bum Song<sup>3</sup>; Sunghak Lee<sup>1</sup>; <sup>1</sup>POSTECH; <sup>2</sup>Hyundai Motors; <sup>3</sup>Agency for Defense Development

This study aimed at investigating the ballistic performance of Zr-based bulk metallic glass/Ti surface composites fabricated by high-energy electron-beam irradiation. The mixture of Zr-based metallic glass powders and flux powders was deposited on a pure Ti substrate, and then electron beam was irradiated on this powder mixture to fabricate an one-layered surface composite. Four-layered surface composites (layer thickness; about 3 mm) were fabricated by repeating

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three times the electron beam irradiation process. The ballistic impact test was conducted on surface composite plates to evaluate the ballistic performance. Since the surface composite layers were observed to block effectively a fast traveling projectile, while many cracks were formed in the composite layers, the surface composite plates were not perforated. The surface composite layer containing ductile  $\beta$  dendritic phases showed the better ballistic performance than the one without containing dendrites because dendritic phases could hinder the propagation of shear bands or cracks.

#### 10:05 AM

**Powder Extrusion Followed by ECAP Consolidation of CNT Reinforced AA 4032 Composites:** Senthil Saravanan M.S.<sup>1</sup>; *Kumaresh Babu S.P*<sup>1</sup>; Sivaprasad K<sup>1</sup>; Susila P<sup>2</sup>; Murty B.S<sup>2</sup>; <sup>1</sup>National Institute of Technology; <sup>2</sup>Indian Institute of Technology

ECAP consolidation was one of recent attempts on composite powders. Powder densification through ECAP is still challenging to get a bulk sample. The consolidation of particles after it undergoes regular ECAP, shows difference in densification and hardness from inner portion to outer layer of the sample. To overcome this problem, hot powder extrusion followed by ECAP (route BA) was attempted. Elemental powders of AA 4032 equivalent composition were used in the present investigation along with carbon nanotubes synthesized by arc discharge method as the reinforcement material. The characterization of the consolidated samples was done using XRD, scanning electron microscope and transmission electron microscope. The crystallite size variation and strain variations were calculated using Williamson-Hall X-ray peak broadening analysis. The relative density of the samples was measured using Archimedes principle. The hardness was calculated in both parallel and perpendicular to the ECAP direction. Tensile properties were also evaluated on consolidated samples. TEM studies evidenced the uniform distribution of CNTs in the matrix. With increasing fraction of CNT reinforcement, the mechanical properties were observed to be increased. Number of passes during ECAP also enhanced the mechanical properties.

#### 10:20 AM

Fabrication of (Al<sub>2</sub>O<sub>3</sub>-Al<sub>3</sub>Nb)/Al Composite Materials by In-situ Reaction Using MA Processed Al/Nb<sub>2</sub>O<sub>5</sub> Powder: Hyun Bom Lee<sup>1</sup>; Hiroyasu Tezuka<sup>1</sup>; Equo Kobayashi<sup>1</sup>; Tatsuo Sato<sup>1</sup>; Kee Do Woo<sup>2</sup>; <sup>1</sup>Tokyo Institute of Technology; <sup>2</sup>Chonbuk National University

A planetary ball milling (PBM) technique was employed to fabricate mechanically alloyed (MA processed) Al-Nb<sub>2</sub>O<sub>5</sub> composite powder. Nano or submicron sized Nb<sub>2</sub>O<sub>5</sub> particles were homogeneously embedded in the Al particles after milling for various periods. None of cracks, by-products and pores were observed in the areas between embedded Nb<sub>2</sub>O<sub>5</sub> particulates and Al matrix powder after milling. The sequence of the in-situ reaction was confirmed by DTA, DSC, XRD measurements, optical microscopy and EPMA. The specific temperature of the in-situ reaction was between 650 and 700°C. Al-based metal matrix composites (MMC) reinforced with the sub-sieve sized 0-Al<sub>2</sub>O<sub>3</sub> particulates and Al<sub>3</sub>Nb intermetallic compound were successfully fabricated by the in-situ reaction process. The reduced Nb by the in-situ reaction was fully reacted with Al to form the Al<sub>3</sub>Nb intermetallic compound during sintering. A number of subsieve sized 0-Al<sub>2</sub>O<sub>3</sub> particulates and Al<sub>3</sub>Nb intermetallic compound formed by the in-situ reaction between Al and Nb<sub>2</sub>O<sub>5</sub> were homogeneously distributed in the Al matrix during sintering. Nano sized  $\theta$ -Al<sub>2</sub>O<sub>3</sub> particulates are preferentially distributed near the Al,Nb intermetallic compound and no by-products are formed in the interfaces with the Al matrix.

#### 10:35 AM Tea Break

#### Symposium K: Composites and Hybrid Materials: Metal-Based Composites II

Friday AM August 6, 2010 Room: 4 Location: Cairns Convention Centre

Session Chair: Xitao Wang, University of Science and Technology Beijing

#### 11:00 AM Keynote

**Ti-Based Solid-Solution Carbide Cermets**: *Shinhoo Kang*<sup>1</sup>; <sup>1</sup>Seoul National University

TiC- and Ti(CN)-based cermets have been used successfully for high precision machining operation. The addition of secondary carbides or nitrides greatly improves Ti(CN)-Ni cermets in terms of not only mechanical properties but also densification of the cermets. The amounts and types of dissolved carbides along with sintering duration are important determinants of core/rim structure, which is

typical in Ti(CN)-Ni cermet systems. Recently considerable attentions are being made on the tool materials composed of solid solution carbides. These carbides provide significant changes in microstructure and properties. In this presentation Ti-based cermets made of (Ti,W)C and (Ti,W)(CN) solid solution carbides will be introduced and their microstructure and properties are compared to WC-Co. Based on the results, the effectiveness of these carbides in tool performance will be discussed.

#### 11:20 AM

Composite Filler Metals for Joining of Temperature-Sensitive Aluminium-Matrix Composites: Bernhard Wielage<sup>1</sup>; Ina Hoyer<sup>1</sup>; *Sebastian Weis*<sup>1</sup>; <sup>1</sup>Chemnitz University of Technology

There has been an increased demand for lightweight composite materials in the last years. In particular, aluminium-matrix composites (AMC) have been applied in many different sectors such as aerospace industry, automobile production, or power-electronics. As a special group of composites, AMCs produced by ECAE (equal-channel angular extrusion) feature a very high strength due to a very finegrained structure. But they are very temperature-sensitive. Therefore, an adapted joining technique is required. In this regard, soldering offers some advantages in comparison to other joining processes like welding or bonding. Because of their low melting range below 300 °C, Sn-based filler metals are suitable for this purpose; Ag and Cu are the common alloying elements. The low strength and creep resistance of the joints are disadvantageous features. An improvement of these properties can be achieved by the addition of ceramic reinforcement particles such as Al<sub>2</sub>O<sub>3</sub> or SiC. Investigations into the interface formation as well as the particle type, content and distribution were carried out. Ti as active element was alloyed to improve the bonding between matrix and particles. The microstructure observed by SEM has been correlated with the results of the tensile tests.

#### 11:35 AM

Microstructure and Interface of Mg3Zn6Y Quasicrystal Particulate Reinforced Mg-8Gd-3Y Alloy: Wu Shiping<sup>1</sup>; <sup>1</sup>Harbin Institute of Technology

The impact of particle size, adding amount of quasi-crystalline and time at maximum temperature of melt on microstructure and mechanical properties of Mg-8Gd-3Y alloys were studied and Mg-Zn-Y quasi-crystalline particles prepared adopting conventional solidification methods were crushed with the method of mechanical crushing. Adding quasi-crystalline particles into the Mg-8Gd-3Y melt with additional method changed the particle size, adding amount of quasi-crystalline particles and time at maximum temperature of melt. Cast solidification microstructure and properties of quasicrystal particle-reinforced Mg-8Gd-3Y alloys were studied by SEM, TEM and tensile test at room temperatures. It is shown that quasi-crystalline particles existed in Mg-8Gd-3Y alloys, and quasi-crystalline particles distributed in the interior grains. The tensile strength of the quasicrystal particle-reinforced Mg-8Gd-3Y alloys increases 24.5%, and the optimum performance parameters are 833-1667µm (particle size), 15% (adding amount of particle) and 3 mins (time at maximum temperature).

#### 11:50 AM

Superconductive Property and Microstructure of MgB<sub>2</sub> Particle-Dispersed Aluminum Based Composite Materials: *Manabu Mizutani*<sup>1</sup>; Kenji Matsuda<sup>1</sup>; Kazuya Makino<sup>1</sup>; Katsuhiko Nishimura<sup>1</sup>; Tokimasa Kawabata<sup>1</sup>; Yoshimitsu Hishinuma<sup>2</sup>; Shigeki Aoyama<sup>3</sup>; Susumu Ikeno<sup>1</sup>; Katsumi Watanabe<sup>1</sup>; <sup>1</sup>University of Toyama; <sup>2</sup>National Institute for Fusion Science; <sup>3</sup>Nikkei Niigata

Superconducting wires have been applied for superconducting magnets in the nuclear magneto-resonance (NMR), Magneto-resonance imaging (MRI) and so on. MgB<sub>2</sub> has the highest critical temperature of superconducting transition (TC=39K) in the intermetallic compound superconductive material. This means that MgB, Superconductive wire doesn't need expensive liquid He for cooling. We used the original method of the three-dimensional penetration casting (3DPC) in this laboratory to fabricate the MgB,/Al composite. Our 3DPC method for fabricating composite materials can disperse particles in the matrix homogenously without any aggregation and control volume fractions of composites within the range of 4 – 40%, even when particle size is less than 1  $\mu$ m. Thus, these composite materials can be processed by machining, extrusion and rolling. In the composite material we made, MgB, particles dispersed to the Al matrix uniformly. The TC was determined by electrical resistivity and magnetization to be about 37~39K. We succeeded in extruding MgB<sub>2</sub>/Al composite billet to f1mm wire.Microstructures of these samples have been confirmed by TEM and SEM method. MgB<sub>2</sub>/Al composite billet and extruded wire were no cracks inside the materials.

#### 12:05 PM

The Effect of a Small Volume Liquid Phase on the Hot Deformation in BaPbO<sub>3</sub>/2024Al Composite: G.H. Fan<sup>1</sup>; L. Geng<sup>1</sup>; <sup>1</sup>Harbin Institute of Technology, China

The poor ductility of the metal matrix composites leads to a high production cost, limiting the wide application of the composites. Many researchers have found



that the increase in plasticity of aluminum composite is related to the presence of the liquid phase along composite interface and matrix grain boundaries, which will release stress concentration and thus accommodate grain boundaries sliding. However, once liquid phase appears during deformation process, the changes of the matrix including microstructure and property have not been studied until now. In the present paper, BaPbO<sub>3</sub>(5Vol.%)/2024Al composite was fabricated by powder metallurgy method. Nanosized Pb particles were formed from the in-situ reaction between BaPbO<sub>3</sub> and 2024Al, and mainly distributed homogeneously inside grains. The hot extrusion deformation of the composite at different temperatures was performed. The dislocation density measurement of the 2024Al matrix and dynamic mechanical analysis (DMA) of the composite were made to investigate the effect of liquid Pb on the hot deformation. The results show that the presence of the liquid phase reduces deformation resistance by decreasing dislocation pileups, and simultaneous decrease in Youg's modulus.

#### 12:20 PM

Isotropic and Anisotropic X-Ray Peak Broadening Models of AA 6061-12 Wt. % TiO<sub>2</sub> Nanocomposite: S. Sivasankaran<sup>1</sup>; K. Sivaprasad<sup>1</sup>; *R. Narayanasamy*<sup>1</sup>; Vijay Kumar Iyer<sup>1</sup>; <sup>1</sup>National Institute of Technology

In this work, nanocrystalline AA 6061 composite reinforced with 12 wt.% TiO<sub>2</sub> (tetragonal) powder was synthesized by mechanical alloying (MA) route. These nano powder were consolidated through conventional powder metallurgy (P/M) route. The as-milled and as-sintered (sintering at 300 and 400°C under nitrogen atmosphere) nanocomposites were characterized by X-ray diffraction (XRD) and high-resolution transmission electron microscope (HR-TEM). The individual contribution of small crystallite sizes and lattice strain to the peak broadening in as-milled and as-sintered nanocomposites were studied using Isotropic and anisotropic models of Williamson-Hall analysis like uniform deformation, uniform stress deformation, and uniform energy density deformation model. The other relevant physical parameters such as stress and energy density values were calculated precisely using the above models. Among the developed model, uniform energy density deformation model (UEDM) is observed to be best fit model for MA powders. This evidenced the more anisotropic nature of ball milled powders. Finally, as-milled and as-sintered lattice parameter of Al matrices were determined by fitting the observed reflections between the calculated lattice parameter for each Bragg's angle and cos20/sin0. Increased lattice parameter was observed in as-sintered composite than that of as-milled condition.

Fri. AM



#### Poster Session: Symposium A: Advanced Steels and Processing

Tuesday PM	Room: Hall 2
August 3, 2010	Location: Cairns Convention Centre

A1 Microstructural Observation on Materials of the Japanese Sword under Fold-Forging Process: Go Takami<sup>1</sup>; Takuya Ohba<sup>1</sup>; Shigekazu Morito<sup>1</sup>; Ananda Das<sup>1</sup>; <sup>1</sup>Department of Materials Science, Shimane University, Japan

Japanese sword is made from a special kind of steel called tamahagane, which is produced in traditional tatara process. The preliminary product of tatara is given to shape of metallic plates by stacking, welding and hammering operations. The prepared tamahagane plates are then cut, fold and forged repeatedly for making the final product, Japanese sword. It is said that the cyclic forge-fold operation is performed to control the carbon content but the relation between the cyclic operations and formation of microstructures is ambiguous. The purpose of this study is to clarify the development of microstructures with successive forge-fold operation on tamahagane, the materials of Japanese sword. Each of the subsequent steps for preparation of Japanese sword has been observed by Optical Microscope and Scanning Electron Microscope with Energy Dispersive X-ray Spectroscopy. A combination of ferrite and pearlite morphology has been found along with a lot of nonmetallic inclusions. The ferrite bands become smaller and finer with increasing the recurrence of forge-fold operations.

#### A2 Spheroidization Behavior of Cementite in a High Carbon Steel: *Tae Kwon Ha*<sup>1</sup>; Changhoon Lee<sup>2</sup>; Kisoo Kim<sup>2</sup>; <sup>1</sup>Gangneung-Wonju National University; <sup>2</sup>POSCO

The effect of initial microstructure, cold reduction ratio, and annealing temperature on the spherodization rate of SK85 high carbon steel sheet was investigated. High carbon steel sheet fabricated by POSCO was soaked at 800°C for 2 hr in a box furnace and then treated at 570°C for 5 min in a salt bath furnace followed by water quenching to obtain a fine pearlite structure. Cold rolling was conducted on the sheets of fine pearlite by reduction ratios of 20, 30, and 40 % and heat treatment for spheroidization was carried out at 600 and 720°C for the various time intervals from 0.1 to 32 hrs. Area fraction of spheroidized cementite was measured with an image analyzer as a function of cold reduction ratios and duration times.

#### A3 Study on Carbonitride Precipitating Process in Nb-V Micro-Alloyed Steel: *Wenqing Liu*<sup>1</sup>; Xiaoyong Zhu<sup>1</sup>; Xiaojiao Wang<sup>1</sup>; Xiangyuan Xiong; <sup>1</sup>Shanghai University

Microalloyed steel is regarded as a new type of high strength low alloy steel , which are develop by adding a small amount of two or more alloying elements in traditional low alloy steel. As the most prevalent used additional elements, Nb, Ti, V can substantially improve the strength of steels by refinement of grain size, especially by precipitation strengthening in ferrite. The 3D atom probe (3DAP) is a particularly helpful instrument with atomic spatial resolution and high componential sensitivity in the characterization of the early stages of precipitation reactions. The carbonitrides precipitate in Nb-V micro-alloying steels was investigated by 3DAP. There are great differences in composition among these precipitates with difference size. It is considerable that the carbon atoms precipitate at the dislocation or vacancy first, the vanadium and niobium atoms precipitate at the place in turn, V-C and V-Nb-C cluster coming into being successively, (Nb,V)C complex phase form last. The composition becomes asymmetry as the precipitates grow up.

#### A4 Microstructure and Properties of 9CrSi Steel by Laser Surface Alloying: Wang Wen Yan<sup>1</sup>; <sup>1</sup>Henan University of Science and Technology

In this paper, a series of experiments were carried out to observe the microstructure, the microhardness, the friction coefficient and wear resistance of the Ni-based nano-h-BN solid self-lubricating coatings which were prepared by both HVOF spraying process and YAG laser cladding. The microstructures and friction properties were investigated by using of XRD, SEM,EDS and pin-on-disk tribo-tester machine (MMS - 1G). Results showed that 5% of the content h-BN's friction coefficient is the most stable whose friction coefficient is stable around 0.470, so we come to a conclusion that this coating is an ideal HVOF coating; Whereas the ideal laser cladding coating is 7.5%h-BN(Ni) coating, whose friction coefficient is around 0.360, this is the best laser cladding coating.

A5 Microstructure and Tensile Properties of ODS Ferritic Steels Produced by Mechanical Alloying in Argon and Hydrogen Gas Environments: *Noriyuki Iwata*<sup>1</sup>; Ryuta Kasada<sup>1</sup>; Akihiko Kimura<sup>1</sup>; Takanari Okuda<sup>2</sup>; Masaki Inoue<sup>3</sup>; Fujio Abe<sup>4</sup>; Shigeharu Ukai<sup>5</sup>; Somei Ohnuki<sup>5</sup>; Toshiharu Fujisawa<sup>6</sup>, <sup>1</sup>Kyoto University; <sup>2</sup>Kobelco Research Institute, Inc.; <sup>3</sup>Japan Atomic Energy Agency; <sup>4</sup>National Institute for Materials Science; <sup>5</sup>Hokkaido University; <sup>6</sup>Nagoya University

Two types of oxide dispersion strengthened (ODS) ferritic steels, with the composition of Fe-15.5Cr-2W-4Al-1Zr-0.35Y<sub>2</sub>O<sub>2</sub> (in weight percent), were produced by mechanical alloying (MA) of Fe, Cr, W, Al, and 50Fe-50Zr powders with Y,O, particles either in argon or hydrogen gas environments and solidification of the MA powders by vacuum hot press. SEM and PSD analyses revealed that after milling for 48 h, the MA powders were composed of agglomerated particles having almost similar distributions with particle size ranging from 1 µm to 50 um. Relatively uniform-sized grains, without any argon bubbles both in grains and on grain boundaries, were observed in the ODS ferritic steel when milling was carried out in a hydrogen gas environment. It was found, in particular, that MA in hydrogen is very effective to improve the ultimate tensile strength and total elongation of the ODS ferritic steels, as measured by tensile tests at room temperature and 700 °C. Present study includes the result of "Development of super ODS steels with high-resistance to corrosion towards highly efficient nuclear systems" entrusted to Kyoto University by the Ministry of Education, Culture, Sports, Science and Technology of Japan (MEXT).

#### A6 An Investigation on the Effects of Isothermal Treatment Parameters on Microstructure Properties of Nanostructured, Low Temperature Bainitic Steels: Sasan Yazdani<sup>1</sup>; Nariman Youzbashi<sup>1</sup>; <sup>1</sup>Sahand University of Technology

In the recent decade nanostructured, low temperature bainitic steels which is composed of a slender plates of bainitic ferrite (20-40 nm) with small amounts of uniformly distributed carbon enriched austenite have been developed by Bhadeshia and Caballero. For the purpose of investigation on the microstructure properties of these steels, samples after austenitising at 950°C for 30 minutes were transformed isothermally at temperatures in the range of 200-300°C for different times. For attaining a microstructure with prominent characteristics, the measurements of hardness and evaluation of microstructure with OM, XRD and TEM showed that, the isothermal treatment times of 6, 16 and 72 hours give an acceptable microstructure for isothermal transformation temperatures of 300, 250 and 200°C respectively.

**A7 High Temperature Aging Response in CASTRIP Steels**: *Sachin Shrestha*<sup>1</sup>; Y. Xie<sup>1</sup>; S.P. Ringer<sup>1</sup>; J.M. Cairney<sup>1</sup>; <sup>1</sup>The University of Sydney, Australian Key Centre for Microscopy and Microanalysis

This study investigates the effect of the heat treatment of CASTRIP steels at temperatures of 700-800°C (below and above temperature for transformation to austenite). The Castrip<sup>®</sup> process is an innovative new twin rolling method for producing steel strip directly from liquid steel. This process can produce thin steel strip with significantly less energy, time, and floor space when compared to conventional slab casting methods. The CASTRIP steel specimens being investigated are plain carbon steel and Nb-microalloyed steel (0.04 wt% and 0.08 wt%), and the role of Nb will be studied. Hardness values of both Nb-microalloyed steel and Nb-free steel obtained against various aging times will be presented. We will show the results of characterisation of the grain structures using optical microscopy, transmitted electron microscopy (TEM), and 3D atom probe tomography (APT) and the relationship between this structure and the aging behaviour observed.

A8 An Investigation on Precipitation Behaviour of Cu-Rich Phase in Super304H Heat-Resistant Steel by Three Dimensional Atom Probe(3DAP): Chengyu Chi<sup>1</sup>; Wenqing Liu<sup>2</sup>; Jianxin Dong<sup>1</sup>; *Xishan Xie<sup>1</sup>*; <sup>1</sup>Department of Material Science and Engineering, University of Science and Technology Beijing; <sup>2</sup>Instrumental Analysis & Research Center, Shanghai University

Super304H, a Cu-containing 18Cr-9Ni-3CuNbN heat-resisting steel is wildly used as an important superheater/reheater tube material for Ultra-Super Critical(USC) power plants all over the world. It is recognized that the Cu-rich phase is an important strengthening second phase for Super304H. However, the detail precipitation behaviour and its strengthening effect are still not very clear. Experimental material which taken from routine production was aged at 650°C for different times. The precipitation of Cu-rich phase in Super304H was studied by the three dimensional atom probe(3DAP) and TEM. Experimental results show that Cu-rich clusters have been formed at the very early stage of 650°C aging. The Cu-rich particle images have been clearly caught just after 650°C aging for 5h. The Cu atoms gradually concentrate to the Cu-rich particles and the other elements (such as Cr, Ni etc) diffuse away from Cu-rich particles to the  $\gamma$ -matrix with the increasing of aging time. The Cu-rich particle size and number density have been determined as a function of aging time. Cu-rich particles still keep nano-size and distribute homogenously in grains even after long time (1000h)



aging, which is one of the most important reasons for keeping good strength of Super304H heat-resistant steel at high temperatures.

A9 650°C Long-Term Structure Stability Study on 18Cr-9Ni-3CuNbN Heat-Resistant Steel: Hongyao Yu<sup>1</sup>; Jianxin Dong<sup>1</sup>; Xishan Xie<sup>1</sup>; <sup>1</sup>Department of Material Science and Engineering, University of Science and Technology Beijing

The Cu-containing austenitic heat-resistant steel 18Cr-9Ni-3CuNbN has been widely used as superheater and reheater tube material for modern ultrasuper-critical (USC) power plants in the world. High temperature structure stability is considered to be an important factor for long-term service. Longterm aging at 650°C for this steel was conducted from 1000 to 10000 hours. Effect of aging time on microstructure was studied by means of SEM, TEM and 3DAP(three dimensional atom probe). Micro-hardness tests were carried out after aging at 650°C for different times to be considered as a representative of strength. Experimental results show that Cu-rich phase, MX and M22C6 are major strengthening precipitates in this steel. With increasing of aging time, fine nano-size Cu-rich phase particles precipitate in the grains and its size keeps in the range of several nanometers to 35nm till 10000h at 650°C. The number of MX increases with aging time and its average size is about 100nm till 10000h. M<sub>22</sub>C<sub>6</sub> carbide mainly precipitates at grain boundaries and coarsens quickly. Investigation results show that the most important strengthening effect for 18Cr-9Ni-3CuNbN steel is contributed by Cu-rich phase and MX in the grains and  $M_{23}C_6$  carbide at the grain boundaries.

#### A10 Application of Quenching-Partitioning-Tempering Process in Hot Rolled Plate Fabrication: Shu Zhou<sup>1</sup>; Ying Wang<sup>1</sup>; Nailu Chen<sup>1</sup>; Yonghua Rong<sup>1</sup>; Jianfeng Gu<sup>1</sup>; <sup>1</sup>Shanghai Jiao Tong University

The quenching-partitioning-tempering (Q-P-T) process, based on modifying the quenching and partitioning (Q&P) treatment, has been proposed for producing high strength steel containing significant fractions of retained austenite and controlled amounts of fine martensite laths. In this study, a set of Q-P-T processes of C-Mn-Si-Ni hot rolled plates are designed and realized in laboratory. The steels after Q-P-T processes present good combinations of high strength and relatively high toughness, and the origin of such mechanical properties is revealed by characterization of microstructure.

#### A11 Effect of a High Magnetic Field on the Formation of Widmanstätten Ferrite in Fe-0.36%C Alloy: Xiang Zhao<sup>1</sup>; <sup>1</sup>Northeastern University

The influence of high magnetic field with different strength on the proeutectoid ferrite transformation in high purity Fe-0.36wt%C during diffusional  $\gamma \rightarrow \alpha$  phase transformation was studied. It was found that the formation of acicular ferrite (i.e., Widmanstätten ferrite) was obviously suppressed by the applied high magnetic field. The stronger the magnetic field, the more evidently the ferrite grains elongate and align along the field direction, which is attributed to additional driving force for phase transformation and demagnetization effect introduced by the applied magnetic field. This is also considered to be related to the preferential growth of proeutectoid ferrite nuclei along field direction caused by magnetic dipolar interaction. Acknowledgment: this research was supported by a grant from the army and NSF. The authors would also like to acknowledge the support provided by the NHMFL through the use of their facility. Acknowledgments to the Projects 50971034 and 50911130365 supported by NSFC.

#### A12 Effect of Processing Parameters on Microstructure Development in X70 Pipeline Steel: *Abdullah Alshahrani*<sup>1</sup>; Elena Pereloma<sup>1</sup>; Ali Dehghan-Manshadi<sup>1</sup>; Tom Schambron<sup>2</sup>; Jim Williams<sup>2</sup>; <sup>1</sup>University of Wollongong; <sup>2</sup>BlueScope Steel Limited

Optimisation of strength and toughness are key quality aims in the production of pipeline steels. In Nb-containing steels the mixed grain structure is a common feature, and can be detrimental to toughness properties such as Charpy V-notch and Battelle Drop Weight Test transition temperatures. In steel hot rolling, the final ferrite microstructure is a consequence of austenite transformation. So, an important goal should be to control the prior austenite microstructure in terms of fine grain size and uniformity prior to transformation. The aim of the study is to evaluate the role of (i) re-heated austenite grain structure; (ii) rolling conditions and (iii) austenite recrystallisation in achievement of fine uniform microstructure in high Mn steel. In particular, different strain, temperature and interpass time at finishing stands have been investigated by conducting plane strain experiments using a Gleeble 3500 thermo-mechanical simulator.Results of data analysis from Gleeble simulations and optical microscopy will be presented, discussed and compared to production data. A13 Effect of Processing Parameters on Microstructure, Crystal Orientation and Micro-Texture of High Niobium-Bearing Microalloyed Steel: *Chengliang Miao*<sup>1</sup>; Chengjia Shang<sup>1</sup>; Mani Subramanian<sup>2</sup>; <sup>1</sup>University of Science and Technology Beijing; <sup>2</sup>Mcmaster University

The present studies are aimed at understanding the effect of cooling rate, prior strain and rolling temperature on the evolution of morphology, crystallographic orientation, microtexture and density of high angle boundaries (HABs) in pipeline steel with 0.09 (wt%) niobium. Fast cooling and large deformation below Tnr both can raise drive force of coherent transformation and weaken variants selection, which can generate higher frequency of HABs (=15degree). All products by coherent transformation in identical prior austenite grain keep orientation relationship (OR) with parent austenite, thereinto, lower bainite has highest density of HABs. Moreover, large deformation by few passes below The can accelerate nucleation of ferrite grains in the austenite boundary by incoherent transformation. These fine grains have not OR with prior austenite grain in any side of boundary, and exhibit significant misorientations between themselves. Dispersion of average density of HABs was found from boundary to interior in prior austenite grain, and higher density of HABs is exhibited near austenite boundary. Highest density of HABs was obtained at 850°C, compared with 940, 800°C, and micro-texture index will be larger as decreasing of rolling temperature. Relatively, strongest {100}<110> texture component was found at 800°C, which is infaust texture component for toughness.

#### A14 Processing, Properties and Microstructure of 1.2%Al TRIP Steels: *Delu Liu*<sup>1</sup>; Jian Zhang<sup>2</sup>; Zhongyi Li<sup>2</sup>; <sup>1</sup>University of Science and Technology Beijing; <sup>2</sup>Maanshan Iron and Steel Co., Ltd

TRIP-600 steels containing 1.2%Al was developed on laboratory scale. Composition of the steels is designed as 0.12%C-0.30Si-1.6%Mn-1.2%Al and small amount of Mo or Cr. The phase diagram, TTT curves and T0 line of bainite formation in the steels are calculated by Thermo-Calc. The TRIP600 strips were prepared successfully through steel-making, hot and cold rolling as well as heat treatment. The strips with thickness 1.7mm were reheated to 830°C, 850°C and 870°C followed by isothermal holding at 420°C then quenching. Tensile strength 660~700MPa, yield strength 390~420MPa and total elongation 30.5~37.5% is obtained for the strips. Microstructure of the steels was investigated by chromatic metallography, SEM, EBSD and X-ray diffraction techniques. The results showed that fraction of the retained austenite in the experimental steels is about 11% and the carbon content in the retained austenite is about 1%(wt). It is pointed out by the present investigation that the chemical composition and processing parameters of the experimental steels could be referred for trial-manufacture of TRIP 600 steels. The important factors which interfere with improving of the TRIP steels include decarbonization in the surface layer and band structure of the strips.

## A15 Effect of Thermomechanical Processing Parameters on the Microstructure Development in Low Si TRIP Steel: Fayez Al Harbi<sup>1</sup>; Elena Pereloma<sup>1</sup>; <sup>1</sup>University of Wollongong

In order to reduce the CO<sub>2</sub> gas emission, the reduction in fuel consumption is necessary. This is achievable by automotive vehicles becoming lighter due to utilisation of high strength steels. However, the aim is to increase the strength of steel without adversely affecting its formability. It is well established for TRansformation-Induced Plasticity (TRIP) steels that improvements of the mechanical properties can be attained by maximising the amount of retained austenite (RA). Although the presence of Si in TRIP steel is essential for the formation of RA, high Si addition has adversary effect on the quality of sheet coatings. Therefore, low Si TRIP steel remains the focus of research for many engineers and scientists. In this work the effect of thermomechanical processing (TMP) parameters on the microstructure of the low Si, Al-added Ti-Mo TRIP steel was investigated using Gleeble 3500 thermo-mechanical simulator. The designed TMP schedules were aimed to achieve the microstructures with different volume fractions of phases present and maximise the amount of RA. Microstructure characterisation, including determination of the amount of RA, was performed by optical metallography, image analysis and X-ray diffraction (XRD). Different etching techniques, such as heat tinting and colour etching, were applied to reveal the phases.

A16 Effects of Alloying Elements on Tensile Strength and the Occurrence of Delamination in Hyper-Eutectoid Steel Wires: Jung Won Lee<sup>1</sup>; *Ui Gu Kang*<sup>1</sup>; Yong Shin Lee<sup>1</sup>; Kyung Tae Park<sup>2</sup>; Won Jong Nam<sup>1</sup>; <sup>1</sup>Kookmin University; <sup>2</sup>Hanbat University

The effects of alloying elements and initial interlamellar spacing on tensile strength and the occurrence of delamination in cold-drawn hyper-eutectoid steel wires were investigated under equivalent drawing conditions. The initial interlamellar spacing showed little influence on the occurrence of delamination. The addition of Cr effectively increased attainable tensile strength, since the added Cr not only increased tensile strength but also delayed delamination. The addition

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of Ni also increased attainable tensile strength, since the effect of the added Ni on delaying delamination prevailed over the tendency to decrease tensile strength and work hardening. Meanwhile, the addition of Si showed increased tensile strength but accelerated the occurrence of delamination at lower drawing strain. The addition of Cr increased the attainable tensile strength more effectively than the addition of Ni in cold-drawn hyper-eutectoid steel wires, although the added Ni markedly delayed delamination-associated strain.

#### A17 Effects of Deformation Strains and Annealing Temperatures on Mechanical Properties of Martensitic Steels: Jong Chul Lee<sup>1</sup>; Shin Woong Jeong<sup>2</sup>; Chang Suk Oh<sup>3</sup>; Sung Joon Kim<sup>3</sup>; *Won Jong Nam*<sup>2</sup>; <sup>1</sup>Kookmin University; <sup>2</sup>School of Advanced Materials Engineering, Kookmin University; <sup>3</sup>Korea Institute of Materials Science (KIMS)

The effects of deformation strains and annealing temperatures on microstructures and mechanical properties of martensitic steels were examined. The amount of cold deformation was changed as 30%, 50% and 60%, and annealing temperatures varied from 500°C to 600°C. In samples cold rolled 30%, the dominant microstructure for an annealing at 500°C was dislocation substructures with uniformly distributed rod-shaped carbide particles. For an annealing at 600°C, the microstructure consisted of equiaxed ultrafine grains, spherical carbide particles and elongated dislocation substructures. A proper annealing temperature for martensitic steels received 30% reduction, showing a good combination of a high strength, 1230MPa, and an adequate total elongation. 9.4%, was found as 500°C. Samples received large amount of cold deformation exhibited higher tensile strength than samples received small amount of cold deformation for all annealing temperatures. This would be attributed to larger dislocation density produced during cold deformation and the unchanged width of elongated dislocation substructures during annealing in samples with larger amount of cold deformation.

A18 Identification of Dynamic and Static Ferrite by EBSD Analysis: *Yong-Min Kim*<sup>1</sup>; Tae-Hong Ahn<sup>1</sup>; Do Hyun Kim<sup>1</sup>; Kwang Kyun Park<sup>2</sup>; Kyu Hwan Oh<sup>1</sup>; Heung Nam Han<sup>1</sup>; <sup>1</sup>Seoul National University; <sup>2</sup>POSCO Technical Research Laboratories

In this research, the static and dynamic ferrites were identified by a statistical analysis of experimental data measured from EBSD (Electron Backscattered Diffraction). It has been known that grain refinement could contribute to enhance strength of steel without expense of elongation or formability. The core technology for producing ultrafine-grained ferritic steels is to impose severe deformation to super-cooled austenite which induces dynamic formation of ferrite grains. By excessive strain on austenite, dynamic ferrite nucleation occurs rapidly from grain boundary of base austenite. The nuclei quickly impinge, but coalescence and growth are inhibited to a large degree by the large misorientation angles between adjacent ferrite grains. Consequently, dynamic ferrite has smaller grain size and greater difference of inter-granular orientation than static ferrite. Standing on these differences of each ferrite, various methods such as grain size, local misorientation, intra-granular misorientation and inter-granular misorientation were applied to classify the dynamic and static ferrite. For intergranular misorientation-based method, especially, a quantitative analysis was carried out by using a deviation of inter-granular misorientation as a criterion of the method. Among the various methods, inter-granular misorientation-based method was regarded as the most reasonable one

#### A19 Influence of Microstructural Variations with Inclusion Modification on Impact and Fracture Toughness in High Co-Ni Secondary Hardening Steels: *Kisub Cho*<sup>1</sup>; Sangha Kim<sup>1</sup>; Hwaseok Kang<sup>1</sup>; Konbae Lee<sup>1</sup>; Hyeongryeol Yang<sup>2</sup>; Hoon Kwon<sup>1</sup>; <sup>1</sup>Kookmin University; <sup>2</sup>Incheon City College

The variations in impact and fracture toughness in secondary hardening allow steels were studied for inclusion modification by La, Ti and (La+Ti) additions. La-added and Ti-added alloys mostly contained La-oxy-sulfides and Ti-carbosulfides, respectively. The (La+Ti) added alloy also contained above two types of inclusions. In addition, Ti additions refined the prior austenite grains. The impact toughness and the fracture toughness( $K_{ic}$ ) exhibited the different results, according to microstructural variations such as grain size, sulfide type inclusions, undissolved primary carbides, fine secondary carbides, etc. In the peak-aged condition with nearly the same strength level, while the (La+Ti) added alloy with average grain size of 35µm was higher impact toughness than the La added alloy of 103µm, the opposite behavior was represented in case of fracture toughness. In some case, the Ti added allow with similar volume fraction of sulfide type inclusions but smaller grain size, compared to the La added alloy, indicated a large drop in fracture toughness in spite of the similar level of impact toughness. Those variations in impact and fracture toughness were analyzed by the microstructural investigation through OM, FE-SEM, TEM and XRD.

A20 Non-Inclusion Induced Crack Initiation of Bainite/Martensite Dual Phases Steels in Very High Cycle Fatigue: *Jialin Gu*<sup>1</sup>; Yang Yu<sup>1</sup>; <sup>1</sup>Tsinghua University

The behaviors of very high cycle fatigue (VHCF) of Bainite/Martensite (B/M) dual phases high-strength low-alloy (HSLA) steels were investigated. Non-inclusion induced crack initiations occurred in the designed steels under ultrasonic fatigue test. Results show the weaker aspect of the steel, including microstructure and inclusion, will dominate the VHCF behavior. Competition mechanism between microstructure and inclusion is advanced and established according to the VHCF behaviors of studied steels, which could give a direction for improving the VHCF property.

#### A21 Effect of Carbon on Frictional Wear Behaviours of High Vanadium High Speed Steel under Dry Sliding Condition: *Liujie Xu*<sup>1</sup>; Shizhong Wei<sup>1</sup>; Yingping Ji<sup>1</sup>; Guoshang Zhang<sup>1</sup>; Jiwen Li<sup>1</sup>; Rui Long<sup>1</sup>; <sup>1</sup>Henan University of Science and Technology

The high vanadium high-speed steel (HVHSS) with about 9wt% vanadium and different carbon contents were prepared using casting process. The effects of carbon on wear properties of HVHSS were studied using pin-on-ring tester, and the failure behaviors were investigated via SEM. Results show the optimal wear resistance is obtained when HVHSS possesses moderate carbon content (2.58wt.%). The cause is that the matrix microstructure of moderate carbon HVHSS is mainly low-carbon lath martensite with good toughness and high hardness, and it can effectively resist micro-cutting and figure wear at the same time, so the role of high-hardness vanadium carbides (VC) can be played enough because of the strong support of matrix. If carbon content is too low, the wear failure of HVHSS is mainly caused by severe micro-cutting and adhesive wear on contact surface because the matrix microstructure of high speed steel is ferrite with very low hardness, which leads to poor wear resistance. While, the matrix microstructure is mainly composed of high carbon martensite with poor toughness when carbon content is too high, therefore, it possesses very poor resistance to cycle fatigue and thermal fatigue, resulting in decrease of wear resistance.

#### A22 A Study on Tool Wear of Sheet Metal Stamping Die Using Numerical Method: Xuan Zhi Wang<sup>1</sup>; Syed Masood<sup>1</sup>; <sup>1</sup>Swinburne University of Technology

Advanced high strength steels (AHSS) are increasingly used in sheet metal stamping in the automotive industry. In comparison with conventional steels, advanced high strength steels (AHSS) stampings produce higher contact pressures at the interface between draw die and sheet metal blank, resulting in more severe wear conditions, particularly at the draw die radius. Prediction of tool wear pattern for sheet metal stamping die is a highly challenging task as there are many control parameters involved in the production. This paper presents a numerical simulation methodology to analyse the influences of various control parameters on tool wear pattern of a sheet metal stamping die with different die radius are profiles. The numerical model was validated by a channel bending test. The results of tool wear patterns provide informative guidelines for the on-site production.

## A23 Back Bead Characteristics during Butt Welding of Thick Plate for Various Backing Conditions: *Cheolhee Kim*<sup>1</sup>; <sup>1</sup>KITECH

In the welded structure, the thick plates were joined by multi-pass welding in butt joint. During the first pass of multi-pass welding, the burn through, lack of fusion and incomplete penetration were easily found as weld defects. So the backing condition should be carefully selected in welding of thick plate because improper backing conditions lead to weld defects. In the job site, the steel backing plate is usually adopted although it reduces the fatigue strength. No backing condition or removable backing would be recommended to increase the fatigue strength but selecting proper backing and welding conditions is complicated. Several backing method, such as the ceramic backing, the water-cooled copper backing and even no backing method, were investigated during GMA welding of thick steel plate in this study. The gas metal arc welding conditions were established for each backing method, and the bead shape and mechanical property was examined after welding.

#### A24 Cold Rolling Process Experimental Research of Difficult-to-Deform Steel: *Jiao Zhijie*<sup>1</sup>; Li Jianping<sup>1</sup>; Sun Jie<sup>1</sup>; <sup>1</sup>State Key Lab of Rolling and Automation

Difficult-to-deform steel, such as austenitic stainless steel and silicon steel, has the work-hardened phenomenon. During cold rolling process, rolling force will increase quickly with accumulated reduction increasing. Especially during cold rolling process experimental researching work, traditional two-high or fourhigh reverse pilot cold rolling mill is used; tension can't be generated. For the work-hardened phenomenon, rolling force will increase quickly, equipment load will be serious, and strip shape will be difficult to control. A new type pilot cold rolling mill was designed; the hydraulic cylinders and clamps are used to tension rolling piece. With tension, rolling force can be decreased and production shape



is easy to control during rolling experiment. Using this new type pilot cold rolling mill, the austenitic stainless steel, silicon steel, TRIP steel and other difficult-to deform steels are rolled. The actual technological data such as roll gap, rolling force, and roll speed can be measured. And with these data, the reduction, friction coefficient, deformation resistance and other process parameters can be calculated.

## **A25** Torsion Textures Simulation by Considering Rigid Body Rotation: *Liu Yandong*<sup>1</sup>; <sup>1</sup>Northeastern University

In this paper, the torsional texture evolution of pearlitic steel wire is simulated by Full Constrains (FC) Taylor model. The simulation results indicate that a simple shear deformation can not explain the experiments results of torsion texture. Bring in additional rigid body rotation and rational shear deformation can emersion the torsion texture very well. The different deformation styles are imposed to the FC Taylor, a group of parameters is given to explain the torsion texture evolution.

### A26 Effect of CuS on the Fatigue Properties of Bake Hardening Steels: *Ildong Choi*<sup>1</sup>; Sunggyu Kang<sup>1</sup>; Jihyun Jang<sup>1</sup>; Sungbok Lee<sup>2</sup>; Moonhi Hong<sup>2</sup>; <sup>1</sup>Korea Maritime University; <sup>2</sup>POSCO

Bake hardening steels have to resist strain aging to prevent the yield strength increment and stretcher strain during press process and to enhance the bake hardenability during baking process after painting. The bake hardening steels need to control the solute carbon and the solute nitrogen to improve the bake hardenability. Ti and/or Nb alloying for nitride and carbide precipitation and low carbon content below 0.003% are used to solve strain aging and formability problem for automotive materials. However, in the present study, the effect of micro-precipitation of CuS on the fatigue properties of extremely low-carbon steel has been investigated. The bake hardenability of Cu-BH steel is slightly higher (5MPa) than that of Nb-BH steel, but the fatigue limit of Cu-BH steel is far higher (45MPa) than that of Nb-BH steel. All samples show the distinct fatigue stages, such as crack initiation, stable crack growth and unstable crack growth.

#### A27 Effect of Grain Boundary Serration on the Tensile Properties of the Super 304H Heat Resistant Austenitic Stainless Steel: *Sung Min Hong*<sup>1</sup>; Dong-Joon Min<sup>2</sup>; Eric Fleury<sup>1</sup>; <sup>1</sup>KIST, Center for High Temperature Energy Materials; <sup>2</sup>Yonsei University

Heat resistant austenitic steels are candidate materials primarily for the superheater /reheater tubing in ultra-super critical (USC) power plant, where oxidation resistance and corrosion become important in addition to creep strength. Recently Cu nano-size precipitates have been introduced in austenitic steel to increase the creep properties up to about 750°C. However further improvement for the high temperature properties is required to enhance the durability of the superheater/reheater tubes. A special heat-treatment was thus applied with the aim of producing serrated grain boundaries. In this article, we will present the effect of the grain boundary serrations produced by a 30minutes holding time at 700°C after solution heat treatment at 1200°C, on the tensile and creep properties of 0.1C,18Cr,9Ni,0.4Nb,0.2Si,0.8Mn,0.1N,3Cu (wt%) austenitic steel in the temperature range up to 750°C. Results of tensile tests indicated that the serrated grain boundaries provide on enhancement of the ductility particularly at high temperature (750°C). Further tests are currently in progress to understand the role of the grain boundary serration on the mechanical properties of Cu containing heat resistant austenitic stainless steel.

## **A28 Effect of Hard Phase on the Texture Evolution of Subgrain in IF and DP Steels during Biaxial Deformation**: *Do Hyun Kim*<sup>1</sup>; Seoung-Bum Son<sup>1</sup>; Jung Han Kim<sup>1</sup>; Heung Nam Han<sup>1</sup>; Kyu Hwan Oh<sup>1</sup>; <sup>1</sup>Seoul National University

Subgrain textures of interstitial free (IF) and dual phase (DP) steels were compared during biaxial tensile deformation. Deformation stage for biaxial tension was specially designed for this study and electron backscattering diffraction (EBSD) was used to perform texture measurement with increasing biaxial strain. The martensite which exists on the ferrite boundaries was uniformly distributed in DP steel. Several individual ferrite grains were selected to measure the average orientations, local misorientations, orientation spread of grains with increasing strain, and these experimental data were compared between IF and DP steels to analyze the hard phase effect.

#### A29 Effect of Punch Deformation Velocity on Plate by FEM Method: Hyunchul Roh<sup>1</sup>; Eu Sun Yu<sup>1</sup>; Tae Jun Ko<sup>1</sup>; Xiaodong Mao<sup>1</sup>; Kyu Hwan Oh<sup>1</sup>; <sup>1</sup>Seoul University

Recently, deformation velocity became an important parameter because of the large size surbo-press machine. We adopted FEM method to make the light weight car body according to increasing use of aluminum and high tensile steel sheet so to know how to increase the deformability. Through this experiment, it was proven that the high velocity makes the concentrated deformation on local place and this leads to the decreased changes of thickness reductions. We observed the generations of necking and crack to use this data to decide the deformability according to the increased punch's mpm from 3 to 30. It was shown that high tensile test speed makes the increase of the yield stress and UTS that makes the strengthening of the material. As a result, thin and high FLD sheet had a pure deformability. We confirmed this result by conducting experiments and the result of the FEM simulation had a similar tendency of the experiment. Conclusively, the high punch velocity makes concentration of the stress on the rugged part and this reads to the high percentage of the deformation like crack or necking.

#### A30 Effect of Rate of Heat Input on Microstructural Features and Mechanical Properties of Medium Chromium Ferritic Stainless Steel Welds: *Muhammed Amuda*<sup>1</sup>; Shahjahan Mridha<sup>1</sup>; <sup>1</sup>International Islamic University Malaysia

Medium chromium ferritic stainless steel conforming to AISI 430 was TIGwelded with different energy input rates 205J/mm = HI = 2304J/mm in argon shielding environment and characterized for microstructural features as well as mechanical properties. Macro profiling of the weld section revealed greater distortion with wider HAZ at energy input rate greater than 1576J/mm corresponding to welding current range 70-100A and welding speeds of 1-3.5mm/s. XRD and OM characterization of the weld section revealed the presence of high temperature delta ferrite and intragranular carbide precipitates in the weld whose percent distribution decreases with increase in heat input rate but with an increase in the amount of intergranular carbide precipitates. The weld grain structure changed from columnar grains to equiaxed grains as the welding speed increases most significantly between 2.5 and 3.5mm/s for a given welding power. The energy input generally lead to lower mechanical property relative to the base metal but the decrease is more pronounced in welds made with energy input greater than 1576J/mm due to the increased intergranular carbide precipitation at the grain boundary. SEM observation of the fractured surface revealed transition between dimple and cleavage fracture mode as the heat input rate increases.

#### A31 Hot-Working Behaviour of Advanced High-Manganese C-Mn-Si-Al Steels: *Leszek Dobrzanski*<sup>1</sup>; Wojciech Borek<sup>1</sup>; <sup>1</sup>Silesian University of Technology

The high-manganese austenitic steels are an answer for new demands of automotive industry concerning the safety of passengers by the use of materials absorbing high values of energy during collisions. The chemical compositions of two high-manganese austenitic steels containing various Al and Si concentrations were developed. Additionally, the steels were microalloyed by Nb and Ti in order to control the grain growth under hot-working conditions. The influence of hotworking conditions on a recrystallization behaviour was investigated. Flow stresses during the multistage compression test were measured using the Gleeble 3800 thermo-mechanical simulator. To describe the hot-working behaviour, the steel was compressed to the various amount of deformation (4x0.29, 4x0.23 and 4x0.19). The microstructure evolution in successive stages of deformation was determined in metallographic investigations using light microscopy. The flow stresses are much higher in comparison with austenitic Cr-Ni and Cr-Mn steels and slightly higher compared to Fe-(15-25) Mn alloys. Making use of dynamic and metadynamic recrystallization, it is possible to refine the microstructure and to decrease the flow stress during the last deformation at 850°C. Applying the true strains of 0.23 and 0.19 requires the microstructure refinement by static recrystallization. The obtained microstructure - hot-working relationships can be useful in the determination of powerful parameters of hot-rolling and to design a rolling schedule for high-manganese steel sheets with fine-grained austenitic structures.

## A32 Research on the Tempering Kinetics of High Strength Steels by In-Situ Resistance Measurement: Xianwen Lu<sup>1</sup>; *Hongyan Li<sup>1</sup>*; Heping Liu<sup>1</sup>; Xuejun Jin<sup>1</sup>; <sup>1</sup>Shanghai Jiaotong University

The resistance change during the tempering of medium-carbon high strength steels was examined in the range of 450~650°C. Tempering kinetics were obtained from measured resistance change during tempering. From the tempering kinetics data, isothermal transformation diagram of the investigated steel was determined and a tempering kinetic model was proposed. The results obtained by in-situ transmission electron microscopy(TEM) will be compared to those by electric resistance measurement for a better understanding of the precipitation behaviour of high strength steels during tempering. In-situ resistance measurement was proved to give a possibility of non-destructive, facility tempering monitoring technique.

**A33 Slide-Bend Forming of Very Thin Metal Sheet Using Slide-Ironing Tool**: Ryo Nakagawa<sup>1</sup>; Takeharu Matsuno<sup>1</sup>; *Yasuo Marumo*<sup>1</sup>; Yuya Hayano<sup>1</sup>; Liqun Ruan<sup>1</sup>; Hidetoshi Sakamoto<sup>1</sup>; Hiroshi Harada<sup>1</sup>; <sup>1</sup>Kumamoto University

Small parts with very thin thickness are commonly fabricated from thin sheets through many forming operations including stretching, deep drawing, ironing, bending and so on. The thinning of sheet thickness causes critical problems on the defects and accuracy of the formed parts. In this study, the features of the slidebend forming of metal foils were studied, in which a simple-shaped tool like a bar or a plate is slid on the surface of a foil workpiece 0.01-0.05mm thick which is on



a flat tool. In the experiments, the bending formation of metal foil with 0.01mm-0.05mm thickness was conducted using several kinds of metal foils. The effects of the tool indentation and the foil thickness on the bending angle of the metal foil were clarified. The relationship between these factors and formability was found and appropriate conditions of bending foil were obtained.

#### A34 Possibility of Removing Platinum from the Used Catalytic Converters in Pyro- and Hydrometallurgical Processes: *Agnieszka Fornalczyk*<sup>1</sup>; Mariola Saternus<sup>1</sup>; <sup>1</sup>Silesian University of Technology

The paper presents characteristics of catalytic converters used in cars and the review of available technologies during recycling process. The possibility of removing platinum from the used catalytic converters applying pyrometallurgical and hydrometallurgical methods were also investigated. Metals such as copper and magnesium were used in the pyrometallurgical research. In the first test the catalytic converters carrier was crushed and located in the basket with copper. During the high temperature process in the carrier was melted with copper As a result platinum was separated from the ceramic substrate. In the second test, magnesium vapours were blown through the whole carrier located in the pit furnace. Three methods of hydrometallurgical platinum recovery from catalytic converters were investigated. In the first test the crushed catalytic converter was treated with aqua regia. In the second test the mixture of acids HF, HCl and HNO, was used as a solving agent. The last test was based on the leaching the crushed material in H<sub>2</sub>SO<sub>4</sub> and NaOH solution. Analysis of platinum contents in the carrier before and after the process was performed by means of atomic absorption spectroscopy. Obtained result were discussed.

#### A35 Effect of Microshot Peening on Surface Characteristics of Spring Steel: Yasunori Harada<sup>1</sup>; *Koji Yoshida*<sup>1</sup>; <sup>1</sup>University of Hyogo

Shot peening is a surface treatment that improves the performance of engineering components. In conventional shot peening, the medium consists of small spheres, which are usually made of high-carbon cast steel; the diameter of the spheres is in the range from 0.3 to 1.2mm. More recently, however, a new type of microshot has been developed to enhance the peening effect. The diameter of the spheres in the new medium is in the range from 0.02 to 0.15mm. In this study, the effect of microshot peening on the surface characteristics of spring steel was investigated. The projective method of the microshot was of the compressed air type. The microshots of 0.1mm diameter were high-carbon cast steel and cemented carbide, and the workpiece used was the commercially spring steel JIS-SUP10. The surface roughness, hardness and compressive residual stress of the peened workpieces were measured. The surface layer of the workpieces was sufficiently deformed by microshot peening. A high hardness or residual stress was observed near the surface. The use of hard microshots such as cemented carbide was found to cause a significantly enhanced peening effect for spring steel.

## A36 The Effect of Shot Peening Operation on Fatigue Behavior of Austempered Ductile Iron (ADI): Sasan Yazdani<sup>1</sup>; Amir Sadighzadeh Benam<sup>1</sup>; Behzad Avishan<sup>1</sup>; <sup>1</sup>Sahand University of Technology

Shot peening is one of the most common surface treatments to improve the fatigue behavior of metallic parts. In this study the effect of shot peening operation on the fatigue behavior of an alloyed austempered ductile iron (ADI) has been studied. Austempering heat treatment consisted of austenitizing at  $875^{\circ}$ C for 90 min. followed by austempering at three different temperatures of 265, 320 and  $400^{\circ}$ C. Rotating–bending fatigue test was carried out on samples after shot peening by 0.4 - 0.6 mm shots. XRD, SEM, micro hardness and roughness tests were used to study the fatigue behavior of the samples. Results indicate that the fatigue strength of samples austempered at 320, 365 and 400°C were increased by 27.3%, 33.3% and 48.4% respectively after shot peening operation.

## **A37** Unconventional Method of Nitriding of 316l Austenitic Steel: *Jaroslaw Jasinski*<sup>1</sup>; Tadeusz Fraczek<sup>1</sup>; Michal Olejnik<sup>1</sup>; <sup>1</sup>Czestochowa University of Technology

In this study 316L austenitic steel after glow discharge nitriding at a temperature of T=673 K, for 4h and two different variants of specimen arrangement in the glow-discharge chamber was investigated. According to variant 1, specimens were placed on the cathode and for variant 2, specimens were placed also on the cathode, but were shielded with a booster screen. In order to assess the effectiveness of nitriding process variants, the profile analysis of obtained surface layers, surface hardness tests and surface layer hardness profile examination, the analysis of surface layer structures, abrasive wear and corrosion resistance tests were carried out. It has been found that application of booster screens for 316L austenitic steel nitriding process, effects in nitrogen diffusion depth increment and thus an increment of surface layer thickness.

A38 Role of Microstructure in Susceptibility of X70 Pipeline Steel to Hydrogen Embrittlement: *Daniel Hejazi*<sup>1</sup>; Ayesha Haq<sup>1</sup>; Elena Pereloma<sup>1</sup>; Druce Dunne<sup>1</sup>; Andrzej Calka<sup>1</sup>; <sup>1</sup>University of Wollongong

Hydrogen can be picked up in steel processing, during fabrication by welding and as a result of in-service exposure to a hydrogen-rich environment. The presence of diffusible hydrogen of only a few ppm is known to induce cold cracking at stress concentrations. Microstructures with more hydrogen traps can take hydrogen atoms out of circulation at low temperatures and hence reducing the susceptibility to HACC (hydrogen assisted cold cracking). In order to investigate the susceptibility of steels to hydrogen embrittlement as a function of their microstructure X70 steel was chosen in different conditions: normalized transfer bar, as-received hot rolled strip and heat affected zone (HAZ). HAZ simulations were conducted using Gleeble thermo-mechanical machine to simulate a thermal cycle typical of ERW( Electro resistance welding)Notched by wire cutting and fatigue pre-cracked samples were subjected to electrochemical hydrogen charging using a solution of H<sub>2</sub>SO<sub>4</sub> (1N) and NaAsO<sub>2</sub> (250mg/L) for various times in order to achieve 2 ppm hydrogen content. Three point bend tests were conducted on asreceived and hydrogen charged samples. The J1c method was selected to measure the fracture toughness. Fractography and fracture toughness of the samples are compared and discussed.

## A39 Mechanical Properties and Marine Corrosion Resistance of P- Bearing ULCB Steels: *Wenfang Cui*<sup>1</sup>; Chunming Liu<sup>1</sup>; <sup>1</sup>Northeastern University

The present paper deals with P-bearing ultra low carbon bainitic steels (ULCB) which were developed for offshore oil platform. The research aims to evaluate the effects of Mo, P and B content on the microstructure, mechanical properties and marine corrosion resistance of the steels. The results show that the higher Mo and B content lead to the formation of lath bainite with high strength and low impact toughness. The microstructure of Mo free steel was quasi poly-ferrite which behaved low strength and high impact toughness. When adding 0.21(mass) % Mo and 0.0038(mass) % B in the steel, granular bainitic microstructure was obtained and the tensile strength, ductility and impact toughness of the steel reached an optimum match. 0.09(mass) % P content in the steel obviously increased yield strength and decreased immersion corrosion rate in 3.5%NaCl in comparison with the steels without or with lower P content. This is attributed to that the complex effect of Cu and P promoted the formation of compact inner scale, which inhibited further corrosion from NaCl solution. Mo element played an important role in avoiding pitting corrosion of ULCB steel.

A40 In Situ Observation of Liquid to Solid Phase Transformation in Q235 Steel Prepared by Electric Pulse Modification Technology: Jianzhong Wang<sup>1</sup>; Jingang Qi<sup>1</sup>; Bing Wang<sup>1</sup>; Zuofu Zhao<sup>1</sup>; <sup>1</sup>Liaoning University of Technology

The modification of liquid metal by electric pulse (EP, EPM) is a novel method for grain refinement, but the EPM mechanism is still a hypothetical theory and far from being fully understood. The direct observation on solidification process of EP-modified metal will obviously benefit this mechanism investigation. In this study, the process of liquid to solid phase transformation in Q235 steel prepared by EPM technology was in situ observed by using a confocal laser scanning microscope (CLSM) combined with an infrared image furnace. It is shown that the EP-modified Q235 melt exhibits an increasing and homogeneous nucleation density during a concentric solidification, and the morphology of liquid-solid interface changes from river-like (No EP) to arc-like. The observed growth velocity of peritectic reaction is 2.8µm/s on an average, showing a sharp decrease compared with those of the unmodified. Furthermore, the morphology of dproduct varies from a dimple-shape of the unmodified to a plane-like in the EP-modified. These observations and analysis experimentally verify the proposed Wang's EPM model and illuminate in situ the effects of EP-modified liquid metal on its liquid to solid phase transformation process.

#### A41 Thermodynamic Relation between Chromium and Aluminum in Liquid Iron: Jong-Oh Jo<sup>1</sup>; Moon-Sic Jung<sup>1</sup>; Min-Kyu Paek<sup>1</sup>; Jong-Jin Pak<sup>1</sup>; <sup>1</sup>Hanyang University

Aluminum is one of the most important alloying elements used for deoxidizing liquid steels. However, thermodynamics of aluminum deoxidation in liquid stainless steels are not fully understood because the thermodynamic relation between aluminum and chromium at high Cr content in liquid iron is not well known. In this study, Thermodynamic relation between chromium and aluminum in liquid iron was studied by the metal-nitride-gas equilibration technique. The effect of chromium on the equilibrium solubility product of AlN in liquid Fe-Cr alloys containing chromium up to 27 mass% was measured under reduced nitrogen partial pressures in the temperature range from 1873 to 1973K. Using available thermodynamic relations between nitrogen and alloying elements of Cr and Al, the effect of chromium on the activity coefficient of titanium in Fe-Cr-Al-N melts was determined.



A42 Thermodynamic Relation between Chromium and Titanium in Liquid Iron: *Jong-Oh Jo*<sup>1</sup>; Min-Kyu Paek<sup>1</sup>; Won-Kyu Lee<sup>1</sup>; Jong-Hyun Park<sup>1</sup>; Jong-Jin Pak<sup>1</sup>; <sup>1</sup>Hanyang University

The thermodynamic relation between chromium and titanium was studied by measuring the effect of chromium on the solubility product of TiN in Fe-Cr-Ti-N melts containing a wide range of chromium up to 26 mass% in the temperature range from 1873 to 1973K. Using Wagner's formalism1, the first- and the second-order interaction parameters between chromium and titanium were determined as a function of temperature from the experimental results. The validity of thermodynamic data determined in the present study was examined by measuring the critical titanium and nitrogen contents for TiN formation in a commercial ferritic stainless steel melt during cooling from 1923K to 1793K.

#### A43 Understanding the Effects of Na<sub>2</sub>F and CaF<sub>2</sub> on the Viscosity of TRIP Mold Fluxes: *Hyuk Kim*<sup>1</sup>; Jin Kyun Park<sup>1</sup>; Il Sohn<sup>1</sup>; <sup>1</sup>Yonsei University

Due to the continued demand for lightweight advanced high strength steels, TRIP steels has become one of the most actively researched topic in the steel industry. In particular, the continuous casting engineer actively plays an essential role in providing and developing mold fluxes that are not detrimentally affected by the reaction of some of the elements in TRIP steel composition. Many combinations of mold flux chemistry have been provided to the steel industry, some with minor success but continuous operation beyond a certain number of heats is still limited in the industry. Typical chemical compositions of TRIP mold fluxes have flux basicities of less than 0.9 and significant amounts of CaF<sub>2</sub> and NaF<sub>2</sub> to maintain high fluidity in the flux and also controlled heat transfer. However, these fluorine compounds have environmental issues and corrosion problems below the mold and may eventually have to be replaced. In this study, the effect of CaF<sub>2</sub> and NaF<sub>2</sub> on the properties of TRIP mold flux has been studied to provide a fundamental understanding for future substitution of these raw materials. In particular, the effect on the viscosity and crystallization behavior has been studied.

#### Poster Session: Symposium B: Advanced High Temperature Structural Materials

Tuesday PM	Room: Hall 2
August 3, 2010	Location: Cairns Convention Centre

**B1 Alloying Behavior of Ni<sub>3</sub>Nb, Ni<sub>3</sub>V and Ni<sub>3</sub>Ti Compounds**: *Hotaruko Sugimura*<sup>1</sup>; Yasuyuki Kaneno<sup>1</sup>; Takayuki Takasugi<sup>1</sup>; <sup>1</sup>Osaka Prefecture University

The site preference of ternary additions in Ni,X-type GCP compounds was determined from the direction of solubility lobe of the GCP phase on the experimentally reported ternary phase diagrams. In Ni, Nb (D0,), Co and Cu preferred the substitution for Ni-site, Ti, V and W the substitution for Nb-site, and Fe the substitution for both sites. In Ni<sub>3</sub>V (D0<sub>22</sub>), Co preferred the substitution for Ni-site, Cr the substitution for both sites, and Ti the substitution for V-site. In Ni, Ti  $(D0_{24})$ , Fe, Co, Cu, and Si preferred the substitution for Ni-site, Nb, Mo and V the substitution for Ti-site. The thermodynamic model, which was based on the change in total bonding energy of the host compound by a small addition of ternary solute, was applied to predict the site preference of ternary additions. The bond energy of each nearest neighbor pair used in the thermodynamic calculation was derived from the heat of compound formation by Miedema's formula. The agreement between the thermodynamic model and the result of the literature search was excellent. Both transition and B-subgroup elements have two possibilities, *i.e.*, the case of substitution for Ni-site or the case for X-site, depending on the relative value of two interaction energies

B2 Characterization of Precipitates in a Type 304 Austenitic Stainless Steel Containing Niobium and Vanadium: Dae Bum Park<sup>1</sup>; Moo-Young Huh<sup>1</sup>; Woo Sang Jung<sup>2</sup>; Seung-Cheol Lee<sup>2</sup>; Dong Hee Lee<sup>3</sup>; <sup>1</sup>Korea University; <sup>2</sup>Korea Institute of Science and Technology; <sup>3</sup>POSCO Specialty Steel Researches on the 18Cr-8Ni stainless steels for the boiler tube have been

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investigated due to its high strength and good corrosion properties. Their mechanical properties are dependent on the formation and growth of precipitates. There are several well-known precipitates in the matrix and at grain boundaries: Carbonitride such as MX,  $M_2X$ ,  $M_6X$  and  $M_{23}C_6$ , intermetallic compounds such as Laves phase,  $\chi$ -phase and  $\mu$ -phase and so on, metallic phase such as Cu. In this study, we investigate the precipitate formation during the solid solution heat treatment and the creep test for 18Cr-8Ni-0.3Nb-0.3V steels. To observe the morphology and the distribution of the precipitate, the high-resolution transmission electron microscopy (HRTEM) is used. From our results, it is observed that the MX-typed Nb-rich precipitate is formed during the solid solution heat treatment,

and then the V-rich carbonitride,  $M_{23}C_6$ , Cu-rich precipitate is also formed during the creep tests. The result will be discussed in detail.

**B3** Construction of a High Temperature Grade 91 Sodium Component Test Loop Evaluation: *Hyeong-Yeon Lee*<sup>1</sup>; Jae-Han Lee<sup>1</sup>; Jae-Hyuk Eoh<sup>1</sup>; Tae-Ho Lee<sup>1</sup>; Yong-Bum Lee<sup>1</sup>; <sup>1</sup>Korea Atomic Energy Research Institute

A sodium test facility of 'CPTL'(Component Performance Test Loop) for simulating thermal hydraulic behavior of the Korean demonstration fast reactor components such as IHX(Intermediate Heat Exchanger), DHX(Decay Heat Removal Heat Exchanger) and sodium pump under development by KAERI(Korea Atomic Energy Research Institute) is to be constructed. The design temperature of the test loop is 600°C and design pressure is 1MPa. The heat exchangers are made of Grade 91 steel and high temperature design evaluations are carried out and design evaluations are carried out with the high temperature design codes of the ASME Section Subsection NH (nuclear grade) and ASME Section VIII Division 1(non-nuclear grade), and the conservatisms of the two design codes are quantified. In this paper, the overall preliminary design features of the CPTL are introduced, comparisons of the two design codes are conducted for a heat exchanger in terms of code conservatism and issues on high temperature design for a Grade 91 steel structure are raised.

**B4** Creep Behavior for Alloy 617 in Air of 950°C: *Woo-Gon Kim*<sup>1</sup>; Song-Nan Yin<sup>1</sup>; Gyeong-Geon Lee<sup>1</sup>; Yong-Wan Kim<sup>1</sup>; <sup>1</sup>Korea Atomic Energy Research Institute

Creep behavior for Alloy 617, which is considered as one of major structural materials of a very high temperature gas-cooled reactor (VHTR), was investigated in air at 950°C. Fracture morphologies were observed from fractured specimens after the creep tests. Creep experimental data was obtained by a series of creep tests with different stress levels at 950°C. Alloy 617 revealed little primary creep strains and unclear secondary creep stage. A tertiary creep stage was initiated from a low strain level and dominant in full creep curves. It revealed a sufficient ductility (>30%) in spite of the creep duration of 14,100h. The creep constants of A, n, m, and C in Norton's power law and Monkman-Grant relationships were determined. From microstructure observations of crept specimens, it was found that a  $Cr_2O_3$  oxidation layer was formed on the surface, and just beneath the  $Cr_2O_3$  layer, a thin internal sub layer oxide was formed with rod shapes. And, below the thin internal sub layer, a thick carbide-depleted zone was developed by a reaction of the chromia and carbide precipitates.

**B5** Effects of Electropulsing Treatment on the Precipitation Behaviour of Grain Boundary Carbides in GH3044 Alloy: *Yang Liu*<sup>1</sup>; Lei Wang<sup>1</sup>; Yuchen Wang<sup>2</sup>; Hongyan Liu<sup>1</sup>; Xuejiao Chen<sup>1</sup>; Yan Yu<sup>1</sup>; <sup>-1</sup>Northeastern University; <sup>2</sup>Fushun Special Steel Co., Ltd

The effects of electropulsing treatment on the precipitation behaviour of grain boundary carbides in GH3044 alloy were investigated. The results showed that the initial temperature of precipitation of  $M_{23}C_6$  type carbides on grain boundary could be decreased by electropulsing treatment under the condition of high current density of 10.0kA/mm<sup>2</sup> with a frequency of 5Hz and pulse width of 15  $\mu$ s. The volume percentage of  $M_{23}C_6$  type carbide was greatly increased to 274.60% as compared with that of the aging treatment at the same temperature. However, the precipitation of  $M_{23}C_6$  type carbide was inhibited by electropulsing treatment under the condition of high frequency of 45Hz with the current density of 2kA/ mm<sup>2</sup> and pulse width of 15 $\mu$ s. The volume percentage of  $M_{23}C_6$  type carbide was decreased to 18.81% as that of the aging treatment at the same temperature. It has been found that the diffusion of solute atom in the alloy can be promoted by the electric effect with the electropulsing. As a result, the thermodynamic condition and dynamic condition of the precipitation of  $M_{23}C_6$  type carbide were changed, and its initial and peak temperatures of precipitation were decreased.

## **B6 Fretting Wear Resistance of ATW Cr<sub>3</sub>C<sub>2</sub>/Ni<sub>3</sub>Al Composite Hardfacing on IC6SX**: *Li Shusuo*<sup>1</sup>; Han Yafang<sup>1</sup>; <sup>1</sup>Beihang University

The present study has been carried out in order to investigate the fretting wear performance of surface wear-resistant  $Cr_3C_2/Ni_3Al$  composite deposited onto IC6SX alloy by manual argon tungsten-arc welding. Tests were conducted on both the unwelded and welded substrate under the same fretting condition at RT, 500°C and 800°C in order to evaluate the fretting wear resistant property of the welding layer at different temperatures. It has been determined that the welded substrate exhibits a very good tribological performance in comparison to the unwelded substrate at 500°C and 800°C, with 51.5% and 57.8% decrease of the fretting sectional area respectively. The increase in the fretting wear resistance provided by the welding layer has been attributed to the presence of a large amount of dispersed Mo and Cr carbides in the Ni<sub>3</sub>Al matrix. The fretting sectional area of the welded substrate is larger than that of the unwelded substrate at RT indicating poor fretting resistance as the reinforce phases of the welding layer is harder and more harmful to the sample surface.



**B7** Study of a New Type High Strength Ni-Based Superalloy DZ468 with Good Hot Corrosion Resistance: Zheng Zhi<sup>1</sup>; Liu Enze<sup>1</sup>; Yu Yongsi<sup>1</sup>; Zhu Yaoxiao<sup>1</sup>; 'IMR,CAS

The mechanical property, hot corrosion resistance and phase stability of a new-type directional solidification nickel-base superalloy named DZ468 that was developed independently by Institute of Metal Research, Chinese Academy of Sciences were investigated in this paper. The rupture properties of DZ468 alloy are nearly the same as those of DZ4125 alloy and its hot corrosion resistance property nearly the same as that of IN738 alloy under the same conditions. There is no finding TCP phase in the DZ468 alloy after aging at 900°C for 1000h. DZ468 alloy displays excellent phase stability.

#### **B8** Study of Sintering Behavior of Ni-Al<sub>2</sub>O<sub>3</sub> FGM Depending on Different Particle Size Variation: *Dong-guk Cho*<sup>1</sup>; Seung-kyu Yang<sup>1</sup>; Jun-chul Yun<sup>1</sup>; Jaisung Lee<sup>1</sup>; Caroline Sunyong Lee<sup>1</sup>; Jae-Chul Lee<sup>2</sup>; <sup>1</sup>Hanyang University; <sup>2</sup>Seoul National University

Ni-Al<sub>2</sub>O<sub>3</sub> composite has been widely studied to improve the strength and toughness of ceramic matrix by dispersing small percentage (~5wt%) of Ni inclusions. To fabricate Ni-Al<sub>2</sub>O<sub>3</sub> FGM where high percentage of Ni powders are incorporated, understanding sintering behavior at various compositions is critical. Therefore, sintering behavior at various compositions of Ni-Al<sub>2</sub>O<sub>2</sub> FGM was studied along with the effect of different particle size. The different sizes of Ni powders used were 100nm and 3nm while Al<sub>2</sub>O<sub>2</sub> powders being 160 nm and 18um respectively. It was found that the particle size affects sintering characteristic. Especially, 18µm-sized Al<sub>2</sub>O<sub>3</sub> powders barely sintered at 1350°C, whereas 100nm-sized Ni powders resulted in agglomerations with few micrometers in diameter. This variation in size changed matrix being ALO. instead of Ni. In case of using 100nm-sized Ni powders, the dispersion of Al<sub>2</sub>O<sub>3</sub> and Ni improved greatly compared to using 3µm-sized Ni powders in 60wt% Ni component of FGM. However, the density of the sample using 100nm-sized Ni powders dropped considerably. Density improvement of composite can be achieved through optimized combination of Nano-sized and Micron-sized Ni powders so that crack problems occurred in FGM fabrication due to density difference between two types of powders, can be minimized.

## **B9 The Effect of Grain Size on Cutting Force in End Milling of Inconel 718C**: *Zhilong Zhao*<sup>1</sup>; Changhui Ai<sup>1</sup>; Lin Liu<sup>1</sup>; <sup>1</sup>Northwestern Polytechnic University

Inconel 718C is a nickel-base alloy that is difficult to machine. This paper presents a study of the influence of grain size in as-cast workpieces on cutting forces of Inconel 718C. The end milling tests were performed to understand the effect of various grain structures on machinability of nickel-base superalloy under wet condition using carbide insert. The collected data of cutting forces were analyzed using polynomial regression methods. The results show that grain refining of Inconel 718C can effectively decrease cutting force and improve the machinability of nickel-base superalloy.

### **B10 The Effect of Refractory Elements on Microstructure and Mechanical Properties of Ni<sub>3</sub>(Si,Ti) Intermetallic Alloys:** *Akiko Kai*<sup>1</sup>; Daiki Imajo<sup>1</sup>; Yasuyuki Kaneno<sup>1</sup>; Takayuki Takasugi<sup>1</sup>; <sup>1</sup>Osaka Prefecture University

The microstructures, mechanical properties and cold workability of quaternary Ni<sub>3</sub>(Si,Ti) intermetallic alloys with L1, structure, which were alloyed with two atomic percent of a refractory element X (X: Hf, Ta and W), were investigated. The Ta-added Ni<sub>3</sub>(Si,Ti) alloy showed an L1, single phase microstructure, while the microstructure of the Hf-added alloy was comprised of Ni Hf and/or Ni.Hf intermetallic dispersions in the L1, matrix, and that of the W-added allow consisted of fcc Ni solid solution phase within the L1, grain. In the homogenized condition, the hardness increased in the order of the Hf-added, the Ta-added and the W-added alloys. The hardening of the Hf-added and the Ta-added alloys was attributed to second-phase dispersion hardening and distinctive solid solution hardening, respectively. Among these alloys, only the W-added alloy was successfully cold-rolled to thin sheet with a thickness of 200 um. It was found that both room-temperature and high-temperature tensile strength of the W-added alloy sheet were enhanced compared with that of the unalloyed Ni<sub>2</sub>(Si,Ti) sheet. Also, high-temperature tensile ductility was significantly improved in the Wadded alloy sheet, by suppressing the propensity of brittle intergranular fracture.

# B11 The Effect of Ti Addition on Phase Equilibria among Ni (A1), Ni<sub>3</sub>Al (L1<sub>2</sub>), Ni<sub>3</sub>V (D0<sub>22</sub>) Phases: *Eiki Hayashi*<sup>1</sup>; Satoru Kobayashi<sup>2</sup>; Kazuhisa Sato<sup>2</sup>; Toyohiko Konno<sup>2</sup>; Yasuyuki Kaneno<sup>1</sup>; Takayuki Takasugi<sup>1</sup>; <sup>1</sup>Osaka Prefecture University; <sup>2</sup>Osaka Center for Industrial Materials Research, Institute for Materials Research, Tohoku University

The effects of alloying addition on phase equilibria among Ni (A1), Ni<sub>3</sub>Al (L1<sub>2</sub>) and Ni<sub>3</sub>V (D0<sub>22</sub>) phases at 950 °C were investigated by means of TEM/EDS analysis and DSC on heat-treated alloys. The three-phase coexisting region of A1 + L1<sub>2</sub> + D0<sub>22</sub> was found to exist around the composition of Ni-4Al-19V (at. %) in the Ni-Al-V ternary system. The addition of Ti, Nb or Ta into the ternary system shifted the three-phase coexisting region to the Ni-rich side. The addition

of Cr or Co, on the other hand, shifted the coexisting region to the Ni-poor side. Ti, Nb and Ta partitioned into the  $L1_2$  phase and the  $D0_{22}$  phase rather than the A1 phase. These results suggest that the A1  $\rightarrow$  L1<sub>2</sub> + D0<sub>22</sub> eutectoid reaction temperature increases (decreases) by addition of Ti, Nb or Ta (Cr or Co). The eutectoid temperatures in the Ni-Al-V-M (M: Ti, Nb, Ta, Cr, Co) systems will be experimentally determined.

#### B12 The Study on Manufacturing and Analysis of High Temperature Properties of Ni Based Superalloys for 5MW Gas Turbine Blade: *Young Seok song*<sup>1</sup>; <sup>1</sup>Doosan Heavy Industries & Construction

Nowadays, the demands of the small sized gas turbine such as 5MW gas turbine engine have been increased. Ni-based superalloys have been widely used for casting material of gas turbine blade since it is required high tensile strength, stress rupture and so on at high temperature. In this 5MW gas turbine for power generation, the turbine blade of hot parts in this gas turbine is hollow type which has complex cooling air passages to cool down the blade. The purpose of this study is to decide the best candidate materials as turbine blade. Therefore, it is necessary to develop a casting process of turbine blade that satisfied the hot deformation characteristics of the Ni-based superalloys. Several superalloys were prepared and investigated by tensile strength, creep rupture, Low cycle fatigue, etc from room temperature to high temperature including the microstructure analysis. As results of these test, we compared IN 738LC, GTD-111, 88Y and another Ni-based superalloy experimentally designed.

### **B13 Microstructure and Properties of a Spray Formed Superalloy Ring**: Zhou Li<sup>1</sup>; Guoqing Zhang<sup>1</sup>; Hua Yuan<sup>1</sup>; <sup>1</sup>Beijing Institure of Aeronatical Materials, China

A nickel base superalloy was spray atomized and deposited, hipped, forged, ring rolled and heat treated. Its microstructure and properties under different condition have been studied in this paper. The spray atomized and deposited superalloy preform has low oxygen pickup, refined grain and microstructure and good inhibited coursing of grain. Spray forming process can improve the forgeability of the alloy because of homogeneity of the chemistry and fineness of microstructure. After hot working, the spray formed superalloy ring has good tensile and stress-rupture properties.

**B14 The Stability of γ'-Co<sub>3</sub>(Al,W) Phase in Co-Al-W Ternary System**: *Yuki Tsukamoto*<sup>1</sup>; Satoru Kobayashi<sup>2</sup>; Takayuki Takasugi<sup>1</sup>; <sup>1</sup>Osaka Prefecture Unversity; <sup>2</sup>Tohoku University

The decomposition of metastable  $\gamma'$ -Co<sub>3</sub>(Al,W) particles in  $\gamma$ -Co matrix during heat treatment at 900°C was investigated in Co-Al-W ternary alloys. Co-Al-W ternary alloys were arc melted, and heat treated at 1300 °C for 20h and 900 °C up to 2000h. Microstructure was observed by means of SEM and composition analysis was performed by EPMA. In the case of alloys existing in the metastable  $\gamma+\gamma'$ +CoAl three-phase coexisting field,  $\gamma'$  cuboidal particles were precipitated within the  $\gamma$ -Co matrix at an early stage of heat treatment at 900 °C. In grain interior, the particles remained even after 2000h at 900 °C. On grain boundaries, however, discontinuous coarsening took place to form  $\gamma+\gamma'$  lamellae, and then CoAl and Co<sub>3</sub>W phases were formed to replace the  $\gamma'$  phase. The effect of alloy composition on the decomposition process will be presented and discussed in terms of supersaturation of alloying elements and diffusion coefficients.

## **B15 Microstructural Characterization of Nb-Al Base ODS Superalloys**: *Shigeharu Ukai*<sup>1</sup>; Akinobu Minami<sup>1</sup>; Shigenari Hayashi<sup>1</sup>; Norihito Sakaguchi<sup>1</sup>; Seiji Miura<sup>1</sup>; <sup>1</sup>Hokkaido University

A Nb metal has unique potential for applying to the advanced high-temperature structural materials because of its high melting temperature and lower specific density than Mo, W and Ta. A disadvantage of the Nb metal is less oxidation resistance associated with a large amount of oxygen solubility. In this study, Nb, Al and Y metal powders were mechanically milled and consolidated by HIP at 1,500 °C and 150 MPa for 0.5 h. The 3.3 at% Y-metal was added to form 1.7 at% Y.O. particles through combining with 5 at% soluble oxygen in Nb matrix. The alloy design of Nb-Al base superalloy is based on strengthening by dispersed Y<sub>2</sub>O<sub>3</sub> particles and oxidation resistant by an alumina scale formation. From a micro-structural analyses by TEM, it was revealed that the manufactured Nb-15Al-1.7Y2O3 (at%) superalloy is composed of two phases: Nb solid solution and Nb<sub>2</sub>Al, as consistent with phase diagram. The Y<sub>2</sub>O<sub>2</sub>-Al<sub>2</sub>O<sub>2</sub> complex oxide is formed in the blocky shape instead of dispersed Y2O3 particles, which could be result of high-temperature consolidation at 1,500 °C during HIP. The mechanical properties of Nb-15Al-1.7Y2O3 superalloy at 1,000°C are also evaluated in relation to its microstructure.

#### B16 Property Responses in Nb-Si-Hf-Ti-Al-W-B-Cr Alloys for High-Temperature Applications: Jiangbo Sha<sup>1</sup>; <sup>1</sup>Beihang University

Multi-component alloys Nb-(11,15)Si-5Hf-30Ti-4Al-4W-2B-(8,16)Cr have been proposed, attempting to develop Nb-Si based alloys with a comprehensive property. The phase constitutions and microstructures, mechanical properties



at room temperature, 1250°C and 1350°C, as well as oxidation resistance at 1250°C, have been characterized. The results show that three phases of the NbSS, Nb5Si3 and Laves Cr2Nb are found in the alloys at a Cr content of 16 at%. With increasing Si and Cr contents the fracture toughness KQ decreases, while the oxidation resistance at 1250°C and strength at 1250°C and 1350°C exhibit an increasing tendency. The 15Si-16Cr alloy shows the highest strength and oxidation resistance, and the lowest toughness; they are 385MPa at 1350°C, 225mg/cm2 at 1250°C for 100 h, and 5.45 MPam1/2 at room temperature, respectively. The 11Si-8Cr alloy only has the highest toughness of 11.92 MPam1/2, its strength and oxidation resistance are the lowest. Finally, the failure modes at room temperature and higher were discussed.

#### **B17 Microstructure Control for Mo5SiB2-Based Alloys by Heat Treatment**: *Seong-Ho Ha*<sup>1</sup>; Kyosuke Yoshimi<sup>1</sup>; Kouichi Maruyama<sup>1</sup>; <sup>1</sup>Tohoku University

Mo-Si-B system has been the focus of research attention since Mo<sub>5</sub>SiB<sub>2</sub> (T<sub>2</sub>), only the ternary compound in the system, has excellent ultra-high temperature yield strength, good oxidation resistance and relatively low density comparable to Ni-base alloys. However, its poor fracture toughness as low as ceramics restricts the practical performance for ultra-high temperature applications. On the other hand, there is the two-phase region of ductile Moss and  $\mathrm{T_2}$  in the Mo-Si-B ternary phase diagram. Therefore, the fracture toughness will be improved by the proper combination of Moss and T<sub>2</sub>. The aim of this study is to investigate the microstructural evolution of Mo-Si-B alloys in and near the Moss and T, two-phase region by heat treatment at 1800°C. The two-phase region is divided into three regions with respect to the primary phases (Moss, Mo<sub>2</sub>B and T<sub>2</sub>) when it is superposed with the liquidus projection that indicates solidification pathways depending on composition. In the as-cast microstructures of the three regions, Moss + Mo<sub>3</sub>Si + T<sub>2</sub> and Moss + T<sub>2</sub> eutectic phases exist in addition to the primary phase. After the heat treatment, Moss-T, two-phase microstructure was successfully obtained and controlled in terms of the size, morphology and distribution of Moss particles.

### **B18 Statistical Analysis of Creep Crack Growth Behavior in Modified 9Cr-1Mo Steel**: *Seon-Jin Kim*<sup>1</sup>; Woo-Gon Kim<sup>2</sup>; Ik-Hee Jung<sup>1</sup>; Yong-Wan Kim<sup>2</sup>; <sup>1</sup>Pukyong National University; <sup>2</sup>KAERI

In this paper, a series of statistical studies were conducted on creep crack growth behavior of modified 9Cr-1Mo steel for next generation reactor. Creep crack growth tests were performed on pre-cracked compact tension (CT) specimens under the applied load ranges of 3800 to 5500N at the identical temperature condition of  $600^{\circ}$ C. The creep crack growth behavior has been analyzed statistically using the empirical equation between crack growth rate (da/dt) and C\* parameter, namely da/dt =B(C\*)q . First, the determination methods of B and q obtained from experiments were investigated by the least square fitting method and mean value method. The probability distribution functions of B and q follow well 2-parameter Weibull distribution. Second, the creep crack growth rate data were generated by Monte-Carlo simulation method assuming the 2-parameter Weibull in B and q parameters. The probability distribution function of creep crack growth rate (da/dt) for arbitrary C\* parameter values seems to follow well Weibull distribution.

#### **B19 High Temperature Oxidation Behavior of Si-Mo Ferritic Ductile Cast Iron**: *Kyeong-Hwan Choe*<sup>1</sup>; Sang-Mok Lee<sup>1</sup>; Kyong-Whoan Lee<sup>1</sup>; <sup>1</sup>Korea Institute of Industrial Technology

High temperature oxidation behavior of Si-Mo ferritic ductile cast irons(ferritic DCI) for automobile parts has been investigated with great interests in recent years. Despite the improvement of dimensional stability at high temperature with the increment of Si content, the accompanying mal-effects of deterioration of casting quality and fracture toughness has retarded the development of ferritic DCI. Meanwhile, the recent lean alloying trend has been also justified and utilized in the field of heat resistant ferritic DCI. In this respect, we have noticed Cr as not only effective oxidation barrier builder but strong carbide former. In the present study, systematic experiments have been carried out by adding different amounts of Cr up to 0.7wt% to the base cast iron. Various effects of Cr on the precipitation and resultant high temperature behavior were examined with and without V content. Detailed discussion regarding to the role of Cr and V on the high temperature performance characteristics of ferritic DCI was given based on the various alloying systems and heat treatment routes.

Poster Session:	
Symposium C:	
Light Metals and Alloys	

Tuesday PMRoom: Hall 2August 3, 2010Location: Cairns Convention Centre

**C1 Technology of Molten Salt Electrolysis of Magnesium Chloride**: *Miseon Choi*<sup>1</sup>; Changkyu Lee<sup>1</sup>; GoGi Lee<sup>1</sup>; Sung-Koo Jo<sup>1</sup>; Jae Young Jung<sup>1</sup>; <sup>1</sup>RIST

The electrolytic production of magnesium from magnesium chloride containing sodium chloride-rich melts has been studied using mono-polar cell, where originally designed in consideration of current efficiency and energy consumption. The anode/cathode geometry, anode/cathode distances, dimensions, current distribution, voltage were optimized mathematically. The molten condition of the electrolyte by temperature change was developed using fact sage to predict operating temperature. The optimum composition of the electrolyte was NaCl 50~60%, CaCl2 20~25% and MgCl2 20~25%, and operating temperature was at about 720°C. The magnesium was formed well at the surface of cathode and floated at the free surface of the molten salt, and chlorine gas was generated at the anode without any inverse reaction between the magnesium which is produced electrolysis process. The magnesium was collected about 200 g/hr by operating an optimized mono-polar cell with 500 A for 24 hours. The metallic magnesium produced from this study had a high purity with 99.92%.

#### **C2** Characterization of Nickel Oxide Layers on the AZ91 Mg Alloys by Plasma Electrolytic Oxidation: *In Jun Hwang*<sup>1</sup>; Kang Min Lee<sup>1</sup>; Bongyoung Yoo<sup>1</sup>; Dong Hyuk Shin<sup>1</sup>; <sup>1</sup>Department of Metallurgy and Materials Science, Hanyang University

The effect of nickel ion in electrolyte on the structural characteristics, surface color, and corrosion resistance were studied. The smaller size of pore could be observed on the oxide layer coated from electrolyte containing nickel ions, with which respond voltage increased on the higher rate compare to that from without nickel ions under constant applied current density. The color of oxide layer on AZ91 Mg alloy could be changed from light grey to moderate olive brown, when nickel ion added in electrolyte. The corrosion resistance was investigated with potentiodynamic polarization analysis. The variation of corrosion current density and polarization resistance of oxide layer by adding nickel ions clearly indicated that incorporation of nickel could enhance the corrosion resistance of magnesium oxide layer synthesized with PEO process, which is strongly related with the change of microstructure of oxide layer by nickel oxide, which is added during PEO process.

## C3 The Grain Refinement and Plastic Formability of Mg-14Li-1Al Alloy: *Peidao Ding*<sup>1</sup>; Bin Jiang<sup>1</sup>; <sup>1</sup>Chongqing University

In this paper, Al-5Ti-1B master alloy was added into LA141 alloy to be expected as a grain refiner. The effect of its addition levels on the grain size of LA141 alloy was investigated. Based on the grain refinement, the LA141 sheets were prepared by extrusion and the following cold rolling, and then microstructure, mechanical properties and plastic formability of the LA141 alloy sheets was researched by taking into account the effects of thickness of the LA141 sheet, the annealing temperatures and Erichsen cupping. The results show that the optimal addition level of Al-5Ti-1B master alloy into LA141 was 1.25 wt% and the finest grain size of LA141 alloy could be gotten. With the increase of the total reduction during cold rolling, the grains of the as-rolled LA141 sheets were flattened gradually. An optimized annealing temperature of 200°C for the cold rolled LA141 sheets was obtained. Under this condition, the microstructure of the LA141 sheets had a good plastic formability.

#### C4 Continuous Casting of Magnesium Alloy Billet Using Electromagnetic Techniques: *Myoung-Gyun Kim*<sup>1</sup>; Jonh-Ho Kim<sup>1</sup>; Joon-Pyo Park<sup>1</sup>; Gyu-Chang Lee<sup>1</sup>; <sup>1</sup>Research Institute of Industrial Science and Technology(RIST)

The use of magnesium alloys for structural components is attractive due to their excellent specific strength and low density. The cost of wrought magnesium products remains still high due to low quality of casting billets. Currently, surface defect, segregation, and inclusions are most important issues for the improvement of the quality of magnesium billets. Continuous casting in conjunction with electromagnetic field is very effective not only in improving productivity, but also in refining grain size, eliminating surface defect and segregation, stabilizing the temperature distribution during solidification and enhancing the casting speed. The latent heat of fusion per weight (J/g) of magnesium is similar to that of other metals, however, considering the heat emitted to the mould surface during continuous casting in meniscus region and converting it to the latent heat of fusion per volume, magnesium will be rapidly solidified in the mould

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during continuous casting, which induces subsequent surface defect formation. In this study, electromagnetic casting and stirring (EMC and EMS) techniques is proposed to control solidification process conveniently by compensating the low latent heat of solidification by volume and to fabricate magnesium billets of high quality surface.

C5 Effect of Casting Method of Al Contents on Microstructure in AM-Type Magnesium Alloys: *Katsumi Watanabe*<sup>1</sup>; Kenji Matsuda<sup>1</sup>; Takumi Gonoji<sup>1</sup>; Tokimasa Kawabata<sup>1</sup>; Katsuya Sakakibara<sup>2</sup>; Yukio Sanpei<sup>2</sup>; Seiji Saikawa<sup>2</sup>; Susumu Ikeno<sup>1</sup>; <sup>1</sup>University of Toyama; <sup>2</sup>Ahresty Corporation

Magnesium alloys have received considerable attention because of their lightweight and recyclability. Mg-Al alloy is usually taken as the matrix alloy among the magnesium alloys. AM-type and AZ-type Mg-Al alloys are used for industrial products widely, and particularly AM-type alloys have the better toughness and impact absorption properties than AZ-type alloys. However, there is little report about the effect of the difference of casting method on age-hardening behavior and microstructure of AM-type alloys cast into sand- and metal(permanent) -mold. The purpose of this study is to investigate the difference of the age-hardening behavior and microstructures in three AM60 alloys cast into steel, copper and sand mold by hardness test and scanning electron microscopy. Furthermore, the effect of Al content is also investigated using three alloys, i.e. AM30 (3%Al), AM60 (6%Al) and AM90 (9%Al) alloys. In this presentation, we will report results about the difference of aging behavior and microstructures in three AM60 alloys cast into steel, copper and sand mold by hardness test and scanning electron microscopy. Furthermore, the effect of Al content is also investigated using three alloys, i.e. AM30 (3%Al), AM60 (6%Al) and AM90 (9%Al) alloys. In this presentation, we will report results about the difference of aging behavior and microstructures in three AM60 alloys cast into steel, copper and sand mold in Mg- 3, 6 and 9 mass% Al alloys.

#### C6 Effect of Quenching on Aging Behavior of Binary Magnesium–Zinc Alloys: *Ryosuke Nakanishi*<sup>1</sup>; Tokimasa Kawabata<sup>1</sup>; Kenji Matsuda<sup>1</sup>; Susumu Ikeno<sup>1</sup>; <sup>1</sup>University of Toyama

The Vickers microhardness measurements and high resolution transmission electron microscopy (HRTEM) observation were carried out to investigate the effect of Zn content on aging behaviour of Mg-Zn alloys. The hardness increased drastically after aging at 473K in the Mg-Zn alloys with higher Zn concentration. And higher maximum hardness was obtained at the earlier aging time in the alloys with higher Zn concentration. The aging response of the Mg-4.7mass%Zn alloy aged at 473 K after quenching into the chilled water was different from that of the specimen aged at the same temperature after quenching at 473 K up to maximum hardness. HRTEM images of the mono layers on {1120} Mg planes, which was considered to Guinier-Preston (G.P.) I zone, were observed in the specimen after quenching into chilled water and  $\beta_1$ ' and  $\beta_2$ ' phase existed in the peak aged specimen. The number of  $\beta_1$ ' phase decreased in the specimen aged at the same temperature after quenching at 473 K, compared to that of the specimen quenched into chilled water. It was proposed that the difference of the aging response was related to the formation of the G.P. zones.

#### C7 Effects of Trace Elements on Microstructure Characterization and Mechanical Properties of Mg-Zn-Sn Based Alloys: *Hyeon-Taek Son*<sup>1</sup>; Dae-Guen Kim<sup>1</sup>; <sup>1</sup>Korea Institute of Industrial Technology

Magnesium alloys have attracted significant interest due to their high potentially lightweight materials for structural applications such as automotive and aerospace industry. The alloy systems for commercial applications are normally based on either an Mg-Al based system, such as AZ91 (ASTM designation, Mg-9.5%Al-0.5%Zn-0.3%Mn), or an Mg-Zn based system, such as ZK61 (Mg-6%Zn-0.7%Zr). However, application of magnesium alloys is limited to components because of low strength and ductility at room and elevated temperature. In the present study, we studied effects of trace elements on microstructure and mechanical properties of Mg-Zn-Sn based alloys. The Mg-Zn-Sn-X alloys prepared in mild steel crucible under the protection of mixed gas of CO2/0.5%SF6 using commercial stock. The melt was held at 700°C for 20 min and then poured into a permanent mould(PM). And as-cast alloys were hot-extruded into a rod that was 8 mm in thickness with a reduction ratio of 30:1. Also Mg-Zn-Sn-X alloys were hot-rolled into a sheet of about 1mm in thickness at elevated temperature.

# **C8 Microstructure and Tensile Properties of Mg-5Sn-5Zn Alloy**: *Jixue Zhou*<sup>1</sup>; Yuansheng Yang<sup>1</sup>; Changwen Tian<sup>2</sup>; Shouqiu Tang<sup>2</sup>; <sup>1</sup>Institute of Metal Research, Chinese Academy of Sciences; <sup>2</sup>New Materials Research Institute of Shandong Academy of Sciences

The microstructure of Mg-5Sn-5Zn alloy in different states, as-cast, solutioned and eged, and tensile properties at room temperature and 150 °C is investigated. The microstructure of as-cast Mg-5Sn-5Zn alloy consists of  $\alpha$ -Mg primary, Mg-MgZn eutectic, divorced eutectic Mg<sub>2</sub>Sn and the second constituent Mg<sub>2</sub>Sn. After solution treatment, all the Mg<sub>2</sub>Zn particles and the majority of MgSn phase are dissolved into the matrix, and the solute distribution within the matrix is homogeneous. After aging, many fine precipitates distributes within the matrix and along the grain boundaries in the alloy. The tensile strength, yield strength and the elongation of the as-cast, solutioned and aged alloys are tested respectively.

**C9 Effect of Sc Addition on Aging Behavior of Mg-Gd Alloys**: *Tokimasa Kawabata*<sup>1</sup>; Kenji Matsuda<sup>1</sup>; Susumu Ikeno<sup>1</sup>; <sup>1</sup>University of Toyama

The Vickers microhardness measurements and transmission electron microscope (TEM) and high resolution TEM observations were carried out to investigate the effect of Sc addition on aging behavior of Mg-Gd alloy. The higher maximum hardness was obtained from the specimen with higher content of Sc. The contrasts of precipitates, which were similar to  $\beta$ " phase (D0<sub>19</sub> type structure) in binary Mg-Gd alloy, were observed in the Mg-Gd-Sc alloys at early stage of aging. The contrasts of the precipitates, which were similar to  $\beta$ " phase (base centered orthorhombic structure) in binary Mg-Gd alloy, were observed dominantly in the specimen before reaching maximum hardness after increase of hardness again. The contrasts of the precipitates with D0<sub>19</sub> type ordered structure were appeared during prolong aging in the specimen higher content of Sc. It was revealed that Adding Sc to the Mg-Gd alloy tends to slow down transformation from the  $\beta$ " phase.

**C10 Microstructure and Aging Behavior in AM60 Magnesium Alloy Cast into Sand and Permanent Molds**: *Hiroshi Yamada*<sup>1</sup>; Mitsuaki Furui<sup>1</sup>; Susumu Ikeno<sup>1</sup>; Yukio Sanpei<sup>2</sup>; Katsuya Sakakibara<sup>2</sup>; Seiji Saikawa<sup>2</sup>; <sup>1</sup>University of Toyama; <sup>2</sup>Ahresty Corporation

AM60 magnesium alloy is an Mg-Al-Mn system standard alloy which has been used for a lot of interior system parts in the automobile in recent years. This alloy is manufactured from the high-pressure die casting process with chiefly high productivity, but practical use and research using the gravity metal mold casting with small cooling rate are less than the die casting method, and there are a lot of unknown things regarding the solidification behavior and the microstructure evolution. In this study, the AM60 magnesium alloy cast into sand and permanent molds about the microstructure evolution during age hardening process after solution treatment and aging treatment was reported on. AM60 magnesium alloy castings gave the solution treatment at 473K for 86.4ks. After that, aging treatment was carried out by three temperatures of 473, 498 and 523K. The age hardening curve obtained, all the specimens in the condition of peak aging hardness was increased by decreasing the aging temperature. In the condition of long aging time, a cellular precipitation grows up from the grain boundary in the crystal grain. Fine cellular precipitation and intergranular precipitation obviously occurs at the lower aging temperature.

#### C11 Microstructure and Mechanical Properties of Mg-9Zn-Zr Magnesium Alloys: Jing Zhang<sup>1</sup>; Fusheng Pan<sup>1</sup>; Qi Ma<sup>1</sup>; Rulin Zuo<sup>1</sup>; <sup>1</sup>Chongqing University

Mg and its alloys are the lightest commercial structural metals. However, the use of wrought Mg alloys has so far been restricted, partly because of their inadequate combined mechanical properties. There are many efforts in recent years to explore new alloys with increased strength while as the same time maintaining an acceptable plasticity. Among these new alloys, Mg-Zn-Zr-Re system alloy has attracted particular attention due to its considerable age hardening response and strengthening effects of Re- and Zn-bearing compounds. However, consumption of Zn atoms by forming these Zn-bearing particles causes decrease of the solid solubilities of Zn in Mg matrix, resulting in reduction of solution and age hardening strengthening effects brought by Zinc. In the present study, higher Zn element content than that in commercial Mg alloys were alloyed in Mg, to compensate the solute lose in Mg matrix. The effects of alloying additions of Zinc, as well as a chosen rare earth element Er, on the resulting microstructures were examined. The microstructure was then correlated to the mechanical properties. Effects of alloying elements and thermomechanical treatment on the microstructure and mechanical properties were analyzed and discussed

#### C12 TEM Observation of Precipitates in Mg-Gd-Sc Alloy with High Content of Sc: *Takafumi Fujii*<sup>1</sup>; Tokimasa Kawabata<sup>1</sup>; Kenji Matsuda<sup>1</sup>; Susumu Ikeno<sup>1</sup>; <sup>1</sup>University of Toyama

Transmission electron microscope (TEM) observation was carried out to investigate the precipitates in Mg-Gd-Sc alloy with high content of Sc up to 13mass%. In the specimen aged at 573 K, the contrasts of plate-like precipitates were appeared from early stage of aging. The contrasts of the plate-like precipitates lied on the {1100} magnesium matrix planes and were similar to ß phase in Mg-Gd alloy aged at 573 K and the precipitates with granular shape were observed with the plate-like precipitates. The contrasts of granular shape were not observed in the Mg-Gd alloys. The contrasts of the plate-like precipitates became longer and the number of the precipitates contrasts increased with increasing aging time up to maximum hardness aged at 573 K and it was revealed that the plate-like precipitates contributed to the increase of hardness in the Mg-Gd-Sc alloy with high content of Sc.



C13 Effect of Temperature on Plastic Buckling Strength of Shot Peened Pipe of Magnesium Alloys: *Izumi Fukuda*<sup>1</sup>; Yasunori Harada<sup>2</sup>; Yuichi Tanaka<sup>1</sup>; <sup>1</sup>Kumamoto National College of Technology; <sup>2</sup>University of Hyogo

This paper describes the effect of temperature on plastic buckling strength of shot peened pipe of magnesium alloys. Workpieces are three cylindrical pipes of AZ31, AZ61 and AZ80 magnesium alloys. The shape of the specimen is 30mm in height, 16mm and 14mm in outer and inner diameters. The shot peening treatment was given on the surface of cylindrical pipe using an air-type peening machine. Plastic buckling strength test was performed under axial compression in the case of both ends with fixed condition, at several temperatures between 293K and 573K and at crosshead speed of 1 mm/min using an Instron-type testing machine. Main results are summarized as follows: Firstly buckling stresses for all pipes of AZ31, AZ61 and AZ80 alloys increased after the shot peening treatment. Secondly the effect of shot peening on plastic buckling strength was found out that there is a few of differences for AZ31, AZ61 and AZ80 alloys. Finally from the investigated result about the influence of test temperature on the buckling stress of shot-peened pipe, the buckling stress of the shot-peened pipe was higher than that of as received one up to the test temperature of 473K, however it tended to be around equal at 573K.

### **C14 Evolution of Textures during Compression in a Hot-Extruded AZ31 Mg Alloy:** *Yong Bum Park*<sup>1</sup>; Myung Jae Lee<sup>1</sup>; Byung Jo Jung<sup>1</sup>; <sup>1</sup>Sunchon National University

The development of textures and microstructures during plastic deformation in a hot-extruded AZ 31 Mg alloy was investigated using compression test with such parameters as deformation temperature, strain and strain rate. It was observed from true stress-strain curves measured that twinning involves changes of the flow stresses. In the early stages of deformation at temperatures lower than 200oC, the occurrence of twins resulted in decrease of the work hardening rate, which increased drastically at a true strain of -0.05. The initial <1010>//ED fibre texture as hot-extruded transformed into <0001>//ED fibre texture due to basal slips in grains whose crystallographic orientations were changed by twinning. The evolution of microtextures during compressive deformation was observed with the aids of the EBSD analysis and discussed in terms of competition between twinning and slip.

#### C15 Hot Extrudability of Semi Solid AM100A Alloy Fabricated by Cooling Plate: *Dae-Hwan Kim*<sup>1</sup>; Yeong-Rok Seong<sup>1</sup>; Seong-Hwa Choi<sup>1</sup>; Young-Hwa Kim<sup>1</sup>; Su-Gun Lim<sup>1</sup>; <sup>1</sup>i-Cube Center, Engineering Research Institute, Gyeongsang National University

The effects of extrusion temperature on the hot extrusion of semi-solid AM100A alloy fabricated by cooling plate over a temperature range of 300–380°C were experimentally investigated. And the mechanical properties of extruded AM100A alloy products were also presented. In order to confirm the effect of temperature on hot extrudability of AM100A alloy, we observed the surface and microstructure of hot extruded AM100A alloys. The surface of the fabricated extrudates with extrusion container temperature of 380°C under the extrusion ratio of 25:1 and ram speed of 2.4mm/sec was significantly sound and the tensile strength of extrudate was approximately 310MPa at room temperature.

#### C16 Influence of Thickness Reduction per Pass on Microstructure and Rolling-Formability of ZK60 Magnesium Alloy Sheet: X.H. Chen<sup>1</sup>; F.S. Pan<sup>1</sup>; J.J. Mao<sup>1</sup>; J. Peng<sup>1</sup>; A.T. Tang<sup>1</sup>; J.F. Wang<sup>1</sup>; <sup>1</sup>Chongqing University

Magnesium alloys have been considered as advanced materials for many engineering applications due to their low density, good machinability, excellent damping capacity, high specific strength and other good properties. Recently, there is a growing need for high-strength Mg alloy sheet in automotive and aerospace. Therefore, it is quite necessary to understand the effects of rolling parameters on ZK60 Mg alloy sheet during hot rolling process since this alloy is one of the most typical high-strength Mg alloys. In this work, the influence of thickness reduction per pass over the range of 10~20% on the microstructure and rolling-formability of ZK60 alloy sheets has been investigated systematically. With increasing reduction per pass, the formability decreases gradually, and the volume of twinning region and shear bands increases at the same total reduction of ~50%. The possible mechanisms underlying these observed results are discussed.

#### **C17** Mechanical Behaviors of Mg-8.5wt%Al Alloy after Hydrostatic Extrusion and Comparison with Indirect Extrusion Process: Sangmok Lee<sup>1</sup>; <sup>1</sup>Korea Institute of Industrial Technology (KITECH)

Mg-8.5wt%Al alloy was subjected to hydrostatic extrusion and mechanical behaviors were investigated. Hydrostatic extrusion was conducted at temperatures of 523 and 573K at ram speeds of 5.0, 10.0 mm/s with extrusion ratio (ER) of 25. Extruded specimens were pulled in tension and ultimate tensile strengths (UTS) and elongation to failures (EF) were measured. It was noted that for the same extrusion temperature of 523K UTS and EF decreased with increasing deformation rate and for the same deformation rate UTS and EF for specimens decreased

with increasing extrusion temperature. These results were also compared with ones obtained from indirect extrusion. The extrusion was possible only at a ram speed of 1.3 mm/s. At this slow deformation rate UTS and EF increased in comparison to the case for hydrostatic extrusion. It is realized that these two mechanical properties are significantly dependent upon the deformation rate. Optical microscopic observation was followed. As deformation rate increased, microstructures were ill-defined and cracks along the grain boundaries were severer. It is presumed that at higher deformation rate the micro fracture of matrix initiated in the proximity of the fine  $Mg_{17}Al_{12}$  precipitates during extrusion, which result in lowering the tensile strength and elongation to failure.

### C18 Low Temperature Extrusion of Magnesium Alloy AZ80 in Hot Hydrostatic Process: *Duk-Jae Yoon*<sup>1</sup>; Eung-Zu Kim<sup>1</sup>; Ki-Sun Lee<sup>1</sup>; Sang-Mok Lee<sup>1</sup>; Seong-Joo Lim<sup>1</sup>; <sup>1</sup>KITECH

Forming limit of AZ80 was explored in hot hydrostatic extrusion and possibility of extending the safe extrusion zone in temperature-speed domain was examined. Extrusion characteristics of AZ80 were studied with hot hydrostatic extrusion process. AZ80 was extruded with hot hydrostatic extruder under temperature condition of 200, and 250°C. The effects of process variables' change were investigated parametrically. Round bars were extruded with conical shaped dies which had 45° of half angle, and corner radii of 1mm, and 10mm. Billets were prepared with different surface roughness of Ra 25, 50, and 100. Extrusion speed were controlled to 5, 10, 15mm/s to observe the strain rate effect. AZ80 was extruded with sound surface in spite of the relatively low temperature and high speed condition. As the extrusion speed and curvature of die corner were increased, surface defect was detected on the extruded bar which was originated from hot shortness. Condition of high hydrostatic pressure and good lubrication enlarged forming limit of the alloy and reduced the redundant work, which were resulted in limited temperature rise in the extrusion. By optimizing process condition of die shape and billet surface roughness, forming limit in hot extrusion of the alloy was extended.

**C19 Mechanical Properties and Microstructure of AZ Magnesium Alloys Anodized by Phosphate Electrolyte**: *Teruto Kanadani*<sup>1</sup>; Shuji Hikino<sup>1</sup>; Atsushi Saijo<sup>2</sup>; Makoto Hino<sup>3</sup>; Koji Murakami<sup>3</sup>; Akira Sakakibara<sup>4</sup>; <sup>1</sup>Okayama University of Science; <sup>2</sup>Hori Metal Finishing Industry Co., Ltd.; <sup>3</sup>Industrial Technology Research Institute of Okayama Prefecture; <sup>4</sup>Okayama University

Magnesium alloys possess many advantageous functional properties and performance like high specific strength, vibration absorption, or high recyclability. Use of magnesium alloys, mainly for vehicle parts as well as electronic appliances, has been booming in recent years because of their lightweight compared to aluminum alloys and good creep resistance relative to plastics. Most magnesium usage for structural applications was in die-casting and most of this was in one alloy, AZ91D. Since magnesium has the electrochemical potential out of all the common commercial metals and is extremely prone to corrosion, it should be necessary that it undergo surface treatment. It is well known that fatigue cracks start near free surface region. Surface microstructure, therefore, should have significant effect on the fatigue strength. This study carried out the environmental friendly anodizing was carried out using a mainly phosphate solution without heavy metal onto various AZ magnesium alloys. The effect of anodizing on mechanical properties and microstructure was examined by repeated tension fatigue tests, tensile tests, hardness tests and electron microscopy.

### **C20 Deformation Behavior of Magnesium Single Crystal in C-Axis Compression and A-Axis Tension**: *Shinji Ando*<sup>1</sup>; Masayuki Tsushida<sup>1</sup>; Hiromoto Kitahara<sup>1</sup>; <sup>1</sup>Kumamoto University

From von-Mises criterion, five independent slip systems are required to deform crystals uniformly. In the case of magnesium, main slip system is a basal slip. However, von-Mises criterion cannot satisfy by the basal slip because it has only two independent slip systems. Therefore, non-basal slip must be activated in deformation of magnesium. In this study, deformation behavior by non-basal slip in magnesium single crystals was investigated by c-axis compression and a-axis tension test. The crystals were yield by second order pyramidal slip in the range from 77K to 573K, and the yield stress shows anomalous temperature dependence (incleased with incleasing temperature) between 203K and 293K. The yield stress by second order pyramidal slip was decreased over 293K. In a-axis tensile test, the single crystal elongated over 3%, however, the crystal fractured les than 1% in c-axis compression. Deformation mechanism by nonbasal slip was discussed.

C21 Texture and Mechanical Properties of AZ31 Magnesium Alloy Sheets Processed by Symmetric/Asymmetric Combination Hot-Rolling: Jumpei Horiuchi<sup>1</sup>; Hirofumi Inoue<sup>1</sup>; Takayuki Takasugi<sup>1</sup>; <sup>1</sup>Osaka Prefecture University

The poor formability in magnesium alloys has been attributed to basal texture formed at rolling. Also, it has been reported that the modification from basal texture results in improving formability but decreasing yield strength. The conventional symmetric rolling enhances yield strength by forming basal texture,



while the asymmetric rolling can improve formability by inclining the c-axis of hcp crystal. In this study, the combination rolling consisting of symmetric and asymmetric hot rolling has been performed to improve formability and maintain high strength of AZ31 magnesium alloy sheet simultaneously. The combination hot-rolled and annealed sheet exhibits a broadened texture having double peak with tilt angles of 0° and 40° from ND toward RD with respect to the c-axis. Correspondingly, this sheet shows relatively high yield strength of 123 MPa and large elongation of 24.7%. As for cup drawing test, the conventional warm-rolled sheet can be barely formed at 175°C, but the combination rolled sheet can be formed at as low as 75°C. These results indicate that the symmetric/asymmetric combination hot-rolling leads to a unique texture with good balance of formability and strength.

#### C22 Twinning Behavior in AZ31-B Polycrystal Texture Subjected to In-situ Bending Test: Itsuya Sato<sup>1</sup>; Seiji Miura<sup>1</sup>; Tetsuo Mohri<sup>1</sup>; <sup>1</sup>Hokkaido University

As a commercial Mg alloy, AZ31-B has been used widely. In the texture of AZ31-B plate, each grain has the c-axis almost parallel to the plate normal. Therefore, at the bending process of the plate, basal slip system can not accommodate a plastic strain parallel to the a-axis. It is known that {10-12} twinning can be formed by applying an extension strain parallel to the c-axis, which is equivalent to the a-axis compression. So in the bending deformation of the AZ31-B plate with a texture microstructure, it is expected that {10-12} twinning occurs. In this study, in-situ bending test of AZ31-B texture was conducted under a scanning laser confocal microscope. In addition, EBSD techniques were used for the analysis of crystal orientations. The process of twinning development observed by in-situ bending test can be summarized as follows. With the increase of deformation strain, total area of twins increases. However, it is noted that the growth of twins is apparent while the number of twins is almost constant. EBSD analysis revealed that twinning behavior seems to obey Schmid's law even in the polycrystal.

**C23** A Study on the Deformation and Fracture Behaviors of Mg Alloy: Tianmo Liu<sup>1</sup>; Liwei Lu<sup>1</sup>; Shan Jiang<sup>1</sup>; <sup>1</sup>National Engineering Research Center for Magnesium Alloys, Chongqing University

The fracture mechanisms of as-extruded AZ31 are investigated by compression and tensile deformation. Upsetting in ambient compression deformation, the microstructure shows that lots of twins formed at coarse boundary, some twins boundaries induce the source of crack, the crack spread through grain boundary, while others play an important role in hindering crack's initiation and propagation, SEM shows that the fracture mechanism is mixed crack of ductile and brittle. Tensile in ambient deformation, withdraw neck comes out, a great quantities of twins are formed at the elongated-grain boundary and exists interact role between twins and crack, SEM indicates that the mechanism is dimpled rupture induced by cavitation's nucleation.

# C24 The Investigation of Various Strain Rate and Temperature on the Microstructure and Mechanical Properties of Hot-Rolled AZ31 Magnesium Alloy: *Dae-Guen Kim*<sup>1</sup>; Hyeon-Taek Son<sup>1</sup>; Moo-Young Huh<sup>2</sup>; <sup>1</sup>Korea Institute of Industrial Technology; <sup>2</sup>Korea University

The increasing demand for better fuel economy, higher operating efficiency and related reduction of exhaust gases in automotive applications have prompted intensive research into lightweight structural materials. Magnesium alloy offers great potential for automobile construction and further technical applications in aeronautics, electronics and other fields due to its low density, high specific strength and high specific stiffness. In spite of the above-mentioned advantages and applications, the use of pure magnesium in industry is limited because of its low deformability by cold working, due to its hexagonal closed-packed (HCP) crystal structure. However, warm forming can improve the formability of magnesium alloy because the pyramidal plane of magnesium HCP crystal structure will be operated by thermal activation and which results in good formability with warm forming. The aim of the present work is to further investigate the hot formability of an AZ31 magnesium alloy in the temperature range between RT(room temperature) and 300°C in terms of flow stress and microstructure of the deformed material.

#### **C25** Dynamic Deformation of Submicrocrystalline Aluminum Alloys: *Young Gun Ko*<sup>1</sup>; Yang Gon Kim<sup>2</sup>; Seung Namgung<sup>3</sup>; Dong Hyuk Shin Shin<sup>3</sup>; Sunghak Lee<sup>4</sup>, <sup>1</sup>Yeungnam University; <sup>2</sup>Hyundai Steel; <sup>3</sup>Hanyang University; <sup>4</sup>POSTECH

The effect of equal-channel angular pressing (ECAP) route on high strain rate deformation behavior of submicrocrystalline Aluminum alloy was investigated. The 8-pass ECAPed specimens deformed via three different routes consisted of fine grains of 0.5  $\mu$ m in size, and contained a considerable amount of second phase particles, which were fragmented and distributed homogeneously in the matrix. In the torsion tests, the maximum shear stress significantly increased with increasing number of ECAP passes, while the maximum shear stress and fracture shear strain were lowest in the specimen deformed via route A among the 8-pass ECAPed specimens. Observation of the deformed area beneath the fractured

surface revealed that adiabatic shear bands of 100  $\mu$ m in width in the specimen deformed via route A, which minimized the maximum shear stress and fracture shear strain, whereas they were hardly formed in the specimens deformed via route B or C. The formation of adiabatic shear bands was explained in terms of critical shear strain, deformation energy required for void initiation, and microstructural homogeneity related to ECAP routes.

#### C26 Research on Non-Isothermal Forming of Complex Shaped AZ31 Magnesium Sheet Products: Xu Chun<sup>1</sup>; <sup>1</sup>Shanghai Institute of Technology

A new deep drawing process with a localized heating was developed to improve sheet forming of magnesium alloy AZ31 which is very difficult to form by conventional methods at room temperature. A magnesium alloy AZ31 sheet of 0.6 mm thickness was used in the present study. Computer component case was conducted to form at warm temperature conditions (250~280°C) for the blank and deep drawing tool (holder and die) and in the hydro mechanical stamp. As a result, the formability of the magnesium alloys can be enhanced at nonisothermal forming. The necklace microstructure of AZ31 has obtained at warm forming. Necklace grains were formed along the deformed grain boundary that means dynamic recrystallization has taken place. Necklace region become larger with a decrease in strain rates.

## **C27 Microstructure Evolution in AZ31 Magnesium Alloy Worked by Torsion at Warm Temperature**: *Kouji Aoyama*<sup>1</sup>; Mitsuaki Furui<sup>1</sup>; Susumu Ikeno<sup>1</sup>; <sup>1</sup>University of Toyama

In my laboratory, a bar of AZ31B magnesium alloy was worked by torsion after heating in the furnace. It was reported that the resistance of compression at warm condition can be decreased by the preliminary torsion working. However, there was a problem that the specimen temperature decreased during processing at the room temperature. In the present study, the temperature was controlled by using a cylindrical electric furnace while processing. The bar of AZ31B magnesium, with 10mm in diameter and 980mm in length, was given homogenization treatment at 673K for 72ks. It was processed to torsion using the AZ31B magnesium alloy at a warm temperature, and microstructure evolution during the torsion deformation was observed. A dynamic recrystallization occured in torsion working at warm temperature. Dynamic recrystallization was slightly seen in the edge of the bar fracture dat a rotation speed of 1rpm at 573K and 623K. The amount of torsion to fracture was increased greatly, dynamic recrystallization was seen in the center of bar, and it was almost seen in all aspects at the rotation speed of 1rpm at 673K.

#### **C28** Preparation and Properties of Novel Mg-Cu-Mn-Zn-Y Damping Magnesium Alloy: *Jingfeng Wang*<sup>1</sup>; Wenwen Wei<sup>1</sup>; Fusheng Pan<sup>1</sup>; <sup>1</sup>College of Materials Science and Engineering, Chongqing University, China

The sintered Mg-3wt%Cu-1wt%Mn (CM31) alloy fabricated by powder metallurgy processing has drawn a lot of attention because of its excellent damping capacity and adequate mechanical property. Unfortunately, the high preparation cost and the poor stability restrain the application of sintered CM31 alloy. Consequently, it is of great significance for the application of CM31 alloy to develop a low cost preparation process. In present paper, a little Y and Zn elements were added into CM31 alloy in order to improve its damping capacity and mechanical property, and the novel Mg-Cu-Mn-Zn-Y damping magnesium alloy was successfully obtained by using conventional melting process and combined with proper extrusion process. At as-cast state, the damping capacity in high strain amplitude of Mg-Cu-Mn-Zn-Y alloy exceeded that of CM31 alloy and closed to that of pure magnesium. Furthermore, the yield strength of Mg-Cu-Mn-Zn-Y alloy was also superior to that of CM31 alloy. After extrusion, the mechanical property of the alloys was remarkably improved in comparison with that of alloys at as-cast state. The novel Mg-Cu-Mn-Zn-Y alloy exhibits a good comprehensive mechanical property and promising damping capacity.

## **C29 Structure and Quasi-Static Compressive Properties of Mg-Based Foam**: *Yuansheng Yang*<sup>1</sup>; Zhengguo Xu<sup>1</sup>; Hongjie Luo<sup>2</sup>; Chunlei Liu<sup>3</sup>; Li Chen<sup>3</sup>; <sup>1</sup>Institute of Metal Research, Chinese Academy of Sciences; <sup>2</sup>Northeastern University; <sup>3</sup>Beijing Institute of Technology

The structure of Mg-based foams prepared by endogenetic foaming process is studied by optical microscopy and scanning electron microscopy. It is found that the Mg-based foam show a uniform structure. The compressive constitutive behavior of the foam is evaluated under quasi-static conditions as a function of pore size and relative density. The experimental results show that the pore size has strong affect on compress property, and the foam with 1.1 mm diameters pore size is better than the foam with 0.6 mm and 2.0 mm pore size. The yield stress, elastic modulus rapidly increased with increase of the relative density of the foam.


**C30 Effect of Aging Treatment on Microstructure and Mechanical Properties of a Two-Phase Titanium Alloy**: Miao Song<sup>1</sup>; Jia Lei<sup>1</sup>; *Yingjie Ma*<sup>1</sup>; Yu Liu<sup>1</sup>; <sup>1</sup>Institute of Metal Research, Chineses Academy of Sciences

In the present work, the microstructure and properties of Ti-Al-Mo -Cr -Sn -Zr -V-Si-Fe alloy as a function of aging temperature was investigated using optical microscopy, scanning electron microscopy (SEM), x-ray diffraction (XRD) and transmission electron microscopy (TEM). Three types of precipitates were found in this alloy, they were a2 phase (Ti3Al), silicates, and Sn rich phase. Ti3Al were observed in all aged specimen covering a range of 500-740°C. A method of quantitative analysis toward a2 based on high resolution imagines and Flourier transformation is used. The results combined with the TEM observation showed that the ordering inaphase were highly related to the property of fracture toughness. Silicates were determined in the formulate (Ti5+xZr3-x)Si3 in the specimens aged at 660-780°C, and it was not found to influence the fracture toughness significantly due to its small amount. The Sn rich phase was emerged with large area during the 780°C aging. And its direct correlation to property was not found. The content of acicular aphase was found almost invariable by calculating through the modification XRD quantitative analysis during the aging

#### **C31 Electron Microscopy Study of Deformation Microstructure in a Metastable Beta Titanium Alloy**: J. Sun<sup>1</sup>; H. Xing<sup>1</sup>; <sup>1</sup>Shanghai Jiaotong University

The deformation behavior of beta-Ti alloys is guite complicated, which can proceed by slip, twinning, formation of stress-induced phases or a combination of these processes. The stability of the ß phase and the stacking fault energy significantly influence the plastic deformation in beta-Ti alloys. In this work, the deformation microstructure of a metastable beta-Ti alloy with chemical composition of Ti-23Nb-0.7Ta-2Zr-1.2O at.% after cold swaging was studied by electron backscattering diffraction (EBSD) and high-resolution transmission electron microscope (HRTEM). The results showed that the deformation structure of the cold-swaged alloy appears to be similar to the swirled structure that commonly found in bcc metals heavily deformed either by wire-drawing or by rotary swaging process, and <110> fiber texture is a typical texture component of bcc metals. HRTEM results further showed <111> dislocations in the deformed alloy. Additionally, {112}<111> mechanical twinning and stress-induced omega transition were further revealed, where the orientation relationships between the  $\omega$  phase and  $\beta$  parent matrix are different from that often observed for the thermal  $\omega$  transition. Both mechanical twinning and  $\omega$  transition are considered to arise from the shear along <111>{112}. A dislocation mechanism for mechanical twinning and stress-induced ω transition is discussed.

#### **C32** Screw Form Rolling of Beta Type Titanium Alloy Preliminary Worked by Torsion: *Kazuhiro Kume*<sup>1</sup>; Mitsuaki Furui<sup>1</sup>; Susumu Ikeno<sup>1</sup>; Yuusuke Ishizaka<sup>2</sup>; Masayuki Yamamoto<sup>2</sup>; <sup>1</sup>University of Toyama; <sup>2</sup>Tsukiboshi Corporation

Beta type titanium alloys which have the same level as the Young's modulus of human bone is used for biomaterials and also wheelchairs, medical equipment and golf club heads, because of their lightness, high strength and excellent compatibility with the human body. Microstructure and mechanical properties of beta type titanium alloys processed by rolling and heat treatment was reported on. Additionally, screw form rolling using beta type titanium alloys was reported on. However, there have hardly reports on the development of those characteristics after preliminary working by torsion. It has been reported that AZ31B magnesium alloy preliminary worked by torsion has the characteristic of improved screw. In this study, a Ti-15%V-3%Cr-3%Sn-3%Al alloy which had beta single phase, typical beta type titanium alloy, preliminary worked by torsion at room temperature using the rotation speed of 1rpm, screw form rolling was done and the characteristics of screw was researched. Also, other materials were worked with a similar processing and were compared with the Ti-15%V-3%Cr-3%Sn-3%Al alloy.

## **C33 Dehydrogenation Behaviours of Titanium Hydride**: *Nur Farhana Hayazi*<sup>1</sup>; Sammy Chan<sup>1</sup>; Mannfu Rau<sup>2</sup>; <sup>1</sup>University of New South Wales; <sup>2</sup>AG Materials Inc.

Titanium hydride (TiH2) has been widely used in thermo-hydrogen processing (THP) of titanium and titanium alloys and in joining ceramics to metallic materials. In THP, hydrogen is used as a temporary alloying element in titanium alloys, which is effective in controlling the microstructure and improving the mechanical properties. The existence of hydrogen in titanium also improves the sintering of titanium powder. Therefore, the understanding of TiH<sub>2</sub> dehydrogenation behaviours is essential prior to the sintering in these fields to prevent the chances of crack and porosity. Effects on different parameters of dehydrogenation on its behavior have been examined. When the heating rate is decreased, the holding time will be increased and as a result, dehydrogenation starts at a lower temperature. The dehydrogenation behaviour of TiH<sub>2</sub> after compaction differs significantly from that the loose powder. Reduction in surface

area of compacted powder retards the dehydrogenation and higher temperature is required for the dehydrogenation. It shows a very low level of porosity and the near full densification has been achieved after dehydrogenation under Argon atmosphere. Characterisation and resulting microstructure of the dehydrogenated sample will be discussed.

#### C34 Application of Laser Remote Welding to the Aluminum Automotive Part: *Won-Ho Choi*<sup>1</sup>; Cheolhee Kim<sup>2</sup>; Ki-Young Park<sup>3</sup>; <sup>1</sup>Shinyoung; <sup>2</sup>KITECH; <sup>3</sup>Institute for Advanced Engineering

Aluminum alloys are extensively used in the automotive industry in response to increasing requirement for weight reduction in car body architecture. Laser welding of Al alloys have many advantages such as low heat input, narrow heat affected zone, minimal thermal distortion and flexibility. Recently, high brightness lasers - thin disk lasers and fiber lasers enable long focal distance welding so laser remote welding with scanning mirrors have been realized in laser welding of Al alloys.In this study, the laser remote welding was implemented to a heat shield part of the automobile by utilizing a 4kW disk laser and a 2-axis scanner. By replacing the conventional resistance spot welding, the weld flange could be reduced from 15mm to 8mm and a cycle time for each welding point could also be reduced for 3.5s to 0.6s.

#### **C35 Effect of Cryorolling and Annealing on Electrical Conductivity of Al-4Cu-TiB, In Situ Composites**: Naga Krishna N<sup>1</sup>; *Gokul Muthupandi*<sup>1</sup>; Sivaprasad Katakam<sup>1</sup>; Kumaresh Babu S.P.<sup>1</sup>; <sup>1</sup>National Institute of Technology

In this paper, the effect of cryorolling followed by short annealing on electrical conductivity of Al-4Cu-TiB<sub>2</sub> in situ composite was studied. Composites with 0, 5 and 10 % TiB<sub>2</sub> reinforcements were cryorolled followed by short annealing treatment for 3 minutes at three different temperatures (150, 175 and 200°C). Set of samples were subjected to ageing treatment at 175°C for 8h. The electrical conductivity measurements were done on these composites as well as on monolithic alloy for comparison. It was observed that the conductivity was slightly decreased in composites with increasing reinforcement due to non-conductive ceramic phase as reinforcement. Moreover, ageing after the short annealing treatment resulted in decreased conductivity, as ageing resulted in precipitation of Al<sub>2</sub>Cu phase that act as scattering sources for conducting electrons. However, among the three short annealing treatments, 175°C treated samples in both as-annealed and as-aged conditions exhibited better conductivity, which may be attributed to the texture component that was developed due to the treatment.

**C36 Effect of Rapid Solidification Processing on Hydrogen Behaviour in Aluminium**: Iya Tashlykova-Bushkevich<sup>1</sup>; Takahiro Shikagawa<sup>2</sup>; Vasiliy Shepelevich<sup>3</sup>; *Goroh Itoh*<sup>2</sup>; <sup>1</sup>Belarusian State University of Informatics and Radioelectronics; <sup>2</sup>Ibaraki University; <sup>3</sup>Belarusian State University

Within the last decade research activities dedicated to hydrogen behaviour in aluminium and its alloys experienced considerable amplification because of a large potential for utilization of advanced aluminium alloys without hydrogen embrittlement in the automotive industry in relation to fuel cell vehicles. However, as far as we are concerned, there are no such works executed at present to reveal hydrogen trapping in aluminium materials prepared through the rapid solidification processing at exceptionally high cooling rates. This work presents investigation of hydrogen desorption kinetics in rapidly solidified foils of aluminium by means of thermal desorption spectroscopy. The aluminium foils exposed to humid air and aluminium binary alloy with small addition of titanium were also examined to assess the impact of cooling rate on hydrogen/microstructure interaction during the solidification of aluminium. Original results are considered in comparison with traditionally processed aluminium foils prepared from the DC cast industrial rolling ingots.

#### C37 Evaluation of Corrosion Properties for FSWed Dissimilar Al Alloy (5052-O:5083-H321): Jae-Cheul Park<sup>1</sup>; Min-Su Han<sup>1</sup>; Seong-Jong Kim<sup>1</sup>; <sup>1</sup>3Division of Marine System Engineering, Mokpo Maritime University

Al allovs usually have good corrosion resistance, but serious casualties and economic loss can arise due to the chloride ions contained in marine environments. To minimize corrosion damage for these vessels during the voyage, painting for the hull and applying sacrificial anodes or(and) an impressed current cathodic protection (ICCP) system can be applicated. The friction stir welding (FSW) was developed by The Welding Institute (TWI) as a new solid-state welding technique for joining Al allovs using frictional heat in 1991. The tool was shifted to the joint direction. friction stir welding (FSW), is joining technique by partly shoftening phenomenon of the material by mechanical stir action and friction heat at welding zone. In this study, we compared the corrosion properties of base metals and optimum FSWed parts. As a result of the electrochemical experiments for FSWed part with 5052-O and 5083-H321 Al alloy in seawater solution are presented. The corrosion protection potential range was found to be from -0.7 V to -1.4 V. This research was financially supported by the Honam Sea Grant and the Ministry of Education, Science Technology (MEST) and Korea Industrial Technology Foundation (KOTEF) through the Human Resource Training Project for Regional Innovation.



C38 Texture Distribution through Thickness in 6xxx Aluminum Alloy Sheet Fabricated by Cross-Roll Rolling Method: *Kwang-jin Lee*<sup>1</sup>; Jae-yeol Jeon<sup>1</sup>; Kee-do Woo<sup>2</sup>; <sup>1</sup>Korea Institute of Industrial Technology; <sup>2</sup>Chonbuk National University

Asymmetric rolling (ASR), in known to take place due to differences in the circumferential velocities of working rolls caused by lubrication mismatch, different angular speeds or different roll diameters. Because asymmetrical rolling process can gain merits such as less rolling pressure distribution, rolling force, rolling torque and a very thin thickness with high rolling precision, it becomes more and more important in the recent years. However, cross rolling is different from conventional asymmetric rolling. The characteristic of cross rolling is that the angle between upper roll and lower roll is different so that the tension applies  $\epsilon_{_{11}}(RD),\,\epsilon_{_{22}}((TD),\,and\,\epsilon_{_{33}}((ND)$  of the sheet. The purpose of the present work is to investigate the texture distribution through thickness in 6xxx series aluminum alloy sheet manufactured by cross rolling method. The cross roller has the angle of 5 degrees between upper and lower roll. Texture distribution through sheet thickness was analyzed by EBSD. The EBSD results revealed that the amount of shear deformation texture components was increased by cross rolling compared to conventional parallel rolling. And concentration ratio of shear deformation texture on the surface was higher than 1/2 and 1/4 thickness of TD plane.

#### C39 Low-Cycle Fatigue Behavior of an Al-Mg-Si Alloy with and without a Small Addition of Sc: *Chihiro Watanabe*<sup>1</sup>; Ryoichi Monzen<sup>1</sup>; <sup>1</sup>Kanazawa University

Low-cycle fatigue behavior of a wrought Al-0.8wt%Mg-0.7wt%Si alloy with and without 0.27wt%Sc has been investigated at room temperature under constant plastic-strain amplitudes in the range of  $10^{-4}$  to  $10^{-3}$ . After peakaging treatments, both the alloys had fine rod-shaped  $\beta$ ' precipitates. In the Sc containing alloy, spherical Al<sub>3</sub>Sc precipitates of about 11 nm in diameter were co-existed. The alloy with Sc exhibited cyclic hardening to saturation, while the alloy without Sc showed clear cyclic softening after initial hardening under all applied plastic-strain amplitudes. Transmission electron microscopy observation revealed that slip band structures were developed in the Sc-free alloy. Within the slip bands, shearing of the  $\beta$ ' precipitates by moving dislocations was often observed. The cyclic softening in the alloy without Sc can then be explained by a loss of precipitation strengthening effect through the precipitation destruction within strongly strained slip bands. In the Sc-bearing alloy, owing to the existence of non-shearable Al<sub>3</sub>Sc precipitates, dislocations were uniformly distributed, resulting of the absence of the cyclic softening.

#### C40 Morphological Variation of Fe/Cr-Rich Intermetallic Phase in Recycled Al-Si Alloy as a Function of Cooling Rate: Time Resolved Radiography: *Bong Hwan Kim*<sup>1</sup>; Sangmok Lee<sup>1</sup>; Hideyuki Yasuda<sup>2</sup>; <sup>1</sup>Korea Institute of Industrial Technology; <sup>2</sup>Osaka University

The increased iron content in recycled Al alloy causes excessive solute of iron to segregate inter-dendritic region during solidification, which results in formation of iron-rich intermetallic phases. Iron-rich intermetallic phases have been considered as a major concern in foundry due to their intrinsic deleterious effect on properties of casting products. Effects of various alloying elements like Mn and Cr on harmful morphologies of iron-rich intermetallic B-Al FeSi phase have been well reported by many researchers. However, resultant morphologies of same phase were reported to be observed in different shapes such as polygonal, script-type, dendritic, etc. This study aims to investigate morphological variation of Fe/Cr-rich intermetallic phases formed in recycled Al-Si alloy. For this purpose, the solidification of recycled Al-Si alloy and Cr added alloy were observed by time-resolved radiography as a function of cooling rate. The morphology of Fe-rich intermetallic B-Al FeSi phase was observed to be modified by cooling rate, while the Fe/Cr-rich, α-AlFeCrSi phase was observed to transform from polyhedron growth to dendritic growth of tetrahedral symmetry as cooling rate increased.

#### C41 Microstructure and Mechanical Properties of Al Alloy Sheets Containing High Mg Contents Fabricated by Twin-roll Strip Casting: *Cheon Boo-Hyeon*<sup>1</sup>; Kim Hyung-Wook<sup>2</sup>; Lee Jae-Chul<sup>1</sup>; <sup>1</sup>Korea University; <sup>2</sup>Korea Institute of Materials Science

The weight reduction of automobile has been the hot issue that can preserve environment and improve the driving performance. Employing aluminum intensive auto-body is an effective means for such purposes. In this study, we produced high-strength aluminum alloy sheets with high magnesium contents by a strip caster equipped with an asymmetric nozzle, which help reduce surface defects and internal segregation. The as-cast sheets with dendritic grains and  $Al_8Mg_5$  segregation were hot-rolled and subsequently annealed at various temperatures. The observed properties were explained on the basis of the microstructural features shown by the alloy sheet.

C42 Preparation of Aluminum-MWCNT Nanocomposite Powders for the Powder Molding by Mechanical Milling with the Aid of PCAs: *Hendrick*<sup>1</sup>; Kwan Hee Han<sup>1</sup>; <sup>1</sup>Yeungnam University

An attempt has been made to prepare aluminum-MWCNT composite powders being suitable for the net shape manufacture via powder molding process. In order to accomplish uniform dispersion of MWCNTs in aluminum powders, we employ mechanical milling in an inert gas atmosphere for up to 72 hr using a planetary milling apparatus equipped with cemented carbide-lined jar and cemented carbide balls. Two types of PCAs(process controlling agents), i.e., polyethylene wax and ethanol are used up to 5% for to control the particle size by preventing excessive cold welding during milling. The average particle size and the specific area are determined with the milling time, and the powder shape change during milling is examined in SEM. On the basis of the experimental observation, the effects of the two kinds of PCAs on the characteristics of milled powders will be presented.

## **C43** The Powder Molding of an Al-Mg-Si-Cu Alloy with Premixed Elemental Powders: *Hansol Lee*<sup>1</sup>; Si-hyung Kim<sup>1</sup>; Kwan Hee Han<sup>1</sup>; <sup>1</sup>Yeungnam University

In this paper we demonstrate that a sound and dense sinter an Al-Mg-Si-Cu alloy can be produced by use of the powder molding and a feedstock made of commercial grade premixed powders, a wax-based thermoplastic binder system and a sintering aid. The greed body is formed under an applied pressure of less than 20MPa. Debinding for the removal of organic binders from the green body and sintering for densification in debound body are performed according to onestep heating schedule in either nitrogen, argon or a gas mixture which contains hydrogen. Preliminary results about the sinter properties such as apparent density, shrinkage, microstructure and mechanical properties obtained under different experimental conditions will be presented.

C44 The Research of Al-Mg Alloy Foam by Melt Foaming Method and Its Mechanical Property: *Rui Zhao*<sup>1</sup>; Byeong-Su Tak<sup>1</sup>; Zan Wang<sup>1</sup>; Yuxuan Li<sup>1</sup>; Bo-Young Hur<sup>1</sup>; <sup>1</sup>Gyeongsang National University

Melt foaming method is one of cost-effective methods. The Al-Mg alloy foam was prepared by the melt foaming method. The preparation process is as the following, a quantity of Al-1%Mg alloy (~1kg) was melted in a crucible at a proper temperature and then 2wt.% Ca particles was added by a stirrer with rotation speed of 500rpm to increase the melt viscosity. After the melt viscosity reach a proper value, the foaming agent TiH, was added and dispersed into the melt with the stirrer revolution speed of 1000rpm, leading to the melt being foamed gradually. After that, the stirrer was pulled out and the foamed melt was kept in the furnace to let a proper cellular structure be formed. Finally, the melt foam was cooled and solidified. Al-Mg foam samples were cut to 30X30X30mm3 and they were polished by emery papers of 1000# and 1200#. After that, Al-Mg foams sample was polished and rusted. The thickness cells were analyzed by use of Metallurgical Microscope and software. The compressive tests on these foams were carried out by Universal Testing Machine. The sound-absorption test was carried out by Acoustic duct. The relationship between pore structures of foams and their mechanical properties is investigated.

#### **C45** The Structure and Mechanical Properties of Al-Cu Alloy Foams: *Zan Wang*<sup>1</sup>; Byeonggu Kim<sup>1</sup>; Rui Zhao<sup>1</sup>; Yuxuan Li<sup>1</sup>; Boyoung Hur<sup>1</sup>; <sup>1</sup>Gyeongsang National University

Metal foam, as one of the most interesting materials in the field of functional materials, has so many excellent properties, such as lightweight, incombustible, thermal insulation, energy absorption and pro-environment, that it can be used in such various fields of automotives, transports, ships and aerospace applications. And in this study, Al-Cu alloy was used to manufacture the closed-cell metal foam, by putting certain tackifier and foaming agent into molten metal. The porosity, pore size and distribution, compressive and impact ability and hardness of the foam were investigated. I-Solution program was used to measure the pore size and distribution; High-Resolution Scanning Electron Microscope and Energy Dispersive Spectrometer was used to analyze the microstructure and composition of the surface. Compressive test was carried out by using Universal Material Testing Machine, and compressive strength decreased with the increasing of the porosity. Impact energy absorption test was tested with IZOD IMPACT TESTER, and the higher the porosity was, the smaller the impact energy absorption was. The hardness was tested by the micro hardness tester and the results, was much higher than that of pure aluminum because of the addition of Cu.

C46 Development of Wrought Magnesium Alloys by an Improved Neural Network Model: *Aitao Tang*<sup>1</sup>; Bin Liu<sup>1</sup>; Weiqing Wang<sup>1</sup>; Jingfeng Wang<sup>1</sup>; Fusheng Pan<sup>1</sup>; <sup>1</sup>Chongqing University

Many new types of magnesium alloys are being developed in the world in order to further improve the mechanical properties and processing performances of the magnesium alloys. However, conventional methods for developing new alloys need a lot of experimental work and take long time. In the present work,



the improved neural network model was developed by refining the types of input variables and using a more reasonable algorithm, and was used to develop new types of wrought magnesium alloys. The work was focused on the design and development of Mg-Zn-Zr-RE, Mg-Zn-Zr-Sr and Mg-Zn-Mn alloys. The results showed that the prediction errors were small by using the improved model when alloy compositions were limited to the range suggested. Three new types of wrought magnesium alloys have been developed by using the model, which had satisfactory comprehensive mechanical properties. It is suggested that the model may also be used to design new types of casting magnesium alloys.

#### **C47 Thermal Analysis of a Bi-Metallic Chill Vent for High Pressure Die Casting**: *Mohammad Imran*<sup>1</sup>; Rajpreet Dhaliwal<sup>1</sup>; Syed Masood<sup>1</sup>; <sup>1</sup>Swinburne University of Technology

High Pressure Die Casting (HDPC) is a widely used metal deformation process used for manufacturing aluminium parts in automotive industry. In high pressure die casting mould, chill vents are used to allow residual air and gases to exhaust out from the mould cavity. The objective of this paper is to develop a bi-metallic chill vent for high pressure die casting using high strength copper alloy material having high thermal conductivity coated with a thin steel layer. Transient thermal analysis was carried out and cooling rate, cooling efficiency and temperature distribution results were compared with copper and tool steel chill vents. It is concluded that copper based bimetallic chill vent could provide much better heat extraction with added strength for the HDPC process. The paper also presents the effects of varying internal diameter of cooling fountain channels in chill vent on cooling efficiency using the transient thermal analysis on ANSYS Workbench simulation system.

### **C48 TiFe Alloy Prepared by Molten Salt Electrolysis Ilmenite**: *Du Jihong*<sup>1</sup>; <sup>1</sup>Northwest Institute for Nonferrous Metal Research

Titanium content in the crust of the abundant elements, the distribution of resources is very broad, more than 30 countries have main the world's titanium resources. There are industrial utilization value that are mainly ilmenite, anatase, brookite, white titanium, perovskite and rutile. The ilmenite and rutile are a large number exploited .And ilmenite is majority . The rutile titanium resources is a very small proportion. With the rutile resources diminishing, These will become a very important issue that ilmenite resources are used economy and reasonable. Therefore, we introduce that TiFe alloy was prepared by salt electrolysis ilmenite. The results showed that reaction of the process is to gradually restore the generation of titanium alloy from ilmenite, ilmenite reduction experienced a priority to generate Fe and gradually TiFe2, TiFe alloy. The first reaction of the resulting alloy is TiFe<sub>2</sub> alloy, Ti and TiFe<sub>2</sub> through inter-diffusion turn to TiFe alloy, So the proliferation reaction is control of steps. When same electrolytic conditions, the ilmenite deoxy more difficult than the mixed oxide electrolysis is due to larger ilmenite particles and impurity that is the solution to the iron titanate

#### **C49 Effect of Bi Addition on Thermal Stability and Tensile Ductility of Mg-3%Zn-0.4%Zr Alloy**: Joong-Hwan Jun<sup>1</sup>; *Min-Ha Lee*<sup>1</sup>; <sup>1</sup>Korea Institute of Industrial Technology

Up to date, various grain refining processes such as severe plastic deformation (SPD) and large strain rolling etc. have been developed and examined. In spite of numerous studies on grain refinement of Mg alloys and their characterization, relatively fewer efforts have been devoted to fabrication of fine-grained microstructure by an addition of grain refining element into Mg alloy. Considering a previous report that Bismuth (Bi) plays a beneficial role in refining grains of Mg-Si alloy effectively, it is reasonable to expect that Bi addition might enhance tensile ductility for Mg-Zn-Zr wrought alloy. The objective of this study is to investigate and compare the thermal stability of recrystallized grains and their tensile ductility at elevated temperature for Mg-3%Zn-0.4%Zr and Mg-3%Zn-0.4%Zr-1.0%Bi alloy sheets. The Mg-3%Zn-0.4%Zr-1.0%Bi alloy shows higher elongation at elevated temperature. By the addition of 1%Bi, the recrystallized grains become smaller and possess higher thermal stability, with the introduction of Mg3Bi2 phase inside the grains. All these microstructural evolutions are thought to be responsible for the enhanced tensile ductility of the Bi-added Mg-Zn-Zr alloy

#### C50 Mill Scale for Synthesis of Fe–Ni and Fe–Ni–Co Alloys through Hydrogen Reduction: *M.K. Paek*<sup>1</sup>; D.H. Kim<sup>1</sup>; K.H. Do<sup>1</sup>; M. Bahgat<sup>2</sup>; J.J. Pak<sup>1</sup>; <sup>1</sup>Hanyang University; <sup>2</sup>Central Metallurgical Research and Development Institute

Posters

This work demonstrated an economic route for synthesis of Fe–Ni and Fe–Ni–Co alloys from secondary iron oxide resources, mill scale. Nickel and Nickel cobalt ferrite powders (NiFe<sub>2</sub>O<sub>4</sub>, Ni<sub>0.5</sub>Co<sub>0.5</sub>Fe<sub>2</sub>O<sub>4</sub>) were prepared through the ceramic route by calcination of a stoichiometric mixture of nickel oxide, cobalt oxide and mill scale as a source for the iron oxide. The produced ferrites compacts were isothermally reduced in pure hydrogen atmosphere from 800 to 1100°C. The reduction was studied by thermogravimetric analysis to determine the kinetics and reaction mechanisms. The initial ferrite compacts and the various reduction

products were characterized by XRD, SEM and reflected light microscope to reveal the effect of hydrogen reduction on composition, microstructure and reaction kinetics of synthesized ferro-alloys. The Arrhenius equation with mathematical formulations for the heterogeneous gas–solid reaction was applied to calculate the activation energy values and to determine the controlling reaction mechanism. Complete reduction of each ferrite compact was achieved resulting in a nanocrystalline Fe–Ni and Fe–Ni–Co alloys.

#### **C51 Effect of Applied Stress on Nucleation and Growth of Precipitates in a Cu-Be-Co Alloy**: *Ryoichi Monzen*<sup>1</sup>; Tadashi Terazawa<sup>2</sup>; Chihiro Watanabe<sup>1</sup>; <sup>1</sup>Division of Innovative Technology and Science, Kanazawa University; <sup>2</sup>Division of Mechanical Science and Engineering, Kanazawa University

The influence of an external stress on the nucleation and growth of G.P. zones and  $\gamma'$  precipitated phase has been investigated for a Cu-1.2wt%Be-0.1wt%Co alloy aged at 220°C. A compressive stress applied in the [001] direction during aging accelerates preferentially the nucleation and growth of the G.P. zones perpendicular to the [001] axis, whereas a tensile stress does not affect those of the G.P. zones. The  $\gamma'$  phase heterogeneously precipitates on the  $\gamma''$  or  $\gamma'_{\perp}$ phase under the compressive stress, different from our previous result that the heterogeneous formation of  $\gamma'$  under no stress occurs on the  $\gamma_i$  phase. The compressive stress causes the preferential nucleation and growth of specific  $\gamma'$ variants among crystallographically equivalent ones, but the tensile stress does not influence those of  $\gamma'$ . The promotion of the nucleation and growth of the G.P. zones perpendicular to the stress axis and specific  $\gamma$ ' variants under compression can be well understood through the interaction energy between the external stress and the misfit strains of G.P. zones and y' phase. The critical diameter of the diskshaped G.P. zone nucleus was estimated as about 1.3 nm from evaluation of the interaction energy.

#### Poster Session: Symposium D: Bulk Metallic Glasses and Nanomaterials

Tuesday PM	Room: Hall 2
August 3, 2010	Location: Cairns Convention Centre

**D1 A New Parameter for Evaluating Glass-Forming Ability of Metallic Glasses:** Je-In Lee<sup>1</sup>; Eun Soo Park<sup>1</sup>; <sup>1</sup>Seoul National University

Recent studies on bulk metallic glasses (BMGs) show that a proper estimation of glass forming ability (GFA) is important in developing new BMG systems. The critical cooling rate for glass formation (Rc) may be a proper measure of GFA. However, an accurate measurement of Rc is not simple, especially at a high cooling rate. Alternatively, the maximum section size of BMG (Dmax) has often been used as a measure of GFA. Several other parameters have been proposed as well to estimate GFA considering thermodynamic and/or kinetic aspects of glass formation, i.e., (1)  $\Delta Tx(=Tx-Tg)$ , (2) K(=[Tx-Tg]/[Tl-Tx]), (3) Trg(=Tg/Tl), and (4)  $\gamma$ (=Tx/[Tl+Tg]). Even though these parameters offer some useful guideline on alloy design, our ability to evaluate GFA is still limited. In the present study, we propose a new parameter for evaluating GFA of metallic glasses based on the combination of thermodynamic, kinetic and atomic structural aspects of glass formation. The proposed parameter shows a higher regression coefficient value and narrower prediction band compared to the four other criteria, implying that this parameter is highly correlated with Rc and GFA. Indeed, this parameter can give some ideas about how we can select alloy composition in order to reduce experimental efforts.

## **D2** Bulk Metallic Glass Formation, Crystallization, and Magnetic Properties of RE<sub>4</sub>Fe<sub>22</sub>B<sub>22</sub>M<sub>2</sub> Alloys: XueHong Cui<sup>1</sup>; Z.W. Liu<sup>1</sup>; D.C. Zeng<sup>1</sup>; <sup>1</sup>South China University of Technology

 $RE_2Fe_{14}B/Fe$  and  $RE_2Fe_{14}B/Fe_3B$  nanocomposites have shown increasing interests due to their enhanced remanence and maximum energy product resulted from the exchange coupling between hard and soft magnetic phases. This paper reports our recent attempt to prepare high density nanocomposites by devitrifying amorphous magnets. The RE4Fe<sub>72</sub>B<sub>22</sub>M<sub>2</sub> (RE=Y, Dy or Nd; M=Nb, Ta or Zr) alloys in ribbon and rod forms were prepared by melt spinning and water cooling copper mold casting methods, respectively. The XRD patterns showed that all ribbon samples were fully amorphous, but the results form rod samples indicated that the composition has an important effect on the glass formability. All rod samples with RE=Y or Nd were partly amorphous, while fully amorphous structure was obtained for Dy<sub>4</sub>Fe7<sub>2</sub>B<sub>22</sub>Nb<sub>2</sub> alloy rod with a diameter of 2 mm. The rare earth and doping elements on the glass formability have been discussed regarding to the underlying physics. The effects of post-annealing on the microstructure and magnetic properties of these alloys have been investigated. The results showed



that the nanocomposite structure of  $Fe_3B+a-Fe+RE_2Fe_{14}B$  phases with good permanent magnetic properties has been obtained by optimal heat treatment. The results presented here provide a viable method to get fully dense bulk hard magnets.

**D3** Effects of Partial Substitution of Zr with Y in Cu-Zr-Al Bulk Metallic Glasses: *J.W. Kim*<sup>1</sup>; E.S. Park<sup>1</sup>; R.T. Ott<sup>2</sup>; <sup>1</sup>Research Institute of Advanced Materials, Department of Materials Science and Engineering, Seoul National University; <sup>2</sup>Materials and Engineering Physics Program, Ames Laboratory (USDOE)

It has been widely accepted that one of the crucial BMG-forming conditions is the presence of a large negative enthalpy of mixing among constituent elements, which makes glass more stable. On the other hand, from the thermodynamics of glass formation we know that increased difference in the enthalpy of mixing among constituent elements leads to greater heterogeneity. Indeed, how to control the degree of heterogeneity in bulk metallic glasses (BMGs) is an open question because most of BMGs have more than one repulsive atomic pair. In the present study, we systematically explore the effects of partial substitution of Zr with Y (Zr-Y: +35 kJ/mol) in Cu-Zr-Al BMGs. In our experiments, a small amount of substitution results in atomic scale heterogeneity in as-cast BMGs, which can lead to the improved plasticity of BMG by inhomogeneity of local viscosity. On the other hand, a large amount of substitution can lead to phase separation in the miscibility gap region forming two different glasses which exhibit nearly zero plasticity. These findings suggest that optimization of heterogeneity by tailoring combination of constituent elements in BMGs could induce further improved plasticity of BMGs.

**D4 Manufacture of Soft Magnetic, Four-Component Fe**<sub>78-x</sub>Y<sub>4+x</sub>Nb<sub>5</sub> **B2 Bulk Amorphous Alloys**: *Michal Szota*<sup>1</sup>; Marcin Nabialek<sup>1</sup>; Marcin Dospial<sup>1</sup>; <sup>1</sup>Czestochowa University of Technology

The samples were prepared in the form of plates and ribbons with a thickness of 0.5mm and 40µm, respectively in an inert gas atmosphere. Four-component alloys in the form of massive amorphous plates were produced using a suctioncasting method consisting of suction of a liquid, homogenous allov onto water cooled copper mould, while the sample in the form of ribbons were produced using classical melt-spinning method. The X-ray diffraction and Mössbauer spectroscopy have been used to confirm amorphous structure of the samples. The investigated materials display good soft magnetic properties: high saturation of the magnetization, low coercivity field and core looses. Set of measurements of Curie temperature for all samples were performed using magnetic weight. Thermal stability of the investigated alloys was obtained using a differential scanning calorimeter (DSC). From the DSC plots several characteristic temperatures including: the glass transition temperature (Tg), the crystallization temperature (Tx), the melting temperature (Tm), the liquidus temperature (Tl), and some GFA parameters ( $\delta$ ,  $\gamma$ m) were derived. Samples in the form of ribbons for all investigated alloys, much more easily reach a ferromagnetic saturation state, have a slightly lower temperature of the beginning of crystallization.

#### **D5 Mechanical Behaviors of Bulk Metallic Glass at Cryogenic Temperature**: *Yoon Kyeu Sang*<sup>1</sup>; Lee Jae-Chul<sup>1</sup>; Sa Hyeon-Jei<sup>1</sup>; <sup>1</sup>Korea Unversity

A number of experiments carried out on various amorphous alloys at cryogenic temperatures demonstrated very interesting mechanical behaviors that were not observable in crystalline metals. Unlike the crystalline metals, amorphous alloys exhibited a higher strength, but became more ductile at cryogenic temperatures. For example, the strength and plasticity of the  $Cu_{57}Zr_{43}$  bulk amorphous alloy are 2.0 GPa and 4%, respectively, at room temperature. However, when tested at cryogenic temperature, it showed a dramatic increase in strength and plasticity, reaching 2.3 GPa and 21%, respectively. In this study, we used a simple Cu-Zr binary alloy to explain the basis of the enhanced plasticity at cryogenic temperatures by exploring the behaviors of the shear band propagation and serrated flow.

### **D6 Medium- to Extended-Range Order of Amorphous Alloy**: *Mirim Lee*<sup>1</sup>; Jae-Chul Lee<sup>1</sup>; <sup>1</sup>Korea University

The local structural states of amorphous alloys have been depicted previously via short-range order (SRO). However, the concept of SRO alone is sometimes inadequate and insufficient to explain the structure-property relation of amorphous alloys. In this study, we propose a new type of structural organization that forms the backbone of amorphous solids: the extended connection of icosahedron (ECOIs) and their extended-range order. The mechanical properties, especially plasticity, of these amorphous alloys are very sensitive to the structural stability of the ECOIs, as characterized by their interconnecting features with neighboring icosahedra. Using molecular dynamics (MD) simulations, we explore specific morphology of ECOIs, and their role in mechanical response to relate the structure-property relation.

**D7** Microstructural Assessment of the Oxidation Behavior of Cu-Based Metallic Glass Powder: *Ming Yan*<sup>1</sup>; Peng Yu<sup>1</sup>; Xiaopeng Li<sup>1</sup>; Graham Schaffer<sup>1</sup>; Ma Qian<sup>1</sup>; <sup>1</sup>The University of Queensland

Oxidation behaviour study bears two folds importance to metallic glass: one is with scientific value, given that the oxidation mechanism for conventional alloys is well established yet this is not the case for metallic glass; the other is of technical purpose, since such studies will realize the atmospheric tolerance limits for processing metallic glass without catastrophic corrosion due to oxidation - this is particularly important to powder metallurgy of metallic glass. In this study, gas-atomized metallic glass powders were intentionally heat-treated in an oxygen atmosphere within a broad temperature window, i.e. from well below glass transition temperature to temperatures higher than crystallization temperature. Analytical techniques, such as elemental mapping (based on scanning electron microscopy), electron backscattered scanning diffraction (EBSD) and electron diffraction (based on transmission electron microscopy), were employed to investigate the microstructual evolution due to oxidations of the differently treated powders. Oxidation mechanism was subsequently discussed based on the microstructural findings.

D8 Relaxation Behavior of Zr-Cu-Al Ternary Bulk Glassy Alloy Studied by Using Positron Annihilation Techniques: *Akito Ishii*<sup>1</sup>; Shinya Mineno<sup>1</sup>; Akihiro Iwase<sup>1</sup>; Yoshihiko Yokoyama<sup>2</sup>; Toyohiko Konno<sup>2</sup>; Fuminobu Hori<sup>1</sup>; <sup>1</sup>Osaka Prefecture University; <sup>2</sup>Tohoku University

Zr-based bulk glassy alloys (BGAs) show high tensile strength and a high Chrapy impact value. In this study, the free volume relaxation process for eutectic Zr<sub>50</sub>Cu<sub>40</sub>Al<sub>10</sub>BGA and hypoeutectic Zr<sub>60</sub>Cu<sub>30</sub>Al<sub>10</sub>BGA during isothermal annealing below glass transition temperature have been investigated by positron annihilation lifetime and coincidence Doppler broadening (CDB) measurements. The positron lifetime for hypoeutectic and eutectic BGA is almost the same, although the density of hypoeutectic BGA is obviously greater than that of eutectic one. The CDB spectra indicate a marked difference between eutectic and hypoeutectic BGA; that is, the spectrum of the hypoeutectic BGA is more similar to that of Zr metal than that of the eutectic BGA. This result exhibits that the fraction of Zr atoms around free volume in hypoeutectic BGA is greater than that in eutectic BGA. The CDB ratio profiles for hypoeutectic BGA during annealing show no appreciable change. We have reported that long-range diffusion around free volume does not take place in eutectic BGA during relaxation [1]. The same trend was observed in hypoeutectic BGA. That is, the relaxation process of free volume for these BGA is almost the same. [1] A. Ishii et al. 2008 Mater. Trans. 49

**D9 Ti Base Metallic Glasses for Telescope Crown Partial Denture**: *Jeong-Jung Oak*<sup>1</sup>; Joonho Cho<sup>2</sup>; Junji Saida<sup>3</sup>; Keiichi Sasaki<sup>2</sup>; Akihisa Inoue<sup>1</sup>; <sup>1</sup>Institute for Materials Research, Tohoku University; <sup>2</sup>Graduate School of Dentistry, Tohoku University; <sup>3</sup>Center for Interdisciplinary Research, Tohoku University

We have attempted development of Ti base metallic glasses for partial denture. Due to the characteristics of titanium and its alloys, i.e. lightweight, excellent corrosion resistance and high strength for engineering structural materials, they have been applied for aircraft engine, biomedical devices as well as chemical processing unit partials. Meanwhile, it is also well known that metallic glasses exhibit the distinguished properties, i.e. low elastic modulus with specific strength, excellent electrochemical stability in corrosion environment as well as good workability as like plastics. The fabrication of Ti base metallic glasses is expected to have a significant potential for application by the combination of good properties of both materials above. The toughness of newly designed Ti base metallic glasses is comparable with that of crystalline alloys in fracture deformation. Moreover, plastic deformation more than 2% is exhibited during compression in Ti base metallic glasses, which is almost equivalent to their elastic deformation. The result might be recognized as a hot issue in novel materials science in Ti base alloys because of the improvement of machining-workability and the possibility of further application as like common alloys. The upgraded properties in Ti base metallic glasses will be discussed in detail.

#### **D10 Effect of Isothermal Annealing on the Corrosion Resistance of an Amorphous Alloy**: *Sang Soo Shin*<sup>1</sup>; Kyung Mook Lim<sup>1</sup>; Eok Soo Kim<sup>1</sup>; Jae Chul Lee<sup>2</sup>; <sup>1</sup>KITECH; <sup>2</sup>Korea University

This study examined the role of excess free volume on the corrosion resistance of an amorphous alloy. Corrosion behaviors were monitored on the amorphous alloys, of which amount of free volume was controlled via the isothermal annealing below the glass transition temperature, using immersion tests and potentiodynamic polarization tests in HCl aqueous solutions. It was found that the corrosion resistance of the amorphous alloy is improved by reducing the amount of excess free volume. The possible reason explaining the experimental result was discussed from the viewpoint of the potential energy (eV) associated with the annihilation of excess free volume.



**D11 Synthesis and Mechanical Property of FeSiBPCu Bulk Nano-Structured Material**: *Yan Zhang*<sup>1</sup>; Sangmin Lee<sup>1</sup>; Hidemi Kato<sup>1</sup>; Akihiro Makino<sup>1</sup>; Akihisa Inoue<sup>1</sup>; <sup>1</sup>Institute for Materials Research, Tohoku University

Bulk nano-structured material (BNM) was fabricated by compacting amorphous  $Fe_{s_3}B_9Si_2P_4Cu_{0,7}$  alloy shown by differential calorimetric measurement to have no glass transition temperature. Spark plasma sintering (SPS), capable of rapid heating and cooling, enabled us to obtain a densely compacted BNM through viscous deformation of amorphous ribbon particles. Vickers hardness of BNM samples, being expected to consist of about 20 nm grains, were 1300 Hv on average, which was approximately 1.7- and 2.8-fold that of the as-melt spun amorphous ribbon and that of the fully annealed ingot, respectively. The stress-induced occurrence of viscous flow was observed by comparing findings of thermomechanical analyses (TMA) and the displacement curves of the SPS, indicating that the flow temperature (Tf) of the BNM sample during SPS was 200 K lower than the crystallization temperature (T<sub>x</sub>) shown by differential scanning calorimetry (DSC) at a heating rate of 1.67 K/s.

## **D12** The Characterization and Gas Sensing Properties of Polythiophene Coated V<sub>2</sub>O<sub>5</sub> Nanotubes: Yu Lu<sup>1</sup>; Wei Jin<sup>1</sup>; Ying Dai<sup>1</sup>; Wen Chen<sup>1</sup>; <sup>1</sup>School of Material Science and Engineering, Wuhan University of Technology

Inorganic one dimensional (1D) nanomaterials based gas sensors are still limited by relatively high working temperature and poor stability. Recently, to overcome these disadvantages, organic-inorganic one dimensional nanomaterials hybrid sensors are intensively investigated. V2O2/PTP core-shell nanotubes were prepared through an in-situ polymerization of thiophene monomers in the presence of prepared V2O5 nanotubes. The hybrids were characterized by Xray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM), which proved the polymerization of thiophene monomer and the strong interaction between polythiophene and VONTs. The gas sensing properties of V<sub>2</sub>O<sub>6</sub>/PTP nanotubes at room temperature were studied. It was found that V<sub>2</sub>O<sub>5</sub>/PTP nanotubes could detect ethanol with much higher sensitivity at room temperature than pure VONTs. Moreover, the influences of thiophene content, reaction time and temperatures on the structure and gas sensing properties of V2O5/PTP nanotubes were investigated. The sensing mechanism of V<sub>2</sub>O<sub>5</sub>/PTP nanotubes to ethanol was presumed to be the synergetic interaction between polythiophene (PTP) and VONTs.

D13 The Bandgap Characteristics of 2-D Square Lattice Photonic Crystals with Square Air Holes: Yang Yibiao<sup>1</sup>; Li Xiujie<sup>2</sup>; Wang Yuncai<sup>1</sup>; Wang Shuanfeng<sup>1</sup>; Han Peide<sup>2</sup>; Liang Wei<sup>2</sup>; <sup>1</sup>Department of Physics, Taiyuan University of Technology; <sup>2</sup>College of Materials Science and Engineering, Taiyuan University of Technology

The photonic bandgaps of 2-D photonic crystals with Square lattice are studied with plane wave expansion method. The effects on bandgap for a square lattice of air holes in dielectric are discussed as a variety of the rotation angle and the refractive index. A kind of Square lattice photonic crystal with rotational square air holes in dielectric which has excellent performance was presented. The simulation results show that complete photonic band gap can be obtained continuously while the dielectric refractive index is greater than 2.61, and as the refractive index increases, the complete photonic bandgap become larger to the extent that it reaches the maximum value when the refractive index is 3.70. Moreover, the rotation angle of dielectric holes remains 30° when the maximum complete photonic band gap appears. For this structure, the maximum complete photonic band gap width is within 0.059~0.060( $\omega a/2\pi c$ ), and the filling fraction f is 0.3276 when the refractive index changes from 3.25 to 3.73 continuously. The gap width to midgap frequency ratio exceeds 13%. These results are significant for the fabrication and applications of two dimensional photonic crystals.

D14 Gold Nanoparticles in Red Ruby Glasses Used for Decoration in Thailand: *Pisutti Dararutana*<sup>1</sup>; Narin Sirikulrat<sup>2</sup>; Pornsawat Wathanakul<sup>3</sup>; <sup>1</sup>The Royal Thai Army Chemical Department; <sup>2</sup>Glass and Glass Products Research and Development Laboratory; <sup>3</sup>Gemmology and Mineral Sciences Special Research Unit

Red ruby glasses have been used for old-style decorations in Thailand such as ancient Thai glass, enamel and glass bead. Most of them were made by gold addition into molten lead glasses. Their composition and structure were determined using scanning electron microscope with energy dispersive X-ray fluorescence spectrometer (SEM-EDX)and proton induced X-ray emission(PIXE). X-ray absorption spectroscopy (XAS) was carried out to study the oxidation of gold. UV-VIS spectra were also recored using an UV-VIS-NIR spectrophotometer. It was found that the presence of chromophore was gold nanoparticles. The details were discussed. D15 Soft Magnetic Properties of Nanocrystalline Fe-Based P/M Cores Mixed with Various Polymer Binders: *Mi Rae Kim*<sup>1</sup>; Kyu Seong Kim<sup>1</sup>; Sung-Min Jeon<sup>1</sup>; Sun-I Kim<sup>1</sup>; Keun Yong Sohn<sup>1</sup>; Won-Wook Park<sup>1</sup>; <sup>1</sup>InJe University

One of the most interesting nanocrystalline magnetic materials is FINEMET type Fe-Si-B-Nb-Cu base alloys. Melt-spun amorphous Fe<sub>73</sub>Si<sub>16</sub>B<sub>7</sub>Nb3Cu1(at.%) allow strip firstly iet-milled to make powders, and then ball-milled for 30 hrs to get homogeneous fine powder. The powders were mixed with polyimide-based binder, water glass and silicon compound of ~ 3 wt.% respectively, followed by cold compaction to form toroidal-shaped soft magnetic cores. Crystallization treatment at 350°C~580°C was performed to control the nano-grain structure. Among the P/M cores, the toroidal Fe-based core mixed with polyimide binder showed the best magnetic properties. Well-coated polymer binder on the powder surface decreased remarkable the coercivity(0.11Oe @0.1T, 50kHz) and the core loss(20w/kg @0.1T. 50kHz, commercialized Amorphous core : 88w/kg) with the insulation of each powder. The transmission electron microscopy revealed that the best magnetic properties of powder cores was obtained after annealing for 1h at 520°C~580°C. The annealing conditions to obtain the grain size of 10~20nm were be optimized considering the stored energy generated during the powderforming process.

D16 Synthesis and Characterization of CdSe Microspheres via Solvothermal Process: Juan Yang<sup>1</sup>; Chuanliang Zang<sup>1</sup>; Xiaonong Cheng; <sup>1</sup>Jiangsu University

CdSe particles with wurtzite structure have been synthesized via solvothermal method using a mixed solution of triethylenetetramine (TETA) and de-ionized water (DIW) without adding reducing agent. It was found that ball-like CdSe precursor with zinc-blende phase could be transformed to wurtzite structure after heat-treating at 580°C in Ar atmosphere and the obtained microspheres were composed with small CdSe particles. The experimental results were compared with CdSe obtained via hydrothermal method using N<sub>2</sub>H<sub>4</sub>•H<sub>2</sub>O as the reducing agent and it was found that CdSe nanorods with wurtzite structure could be obtained. It was speculated that TETA in the mixed solution played the role of reducing agent and surfactant. Both the as-prepared products and the annealed powders were systematically characterized by powder X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), Fourier transform infrared absorbance spectroscopy (FTIR) and thermogravimetric analysis (TGA).

**D17 High Strength and High Ductility in Electrodeposited Nanocrystalline Ni–W Alloy**: *Kazutaka Fujita*<sup>1</sup>; Taiji Suidu<sup>2</sup>; Tohru Yamasaki<sup>3</sup>; <sup>1</sup>Department of Mechanical Engineering, Ube National College of Technology; <sup>2</sup>Advanced Course of Production Systems Engineering (Student), Ube National College of Technology; <sup>3</sup>Department of Materials Science and Chemistry, University of Hyogo

The tensile specimen of nanocrystalline Ni-W alloys with 50  $\mu$ m x 20  $\mu$ m in area and 4 mm in length at the parallel part was made by using electrodeposite together with UV (ultraviolet light) lithographic techniques. The composition and grain size were Ni-16.9 at. % W and about 6 nm, respectively. The nominal tensile strength and Young's modulus were about 2.8 GPa and 120 GPa, respectively. The elastic strain and total strain until fracture were about 2% and 3%, respectively. The stress-strain curve showed a plastic strain with work hardening. The macroscopic fracture part yielded necking and the microscopic fracture surface showed dimple pattern with a size of about 200 nm. As mentioned above, this electrodeposited nanocrystalline Ni-W alloy showed high strength and high ductility.

D18 Influence of Solidification Speed on Quality and Quantity of Structural Defects in  $Fe_{61}Co_{10}Zr_{2.5}Hf_{2.5}Y_2W_2B_{20}$  Amorphous Alloy: *Michal Szota*<sup>1</sup>; Marcin Nabialek<sup>1</sup>; Marcin Dospial<sup>1</sup>; <sup>1</sup>Czestochowa University of Technology

The study of an influence of solidification speed on structural defects in bulk  $Fe_{61}Co_{10}Zr_{2.5}H_{2.5}Y_2W_2B_{20}$  amorphous alloy was performed. The investigated samples were prepared as a 0.5mm and 1mm thick plates. The microstructure was examined using X-ray diffraction and Mössbauer spectroscopy. The quality and quantity of structural defects were determined by indirect method from measurements of magnetization close to ferromagnetic saturation. On the basis of the results obtained from studies carried out in strong magnetic fields, it was found that the thickness of the amorphous material and production method have a significant influence on the size and type of defects appearing in it. It was found that both samples contain two-dimensional quasi-dislocation dipoles with slightly different size.

D19 FePBNbCr Soft Magnetic Glassy Alloys "SENNTIX" with Low Loss Characteristics for Commercial Inductor Cores: *Hiroyuki Matsumoto*<sup>1</sup>; Akiri Urata<sup>1</sup>; Yasunobu Yamada<sup>1</sup>; Akihisa Inoue<sup>2</sup>; <sup>1</sup>NEC TOKIN Corporation; <sup>2</sup>Tohoku University

The inductor for a power supply is expected to having higher efficiency and capability of dealing satisfactorily with large current. Additionally, high corrosion resistance characteristics are also required for commercial inductor in a practical use. Thereby, we focused on Fe-based glassy metal alloys with both

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high magnetization and low magnetic anisotropy, and developed the novel glassy metal alloys with a chemical composition Fe(97-x-y)PxByNb2Cr1. In this glassy metal alloy, 1at% Cr is the optimum composition for realization higher corrosion resistance as well as the high magnetic flux density. The glassy metal alloy Fe(97-x-y)PxByNb2Cr1 (x-5.6.7.8.9.10.11.13, y-7.9.10.11.12.13.14.15) exhibits the high glass-forming ability leading to the large thickness of 110-150  $\mu$ m and low coercive force of 2.5-3.1 A/m due to higher structural homogeneity in wide range of chemical composition. The large critical thickness of this alloy should be caused by the high glass-forming ability (GFA) due to the existence of the super cooled liquid region ( $\Delta$ Tx) of roughly 30 K. Therefore Fe77P7B13Nb2Cr1 powder/resin composite core reveals much lower core loss of 650 W/m3 than the conventional amorphous Fe73Si10B15Cr2 powder/resin composite core by approximately 1/3.

#### D20 Nanoscale Structure and Magnetic Properties of Nd-Fe-Nb-Zr-B Permanent Magnets: Xiuyun Chen<sup>1</sup>; <sup>1</sup>Tsinghua University

Nd2Fe14B/ $\alpha$ -Fe nanocomposite permanent magnet is a new type of permanent magnets. In a new kind of alloy the samples with several Nd content have been studied. The sample with Nd content of 10.9 at% has an optimum volume fraction and a relatively smaller grain size, which causes a relatively higher intrinsic coercivity, remanence and therefore a highest maximum energy product. Adding Zr to the alloy is effective to refine grains, grain size refinement has a significant influence on coercivity. Addition of Zr also can enhance the ability of glass-forming. The sample with Zr content of 1.0 at% has the smallest grain size of 17 nm and therefore the intrinsic coercivity is remarkably enhanced. MFM (Magnetic Force Microscope) was used to observe the magnetic microdomain structure in the nanophase alloy. The length of the magnetic contrast shows a significant dependence on the microstructure and phase constitution, and the longer length is correspond with the larger exchange coupling effect between the soft and hard magnetic phases.

**D21** Synthesis of Fe Stainless Steel Alloy NanopowderS by Electrical Explosion of Wire in the Liquid: *Jin-Chun Kim*<sup>1</sup>; L.H. Bac<sup>1</sup>; Ji-Soon Kim<sup>1</sup>; Young-Soon Kwon<sup>1</sup>; <sup>1</sup>University of Ulsan

Fe-stainless steel alloys have been used in various fields of industry due to a variety of usefully corrosion properties. For instance, it has excellent resistance to stain or rust due to its chromium content, usually from 12 to 20 percent of the alloy. The Fe stainless steels have several types depending on their microstructure. The Fe-stainless steel alloys nanostructured and nanoparticles have received much attention in the recent decade because they exhibit good mechanical properties. In this paper, we report to synthesize the Fe 304 stainless stell alloy nanopowder by electrical explosion of wire in liquid. X-ray diffraction was used to investigate the phase of nanopowder. The results show that nanopowder is in Fe solid solution. Particles size and morphology were observed by electron transmission microscope. The nanoparticles are in nearly spherical shape with average size of 50 nm. The compositions of the wire before exploding and the nanopowder were determined using electron probe microanalysis.

## **D22** Effect of Electric Field on Cuo Nanoneedle Growth during Thermal Oxidation and Its Growth Mechanism: *Xiaozhu Li*<sup>1</sup>; Chunxu Pan<sup>1</sup>; <sup>1</sup>Shaoguan University

It is unreasonable to explain the growth of CuO nanoneedles by using the vapor-liquid-solid (VLS) or vapor-solid (VS) model during thermal oxidation, because the growth temperature is much lower than the melting points of Cu and its oxides, and no catalyst is required. In our previous work, we proposed a novel "solid state based-up diffusion growth mechanism", which indicates that the driving force for growing metal oxide nanoneedles is a local electrical field set up by the ionization phenomenon of the metal and O atoms at the solid/gas interface, and it exhibit a self-limiting character. In this work, different electric field strengths were added along the growth direction during growth of CuO nanoneedles. The results show that: 1) the length of CuO nanoneedles increased with the electric field, but when the voltage was greater than a certain value, the growth stopped: 2) the diameter of CuO nanoneedles from top to root became more uniform. Therefore, it is further demonstrated the "solid state based-up diffusion growth mechanism" for CuO nanoneedles prepared by thermal oxidation. The recent study also provides a possibility for controlling the growth of metal oxide nanowires which will promote their potential applications in nano-devices.

#### **D23 Effect of Organic Capping on the Magnetic Properties of Au Nanoparticles**: *Hao Zhang*<sup>1</sup>; Kiyonori Suzuki<sup>1</sup>; Kei Saito<sup>1</sup>; Jose Garitaonandia<sup>2</sup>; Eider Goikolea<sup>2</sup>; Maite Insausti<sup>2</sup>; <sup>1</sup>Monash University; <sup>2</sup>Euskal Herriko Unibertsitatea

One of the latest discoveries in magnetism in recent years is the chemically induced magnetism in organically-capped nanoparticles (NPs) of intrinsically nonmagnetic metals. It has been found that ferromagnetism is induced when Au NPs are capped with some organic compounds containing S. The ferromagnetism of Au NPs is believed to be due to the charge transfer of the d electrons of Au atoms to the surface S atoms of the capping compound. Hence, the magnetism induced should depend highly on the capping conditions. In this study we have investigated the magnetic properties of organically-capped Au NPs prepared with a range of processing conditions and the effect of the capping compound on the magnetic properties is discussed. The Au-regioregular poly (3-hexylthiophene) NPs were synthesized by a water/toluene two-phase one-pot Brust method where the concentration of reagent, reaction temperature, and stirring time are systematically altered. It was found that the average magnetic moment of the surface Au atoms depends on the reagent concentration. Since the creation of bonds between the surface Au atoms and the polymer chain depends on the reagent concentrations, our results suggest that the average magnetic moment is governed by the number of surface Au-S pairs.

#### **D24 Effects of Particle Size on the Heat Transfer in the Copper Nanopowder:** *C. Y. Ho*<sup>1</sup>; M.Y. Wen<sup>2</sup>; C. Ma<sup>1</sup>; <sup>1</sup>Hwa Hsia Institute of Technology; <sup>2</sup>Cheng Shiu University

Nanoparticles show a variety of size-dependent properties due to the dramatic changes in the ratio of surface area to volume. The characteristics of nanoparticles different from the bulk material make the properties of the powders composed of these nanoparticles attract more attention. The heat transfer in the powder composed of Cu nanoparticles is experimentally investigated in this paper. The temperature histories at two different locations in the slender tube are recorded using thermal couples. The results show that the heat transfer depends on the bed porosity and the thermal conductivities of the solid and the gas. The effects of particle size on heat transfer in the copper nanopowder are also discussed in this paper.

#### **D25 High Bs FeSiBPCu Nanocrystalline Wide Ribbons Using Industrial Raw Materials**: *Akiri Urata*<sup>1</sup>; Hiroyuki Matsumoto<sup>1</sup>; Shigeyoshi Yoshida<sup>1</sup>; Akihiro Makino<sup>2</sup>; <sup>1</sup>NEC TOKIN Corporation; <sup>2</sup>Institute for Material Research, Tohoku University

Recently, FeSiBPCu nanocrystalline soft magnetic alloys with both high Bs and good magnetic softness has been developed, which contains no Nb or Zr. These alloys are promising for a novel core material in electronic devices such as transformers and motors. In this study, FeSiBPCu nanocrystalline wide ribbons using industrial raw materials have been investigated aiming for an industrialization of the alloys. Fe<sub>84.8</sub>Si<sub>1</sub>B<sub>10</sub>P<sub>3</sub>Cu<sub>1.2</sub> ingot was prepared by induction melting using the mixture of industrial raw materials of Fe, Cu, Fe-99mass%Si, Fe-21mass%B and Fe-19mass%P in an Ar atmosphere. A single-roller meltspinning method in air was used to produce the rapidly solidified ribbons with 15 mm in width and about 20  $\mu$ m in thickness. The alloy ribbon annealed at 698 K has the high Bs of 1.82T and the low Hc of 8.5 A/m for a crystallized  $\alpha$ -Fe structure with homogeneous grains with around 15 nm in diameter. In addition, the alloy has a large economical advantage by using industrial raw materials about the cost.

## **D26 Patterning and Sintering Properties of Cu Nanocolloid**: *Sangsun Yang*<sup>1</sup>; Jae-Cheol Yoon<sup>1</sup>; Yong-Jin Kim<sup>1</sup>; Ji-Hun Yu<sup>1</sup>; <sup>1</sup>Koera Institute of Materials Science

Ink-jet printing is a kind of noncontact and direct process as a pattern on demand type. It is also possible to make a conductive metal nanoparticles pattern simply, continuously and economically. Therefore, there are many kinds of merits on ink-jet printing process considering the application field like TFT, PCB, FPD, RFID, Solar cell, etc. To apply nanoparticles to electrical printing technology, long time stability of nanoparticles without contamination in liquid is indispensable. Wire electric explosion process has been found about two hundred years ago and developed for the synthesis of nanoparticles from several ten years ago. Wire explosion process in liquid is a kind of new process to prepare stable nanoparticle colloids with high purity. Cu nano-colloid was prepared by wire electric explosion in de-mineralized water and anhydrous ethanol. The size of Cu nanoparticles in colloid is about 20 nm. Cu nano-colloid showed long time dispersion stability without the addition of surfactants and additives. We are also going to introduce a new low temperature sintering method using the reaction between Cu organic and polymer.

#### D27 Controllable Hydrothermal Synthesis of $\alpha$ -, $\beta$ -, $\gamma$ -, and $\delta$ -MnO<sub>2</sub> Nanomaterials Based on KMnO<sub>4</sub>/MnSO<sub>4</sub> Aqueous Reaction System: Chuan Yang<sup>1</sup>; Ying Dai<sup>1</sup>; Xinmei Pei<sup>1</sup>; Wen Chen<sup>1</sup>; <sup>1</sup>Wuhan University of Technology

We report a facile hydrothermal synthetic approach to selectively produce  $\alpha$ -,  $\beta$ -,  $\gamma$ -, and  $\delta$ -MnO<sub>2</sub> nanomaterials with different morphologies based on the redox reaction of KMnO<sub>4</sub>/MnSO<sub>4</sub>. Unlike other works to control phases mainly by changing the types of reaction reagents and ion concentrations, in our work, reaction condition such as temperature, pH, and PVP have been adjusted to control the growth of different four types of MnO<sub>2</sub>, such as  $\alpha$ -MnO<sub>2</sub> nanowires,  $\beta$ -MnO<sub>2</sub> nanowires,  $\gamma$ -MnO<sub>2</sub> nanoparticles/nanowires,  $\delta$ -MnO<sub>2</sub> nanoflakes. It's interesting to note that pH contributes to form tunnel or layered structures. Under a strong alkaline (pH=14) condition, the lamellar structure  $\delta$ -MnO<sub>2</sub> tends to be formed. In

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a strong acid environment,  $\alpha$ -,  $\beta$ -,  $\gamma$ - MnO<sub>2</sub> with 2×2, 1×1, 2×1 tunnel structures can be easily controllably obtained, respectively.  $\beta$ -MnO<sub>2</sub> is a thermodynamically stable phase and  $\alpha$ -,  $\gamma$ -MnO<sub>2</sub> tend to convert to  $\beta$ -type at higher temperature. With the addition of PVP,  $\gamma$ -MnO<sub>2</sub> nanomaterials can be obtained via the same processing condition of  $\alpha$ -MnO<sub>2</sub> nanowires. The controlled growth of MnO<sub>2</sub> nanomaterials is achieved and the related growth mechanism is discussed.

**D28** Current Steps in Poly(3-Hexylthiophene)/ZnO Nanobelt Hybrid Diodes: Wen Guo<sup>1</sup>; *Ya Yang*<sup>2</sup>; Junjie Qi<sup>2</sup>; Yue Zhang<sup>2</sup>; <sup>1</sup>State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing; <sup>2</sup>Department of Materials Physics and Chemistry, University of Science and Technology Beijing

We report the poly(3-hexylthiophene) (P3HT)/ZnO nanobelt hybrid p-n junction diodes characterized by using a conductive atomic force microscope (C-AFM). The diodes exhibited a turn-on voltage of about 2.5 V and ideality factor of about 11.6. The obvious current steps in the I-V characteristics under the reverse bias were clearly observed at room temperature. The origin of these steps is suggested to be attributed to the charge injection-trapping induced by nanoparticles on the surface of the ZnO nanobelt. The  $P_3$ HT/ZnO nanobelts are promising in potential applications as memory and logic nanodevices.

#### **D29** Combined Field and Thermionic Emission Process in ZnO Nanostructure Cold Emission Cathode: *Qi Zhang*<sup>1</sup>; Junjie Qi<sup>1</sup>; Yue Zhang<sup>1</sup>; Qinliang Liao<sup>1</sup>; Xin Li<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing

With recent research, the author intends to outline the framework of the field emission of ZnO nanostructures. Many groups' reports ignored the thermionic emission process in the low electric field. A recently published field emission cathode parameter extraction method [X. He et al., J. Appl. Phys. 102, 056107(2007)] provided unambiguous and reliable cathode. The method utilized Richardson-Laue-Dushman law in low electric field and Fowler-Norheim equation in high electric field to solve a one-dimensional model including both thermionic and field emission. In this letter, the model gave a much better agreement with the experimental data of ZnO cathode under the applied field and acquired a revised surface field enhancement factors and work function of ZnO cathode in the electron emission process. Finite temperature thermal contributions to the current emission were investigated in theory by using the revised work function, revealing that finite temperature thermal contributions can be of great significance in the operation of ZnO field emission.

#### D30 Investigation on the Plasma-Induced Electron Emission Properties of ZnO Nanorod and Carbon Nanotube Arrays: *Qingliang Liao*<sup>1</sup>; Yue Zhang<sup>1</sup>; Yunhua Huang<sup>1</sup>; Junjie Qi<sup>1</sup>; Zheng Zhang<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing

The plasma-induced emission properties of ZnO nanorod and carbon nanotube (CNT) arrays were investigated under the pulse electric field. The formation of plasma on the array surface was found and intense current electron beams were obtained from the two kinds of arrays. The plasma-induced emission properties of the ZnO nanorod and CNT arrays have big differences. Under the same electric field, the CNT arrays have higher emission current than the ZnO nanorod arrays. The distribution of electron beams produced by the two kinds of arrays was diagnosed by plasma radiation and Cerenkov radiation. With the emission currents changing, the electron emissions of the ZnO nanorod arrays always are very uniform; but that of the CNT arrays is lower than that of the CNT arrays. Accordingly, the emission stability of the ZnO nanorod arrays is better than that of the CNT arrays.

#### **D31** Preparation and Characterization of BNC Compounds with Cylindrical and Bamboo Nanotubes: *De Ming Zhu*<sup>1</sup>; Erich Kisi<sup>1</sup>; <sup>1</sup>The University of Newcastle

Doping carbon nanotubes (CNTs) with B and N can result in promising electronic properties. In contrast to CNTs, the properties of these hetero-atomic nanotubes are primarily determined by chemical composition rather than geometry, and are thus relatively easy to control. Nanostructures in the CBN system have indeed attracted much attention recently, because of their great potential in nano-transistors working at high temperature. High-yield cylindrical and bamboo nanotubes from CBN compounds have been prepared by highenergy ball milling graphite and hexagonal boron nitride powder using hardened steel balls and vial, followed by high temperature annealing. Graphite and boron nitride powder was first ball milled at room temperature for 72 hours to produce nano-porous particles with a metastable structure. Upon elevated temperature annealing (1100 to 1300°C), these nano-porous structures were chemically activated to form CBN compounds and generate nanotubes in two different structures. Detailed structures and nanostructures were characterized using xray diffraction analysis, scanning electron microscopy and transmission electron microscopy. The cylindrical tubes have diameters from 40 to 70 nm and lengths

up to 2  $\mu$ m. The bamboo tubes have diameters from 60 to 100 nm and lengths up to 10  $\mu$ m. The formation mechanisms of different nanostructures are discussed.

#### **D32** Shape-Controlled Synthesis and Formation Mechanism of Cobalt Nanopowders by a PVP-Assisted Method: Bingcong Zhang<sup>1</sup>; *Hongying Yu<sup>1</sup>*; Dongbai Sun<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing

Three different kinds of morphologies including spherical, chainlike and wirelike cobalt nanopowders, have been synthesized by chemical reduction of coblat chloride solution with hydrazine hydrate in basic solution. The products were characterized by X-ray diffraction (XRD) and field emission scanning electron microscopy (FESEM). It was revealed that the morphologies of the nanopowders depend on the concentration of  $Co^{2+}$  and the way of adding reducing agent (hydrazine hydrate). These two features affected the nucleation sites and the number of nucleuses. When the concentration of  $Co^{2+}$  was low, nucleuses formed in the soft template, while the reducing of the reducing agent added drop wise, a little number of nucleuses was formed. Based on that, a mechanism of formation, as a basis of gram-scale syntheses, was proposed.

#### D33 Controlled Growth of 1-D Nanomaterials Base on Electro-Deposited Nanocrystalline Films: An Overview: *Chunxu Pan*<sup>1</sup>; Xiaozhu Li<sup>1</sup>; <sup>1</sup>Wuhan University

This paper introduces a novel process for growing one-dimensional nanomaterials. That is, firstly, preparing a pure metal nanocrystalline film by using a pulse electro-deposition technique, then, using this film as a catalyst for synthesizing variant one-dimensional carbon nanomaterials, such as carbon nanotubes (CNTs) and carbon nanofibers (CNFs), and one-dimensional metal oxide nanoneedles, such as ZnO, CuO and Fe3O4. Comparing with the nanoparticle catalyst, this process exhibits the following advantages: 1) The 1-D nanomaterials grow according to the "base growth" model and there is no catalyst at the tip of the 1-D nanomaterials, due to close combination between the nanocrystalline film and the substrate. Therefore, post purification is not required. 2) It provides a possibility for diameter control of the 1-D nanomaterials through grain size control of the 2-D nanocrystalline film by adjusting the pulse electro-deposition parameters. 3) It is more easily to obtain a large area, uniform, vertical and good shape 1-D nanomaterials on a surface. It is expected to solve the application problems in making micro - nano devices.

#### D34 The Electrical Characterization of Single ZnO Nanowries Field-Effect Transistors: *Huifeng Li*<sup>1</sup>; Yunhua Huang<sup>1</sup>; Xiaoqin Yan<sup>1</sup>; Xiujun Xing<sup>1</sup>; Jia Su<sup>1</sup>; Yue Zhang<sup>1</sup>; <sup>1</sup>University of Science and Technology Beijing

Using nanomanipulator and scanning electron microscopy (SEM) system with a tungsten tip, mechanical and electronical property of a single ZnO nanobelt/nanowire has been investigated. It demonstrated that the nanobelt was of sizable toughness due to its perfect monocrystalline micro-/nanostructure. The experimental results show that ZnO nanowire resistivity was about  $1.1 \times 10$  2 O•cm with ohmic contact, and  $1.3 \times 10$  3 O•cm with Schottky contacts. The local change of electron density induced by chottky contacts or ohmic contact with tip and semiconductor/metal materials significantly affects the current transport through the nanowire. Single ZnO nanowires are configured as field effect transistors (FET) and their electrical properties are characterized, too. The electrical measurements using FET based on individual ZnO nanowire show a pronounced n-type gate modulation with an electron concentration of ~10 18 cm<sup>-3</sup> and an electron mobility of ~5.1 cm<sup>2</sup> / V s at a bias voltage of 1 V.

#### **D35** Mechanical Properties of Electrodeposited Ni-SiO<sub>2</sub> Nanocomposite: *Hiroyuki Miyamoto*<sup>1</sup>; Koshiro Ueda<sup>1</sup>; Toshiyuki Uenoya<sup>1</sup>; <sup>1</sup>Doshisha University

Nanocomposite materials consisting of a nanocrystalline Ni matrix (grain size 50-60 nm) and nano-size Al<sub>2</sub>O<sub>3</sub> particulates up to 1.5 mass.% have been synthesized by pulse electrodeposition. Commercial Al<sub>2</sub>O<sub>3</sub> particulates having average particle size of 30 nm were dissolved in a liquid electrolyte using supersonic homogenizer before and during electrodeposition. It was found by TEM that several Al.O. particles were aggregated inside grains and formed larger particles having almost grain diameter. Hardness of Ni-Al<sub>2</sub>O<sub>3</sub> nanocomposite became about 2 times that of nanocrystalline pure Ni. However, tensile strength of Ni-Al2O3Al<sub>2</sub>O, nanocomposite decreased to about 1/2 times that of nanocrystalline Ni. Fracture surface exhibited a typical dimpled surface with dispersed Al<sub>2</sub>O<sub>2</sub> particles on it. EDS analysis revealed that concentration of Al<sub>2</sub>O<sub>2</sub> on the fracture surface was 3 times higher than that of the bulk concentration. suggesting that the aggregated Al<sub>2</sub>O<sub>3</sub> particles caused initiation of cracks and the reduction of tensile stress. Thermal stability was improved; that is, grain growth was suppressed in nanocomposite whereas a certain degree of grain growth was observed in nanocrystaline pure Ni at 573 K. It is considered that higher hardness of nanocomposite is caused by particle strengthening by Al<sub>2</sub>O<sub>3</sub> rather than by composite strengthening.



D36 The Experimental Study of the Electron Emission with SiC Nanowires Cathode Used in High Power Microwave: Chen Zhongdao<sup>1</sup>; <sup>1</sup>National University of Defense Technology

The high power microwave source cathode materials is the key component of the technology of the high power microwave. And the research on the cathode materials is an important advanced project nowadays. In this paper, the silion carbide(SiC) nanowires have been prepared by pyrolysis of a polymer precursor with ferrocene as the catalyst by a CVD route, and the experimental study of the electron emission with the SiC nanowires cathode samples was carried out. The results show that the SiC nanowires cathode has higher electron emission current density, better electron beam quality and longer service life compared with the other cathode uesed in high power microwave source. So SiC nanowires is a valuable and potential material to be extensive used as the high power microwave source cathode.

#### Poster Session: Symposium E: Solidification, Deformation and Related Processing

Tuesday PM	Room: Hall 2
August 3, 2010	Location: Cairns Convention Centre

E1 Effect of Polygonal Rotor Process on Solidification Structure of Lead-Free Bismuth Bronze: *Takahisa Kose*<sup>1</sup>; Yasuhiro Uetani<sup>2</sup>; Katsuyuki Nakajima<sup>3</sup>; Kenji Matsuda<sup>1</sup>; Susumu Ikeno<sup>1</sup>; Katsumi Watanabe<sup>1</sup>; <sup>1</sup>University of Toyama; <sup>2</sup>Toyama Prefectural University; <sup>3</sup>Joetsu Bronz1 Corporation

Semi-solid slurry of lead-free bismuth bronze with fine solid granules for rheocasting was tried to make by the use of an original new melt treatment, in which the revolution of a regular polygonal rotor gives strong shear flow to the melt flowing down along/upon the circular arc plane of chilled block for generation of solidification nucleus, and at the same time, the melt containing obvious nuclei is ejected from the gap between a rotor and a chilled block. The optimum manufacturing conditions were obtained by investigating the effects of rotational speed, gap spacing and difference of rotor and chilled block material on the structure of a solidified small ingot. When the shear flow due to rotations larger than 50rpm were added to the melt just above liquidus temperature by using SUS-304 rotor with a diameter of 150mm and chilled block, the ingot structures were changed from the dendrite at 0rpm to the rosette at 50rpm and the granular with mean size of about  $50\mu$ m at 100 to 250 rpm. When the gap increased to 11mm, it became a granular structure of the rosette. The change wasn't seen though the material of the rotor and the block was changed into copper.

#### **E2** The Correlation between the Liquid Structure and the Solidification Microstructure of Sn-Cu Lead-Free Solders: *Xuemin Pan*<sup>1</sup>; <sup>1</sup>Dalian University of Technology

The liquid structure of two lead-free solder Molten alloys, Sn-0.5Cu and Sn-1.8Cu (wt.%, mass fraction), have been investigated using X-ray diffraction method. The main peak for liquid structure of Sn-0.5Cu is similar to that of pure Sn. A pre-peak has been found in the low Q part on the structure factor S(Q) of Sn-1.8Cu tested under 320°C and the pre-peak decreases its intensity with increasing temperature, but it disappeared finally when the testing temperature reached 350°C. The microstructure of the solder matrixes as well as interfacial reaction between liquid solders and Cu substrates was also studied. The structural unit size corresponding to the pre-peak almost equals to magnitude of crystal planar distance of Cu6Sn5 phase. The appearance of a pre-peak maybe due to existence of clusters with Cu6Sn5-phase-like structure in melt. Quantity and size of clusters increases with decreasing temperature but their structural unit size remains constant. Cu6Sn5 phases develop from incorporating and growing of the clusters during solidification, thus result in the correlation between liquid structure.

### **E3** Visualization of the Thixotropic Fluid Flow: Sang Soo Shin<sup>1</sup>; Eok Soo Kim<sup>1</sup>; Kyung Mook Lim<sup>1</sup>; Jae Chul Lee<sup>2</sup>; <sup>1</sup>KITECH; <sup>2</sup>Korea University

The objective of the study is to study the difference in the flow behaviors between the thixotropic fluid and the Newtonian fluid. The water(Newtonian fluid) and the paint(thixotropic fluid) were selected as the model fluids for representing the molten metal and the semi-solid slurry, respectively. The flow characteristics of these two fluids were analyzed via the experimental and computational techniques. The effect of various rheological parameters on the thixotropic behaviors of the semi-solid metal (A357 Al alloy) and the paint were studied using a Searl type high temperature viscometer. Thixotropy of the slurry was measured as a function of solid fraction, shear rate, and time at rest. Also, two different fluids, i.e. thixotropic fluid and Newtonian fluid, were chosen as the model fluids for monitoring the differences in flow patterns during die filling. When compared the analytical results with what were observed from the experiments, the analytical results based on the k- $\varepsilon$  turbulence model agreed well with the experimental results.

E4 The Effect of Cu on the Microstructure and the Elevated Temperature Properties of Ferritic Heat Resistant Cast Iron: *Kyeong-Hwan Choe*<sup>1</sup>; Sang-Mok Lee<sup>1</sup>; Myung-Ho Kim<sup>2</sup>; Kyong-Whoan Lee<sup>2</sup>; <sup>1</sup>Korea Institute of Industrial Technology; <sup>2</sup>Inha University

Ferritic heat resistant cast iron contains 4~6% silicon and 0.4~2% molybdenum and has good oxidation resistance, structural stability and high temperature strength, however, due to high Si contents, it has poor castability and machinability. Impact properties of ferritic ductile iron are influenced by nodule count and ferrite grain size. The solubility of Cu in a-ferrite is very low, so precipitated Cu may be nucleation site of ferrite during eutectoid transformation. However, smaller grain may deteriorate high temperature strength. In this study, we carried out systematic experiments to understand about the behavior of Cu on the microstructure and elevated temperature properties of ferritic heat resistant cast iron. 3.0wt%C-4.8wt%Si-0.5wt%Mo was selected for base alloy and different amounts of Cu up to 2.0wt% were added. Metallurgical assessments were conducted by combined analysis of elevated temperature properties measurements such as transformation temperature, oxidation properties and strength and relevant microstructural observations.

E5 Effect of Rare-Earth Elements on the Ignition-Proof Behavior of Industrial Pure Magnesium: *Weimin Zhao*<sup>1</sup>; Zhongfang Shi<sup>1</sup>; Zhifeng Wang<sup>1</sup>; Yongyan Li<sup>1</sup>; Jian Ding<sup>1</sup>; Bo-Young Hur<sup>2</sup>; Rui Zhao<sup>2</sup>; <sup>1</sup>Hebei University of Technology; <sup>2</sup>Gyeongsang National University

This paper deals with the effect on flame retardancy of industrial pure magnesium with different kinds of Rare earth intermediate alloys(Mg-Y, Mg-Dy). The result shows that the 0.5%Y can increase the ignition point of pure magnesium about 30°C, however, the ignition point of pure magnesium can be improved about 50°C by the co-activition of 0.5%Y and 5%Dy. The result of SEM indicates the oxide film of Mg0.5Y5Dy is more tenacious than the oxide film of Mg0.5Y. The EDS analysis of the oxide of Mg-0.5Y-5Dy indicates that the oxide film of Mg0.5Y-5Dy is made of Mg0,  $Y_2O_3$  and  $Dy_2O_3$ . Among the three oxides Dy<sub>2</sub>O<sub>3</sub> occupies the most amount.

E6 Restoration Process of Face-Centered-Cubic Metals Subjected into Kinetic Spraying: *Kicheol Kang*<sup>1</sup>; Gyuyeol Bae<sup>1</sup>; Wanghyun Young<sup>1</sup>; Changhee Lee<sup>1</sup>; <sup>1</sup>Hanyang University

Kinetic spraying process (or cold gas dynamic spraying process) utilizes the kinetic energy of in-flight particles accelerated into supersonic velocity by the process gas of high pressure for the deposition. As different face-centered-cubic (FCC) metals, such as aluminum, nickel, and copper, are deposited in the kinetic spraying process, they undergo the severe plastic deformation and restoration process. Due to the difference in stacking fault energy (SFE) of FCC metals, different deformation modes, e.g., deformation twinning and dislocation slip, are operated while the FCC metals impact. The restoration behavior, such as static recovery and recrystallization, of FCC metals is also highly influenced by the SFE during kinetic spraying. In this study, deformation behavior, and microstructure of kinetic sprayed FCC metal coatings were analyzed. The restoration process of FCC metals were investigated considering metallurgical factors, e.g., SFE, and dislocation mobility of FCC metals.

E7 Effect of Various Processing Route on Microstructure and Mechanical Properties of Spray-Deposited Al-8.6Zn-2.6Mg-2.2Cu Alloy: *Feng Wang*<sup>1</sup>; Baiqing Xiong<sup>1</sup>; Yongan Zhang<sup>1</sup>; Baohong Zhu<sup>1</sup>; Hongwei Liu<sup>1</sup>; Zhihui Li<sup>1</sup>; Xiwu Li<sup>1</sup>; <sup>1</sup>General research institute for nonferrous metals

Abstract: Spray deposition is a novel process which is used to manufacture rapidly solidified bulk and near-net-shape preforms. In this paper, Al-8.6Zn-2.6Mg-2.2Cu alloy was synthesized by the spray atomization and deposition technique. The microstructural development during subsequent hot extrusion, hot rolling and canned forging was investigated by means of scanning electron microscope(SEM), transmission electron microscope(TEM) and X-ray diffraction. The results indicate that the microstructure of the alloy mainly is composed of the Al matrix, the Mg(ZnCu)2 compounds with various shape. The fragmentation of the Mg(ZnCu)2 phases in the alloy has been regarded as one of the main feature during extrusion, rolling and forging. Under T6 temper condition, the hot-extruded Al-Zn-Mg-Cu alloy displays superior strength than hot-rolled and canned-forged ones.

**E8** Electromagnetic Continuous Casting Process for Near Net Shaped Aluminum Alloy Billet: *Jong Ho Kim*<sup>1</sup>; Myoung Gyun Kim<sup>1</sup>; Joon-Pyo Park<sup>1</sup>; Gyu-Chang Lee<sup>1</sup>; <sup>1</sup>RIST

A new method and apparatus for the fabrication of high-quality, free-shaped aluminum alloy billets is developed by the combination of continuous casting and



electromagnetic casting/stirring technique. Traditional machine for continuous casting process involves round, square and rectangular billets; therefore it requires additional multistep forging process to fabricate final products. A new process for the designed free-shaped aluminum billets offers some advantages: the process of extrusion and forging is simplified and the cost of plastic working can be greatly reduced. In order to reduce the peculiar problems such as surface crack and internal defect due to inhomogeneous heat transfer through solidified billets, electromagnetic casting and stirring technique were adopted. Developed continuous casting machine for free-shaped billets consists of mold, tundish, cooling system, electromagnetic casting and stirring apparatus. Prior to determine the geometry of mold, the effect of electromagnetic field induced by electromagnetic casting and stirring was studied by numerical simulation and induced current, heating effect, field intensity were characterized by changing the geometrical parameters.

### **E9 Evaluation of Hydrogen Properties for Ternary Nitride Li3BN2**: *Naoki Ito*<sup>1</sup>; Yusuke Michikane<sup>1</sup>; Hiroyuki Takeshita<sup>1</sup>; <sup>1</sup>Kansai University

LiNH2-LiH mixture and LiBH4, which respectively generate hydrogen by the reactions of Li3N+2H2=LiNH2+2LiH and 2LiH+2B+3H2=2LiBH4, are attractive for hydrogen storage materials because they have high gravimetric H storage capacities. However, the dehydrogenation products such as Li3N, LiH and B are not thermodynamically stable enough to promote the decomposition of the hydrides at low temperatures. If the dehydrogenation products are more stable, the absolute values of the enthalpies of the reactions can be decreased, leading to the dehydrogenation under moderate conditions. So, we focus on ternary nitride Li3BN2 for the dehydrogenation product. If Li3BN2 absorbs H2, by the reaction of Li3BN2+2H2 $\rightarrow$ 2LiNH2+LiBH4, 11.9mass% of H2 can be stored and its thermodynamic properties are expected to be improved. For this reason, we have evaluated the hydrogenation properties of ternary nitride Li3BN2.

#### E10 Production Magnesium Alloy Strip with Boss and Rib Section by Melt Drag Process and Experimental Conditions: *Shinichi Nishida*<sup>1</sup>; Kazuki Fukudome<sup>1</sup>; Junpei Kudo<sup>1</sup>; Mitsugu Motomura<sup>1</sup>; <sup>1</sup>Waseda University

This study aims to produce magnesium alloy strip with boss and rib directly from molten metal. Magnesium alloy is the lightest structural material, so it is expected to widely use for small electronic device and etc.. And we studied about melt drag process. Melt drag process is one of single roll strip casting process. We use model melt drag experimental device to produce rapid solidified magnesium strip with boss and rib. Substrate is used on model experimental device instead of roll to easily research shape of substrate. Diameter of boss is 5 mm, 7.5 mm and 10 mm. Height of boss is 6 mm. We revealed on this study that the experimental conditions to get good shape boss and rib, improvement substrate shape for good boss and rib, microstructure and etc.

# E11 Researches Regarding the Influence of the Physical-Chemical Factors of Elaboration, Casting and Solidification on the Steel Ingots Structure Homogeneity: *Ilie Butnariu*<sup>1</sup>; Ioana Butnariu<sup>1</sup>; Dana Butnariu<sup>1</sup>; <sup>1</sup>University POLITEHNICA Bucharest

The casting and solidification processes belong in the most cases, nonstationary thermoconductibility processes, namely: cooling solidified crusts, ingot cooling, melting of the micrcooler and of the inoculater in the liquid metal during the directed solidification. The necessity to address the theme lies in the importance of thorough knowledge of phenomena and processes taking place in casting and solidification of steel ingots, continuously improvement of technology, understanding of the complex phenomena of mass transfer and energy and the ways of action for contiounsly improvement the quality and cast and Forged Products. Research goal was to determine the parameters dynamic variation and transfer believed representatively, depending on the specifical technological parameters in the technological processes of casting and solidification processes, to determine ways of influencing them to obtain steel products of high quality, in terms of reproducibility and efficiency maximum technical –economic.

### **E12 Deformation Characteristics in Alpha Type Brass Worked by Torsion**: *Masahiro Shinsen*<sup>1</sup>; Mitsuaki Furui<sup>1</sup>; Susumu Ikeno<sup>1</sup>; Takekazu Nagae<sup>1</sup>; <sup>1</sup>University of Toyama

In the present work, bar samples of pure copper and Cu-Zn alpha single phase alloys setting 20mm in diameter and 150mm in length were processed by torsion at the rotation speed of 1rpm and twist angles of 60°. Then, microstructure and deformation characteristics of deformed samples were observed. Microstructures of non-deformed samples were isometric, however, deformed samples were streaky. Square elements of  $10 \times 10$ mm mesh in sample surface were developed as parallelograms. The trend angle between stretchable direction in microstructures and torsion axis increased more twist angles. It coincided with between the angle and deformed direction of microstructures. Shear strain was calculated by theoretical formula from twist angles. The value corresponded rough with measurements from the mesh variation. Pure copper and Cu-Zn alpha phase alloys were similar in deformation microstructure and broke near the grip, but the fracture twist angle in copper was larger than Cu-Zn alloys and the trend fracture angle decreased with the increase of concentration of Zn. This trend was different from fracture elongation obtained in the tensile test.

#### E13 Formability of Pure Titanium Long-Cup by Multi-Stage Deep Drawing: Yasunori Harada<sup>1</sup>; *Toshiyuki Araki*<sup>1</sup>; Takahiro Tsuda<sup>2</sup>; <sup>1</sup>University of Hyogo; <sup>2</sup>Nikkoh Metal

The pure titanium long-cups were formed by preventing seizing in cold multistage deep drawing processes by the use of oxide coating. For the prevention, pure titanium sheets are treated by heating oxide coating. The coating is very effective in preventing the occurrence of seizure and galling. The fresh and clean titanium is not in direct contact with the die during the forming due to the existence of the oxide layer. In multi-stage deep drawing, two kinds of pure titanium sheets, the JIS grades 1 and 2, with different oxidant contents were employed. Various cups were drawn by exchanging the punch and ringed die. The die was flat in the first stage, and was taper without a blankholder in the subsequent stages. The effects of the coating conditions on the occurrence of seizure in multi-stage deep drawing were examined. It was found that the coating titanium sheet has sufficient ability in preventing the seizure in multi-stage deep drawing processes. The pure titanium long-cups were successfully formed by a multi-stage deep drawing operation in cold.

## **E14 Tantalum Texture Evolution via Laser Heat**: *Eu Sun Yu*<sup>1</sup>; Tae Jun Ko<sup>1</sup>; Xiaodong Mao<sup>1</sup>; Hyun Chul Roh<sup>1</sup>; Kyu Hwan Oh<sup>1</sup>; <sup>1</sup>Seoul National University

The texture on a wide variety of sputtering target have been extensively studied since the deposition state of thin film can be governed with surface texture arrangement of sputtering target. Among various sputtering targets, Tantalum sputtering target has been researched due to their remarkable versatility, such as chemical processing plants, diffusion barrier layer with nitride, aerospace and military industries. Tantalum sputtering target have been fabricated through melting-deformation, sintering process, etc. Very recently, Si/B doped Tantalum target is being studied to make the tantalum nitride thin film as an active diffusion barrier. In this presentation, we presented Ta texture evolution via heat treatment, which lead to modified texture. We treat cold rolled Ta sputtering target using laser irradiation, which lead to novel texture evolution. After laser treatment, we found oriented texture paralleled to laser irradiation direction from initial (100) Ta texture dominantly. Furthermore, we also observed enlarged and uniformed Ta grains rather than initial cold rolled texture.

### E15 Cold Joining of Dissimilar Metal Sheets by Shot Peening: Yasunori Harada<sup>1</sup>; <sup>1</sup>University of Hyogo

The cold joining of dissimilar metal sheets using a shot peening process was investigated. In shot peening the substrate undergoes large plastic deformation near the surface due to the hit with many shots. Consequently, plastic flow areas formed by cold working may form the surface layer. The dissimilar sheets with the concavo-convex edge are connected, and then the contact area is shot-peened. In this joining, the convex edges of the sheet are stacked on the other sheet. Namely, in the joining area, the two sheets are on top of each other. This method is similar to joining by caulking. In this experiment, the shot peening treatment was performed by using an air-type peening machine. The micro-shots used were made of high carbon cast steel and cemented carbide. Air pressure was in the range of 0.6 - 0.8 MPa and peenig time was in the range of 30 - 150 s. The sheets were commercial low-carbon steel, stainless steel, and pure aluminum. The influences of processing conditions on the joinability were mainly examined. The joint strength increased with the kinetic energy of shots. It was found that the present method was effective for cold joining of dissimilar metal sheets.

## E16 Cyclic Softening of Cu-Ni-Si Alloy Single Crystals under Low-Cycle Fatigue: *Toshiyuki Fujii*<sup>1</sup>; Hiroshi Kamio<sup>1</sup>; Yoshifumi Sugisawa<sup>1</sup>; Susumu Onaka<sup>1</sup>; Masaharu Kato<sup>1</sup>; <sup>1</sup>Tokyo Institute of Technology

Cu-2.2 wt% Ni-0.5 wt% Si alloy single crystals were grown by the Bridgman method and aged at 723 K for 10 h to 72 h to form Ni<sub>2</sub>Si precipitates. Fully reversed tension-compression fatigue tests were conducted on the aged single crystals with a single slip orientation under constant plastic-strain amplitudes at room temperature. After initial cyclic hardening, cyclic softening occurred at constant strain amplitudes between 2.5 x  $10^4$  and 2.5 x  $10^2$ . Based on the peak stress amplitude in each cyclic hardening/softening curve, a pseudo cyclic stress-strain curve (CSSC) was obtained. The CSSC was found to exhibit a plateau region with the stress level of about 167 MPa. Transmission electron microscopic observation revealed the formation of persistent slip bands (PSBs) in the plateau regime. It was found that the Ni<sub>2</sub>Si particles were intensively sheared by glide dislocations and were eventually dissolved into the Cu matrix. The cyclic softening can be understood as a strain localization phenomenon related to dissolution of the Ni<sub>2</sub>Si particles during cyclic deformation.

E17 Influence of Roll Speed Schedule on Transverse Wall Thickness Evenness of Shell Elongated by Mandrel Mill: *Shengzhi Li*<sup>1</sup>; Zhichao Zhang<sup>1</sup>; <sup>1</sup>Anhui University of Technology

The steel tube continuous rolling process with 8-stand full-floating mandrel is simulated with the aid of commercial FE code MSC.SuperForm, and the relationship is analyzed between the distribution characteristics of the transverse wall-thickness and the different Speed Schedule. The result shows that the shell tube's transverse wall-thickness evenness whose influence is remarkable can be enhanced by changing the speed schedule of the rollers. Furthermore, by adopting meliorated method to measure the wall-thickness of the shell, the error is reduced while data reliability is increased. When using the existing equipments, it is an effective way to improve the transverse wall-thickness accuracy of the shell tube by adjusting the speed schedule. Compared with the present roller speed schedule, the transverse wall-thickness accuracy can be increased by 10% for rolling of shell with 152.5mm in OD and 6mm in wall thickness under the 3# speed schedule put forward in this paper.

**E18 Degradation Mechanism of Tungsten Electrode for Fusing Joining:** *Sho Ishii*<sup>1</sup>; Nobuhide Itoh<sup>2</sup>; Goroh Itoh<sup>2</sup>; Shingo Mukae<sup>3</sup>; <sup>1</sup>Undergraduate Student, Department of Mechanical Engineering, Ibaraki University, Japan; <sup>2</sup>Department of Mechanical Engineering, Ibaraki University, Japan; <sup>3</sup>Nippon Tungsten Co., Ltd.

In these years, connecting wires in home electric appliances are becoming more complicated and automobile vehicles are becoming more highly electrically equipped. With these trends, fusing has come to be applied more frequently as a joining method between electrical parts and wires, where direct joining of conducting materials is quickly performed through fusing the surrounding insulating polymer coatings. For fusing, spot welding machines are generally used with a pair of tungsten or molybdenum electrodes. Since the work materials are usually highly electrically conductive such as copper and aluminum, resulting in extremely large electric current, degradation of the electrodes is prone to occur. In this study, tungsten electrodes with two types of microstructures (recrystallized equi-axed and unrecrystallized fiber) were subjected to repeated fusing joining tests where the work was a tough pitch copper sheet. The surface appearance and microstructure of the electrodes after the test were observed with an SEM, focusing on the relationship between the microstructure and the cracks that were introduced during the joining. Finally the effect of microstructure on the degradation behavior was summarized.

#### E19 Development of High Strength and High Conductivity Cu-Ag-Zr Alloy: Hoon Cho<sup>1</sup>; <sup>1</sup>Korea Institute of Industrial Technology

The development of high-performance instruments and patient-friendly system for medical equipment has been remarkable in recent years. However, there is an urgent demand to reduce cable diameter given the needs for easy operation and frequent insertion into a patient's body. The development trend for diagnostics is of reducing the diameter of coaxial signal cables that is offering superior electrical and mechanical properties, such as 75% IACS(International Annealed Copper Standard, electrical conductivity) and 700 ~ 800 MPa in tensile strength has to be developed. The Cu-Ag alloys are very well-known that the outstanding combinations of high strength and high conductivity materials can be achieved. However, although the Cu-Ag binary alloy has shown the promising strength and conductivity, the actual solubility of Ag in Cu matrix is normally higher than its equilibrium solubility. This non-equilibrium high solubility of Ag can deteriorate the mechanical and electrical properties. Therefore, in the present study, the ternary Cu-Ag-X alloy which X can reduce the solubility of Ag to equilibrium level has been considered in order to increase the conductivity of the alloy.

#### E20 Simultaneous Enhancement of Electrical Conductivities and Mechanical Properties in Cu-Ti Alloy by Hydrogenation Process: *Atsunori Kamegawa*<sup>1</sup>; Toru Iwaki<sup>1</sup>; Masuo Okada<sup>1</sup>; <sup>1</sup>Tohoku University

Effects of hydrogenation process of the microstructure, electrical conductivity and mechanical properties for the Cu-(1~5) mass%Ti alloys were investigated. During hydrogenation process at 350°C, 7.5 MPa for 48 h, the disproportionation reaction occurred with forming of Ti hydrides in the alloy. With decomposition of Ti hydrides by the subsequent desorption process, Ti resolved into Cu matrix. In consequence, the original solid solution phase of the alloys formed. This means that the HDDR(Hydrogenation-Disproportionation-Desorption-Recombination) phenomena could take place in the Cu-Ti alloys. The grain size of the alloy is found to be about 20–50 nm after HDDR treatments such as hydrogenation treatment at 350°C under hydrogen pressure of 7.5 MPa for 48 h, followed by the hydrogen desorption treatment at 530°C for 4 h in vacuum. It is found that remarkable simultaneous improvements of mechanical strength of 1094 MPa and electrical properties of 21%IACS are obtained in the hydrogenated Cu-3mass%Ti alloy. On the other hand, nominal stress of this alloy after HDDR process decreased to 607MPa.



#### Poster Session: Symposium F: Modelling and Simulation of Microstructures and Processes

Tuesday PMRoom: Hall 2August 3, 2010Location: Cairns Convention Centre

**F1 A Monte Carlo Simulation of Melting in Prototype Crystal**: *Kazufumi Sato*<sup>1</sup>; Satoshi Takizawa<sup>1</sup>; Tetsuo Mohri<sup>1</sup>; <sup>1</sup>Hokkaido University

We investigate the melting transition of the solids interacting with a simple pairwise potential using conventional and Wang-Landau Monte Carlo simulation. In the simulations, the atomic displacement is discretized for describing the atomic vibration and each atom is confined within its Voronoi polyhedron. The melting point can be uniquely determined by Wang-Landau approach while the temperature hysteresis inevitably appears in the conventional method. The obtained results show the typical feature of first-order transition which is the discontinuous change in the internal energy and in the slope of free energy. The transition temperature is in good agreement with previous simulational studies. We discuss the relation between the limit of superheated state and intrinsic instability of model system through the comparison with two results.

#### F2 First-Principles Calculation of Water Molecules with Adsorbed Ions on the Fe(001) Surface: *Norio Nunomura*<sup>1</sup>; Satoshi Sunada<sup>1</sup>; <sup>1</sup>University of Toyama

The behavior of water molecules with sulfate on the Fe (100) surface has been investigated using first-principles method based on density-functional theory (DFT) with numerical atomic orbital as a basis set for the description of valence electrons and nonlocal pseudopotentials for the atomic core. The Kohn-Sham equations are solved self-consistently within the generalized gradient approximation (GGA) for the exchange-correlation potential. We report results for the adsorption structure and bonding nature as caused by the adsorptioninduced variations in the electron density and in the projected density of states. We have found that the structure of absorbed sulfate is stabilized by hydration. The mechanism of proton conduction through hydrogen bonding has been obtained from our calculations.

#### **F3 First-Principles Investigation of the Electronic Structure and Magnetic Properties for Co-Doped Fe<sub>3</sub>O<sub>4</sub>**: *Yuhua Hou*<sup>1</sup>; Yujun Zhao<sup>1</sup>; Dechang Zeng<sup>1</sup>; Zhongwu Liu<sup>1</sup>; Lishi Wen<sup>1</sup>; <sup>1</sup>South China University of Technology

The electronic structure and magnetic properties of spinel structural of  $(Co_x Fe_{1-x})A(Co_{1-x}Fe_{1+x})BO_4$  (x is defined as the fraction of A sites occupied by divalent cations Co2+) scenario are investigated theoretically from first-principles, using Generalized Gradient Approximation (GGA) method for systems with strong Coulomb correlations is described which gives a correct description of the electronic structure. The GGA+U method gives a qualitative improvement compared with the GGA not only for excited-state properties such as energy gaps but also for ground-state properties such as magnetic moments and interatomic exchange parameters, We concentrate on establishing the nominal valence of the transition metal elements and the ground state structure, based on the study of variation of the cation distribution (x=0.0,0.25,0.5,0.75,1.0) over the A and B sites, The results show that Site-preference calculation on bulk systems pointed out that Co<sup>2+</sup> ions strongly prefer the octahedral B sites, and the electronic structure and magnetic properties of Cobalt ferrites highly depend on cation distribution even though the chemical composition of the compound does not change, which are in good agreement with available experimental data and others theoretical results.

F4 Analysis of Leaning Problem of Gold Wire in Wire Bonding Process: Seoung Bum Son<sup>1</sup>; Suk Hoon Kang<sup>1</sup>; Do Hyun Kim<sup>1</sup>; Jung Han Kim<sup>1</sup>; Jong Soo Cho<sup>2</sup>; Jeong Tak Moon<sup>2</sup>; Kyu Hwan Oh<sup>1</sup>; <sup>1</sup>Seoul National University; <sup>2</sup>MK Electron

Gold wire mainly performs interconnecting between IC chips for electrical conduction. After gold wires are bonded, it is found that a few wires are leaned to others. Unexpected connection between wires causes a short-circuit problem, and this leads to IC chips failure. In this research, FEM and EBSD methods are used to analyze how wire leaning problem is occurred. By observing SEM of drawing dies, it is found that dies have unsymmetrical shapes near wire reduction area which can lead to asymmetrical deformation of wire. By EBSD analysis, it is turned out RD//<100> texture is off center in the wire which is supposed to be exact in the center of the wire in the case of general cold drawn wire. Considering RD//<111> texture is typical drawing texture and RD//<100> texture is undeformed, it is thought that out of centered RD//<100> texture means asymmetric deformation of wire in the drawing process. After that, elastic modulus of each orientation is calculated and applied to FEM analysis. Through



FEM, under applying tensile stress, we changed the location of RD//<100> in the gold wire from center to surface, and studied how location of <100> orientation has effect on leaned shapes of bonded wire.

F5 Effect of Inclusion Shape and Material Properties on Drawing Stress of Ultra Fine Gold Wire: *Young-Kwang Lee*<sup>1</sup>; Seoung-Bum Son<sup>1</sup>; Sang-Yeop Kim<sup>2</sup>; Tae-Jin Kwon<sup>2</sup>; Jong Soo Cho<sup>2</sup>; Jeong-Tak Moon<sup>2</sup>; Hee-Suk Chung<sup>1</sup>; K.H. Oh<sup>1</sup>; <sup>1</sup>Seoul National University; <sup>2</sup>MK Electron

Effect of inclusion type on failure of ultra-fine wire has been analysed by FEM simulation and exeprimental test. Large portion of inclusion come from particle of guide, die and dust from air during drawing process, inclusion has been specified into two type with material property - hard type as  $Al_2O_3$  and  $SiO_2$  and soft type as SUS304 steel. FEM simulation has been performed with change of inclusion size, material property of hard/soft type inclusion, of inclusion shape. In drawing process of FEM model, soft type of inclusion shows deformation along drawing direction, on the contrary, initial shape of hard type inclusion remained after drawing process of gold wire. On experimental result, cross-section of failed gold wire with hard inclusion particle shows rough surface and cross-section with soft inclusion particle shows smooth surface. Inclusion size and thickness/height ratio affects differnetly on drawing stress with soft/hard type of inclusion.

#### F6 Determination of Interphase Thickness and Mechanical Properties of Effective Nanofillers in Polymer Nanocomposites by Molecular Dynamic Simulation: *Wen Xu*<sup>1</sup>; Qinghua Zeng<sup>1</sup>; Aibing Yu<sup>1</sup>; Donald Paul<sup>2</sup>; <sup>1</sup>The University of New South Wales; <sup>2</sup>The University of Texas at Austin

The properties of interphase in polymer composites are often different from those of bulk polymer matrix, which may include chemical, physical, microstructural, and mechanical properties. The nature of interphase is critical to the overall properties and performance of polymer materials, in particular in nanofiller reinforced composites. Experimental efforts have been made to determine the effective interphase thickness and its properties, for example, by nanoindentation and nanoscratch techniques. Yet, it is very difficult to quantify the interphase and its properties because of its nanoscale nature and the unclear boundary. In this regard, computer simulation, e.g., molecular dynamics, provides an effective tool to characterize such interphase and the properties. In this work, molecular dynamics simulations are applied to quantify the interphase thickness in clay-based polymer nanocomposites. Then, the properties of the so-called effective nanofiller (i.e., the physical size of nanofiller plus the size of interphase) will be determined by a series of simulations.

## F7 Effects of Hub Contact Shape on Contact Pressure and Fatigue Life in a Press-Fitted Shaft: *Dong-Hyong Lee*<sup>1</sup>; Seok-Jin Kwon<sup>1</sup>; Won-Hee You<sup>1</sup>; <sup>1</sup>Korea Railroad Research Institute

In the shrink or press-fitted shafts such as railway axles, rotor of a steam turbine or coupling, fretting damage takes place at the contact edge by cyclic stress and local movement between shaft and boss. And a high stress concentration takes place in the close of contact edge due to relative slip between shaft and boss in a press-fitted shaft. The object of this paper is to propose a numerical finite element procedure and to quantify the effects induced by varying the shape of boss on contact pressure, contact stress and microslip. Numerical asymmetricaxisymmetric finite element models were developed in order to predict the contact stress state of press-fitted shaft induced by using three types of boss shape. The results showed that the best performance, in terms of stress and pressure distribution state on the contact surface of shaft, can be obtained by using a proper taper values for the boss element. Boss taper design could be a useful tool, able to increase the load capabilities of press fits in terms of wear and fatigue behavior.

### **F8** Polyanion Transport at Nanoscale Heterostructure Interfaces: *Yongkai* Zhou<sup>1</sup>; Stefan Adams<sup>1</sup>; <sup>1</sup>National University of Singapore

Recently, we demonstrated computationally and experimentally that ionic conduction in  $Sc_2(WO_4)_3$  can be traced back to mobile  $WO_4^{2^2}$ . Here, we investigate how the ionic mobility in such polyanionic conductors is enhanced by defects in the bulk material or via heterogeneous doping at interfaces. For a variety of anion conductor heterostructures, conductivity is enhanced by (i) the redistribution of mobile species across the interface (e.g.  $CaF_2 / BaF_2$  or (ii) expanded conduction channels due to structural misfit. Here, defect formation energies in bulk  $Sc_2(WO_4)_3$  and in the vicinity of the interface are compared and the diffusion of polyanions at nanoscale heterostructure interfaces is studied by Molecular Dynamics simulations. A 3D periodic 2784 atom heterostructure was built attaching cleavage surfaces of the  $WO_4^{2^2}$  conductor  $Sc_2(WO_4)_3$  (010) to  $CaWO_4$  (101). An increase of the  $WO_4^{2^2}$  diffusion coefficient by several orders of magnitude is observed in the highly disordered interface region and the mobility remains significantly enhanced throughout the nanostructured heterolayer. Experimental studies to verify our simulations are in progress. F9 Phase Field Simulations of Dendritic Growth Using an Extended Cahn-Hilliard Model for Cubic Anisotropy: *Joo-Youl Huh*<sup>1</sup>, Sung-Kyun Park<sup>1</sup>; In-Sung Cho<sup>2</sup>; Ho-Young Hwang<sup>2</sup>; <sup>1</sup>Korea University; <sup>2</sup>KITECH

Phase field models based on a diffuse interface concept are powerful techniques to simulate the structural evolution during materials processing since an explicit tracking of the interface at every time step is not required. Although the conventional phase model has been extensively used to simulate dendritic growth during solidification of pure metals and alloys, it was only applicable to the systems of which interfacial energy anisotropies are relative small. When the anisotropy of interfacial energy is large enough to exhibit a concavity in the polar plot of the reverse of the interfacial energy, missing orientations occur in the equilibrium shape and phase field simulations fail to converge. In this study, we employed two recently developed techniques to simulate dendrite growth of pure metals with high interface energy anisotropy; one by regularizing the gradient energy coefficient and the other by extending the Cahn-Hilliard model to include the terms containing fourth-rank tensors. Two-dimensional simulations were performed for a wide range of cubic anisotropy to compare the two techniques for the growth rate of dendrite and the selection of secondary arm spacing.

#### **F10** Analysis of {10-12} Twin Structure by Molecular Dynamics Method: *Kounosuke Nakamura*<sup>1</sup>; Hiromoto Kitahara<sup>1</sup>; Shinji Ando<sup>1</sup>; <sup>1</sup>Kumamoto University

{10-12} twin is common twin in pure hcp metals. In this study, initiation and development of {10-12} twin in hcp metal was simulated by molecular dynamics method. Two types of model crystals were stretched along the y-axis by applying displacement of 0.01a (a is lattice constant of basal plane) every 2000 MD steps and relaxed atoms by molecular dynamics method. Y-boundary of the model was applied fixed boundary condition and X and Z-boundary were free boundary condition. Lennard-Jones type interatomic potential was employed in this simulation. In the model single crystal, {10-11} pyramidal slip initiated at the crack tip and the slip was stopped at y-boundary atoms. After that, a {10-12} twin was initiated at a front of the slip. With increasing external strain, the twin grew with increasing external strain. In the model bi-crystal, {10-10} prismatic slip occurred at the crack tip in the Crystal 1 and a-dislocation of the slip stopped at a grain boundary between the Crystal 2 at the grain boundary. From the simulation results, 'shuffling' process of twin deformation was estimated.

#### **F11 Role of Sulphur on the Weld Pool Development during A-TIG Welding:** *Yuzhen Zhao*<sup>1</sup>; Haiyan Zhao<sup>1</sup>; <sup>1</sup>Tsinghua University

A 3D mathematical model was carried out to understand the influence of the fluid flow patterns and heat flow on the development of the weld pool with sulphur content during A-TIG welding of Type 304 stainless steel. The rule how soluble sulphur content affects the weld pool at different welding current was studied. It is shown that sulphur affects the weld shape by changing the flow patterns. The weld depth/width ratio increase first sharply and then remain nearly a constant with increasing sulphur content. The depth/width ratio under the low current is greater than that under the high current with the same sulphur content. Sulphur can cause significant changes in the weld shape by varying the sign of the temperature coefficient of the surface tension. The situation with the maximum surface tension moves from the edge to the center gradually with increasing sulphur content. As sulphur content exceeds a critical value, the situation with the maximum surface tension occurs in the center and positive temperature coefficient dominates the flow patterns and result in deep and narrow weld pool. There are no observable changes in the flow patterns and the overall shape of the weld pool with further increasing sulphur content.

# F12 Simulation of Texture Evolution in Equal Channel Angular Pressing of Aluminum Single Crystal: *Cheng Lu*<sup>1</sup>; Guanyu Deng<sup>2</sup>; Nam Huynh<sup>1</sup>; Hongtao Zhu<sup>1</sup>; Xianghua Liu<sup>2</sup>; Kiet Tieu<sup>1</sup>; <sup>1</sup>University of Wollongong; <sup>2</sup>Northeastern University

Equal channel angular pressing (ECAP) has attached much attention during last decade due to its capability of significant grain refinement. Texture in ECAP dominates many aspects of material behavior, such as strength, grain refinement, and plastic anisotropy. However, a deeper theoretical understanding of texture evolution is still lacking. In this paper, a crystal plasticity finite element model has been developed to analyze texture evolution during ECAP of aluminum single crystal for two different intersection angles of ECAP die (90 degree and 120 degree). Textures predicted by the developed model are in good agreement with the experimental observations. The simulation results indicate the inhomogeneous orientation distributions in the samples for both angles. However, the orientation rotation patterns are different. 90 degree die exhibits a higher rotation around the transverse direction than 120 degree die.



F13 The Effect of the Doping Profile in Aluminum Back-Surface-Field on the Electronic Properties of C-Si Solar Cells: *Jicheng Zhou*<sup>1</sup>; Yongming Chen<sup>1</sup>; <sup>1</sup>Central South University

The electronic properties of the solar cells were greatly influenced by the aluminum atomic concentration in Al-BSF region under that the Al-BSF is doped heavily. The effects of thickness and doping profile of heavily-doped Al-BSF, as well as the trap levels of impurities in Al-BSF, on electronic properties of n+pp+ monocrystalline solar cells, were investigated by PC1D software. The results show the electronic properties of the solar cells are hardly affected by the doping profile of Al-BSF, but mainly depend on the Al/B atomic amount in Al-BSF and the back surface doping density. The optimum thickness of Al/B-BSF is about 10 $\mu$ m with the average Al/B atomic concentration of Al-BSF less than ~6.56×10<sup>18</sup> cm<sub>3</sub>, and it declines sharply as the average Al/B atomic concentration of Al-BSF is more than ~1.15×10<sup>19</sup> cm<sub>3</sub>. The oxygen trap level has an obvious impact on the electronic properties in case of the back surface recombination velocity (BSRV) less than ~1×10<sup>5</sup> cm/s or the average Al/B atomic concentration less than ~1×10<sup>18</sup> cm<sub>3</sub>.

## F14 Theoretical Study of Growth Mechanism of Goethite in the Presence of Surfactants: *Jeffrey Yue*<sup>1</sup>; Xuchuan Jiang<sup>1</sup>; Aibing Yu<sup>1</sup>; <sup>1</sup>Materials Sciences & Engineering, UNSW

Goethite (alpha-FeOOH) nanorods could be prepared by a surfactant (CTAB) directed approach in aqueous solution at room temperature. The obtained goethite nanorods have a diameter of ~20 nm and length up to 300 nm. It is observed that the surfactant, cetyltrimethylammonium bromide (CTAB), plays a key role in the growth of goethite nanorods at ambient conditions. Molecular dynamics (MD) method can be used to understand the underlying principles governing particle formation and growth through the analysis of the interaction energies between crystal surfaces and surfactant molecules or other metal nanoclusters (Au, Ag, Pt, and Pd). The results show that the goethite surface could strongly interact with gold and platinum nanoparticles by forming a nanocomposite material without the use of any linking media. This will be useful for understanding the growth mechanism of anisotropic particle growth and their surface coating with heterogeneous materials for desired functional properties.

## **F15 Research on Winding Technology of Thin Helical Tube of Pd-YAlloy**: *Ma Guang*<sup>1</sup>; Zheng Jing<sup>1</sup>; Li Yin'e<sup>1</sup>; Wang Yi<sup>1</sup>; <sup>1</sup>Northwest Institute for Nonferrous Metal Research

The thin helical tube of Pd-Y alloy was mainly used to high purity hydrogen preparation. The physical phenomenon was comprehensively considered during the winding process on the thin helical tube of Pd-Y alloy. Analysis the reason of wrinkling and depression is emerged on the inner of thin helix tube during the winding process on the thin helical tube of Pd-Y alloy. Stress state was analysed on the winding process of the thin helical tube through the elastoplastic finite element method; the stress distribution law was obtained. And then, the stress state was comparative studied on the thin helical tube of Pd-Y alloy by different tension action, the control conditions of tension was presented, it was between sixty-five to seventy-five Newton. Therefore, the numerical simulation study provided theory basis for the process control of the thin helical tube's manufacture. It was no wrinkling and depression by winding simulation test on the thin helical tube of Pd-Y alloy, satisfies requirement of using.

#### F16 Modelling Study of the Influence of Subflux Controller of Turbulence on the Molten Steel Flow in Tundish: *Tomasz Merder*<sup>1</sup>; <sup>1</sup>Silesian University of Technology,

The objective of the study is to diagnose the current condition of the two-strand tundish. The investigated object is a "T"-type tundish. The nominal capacity of the tundish is 7.5 tonne of liquid steel. By the mathematical simulation, fluid flow and heat transfer of molten-steel in a tundish of a billet caster under different conditions (bare tundish and tundish with flow control device) were analyzed. Three variants subflux controller of turbulence configurations in the tundish were tested. Numerical simulations were carried out with the finite-volume commercial code FLUENT using the realizable k- $\varepsilon$  turbulence model. In effect of mathematical calculations liquid steel velocity, temperature, turbulent kinetic energy and characteristic Residence Time Distribution have been obtained. The RTD curve is used to find the different volumes such as plug volume, dead volume and mixed volume inside the tundish. The ratio of mixed to dead volume, which indicate the mixing capability of a tundish, is estimated. The results showed that the tundish with subflux controller of turbulence has an important effect on the flow pattern and temperature distribution.

## **F17** Numerical Simulation on Adiabatic Shearing Behavior of Vanadium Alloy V-5Cr-5Ti Hat-Shaped Specimen: *Yixia Yan*<sup>1</sup>; <sup>1</sup>Institute of structural Mechanics, China Academy of Engineering Physics

In this work, integrated with experiment results observed in the Split Hopkinson Pressure Bar (SHPB), LS-DYNA program is adopted to simulate the loading process of Vanadium alloy V-5Cr-5Ti hat-shaped specimen conducted on SHPB in two dimensions. Based on the Johnson-Cook material constitutive relation and criterion of Johnson-Cook failure, the initiation and propagation process of an adiabatic shear band (ASB) and the corresponding distribution of temperature field in the Vanadium alloy specimen are analyzed. The field of stress, strain and temperature in the tip of an ASB, and the spread speed, the width as well as the type of the ASB are all studied. It is shown that the formation of the ASB is related to the loading velocity and the size of the hat-shaped specimen. And formation of the hat-shaped specimen.

## **F18** Prediction of Durability of Mold in Continuous Casting by Finite Element Method: *Tae Jun Ko*<sup>1</sup>; Hyunchul Roh<sup>1</sup>; EuSun Yu<sup>1</sup>; Xiaodong Mao<sup>1</sup>; Kyu Hwan Oh<sup>1</sup>; <sup>1</sup>Seoul National University

The condition of mold is very important factor which determines the quality of slab and has relation with productivity in continuous casting. So, prediction of durability of mold using by computing simulation, which is suitable for case study and spend low cost, is necessary to prevent failure of mold. The following are the process of crack-generation in brief. On casting, difference of temperature between hot face and cold face is generated. Then, hot face suffers compression and plastic deformation is occurred. That plastic deformation is accumulated as casting cycle processes by. After casting, temperature of hot face goes down and hot face suffers tension because of accumulated plastic deformation. Even as amount of accumulated plastic deformation exceeds elongation of mold, crack is induced which lead failure. In this research, we use finite element method (FEM) to predict durability of mold, and we consider the effect of casting condition to durability of mold.

#### **F19 Optimum Pass Design of Bar Rolling for Producing Bulk Ultrafine-Grained Steel by Numerical Simulation**: *Tadanobu Inoue*<sup>1</sup>; <sup>1</sup>National Institute for Materials Science

The groove design for creating ultrafine-grained low-carbon steel through a caliber rolling process was studied from the viewpoint of a large strain accumulation and cross-sectional shape variation in a bar. A three-dimensional finite element analysis was employed for this purpose. Caliber rolling process of square/foval (flat-like-oval)/square type was proposed as a method to introduce a large strain efficiently in material. The relation among the foval configuration, a strain and cross-sectional shape was examined in the groove rolling. The influence of the equivalent strain distribution by 1st pass (foval rolling) was considered to clarify the strain distribution and a cross-sectional shape by 2nd pass, and then the foval configuration to accumulate a large strain efficiently was shown. The optimum pass schedule to fabricate an ultrafine-grained steel of 13mm square bar from a 24mm square bar was proposed in warm caliber rolling.

### **F20** Residual Stresses in Cold Spray Process Using Finite Element Analysis: *Thanh-Duoc Phan*<sup>1</sup>; Syed Masood<sup>1</sup>; Mahnaz Jahedi<sup>2</sup>; Saden Zahiri<sup>3</sup>; <sup>1</sup>Swinburne University of Technology; <sup>2</sup>CSIRO; <sup>3</sup>CSIRO Manufacturing and Materials Technology

In cold spray process, the simulation of coating deposition and the analysis of the arising residual stresses is vital for an optimisation of process conditions. However, there are not many published literatures on the role of residual stresses in a cold sprayed coating. Additionally, the multi-particles coating deposition behaviours is also not well known, especially when coating of pure titanium powder on steel substrate is considered. This paper presents an investigation on the development of a finite element (FE) model of a cold spray process with defined initial and boundary condition in order to determine the optimum operating conditions to deposit titanium particles on steel substrate. The FE model is used to predict the residual stress developed in the coating by examining a fluid/structure interaction model. The predicted results reveal that Finite Element Method (FEM) can be used to study the residual stress in a cold-sprayed coating as well as to find the optimum operating conditions to deposit to study the residual stress in a cold-sprayed coating as well as to find the optimum operating conditions to deposit optimum opt

## **F21 Rigid-Plastic Finite Element Analysis of Cross Wedge Rolling**: *Dyi-Cheng Chen*<sup>1</sup>; Ci-Syong You<sup>1</sup>; Gua-Nying Lai<sup>1</sup>; Syue-Cheng Ji<sup>1</sup>; <sup>1</sup>National Changhua University of Education, Taiwan

In processes of cross wedge rolling, many factors must be controlled to obtain the required plastic strain and desired tolerance values. The major factors include the on stamping die relative velocity, the forming angle, the broaden angle and section reduce rate. This paper employs the rigid-plastic finite element (FE) DEFORMTM 3D software to investigate the plastic deformation behavior of aluminum alloy (A7075) workpiece as it is rolling for cross wedge rolling. Under various rolling conditions, it analyses the damage factor, the effective strain, the effective stress and the die radius load distribution of the workpiece. Furthermore, using simulative software to analyze its rolling process microstructure changing. The analytical results confirm the suitability of the current finite element software for cross wedge rolling.



F22 Thermal-Structural Finite Element Analysis of Injection Moulding Dies with Optimized Cooling Channels: *Abul Saifullah*<sup>1</sup>; Syed Hasan Masood<sup>1</sup>; Igor Sbarski<sup>1</sup>; <sup>1</sup>Industrial Research Institute Swinburne

One of the most important aspects of mould design in injection moulding is the provision of suitable and adequate cooling arrangements. Proper cooling channel design in the mould is crucial factor because it not only affects cycle time but also the part quality of the injection moulded plastic part. A new cooling channel design with copper tube insert can reduce cycle time and part quality by optimal and uniform heat transfer in the mould. In this research work a comparative FEA thermal-structural analysis has been performed with ANSYS simulation software to understand robustness and longevity of an industrial plastic part mould with these cooling channels in comparison with conventional straight cooling channels. Result shows that by inserting copper tube in the cooling channels mould not only reduces cycle time but also increased life time of mould.

#### Poster Session: Symposium G: Thin Films and Surface Engineering

Tuesday PM	Room: Hall 2
August 3, 2010	Location: Cairns Convention Centre

**G1 Effect of Subsequent-Annealing Temperature on Surface Properties of Plasma Electrolytic Oxidation-Treated Mg Alloy**: *Young Gun Ko*<sup>1</sup>; Seung Namgung<sup>2</sup>; Kang Min Lee<sup>2</sup>; In Jun Hwang<sup>2</sup>; Dong Hyuk Shin<sup>2</sup>; <sup>1</sup>Yeungnam University; <sup>2</sup>Hanyang University

The influence of the subsequent-annealing (SA) temperature on the plasma electrolytic oxidation (PEO)-treated Mg-based alloy was investigated in terms of surface properties associated with hardness and corrosion. For this purpose, a series of the SA treatments were performed on the PEO-treated samples at four different temperatures, i.e., 100, 150, 200, and 250°C for 10 hrs. When compared to the sample without SA, the samples annealed at temperatures higher than 200°C showed a difference in surface morphology due to the volume expansion accompanied by the dehydration reaction where the part of Mg(OH)<sub>2</sub> changed into MgO, working as harder phase. From the results of nano-indentation tests, the applied loads of the samples were seen to increase with increasing SA temperatures. However, the electro-chemical and corrosion properties of the sample annealed at 150°C were higher than those of the samples annealed at three temperatures.

#### G2 Effect of Pre-Deforming on Plasma Nitriding Response of 304 Stainless Steel: Akio Nishimoto<sup>1</sup>; Katsuya Akamatsu<sup>1</sup>; <sup>1</sup>Kansai University

This paper reports a study on the effect of a modified layer caused by predeforming on the low temperature plasma nitriding of AISI 304 austenitic stainless steel. The aim of using the deformed layer is to produce a thicker nitrided layer and to decrease the nitriding temperature due to the much faster diffusion of nitrogen. The pre-deformed sample was prepared by the rolling in 0, 1, 2, 3, and 4% ratios. Plasma nitriding was carried out at 673 and 723 K for 18 ks under 600 Pa pressures in presence of N<sub>2</sub> : H<sub>2</sub> in 50 : 50 ratio. The microhardness, thickness and phase composition of nitrided layers formed on the surface of pre-treated and non-treated samples were investigated using Vickers microhardness tester, optical microscope and X-ray diffraction techniques, respectively. After nitriding, maximum hardness ~1150 HV was achieved on the pre-deformed sample. XRD pattern showed that most dominant phase of the nitrided layer consisted of the expanded austenite (S phase). In addition, the pre-deforming by the rolling had a great influence on the hardness and thickness of the S phase. That is, the hardness and thickness of the S phase increased by applying the pre-deforming.

#### **G3** Aluminizing of TiAl Based Alloy Using Thermal Spray Coating: *Tomohiro* Sasaki<sup>1</sup>; Yagi Takahiro<sup>1</sup>; Takehiko Watanabe<sup>1</sup>; <sup>1</sup>Niigata University TiAl based alloy, including laminar structure of Ti3Al and TiAl, is prospective

heat resistant material for aeroengine and automobile components because of

the high relative creep resistance. However the oxidation resistance of this alloy

is not enough for practical use at higher temperature above 800°C. Therefore

several attempt of addition of alloy elements or surface coating have been made

to improve the oxidation resistance. On the other hand, this alloy has another

problem on the low ductility at room temperature. In order to produce high

precision components, grinding or high-speed milling will have to be performed.

However, the defects such as crack or pealing caused by the laminar structure

easily occur during machining. In addition, these defects lead to decline the

mechanical performance. Thus, a surface coating to improve oxidation resistance

without seriously damaging the mechanical properties is needed. In this study,

aluminizing on the machined surface of TiAl based alloy has been conducted

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by two processes of pure aluminum coating using thermal spray and diffusion treatment. We will report the formation process of coating layer in the diffusion treatment and the effect of aluminizing condition for the surface property.

### **G4** Characteristics of a Polymer Microparticle Irradiated by a Laser: *C. Y. Ho*<sup>1</sup>; M. Y. Wen<sup>2</sup>; <sup>1</sup>Hwa Hsia Institute of Technology; <sup>2</sup>Cheng Shiu University

This paper analytically investigates characteristics of a polymer microparticle irradiated by a laser. Micrometer- and nanometer-sized particles possess characteristic physical and chemical properties different from those of bulk materials. Based on the measurement of radiation pressure exerted on a particle, the absorption analysis of a polymer microparticle with a diameter  $5\mu$ m had been conducted. Assuming the polymer microparticles to be spherical and neglecting net charges on the particle, the classical electromagnetic wave theory is employed to analyze the scattering and absorption of a polymer microparticle for laser. The effects of parameters on the scattering and absorption of a polymer microparticle are discussed in this paper

**G5** Characterization Performance of Laser Melted Commercial Tool Steels: *Miroslaw Bonek*<sup>1</sup>; Leszek Dobrzanski<sup>1</sup>; <sup>1</sup>Silesian University of Technology

Investigations include alloying the hot-work tool steel surface layer with the carbides, using the high power diode laser (HPDL). The structural mechanism was determined of surface layers development, effect was studied of alloying parameters, gas protection method, and thickness of paste layer applied onto the steel surface on structure refinement and influence of these factors on the mechanical properties of surface layer, and especially on its hardness, abrasive wear resistance, and roughness. The fine grained, dendritic structure occurs in the remelted and alloyed zone with the crystallization direction connected with the dynamical heat abstraction from the laser beam influence zone. The remelted zone structure is characterized by the significant martensite dispersion with its lathes length several times shorter than of those developed during the conventional quenching. The fine grained martensite structure is responsible for hardness increase of the alloyed layer. The dependence is presented of microhardness change on the laser beam effect on the treated surface, and especially the hardness increase in the alloved layer. It has the important cognitive significance and gives grounds to the practical employment of these technologies for forming the surfaces of new tools and regeneration of the used ones.

G6 Development of Laser Surface Treatment with Excellent Corrosion Resistance and Conductivity Performance on Magnesium Alloy Products: *Makoto Hino*<sup>1</sup>; Yutaka Mitooka<sup>1</sup>; Koji Murakami<sup>1</sup>; Katsuji Nishimoto<sup>2</sup>; Teruto Kanadani<sup>3</sup>; <sup>1</sup>Industrial Technology Research Institute of Okayama Prefecture; <sup>2</sup>ARRK Okayama Co. Ltd.; <sup>3</sup>Okayama University of Science

Magnesium alloy has been applied to the bodies of electronic equipment due to its excellent characteristics such as high rigidity, thinness, light weight, good heat dissipation, good damping capacity, good electro-magnetic shielding effectiveness, and good recycling. However, magnesium has the lowest electrochemical potential of common commercial metals and is extremely prone to corrosion, it is necessary to apply surface treatment. When treating the surface of such magnesium bodies, some degree of conductivity is required to prevent charging of the electronic circuit and to improve the electromagnetic shielding effectiveness. Magnesium alloy treated by anodizing from the phosphate electrolytic solution was excellent corrosion resistance, but this coating was insulator. For the purpose of this study is to improve both corrosion resistance and conductivity of magnesium alloy products, anodic oxidation coatings from the phosphate electrolytic solution were removed by laser processing. The area where anodized coating was removed by the appropriate laser processing condition showed the excellent corrosion resistance as well as good conductivity. This improvement of the conductivity is attributable to the removal of the anodized coatings, and excellent corrosion resistance is based on the sacrifice corrosion protection by anodizing from the phosphate electrolytic solution.

## G7 The Effect of Grain Size and Film Thickness on the Coefficient of Thermal Expansion of Copper and Silver Thin Films: *Seul-Gi Hwang*<sup>1</sup>; Young-Man Kim<sup>1</sup>; <sup>1</sup>Chonnam National University

When thin films are deposited, stresses arise inevitably. The stresses include both intrinsic and extrinsic (thermal) stresses. The intrinsic stress is the stress arising during deposition, which is dependent on the deposition methods and conditions. The extrinsic (thermal) stress arises from the temperature change during deposition and/or service. If thin films encounter frequent temperature changes, thermal stress may lead to damage or deformation to the thin film structures in electronic devices. Therefore, the knowledge of the thermo-mechanical properties of thin films, such as the CTE, will be important for determining the stability and reliability of the devices that includes thin film structures. In this study, Cu and Ag thin films underwent thermal cycling to determine their Coefficient of Thermal Expansion (CTE). The thermal stresses of the Cu and Ag thin films with various microstructures (different grain size and film thickness) were measured using a curvature measurement system. The thermal expansion coefficients of the films

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were obtained from the slope of the stress-temperature curve with the knowledge of the Young's modulus and Poisson's ratio. As a result, the coefficient of thermal expansion increased with increasing grain size. However, the film thickness did not show a remarkable difference.

#### **G8 Effect of Ultrasonic Surface Peening on Fatigue Property of 7B04 High Strength and Toughness Aluminum Alloy**: *Zhu Qifang*<sup>1</sup>; Sun Zeming<sup>1</sup>; Ma Tongda<sup>1</sup>; Vacily A. Klimenov<sup>2</sup>; Viacheslav Borozna<sup>2</sup>; Zhu Baohong<sup>1</sup>; <sup>1</sup>General Research Institute of Non-ferrous Metals; <sup>2</sup>Tomsk Polytechnic University

The present paper discussed the influence of ultrasonic surface peening on fatigue property of 7B04 high strength and toughness aluminum alloy (hereinafter as referred to as 7B04 alloy). The result showed that a nano layer formed with the thickness of  $10-20\mu$ m on alloy surface was comprised of crystals with grain size of 10-50 nm, meanwhile, the grain size increased from the surface to matrix, which formed a gradient structure, and grain orientation was in accordance with the direction of peening. On the other side, a residual compressive stress layer of 200 MPa, which caused that those potential or already existed micro surface cracks were compressed to closure, was formed on the surface of sample after ultrasonic peening. Consequently, the fatigue life of 7B04 alloy increased 5-10 times.

#### **G9** Mechanical Properties of Nitrided Martensitic Stainless Steel 431 by Multi Grid Active Ion Nitriding: *Bang Hyun Bae*<sup>1</sup>; Jo Hyung-Ho<sup>1</sup>; Jung Uoo-Chang<sup>1</sup>; Cha Byung-Chul<sup>1</sup>; Kim Wang Ryeol<sup>1</sup>; <sup>1</sup>KITECH

Nano-nitriding technology was applied to Stainless steel 431 and enhances mechanical properties of Stainless steel 431 which has high surface hardness and excellent corrosion resistance. The assessment of nitride layer, which is formed by appling nano-nitriding technology is performed. The material of Stainless steel 431 to ø20mm x 8mm of disc type with non-heat treatment and heat treatment was treated by nano-nitriding as a function of temperature, time, gas and pressure. The treated surface of Stainless steel 431 was studied by X-ray diffraction (XRD), optical and filed emission scaning electron microscopy (FE-SEM)). The mechanical properties of these sample were studied by wear tester and Vickers hardness tester. The value of hardness of the nano-nitriding is increased two to three times comparing to the value of substrate hardness. By nano-nitriding for a long time, the hardness difference between heat treatment and non heat treatment is quite small. The result of wear test shows that the difference of friction coefficient between heat treatment and non heat treatment is big.

#### **G10** Preparation of Colored Oxide Film on Stainless Steel to Improve Corrosion Resistance: *Jaemin Kim*<sup>1</sup>; Junghoon Lee<sup>1</sup>; Jinyoung Lim<sup>1</sup>; Jangbeom Han<sup>1</sup>; Sujeong Lee<sup>1</sup>; Wooyoung Jung<sup>1</sup>; Wonsub Chung<sup>1</sup>; <sup>1</sup>Pusan National University

The coloring process is one of the attractive surface finishing techniques for stainless steel. By the coloring process, an added value from the visually beautiful appearance for decorative purposes could be obtained, and also an improvement of corrosion characteristics from thick colored oxide film could be expected. Coloring treatments were applied to stainless steel 304 to improve corrosion resistance in salt water. Several electrochemical coloring techniques, such as potentiostatic, triangular scan and square wave current pulse were carried out in hot chromic acid solution. Composition and structure of colored oxide film were also analyzed using GDS and XPS. Surface morphology was also observed using FE-SEM. Corrosion resistance was tested by potentiodynamic polarization in 3.5 wt.% NaCl solution. From the difference of oxide film thickness, the surface of stainless steel appears different colors by interference of light. During the growth of colored oxide film, grainboundary was dissolved. After coloring treatments, corrosion resistance was improved than bared stainless steel.

#### **G11** Antibacterial and Corrosive Properties of Stainless Steel Implanted by Silver and Zinc Ions: Hongwei Ni<sup>1</sup>; Rongsheng Chen<sup>1</sup>; Weiting Zhan<sup>1</sup>; Hanshuang Zhang<sup>1</sup>; <sup>1</sup>Wuhan University of Science and Technology

Stainless steel has been widely used in kitchenware and medical apparatus The incidence of infections caused by the use of bacterial colonized stainless steel has led to the development of stainless steel with antibacterial properties. Silver or zinc ions were the usual antibacterial agents in preparation of antibacterial stainless steel. But few reports studied the antibacterial and corrosive performance of stainless steel with both silver and zinc ions. Stainless steel implanted by silver and zinc ions simultaneously was firstly reported in this work with a metal vapour vacuum arc source at an extracting voltage of 50 KV. The implanted layer was characterized by X-ray photoelectron spectroscopy. The depth profile showed that the sputtered depth of both silver and zinc ions were about 80 nm below the surface. Atomic force microscope revealed that the surface roughness decreased with the increasing of implantation dose. The antibacterial rates were estimated by standard plate count method. Excellent antibacterial activities (>99%) against both Gram-negative Escherichia coli and Gram-positive Staphylococcus aureus were obtained for Ag/Zn implanted stainless steel. Electrochemical polarization curves indicated that the corrosion resistance of stainless steel was slightly enhanced after implantation of silver and zinc ions.

G12 Annealing Effects of Mg-Ni Alloy Thin Films in Diluted Hydrogen Gas: *Tae-Won Kim*<sup>1</sup>; In-Ki Kim<sup>1</sup>; Gi-Soek Heo<sup>1</sup>; Jong-Ho Lee<sup>1</sup>; <sup>1</sup>KITECH

Mg–Ni alloy thin films have attracted great attention for the applications like switchable mirror, display, optical switches, gas sensors because these films demonstrate reversible optical and resistance change by exposuring to hydrogen containing gas. In this research, we have studied the hydrogenation effect on Mg-Ni alloy thin films fabricated by combinatorial sputter system, with which we could make composition spread Mg-Ni films. Mg-Ni alloy thin films with composition range of 91.7~37at% (Mg/(Mg+Ni)) fabricated on glass substrate (corning 1737) at room temperature. The Mg-Ni alloy thin films annealed in hydrogen diluted Ar gas. The surface morphology, optical, electrical and structure properties of the thin films were investigated by using field emission scanning electron microscopy (FE-SEM), UV/VIS, Hall effect measurement system and XRD. The Mg-Ni alloy films with the composition ratio (Mg/(Mg+Ni) of 6.6:1~4.9:1 showed significant change in visible range transmittance as well as electrical resistivity after hydriding above 150 C. These results demonstrate that the films could be used as a active layer for switchable mirror.

**G13 Electrochromic Properties of Nano-Columnar Nickel Oxide**: Chih-Ming Wang<sup>1</sup>; *Kuo-Sheng Kao*<sup>2</sup>; Da-Long Cheng<sup>2</sup>; Chien-Chuan Cheng<sup>3</sup>; Po-Tsung Hsieh<sup>4</sup>; Tai-Yu Shih<sup>1</sup>; Chih-Yu Wen<sup>1</sup>; <sup>1</sup>Cheng Shiu University; <sup>2</sup>SHU-TE University; <sup>3</sup>De Lin Institute of Technology; <sup>4</sup>National Cheng Kung University

Electrochromic properties of transition metal oxides had much attention in recent years. The electrochromic thin films can be assembly as electrochromic devices (ECDs) and then used for applications in devices such as mirrors, panels and smart windows. A kind of complementary ECD is popular in resent years. Therefore, a specific investigation on nickel oxide (NiO) electrochromic properties is completed in this study. The crystalline structure of the NiO films was analyzed using XRD (PANalytical X'Pert PRO) with Cu-Ka radiation. The atmosphere of oxygen concentration increasing has changed the NiO films crystalline from (200) to (111). The thicknesses and surface microstructures of the NiO films were investigated using a scanning electron microscope (SEM, Philips/FEI XL40 FEG). It is observed that films are relatively smooth deposited without oxygen. The characterization of the electrochromic properties was carried out in a twoelectrode cell with an electrochemical analyzer (CHI 611B). The NiO changes the transmittance of NiO films in the wavelength range of 300-1500 nm and the color of the film changes from transparent to brown. The nano-crack exhibits in the NiO film did enhance the electrochromic properties.

#### G14 "In-situ" Preparation of A TiO<sub>2</sub>/Eu<sub>2</sub>O<sub>3</sub> Composite Film upon Ti Alloy Substrate by Micro-Arc Oxidation and Its Photocatalytic Property: Yongqian Wang<sup>1</sup>; Chunxu Pan<sup>1</sup>; <sup>1</sup>Wuhan University

Micro-arc oxidation (MAO) has been developed as an "in-situ" process for sintering a ceramic coating on the surface of the metallic substrates. In this paper, a TiO<sub>2</sub>/Eu<sub>2</sub>O<sub>2</sub> composite film was prepared upon a titanium alloy (Ti-6Al-4V) substrate by using MAO technique. The microstructures and photocatalytic property of the films were characterized by using X-ray diffractometer (XRD), scanning electron microscope (SEM), X-ray photoelectron spectroscopy (XPS), UV-visible absorption spectra, etc. The experimental results showed that photocatalytic performance of the TiO<sub>2</sub>/Eu<sub>2</sub>O<sub>3</sub> composite film exhibited two times higher than that of the film without the Eu<sub>2</sub>O<sub>2</sub> additive. The reason is that in Eu<sub>2</sub>O<sub>2</sub>, ion Eu3+ has an incomplete 4f orbital track and an empty 5d orbital track, which tend to produce multi-electron configuration and therefore can effectively inhibit the recombination of photo-generated electrons and holes. In addition, the groundstate energy and the excited-state energy of ion Eu3+ are very closer, which make the transition of f-electron from the ground-state to the excited-state under the visible light irradiation, and then exhibits a higher absorption in the visible light range. Eu<sub>2</sub>O<sub>3</sub> acts as a catalyst or an assistant catalyst during the MAO process.

#### G15 Production of Stainless Cast Iron Base Deposits with Dispersed Titanium Carbide Particles by Plasma Spraying: Yasuhiro Hoshiyama<sup>1</sup>; Tsutomu Miyazaki<sup>1</sup>; Hidekazu Miyake<sup>1</sup>; <sup>1</sup>Kansai University

Fe-C-Ti-Cr-Ni alloy powder in diameter of 32-53µm made by argon atomization is low-pressure plasma sprayed to produce stainless cast iron base deposits with finely dispersed titanium carbide particles. The as-sprayed deposit formed on a water-cooled substrate consists of  $\gamma$ Fe,  $\alpha$ Fe, TiC and Cr<sub>3</sub>C<sub>2</sub>. Heat treatment of the as-sprayed deposit above 873K results in the formation of Cr<sub>7</sub>C<sub>3</sub>. The fine precipitates of about 0.2µm in the as-sprayed deposit formed on a watercooled substrate are carbide. The as-sprayed deposit on a non-cooled substrate and deposits which are obtained by heat treatment of the as-sprayed deposit are composed of  $\gamma$ Fe,  $\alpha$ Fe, TiC, Cr<sub>3</sub>C<sub>2</sub> and Cr<sub>7</sub>C<sub>3</sub>. As heat treatment temperature increases, carbide precipitates corsen. The hardness of deposit decreases with increasing heat treatment temperature. The wear resistance of as-sprayed deposit formed on a non-cooled substrate is higher than that of the deposit heat-treated at 1273K. The as-sprayed deposit and deposit heat-treated at 1273K have higher wear resistance than a commercial stainless steel.



#### G16 The Stability Evaluation in Sea Water of Zn Thermal Spray Coating and

Its Sealing: Seong-Jong Kim<sup>1</sup>; Seung-Jun Lee<sup>1</sup>; <sup>1</sup>Mokpo Maritime University

Marine transportation is an international competitive industry that is strategically important for the nation. This study applied thermal spray coating technology with excellent electrochemical characteristics and cavitation resistance. Spray coating with Zn wires for corrosion resistance was applied to steel used as a ship material, and the electrochemical and cavitation characteristics were compared according to the existence of fluorine-silicon sealer. Cavitation test was used a ultrasonic vibration generator with the requirements of ASTM-G32. The electrochemical apparatus consisted of a Pt coil as the counter electrode and Ag/AgCl reference electrode. The natural potential measurement test was performed for 86,400 seconds. Anodic and cathodic polarization trend were tested from the open circuit potential to +4.0V and -3.0V. For potentiostatic experiment to compare corrosion resistance, the changes in current density for 3.600 seconds at a constant potential and the values after 3,600 seconds were compared in various applied potential conditions. For Tafel analysis, the corrosion potential and corrosion current density were obtained by polarizing  $\pm$  0.25V.At the results of electrochemical and cavitation experiments, the sealed specimen presented good electrochemical and anti-cavitation characteristics.

#### **G17 Mechanical Stress in Thermally Evaporated Amorphous Ge33As12Se55 and As40S60 Thin Films**: *Douglas Bulla*<sup>1</sup>; Rongping Wang<sup>1</sup>; B Luther-Davies<sup>1</sup>; <sup>1</sup>Australian National University

We report the investigation of stress behaviour in thin films of Ge33As12Se55 and As40S60 chalcogenide glasses deposited by thermal vacuum evaporation from bulk material and submitted to thermal treatments. The films, ~3 µm thick, were deposited on Si wafers at room temperature in a rate of ~3 Å/sec. The post deposition thermal treatments were carried out in vacuum at temperature slightly lower than the specific glass transition temperature. The stress in the deposited films (s) was estimated by the curvature of the Si wafer substrate using the Stoney's formula. The wafer curvature was measured by optical reflectometry before deposition, just after deposition and after annealing. Both films presented low intrinsic stresses ( $|s| \sim 10$  MPa) and relatively higher thermal stresses ( $|s| \sim 30$ MPa). The mechanical stress for Ge33As12Se55 film evolve from an intrinsic compressive to a tensile thermal stress after annealing, and for As40S60 films the reverse stress behaviour was observed. The results of the stress measurements are in agreement with the variations in film thickness and optical properties measured before and after annealing. Also, the effects of the annealing process on the films glass network arrangements were investigated by x-ray diffraction and the results were compared with the stress measurements.

#### **G18 Structural Stabilities in GaAs Nanocrystals Grown on Si (111) Surface**: *Hidehiro Yasuda*<sup>1</sup>; Kimihisa Matsumoto<sup>2</sup>; Takuya Furukawa<sup>1</sup>; Masaki Imamura<sup>1</sup>; Noriko Nitta<sup>1</sup>; Hirotaro Mori<sup>3</sup>; <sup>1</sup>Kobe University; <sup>2</sup>Toyama Prefectural University; <sup>3</sup>Osaka University

Structural stabilities in GaAs nanocrystals grown on the Si (111) substrate have been studied by transmission electron microscopy in order to see the structure and growth mechanism. The GaAs nanocrystals grown epitaxially on the Si (111) surface kept at 573 K have thin shapes consisting of a flat surface which parallels to the Si (111) surface. The crystalline structure of the initial growth layer below approximately 10 nm in thickness is the zinc-blend structure, but with increasing thickness the structure changes to the wurtzite structure to relax the lattice strain. The wurtzite structure is due to the changes in the sequence of stacking on the (111) lattice planes of the zinc-blend structure parallel to the Si surface. On the other hand, the nanocrystals grown at 673 K are stabilized as the zinc-blend structure over all of the thickness. During high temperature growth, the lattice strain in the interface between GaAs nanocrystals and Si substrate is relaxed over all of the GaAs layer, but during lower temperature growth the relaxation of the interfacial strain is limited only in the thin GaAs layer near the interface.

### **G19 Study of Deposition of Aluminum Nitride Thin Films by Hollow Cathode Electron Beam Vapor Deposition Method**: *Mu Zongxin*<sup>1</sup>; <sup>1</sup>Dalian University of Technology

Aluminum nitride (AIN)thin films were deposited on (100) oriented silicon wafers substrates by a hollow cathode electron beam vapor deposition system (HCEBVD) under various Ar/N2 flow ratio. The films were characterized by tapping mode atomic force microscopy (AFM),glancing incident X-ray diffraction (GIXRD) techniques and ultraviolet/visible (UV/VIS) spectrophotometer. It was found that the thin films are polycrystalline and have a hexagonal wurtzite structure with (002) preferred orientation, as revealed by GIXRD. AFM analysis indicates that the surface of the thin films is smooth, with Ra=0.7 nm, which is suitable for application in surface acoustic wave devices. EDS analysis gives the chemical composition of the coatings. The film thickness and optical refractory properties of the AIN thin films were investigated.

**G20 Synthetic Properties of the C-Axis Tilted AlN Thin Films**: *Chung-Jen Chung*<sup>1</sup>; Ching-Liang Wei<sup>2</sup>; Po-Tsung Hsieh<sup>1</sup>; Chao-Yu Huang<sup>1</sup>; Jen-Fin Lin<sup>1</sup>; Ying-Chung Chen<sup>2</sup>; Chien-Chuan Cheng<sup>3</sup>; <sup>1</sup>National Cheng Kung University; <sup>2</sup>National Sun Yat-Sen University; <sup>3</sup>De Lin Institute of Technology

Aluminum nitride (AlN) is one of the most popular piezoelectric materials for high frequency resonators, filters and sensors. The piezoelectric property, i.e. electromechanical coupling coefficient, of AIN thin film is highly related to its crystalline orientation. AIN thin films with various c-axis-tilted angles can be fabricated by the RF sputtering technique. The crystallization and grain growth orientations of AlN thin film are examined by XRD, SEM, and TEM, while the bonding condition and nano-mechanical properties are investigated by a raman system and a nano-indentation system. From the SEM images and the O-2O Xray patterns, the AIN thin films possess 0 to 30 degree tilted columnar structure and (002) c-axis preferred orientation. TEM examinations show detailed crystalline structures. From the nano-indentation tests, the elastic modulus gradually decreases as the c-axis-tilted angle increases. The hardness seems to have a similar trend but has a sudden rise for the 30 degree tilted AIN. Stronger raman shift intensity at 940 cm<sup>-1</sup> is detected for the AIN with larger c-axis tilted angle. The synthetic properties of the c-axis tilted AIN thin films will be discussed thoroughly in this paper.

#### G21 The High Temperature Oxidation Behavior of SiO<sub>2</sub> Protective Layer Coated IN738LC Using CCVD: Kyoung-Soo Park<sup>1</sup>; Youngman Kim<sup>1</sup>; <sup>1</sup>CNU

Gas turbines should be operated at high temperatures to increase their fuel efficiency. The turbine materials should be able to endure the harsh environments of high temperature oxidation. Thermal Barrier Coating (TBC) on the surfaces of turbine blades is commonly used to protect gas turbine materials by reducing the level of thermal conduction and protecting from high temperature oxidation. To prolong the lifetime, the protective coatings were processed on TBC of turbine blade material. In this study, a SiO<sub>2</sub> protective layer was processed using 0.03 mol concentration of TEOS (tetraethyl orthosilicate, C8H2004Si) by Combustion Chemical Vapor Deposition (CCVD). The Ni-base superalloy(IN738LC) specimens coated with SiO<sub>2</sub> protective layer were burned by the flame from the CCVD equipment for 10 min. at the temperature range of 1150  $\eta$  to 1350  $\eta$ in air under similar conditions for gas turbine operation. SEM, EDX and XRD analysis were performed to analyze the changes in the SiO, protective layer. After burning the amounts of Ti and Cr elements were increased and those of Al, Co and Si decreased. Oxides of Ti and Cr were observed on the surface of the burned specimens and Ti and Cr might be important elements against oxidation

### **G22** Effect of Nitridation on GaN Film Grown on Glass Substrate by ECR-PEMOCVD Method: *Fu-Wen Qin*<sup>1</sup>; Ai-Min Wu<sup>1</sup>; Feng-Chun Liu<sup>1</sup>; Bao-Dan Liu<sup>1</sup>; Xin Jiang<sup>1</sup>; <sup>1</sup>Dalian University of Technology

Nitridation of Corning 7101 glass substrate and the following GaN deposition were carried out in the electron cyclotron resonance plasma enhanced metalorganic chemical vapor deposition (ECR-PEMOCVD) system equipped with in-situ RHEED monitoring. The N2 and trimethyl-gallium (TMGa) were used as N and Ga sources, respectively. Glass substrates were cleaned by H2 plasma for 10 min at 650W microwave power after the substrate temperature was raised to 430°C. Keeping the temperature and microwave power unchanged, the nitriding time was varied from 0~75 min with a constant N2 gas flow rate of 70 sccm. Then the GaN film was deposited for 200 min under the following condition: TMGa gas flow rate at 0.4 sccm. RHEED pattern and XRD spectrum showed that the nitridation can effectively improve the C-axis orientation of as-prepared GaN film. AFM analysis indicated that the average grain size increased significantly with 5 min of nitriding, but degraded as nitriding time increased. The optimum nitriding time was achieved as 5 min. The effect of nitridation on the GaN film deposition mechanism were discussed.

### **G23 Formation of Various Size InSn Metal Sphere by Post-plasma Treatment**: *Dong-Hoon Han*<sup>1</sup>; Soon Ho Kwon<sup>1</sup>; Jung Joong Lee<sup>1</sup>; <sup>1</sup>Seoul National University

Owing to the low contact resistance, high switch density and wear stability, the liquid-metal droplet switch is regarded to be an excellent candidate for the next generation micro electro-mechanical switch and reconfigurable circuit interconnections. Since injecting liquid metal is not suitable to make such liquid metal droplet switches by the integrated circuit process, a new process which consists of deposition and post-heat treatment is proposed. As the first process step indium-tin eutectic alloy was deposited on a blank Si (100) wafer and heat treated with or without plasma to make metal droplets. Hydrogen plasma was used for the investigation of the plasma effect by the heat treatment. The morphology of the as-deposited and heat treated samples was observed by field emission scanning electron microscopy (FESEM). As-deposited indium-tin film showed a cauliflower-like morphology, while it was changed to breath figure pattern which was formed with various size droplets. It was found that by using plasma the sample showed more sphere-shaped droplets after the heat treatment.



Also, it was observed that droplet size was dependent on film thickness and posttreatment plasma power.

#### G24 The Tribological Properties of Mo<sub>2</sub>N/Cu Films with Mo-Cu Compound Target by Applying Inductively Coupled Plasma: *Jin-nam Kim*<sup>1</sup>; Somi Park<sup>1</sup>; Shinyoung Kim<sup>1</sup>; Jung Joong Lee<sup>1</sup>; <sup>1</sup>Seoul National University

 $Mo_2N/Cu$  films were deposited on silicon substrate and tappet with Mo 95wt%-Cu 5wt% compound target by inductively coupled plasma sputtering system. The film properties such as hardness, adhesion and wear resistance were measured. The structure of the films was confirmed by SEM and XRD. The (200) oriented  $Mo_2N$  films were obtained in high nitrogen flow rate (>1.5sccn) and low substrate bias (<-20V). While Mo films became predominant low nitrogen flow rate (<1sccm) and high substrate bias (>-50V). Hardness of the films varied between 10GPa and 40GPa when substrate bias and nitrogen flow rate were changed in the range of 0V~100V and 1sccm~2.5sccm respectively. Adhesion of the films increased up to 50N while hardness of the film reduced to 18GPa when the substrate bias was induced -50V. To increase adhesion, bi-layer coating which is consisted of soft layer having good adhesion and hard layer having poor adhesion was deposited. Wear test was performed in the dry and oil circumstance.

## **G25** Effect of Interlayer Thickness on Stress and Dielectric Properties of **MgTiO<sub>3</sub> Modified (BaSr)TiO<sub>3</sub> Multilayer Thin Films**: *Shengbo Lu*<sup>1</sup>; Zhengkui Xu<sup>1</sup>; <sup>1</sup>City University of Hong Kong

 $(BaSr)TiO_3/MgTiO_3/(BaSr)TiO_3$  (BST/MT/BST) multilayer composite thin films were deposited on LaNiO<sub>3</sub> covered Pt/Ti/SiO<sub>2</sub>/Si substrates by pulsed laser deposition (PLD). Dense and crack-free multilayer thin films were successfully prepared by PLD. The thickness of MT interlayer has a significant impact on the residual stress and dielectric properties of the BST/MT/BST multilayer thin films. It was found that the residual stress in the multilayer thin films decreases significantly with increasing MT interlayer thickness, mainly due to a close match of thermal expansion coefficients between the BST and MT. On the other hand, dielectric constant, loss tangent and tunability decrease with increasing MT thickness, mainly attributed to a series dilution effect. The results indicate that the series dilution effect plays a more important role than stress effect does in determining the dielectric properties of the multilayer thin films. The BST/MT/BST multilayer thin film with a 50nm MT interlayer exhibits the best combination of tunability (30%) and loss tangent (0.016), which is desirable to potential tunable device applications.

### **G26 Effect of Atomic Layer Deposited Ni Thin Films on the Mechanical Properties of Nitinol Shape Memory Alloy**: *Young-Keun Jeong*<sup>1</sup>; Se-Hun Kwon<sup>1</sup>; Myung-Chang Kang<sup>1</sup>; <sup>1</sup>Pusan National University

A thin film of elementary nickel was deposited by atomic layer deposition (ALD) on nitinol shape memory alloys for a biomedical application. To prepare Ni thin films on nitinol shape memory alloy, the deposition cycle for Ni ALD consisted of two sequential chemical reaction steps: an oxidizing step and a reducing step. An atomic nickel oxide layer was made by using bis(cyclopentadienyl)-nickel and water; then nickel oxide layer was reduced to Ni metal by exposure to hydrogen plasma. Auger electron spectroscopy analysis detected negligible oxygen content in the grown films. Also, carbon impurities in the film reduced from 16 atom % to less than 5 atom % during the reduction reaction. In this presentation, the mechanical properties of Ni coated nitinol shape memory alloy will be discussed and compared with uncoated pure nitinol shape memory alloy.

#### **G27** Deposition of GaN Films on Freestanding CVD Thick Diamond Films: Dong Zhang<sup>1</sup>; Yi-Zhen Bai<sup>1</sup>; Fu-Wen Qin<sup>1</sup>; Ji-Ming Bian<sup>1</sup>; <sup>1</sup>Dalian University of Technology

High quality GaN films are deposited on freestanding thick diamond films by electron cyclotron resonance plasma enhanced metal organic chemical vapor deposition (ECR-PEMOCVD). The characteristics of GaN films were investigated by x-ray diffraction analysis (XRD), reflection high energy electron diffraction (RHEED) and atomic force microscopy (AFM). The high quality GaN films with small surface roughness of 8.3 nm and high c-orientation are successfully achieved at the optimized nitriding time with the diamond substrate. These properties of GaN films with small surface smoothness and high c-orientation are well used as piezoelectric films for surface acoustic wave (SAW) devices.

**G28 Electrodeposition of CuSn Alloy from Noncyanide Sulfosuccinate Bath**: *Toshihiro Nakamura*<sup>1</sup>; Tomio Nagayama<sup>1</sup>; Takayo Yamamoto<sup>1</sup>; Yasushi Mizutani<sup>1</sup>; Hidemi Nawafune<sup>2</sup>; <sup>1</sup>Kyoto Municipal Industrial Research Institute, Industrial Technology Center; <sup>2</sup>Frontiers of Innovative Research in Science and Technology, Konan University

Recently the regulation of nickel allergy has become strengthen in Europe and other countries. Cu-Sn alloy (40–55 wt.% Sn) is called "speculum alloy" or "white bronze" and has silvery-white appearance. We developed noncyanide Cu-Sn alloy plating bath consist of sulfosuccinic acid, L-methionine and polyoxyethylene- $\alpha$ -naphthol, from which silvery-white Cu-Sn alloy (40-55wt.%

Sn) were obtained. The Cu-Sn alloy films anticipated for use as an alternative to nickel undercoating for decorative gold or chromium electroplating.

#### **G29 Electrodeposition of ZnTe Compound Semiconductors from Aqueous Solution**: *Takashi Ikeda*<sup>1</sup>; Takeshi Ohgai<sup>1</sup>; Yasuyuki Kawanaka<sup>1</sup>; Keizo Takao<sup>1</sup>; Akio Kagawa<sup>1</sup>; <sup>1</sup>Nagasaki University

ZnTe compound semiconductors were synthesized in acidic aqueous solution using a pulsed current electrodeposition technique. Optimum condition to obtain ZnTe deposits was determined by the cathodic polarization curves measured at a wide potential range. During the co-deposition of Zn and Te, under potential deposition (UPD) of Zn was observed. Increasing the solution temperature up to 353 K, UPD of Zn was promoted by the formation of Zn(OH)<sub>2</sub>. Crystal phase, structure and chemical composition of electrodeposited ZnTe was controlled by the solution composition and electrolysis condition. Band gap energy of ZnTe films annealed at 573 K was close to 2.26 eV.

## **G30** Epitaxial Growth of BaTiO<sub>3</sub> Thin Films on Mgo using Pulsed Laser Deposition: *Rongping Wang*<sup>1</sup>; Douglas Bulla<sup>1</sup>; Steve Madden<sup>1</sup>; Andrei Rode<sup>1</sup>; Barry Luther-Davies<sup>1</sup>; <sup>1</sup>Australian National University

Due to the large contrast in refractive index and good lattice-matching, epitaxial growth of electro-optic BaTiO<sub>3</sub> thin film on MgO wafer has great potential as an electro-optic modulator for the application in integrated optics. We therefore prepared BaTiO<sub>3</sub> thin films on MgO(001) single crystalline substrates using pulsed laser deposition method. We evacuated the chamber down to  $3X10^{-5}Pa$  and introduce oxygen to various pressures. With a fixed growth temperature of 800°C, the as-grown films show a (111) and (001) preferential orientation at low deposition pressure of 1X10-2Pa, and become dominated by (001) orientation at 0.1Pa. When the deposition pressure is kept at 0.1Pa, the as-grown films shows (111) and (001) preferential orientation. X-ray pole figure analysis along BaTiO<sub>3</sub> (111) direction indicated that the film prepared at 0.1Pa and 800°C have four symmetric distribution of the diffraction intensity, confirming the epitaxial growth of the film. The optical properties such as transmission, the dispersion of the refractive index are also investigated.

#### **G31** Fabrication of Co/Cu Multilayered Nanowires Using a Pulsed Current Deposition Technique: *Keisuke Hashiguchi*<sup>1</sup>; Takeshi Ohgai<sup>1</sup>; Takao Morimura<sup>1</sup>; Keizo Takao<sup>1</sup>; Akio Kagawa<sup>1</sup>; <sup>1</sup>Nagasaki University

Co/Cu multilayered nanowires with 40 nm in diameter were fabricated using a pulsed current deposition technique into a nanoporous template with numerous nanochannels. To determine the optimum electrodeposition condition of Cu and Co into the template, cathodic polarization behavior was examined at a wide range of cathode potential. Time-dependence of deposition current was monitored to determine the growth rate of Co and Cu nanowires. Co layer and Cu layer thicknesses were adjusted to several tens nanometers, by controlling the deposition times. With decreasing the each layer thickness, the coercive force of Co/Cu multilayered nanowires was decreased and the soft magnetic property was improved.

### **G32** Fabrication of Densely Distributed Silver Indium Selenide Nanorods by Ag+ Ion Irradiation: *Dinesh Pathak*<sup>1</sup>; <sup>1</sup>Physics, GNDU, Amritsar

We prepared polycrystalline AgInSe<sub>2</sub> thin films by vacuum evaporation on Si(100) substrate at a high temperature using the stochiometric powder. The thin films were characterized by X-ray diffraction and Uv-vis-NIR spectroscopy .For the fabrication of densely distributed one dimensional nanostructures of Silver Indium selenide on Si substrates, the thermally evaporated films of AIS on Si (1 0 0) substrate were irradiated by incident 200MeV Ag+ ions at a fluence of 5 X 1011 Ion/cm<sub>2</sub>. At elevated substrate temperatures AIS were featured by the nanorods -like structure. The optical and structural properties of the irradiated films were studied using UV–visible absorption spectroscopy, atomic force microscopy (AFM), Field emission scanning electron microscopy (FESEM ) and XRD. The controlled fabrication of such densely distributed one dimensional nanorods on Si substrate using ion beam technique, we believe, would open up a variety of applications such as nanoelectronics and optoelectronics devices.

#### **G33 Focused Ion Beam Makes an Angled Nano-Tunnel with High Aspect Ratio on Poly(methyl methacrylate)**: *Eun Kyu Her*<sup>1</sup>; Hee-Suk Chung<sup>2</sup>; Myoung-Woon Moon<sup>3</sup>; Kyu Hwan Oh<sup>2</sup>; <sup>1</sup>Ohio State University; <sup>2</sup>Seoul National University; <sup>3</sup>Korea Institute of Science and Technology

Angled nano-scale tunnels with high aspect ratio were fabricated on the Poly(methly methacrylate) (PMMA) using a focused ion beam (FIB). The fabrication parameters such as ion fluence, incidence angle, acceleration voltage of the Ga+ ion beam were first studied on PMMA surface for exploring the formation of the nano-scale configurations such as nano-holes and cones with diameter ranging 50 to 150 nm at the ion beam acceleration voltage of  $5 \sim 20$  kV. It was also found that PMMA surface exposed to FIB was changed into an amorphous graphitic structure. Angled nano-scale tunnels were fabricated with



high aspect ratio of  $700 \sim 1,500$  nm in depth and 60 nm in mean diameter at an ion beam acceleration voltage of 5 kV and under a specific ion beam current. The angle of the nano-tunnels was found to follow the incident angle of the ion beam tilted from 0° to 85°, which has the potential for creating a mold for anisotropic adhesives by mimicking the hairs on a gecko's feet.

#### G34 Multi-Layer Coating for Optical Mold of Strengthening by Electroplating Ni-W and Electroless Plating Ni-Mo-P by Nonisothermal Method: Yun-Feng Chang<sup>1</sup>; kung-Hsu Hou<sup>1</sup>; <sup>1</sup>National Defense University, Taiwan

Optical mold development and manufacturing process for precision optical components, one of the key technologies in recent years to improve the life of the international community has begun to seek higher temperature resistance, corrosion resistance strength electroforming material, such as Ni-W, and Ni - Mo-P ternary alloy plating, etc. To enhance the glass-molding die life, creating two kinds of multi-layer coating process to enhance mechanical strength and high temperature, wear-resistant properties. First, electro-deposition approach to the development of thick-film Ni-W coating, and then electroless plating by nonisothermal method of Ni-Mo-P thin-film, to form a multi-layer coating. For the coating of the composition, mechanical properties such as analysis of the first assessment. And then for the multi-layer coating of high temperature resistance, thermal stability and to conduct a comprehensive assessment of mill-run to serve as a reference basis for the development of optical mold.

# **G35** Multilayered Approach to Step-Edge Josephson Junctions: *Olga Shcherbakova*<sup>1</sup>; A. V. Pan<sup>1</sup>; S. V. Pysarenko<sup>1</sup>; S. Fedoseev<sup>1</sup>; S. X. Dou<sup>1</sup>; J. Du<sup>2</sup>; S. Lam<sup>2</sup>; C. Foley<sup>2</sup>; <sup>1</sup>Institute for Superconducting and Electronic Materials, University of Wollongong; <sup>2</sup>CSIRO Materials Science and Engineering

Fabrication technology of Josephson junction is an important research field due to their potential applications in electronic devices, sensors, and metrology. Most important aspects for their applications are reproducibility and high critical current of the junctions. These issues are of a particular concern in high temperature superconducting (HTS) YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub> (YBCO) thin films. Multilayered approach to YBCO-based Josephson junction manufacture is introduced. Properties of monolayer YBCO and multilayer YBCO/NdBCO/YBCO junctions of similar thickness grown by pulsed-laser deposition on MgO (100) substrates has been analysed and compared. The benefits of the multilayered structure are presented and discussed.

#### G36 Numerical Simulation for Surface Modification of Thermal Barrier Coatings by High Current Pulse Electron Beam: *Ying Qin*<sup>1</sup>; Wei Qu<sup>1</sup>; Xianxiu Mei<sup>1</sup>; Shengzhi Hao<sup>1</sup>; Jijun Zhao<sup>1</sup>; Wen Lu<sup>1</sup>; Chuang Dong<sup>1</sup>; <sup>1</sup>Dalian University of Technology

High-current pulsed electron beam is a promising technique for surface sealing of initially rough and porous thermal barrier coatings prepared by PVD. This technique is characterized by low energy (5~20keV), short pulse time (10~200µs), and high current (50~200A). Due to the rapid remelting and solidification, the coating outer layer becomes smooth, dense, and wear and corrosion resistant, and the protective performance for turbine blades is quite enhanced. Due to the complex multi-layered structures, the high-current pulsed electron beam treatment requires some parameter inputs which are related to the temperature and stress fields induced by the electron energy deposition in the coatings. In the present work, two-dimension temperature field model is adopted to describe the temperature evolution and distribution in thermal barrier coatings by high current pulse electron beam. The simulation results indicate that the melting layer reaches about 3~5 µm. The heating rate is up to 107~108Ks-1 and the temperature gradient is about 109Km-1. These simulated results have been confirmed by experiments. Stress field simulated using ANSYS reveals that the maximum thermal stress in the thermal barrier occurs at the interface between the bond coating and thermally grown oxide layer.

**G37 PEM Fuel Cell Separator with Thermally Nitrided Low Carbon Steel:** *Dae-Geun Nam*<sup>1</sup>; Chang-Yong Choi<sup>1</sup>; Jae-Ho Jang<sup>1</sup>; Yeong-Do Park<sup>2</sup>; Namhyun Kang<sup>3</sup>; <sup>1</sup>Korea Institute of Industrial Technology; <sup>2</sup>Dong-Eui University; <sup>3</sup>Pusan National University Separator is one of the most important parts in PEM fuel cell. Stainless steels are

widely used as separator for its good mechanical properties and mass production.

However, for a good chemical compatibility, stainless steels need to have high

chromium content or surface treatment, which makes separator high cost. Low

cost of separator is important for commercial use. In this study, conventional low

carbon steel is used as base metal of separator. Low carbon steel is low at cost,

but has poor chemical properties for separator. For a good corrosion resistance,

low carbon steel needs to be surface treated. To make a uniform surface treated

layer on low carbon steel, chromium is conventionally electroplated on the steel and thermally nitrided. Surface treated low carbon steel is investigated using

microstructure and element analysis tools. Interfacial contact resistance and

polarization test is applied for the properties of fuel cell separator. The results

show that chromium nitrided layer uniformly formed on low carbon steel. And the surface treated steel showed a good corrosion resistance as a separator.

#### G38 Roughness Effect of (Ti,Cr)N Coatings for Bipolar Plate of Polymer Electrolyte Membrane Fuel Cell: *Hyoseok Choi*<sup>1</sup>; W.H. Hong<sup>1</sup>; J.J. Lee<sup>1</sup>; <sup>1</sup>Seoul national university

(Ti,Cr)N coatings were synthesized on a 316L stainless steel substrate by inductively coupled plasma (ICP)-assisted reactive DC magnetron sputtering. Well-crystallized (Ti,Cr)N films were obtained at N<sub>2</sub>/Ar=0.2. Surface roughness of the coatings was controlled by applying Ar plasma treatments at various substrate biases after the deposition process. The Davies method was used for the measurement of the interfacial contact resistance between the sample and carbon paper. The ICR values at a compaction force of 150 N/cm<sup>2</sup> increased from 4.5 mO\*cm<sup>2</sup> to 40 mO\*cm<sup>2</sup> with decreasing surface roughness of the coatings. It seems to be decrease of the real contact area between the coating and carbon paper. The corrosion resistance of the coatings was investigated by potentiodynamic and potentiostatic tests in 0.1N H<sub>2</sub>SO<sub>4</sub> + 0.2 ppm HF solution at 80°, simulating the PEMFC operation conditions. Despite of different surface roughness of the coatings, corrosion resistance shows similar values. The (Ti,Cr)N coatings exhibited excellent stability in both the anodic and cathodic environment.

## G39 Raman Scattering Studies in Oxygen-Vacancy Induced Ferromagnetism of Co-Doped ZnO Films: *Shijian Chen*<sup>1</sup>; Kiyonori Suzuki<sup>1</sup>; <sup>1</sup>Department of Materials Engineering, Monash University

The nature of defect responsible for room-temperature ferromagnetism in Co-doped ZnO remains elusive. As a common defect, oxygen vacancies are ubiquitous and characteristic of the growth and post-annealing processes in ZnO based materials. Oxygen vacancies in these oxides will introduce spatial disorder and generate lattice distortions, thereby distorting the electronic configuration and affecting the interaction between localized spins. One of the direct consequences of lattice distortion is the modification of the lattice vibrational modes which can be detected with Raman spectroscopy. Here we successfully induced room-temperature ferromagnetism in Co-doped ZnO films upon post hydrogen thermal treatment. High quality Zn1-xCoxO thin films were grown by pulsed laser deposition. The as-prepared films showed paramagnetic characteristics, while after having been annealed at 600°C for 3 h in H, the films exhibited apparent ferromagnetic behaviors with coercivity ~200 Oe. X-ray diffraction and X-ray photoelectron spectroscopy results showed no creation of any magnetic cobalt related secondary phase. Raman spectroscopy was used to study the effect of hydrogen thermal RTF treatment on the structural properties as well as magnetization in the films. It was found the presence of plenty of oxygen vacancies in the post annealed films is critical to the observed ferromagnetism.

G40 Remote Atmosphere Plasma Surface Energy Control on the Using the HMDSO(HexaMethlDisilOxane) Monomer: A-Ruem Han<sup>1</sup>; Jung-Han Song<sup>1</sup>; Jeanho Park<sup>1</sup>; Nak-Kyu Lee<sup>1</sup>; Geun-An Lee<sup>1</sup>; Seogou Choi<sup>1</sup>; *Hye-Jin Lee<sup>1</sup>*; <sup>1</sup>Korea Institute of Industrial Technology

We developed a novel process technique and system using the Remote Atmosphere pressure Monomer Plasma (RAMP). The RAMP can treat the hydrophobic and hydrophilic surface using argon, oxygen, nitrogen gas and monomer HMDSO(HexaMethlDisilOxane). The tests are carried out performed to find the uniform condition by the RF power (RF 40W, 50W, 60W) and distances (d=3mm, 4mm, 5mm) of from the RAMP gun of size 10cm to the specimen as soda lime slide glass (26x76x1mm) for uniform surface processing capability test. We obtain the optimum condition for the uniform hydrophobic surface energy of the RF power of 40W and distance as 4mm. And the treated RAMP specimen and the treated Remote Vacuum Monomer Plasma (RVMP) specimen were compared to equipment efficiency verification. Based on this result, we have demonstrated that the plasma treatment using the monomer can be used under the atmosphere pressure instead of vacuum state.

#### **G41 The Influence of Iron and Manganese on Microstructure of Aluminium** – **Silicon Alloy**: *Mehdi Mazar Atabaki*<sup>1</sup>; Alireza Darvishi<sup>2</sup>; Ali Maleki<sup>2</sup>; <sup>1</sup>UTM; <sup>2</sup>Yazd University

The effect of iron and manganese concentration on the morphology of complex intermetallics and their influences on the mechanical properties and microstructure has been studied in an Al-%16.67Si alloy with three levels of iron (0.4, 1.2, 1.8 wt.%), and three different levels of manganese ranging from 0.35 to 0.59 wt.%. The intermetallic compounds form at high iron contents or at high concentrations of manganese with low iron. In this study different amount of additives, iron/manganese, with regards to proportion of 1:2 has been considered. Results of this study showed that when amount of iron and manganese in the alloy increased, the fraction of intermetallics dramatically augmented; which this led tensile strength dropped to 187 MPa. The iron and manganese also lead to the formation of complex intermetallic properties, especially tensile strength, and also lead to the formation of excessive shrinkage porosity defects in casting.

The microstructural investigation by Climex software showed that the biggest intermetallic size reached to 12750  $\mu$ 2m with increasing the amount of iron from 1.2% to 1.8%. It was also showed that the volume percentage of intermetallic compounds increases as the iron, and manganese content increases.

#### G42 The Effects of Heat Treatment on the Bonding Strength of Surface-Activated Bonding (SAB)-Treated Copper-Nickel Fine Clad Metals: *Kyunghoon Kim*<sup>1</sup>; Sungchul Lim<sup>1</sup>; Hyoukchon Kwon<sup>1</sup>; <sup>1</sup>Korea Institute of Industrial Technology

Surface activated bonding (SAB) is a novel method for the precise joining of dissimilar materials. It is based on the concept that two atomically clean solid surfaces can develop a strong adhesive force between them when they are brought into contact at high vacuum condition without high deformation at a 40–90%. With this SAB process, the effects of heat treatment on the bonding strength of surface-activated bonding (SAB)-treated copper-nickel fine clad metals were investigated. An increase in the SAB rolling load of the copper-nickel fine clad metals were interface roughness between the clad materials in the SAB rolling load decreased the interface voids formed by initial microrange surface roughness between the clad materials in the SAB cladding process. Unlike conventional cold rolling, outstanding interface diffusion between the clad materials was not observed after heat treatment. In addition, the peel strength increase of the clad metals compare with initial peel strength increased with SAB rolling load (<1% reduction ratio at a roll load of 5000 kgf ) up to 3.99 N/mm after heat treatment.

G43 The Optimization of SMR-Based Filter by Thermal Annealing Treatment: *Ching-Liang Wei*<sup>1</sup>; Ying-Chung Chen<sup>1</sup>; Chien-Chuan Cheng<sup>2</sup>; Kuo-Sheng Kao<sup>3</sup>; Chung-Jen Chung<sup>4</sup>; Wei-Tsai Chang<sup>1</sup>; <sup>1</sup>National Sun Yat-Sen University; <sup>2</sup>De Lin Institute of Technology; <sup>3</sup>SHU-TE University; <sup>4</sup>National Cheng Kung University

In this study, an SMR-based filter was fabricated by RF/DC magnetron sputtering and photolithography, and a thermal annealing treatment was adopted to improve the frequency response. The SMR-based filter is composed of a ZnO piezoelectric thin film onto W/SiO<sup>2</sup> Bragg reflector. ZnO thin films were prepared by two-step sputtering with various deposition temperatures to obtain good piezoelectric properties. ZnO piezoelectric layers at the deposition temperature of 200°C exhibit a highly c-axis preferred orientation, good crystalline characteristics and low surface roughness. The filter is thermal treated by RTA technique in O<sup>2</sup> ambient. The thermal annealing treatment improves the film properties of ZnO layers, resulting in a higher c-axis preferred orientation and a lower surface roughness of ZnO films than those of as-deposited ZnO films. The atomic ratio of Zn to O in ZnO film approaches one at the annealing temperature of 400°C, which results in a comparatively oxidized stoichiometric ZnO film. Finally, the frequency response of the annealed filter is improved, and a lower insertion loss is obtained.

G44 High Quality FBAR Sensor Operating in Liquid: *Chien-Chuan Cheng*<sup>1</sup>; Re Ching Lin<sup>2</sup>; Wei-Tsai Chang<sup>3</sup>; Ying-Chung Chen<sup>3</sup>; Kuo-Sheng Kao<sup>4</sup>; <sup>1</sup>De Lin Institute of Technology; <sup>2</sup>Feng-Chia University; <sup>3</sup>National Sun Yat-Sen University; <sup>4</sup>Shu-Te University

The rising requirement for the nanomedicine of bio-sensor has motivated the development of high-sensitive, low-cost and measurement toleration. The film bulk acoustic resonator (FBAR) has become a promising component for realizing easy fabrication, especially in high- sensitivity mass sensor. However, bio-sensor is operated in organic solvent and acid/alkali solution and therefore acid/alkali toleration and quality factor in liquid of FBAR sensor is development emphasis in recent. In this study, platinum (Pt) and zinc oxide (ZnO) had been adopted as electrode and piezoelectric layer of an FBAR sensor. Based on the off-axis deposition of the ZnO film, longitude and shear modes resonance phenomenon can be approached to sensitive in air and liquid respectively. The preferred orientation and crystal properties of the ZnO film were evaluated by X-ray diffraction (XRD). The crosssections of the grain structures of ZnO films were observed by scanning electron microscopy (SEM). The HP8720 network analyzer and CASCADE probe station were used to measure the frequency responses of FBAR devices. The result of FBAR frequency response in liquid, the quality factor of longitude mode is decayed apparent and shear mode is kept high value. The Pt electrode of FBAR sensor has to withstand attack of acid/alkali solution.



#### Poster Session: Symposium H: Advanced Ceramics

Tuesday PM	Room: Hall 2
August 3, 2010	Location: Cairns Convention Centre

H1 Research on the Powder Injection Molding of TiC Based Cermets: *Xianyong Bao*<sup>1</sup>; Bei Li<sup>2</sup>; Cheng Shu<sup>3</sup>; Meiqin Zeng<sup>1</sup>; Min Zhu<sup>1</sup>; <sup>1</sup>South China University of Technology; <sup>2</sup>Shenzhen FIYTA Holdings Ltd.; <sup>3</sup>Changchun University of Science and Technology

Compared TiC based Cermets with WC based Cermets when used in body touch watch case and its accessories, the former has low density, good human affinity and other advantages. But due to the difficult process of TiC based Cermets, when this kind of material is used in watch case and its accessories, the problems of precision forming of sophisticated shape watch parts need been solved. This paper studied on the injection molding of complex watch parts and the result revealed that the composite binder which composed by a variety of wax and polymer has high intensity, can be removed by solvent debinding process and not easy to crack and so on, the plastic injection molding simulation software can be used for qualitative analysis of injection molds, composite solvent debinding process can control precision molding dimension accurately and reduce molding defects. The powder injection molding has technological advantages and developmental potential in manufacturing clean molding products of TiC based cermets with sophisticated geometric shape.

### **H2 Self-Assemble Process in Liquid Phase**: *Junhui Xiang*<sup>1</sup>; <sup>1</sup>Graduate University of Chinese Academy of Sciences

Self-assembly of the crystal units into an organized three-dimensional architecture is an attractive, strategic goal, which can be expected promising applications in information storage, microelectronics, optics, biomedical implants, catalysis, and so on. Many organisms, including mollusks, echinoderms, calcisponges, corals, certain algae, and so on, form their hierarchical skeleton with calcium carbonate minerals in an organic-aqueous environment. Their skeleton has complex morphology over several hierarchy levels with super properties. The super property is structure-dependent as a result of accurate controlling of subunits size, shape and arrangement. During the past decades, different materials with specific size, shape, orientation, composition, and hierarchical structure have been explored by different inducing factors, including surfactants, double-hydrophilic block copolymers, biopolymers, dendrimers, electric field, magnetic field, and so on. In this research, the authors construct an organic-aqueous biphase interfaces system to mimic the living environment, and the hydroxyl-terminated SAMs is employed as an organic surface to induce the nucleation and modulate the morphology of crystals. CaCO<sub>3</sub> was crystallized by a slow gas-diffusion procedure in such a system, and the relationship between the structure and the property is investigated.

#### H3 Sintering and Nonlinear Dielectric Properties of Ba<sub>0.6</sub>Sr<sub>0.4</sub>TiO<sub>3</sub> Ceramics Prepared by Citrate Method: Xiao-Fei Zhang<sup>1</sup>; Qing Xu<sup>1</sup>; Wen Chen<sup>1</sup>; Han-Xing Liu<sup>1</sup>; <sup>1</sup>Wuhan University of Technology

Barium strontium titanate (Ba<sub>1,x</sub>Sr<sub>x</sub>TiO<sub>3</sub>, BST) has drawn considerable interest as a promising candidate material for tunable capacitive elements. BST compositions with a paraelectric state at working temperatures have been regarded to be preferred for the application. Improving the sinterability of the BST compositions is expected to offer a larger space for their application in thick film devices. In this work,  $Ba_{0.6}Sr_{0.4}TiO_3$  powder was synthesized by a citrate method. It was found that careful controlling heating rate during the calcining process allowed for producing Ba06Sr04TiO3 powder with a pure perovskite phase at 550°C. The powder showed fine and uniform morphology with mean particle size of about 100 nm. Microstructure and nonlinear dielectric properties of the ceramic specimens were investigated with respect to sintering temperature. The results indicate that the fine morphology of the powder effectively promoted the reduction of the sintering temperature of the ceramic specimens. The ceramic specimen sintered at 1260°C attained about 95% of the theoretical density. At room temperature, the ceramic specimen showed a dielectric constant of 3010 and a dielectric loss of 0.48% at 10 kHz together with a tunability of 32.4% at 10 kHz and 20 kV/cm.

## **H4 Solid-State Synthesis and Properties of Y-Doped BaCeO**<sub>3</sub>: *Ying Li*<sup>1</sup>; Yushi Ding<sup>1</sup>; Changzhen Wang<sup>1</sup>; Xiaoming Song<sup>1</sup>; <sup>1</sup>Northeastern University

In this paper, an attempt has been made to prepare the BaCe<sub>1-x</sub>Y<sub>x</sub>O<sub>3-a</sub> (x = 0.1, 0.15) solid electrolytes via a solid-state synthesis method. The raw powders of BaCO<sub>3</sub>, CeO<sub>2</sub> and Y<sub>2</sub>O<sub>3</sub> were mixed and refined by ball milling, and then pressed into tablets, followed by calcining in air. X-ray diffraction analysis indicated



that the BaCe<sub>0.9</sub>Y<sub>0.1</sub>O<sub>3-a</sub> and BaCe<sub>0.85</sub>Y<sub>0.15</sub>O<sub>3-a</sub> solid solutions were synthesized at 1250°C and 1400°C, respectively. To conduct the electrochemical impedance spectroscopy measurements, the obtained Y-doped BaCeO<sub>3</sub> tablets were grinded into fine powders, then pressed into tablets and sintered to make them compact. Results show that the sintered tablets exhibit useful protonic conduction. Furthermore, the solid electrolyte tubes were prepared from the finely grinded BaCe<sub>0.9</sub>Y<sub>0.1</sub>O<sub>3-a</sub> and BaCe<sub>0.85</sub>Y<sub>0.15</sub>O<sub>3-a</sub> powders and shaped with isostatic pressure (or hot compression casting). They were strong and dense after subsequent sintering.

## H5 Synthesis of Ba S-Phase SiAlON by Nitridation Using Ammonia Gas: *Toru Wakihara*<sup>1</sup>; Akio Ihara<sup>1</sup>; Junichi Tatami<sup>1</sup>; Katsutoshi Komeya<sup>1</sup>; Takeshi Meguro<sup>1</sup>; <sup>1</sup>Yokohama National University

SiAlON phosphors have shown promising potential because of their excellent photoluminescence property; however, it needs a high temperature firing. It is important to establish a method for the fabrication of SiAlON powders at lower temperatures, while keeping its characteristic high performance. In recent years, ammonia nitridation, which uses a mixture of  $NH_3$  and carbohydrate as the reactant gas, has been proposed as a synthetic technique for producing high-purity nitride powders. The ammonia nitridation technique is quite a simple process since it does not require mixing, milling or a decarburization process. In this study, Ba S-pahse SiAlON was synthesized by ammonia nitridation of Ba ion-exchanged zeolite and low temperature synthesis (1200°) of pure Ba S-phase SiAlON has been performed.

#### H6 Synthesis of Blue-Emitting Ce<sup>3+</sup>-Activated La-Si-Al-O-N Phosphors for White Light-Emitting Diodes: *Atsuro Yaguchi*<sup>1</sup>; Takayuki Suehiro<sup>1</sup>; Naoto Hirosaki<sup>2</sup>; Tsugio Sato<sup>1</sup>; <sup>1</sup>IMRAM Tohoku University; <sup>2</sup>Nitride Particle Group, Nano Ceramics Center, National Institute for Materials Science

Silicon nitride-based oxynitride phosphors have attracted much attention in recent years, especially in the field of solid-state lighting, i.e., white LEDs. Ce3+activated La-Si-Al-O-N host materials such as LaSi<sub>3</sub>N<sub>5</sub>, LaSi<sub>6-z</sub>Al<sub>1+z</sub>N<sub>10</sub>. <sub>2</sub>O<sub>2</sub> (JEM) exhibit efficient blue broadband emission, whereas the processing of these nitrogen-rich compositions requires the use of oxygen/moisture-sensitive rare earth nitrides and the consequent complicated synthesis procedures. To establish a simple and facile synthesis route for obtaining these phosphors, we attempted to synthesize JEM-Ca-a-SiAlON multiphase phosphors from the system CaO-La2O3-Si3N4-AlN, which can be handled under ambient atmosphere. Highly phase-pure LaSi<sub>2</sub>N<sub>5</sub> phosphors were also obtained with minor amounts of secondary phases such as JEM,  $\rm La_3Si_8O_4N_{11},\,Si_3N_4$  from the system La<sub>2</sub>O<sub>3</sub>-Si<sub>3</sub>N<sub>4</sub>-AlN. The highest emission efficiency was obtained with the composition La,  $O_3$ —3Si,  $N_4$ , and the external quantum efficiency of 42.2% under 365 nm excitation was attained. By partially substituting Ca<sup>2+</sup> for La<sup>3+</sup> in the La<sub>2</sub>O<sub>3</sub>-3Si<sub>3</sub>N<sub>4</sub> system, we succeeded in broadening of the excitation spectrum and redshifting of the emission bands for the LaSi<sub>2</sub>N<sub>5</sub> phase, with the improved quantum efficiencies and phase purity. The developed Ce3+-activated multicomponent La-Si-Al-O-N phosphors demonstrated the promising applicability as blue components for NUV-converting white LEDs possessing high color rendering properties.

#### **H7 Effect of LiNbO<sub>3</sub> on Piezoelectric Properties of K**<sub>0.5</sub>**Na**<sub>0.5</sub>**NbO<sub>3</sub> Ceramics**: *Minghe Cao*<sup>1</sup>; Zhuo Li<sup>1</sup>; Fan Li<sup>1</sup>; Hua Hao<sup>1</sup>; Hanxing Liu<sup>1</sup>; <sup>1</sup>Wuhan University of Technology

Lead-free  $(1-x)K_{0.5}Na_{0.5}NbO_3-xLiNbO_3$  piezoelectric ceramics have been prepared by a conventional solid state process. The phase structure and the electrical properties of the ceramics were studied. A polymorphic phase transition (PPT) between the orthorhombic and tetragonal phases was identified in the composition range of 0.08 < x < 0.10. The ceramics near the PPT show better piezoelectric properties. At a level of 10mol% LiNbO3, the sample sintered at 1100°C for 1h exhibits optimal piezoelectric properties,  $\rho=4.30g/$  cm3,d33~130pC/N, kp~0.40, TC~471°C, TO-T~40°C.

H8 Influence of Fiber Properties on the Performance of Piezoelectric 1-3 Composites: Wei-wei Peng<sup>1</sup>; Jing Zhou<sup>1</sup>; Lei Xu<sup>1</sup>; Wen Chen<sup>1</sup>; <sup>1</sup>State Key Laboratory of Advanced Technology for Materials Synthesis and Processing, and School of Material Science and Engineering, Wuhan University of Technology

Lead zirconate titanate (PZT)-based materials have wide range of applications

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as sensors, actuators and tranceducers. In this work, 1-3 composites, which required for different divices applications, with the (PZT)-based (PZT-5, PMnS-PZN-PZT and PSN-PZN) piezoelectric fibers produced via a VPP method have been investigated. The relationships between the dielectric and piezoelectric properties of the 1-3 composites and different ceramic fibers with various height-to-width radios have been compared. As the aspect radio raised up, the relative dielectric constant eT, the mechanical quality factor Qm and dielectric loss tand of 1-3 composites with 30% fiber loading change slightly, while the value of the piezoelectric constant d33, piezoelectric voltage constant g33 and the acoustic impedance Z increased, and the anisotropic properties kt/kp was always more than 1 and reached the extreme value around the aspect ratio of 2.5. We conclude that the properties of the 1-3 composites are well followed with the properties of the piezoelectric fibers, so the performance of the 1-3 composites can be improved for various divices by tuning the fiber properties.

#### H9 Research on Microstructure and Thermal Conductivity of Al-Intercalated Vermiculite-Forsterite Refractories: Chunfeng Wang<sup>1</sup>; Huazhi Gu<sup>1</sup>; Hongxi Zhu<sup>1</sup>; *Shan Ge*<sup>1</sup>; <sup>1</sup>Wuhan University of Science and Technology

Expanded vermiculite is an important thermal insulation material with layered micro-porous structure. In this paper using natural vermiculite as raw materials, heat-treated for activation, Al-pillared vermiculites are prepared using polyhydroxyl aluminium as intercalating agent through sol-gel method. Then the Al-pillared vermiculites are mixed with forsterite, and Al-pillared vermiculite-forsterite refractories are obtained. The phase and microstructure are analyzed by means of XRD, SEM, TG, et al. The results reveal that the basal spacing of vermiculite expanded by intercalation of Al-species, and the interlayer of vermiculite inlaid with a large amount of nanometer Al-species particles. The Al-pillared vermiculites. Firstly, the unusual slice layer of vermiculite can reflect the flow of heat; secondly, the pore space and pore volume in the interlayer of Al-pillared vermiculite become smaller, and convective heat transfer is blocked, so the thermal insulation property of vermiculite-forsterite refractories made from Al-pillared vermiculites is further reinforced.

### H10 Investigation of Micro-Defects in Glass and Damage Evolution: Yan Qiu<sup>1</sup>; <sup>1</sup>China Building Materials Academy

The strength and the lifetime of the glass components strongly depend on the defect size and location. However, the initiation and evolution of a micro-crack in the glass as well as its effect on the failure and lifetime is not very clear. In this work, spherical indentation and Vickers indents were induced on the glass surface in air and in water environments respectively, and the indentation damage process was recorded by camera in situ during the indentation loading and unloading. The relationship between load the contact area and the indentation crack size was studied to understand the influence of surface defects. And the evolution of the inner micro-crack induce by laser was also investigated to compare the difference from the surface defect and inner defect. The critical crack size for glass plate under uniform bend load was determined to estimate the lifetime The experiments show that, comparing to the Vickers crack, the cone crack from the spherical indentation exhibits different damage evolution and propagation direction. The residual strength of the damaged glass specimens was measured. It is concluded that the micro-defects on the glass surface governs the fracture strength of the glass and strongly influenced by the environment.

#### H11 Characteristics of Grain Growth in Superplastically Deformed 3Y-TZP: *Ha-Guk Jeong*<sup>1</sup>; Kenji Higashi<sup>2</sup>; <sup>1</sup>Korea Institute of Industrial Technology; <sup>2</sup>University of Osaka Prefecture

Several studies have noted that the grain growth of polycrystalline materials during superplastic deformation is one of intrinsic characteristics. Strain-enhanced grain growth, namely, dynamic grain growth, also, is a widespread property of superplastic deformation. To date, however, enough microstructural studies have not been dedicated in superplastic ceramic materials, in special in Y-TZP. In addition, fundamental deformation mechanisms for superplasticity in ceramics, in particular under a condition of tension, have been reported so far. The present work has therefore paid attention to the microstructure including strain-enhanced grain growth in 3Y-TZP deformed superplastically. In the present material, grain growth occurred during tensile deformation, and grain growth rate did vary with strain rates in fairly grained 3Y-TZP. Furthermore, a variation in grain growth rate along the gauge length of the deformed specimen was also observed. Fracture surface of the specimen, which shows relatively large elongation, exhibits the evidence of ductile-like fracture containing a remarkable protrusion of grains.

#### H12 Characterization and Energy Storage Density of BaTiO<sub>3</sub> - Ba(Mg<sub>1/3</sub>Nb<sub>2/3</sub>)O<sub>3</sub> Ceramics: Yiqiu Li<sup>1</sup>; Hanxing Liu<sup>1</sup>; Zhonghua Yao<sup>1</sup>; Jing Xu<sup>1</sup>; Yunjiang Cui<sup>1</sup>; Yan Liu<sup>1</sup>; Hua Hao<sup>1</sup>; Minghe Cao<sup>1</sup>; Zhiyong Yu<sup>1</sup>; <sup>1</sup>Wuhan University of Technology

The energy storage density of (1-x) BaTiO<sub>3</sub> – x Ba(Mg<sub>1/3</sub>Nb<sub>2/3</sub>)O<sub>3</sub> (x = 0, 0.1, 0.2, 0.3) ceramics was investigated. The crystalline phases and microstructure of samples were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM), respectively. The energy storage density was calculated from the P-E hysteresis loops measured at room temperature. Experimental results show that the energy storage density of 0.9 BaTiO<sub>3</sub> – 0.1 Ba(Mg<sub>1/3</sub>Nb<sub>2/3</sub>)O<sub>3</sub> ceramics is highest among all compositions. At 16 kV mm-1 electric field, the energy storage density of 1.6 compared with pure BaTiO<sub>3</sub>. The improvement of the energy density can be due to two factors: one is that the breakdown strength was improved due to the decrease of the grain size in 0.9 BaTiO<sub>3</sub> – 0.1 Ba(Mg<sub>1/3</sub>Nb<sub>2/3</sub>)O<sub>3</sub> ceramics, the other is that the remnant polarization of the ceramics was decreased. This results indicates that 0.9 BaTiO<sub>3</sub> – 0.1 Ba(Mg<sub>1/3</sub>Nb<sub>2/3</sub>)O<sub>3</sub> ceramics have advantages



compared with pure BaTiO<sub>3</sub> for energy storage applications, and with further improvements in microstructure and reduction of sintering temperature, could be a good candidate for energy storage capacitors.

#### H13 Microstructure and Strength of 3%Yttria-Stabilized Zirconia Fabricated by Two Step Sintering Process: Jai-Won Byeon<sup>1</sup>; *Yong In Kim*<sup>1</sup>; Si-Hwa Sung<sup>1</sup>; C.Y. Hyun<sup>1</sup>; <sup>1</sup>Seoul National University of Technology

3%yttria-stabilized zirconia (3YSZ) ceramic has been developed for structural applications such as cutting tools and medical parts due to its high fracture toughness and high strength. Further improvement of strength can be made by optimizing sintering process for inhibiting grain growth. In this study, in order to obtain a fully sintered body with very fine grain and high strength, two-step sintering (TSS) process was applied. Two types of commercial 3YSZ powder with mean size of about 20 micrometer and 20 nanometer were used for preparation of green body. TSS consists of first sintering at a low temperature and subsequent second sintering at a high temperature. Microstructural evolutions including relative density, shrinkage rate, grain size, phase fraction were investigated in a wide range of temperature (i.e., 1100°C~1450°C) and time (i.e., 0.5~10 hour). Fully sintered body with a grain size of about 200nm was obtained through TSS. Flextural strength determined by four point bending test was discussed in relation with the microstructure. "This work is financially supported by Korea of Environment (MOE) as ET-Human Resource Development Project".

#### H14 Fabrication of Eco-Friendly Porous Alumina Ceramics with Unidirectional Pores by Freeze-Drying: Weijiang Xue<sup>1</sup>; Yang Sun<sup>2</sup>; Yong Huang<sup>1</sup>; Chang-An Wang<sup>1</sup>; Jinlong Yang<sup>1</sup>; Zhipeng Xie<sup>1</sup>; <sup>1</sup>Tsinghua University; <sup>2</sup>University of Science and Technology Beijing

For the slurry system containing sodium alginate, alumina powder, the influence of different amount of dispersants on slurry viscosity was investigated. When adding the dispersant TAC by 5 percent of the alumina power mass, the viscosity of the slurry is lowest. Porous alumina ceramics with unidirectional pores, some strength and good permeability were prepared by water freezing in the slurry unidirectionally and freeze-drying leading to formations of pores due to ice sublimation and then sintering. The researches showed that green bodies could gain strength and the pore structures could also be preserved by adding sodium alginate to the raw material. The compressive strength of the porous alumina ceramic with 66.7% porosity reached 16.03MPa. Influences of solid loading and sintering temperature on porosity, pore size distribution, compressive strength and permeability were also researched.

#### H15 Preparation and Luminescent Properties of Na<sub>5</sub>Eu(MoO<sub>4</sub>)<sub>4-x</sub>(PO<sub>4</sub>)<sub>x</sub> Red Phosphors for White Light-Emitting Diodes Application: Cuisheng Xi<sup>1</sup>; Shikao Shi<sup>1</sup>; Huili Gong<sup>1</sup>; Ji Zhou<sup>2</sup>; <sup>1</sup>Hebei Normal University; <sup>2</sup>Tsinghua University

White light-emitting diodes (LEDs), as the promising solid-state lighting sources to replace conventional incandescent and fluorescent lamps, have attracted much attention due to their high reliability, long lifetime, low energy consumption and environment-friendly characteristics. The common process to achieve white light uses combination of the red/green/blue tricolor phosphors with a GaN/InGaN chip. However, the red phosphor Y2O2S:Eu3+ is chemically unstable and not desirable in luminescence efficiency, as compared with blue (BaMgAl<sub>10</sub>O<sub>17</sub>:Eu<sup>2+</sup>) and green (ZnS:Cu<sup>+</sup>, Al<sup>3+</sup>) phosphors. Hence, the search for new red phosphors that can be efficiently excited around 400 nm is urgent. In this work, a series of new red phosphor,  $Na_sEu(MoO_4)_{4,x}(PO_4)_x$  (x<0.10), were prepared by high-temperature solid-state reaction, and their crystal structure and photoluminescence properties were investigated. The powder X-ray diffraction patterns of the samples show that the phosphors are of single phase and consistent with JCPDS 72-2158  $[\mathrm{Na}_{5}\mathrm{La}(\mathrm{MoO}_{4})_{4}],$  and the doped  $\mathrm{PO}_{4}3\text{-}$  ion has little influence on the host structure. With the introduction of PO<sub>4</sub><sup>3-</sup> ions, the excitation and emission intensities of these phosphors are both enhanced. In particular, the phosphor Na<sub>5</sub>Eu(MoO<sub>4</sub>)<sub>3.96</sub>(PO<sub>4</sub>)<sub>0.04</sub> exhibits the optimum red emission under 395 nm light excitation. The novel phosphor system may be a potential candidate as red components for white LEDs.

## H16 Formation of AlN Polycrystals and Nanotubes in NH<sub>3</sub> with Bi: *Haruhiko Morito*<sup>1</sup>; Tomoyuki Ide<sup>1</sup>; Taiki Karahashi<sup>1</sup>; Hisanori Yamane<sup>1</sup>; <sup>1</sup>Tohoku University

A bulk layer and nanotube of AlN were prepared by heating Al and Bi chunks in  $NH_3$  flow. It was found that Bi additives enhanced the reaction of Al and  $NH_3$ . A bulk layer of AlN polycrystals was synthesized on a BN crucible surface by heating Al chunks with 5 or 16 mol% of Bi at 1273 K for 3 h in  $NH_3$  gas flow. A large quantity of nanotubes about 20-100 nm thick were formed on the sample surface.

#### H17 Standardization of Silicon Nitride Materials for Rolling Bearing Balls: Katsutoshi Komeya<sup>1</sup>; <sup>1</sup>Yokohama National University

The first practical applications of Si<sub>3</sub>N<sub>4</sub> bearing balls have been achieved in 1983. ASTM standard F2094 "Standard specification for silicon nitride bearing balls" was established in 2001 to standardize bearing balls. ISO standards were independently proposed at ISO/TC4 and ISO/TC206 in 2004. In the 12th Plenary Meeting TC206 in 2005, NWIP "Silicon nitride materials for rolling bearing balls" was adapted and TC206/WG36 (convenor:Katsutoshi Komeya) was established. This theme was then eagerly discussed for two and half years in the WG. On March of 2008, the convenor has completed the final document for the standardization to the DIS stage and submitted it to the Secretariat, ISO/ TC206. Finally ISO standard, ISO 26602 "Silicon nitride materials for rolling bearing balls" was established in 2009. This international standard specifies the requirements for pre-processed Si<sub>2</sub>N<sub>4</sub> materials for rolling bearing balls and provides a classification defining physical and mechanical properties of Si<sub>3</sub>N<sub>4</sub> preprocessed bearing ball materials. The materials are classified in three categories by specifying characteristics and microstructures. Methods for sample preparation and microstructure observation are provided in the Annex. The presentation will introduce Si<sub>3</sub>N<sub>4</sub> ceramics for bearing applications, the discussion process for the standard draft and the content of the final document.

### H18 Preparation of Magnesium Carbonate Whisker by Using Magnesite Tailings: Nan Wang<sup>1</sup>; <sup>1</sup>Northeastern University

Magnesium carbonate whisker was prepared by thermal decomposition of  $Mg(HCO_3)_2$  solution that was prepared by hydration and carbonation treatment of light burnt magnesia derived from magnesite tailings, and the influence of thermal decomposition conditions on morphology of magnesium carbonate crystal was investigated. The results showed that thermal decomposition products was  $MgCO_2 \cdot 3H_2O$ , and its morphology was appreciably influenced by the additives added to  $Mg(HCO_3)_2$  solution. When potassium dihydrogen phosphate was added, petal shaped magnesium carbonate crystal was obtained, and spherical magnesium carbonate one was obtained when ammonium carbonate was added. Magnesium carbonate whisker was successfully prepared when a kind of soluble magnesium salt was added, and magnesium carbonate whisker with length of 20 to 60µm and aspect ratio of 10~20 were obtained when the thermal decomposition temperature was 50°C with stirring intensity of 200 rpm.

#### Poster Session: Symposium I: Biomaterials, Smart Materials and Structures

Tuesday PM	Room: Hall 2
August 3, 2010	Location: Cairns Convention Centre

11 Biodegradable Plastic-An Alternative to Commonly Used Plastic in Automotive Applications: *Mohammad Mazumder*<sup>1</sup>; Igor Sbarski<sup>1</sup>; <sup>1</sup>Swinburne University

Worldwide automakers are constantly looking for economic, fuel efficient and light weight vehicles to meet the customer demand. This, inturn pushing the automakers to look for materials that are alternative to stainless steel and having the strength and toughness for automotive applications. Plastic is one such material which meets the properties required by automotive industries. The uses of plastic in automotive has been increased dramatically in recent years. Though uses of plastic added a new dimension to automotives however, it is having negative impact in the environment due to difficulties in recycling from the automotive shredder residue. Each year few million tons of automotive plastic parts are dumped into the landfills causing soil erosion and environmental pollution. Globally, automakers are facing immense pressure to minimize these environmental impacts. The purpose of this research is to provide a possible solution to minimize these environmental effects by using biodegradable plastic. The compatibility of biodegradable plastic in comparison with commonly used automotive plastics has been presented in this paper

### **12** Change in Biological Apatite Orientation in Beagle Mandible: *Wataru Fujitani*<sup>1</sup>; Takayoshi Nakano<sup>1</sup>; <sup>1</sup>Osaka University

Preferred orientation of biological apatite (BAp) c-axis in bone is one of the most important factors controlling the bone function including the mechanical function. Thus, it is important to understand the mechanism for the formation and change in the BAp orientation of mammalian mandible in the dental field in the case of a clinical diagnosis and the application of dental implants. In this study, we focused on the effects of an applied stress distribution on the BAp orientation as well as bone density in beagle mandible. For change in the stress distribution on the mandible, all the teeth of one sided mandible were extracted



to remove a biting stress. The preferred BAp c-axis orientation was analyzed by a microbeam X-ray diffractometer. Mandible generally exhibits one-dimensional BAp orientation along mesiodistal axis. In contrast, just under the teeth root, BAp was oriented along the applied biting stress, especially, in the teeth for mastication. However, this BAp orientation along the biting direction decreased after removing the biting stress. It was concluded that the BAp orientation is sensitively dominated depending on the in vivo local stress distribution in beagle mandible.

#### **I3 Effect of Magnetic Field on the Electrocrystallization of Silver Incorporated Hydroxyapatite**: Tapash Rautray<sup>1</sup>; Ramaswami Narayanan<sup>1</sup>; Tae Kwon<sup>1</sup>; *Kyo Kim*<sup>1</sup>; <sup>1</sup>Kyungpook National University

An electrochemical method of producing nanocrystalline hydroxyapatite coatings on titanium surface is reported. The bath contained CaCl, and K, HPO, in the molar ratio 1.67:1. The electrolyte was maintained at physiological pH and was agitated with a magnetic stirrer throughout the time of electrolysis. Coatings were deposited for 30 minutes at 20, 35, 50, 65 and 80 mA/cm<sup>2</sup> under a magnetic field of 2 tesla. Small globules of hydroxyapatite covered the coating surface completely as evidenced by X-ray diffraction. The thickness of hydroxyapatite formed under N-N magnetic field condition were suppressed as compared to the respective current densities of hydroxyapatite formed without magnetic field. This shows that N-N magnetic field retards the formation of HA, whereas N-S magnetic field favours hydroxyapatite formation especially under 50 mA/cm<sup>2</sup> and 65 mA/cm<sup>2</sup> current densities. From the SEM micrographs, it is evident that, branched cracks are formed under N-N magnetic field condition whereas no cracks are visible under N-S and no-field conditions. As evidenced from XRD, thick HA deposits are formed at 50 mA/cm<sup>2</sup> and 65 mA/cm<sup>2</sup> under NS magnetic field whereas with the same current densities in N-N magnetic field, the crack patterns are more severe.

## 14 Multiscale Bone Remodeling Simulation of Implant-supported Fixed Dental Prosthesis: Chaiy Rungsiyakull<sup>1</sup>; Qing Li<sup>1</sup>; <sup>1</sup>The University of Sydney

Osseointegrated dental implants are accepted as clinically desirable and have predictable outcomes for the management of partially and fully edentulous patients. A better osseo-integration is believed that can improve adaptive bone remodelling and minimize healing time. In this regard, fully porous coated (FPC) implant has been making significant success in implantation in-vivo over the last decade and is believed that these coating create a better osseointegrating environment. However, it still remains unclear how the implant biomaterials and corresponding surface morphologies would affect the bone remodelling activities. This paper aims at providing a preliminary understanding in biomechanics with respect to the effect of implant supported cantilever partial denture on bone remodelling. 2D macro- and micro-scale finite element models are created for a typical dental implantation setting. Under a certain mastication loading (<200N), global responses from macro-scale models (single crown and 2-unit cantilever bridge) are first obtained and then they are transferred to micro-scale models (with coated surface morphology details) for micro-scale analysis. A strain energy density (SED) obtained from 2D micro-scale finite element analysis is used as a mechanical stimuli to determine the bone remodeling in term of the change of apparent bone density over a period of 48 months.

#### **15** The Dynamic Performance of Magnetostrictive Actuators for Heavy Load: *Zhang Tianli*<sup>1</sup>; Mao Jianqin<sup>1</sup>; Jiang Chengbao<sup>1</sup>; Li Lin<sup>1</sup>; Xu Huibin<sup>1</sup>; Zhang Heng<sup>1</sup>; <sup>1</sup>Beihang University

Based on the demand of optics system, heavy load platform and mini-type and integration system, a kind of giant magnetostrictive actuator for heavy load is designed. It owes wide working range and the low losses in the coil to the accurate lay out of the entire magnetic circuit on the basis of the finite element (FEM) calculations, and the robust construction predestines the actuator for purposes such as fine positioning and active vibration control as well as oscillatory excitation of heavy structures. Dynamical excitations of the actuators have been investigated under various frequences and loads conditions. The effects of heavy load to the dynamic performance of magnetostrictive actuator are analysed.

**I6** Simultaneous Incorporation of 5-Fluorouracil and Leucovorin into Chitosan Nanoparticle as Drug Carrier and Its Characterization: *Puwang Li*<sup>1</sup>; Lingxue Kong<sup>1</sup>; Zheng Peng<sup>2</sup>; Yichao Wang<sup>1</sup>; <sup>1</sup>Centre for Materials and Fibre Innovation; <sup>2</sup>Agricultural Product Processing Research Institute, Chinese Academy of Tropical Agricultural Sciences

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Combined drug loaded systems offers many advantages over traditional single drug loaded delivery systems due to the multiple and improved therapy efficiency. Therefore, a combined drug loaded system containing 5-fluorouracil (5-FU) and leucovorin (LV) was designed and prepared by ion gelation method in this work. The interaction between polymer and drugs was investigated by Fourier transform spectroscopy (FTIR), the morphology of the drug free and drug loaded nanoparticles was investigated by transmission electron microscopy (TEM), the encapsulation efficiency and the loading capacity as well as the in

vitro release profile in simulated gastrointestinal fluid were evaluated. FTIR results confirm the presence of strong interactions between polymer and drugs, and the XRD results demonstrate that both drugs distribute in the nanoparticles matrix in amorphous phase. The encapsulation efficiency and loading capacity of both drugs are affected by the initial concentration of the drugs. The initial concentration of the drugs also poses great effect on the release profile of the combined drug loaded nanoparticles. In vitro release confirms that the combined drug loaded nanoparticles could provide a constant and continue release. All these pronounced nature prove that the combined drugs loaded nanoparticles is a promising vehicle for the chemotherapy of colorectal cancer.

### 17 Martensitic Transformation and Shape Memory Effect of Ti-Zr-Nb Alloys: Yan Li<sup>1</sup>; <sup>1</sup>Beihang University

 $Ti_{50}Zr_{50-x}Nb_x(x=1~10, at.\%)$  alloys have been investigated as shape memory alloys, with regard to the microstructure, martensitic transformation and shape memory effect (SME). The results show that with the increasing of Nb content, the phase composition of Ti-Zr-Nb alloy transforms from  $\alpha$ ' to  $\langle \alpha \rangle$ ", and only  $\alpha$  phase appears when Nb content is more than 8%. The martensitic transformation temperature is in the range of 350~500°C. A series of pre-strain from 8% to 14% are applied to the Ti-Zr-Nb alloys by compression, and the lowest yield stress occurs in the  $Ti_{50}Zr_{42}Nb_6$  alloy, while the maximum recovery strain of 2.9% is obtained in  $Ti_{50}Zr_{42}Nb_8$  alloy. The Ti-Zr-Nb alloys show promising as high-temperature SMAs for applications such as in aerospace and automotive areas.

#### **18 Mechanical Properties of Ti-48at%Ni Shape Memory Alloy Melt-Spun Ribbon**: *Hongyan Xing*<sup>1</sup>; Shuichi Miyazaki<sup>1</sup>; <sup>1</sup>The College of Mechanical Engineering, Tianjin University of Science and Technology

In this study, the mechanical properties, such as shape memory behavior and fracture stress in the Ti-48at%Ni melt-spun ribbons were investigated. Tensile tests of as-spun ribbons were carried out at room temperature. The elongation and the yield stress increased with increasing rotation speed less than 4000rpm. The maximum shape recovery strain shows a value of 5.4% under the applied stress of 500Mpa.

**19 Microstructure and Martensitic Transformation Behavior of CoNiGa High-Temperature Shape Memory Alloy**: *Yan Xin*<sup>1</sup>; Yan Li<sup>2</sup>; Liang Chai<sup>2</sup>; <sup>1</sup>North China Electric Power University; <sup>2</sup>Beihang University

Microstructures and martensitic transformation behavior of  $Co_{48}Ni_{24+x}Ga_{28-x}$ (x=0, 2, 4, 6) high-temperature shape memory alloys have been investigated. The results show that all the alloys are composed of a non-modulated tetragonal martensite and a face-centered cubic (FCC)  $\gamma$  phase at room temperature, which have been identified by optical observation, X-ray diffraction (XRD) and selected-area electron diffraction (SAED) measurements. The volume fraction of the  $\gamma$  phase increases from 12.5% to 75.8% with increasing Ni content, but the crystal lattices of it almost keep invariable. The Co-Ni-Ga alloys exhibit a thermoelastic martensitic transformation (MT) between the cubic L2<sub>1</sub> parent phase and the tetragonal martensite phase. The martensitic transformation temperatures(Ms) of dual-phase  $Co_{48}Ni_{24+x}}Ga_{28-x}$  alloys increase from 110.2°C for x=0 to 272.3°C for x=4, which is contributed to the enlargement of the electron concentration (e/a) and tetragonality of martensite phase, induced by the increasing of Ni content. No martensitic transformation can be observed in  $Co_{48}Ni_{30}Ga_{22}$  because of the overmuch  $\gamma$  phase.

**I10 New SMA Actuator with High Energy Efficiency and Quick Response:** *Kwang Jee*<sup>1</sup>; Jun Han<sup>1</sup>; Woo Jang<sup>2</sup>; <sup>1</sup>Korea Institute of Science and Technology; <sup>2</sup>Chosun University

The main drawbacks of SMA (shape memory alloys) actuators are a slow response and a waste of electric power. The power should be continuously supplied for SMA elements to remain austenite state until the elements begin to cool. The problems are more serious when batteries and thick SMA elements are used. We develop a new SMA actuator which consumes little energy and responds very quickly. The actuator consists of a specially designed bias spring and two SMA elements which exert the force in opposite direction to each other. The bias spring, unlike a common spring which has one stable position, has two stable positions. A SMA element (1), for instance, contracts on heating by electricity, the actuator takes one stable configuration. No further power supply is necessary to maintain the stable position, providing enough time for the SMA element (1) to cool. When the other SMA element (2) is heated, it contracts with enough force to move the bias spring and to expand the already-cooled SMA element (1). Power supply stops immediately after the actuator takes the other stable configuration, causing power saving and quick response.

**I11 Self-Accommodation Morphology in Ti-Nb-Al Shape Memory Alloy**: *Tomonari Inamura*<sup>1</sup>; Hideki Hosoda<sup>1</sup>; Hee Young Kim<sup>2</sup>; Shuichi Miyazaki<sup>2</sup>; <sup>1</sup>Tokyo Institute of Technology; <sup>2</sup>University of Tsukuba

The self-accommodation microstructure of a  $\beta$ -titanium shape memory alloy was investigated by a new method of crystallographic analysis and transmission



electron microscopy (TEM) observation. In the crystallographic analysis, the deviations from the exact twin-orientation and the kinematical compatibility condition were quantitatively evaluated among 132 pairs of the habit plane variants (HVs) to predict the preferential pair of HVs in the self-accommodation structure. The analysis showed that there is no pair of HVs which has exact twin-orientation without breaking the kinematical compatibility condition and the invariant plane condition. The deviation angle from the exact twin-orientation and the kinematical compatibility condition was minimized in the pair of HVs that has {111}typeI or <211>typeII twin as a boundary between the HVs. TEM observation showed that the self-accommodation microstructure in the alloy is consisted of these pairs of HVs. The validity of the new method to predict the preferential pair of HVs was confirmed together with the self-accommodation microstructure of the Ti-Nb based new shape memory alloy.

### **112 Shape Memory Effect in Fe-Pd Magnetic Shape Memory Alloy Thin Films**: *Junhyun Han*<sup>1</sup>; Hyun-Tae Ahn<sup>2</sup>; Kwang-Koo Jee<sup>1</sup>; Tae-Hyun Kim; <sup>1</sup>Korea Institute of Science and Technology (KIST); <sup>2</sup>Korea University

Ferromagnetic shape memory alloys have received much attention recently because large strain and high frequency can be obtained by the application of an external magnetic field. Such a magnetic shape memory effect (MSME) is achieved by conversion of martensite variants through twin boundary motion so that the new crystal orientation allows the magnetic moment to be better aligned with the external magnetic field. To date, MSME has already been demonstrated in the Ni-Mn-Ga systems, Fe-Pt systems, and Fe-Pd system in bulk form. In this work, Fe-Pd thin films with a variation in Pd content are obtained by magnetron sputtering. The shape memory effect (SME) and MSME are demonstrated in films by using the substrate curvature method.

#### **I13 The Dependence of the Yield Behavior of Martensite on the Transformation Temperature of NiTi Based Shape Memory Alloys**: Mingyuan Jiang<sup>1</sup>; *Xinqing Zhao*<sup>1</sup>; <sup>1</sup>Beihang University

The relationship between the self-orientation yield and the thermoelastic martesitic transformation in NiTi based shape memory alloys has been a key issue of the academic research and the application of shape memory alloys. In recent experimental research, it was found that the self-orientation yield behavior is closely related to the transformation temperature. The same alloy can exhibit different self-orientation behavior, if the alloy keeps different original states even at the same temperature. In the present study, the present authors prepared NiTi based shape memory alloys with different transformation temperatures and discussed the relationship between the self-orientation yield and the thermoelastic martesitic transformation in NiTi based shape memory alloys, on the basis of the preparation of NiTi based shape memory alloys with single phase microstructure. By the present research, it is attempt to clarify the mechanism for the dependence of martensite self-orientation on the transformation temperature and on the mechanical behavior of shape memory alloys.

#### **I14 A Study of a Retention of Antimicrobial Activity by Plasma Polymerized Terpinen-4-ol Thin Films**: Kateryna Bazaka<sup>1</sup>; *Mohan Jacob*<sup>1</sup>; Elena P. Ivanova<sup>2</sup>; <sup>1</sup>James Cook University; <sup>2</sup>Swinburne University of Technology

Organic polymers deposited by means of luminous chemical vapour deposition offer many advantages over inorganic materials for applications in electronic and biomedical devices, including simple manufacturing, flexibility, and low cost. Non-equilibrium plasma conditions allow for fabrication of chemically functionalized polymer thin films from a variety of organic precursors that may not undergo polymerization using conventional methods. Furthermore, such conditions permit the retention of some of the original functionalities and structure of the monomer while taking advantage of the intrinsic properties of the substrate. Resultant films are characterized by spatial uniformity, good substrate adhesion and smooth, defect-free and uniform surfaces. Terpinen-4-ol is the main constituent of Melaleuca alternifolia essential oil known for its biocidal and antiinflammatory properties. This study investigates the possibility of fabricating polymer thin films from Terpinen-4-ol by means of RF plasma polymerisation for the prevention of the growth of Pseudomonas aeruginosa and comparing the properties of the resultant films against their biologically active precursor. Because the degree of monomer fragmentation is strongly dependent on the magnitude of the excitation signal, biocidal properties of the polymer films are studied as a function of deposition power. Films are characterized using Atomic, Scanning Electron and Confocal microscopies.

### **I15 Effect of Electropolishing Process on Corrosion Resistance in Co-Cr Alloy:** Je Min Park<sup>1</sup>; *Kwang Koo Jee*<sup>1</sup>; Yoon Bae Kim<sup>1</sup>; Wan Cheol Kim<sup>2</sup>; Sang Ho Kim<sup>3</sup>; <sup>1</sup>KIST; <sup>2</sup>Hongik University; <sup>3</sup>M.I.TECH Inc.

The Co-Cr alloys have clinical histories in dental and orthopaedic implants, and recently in cardiovascular stent applications because the alloys exhibit a high elastic modulus and radiopacity. In order to improve mechanical and corrosion resistance of the alloys, electropolishing is employed as the final process. Electropolishing, an anodic dissolution process in the transpassive state, is sensitively affected by process conditions such as current density, machining time, electrode gap. In this study, effect of the electropolishing conditions on surface roughness and corrosion resistance is investigated in Co-Cr alloys (L605). The most smooth surface is obtained when electropolishing is performed at 15-20V for 15-30 sec with a electrode gap of 3.5mm. It is found out that electropolishing reduces corrosion rate about one-tenth as much.

#### **I16 Effect of Nitrogen Addition on Mechanical Property of Ti-Cr-Sn Alloy**: *Yuichi Nakahira*<sup>1</sup>; Tomonari Inamura<sup>1</sup>; Hideki Hosoda<sup>1</sup>; Shuichi Miyazaki<sup>1</sup>; <sup>1</sup>Tokyo Institute of Technology, Japan

Effect of nitrogen (N) addition on mechanical properties of Ti-Cr-Sn alloy was investigated where N atoms are expected to occupy interstitial sites. The Ti-Cr-Sn alloy is a new Ni-free Ti-based biomedical shape memory alloy which can be applicable for medical applications. However, the alloys exhibit imperfect shape recovery due to the low critical stress for slip deformation. Therefore, the improvement of strength is desirable and in this work hardening by interstitial N was focused. Ti-7mol%Cr-3mol%Sn was selected and less than 2mol% of N were systematically added. The alloys were made by Ar-1%H2 arc melting method using high purity materials, homogenized, cold rolled and solution treated at 1273K for 1.8ks. The alloys were characterized by optical microscopy, 0-20 X-ray diffraction analysis, and tensile tests at room temperature. It was found by XRD that the alloys were basically  $\beta$  (bcc) phase, and other peaks which must come from precipitates were also confirmed for some N-added alloys. The grain size was not largely affected by N addition. Tensile tests revealed that N addition improves strength but the elongation was not severely degraded. Therefore, N addition is concluded to enhance the mechanical properties of Ti-Cr-Sn alloys

### **117 Effect of Temperature and Strain Rate on Microstucture of Dynamically Recrystallized Ni<sub>45</sub>Co<sub>5</sub>Mn<sub>367</sub>In<sub>133</sub> Alloy:** *Gang Wang*<sup>1</sup>; Wenru Zhao<sup>1</sup>; Yandong Liu<sup>1</sup>; Chunyan Wang<sup>1</sup>; Yandong Wang<sup>1</sup>; Liang Zuo<sup>1</sup>; <sup>1</sup>Northeastern University

In recent years, there has been an increasing interest in ferromagnetic shape memory alloys (FSMAs) due to their unique ability to produce very large output strains and rapid response frequency. NiMnCoIn is a new-type FSMAs in which a reversible magnetic-field-induced phase transformation was observed. The microstructural evolution in the process of dynamic recrystallization in polycrystalline  $Ni_{45}Co_5Mn_{36,7}In_{13,3}$  was studied in the present paper. The experimental results showed that the high deformation temperature and slow strain rate were necessary to achieve perfect dynamic-recrystallizing microstructure in  $Ni_{45}Co_5Mn_{36,7}In_{13,3}$  alloy. Precipitates with two sizes were observed. The content of Co was higher than the matrix alloy, while the content of In was lower than the matrix alloy.

#### I18 Phase and Morphology of Carbides in ASTM F75 Co-Cr-Mo-C Alloys Formed at 1473 to 1623 K: *Shingo Mineta*<sup>1</sup>; Alfi Rano<sup>1</sup>; Shigenobu Namba<sup>2</sup>; Takashi Yoneda<sup>3</sup>; Kyosuke Ueda<sup>1</sup>; Takayuki Narushima<sup>1</sup>; <sup>1</sup>Tohoku University; <sup>2</sup>Kobe Steel Ltd.; <sup>3</sup>Yoneda Advanced Casting Co., Ltd.

The phase and morphology of carbides in as-cast and heat-treated ASTM F75 Co-Cr-Mo alloys with various carbon contents were investigated. The chemical compositions of specimens used in this study were Co-28Cr-6Mo-xC (x = 0.12, 0.15, 0.25, and 0.35mass%). The as-cast alloys were solution-treated at 1473 to 1623 K for 0 to 43.2 ks. After solution treatment, complete precipitate dissolution occurred in all four alloys. Under incomplete precipitate dissolution conditions. the phase and morphology of precipitates depended on the heat treatment conditions and the carbon content in the alloys. An intermetallic  $\sigma$ -phase and an M., C. type carbide were detected in the Co-28Cr-6Mo-0.12C alloy. In the alloys with carbon contents of 0.15, 0.25, and 0.35mass%, an M23C6 type carbide and a  $\pi$ -phase (M<sub>2</sub>T<sub>3</sub>X type carbide with  $\beta$ -Mn structure) were observed. The  $\pi$ -phase was detected at high temperatures such as 1548, 1573, 1598 and 1623 K. The amounts of  $\pi$ -phase was increased with increasing solution treatment temperature and decreased with holding time. At the high temperatures, the shape of carbides was distinctive starlike. TEM analysis revealed that starlike precipitate with stripe pattern consisted of the  $M_{\gamma_3}C_6$  carbide and  $\gamma$ -phase, and starlike-dense precipitate was the  $\pi$ -phase.

## **I19 Phase Constitution of Ti-Cr-Au and Ti-Cr-Au-Zr Alloy Systems**: *Yuri Shinohara*<sup>1</sup>; Takuya Ishigaki<sup>1</sup>; Tomonari Inamura<sup>1</sup>; Hideki Hosoda<sup>1</sup>; Shuichi Miyazaki<sup>2</sup>; <sup>1</sup>Tokyo Institute of Technology; <sup>2</sup>University of Tsukuba

In order to develop superior biomedical  $\beta$  Ti alloys with good biocompatibility, corrosion resistance and mechanical properties, the Ti-Cr-Au ternary and Ti-Cr-Au-Zr quaternary systems were selected and the phase constitution was investigated by using  $\theta$ -2 $\theta$  X-ray diffraction analysis and optical microscopy. The composition range was 3-10mol%Cr, 0-8mol%Au, and 0-42mol%Zr. The alloys were fabricated by arc melting method in Ar-1%H<sub>2</sub> using high purity elemental materials, solution-treated, cold-rolled and solution treated at 1273K for 1.8ks in vacuum followed by quenching into water. Mechanical property was estimated by micro Vickers hardness tests at room temperature. It was found that, in the Ti-Au-Cr ternary system,  $\beta$  phase was stabilized by addition of Cr, and that



A15 (WO<sub>3</sub>-type) intermetallic phase Ti<sub>3</sub>Au was formed when the Au content was 6mol% or higher. Since Ti<sub>3</sub>Au phase is hard and brittle, the hardness of the alloys became high due to Ti<sub>3</sub>Au precipitates. On the other hand, in the Ti-Cr-Au-Zr quaternary system, Zr addition was found to stabilize  $\beta$  phase. The obtained microstructures were sufficiently corresponded to the results by XRD.

# **I20** Phase Transformations in **B2** Phase of Co-Rich Co-Al Binary Alloys: *Kodai Niitsu*<sup>1</sup>; Toshihiro Omori<sup>1</sup>; Makoto Nagasako<sup>1</sup>; Katsunari Oikawa<sup>1</sup>; Ryosuke Kainuma<sup>1</sup>; Kiyohito Ishida<sup>1</sup>; <sup>1</sup>Department of Materials Science, Graduate School of Engineering, Tohoku University

In the  $\gamma(A1) + \beta(B2)$  two phases region of Co-Al binary system, metastable  $\gamma'(L1_2)$  and A2 phases have been reported. Phase transformation behaviours of these phases, however, have not been well investigated. In present study, the phase transformations appearing in the  $\beta$  phase of Co-21 and -23 at.% Al alloys were examined mainly using TEM, TEM-EDX and DSC. The alloys were water-quenched from 1380°C in  $\beta$  single phase region and then some specimens were additionally aged at various temperatures. The microstructures obtained from the as-quenched specimens were strongly affected by quenching condition. When the size of specimen is large and quenching rate is relatively low, bainitic hcp plates precipitate. On the other hand, when the size of specimen is small, a martensite-like structure, instead of the hcp plates, was observed by optical microscopy. Regardless of the quenching condition, a spinodal-like microstructure composed of A2 and B2 phases was also detected and the A2 phase changes to the hcp phase during further ageing. The characteristics of martensite-like structure will also be discussed in the presentation.

#### **I21 Properties of Polyethylene Foam Incorporated with Extracted Lignin from Pulping Black Liquor**: *Aekartit Boonprasertpoh*<sup>1</sup>; Duanghathai Pentrakoon<sup>1</sup>; Warinthorn Chavasiri<sup>1</sup>; <sup>1</sup>Chulalongkorn University

This research was conducted to assess physical and mechanical properties of polyethylene foam incorporating with various amount of lignin from 5 to 20 parts per hundred. The lignin was extracted from pulping black liquor by precipitating with sulfuric acid at pH 3. A Fourier Transform Infrared Spectroscopy (FTIR), Gel permeation chromatography (GPC), and Thermo Gravimetric Analyzer (TGA) were employed to characterize the extracted lignin. The preparation of polyethylene foam was carried out by a two stage process using two-roll mill and compression moulding. Visual inspection, density measurement, and Scanning Electron Microscope (SEM) were used to investigate physical properties while compression test and compression set were used to investigate mechanical properties. The foam density increased slightly with amount of lignin. From SEM micrographs, it exhibited that all PE foams had a closed-cell structure where the cell sizes varied with amount of lignin. Foam incorporated with 10 phr of extracted lignin showed the finest cell distribution with an average cell diameter of 0.51 millimeters.

## **I22** Safe, Stable and Effective Nanotechnology – A Phase Map for Zinc Sulphide Nanoparticles: *Christopher Feigl*<sup>1</sup>; Salvy Russo<sup>1</sup>; Amanda Barnard<sup>2</sup>; <sup>1</sup>RMIT University; <sup>2</sup>CSIRO

Following its conception in the middle of the 20th century, nanotechnology has grown to become a critical part of our emerging technologies and is expected to create revolutionary capabilities in many fields. However the highly anticipated benefits are intimately linked to serious risks. Care and foresight must be taken to ensure that new and emerging nanotechnologies are safe, stable and effective. Unfortunately, unlike bulk materials, assessing the stability and toxicity of nanomaterials is a formidable task, owing to the complex thermodynamic and kinetic processes which govern the structure and properties of nanomaterials. Using density functional theory (DFT) calculations and a shape dependent thermodynamic model for the Gibbs free energy of a nanoparticle, we are attempting to map the equilibrium morphologies of zinc-sulphide nanoparticles (ZnS NPs) as a function of their size, temperature, pressure and chemical environment. This will facilitate phase prediction and in turn assist in the development of safe, stable and efficient nano-devices and the formation of appropriate material safety guidelines with regard to the use of ZnS NPs. We are able to report on the progress of DFT calculations and morphology predictions for nanoparticles of ZnS in the zinc-blende phase.

Posters

**123** Synthesis and Characterization of Mesoporous Carbon with MoO<sub>3</sub> Loading via Ultrasonic Assembly: Xi Long<sup>1</sup>; Wen Chen<sup>1</sup>; Shaojiang Chen<sup>1</sup>; Chunxia Zhao<sup>1</sup>; <sup>1</sup>State Key Laboratory of Advanced Technology for Materials Synthesis and Processing, and School of Material Science and Engineering, Wuhan University of Technology

Mesoporous carbon with MoO<sub>3</sub> loading (MoO<sub>3</sub>/CMK-3) was obtained via ultrasonic assembly with CMK-3 as the host material and MoO<sub>3</sub> as the guest material which was yield from MoO<sub>3</sub>  $\cdot$  pH<sub>2</sub>O<sub>2</sub>  $\cdot$  qH<sub>2</sub>O sol precursor. The microstructures of such MoO<sub>3</sub>/CMK-3 composites were characterized by X-ray diffraction (XRD), nitrogen adsorption and desorption, X-ray photoelectron spectra (XPS), Fourier-transform infrared (FTIR), field emission scanning electron

microscope (FE-SEM) and transmission electron microscopy (TEM). The results show that the method of ultrasonic assembly was efficient to highly disperse  $MOO_3$  nanoparticales into the channels of mesoporous carbon. The  $MOO_3$ /mesoporous carbon composites material retains an ordered mesoporous structure, the pore sizes are between 3.19 to 3.34 nm, the surface areas are between 924 to 1065 m<sup>2</sup> g<sup>-1</sup> and total pore volumes are around 0.73 and 0.89 cm3 g<sup>-1</sup>. With the assembly of  $MOO_3$ , the pore size, surface area and the total pore volume of the composites were reduced. FT-IR spectrum confirms that the infrared spectra peaks at 850 cm<sup>-1</sup> and 999 cm<sup>-1</sup> are due to the stretching vibration of carboxyl group Mo=O bond and  $MOO_2$  bond. The strong characteristic peaks of  $MOO_3$  verified the  $MOO_3$  particles were assembled into mesoporous carbon successfully.

#### **124** The Study of Cotton Finishing by Artemsia Argyi Oil Microcapsules: He Guanru<sup>1</sup>; Li Ly<sup>2</sup>; Au Wai-man<sup>2</sup>; Thomas K.S. Wong<sup>3</sup>; *Yang Zhuohong*<sup>1</sup>; Jiang Qiongling<sup>1</sup>; <sup>1</sup>College of Science, South China Agricultural University; <sup>2</sup>Institute of Textile and Clothing, Hong Kong Polytechnic University; <sup>3</sup>College of Nursing, Hong Kong Polytechnic University

In this paper, Gelatin-arabic gum microcapsules containing Artemsia argyi oil were prepared by complex coacervation firstly, along with its application to textile finishing using 2D resin as a crosslinking reagent was investigated. Scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR) were used to investigate the formation of ether bonds between 2D resin and cellulose and/ or gelatin-arabic gum microcapsules. The washing durability, strength retention, wrinkle-free performance, formaldehyde content, release property and antibacterial property of the finished fabrics were also tested. The results showed that the crease recovery angle of finished fabric was above 300°C, with the strength retention was above 60% and the released formaldehyde concentration was lower than 75 ppm. While the releasing amount of Artemsia argyi oil was less than 50% after 9 days. Moreover more than 70% antibacterial property can also be obtained even after 30 washing cycles.

## **125** Antioxidant Extracted from Clove Oil for Polyethylene Film: Kittipon Tiabuakaew<sup>1</sup>; Chutima Aiemsa-art<sup>1</sup>; *Duanghathai Pentrakoon*<sup>1</sup>; Warinthorn Chavasiri<sup>1</sup>; <sup>1</sup>Chulalongkorn University

A prospect of replacing commercial antioxidant agent such as butylated hydroxytoluene (BHT) by antioxidant extracted from clove oil extracted (i.e. eugenol) in polyethylene film was studied. The extracted eugenol was characterized using Gas chromatography. A free radical scavenging activities of eugenol and BHT were also investigated using 2, 2-diphenyl -1-picryhydrazyl (DPPH) assay. It revealed that a concentration of eugenol resulting in a 50% inhibition of the free radical, IC50, (0.11 mg/ml) was lower than the one of BHT (0.14 mg/ml) indicating better radical scavenging activity. The film samples were prepared with antioxidant agent from 0.2 to 1.0 phr mixed with LDPE using two roll mill and compression moulding. An Oxidative Induction Time (OIT) of film using Differential Scanning Calorimeter at 180°C was conducted. The OIT values of LDPE film, LDPE film having BHT (0.6 phr), and LDPE film having eugenol (0.6 phr) were 47, 72 and 84 mins, respectively. This exhibited that the LDPE film having eugenol had the best antioxidative efficiency. Insignificant changes of tensile strength and tear strength were presented. While the water vapor transmission rate and oxygen transmission rate values for LDPE film having antioxidant agent were greater that the ones without antioxidant agent.

#### 126 Compression Behavior and Texture Development of Ferromagnetic Shape Memory Alloy (FSMA) NiMnGa/ Polymer Composites: *Motoki Okuno*<sup>1</sup>; Tomonari Inamura<sup>1</sup>; Hideki Hosoda<sup>1</sup>; <sup>1</sup>Tokyo Institute of Technology

Ferromagnetic shape memory alloy NiMnGa is expected as a new actuator material exhibiting large strain around 6% for 10M martensite and high response around kHz. A drawback is that the strain is not recoverable by reducing magnetic field only, and another is the brittleness of polycrystalline NiMnGa. Then, we have been developing NiMnGa/polymer composites. However, the magnetostrain appeared in the composites was very small. This might be due to the random crystallographic orientations of NiMnGa particles. If all the NiMnGa particles have a similar crystallographic orientation, the composites should exhibit comparable large actuation strain. In this work, arrangement of crystallographic orientation of NiMnGa particles was aimed through texture development by compressive deformation. NiMnGa particles with the size of  $100{\sim}160\mu m$  were embedded in polymers: 50vol%NiMnGa in silicone rubber and 30vol%NiMnGa in epoxy. The texture development was evaluated by X-ray diffraction pole figure measurements. It was found that the silicone rubber composite exhibited (004) texture after compression, but that the epoxy composite did not. The difference must be due to the difference in elastic modulus of polymers. The stress for rearrangement of martensite variants was estimated to be 20-40MPa for the composites.



**127** Computational Modelling of Deformation of NiTi Plates with Circular Holes: *Bashir Samsam*<sup>1</sup>; Yinong Liu<sup>1</sup>; Gerard Rio<sup>2</sup>; <sup>1</sup>The University of Western Australia; <sup>2</sup>Université de Bretagne Sud

This study presents a numerical model for deformation behaviour of nearequiatomic NiTi holey plates using finite element method. Near-equiatomic NiTi alloy deforms via stress-induced martensitic transformation, which exhibits a typical hystoelastic mechanical behaviour. In this model, the transformation stress is decomposed into two components: the hyperelastic stress, which describes the reversible aspect of the deformation process, and the hysteretic stress, which describes the irreversible aspect of the process. It is found that, with increasing the level of porosity, the apparent elastic modulus before and after the stress plateau decrease, the nominal stresses for the A  $\leftrightarrow$  M transformation decrease and the strain increases, and the pseudoelastic stress hysteresis decreases. In particular, the transformation strain increases by about 50% by introducing 25% porosity. In addition, the stress-strain slope over stress plateau also increases with increasing the number of holes. While the porosity percentage has a strong effect on global stress-strain behaviour of the holey plate, regularity of holes has negligible effect.

## 128 Electrochemical Synthesis of Sensitive Layer of Polyaniline: Effects of Acid Doped on Ethylene Gas Sensing: *Prasit Pattananuwat*<sup>1</sup>; Daungdao Ahtong<sup>1</sup>; <sup>1</sup>Chulalongkorn

Ethylene gas is released from plants as a hormone during a certain stage of life cycle and induces ripeness in fruits and blooming of flowers. The early detection and control amount of ethylene gas in storage can prevent wastage of the entire stock. The aims of this research were to fabricate ethylene sensor based on polyaniline (PAni) and to investigate the effect of acid doping on an improvement of its sensitivity. Electrochemically synthesized PAni was prepared via an in situ radical polymerization by repeating potential cycling in the range of -0.3 to 1.0 V relative to the silver reference electrode and platinum counter electrode. The PAni films were deposited on interdigited electrode of gold substrate. These PAni were doped with five different acids doping (such as HCl, H<sub>2</sub>SO<sub>4</sub>, H<sub>2</sub>PO<sub>4</sub>, TSA and DBSA) at particular concentration. The influences of types and amount of acid doping on the sensitivity of ethylene gas were investigation. The PAni-dope films were characterized by Fourier transform infrared spectroscopy (FT-IR) and UVvisible spectroscopy (UV-vis). The morphology of PAni film was investigated by scanning electron microscope (SEM). The magnitude sensing of doping PAni in terms of types and amount of acid against ethylene gas will be presented.

## **129 Kinetic Modelling of Nanoparticle Evolution**: Alireza Seyed-Razavi<sup>1</sup>; Amanda Barnard<sup>2</sup>; <sup>1</sup>RMIT University; <sup>2</sup>CSIRO

Nanotechnology is a promising field that is poised to make important contributions to a number disciplines, and potentially revolutionise the scientific and technological landscape. Nanoparticles and other low-dimensional nanostructures are of particular interest, as their characteristics may differ significantly to those of their bulk counterparts. To exploit these differences to full advantage, it is highly desirable to observe the evolution of these minute structures, and ultimately gain insight into how one may tailor specific characteristics, for given applications. However, the evolution of nanoparticle is a complicated issue, which can be difficult to probe experimentally. A theoretical model, capable of describing the various phenomena that occur at the nanoscale (such as coarsening, ripening, aggregation and coalescence), and with sufficiently accuracy so as to be comparable to experiments, would be invaluable in this regard. The first step to achieving this goal is the coupling of variuos classical theories of nanoparticle growth; such as the Lifshitz-Slyozov-Wagner (LSW) and the Burton-Cabrera-Frank (BCF) theories, as outlined in this presentation.

#### **I30 Magnetoelastic Behaviours in Magnetically Annealed Tb-Dy-Fe-Co Polycrystals:** Changsheng Zhang<sup>1</sup>; *Tianyu Ma*<sup>1</sup>; Mi Yan<sup>1</sup>; <sup>1</sup>Zhejiang University

Giant magnetostrictive material Tb-Dy-Fe has attracted much technological and scientific interest because of their giant magnetostriction and high magnetoelastic energy transfer efficiency. The practical application of Terfenol-D always requires a large magnetostriction in a modest external field. Materials with <112> or <110> crystal orientation prepared by various unidirectional solidification methods are known to exhibit improved magnetostrictive performance when the initial magnetic state is changed by applying a compressive pre-stress or magnetic annealing. Here we present the influence of magnetic annealing on the magnetoelastic behaviours of a wide-operating-temperature-range magnetostrictive alloy Tb0.36Dy0.64(Fe0.85Co0.15)2. The results show that: 1) magnetostrictive response exhibits a strong dependence on the direction of annealing field. 2) The magnetically annealed polycrystals exhibit different anisotropic magnetostrictive behaviours when the magnetization direction is changed. 3) Totally different stress dependences of magnetization and magnetostriction are observed after perpendicular field annealing. 4) The perpendicular magnetic annealing also results in enhanced Young's modulus and damping capacity. Magnetic force microscopy (MFM) images present direct

evidence to show the changes of magnetic domain configurations after magnetic annealing, which should be responsible for the variation of such magnetoelastic behaviours.

131 Study on Microstructure of Ti-Nb-Sn/HA Composite of Biomaterials Fabricated by High Energy Mechanical Milling and Pulse Current Activated Sintering: *Kee-Do Woo*<sup>1</sup>; Xiaopeng Wang<sup>2</sup>; Duck-Soo Kang<sup>1</sup>; Sang-hyuk Kim<sup>1</sup>; Zhiguang Liu<sup>2</sup>; <sup>1</sup>Chonbuk National University; <sup>2</sup>Harbin Institute of Technology

Ti and its alloys such as Ti-6Al-4V alloy have been widely used for biomaterials due to their excellent biocompatibility, low density, excellent corrosion resistance and good balance of mechanical properties. However, some problems of Al and V, which are contained in mostly used Ti-6Al-4V (a + ß type) have been reported. Additionally, Ti-6Al-4V alloy has high Young's modulus compared to natural bone. Therefore, Ti-35wt%Nb-2.5wt%Sn/5,10,15wt%Hydroxyapitite( HA ) composites which are biocompatible alloy because this alloys don't include Al, V elements. Our specimens can be successfully fabricated by pulse current activated sintering (PCAS) using high energy mechanical milled(HEMM) powder. The particle sized and microstructures of the milled powders and sintered specimens were studied by OM, XRD, SEM and TEM. As result of XRD, a - and ß- Ti co-existed in the 8h milled powder. The milled powders have been transformed completely from a –Ti to ß- Ti after milling for 12h. The sintered specimen which is milled for 12h shows Ti, Nb, Sn and HA were distributed homogenously. And composites which have nano-sized grain were fabricated. Also the HA acted with Ti during sintering to form CaTiO, which is useful phase for biocompatibility.

#### Poster Session: Symposium J: Materials Characterisation and Evaluation

Tuesday PM	Room: Hall 2
August 3, 2010	Location: Cairns Convention Centre

J1 Effect of Boron Contents on Elevated Temperature Creep Rupture Strength of 9Cr-1.5Mo Alloy: Bumjoon Kim<sup>1</sup>; Haksu Yun<sup>1</sup>; Jonghoon Lee<sup>1</sup>; Byeongsoo Lim<sup>1</sup>; <sup>1</sup>Sungkyunkwan University

The high Cr ferritic martensitic steels are widely used for the component materials in the steam turbine power plants. 9Cr-1.5Mo steels are used for high temperature applications such as main steam pipe, turbine header, blade in power plants, because of their good mechanical properties and oxidation resistance. For thick-section components such as headers and pipes, high creep rupture strength and oxidation resistance are required. Even with their good creep resistance at elevated temperatures, still a number of research works are being carried out to improve the creep properties of 9Cr-1.5Mo alloy. One of the efforts is adding small amount of boron into 9 to 12 % Cr steels. In this study, the creep behaviour of 9Cr-1.5Mo steel with boron addition was investigated by small punch (SP) creep test at the elevated temperature range of 600~650°C. The amount of boron addition was varied within the range of 0.0033~0.0196 wt%. The relationship between the creep rate and rupture life with boron addition was examined and compared at each test temperature of 600, 625 and 650°C. Also, to investigate the effect of boron on the cavity behaviour, the rupture part of SP creep specimens were investigated by the SEM.

J2 Evaluation of Aging Embrittlement of Austenitic Stainless Steels JN1, JJ1 and JK2 by Cryogenic Small-Punch Testing: *Maribel Saucvedo-Muñoz*<sup>1</sup>; Toshiyuki Hashida<sup>2</sup>; Shin-Ichi Komazaki<sup>3</sup>; Victor Lopez-Hirata<sup>1</sup>; <sup>1</sup>Instituto Politecnico Nacional (ESIQIE); <sup>2</sup>Fracture Research Institute, Tohoku University; <sup>3</sup>Muroran Institute of Technology

Small-punch tests were conducted at 4, 77 and 293 K on three types of austenitic stainless steels JN1, JJ1 and JK2, which were solution treated, waterquenched and then aged at 923, 973, 1023 and 1073 K for 5 hours. Small-punch test energy was employed for the evaluation of the aging-induced embrittlement behavior in these materials. Fracture surface of small punch test specimen for the solution treated steels exhibited a ductile fracture, showing the highest SP test energy values. The presence of intergranular brittle fracture was observed in aged specimens. Small-punch test energy decreased significantly as the aging process progressed. The highest and lowest decrease in small-punch test energy with aging temperature occurred in JN1 and JK2 steels, respectively. The decrease in small-punch test energy showed to examine appropriately the aging-induced embrittlement behavior for these steels was explained based on the volume fraction of intergranular precipitates in aged samples.



J3 Evaluation of Fretting Wear Behavior on the Simulated Supporting Structures of a Dual-Cooled Nuclear Fuel Rod: Young-Ho Lee<sup>1</sup>; Hyung-Kyu Kim<sup>1</sup>; <sup>1</sup>Korea Atomic Energy Research Institute

A dual-cooled fuel (i.e. annular fuel) has been proposed to substantially increase in power density and safety margins compared to a solid fuel in operating PWR plants. As this fuel rod has larger outer diameter than the conventional solid rod to accommodate sufficient internal flow, new supporting structure geometries should be designed and their reliabilities (i.e. vibration characteristics, fretting wear resistance, etc.) are also examined with both analytical and experimental methods. In this study, the supporting structure characteristics and fretting wear behaviors are analyzed and examined by using two kinds of simulated supporting structures that have embossing and cylindrical shapes. Their supporting structure characteristics were examined by using a specially designed test rig and their results were compared with that of analytical method. Also, fretting wear behavior of simulated supporting structures was experimentally examined with considering the effect of contact shapes and their stiffness values. Based on the test results, the relationship between the supporting structure characteristics and their fretting wear behaviors was discussed in detail.

## J4 Evaluation of Residual Stress of Railway Wheel Regarding to Deterioration: *Seok-Jin Kwon*<sup>1</sup>; Jung-Won Seo<sup>1</sup>; Dong-Hyung Lee<sup>1</sup>; <sup>1</sup>Korea Railroad Research Institute

The wheels for railway vehicle in running had experienced the wheel failure due to fatigue crack, overheat braking and shelling. Severe heating of the wheel tread during braking was believed to be a contributing the variation of residual stress which is related to wheel failure. It is necessary to evaluate the residual stress due to deterioration of wheel tread in order to ensure the safety of wheel. In the present paper, the residual stress of railway wheel for deterioration using x-ray diffraction system is evaluated. The result shows that the residual stress of wheel is depend on the running distance and thermal gradient during brake application.

### J5 Failure Analysis on the Rivet: H.Y. Yu<sup>1</sup>; *D.C. Zeng*<sup>1</sup>; Z.W. Liu<sup>1</sup>; <sup>1</sup>South China University of Technology

This paper presents a detailed failure analysis on a rivet, which broke into two parts from the joint place between head and shank under a load of seven tons during service, by the combination of optical microscopy, SEM and EDS. The metallographic examination shows some micro-inclusions in the sample and many micro-cracks along the grain boundaries. SEM and EDS results show that the surface of the rivet was plated with a zinc coating and it was eroded severely. The fracture surface exhibits the characteristic of quasi-cleavage fracture, indicating an intergranular brittle fracture mode. Some micro-pores were found on the facets of intergranular cracked grains. Most importantly, the Crow-toe Pattern, one of river patterns or hairline seams, which is a typical microstructure of hydrogen embrittlement, was observed on the fracture. Based on above analysis, it is concluded that the failure of the rivet is caused by the hydrogen embrittlement. The hydrogen, most likely, comes from the zinc plating process. Due to the aggregation and diffusion of hydrogen into the matrix, the brittle cracking happens in the regions with stress concentration because of the volume expansion effect under the external force.

#### J6 Mechanical Properties of Ag Nanopowder for Artclay Prepared by Liquid Phase Reduction Method: *JongSeong Lee*<sup>1</sup>; Sun Moon<sup>1</sup>; Hyun Lee<sup>1</sup>; <sup>1</sup>Chosun University

Ag nanopowders for Artclay were prepared by liquid phase reduction method of adding reductant(NaBH<sub>4</sub>) to AgNO<sub>3</sub> aqueous solution, for improved dispersibility surfactant(Tween 20) was added to the solution during synthesis process. N<sub>2</sub>H-----4 and NaBH<sub>4</sub> that is one of reductants being used generally on liquid phase reduction method was employed under this process, the influence on a change of Ag nanopowder's properties according to kinds of reductant looked around. In order to mechanical properties, bulk that was produce by mixing colloid binder with synthesized Ag power. Irrespective of reductant, the pure Ag powders that have the property of square structure without any impurity mixing was confirmed as the result of XRD, Ag particle prepared by sodium NaBH<sub>4</sub> shows that the spherical shape has the size of 50 nm and less and has the uniformed particle size distribution as the result of observed micro structure by using of TEM and particle size analyzer but the power prepared by N<sub>2</sub>H<sub>4</sub> has the size of around 5.02µm. As the result of tensile and micro hardness test, Ag bulk prepared by sodium borohydride showed that hardness and ductility is around 2 times and 1.1 times more high than that of Ag bulk prepared by N<sub>2</sub>H<sub>4</sub>.

J7 Mechanical Properties of Metallized Single Nanofibers: *HaeRim Kim*<sup>1</sup>; Naotaka Kimura<sup>1</sup>; Hyun-Sik Bang<sup>1</sup>; Byoung-Suhk Kim<sup>1</sup>; Yoshimi Watanabe<sup>2</sup>; Ick-Soo Kim<sup>1</sup>; <sup>1</sup>Shinshu University; <sup>2</sup>Nagoya Institute of Technology

We report that both metallized polyurethane (PU) nanofiber webs and metallized single PU nanofibers can be successfully prepared by a combined technique of electrospinning and metallization. In our previous study, it was found that the metallized nanofiber webs exhibited higher mechanical properties depending on the thickness of the deposited metallic layers. However, evaluating the mechanical properties for the nanofiber webs has remained lots of problems to be figured out. In this study, the mechanical properties of metallized single PU nanofibers are investigated by using recently developed tensile test machine. The tensile strength of 50nm Cu-metallized PU single nanofiber (~ 338 MPa) is clearly higher than that of pure PU single nanofiber (~ 132 MPa), whereas the Young's modulus of 50nm Cu-metallized PU single nanofiber is a little smaller than that of pure PU single nanofiber, suggesting the incomplete metal coating and the formation of metal-PU composite layers at the surface of PU nanofibers, which might give rather tough and rubbery composite nanofibers. In addition, the metal alloy nanofibers (for instance, Cu-Ni, Cu-Sn, etc) are prepared and its mechanical properties are studied.

#### J8 Microstructure and Properties of Ni-Based Nano-Self Lubricating Coating: Wang Wen Yan<sup>1</sup>; <sup>1</sup>Henan University of Science and Technology

In this paper, a series of experiments were carried out to observe the microstructure, and test the microhardness, the friction coefficient and wear resistance of the Ni-based nano-h-BN solid self-lubricating coatings which were prepared by both HVOF spraying process and YAG laser cladding. Microstructure and tribological propertied were investigated by using of XRD, SEM, Microhardness tester and High-temperature High-speed pin-on-disk tribotester machine (MMS - 1G). Results showed that 5% of the content h-BN's friction coefficient is the most stable whose friction coefficient is stable around 0.470, so we come to a conclusion that this coating is an ideal HVOF coating; Whereas the ideal laser cladding coating is 7.5%h-BN(Ni) coating, whose friction coefficient is around 0.360, this is the best laser cladding coating.

## J9 Microstructure Features and Contact Fatigue Crack Growth on Rail: Jungwon Seo<sup>1</sup>; Seokjin Kwon<sup>1</sup>; Hyenkue Jun<sup>1</sup>; Donghyeong Lee<sup>1</sup>; <sup>1</sup>Korea Railroad Research Institute

Rolling contact fatigue damages on the surface of rail such as head check, squats are one of growing problems. Since rail fracture can cause derailment with loss of life and property, the understanding of rail fracture mechanisms is important for reducing damages on the surface of rail. In this study, the fatigue crack at the upper surface of rail was investigated by means of failure analysis. The investigation indicate that the crack grows at about 70° to the surface of the rail and this crack changed its growth direction; either to branch downwards at about 20° or to branch upwards. Since the downward branches lead to fracture of the rail, they are more dangerous to the integrity of rails. It has been observed that White Etching Layer(WEL) occurs within the surface of broken rail. It was found that the fatigue crack initiation and propagation was accelerated by the WEL.

#### J10 Properties of PH 13-8 Mo Steel for Fatigue Application in Helicopters: Sunny Lok Hin Chan<sup>1</sup>; Ung Hing Tiong<sup>1</sup>; Graham Clark<sup>1</sup>; <sup>1</sup>RMIT University

While many fixed-wing aircraft have adopted damage-tolerant design in recent years, helicopter design is still based predominantly on a safe life approach, in which relatively simple Stress Life (S-N) data underpins the tools used for life prediction. Due to the unique loading spectra of helicopters, its structures experience a high number of loading cycles as compared to fixed-wing aircraft, presenting a more challenging fatigue life management problem. To minimise the fatigue damage, the helicopter community tends to design components such that most of the loading experienced falls below the fatigue limit of the selected material. These materials are usually of high strength and have good fatigue properties, although the large number of cycles raises the possibility of fatigue in the "gigacycle" regime where the fatigue limit drops to a new, lower level. This paper discusses the suitability of a high quality PH 13-8 Mo steel for critical helicopter usage; considering its fatigue performance particularly at high R ratio and other properties such as density, corrosion properties and cost in terms of the different conditions that might be faced in Australian helicopter operations.

## **J11 Static and Dynamic Mechanical Behaviors of Vanadium Alloy V-5Cr-5Ti**: *Xicheng Huang*<sup>1</sup>; Wenjun Hu<sup>1</sup>; Yixia Yan<sup>1</sup>; <sup>1</sup>China Academy of Engineering Physics

In this work we focus on the static and dynamic properties of vanadium alloy V-5Cr-5Ti over a range of temperature from RT to 1000 degree at strain rates ranged from 10<sup>-4</sup>/s~10<sup>3</sup>/s. To understand dynamic plastic flow behaviours of vanadium alloy under high strain-rate, uniaxial compression and tension tests are performed using SHB technique. Experimental data show that:1) V-5Cr-5Ti behaves strain-rate sensitive, including yield stress and UTS; 2) V-5Cr-5Ti displays macroscopically or phenomenally the characteristics of ductile materials at room temperature, such as large plastic flow or distinct strain-hardening and the necking phenomenon. The main fracture mode is brittle cleavage fracture microscopically, i.e. Mode I fracture dominates in BCC metal V-5Cr-5Ti at RT. On the grain boundaries large numbers of dimples are observed; 3)at high temperatures (1000°C) the tensile fracture of the material exhibits brittle and the necking is not observed. The stress-strain curves, UTS and failure strains of V-



5Cr-5Ti for various strain rates are presented. The temperature-rate-dependent constitutive relations are obtained in the form of JC model which is widely used in numerical simulation of dynamic processes. The obtained constitutive relations are applied in FEA of testing and processing.

#### J12 Surface Damage in Riveted Aircraft Aluminium Alloy Lap Joints, in the Presence of Lubricants: *Aditya Jaya*<sup>1</sup>; Ung Hing Tiong<sup>1</sup>; Graham Clark<sup>1</sup>; <sup>1</sup>RMIT University

This paper discusses an investigation into the effect of lubrication on surface damage in riveted lap joints typical of those experiencing fatigue loading in aircraft structure. The study focussed on the fracture surfaces of failed specimens. The specimens were made from 2024-T3 clad, and 2024-T3 bare aluminium alloys. No surface protective coating was applied, so that the specimens simulate the surface condition of riveted lap joints in ageing aircraft, where the coatings have degraded completely, and therefore feature direct metal-to-metal contact. Two different forms of lubricant were selected and applied to some of the specimens. One was in the form of an oil-based lubricant, while the other is in the form of soft waxy film. Both are in wide use in the aerospace industry to protect aircraft metallic structures against corrosion. All specimens were fatigue tested at constant amplitude, but at various stress levels. The paper describes detailed Scanning Electron Microscope (SEM) examination of the fractured surfaces to identify the surface damage at the crack initiation sites, and along the crack length. The results were compared with those obtained from control specimens that were not treated with any lubricants.

#### J13 The Study of Corrosion and Mechanical Properties for Optimum FSWed Dissimilar Aluminum Alloy by SSRT: Seong-Jong Kim<sup>1</sup>; Jae-Cheul Park<sup>1</sup>; Seok-Ki Jang<sup>1</sup>; <sup>1</sup>Mokpo Maritime University, Division of Marine System Engineering

The construction of aluminum ships has increased since the introduction of international regulations on the control of marine environmental pollution, which include the use of environmentally friendly materials. Friction stir welding (FSW) was developed by The Welding Institute (TWI) in 1991 to solve this problem of joining light metals such as aluminum alloys. In this study, we evaluated the friction stir weldability for dissimilar aluminum alloys such as 5052-O and 6061-T6, using slow strain rate test. The SSRTs were carried out at a strain rate of 0.003mm/min, during which a constant potential was maintained. The specimens used for the SSRTs were exposed to natural seawater and then fitted with a jig for loading into a CERT machine (R&B Inc.). Result of the SSRT for FSWed part, the mechanical properties presented relatively higher value than only seawater condition at the range of -1.48V  $\sim$  -0.7 V.

#### J14 A Study on Electrochemical Behavior in Sea Water for KR-RA Steel: Seong-Jong Kim<sup>1</sup>; Seung-Jun Lee<sup>1</sup>; <sup>1</sup>Mokpo Maritime University

With the increasing of the trade between nations, ship is in charge more than 80% of world trade. In addition, the cavitation and erosion corrosion of rudder which exposed to corrosive marine environments has been emerged as an important problem. To find a electrochemical behaviour for KR-RA steel, this study performed various experiments in sea water environment. The electrochemical apparatus consisted of a Pt coil as the counter electrode and Ag/AgCl(saturated KCI) reference electrode. The natural potential measurement test was performed for 86.400 seconds. Anodic and cathodic polarization trend were tested from the open circuit potential to +3.0V and -2.0V. For potentiostatic experiment to compare corrosion resistance, the changes in current density for 1.800 seconds at a constant potential and the values after 1.800 seconds were compared in various applied potential conditions. For Tafel analysis, the corrosion potential and corrosion current density were obtained by polarizing  $\pm$  0.25V. At the comparison of current density after potentiostatic experiment during 1800s, the lowest current densities occurred at potentials of -0.8V. Optimum protection potential range in KR-RA steel is  $-1.0V \sim -0.7V$ .

#### J15 Black Molybdate Coating on EG Steel for Solar Application: Chang Hyun Nam<sup>1</sup>; Byung-Il Kim<sup>1</sup>; Yun Il Choi<sup>2</sup>; Chan Jin Park<sup>2</sup>; Young Geun Kim<sup>3</sup>; Ji-Myon Lee<sup>1</sup>; <sup>1</sup>Sunchon National University; <sup>2</sup>Chonnam National University; <sup>3</sup>POSCO

Enhancement of corrosion resistance of electro-galvanized (EG) steel by surface treatment using rare metal such as molybdate and tungstate aqueous solutions was studied using EIS, SEM, and XPS. Sample used in this study were commercially available 0.8 mm thick EG steel plate. The sample, cut into 400 mm2, were dipped in the molybdate or tungstate aqueous solution for 30 min ~ 10 sec at 45°C and pH of 2. As increasing the dipping time in NaMoO<sub>4</sub> solution, the concentration of Mo on the surface was increased to as increase to 2.76 %. When we added a reaction accelerator, such as NaNO<sub>2</sub> and NaNO<sub>3</sub>, the treatment time could be decrease up to 10 sec. Moreover, when NiSO<sub>4</sub> was added in the solution, the color of treated sample was turned into yellow, brown, and black color depending on the treatment time. The spectral reflectance of the black molybdated sample was measured to be less than 5 %, indicative of possibility of solar energy applications. We also found that the polarization resistance of the blackened sample was enhanced by more than 2 orders of magnitude, indicating that the corrosion resistance was enhanced compared with the non-treated sample.

#### J16 Characterising the Effects of Cold Spray Process Gas and Temperature on CP Titanium Structure: *Natasha Wright*<sup>1</sup>; Stefan Gulizia<sup>1</sup>; Mahnez Jahedi<sup>1</sup>; Aaron Seeber<sup>1</sup>; <sup>1</sup>CSIRO

During Cold Spray process small powder particles are propelled to supersonic velocities in gas streams using a converging-diverging de Laval nozzle. Particles bond to the substrate on impact to form a strong bond, however, little is understood regarding the effects of process gas temperature on the deposited structure. The characterisation of these types of materials holds many challenges. Of particular importance is the identification of suspected crystalline phase changes in the higher gas temperature deposited structures. It was hypothesized these trace crystalline phases were the cause of significant changes in both the visible appearance and mechanical performance of the finished titanium product. In this study the effects of process gas type and temperature on the structure of deposited CP titanium were investigated. Cold Spray deposits were produced at various gas temperatures using both nitrogen and helium gas. Oxygen and nitrogen content in the deposited layer was studied using Leco oxygen-nitrogen analyser. SEM and high resolution microprobe were employed to determine changes in both morphology and chemistry depending on the process gas type and temperature. Finally laboratory based X-ray microdiffraction and Synchrotron Xray diffraction provided the required crystalline phase information to finally solve the colour and mechanical performance change puzzle.

#### J17 Evaluation of Surface Defects of Wheel and Rail for Korean High-Sspeed Railway: Lee Chanwool; <sup>1</sup>Korea Railroad Research Institute

Wheels of the railway vehicle play the important role for driving train through wheel-rail interaction. Especially wheel profile is one of the most important design factors to rule the running stability and safety of train. Accordingly control of RCF-related defects is an ongoing concern for both safety and cost reasons. This process is referred to as ratcheting. Wear of wheel and rail surfaces occur due to a mixture of adhesive, abrasive and corrosive processes. In wheel/rail systems with little wear, such failure is manifested by the appearance of closely spaced micro-cracks. In the present paper, evaluation of surface defects of wheel and rail for Korean high-speed railway. The main research application is the wheel-rail maintenance of Korea high-speed train.

### J18 Recrystallization Texture Formation Behavior of Differential Speed Cold-Rolled Pure-Ta for Sputtering Target: *Won-Yong Kim*<sup>1</sup>; Han-Sol Kim<sup>1</sup>; <sup>1</sup>Korea Institute of Industrial Technology

Ta sputtering can provide better performance and reliability as a diffusion barrier material for Cu interconnection in semiconductor packaging. The quality of thin layer deposited by sputtering is usually dependent upon sputtering conditions and materials characteristics including purity, microstructure and texture. We have investigated texture formation behavior of high purity Ta in order to control desired microstructure and texture in relation with uniform deposition in semiconductor packaging process. The material used in this work was highly pure (99.99%) Ta ingots produced by electron beam melting technique. Differential speed cold rolling methods were employed in order to produce characteristic microstructures depending on applied stress state of cold rolling. The cold rolled plates were sealed in vacuum into quartz tube to prevent oxidation or contamination during recrystallization annealing. And then the cold rolled sheets were annealed at 1273K, 1373K and 1473K, respectively, Scanning electron microscopy equipped with electron back-scattered distribution has been used to observe microstructure and texture. The major texture component of differential-speed rolling was characterized by <001>//ND fibre. With increasing annealing temperature, the intensity of initial texture components were drastically reduced, while <111>//ND component was strongly developed. Details will be discussed in relation with recrystallization behavior and deformation texture.

#### J19 Research on Corrisive Wear Behaviours of Super High Chromium Cast Iron (SHCCI) under Acid Medium Condition: *Shizhong Wei*<sup>1</sup>; Kun Wu<sup>1</sup>; Liujie Xu<sup>1</sup>; Jiwen Li<sup>1</sup>; Guang Du<sup>1</sup>; <sup>1</sup>Henan University of Science and Technology

The components in slurry pump suffer serious corrosive abrasion in the phosphorus fertilizer manufacturing process because they undergo impact of particles and corrosion of acid medium at the same time. The conventional abrasive resistant materials, such as high chromium cast iron, wear resisting steel and abrasion resistant cast iron, have not meet the requirement. In order to solve the question, the SHCCI with chromium content of about 37wt.% was developed by means of casting method. The microstructure was researched by use of SEM and XRD. Compared with conventional high chromium cast iron with 26wt.% Cr (Cr26), the corrisive wear property of SHCCI was researched using MCF-30 type erosion abrasion tester under 1 mol/L H<sub>3</sub>PO<sub>4</sub> medium condition. The results show that the microstructure of SHCCI is composed of  $M_7C_3$ ,  $M_{23}C_6$ , martensite and austenite. The relative wear resistance of SHCCI is three times higher than that



of Cr26. The excellent corrisive wear resistance of SHCCI is caused by the high electrode potential enhancing corrosion resistance capability of matrix, and large amounts of high hardness chromium carbides resisting wear as well.

J20 Coarsening Process of Decomposed Phases in Cu-Ni-Cr Alloys: Victor Lopez-Hirata<sup>1</sup>; Felipe Hernandez-Santiago<sup>1</sup>; Jorge Gonzalez-Velazquez<sup>1</sup>; Maribel Saucedo-Muñoz<sup>1</sup>; <sup>1</sup>Instituto Politecnico Nacional (ESIQIE)

A study of the coarsening process of the decomposed phases was carried out in the Cu-34wt.%Ni-4wt.%Cr and Cu-45wt.%Ni-10wt.%Cr alloys using transmission electron microscopy. As aging progressed, the morphology of the coherent decomposed Ni-rich phase changed from cuboids to platelets aligned in the <100> Cu-rich matrix directions. Prolonged aging caused the loss of coherency between the decomposed phases and the morphology of the Ni-rich phase changed to ellipsoidal. The variation of mean radius of the coherent decomposed phases with aging time followed the modified LSW theory for thermally activated growth in ternary alloy systems. The linear variation of the density number of precipitates and matrix supersaturation with aging time, also confirmed that the coarsening process followed the modified LSW theory in both alloys. The coarsening rate was faster in the symmetrical Cu-45wt.%Ni-10wt.%Cr alloy due to its higher volume fraction of precipitates. The activation energy for thermally activated growth was determined to be about 182 and 102 kJ mol-1 in the Cu-34wt.%Ni-4wt.%Cr and Cu-45wt.%Ni-10wt.%Cr alloys, respectively. The size distributions of precipitates in the Cu-Ni-Cr alloys were broader and more symmetric than that predicted by the modified LSW theory for ternary alloys.

J21 Crystallography and Morphology of Antiphase Boundary-Like Structure Induced by Martensitic Transformation in Ti-Ni Shape Memory Alloy: *Mitsuhiro Matsuda*<sup>1</sup>; Kazuhiko Kuramoto<sup>1</sup>; Yasuhiro Morizono<sup>1</sup>; Sadahiro Tsurekawa<sup>1</sup>; Toru Hara<sup>2</sup>; Minoru Nishida<sup>3</sup>; <sup>1</sup>Kumamoto University; <sup>2</sup>National Institute for Materials Science; <sup>3</sup>Kyushu University

An antiphase boundary (APB)-like structure in the B19' martensite of Ti-Ni alloy has been investigated by transmission electron microscopy. The APB-like structure has the atomic shifts on both the  $(010)_{R10}$  plane along the *c*-axis and the  $(001)_{B19}$  plane along the *b*-axis; a kind of ledge and step structure on the b-c plane, in addition to the displacement along the a-axis. The displacement vector can be expressed as  $R = \langle 0.1648 \ 1/2 \ -0.4328 \rangle$  in terms of the conventional atomic coordinates of Ti and Ni in the B19' martensite. The APB-like contrast is diminished by reverse transformation with heating above A<sub>f</sub> temperature. This supports that the APB-like structrue is irrelevant with the B2 ordered structure, that is, the structure is not inherited from the APB in the B2 parent phase. During the martensitic transformation from B2 to B19' structures, the accidental impingement of differently nucleated martensitic domains produces the APBlike interface. Therefore, APB-like structure has both natures, that is, APB and stacking fault from the viewpoint of the morphology, formation mechanism and atomic shift at the interface. We conclude that the APB-like contrast is defined as the stacking fault with APB-like morphology induced by the desplacive transformation.

### J22 Defect Assessments in Known Flaws Using Infrared Thermography: *Jeongguk Kim*<sup>1</sup>; Sung Cheol Yoon<sup>1</sup>; <sup>1</sup>Korea Railroad Research Institute

In order to assess the detectability of known flaws using the infrared thermography technique, two different types of calibration panels were prepared with various dimensions of artificial flaws. The spherical or rectangular flaws with different diameters and depths were prepared from the panels. The panels were composed of polymer matrix composites (PMCs) and structural steel (SS400), respectively. The thermographic detection of artificial flaws on both panels was performed using the infrared thermography method with a high-speed infrared camera. The lock-in thermography with flash lamp was used for the integrity evaluation of both panels. Through lock-in thermography, the optimal frequency of heat source was determined for the best flaw detection. In this investigation, the lock-in thermography was employed to develop a nondestructive evaluation tool for the detection of flaws in PMC and steel panels.

#### J23 Determination of Ni,Cr and Cu in Low-Alloy Steels by Inductively Coupled Plasma Quadrupole Mass Spectrometry with Dynamic Reaction Cell: *Kang Dehua*<sup>1</sup>, <sup>1</sup>Ansteel

obtained between the certified values and the experimental results. The precision

An inductively coupled plasma quadrupole mass spectrometer equipped with a dynamic reaction cell (DRC) was successfully used for the accurate determination of Ni,Cr and Cu in low-alloy steel samples, using the methane as the reaction gas.The method obviated the interference of 44Ca16O+,42Ca18O+ on 60Ni+, 36Ar16O+, 40Ar12C+, 35Cl16O1H + on 52Cr+, and 23Na40Ar+ on 63Cu+ respectively by using the DRC technology.The effects of the operating conditions of DRC system were optimized to get the best signal to noise ratio. The matrix match method and the internal solution was used to calibrate the matrix effect and the instrument drift.Validation of the method was carried out by the determination of Ni Cr and Cu in steel standard reference materials Good agreement was

between sample replicates was better than 5.0% for all the determinations and the recoveries were between 95.0%-110.0%.

J24 Effect of Alumina Starting Materials on Spheroidization in Flame Fusion Method: SunHui Eom<sup>1</sup>; *Jae-Hwan Pee*<sup>1</sup>; Jong-Keun Lee<sup>2</sup>; Woo-Seok Cho<sup>1</sup>; <sup>1</sup>Korea Institute of Ceramic Engineering and Technology; <sup>2</sup>Deahan Ceramics Co., Ltd.

The thermal conductive sheet of high efficient for the heat sink is important in the electrical part. The efficiency of thermal conducting depends on the filling rate of the alumina. To get the high filling rate, the production method has been developed actively. In the production of the spherical alumina, thermal spray using liquefied petroleum gas as the combustion gas has been studied. In the thermal spray method, the produced particle size is 50µm and less. However the melting behavior in the flame and spheroidization rate of starting powder is not studied. Also the spherical alumina produced by thermal spray method has lots of pores and is not densified. Because starting alumina powders was not fully melt in the thermal flame. The low density of spherical alumina decrease the thermal conducting rate. In this paper, the effect of alkaline metal contents in the starting materials and particle size of alumina powder on the spheroidization in the thermal spray process was studied. The high flame burner with oxygen and LPG was used to increase the flame temperature of thermal spray. By using the scanning electron microscope, the spherical alumina observed microstructure. And the spherical alumina was characterized by XRD and BET.

J25 Effects of Doping Elements on Residual Oxygen/Nitrogen Contents in Red Pigment of Tantalum Nitrides (Ta<sub>3</sub>N<sub>5</sub>): Park Eun-young<sup>1</sup>; *Pee Jae-Hwan*<sup>1</sup>; Kim YooJin<sup>1</sup>; Cho Woo-Seok<sup>1</sup>; <sup>1</sup>Korea Institute of Ceramic Engineering and Technology

The cadmium group pigment which is the toxic material is restricted by the RoHS. Especially the tantalum nitride synthesized from TaCl<sub>5</sub> or Ta<sub>2</sub>O<sub>5</sub> by nitridation process show red color tone. In this research, we prepare the amorphous tantalum precursor with fine particle size rather than Ta<sub>2</sub>O<sub>5</sub> in order to improve the nitridation rate of the starting material. Also the effect of doping on the red color value and nitridation rate of Tantalum based materials was studied. Various fine particle size of tantalum precursor with doping elements was prepared in the neutralization process with the starting materials of tantalum chloride. In the nitridation process, tantalum precursor with various doping elements were set in the tube furnace with the N<sub>2</sub> and NH<sub>3</sub> gas flowing. The formation temperature of tantalum nitride (Ta,N,) was set at 1,000°C for 5h. The synthesized tantalum nitrides were characterized by CIE Lab colorimeter to evaluate color value. A crystalline of synthesized powder was analyzed by the XRD. The microstructure and morphology was observed with the SEM. The residual content of nitrogen / oxygen in the synthesized powder was analyzed by O/N analyzer. Also thermal decomposition behavior of tantalum nitride was characterized by the TG-DTA.

J26 Electronic Spin States in Novel Superconducting Arsenides: Svetlana Kharlamova<sup>1</sup>; Sergey Bud'ko<sup>2</sup>; Stanislav Sinogeikin<sup>3</sup>; Alexander Goncharov<sup>1</sup>; Viktor Struzhkin<sup>1</sup>; <sup>1</sup>Carnegie Institution of Washington; <sup>2</sup>Ames Laboratory, Iowa State University; <sup>3</sup>Carnegie Institution of Washington and Argonne National Laboratory

The one of the most interesting topics in condensed matter physics is relation between superconductivity and magnetism, dielectric and bonding properties of solids. Novel iron arsenides AFeAsO<sub>1-x</sub>Fx (A=La, Ce, Sm, Pr, Nd, Sr, Ba, etc.) are considered as a second important class of high-Tc superconductors. They possess extremely interesting electronic and magnetic properties. To reveal the evolution of superconductivity and magnetism, and to investigate the interplay between these two collective phenomena, electronic structures and spin states of these materials at extreme conditions we performed high-pressure low temperature studies of the SrFe<sub>2</sub>As<sub>2</sub>, CaFe<sub>2</sub>As<sub>2</sub>, and Fe<sub>2</sub>TiO<sub>4</sub> at high-pressure and different temperatures using X-ray emission spectroscopy and X-ray diffraction method. I will present the results of new research findings.

## **J27 Fabrication of Niobium Powder for Solid Electrolyte Capacitors**: *Jae Sik Yoon*<sup>1</sup>; Man Bok Park<sup>2</sup>; Dong Ju Yoon<sup>2</sup>; Byung Il Kim<sup>2</sup>; <sup>1</sup>Korea Basic Science Institute; <sup>2</sup>Sunchon National University

Niobium powder was fabricated by sodiothermic reduction process using  $K_2NbF_7$  as a raw materials, KCl and KF as the diluents and sodium as a reducing agent. The apparatus for the experiment was designed and built specifically for the present study. Varying properties of niobium powder depending on reaction temperature and excess of reducing agent were analyzed. The niobium particle size increased significantly as reduction temperature increased from 993K to 1093K. The particle size was fairly uniform at given reaction temperature, varying from 0.2 $\mu$ m to 50nm depending on the reaction temperature. The yield of niobium powder increased from 58% to 83% with a increase in reaction temperature. The average particle size of niobium powder was improved from 70nm to 0.2 $\mu$ m with increase in the amount of sodium excess. In addition, the yield of niobium powder was 82% in the 5% sodium excess.



J28 Grain Boundary-Dependent Selection Criteria for Nucleation of Gamma-Massive Grains in TiAl-Based Alloys: *Ananthi Sankaran*<sup>1</sup>; Emmanuel Bouzy<sup>2</sup>; Matthew Barnett<sup>1</sup>; Alain Hazotte<sup>2</sup>; <sup>1</sup>Deakin University; <sup>2</sup>Paul Verlaine Université

Rapid cooling of TiAl-based alloy from alpha phase (disordered hexagonal, A3) generates  $\gamma$  phase (ordered tetragonal, L1o) grains mostly over the a/a grain boundaries through massive transformation. The current work deals with the identification and the validation of different nucleation mechanisms during  $\gamma$ massive transformation in TiAl-based alloy. Special attention has been given to the variant selection criteria for the nucleation of the massive structures along different types of a/a grain boundaries. The  $\gamma$  massive domains formed along the grain boundaries were analysed using high resolution electron backscattered diffraction (EBSD). Statistical studies were made on different nucleation sites and different mechanisms are proposed. Two-dimensional studies of the nucleation mechanism show that the minimization of the interfacial energy is the predominant criteria during the grain boundary nucleation. In order to verify this nucleation criterion in three-dimensions, serial sections were made and EBSD maps were taken and analysed in each section. The variant selection observed during the nucleation and the growth of the  $\gamma$  massive grains is further discussed after getting a broader view under three dimensional investigations. Similar fast quenching experiments are done on beta-Ti alloys and their results are investigated.

J29 Grain Growth of Cu-Al-Mn Shape Memory Alloy: *Tomoe Kusama*<sup>1</sup>; Toshihiro Omori<sup>1</sup>; Yuji Sutou<sup>1</sup>; Ryosuke Kainuma<sup>2</sup>; Kiyohito Ishida<sup>1</sup>; <sup>1</sup>Graduate School of Engineering, Tohoku University; <sup>2</sup>The Institute of Multidisciplinary Research for Advanced Materials, Tohoku University

Polycrystalline Cu-Al-Mn shape memory (SM) alloys with low Al composition of about 17 at.% show an excellent ductility and SM properties. Sutou et al. reported that the superelasticity of the Cu-Al-Mn alloys strongly depends on the grain size relative to the size of specimens and that the superelastic strain increases with increasing the relative grain size. Therefore, the grain growth is key information for the microstructural control in Cu-Al-Mn SM alloys. In this study, the grain coarsening of Cu-Al-Mn SM alloys was investigated. Cu-17Al-11.4Mn sheet specimens were solution-treated at 800 °C or 900 °C in the  $\beta$  (bcc) single phase region for various periods, and the rate of grain growth at each temperature was determined. Moreover, secondary recrystallization was observed by the combination of precipitation of the  $\alpha$  (fcc) phase at temperatures between 500 °C and 700 °C and dissolving it at 900 °C. In these specimens, the grain size larger than 1 mm was obtained and this technique is considered to be effective for enhancement of the SM properties of the Cu-Al-Mn alloys.

### **J30 HRTEM Study of TiO<sub>2</sub> Photocatalysis Process**: Jun Zhang<sup>1</sup>; *Chunxu Pan*<sup>1</sup>; <sup>1</sup>Wuhan University

The microstructural evolution of P25 (TiO<sub>2</sub>) in a photocatalysis process for the degradation of methylene blue was study by using a high-resolution transmission electron microscopy (HRTEM). Four samples were examined including the pristine P25 nanoparticles, after adsorption of methylene blue in dark environment, after degradation of methylene blue under UV light irradiation, and exposure in air for one month. It was found that the crystal lattice varied as follows: 1) the pristine TiO, nanoparticles exhibited a perfect lattice; 2) after the adsorption and degradation, many methylene blue crystal as 1 nm nanodot were observed on the surface of TiO, particles, simultaneously, the TiO, lattice became fuzzy; 3) when the TiO, was exposure in air for one month, the methylene blue nanodot disappeared and the TiO, lattice again became integrated as the pristine one. The results revealed that the adsorption of methylene blue nanodot induced the lattice distortion in a region of several atom layers on the surface of TiO. nanoparticle which essentially increase surface chemistry energy and enhance surface chemistry action of TiO<sub>2</sub> for promoting the photocatalysis process. When the methylene blue was completely degraded, the methylene blue nanodot disappeared from the TiO<sub>2</sub> surface.

### **J31 Precipitation Behavior in AL**<sub>6</sub>**XN Austenitic Stainless Steel**: *H Xing*<sup>1</sup>; L J Meng<sup>1</sup>; J Sun<sup>1</sup>; <sup>1</sup>Shanghai Jiao-tong University

The austenitic stainless steels have been investigated as potential materials used for reactor core components of supercritical water-cooled reactor because of their excellent stress-corrosion resistance and high strength at elevated-temperature. However, precipitation of second phases may occur in the steel at elevated temperatures, which causes loss of toughness and ductility and reduction of corrosion resistance by removing alloying elements in the matrix. In this work, the precipitation behavior in the AL<sub>o</sub>XN austenitic stainless steel after solution treatment has been studied by electron microscopes. The steels were aged at temperatures 500~750°C for a long period up to 3600 hours. The results showed that second phase precipitations were hardly observed at 500~550°C. The precipitation of carbides mainly occurs at grain boundaries at 600°C. When the temperature increasing to 650°C, high density precipitates were found both at grain boundaries and within the grains. The electron diffraction and energy-

dispersive spectroscopy analyses confirmed that the precipitates are the sigma phase. The similar analyses were also performed in the steel aged at 700~750°C. Additionally, the phase diagrams calculations were carried out by Jmatpro software for the  $AL_oXN$  steel and the theoretical results are discussed with the experimental results in this work.

#### J32 Shape-Controlled Syntheses of Silver Nanoparticles: Role of the Seeds:

Chuyang Chen<sup>1</sup>; Xuchuan Jiang<sup>1</sup>; Aibing Yu<sup>1</sup>; <sup>1</sup>University of New South Wales Noble metal nanoparticleshave attracted considerable attention because of their unique properties (optical, electronic and chemical properties) and potential applications in many areas such as optical probes, and surface enhanced Raman Spectrum. Despite many success in synthesis of anisotropic nanoparticles (rods, plates), one of the principal problems in the seed-mediated synthesis of noble metal nanomaterials still exists, i.e., the mixed shapes of the synthesized particles. This is supposedly due to the coexistence of diverse seed structures, including single, twinned and multiply twinned structures, which could convert to each other if sufficient energy is provided or surface modification is satisfactory for the conversion conditions. This study demonstrates a facile and efficient seedmediated method to prepare silver nanoparticles with desired morphologies and sizes in aqueous solution. This can be achieved by modification of the asproduced silver seeds through heat treatment, oxidative etching, and digestive ripening. The shape and size of the generated particles will be characterized by advanced techniques (TEM, HRTEM), and the particle formation and growth is tracked by UV-vis spectrometry. The role of the seeds in the particle formation and growth will be discussed. This strategy would be useful for the shape-controlled synthesis of metal nanoparticles for desired functional properties.

## J33 Testing of Aluminium Carbide Formation in Hall-Heroult Electrolytic Cell: Piotr Palimaka<sup>1</sup>; *Stanislaw Pietrzyk*<sup>1</sup>; <sup>1</sup>AGH University of Science and Technology

The trend in the aluminium smelting industry today is to operate cells with graphitized carbon cathode linings, increased current density and acidic bath chemistry. The resulting problem is an accelerated wear of graphitized cathode blocks, thougt to be caused by formation and subsequent dissolution of aluminium carbide at the cathode lining surface. The cycle of formation and subsequent dissolution aluminium carbide is recognized as one of the most important mechanism causing pathole and surface wear, which results in limiting of the cell liftime and loss efficiency. A special laboratory test method was developed to elucidate the mechanism of aluminium carbide formation in electrolytic cell. The aluminum carbide formation in the region between the carbon surface and aluminium as well as between the carbon surface and electrolytic bath has also been studied using X-ray diffraction, as well as optical and scanning electron microscopy. Solid aluminium carbide layer was observed at the carbon surface. A possible mechanism which explains the presence of aluminium carbide at the metal-bath interface is the transfer of dissolved aluminium carbide in the bath from metal-carbon interface.

#### **J34 Texture Distribution and Development through the Thickness of Grain Oriented Silicon Steel**: *Zhi Fen Wang*<sup>1</sup>; <sup>1</sup>Research and Development Center, Wuhan Iron and Steel (Group) Corporation

The texture distribution and development of grain oriented silicon steel with different production process have been investigated by electron backscatter diffraction and ODF technique. The results indicated that below the surface the intensity of {001}<110> a-fiber was strongest, and {110}<001> texture and  $\alpha$ -fiber were weaker. After cold rolling and second normalizing, in the corresponding position the {110}<001> texture and  $\alpha$ -fiber appeared again. The {110}<001> grains and  $\alpha$ -fiber after decarburizing annealing was helpful to the abnormal growth of second grain nucleation.

#### J35 The Characterisation of Bauxite Residue Treated with High Power Ultrasound and Magnetic Separation Process: *Yesim Gozukara*<sup>1</sup>; A. D. Farmer<sup>1</sup>; Paul Gwan<sup>1</sup>; <sup>1</sup>CSIRO, Materials Science & Engineering

The possibility of using high power ultrasound to break down the red mud gelation and the bonding of clay and iron particles to allow magnetic or alternative separation of a substantial haematite fraction from the waste has been investigated in this work. The combination of ultrasonics and wet high intensity magnetic separation, has produced good recovery and quality of  $Fe_2O_3$ , substantially reducing the waste volume and, simultaneously producing useful high- and low-iron fractions. The concentration of five metals present in these treated red mud samples were analysed by ICP-AES with primary elements of interest being aluminium and iron. Determination of new methods of fusion and microwave assisted digestion techniques were also investigated. Both methods were found to give good recoveries and good precision in the analysis of the standard material with microwave technique being considerable faster than the fusion method. We report here on the underlying physics that offers industrial promise and the analysis process required to deal with a very difficult material.



J36 Thermo-Mechanical Behavior of ITO Layer on PET Substrate for Rollto-Roll Process with Varying Thermal Conditions: Jae Oh Bang<sup>1</sup>; *Hyo-Soo Lee*<sup>1</sup>; <sup>1</sup>Korea Institute of Industrial Technology

The PET-ITO substrate has been usually applied to many flexible applications such as flexible printed circuit, display panels etc., where it has a main role to enhance the performance in the components of touch screen panels, solar cell panels and so on. Today's flexible technology is issued on forming fine pattern, pattern alignment and mass-productivity on PET-ITO substrates, which is strongly related with thermal shrinkage and expansion of the substrate. Many studies have been more focused on the subjects such as ITO deposition process, ITO crack propagation rather than thermo-mechanical behavior of ITO layer on PET substrate, where the thermo-physical properties of PET-ITO substrate are main key factor decreasing failure cost of roll-to-roll process. In this study, we used bare PET, PET-ITO and finely patterned PET-ITO for analyzing thermal strain quantitatively under temperatures and constant load ranging 1~10N by micro tensile machine. It was observed that the ITO layer on PET substrates decreased thermal strain of PET-ITO substrate dominantly with increasing volume fraction of ITO layer. Therefore, we could suggest reasonable values on thermal strain of PET-ITO substrate by approaching experimental works, which was expected to be applied to so many PET-ITO products with different design of ITO patterns.

### **J37 Thermodynamic Evaluation of the Fe-Pr Binary System**: *Guojun Zhou*<sup>1</sup>; Dechang Zeng<sup>1</sup>; <sup>1</sup>South China University of Technology

The Fe–Pr binary system was thermodynamic evaluation by means of the CALPHAD method based on phase diagram experimental data from the literature and a few values of the mixing enthalpy in the liquid phase obtained by the Miedema theory technique. Each of the selected data values is given a certain weight, which is chosen and adjusted based on the thermodynamic data and diagram phase data. A consistent thermodynamic description of the Fe–Pr binary system is presented: only one intermediate compound, Pr2Fe17, is stable in the system and forms peritectically at 1105°C. An eutectic reaction L↔Pr+Pr2Fe17 occurs at 667°C and the eutectic liquid contains 82 at% Pr, five solid solution phases (Fe-rich  $\alpha$ Fe,  $\gamma$ Fe and  $\delta$ Fe, Pr-rich  $\alpha$ Pr and  $\beta$ Pr) and the liquid solution phase were considered in the evaluation. The intermediate phase was treated as stoichiometric compound, the solid solutions as ideal and the liquid solution phase by the Redlich–Kister formalism. The calculated phase diagram and thermodynamic properties are in good agreement with available experimental data.

#### J38 A Numerical Prediction of the Forming Limit Diagram of the Magnesium Alloy Considering Damage Evolution from Void Growth: *Jung-Han Song*<sup>1</sup>; Geun-An Lee<sup>1</sup>; Hye-Jin Lee<sup>1</sup>; Kyoung-Tae Kim<sup>1</sup>; Sung-Min Bae<sup>2</sup>; <sup>1</sup>KITECH; <sup>2</sup>Hanbat University

Magnesium alloy is widely used in automobile parts as well as electronic and communication devices for its ultra light weight It is important to predict and evaluate the formability of magnesium alloy. Recently, researches have been vigorously carried out to identify mechanical properties of magnesium alloy both experimentally and numerically. However, it still remains a difficult problem to predict the formability because conventional computational simulation of the forming process has some limitations with the material model.In this paper, the novel fracture criterion is utilized to predict the FLD in conjunction with finite element analyses for sheet forming. The principal scheme of the fracture criterion in this paper is that growth of the micro voids leads up to fracture in viewpoint of micro-mechanics. The numerical FLD is verified by results of the out-of plane stretching test using hemispherical punch. The verification is also conducted from the experiment. The forming limit diagram obtains from the numerical results shows in close coincidence with the one obtained from the experiment. Especially, the purposed scheme is appropriate to predict FLDs for a restricted material with low ductility after instability point or ultimate tensile strength.

#### **J39 Preparation and Characterization of (1-x)PZT-xBNbT Ceramics**: *Navavan Thongmee*<sup>1</sup>; Anucha Watcharapasorn<sup>1</sup>; Sukanda Jiansirisomboon<sup>1</sup>; <sup>1</sup>Chiang Mai University

Lead-based ferroelectric materials is Pb(Zr, Ti)O<sub>3</sub> (PZT) and lead-free ferroelectric material based on bi-layered perovskite structure is Bi<sub>4</sub>Ti<sub>3</sub>O<sub>12</sub> (BIT) are two important materials often employed in non-volatile random access memories (NvFeRAM). PZT having a perovskite structure is widely known as one of the most important ferroelectric material. However, PZT still has several problems, especially severe polarization fatigue after bipolar switching pulses. Recently, the researchers studied ferroelectric properties of Nb<sup>5+</sup>-substituted into B-site of BIT ceramics. The results showed that 2P<sub>r</sub> and 2E<sub>c</sub> of Bi<sub>4</sub>Ti<sub>3</sub>O<sub>12</sub> doped with 3 mol% niobium (BNbT) are higher than those of BIT pure. It also showed good switching endurance under bipolar pulse and improved ferroelectric properties by reducing defects such as oxygen vacancies. This present study aims to fabricate and characterize a series of new-complex structured ceramic with formula (1-x)Pb(Zr<sub>0.5</sub>Ti<sub>0.4x</sub>)O<sub>5</sub>-xBi<sub>3.90</sub>Ti<sub>2.97</sub>Nb<sub>0.03</sub>O<sub>12</sub> or (1-x)PZT<sub>x</sub>BNbT (when

x = 0, 0.1, 0.3, 0.5, 0.7, 0.9 and 1.0) using a solid-state mixed oxide method and sintered at temperatures in between 950-1050°C. Densities of the ceramics were measured using Archimedes' method. Phase analysis was done using an Xray diffractrometer (XRD). Microstructure investigation was carried out using a scanning electron microscope (SEM).

J40 Property Evaluation of Cu Sputtering Target Prepared by Spark Plasma Sintering Process: *Seung Min Lee*<sup>1</sup>; Hyun Kuk Park<sup>1</sup>; Jun Mo Yang<sup>2</sup>; Se Weon Choi<sup>1</sup>; Kee Do Woo<sup>3</sup>; Ik Hyun Oh<sup>1</sup>; <sup>1</sup>Korea Institue of Industrial Technology(KITECH); <sup>2</sup>National Nanofab Center; <sup>3</sup>Chonbuk National University

Sputtering target is a material that used for physical vapor deposition and laser of electron beam deposition processing. It is one of the key material for making a micro-electrode and wiring at a manufacturing process of semiconductor and display. Efficiency of target is important factors: density, grain size, composition and purity that have influence with the characteristics of the thin film. High density and microstructure of Target decrease arc discharge which occur nonuniform of the thin film and particle while sputtering process. Spark Plasma Sintering is possible to sinter at low temperature and short time because of activating the powder surface applying a high-voltage pulse current between gaps in powder. It has advantages that fabrication of microstructure compact and high densification therefore, SPS has been studying and applied on the development of the target material recently. The objective of this research is to investigate the microstructure and mechanical properties of Cu compact accompanied by temperature, time and pressure for the fabrication of Cu sputtering target by optimized SPS process. Sintering process is progressed at 10~60MPa, 600~900°C in a vacuum atmosphere and analyzed purity, density, structure of compact by FE-SEM, TEM, SIMS.

J41 The Laser Surface Remelting of Austenitic Stainless Steel: *Zbigniew Brytan*<sup>1</sup>; Miroslaw Bonek<sup>1</sup>; Leszek Adam Dobrzanski<sup>1</sup>; D. Ugues<sup>2</sup>; M. Actis Grande<sup>2</sup>; <sup>1</sup>Silesian University of Technology; <sup>2</sup>Politecnico di Torino

The laser surface remelting (LSR) process was successfully applied to restore localized corrosion resistance in sensitized stainless steel and also as a useful method to improve passivity of some martensitic stainless steels. The LSR process can be successfully applied to repair cracks and defects at the surface of highly thermo-mechanically loaded parts of stainless steel. The purpose of presented study was to evaluate the microstructure and properties of laser surface remelted stainless steels. The wrought austenitic stainless steel and sintered in vacuum type 316L were studied. The laser treatment was performed by the high power diode laser (HPDL) and the influence of beam power of 0.7-2.1kW on the properties of surface layer was evaluated. The geometrical characteristics of weld bead were studied and the x-ray analysis as well as microhardness, surface roughness and corrosion resistance were measured. The increase of laser beam power of LSR process resulted in hardness growth of sintered stainless steel due to porosity reduction and fine dendritic and cellular-dendritic microstructure formation. The corrosion resistance of remelted surface was increased for sintered material when remelted at 2.1kW. The wrought stainless steel revealed impairment of pitting corrosion when remelted at lower beam power.

## J42 The Microstructure and Properties of Ni-Based Superalloy after Service Exposure in Gas Turbine: *Keun Bong Yoo*<sup>1</sup>; Hansang Lee<sup>1</sup>; <sup>1</sup>Korea Electric Power Research Institute

Many investigations about superalloys and coatings have been done in the laboratory, but evaluating the degradation condition of hot section components during service is still important not only for repair and reuse but also for outage prevention. Time dependent degradation of blades for gas turbine was investigated. The degradation analysis for used blades was divided into microstructure changes by position of the blade and mechanical test of high temperature tensile test. In the micrstructure analysis, the rafting and coarsening of  $\gamma$ ', MC decomposition and TCP phase formation occurred and progressed with increasing service time, and especially the leading and trailing edge of top layer should be a check points for used blade. High temperature tensile test results of 25,000 and 52,000 hrs used blades were also compared with service time and position in each blade.

**J43** A New Micromechanical Elasto-Plastic Constitutive Model for Fiber-Reinforced Composite Laminates: *Y.X. Zhang*<sup>1</sup>; H.S. Zhang<sup>2</sup>; <sup>1</sup>The University of New South Wales at the Australian Defence Force Academy; <sup>2</sup>China Academy of Aerospace and Aerodynamic

Fiber-reinforced composite laminates are regarded as promising materials in many industry areas, especially for aerospace applications due to their superior materials properties. To provide accurate prediction of the structural behavior and damage evolution and progressive failure process of fiber-reinforced composite laminates, an effective micromechanics constitutive model which can account for material nonlinearity of the composites is required. A micromechanical elasticplastic bridging constitutive model is developed in this paper for accurate analysis of fiber-reinforced composite laminates. In the bridging constitutive model,



bridging matrix elements are used to represent the elastic behavior. The bridging matrix elements are deduced based on the Equivalent Inclusion Average Stress Method and the Mori-Tanaka theory, and the interaction between the average stresses in resin with those in fibers are included. A transient plastic bridging matrix is then developed to describe accurately the elastic-plastic material properties of the fiber reinforced composite, and the effects of the material parameters of resin and fiber on the bridging matrix elements are considered. The micromechanical elastic-plastic bridging constitutive model is used to analyse CFRP/GFRP-reinforced resin/epoxy composite laminates. The agreement of the numerical analysis results with those obtained from experiment investigation demonstrates the efficiency and accuracy of the proposed model.

#### Poster Session: Symposium K: Composites and Hybrid Materials

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#### K1 Anatase-Type Ti<sub>1-2x</sub>Nb<sub>x</sub>Al<sub>x</sub>O<sub>2</sub> Solid Solution / Silica (SiO<sub>2</sub>) Composite Nanoparticles: Synthesis, Phase Stability, and Photocatalytic Performance: *Masanori Hirano*<sup>1</sup>; Norio Kuno<sup>1</sup>; <sup>1</sup>Aichi Institute of Technology

Composite nanoparticles composed of amorphous silica and anatase-type titania solid solutions co-doped with niobium and aluminum  $(Ti_{1-2X}Nb_{X}Al_{X}O_{2})$ were directly synthesized from precursor solutions of TiOSO<sub>4</sub>, NbCl<sub>5</sub>, Al(NO<sub>3</sub>)<sub>3</sub>, and tetraethylorthosilicate under mild hydrothermal conditions at 180°C for 5 h using the hydrolysis of urea. The crystallite size of anatase was gradually decreased with increased silica content in the composite nanoparticle formed under basic hydrothermal condition. Their photocatalytic activity and adsorptivity were evaluated separately by the measurement of the concentration of methylene blue (MB) remained in the solution in the dark or under UV-light irradiation. The composite nanoparticles with composition  $\text{Ti}_{0.9}\text{Nb}_{0.05}\text{Al}_{0.05}\text{O}_2$  solid solution/SiO<sub>2</sub> =100/10 showed good photocatalytic activity. By the presence of amorphous silica, crystallite growth of anatase after heating in air was also suppressed to maintain the crystallite size of the as-prepared condition. The phase stability of anatase-type solid solutions was improved by the formation of composite nanoparticles with amprphous silica. The anatase-to-rutile phase transformation was retarded to maintain the anatase-type structure up to more than 1000°C without a trace of rutile phase.

#### K2 Development and Applications of the Self-Organized Composite Cu-Based Alloy Powders: *Cuiping Wang*<sup>1</sup>; Yan Yu<sup>1</sup>; Xingjun Liu<sup>1</sup>; Ikuo Ohnuma<sup>2</sup>; Ryosuke Kainuma<sup>2</sup>; Kiyohito Ishida<sup>2</sup>; <sup>1</sup>Xiamen University; <sup>2</sup>Tohoku University

The self-organized composite alloy powders were designed on the basis of the Cu-based liquid immiscible alloy systems by using the CALPHAD (CALculation of PHAse Diagrams) method, and then were fabricated by conventional gas atomization technique under gravity conditions. The liquid phase with minor volume fraction always forms the core part of the self-organized composite alloy powders. The result shows that the formation of the core-type composite microstructure is strongly connected with the existence of a stable miscibility gap of the liquid phase in the Cu-based immiscible alloys, and can be explained by a mechanism that the minor droplet as the second phase are forced to move to the thermal center due to Marangoni motion, which is caused by the temperature dependence of interfacial energy between two immiscible liquids. The Cu-Bi-Sn and Cu-Ag-Ni composite alloy powders with core-type microstructure show promising applications in the field of BGA package and conductive filler.

#### K3 Effect of Interfacial Reaction on High Temperature Properties of Fe-Cr-Si Fiber Reinforced AC8A Aluminum Composites: *Nobuyuki Fuyama*<sup>1</sup>; Akira Terayama<sup>1</sup>; Toshio Fujii<sup>1</sup>; Tohru Shiraishi<sup>2</sup>; Yuki Miyake<sup>3</sup>; Gen Sasaki<sup>4</sup>; <sup>1</sup>Hiroshima Prefectural Technology Research Institute; <sup>2</sup>NHK Spring Co., Ltd.; <sup>3</sup>Kolbenschmidt K.K.; <sup>4</sup>Hiroshima University

As for a metallic fiber (Fe-Cr-Si) with excellent high temperature strength, use as a reinforced material of the engine piston head is expected. However, the high reactivity of Al matrix with most metals has disturbed the use of metallic fibers in aluminum composites until now. In this study, the influence of the reaction products at the fiber/matrix matrix on the high temperature properties of the composites was investigated by different solution treatment conditions. As a result, hardness and strength increased with an increment of the temperature of solution treatment (Tst). Reaction products (Al-Fe inter metallic compounds) by solution treating were formed along the fiber/matrix interface at 773K or more. The composites without interfacial reaction products (Tst=763K) showed excellent rotating-bending fatigue life at 573K. The fatigue crack propagation of

this composite occurred at the necking region of metal fiber because no cracks were observed in the interfacial reaction products.

K4 Effects of Deformation-Induced Heating on Bond Strength of Rolled Metal Multilayer: *Dengke Yang*<sup>1</sup>; Peter Hodgson<sup>1</sup>; Cui'e Wen<sup>1</sup>; <sup>1</sup>Deakin University

The bond strength of various metal multilayers produced by cold rolling of metal foils with different thermal conductivity was investigated. Results indicated that under the same conditions of deformation and surface preparation, the metallic multilayer system with low thermal conductivity exhibited relative high bond strength while high thermal conductivity metal system may fail to be roll-bonded together. The relationship between the deformation-induced localized heating and the bond strength were discussed. The deformation-induced localized heating in the low thermal conductivity metal multilayer systems may provide opportunities for achieving a successful accumulative roll bonding or a "cold roll/heat treatment/cold roll" process to synthesize metallic multilayer materials.

K5 Fabrication and Properties of a Combined Structural Cu Sheet for Interconnect Material: *Je Sik Shin*<sup>1</sup>; Hyung Kwon Moon<sup>2</sup>; Bong Hwan Kim<sup>1</sup>; Hyo Soo Lee<sup>1</sup>; Hyouk Chon Kwon<sup>1</sup>; <sup>1</sup>Korea Institute of Industrial Technology; <sup>2</sup>Sungkyunkwan University

With the increasing demand for higher performance packages in microelectronics industry, Cu alloys are now widely accepted as a novel interconnect material to replace Al alloys due to their lower electrical resistivity and higher heat dissipation capability and electromigration resistance. For a high integration device, the manufacturing of Cu-base materials, which have simultaneously both high electrical conductivity and high strength, is desirable. These properties generally do not coexist in the same material. In this study, it was tried to solve this problem by developing a combined structural Cu sheet. The combined structural Cu sheet was fabricated by forming the high electrical conduction paths of Ag on the surface of a high strength thin film matrix of commercial Cu alloy by damascene electroplating process. As a practical application, the manufacturing of a leadframe for LED module was tried and the machinability and performance of the combined structural Cu leadframe were systematically evaluated.

K6 Interfacial Characteristics of WC Particles Reinforced Hadfield Steel Matrix Composites: *Guoshang Zhang*<sup>1</sup>; Yimin Gao<sup>2</sup>; Jiandong Xing<sup>2</sup>; Shizhong Wei<sup>1</sup>; Jiwen Li<sup>1</sup>; Liujie Xu<sup>1</sup>; <sup>1</sup>Henan University of Science and Technology; <sup>2</sup>Xi'an Jiaotong University

In order to improve the wear resistant properties, WC ceramic particles were used to reinforce the wear surface of Hadfield steel. WCp/Hadfield steel composites were fabricated by optimized solid state sintering process of powder metallurgy. Interface structure, constituent phase and the forming mechanism of the composites were investigated systemically. The results showed that: The WCp/Hadfield steel composites, with uniformly distributed particles and well bonding interface between particles and Hadfield steel, were obtained by optimized solid state sintering process of powder metallurgy. In the WCp/Hadfield steel composites, the interface between WC particles and Hadfield steel matrix is shelly shape, in which W, Fe and Mn elements diffuse between two phase. The interface is a metallurgical bond, in which a new phase, namely Fe3W3C is formed. The micro-hardness of the interface layer is between that of WC and the steel matrix, which can provide a guarantee for the property transition between WC particles and Hadfield steel matrix. The diffusion reaction mechanism of the interface was also systematically studied.

### K7 Interfacial Reaction and its Effection on the Hot-Pressed WCp/2024Al Composite: Qiushi Liang<sup>1</sup>; <sup>1</sup>General Research Institute for Nonferrous Metals

12vol.%WCp/2024Al composites were fabricated by hot-pressing of the powder mixtures at different temperature. The investigation of the interfacial reaction between WC phase and Al alloy matrix was performed by XRD, TEM and EDS. A multiple layers interface structure, which is composed of Al/WAl<sub>12</sub>/Al<sub>4</sub>C<sub>3</sub>/WC, was found to be formed by the interfacial reaction products of the hot-pressed composite. Further research shows that the Al<sub>4</sub>C<sub>3</sub> layer has the effect of slowing down the interfacial reaction of the WCp/2024Al composite during hot-pressing. The interfacial reaction is harm for mechanical property of the WCp/2024Al composites.

K8 Superconductive Property of MgB<sub>2</sub> Particle-Dispersed Mg-Based Composite Material: *Shimizu Yusuke*<sup>1</sup>; Kenji Matsuda<sup>1</sup>; Manabu Mizutani<sup>1</sup>; Katsuhiko Nishimura<sup>1</sup>; Tokimasa Kawabata<sup>1</sup>; Yoshimitsu Hishinuma<sup>2</sup>; Shigeki Aoyama<sup>3</sup>; Susumu Ikeno<sup>1</sup>; <sup>1</sup>University of Toyama; <sup>2</sup>National Institute for Fusion Science; <sup>3</sup>Nikkei Niigata

As has been known, MgB<sub>2</sub> is the Type II superconductor, and its critical temperature of superconducting transition (Tc) is 39 K. MgB<sub>2</sub> has the highest Tc in the intermetallic compound superconductive materials. We used the original method of the three-dimensional penetration casting (3DPC) in this laboratory to fabricate the MgB<sub>2</sub>/Al composite. In the composite material we made, MgB<sub>2</sub>



particles dispersed to the Al matrix uniformly. The Tc was determined by electrical resistivity and magnetization to be about 37~39K. In this work, we have chosen Mg or Mg alloy for the matrix. These Mg- based MgB<sub>2</sub> composite materials have been also fabricated by the 3DPC method combined with semisolid casting (SS-3DPC), and those composite materials have shown clear signals of Tc. Critical current density (Jc) of those composite materials has been calculated from the width of the magnetic hysteresis based on the extended Bean model. Microstructures of these samples have been confirmed by TEM and SEM method.

#### K9 A Study on Improvement of Fatigue Life for Woven Glass Fabric/Epoxy Laminate Composite Applied to Railway Vehicle: *Hee-Young Ko*<sup>1</sup>; Kwang-Bok Shin<sup>1</sup>; Jung-Seok Kim<sup>2</sup>; <sup>1</sup>Hanbat National University; <sup>2</sup>Korea Railroad Research Institute

In this study, the fatigue characteristics and life of woven glass fabric/epoxy laminate composite applied to railway vehicle was evaluated. The fatigue test was conducted by tension-tension load with stress ratio R of 0.1 and frequency of 5Hz. The material used to fatigue test was two types of woven glass fabric/epoxy laminate composite with and without the reinforcement of carbon/epoxy ply. Also, the fatigue life of woven glass fabric/epoxy laminate composite with 6005 used to the carbody and underframe structures of railway vehicle. The test results showed that the failure strength and life of woven glass fabric/epoxy laminate composite with that of aluminum 6005 used to the carbody and underframe structures of railway vehicle. The test results showed that the failure strength and life of woven glass fabric/epoxy laminate composite with the reinforcement of three carbon/epoxy plies had a remarkable improvement in comparison with that of bare specimen without reinforcement.

#### K10 A Study on Structual Strength in Body Structure of Composite Material: Sung Cheol Yoon<sup>1</sup>; Joon Hyung Ryu<sup>1</sup>; Jeongguk Kim<sup>1</sup>; <sup>1</sup>Korea Railroad Research Institute/Railroad Safety Research and Testing Center

This study introduces the Structural Analysis and testing results of the composite Structure which is applied to tilting train in Korea. As problems like finding energy saving cars and looking for ways to reduce vehicle repair and maintenance costs continue to happen, studies on lightening vehicle structures to lower production expenses of the vehicle itself have been continuously promoted. The introduction of composite materials are welcomed in the market and will even likely be enjoyed by the next generations since they have the appropriate strength, as well as the lighter weight. The new composite materials, which became popularly used as car materials since the late 1990's has a huge effect on the vehicle's weight, has superior durability and erosion resistance, has the power to reduce manufacturing costs that are usually driven by the monolithic molding structure, and many other advantages that railroad cars locally and abroad can benefit from. The composite Structure is made of aluminum honeycomb structure materials like a sandwich. This study aims to perform a car body load test to assess its structural safety.

#### K11 Thermal Decomposition and Kinetic Study on Different Types of Glass Fiber/Unsaturated Polyester Pipe Waste: *Kaew Saetiaw*<sup>1</sup>; Duangdao Aht-Ong<sup>1</sup>; <sup>1</sup>Chulalonkorn University

Three types of unsaturated polyester matrix such as orthophthalic, isophtalic and vinyl ester reinforced with 50 wt% glass fiber laminated composite waste have been subjected to thermochemical conversion by gasification process. Investigation of thermal decomposition behavior of these composite wastes was first conducted by Thermo Gravimetric Analyzer (TGA). The result showed that major decomposition range of the unsaturated polyester matrix was from 250 to 400°C, depending on the difference types of polyester matrix. These composite wastes were then gasified in a fixed bed reactor at final temperature of 600, 700 and 800°C under nitrogen mixed with oxygen gases at a total flow rate of 100 mL/min. Solid product obtained was brittle black char containing residual glass fiber. Solid yield was decreased with increasing temperature while volatile matter dropped rapidly between temperature of 300 and 400°C then remained constant from about 500°C. On the other hand, liquid and gas yields increased with reaction temperature. Condensable liquids were mainly aromatic and oxygen containing organic compounds. Gaseous products were rich in carbon monoxide and carbon dioxide while other gases such as hydrogen and methane were found in lesser amounts

## K12 Characterization and Properties of Recycle Cellulose Fibre- Reinforced Epoxy-Hybrid Clay Nanocomposites: *Hatem Alamri*<sup>1</sup>; I.M. Low<sup>1</sup>; <sup>1</sup>Curtin University

Posters

Natural fiber reinforced composites have attracted researchers for their desirable properties of toughness, high modulus, low density, recyclability and renewability. Similarly, polymer-nano-clay composites have been widely investigated for their significant improvement in strength, modulus, impact strength, barrier properties, heat resistance and thermal stability. However, in this paper, Epoxy eco-composites reinforced with recycled cellulose fiber (RCF) and nano-clay platelets and nano-tubes have been fabricated and investigated. The influence of RCF/nano-clay dispersion on the mechanical and fracture

properties of theses composites have been characterized in terms of hardness, flexural strength, fracture toughness, indentation responses, impact-fracture and crack-growth resistance. Scanning electron microscopy (SEM) is used to study the microstructures of these materials and the fracture mechanisms.

#### K13 Effects of Carbon Black and Carbon Nanotube on Mechanical and Thermal Properties of 80NR/20SBR Composites: *Anyaporn Boonmahitthisud*<sup>1</sup>; Saowaroj Chuayjuljit<sup>1</sup>; <sup>1</sup>Chulalongkorn University

In this research, mechanical and thermal properties of natural rubber (NR), styrene butadiene rubber (SBR) and 80NR/20SBR blend filled with carbon black and carbon nanotubes (CNT) were compared. The carbon black and CNT were added into the rubbers at 3-10 phr and 0.3-1 phr, respectively. The effects of filler loadings on these properties were studied. The composites were prepared by latex compounding method. The obtained homogeneous latex was cast into thin sheet on a glass mold, air dried for 1 day and then cured at 110°C for 3 h. The cured specimens were examined for their tensile properties, tear strength, dynamic mechanical properties, thermal behaviours and morphology. The results showed that the mechanical and thermal properties of the rubbers were improved as a result of the incorporation of an appropriate amount of the fillers. The influence of CNT on the properties of the rubbers is different from carbon black, which can be ascribed to the structure and aspect ratio difference between carbon black and CNT.

#### K14 Materials Properties and Machining Characteristics of Hybrid Al<sub>2</sub>O<sub>3</sub>/ CNTs Nanocomposites for Micro-EDM: *Myung-Chang Kang*<sup>1</sup>; Hyun-Seok Tak<sup>1</sup>; Chang-Seung Ha<sup>1</sup>; Young-Keun Jeong<sup>1</sup>; <sup>1</sup>Pusan National University

Micro electro discharge machining (EDM) is an effective method of machining in fabricating micro scale structures and components regardless of any hardness of workpiece which is machined only if the material can conduct electricity. For micro EDM, first of all, micro-electrode fabrication is needed and wire electrode discharge grinding (WEDG) system is proposed for tool electrode fabrication method. This paper described the results of application of hybrid Al<sub>2</sub>O<sub>3</sub>/CNTs nanocomposites to EDM process for micro fabrication. Alumina matrix composites reinforced with CNTs were fabricated by CNT purification, mixing, compaction, and sintering processes. Four different Al<sub>2</sub>O<sub>3</sub>/CNTs nanocomposites were successfully synthesized and their relative and bulk density, electrical conductivity, hardness, X-ray diffraction profiles (XRD) and fracture toughness were evaluated in order to evaluate machining characteristics. In addition, actual micro-EDM was conducted with RC circuit and machining conditions varied to investigate the machining characteristics of machined hybrid Al<sub>2</sub>O<sub>3</sub>/CNTs nanocomposites by field emission scanning electron microscope (FE-SEM).

#### K15 A Research on Interfacial Reaction of Brazing Joint of Alumina Ceramics to Metals: *Xiangdong Ma*<sup>1</sup>; Xinyang Wang<sup>1</sup>; Jiwen Li<sup>1</sup>; Shizhong Wei<sup>1</sup>; <sup>1</sup>Henan University of Science and Technology

Brazing with active filler alloys containing some active elements, which promotes wetting of ceramics surfaces, is one of the most widely methods for joining ceramics to metals. The joints formed by brazing  $Al_2O_3$  to metal by using copper-titanium-nickel (Cu-Ti-Ni) as brazing filler were investigated. The metals/ceramics joints were produced at a vacuum level of 10-2-10-3 Pa at 1000°, using a constant holding time of 10 min. The fracture surfaces were studied both morphologically and structurally using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction analysis (XRD). In the brazing process, copper and titanium in the braze alloy form a series of reaction products. The formation of Ti<sub>3</sub>Cu<sub>3</sub>O and Ti<sub>2</sub>Ni at the interface is characteristic of these joints. The estimated free energies of formation of the Ti<sub>3</sub>Cu<sub>3</sub>O and Ti<sub>2</sub>Ni are -119kcal/mol and -245.92 kJ ~-263.78kJ/mol at 1200~1288K respectively. The highly negative values for the free energies of formation suggest that these compounds are thermodynamically stable.

K16 Crack Propagation Monitoring of DCB Composite Specimens Using Distributed Optical Fiber Sensor: *Hyuk-Jin Yoon*<sup>1</sup>; Kwang-Yong Song<sup>2</sup>; Jung-Seok Kim<sup>1</sup>; Kwang-Bok Shin<sup>3</sup>; Seung-Chul Kim<sup>1</sup>; <sup>1</sup>Korea Railroad Research Institute/Railroad Structure Division; <sup>2</sup>Chung-Ang University/Department of Physics; <sup>3</sup>Hanbat National University

Nowadays in the railway industry, composite structure is broadly applied to the car body, bogie frame and collision absorbtion structure and etc. To ensure the safety of these core composite structures, delamination characteristics must be considered first of all. The delamination, space debonding between layer and other layer, is dominated by resin and fragile part that has many possibilities of breakage due to its load bearing power that much less than the reinforced fiber. To evaluate the interlaminar fracture toughness, DCB(Double Cantilever Beam) test is normally conducted. In the conventional DCB test, test is usually conducted by observing the crack's growth with a microscope, but in this paper, new technique is tried to monitor the crack propagation in real time using optical fiber embedded in the kevlar/epoxy specimen. Crack tip position was monitored using strain distribution field data from the optical fiber and compared with the microscope



data. BOCDA (Brillouin Optical Correlation Domain Analysis) technique was used to measure the strain distribution of the optical fiber and spatial resolution, strain accuracy were about 9mm,  $\pm 20$  each.

## **K17 Design of a Composite Side Beam for the Railway Bogie Frame**: *Jung Seok Kim*<sup>1</sup>; Huk Jin Yoon<sup>1</sup>; Kwang bok Shin<sup>2</sup>; <sup>1</sup>Korea Railroad Reserch Institute; <sup>2</sup>Hanbat National University

This study explains the design, manufacturing and test for a composite beam. It has been developed to be applied to the railway bogie frame. The bogie frame is a most important component for safety of a railway vehicle. In this study, the dimensions and stacking sequence of the composite beam has been determined by parametric study and manufactured using autoclave and RTM method. The two manufactured composite beams were tested under static load of 140kN and 182kN to evaluate structural safety and stiffness. In addition, the nondestructive test using radiograph was performed to investigate the internal damage of it before and after the test.

### K18 Effectiveness of Residual Stress on Forming Copper Patterns of Printed Circuit Board: *Hyo-Soo Lee*<sup>1</sup>; Hyuk-Chon Kwon<sup>1</sup>; <sup>1</sup>KITECH

The compressed residual stress was easily relaxed with applying heattreatment for a few hours. However, we observed that the compressed residual stress of copper foil tended to be relaxed, constant, and compressed again during heat-treatment process, which is mainly considered as that the grain of copper is grown restrictively within a thin foil layer.We suggested a quantitative method for controlling grain size, grain distribution and relaxing stress of copper foil, which was very helpful for increasing an etching factor to decrease pattern width. We fabricated copper patterns with a width of 35µm and analyzed the etching factor, which was improved by about 57% from 0.90 of as-received samples to 1.41 with heating at 100°C for 1~2 hours. The residual stress of copper foil was compressed 70MPa as-received and could be relaxed to -10MPa with controlling heat treatment of 100~150°C for 1~2hrs, which is coincident with the behavior of etching factor. The copper foil with relatively lower stress showed a homogeneous microstructure, which was good to form a rectangular shape of pattern, namely, possibly to obtain higher etching factor. We could in this study implement fine pattern formation by making grains uniform and controlling residual stress through heat treatment.

#### K19 Electronic Structure of Bilayer (Fe, Ni) Metallic α-Al<sub>2</sub>O<sub>3</sub>(0001) Catalysts towards CH<sub>4</sub> Adsorption and Dissociation: *Kenneth Wong*<sup>1</sup>; Qinghua Zeng<sup>1</sup>; Aibing Yu<sup>1</sup>; <sup>1</sup>University of New South Wales

Density functional theory calculations are performed on the monometallic (Fe or Ni) bilayer modified  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>(0001) surface. Comparison has been made to their structural and electronic behaviors upon CH<sub>4</sub> adsorption and dissociation. Local density of states and frontier orbital analysis show that C-H activation proceeds through weak chemical interactions with the metallic 3d electrons. We found that electron transport within the sp and 3d type orbitals of the catalyst is important for the equilibration of the system. Such electron transport also promotes electron donation to the  $\sigma$ \*(C-H) antibonding orbital for C-H bond activation. The calculated adsorption energies showed that the CH+H intermediate is most stable on the Fe/ $\alpha$ -Al<sub>2</sub>O<sub>3</sub> catalyst and is suspect to deactivation via carburization. Furthermore, C-H bond activation is most pronounced in cases where the CH<sub>4</sub> molecule has one or two H atoms directed towards the catalyst surface.

## K20 Strength and Stiffness Estimation for Center Frame of Low-Floor Vehicle: *Yeon Su Kim*<sup>1</sup>; <sup>1</sup>Korea Railroad Research Institute

A low-floor vehicle has no steps to get on the main cabin to provide the old and the handicapped with easy access. Korean government made a plan to purchase about 10,000 low-floor vehicles for mobility enhancement of the handicapped, the old, the weak, children, passengers with their infants or old parents, and passengers with carts and strollers to use public transport until 2013. A chassis frame is composed of a front frame, a center frame and a rear frame in low-floor vehicle. Since a center frame should support passenger load varied frequently in services, it should be designed to have enough strength and stiffness. In addition, its weight should be minimized as much as possible to reduce car weight. The center frame was designed to have light-weight sandwich structure with glass fabric/epoxy resin skins, aluminum honeycomb cores and steel inner-frames. For the designed center frame, in this paper, regression equations for maximum equivalent stresses (Von-Mises stress) and maximum vertical deflection were proposed by finite element analysis, and discussed by comparison with the results of static load test. On the basis of the results, the strength and stiffness were evaluated in composite materials part and steel part of the center frame.

### **K21** Study on Mechanical and Conductivity of Geopolymer Composites: *Cui Xuemin*<sup>1</sup>; <sup>-1</sup>Guangxi University

As compared to traditional cement, geopolymers material possesses the following merits: abundant raw materials resources, easy preparation, energy saving. These properties make geopolymer a extensive application development,

is a kind of "green material". At present, the study of geopolymer is the initial stage when geopolymer replace the traditional cement for new building materials or binders, the reports of electrical properties are little. The conductivity of geopolymer is not high under the arid condition. The electrical materials are prepared by adding organic conductive polymer. In this work, we prepared the geopolymer composites with metakaolin, sodium silicate, PEO as raw materials. The effect of different content PEO, different molecular weight and water content on conductivity of composites was studied, and the influence of PEO on mechanical properties was studied at the same time. The results indicated that the addition of PEO could greatly improve the conductivity of geopolymer composites that had a maximum value at room temperature when PEO content is 5%, but compressive strength has declined; suitable water content could promote the conductivity of geopolymer composites.

# K22 Synthesis and Characterization of the Layered Hybrids( $C_nH_{2n+1}NH_3$ )<sub>2</sub> MCl<sub>4</sub> (M=Co,Cu,Zn and n=2,4,6,8,10,12): *Liling Guo*<sup>1</sup>; Xian Wu<sup>1</sup>; Hanxing Liu<sup>1</sup>; <sup>1</sup>School of Material Science and Engineering, Wuhan University of Technology

A series of hybrids  $(C_nH_{2m+1}NH_3)_2MCl_4$  with quantum-well energy-band structure was synthesized by chemical reaction in HCl solutions. M=Co,Ni,Cu,Zn and n=2,4,6,8,10,12 were chosen to adjust the quantum-well depth and the barrier width, respectively. Chemical analysis of C, H and N confirms that the hybrids form in good agreement with the above molecular formulas. The X-ray diffraction patterns and scanning electron microscopy images of the products demonstrate that they crystallize well and have typical layered structures. However, the analogues of M=Ni can not be successfully synthesized in the same way. Concerning the energy-band structure, the colors of the products reveal that the quantum-well depth is more important than the barrier width for controlling the properties of the hybrids.

#### Poster Session: Symposium L: Energy Generation, Harvesting and Storage Materials

Tuesday PM	Room: Hall 2
August 3, 2010	Location: Cairns Convention Centre

L1 Determination of Reaction Mechanism in Li-N-H Hydrogen Storage System by TEM: *Miki Dohkoshi*<sup>1</sup>; Shigehito Isobe<sup>1</sup>; Yongming Wang<sup>1</sup>; Hikaru Yamamoto<sup>1</sup>; Hiroki Miyaoka<sup>1</sup>; Naoyuki Hashimoto<sup>1</sup>; Somei Ohnuki<sup>1</sup>; Takayuki Ichikawa<sup>1</sup>; Yoshitsugu Kojima<sup>1</sup>; <sup>1</sup>Hokkaido University

Reaction in Li<sub>2</sub>NH system is generally expressed as "LiH + LiNH<sub>2</sub>  $\leftrightarrow$  Li<sub>2</sub>NH + H<sub>2</sub>" with a release of 6.5 mass% H<sub>2</sub>. However, the detail of this reaction mechanism is not clear. Four kinds of models can be suggested to explain the reaction mechanism, the final condition after hydrogenation of Li<sub>2</sub>NH is that (1) LiH is inside LiNH<sub>2</sub>, (2) LiNH<sub>2</sub> is inside LiH, (3) both LiH and LiNH<sub>2</sub> exist randomly, (4) both LiH and LiNH<sub>2</sub> are finely separated. In order to determine an appropriate model for this system, observation of hydrogenation was carried out by transmission electron microscopy (TEM). Three kinds of samples were prepared: Li<sub>2</sub>NH under hydrogen atmosphere of 1.0 MPa for 1, 10, 100 minutes at room temperature, in order to compare to three samples with different hydrogenation ratio. TEM analysis would indicate distribution of LiH, LiNH<sub>2</sub> and Li,NH in those samples, and a suitable model could be determined.

L2 Hydrogen Permeation Properties of Pd-Coated Ni<sub>37.5</sub>Nb<sub>27.5</sub>Zr<sub>25</sub>Co<sub>5</sub>Ta<sub>5</sub> Amorphous Membranes: *Hong-Seok Chin*<sup>1</sup>; Yoon-Bae Kim<sup>2</sup>; Yu-Chan Kim<sup>2</sup>; Jin-Yoo Suh<sup>2</sup>; Wooyoung Lee<sup>1</sup>; Tae-Whan Hong<sup>3</sup>; Eric Fleury<sup>2</sup>; <sup>1</sup>Yonsei University; <sup>2</sup>Korea Institute of Science and Technology; <sup>3</sup>Chungju National University

The widespread of hydrogen-related energies necessitates the production of hydrogen at a large scale and competitive cost. One method of production consists in separating, at high temperature, hydrogen molecules from a gas by means of the permeation technique. Pd-based membranes are currently the most commonly used materials for this application however the price of Pd elements stimulates the search for alternative candidate materials. For that purpose, this study was undertaken to develop new compositions of metallic amorphous alloys with performance at least similar to those of Pd-based membranes. In this paper, we will present the hydrogen permeation properties of Ni-Nb-Co-Zr-Ta amorphous alloy prepared in the form of thin ribbon of about ~30 µm thick. At low temperature, the hydrogen permeability of the Ni-Nb-Co-Zr-Ta alloy was found to be similar to that of Pd-Cu alloy while for temperature larger than 400°C, the hydrogen permeability decreased slightly. The variation in the properties of the Ni-Nb-Co-Zr-Ta amorphous alloy will be discussed based on microstructural modifications detected by XRD, XPS and TEM analyses.



L3 Hydrogen Storage in Nitrides by the Use of Ammonia as a Hydrogen Carrier: *Hayao Imamura*<sup>1</sup>; Naotaka Shimomura<sup>1</sup>; Fumiya Nakamura<sup>1</sup>; Keisuke Watanabe<sup>1</sup>; Taichi Kanekiyo<sup>1</sup>; Toshiki Matsui<sup>1</sup>; Yoshihisa Sakata<sup>1</sup>; <sup>1</sup>Yamaguchi University

Hydrogen storage in calcium nitride, magnesium nitride or lithium nitride has been undertaken by the use of ammonia, in which the possibility of ammonia as a vector for hydrogen carriers has been studied. When the calcium nitride obtained by thermal decomposition of calcium amide was brought into contact with ammonia gas (300 Torr) at room temperature, ammonia uptake readily occurred. The ammonia was absorbed in the form of the decomposed state in the nitride. When the sample was heated, the absorbed ammonia was released in the form of hydrogen and nitrogen. The efficiency of hydrogen storage using ammonia was relatively high. This type of hydrogen storage has been extensively studied and characterized.

L4 Hydrogenation/Dehydrogenation in MgH<sub>2</sub>-Activated Carbon Composites Prepared by Ball Milling: *Yi* (*Alec*) *Jia*<sup>1</sup>; Jin Zou<sup>2</sup>; G.Q. (Max) Lu<sup>1</sup>; Xiangdong Yao<sup>3</sup>; <sup>1</sup>ARC Centre of Excellence for Functional Nanomaterials (ARCCFN),the University of Queensland; <sup>2</sup>Centre for Microscopy & Microanalysis (CMM), University of Queensland; <sup>3</sup>Queensland Micro- and Nanotechnology Centre (QMNC), Griffith University

Mg-x wt% activated carbon (AC) composites (x=1, 5, 10, 20) were prepared by ball milling and their hydrogen storage behaviors were investigated. It was found that MgH<sub>2</sub>-5wt% AC could absorb about 6.8 wt% H<sub>2</sub> within 7 min at 573K and 2 h at 473K respectively. It is also demonstrated that MgH<sub>2</sub>-5wt% AC exhibited good hydrogen desorption property that could release 6.5 wt% at 603K within 30min. Even at a relative low temperature of 573K, the synthesized composites still could release the entire absorbed 6.8wt% hydrogen. Furthermore, Differential Scanning Calorimetry (DSC) measurement on the desorption temperature was performed and the results indicated that the onset and peak temperatures both reduced with increasing AC adding amount, for example, the dehydrogenation peak temperature shifted from 348.8°C for 1 wt% AC to 315.9°C for 20 wt% AC. X-ray diffraction patterns (XRD) and transmission electron microscopy (TEM) observations revealed that the grain size of the synthesized composites decreased with increasing AC amount. This may contribute to the improvement of hydrogen storage in Mg-AC composites.

#### L5 In-Situ TEM Observation for Dehydrogenation Mechanism in MgH<sub>2</sub> with Catalyst: *Eri Morita*<sup>1</sup>; Akifumi Ono<sup>1</sup>; Shigeshito Isobe<sup>1</sup>; Yongming Wang<sup>1</sup>; Naoyuki Hashimoto<sup>1</sup>; Somei Ohnuki<sup>1</sup>; <sup>1</sup>Hokkaido University

Mg has been regarded as a promising candidate for a base metal of hydrogen storage alloys, which have large capacity for hydrogen storage, however, the reaction kinetics of hydrogen absorption/desorption is too slow. It has been suggested that oxide catalyst is effective for improving the hydrogenation and dehydrogenation kinetics of Mg. In this study, the change of the high resolution image before and after dehydrogenation was observed by transmission electron microscope (TEM) to examine the details of the catalytic reaction. In addition, insitu TEM observation on the catalytic effect of Nb<sub>2</sub>O<sub>5</sub> in MgH<sub>2</sub> was carried out. In the case of MgH<sub>2</sub> catalyzed with 1 mol% of Nb<sub>2</sub>O<sub>5</sub>, the dehydrogenation started at 150°C and Mg nano-size particles were formed. However, Nb<sub>2</sub>O<sub>5</sub> was not confirmed in diffraction patterns and images. In the case of MgH<sub>2</sub> catalyzed with 10 mol% of Nb<sub>2</sub>O<sub>5</sub>. Those results suggested that the dehydrogenation started from the interface of MgH<sub>2</sub> and Nb<sub>2</sub>O<sub>5</sub>.

L6 Behavior of Hydrogen in Electrolitically Charged Aluminum: *Tomohiro Tsutsumi*<sup>1</sup>; Takahito Watakabe<sup>2</sup>; Goroh Itoh<sup>3</sup>; Nobuhide Itoh<sup>3</sup>; <sup>1</sup>Undergraduate Student, Department of Mechanical Engineering, Ibaraki University; <sup>2</sup>Graduate Student, School of Science and Engineering, Ibaraki University; <sup>3</sup>Department of Mechanical Engineering, Ibaraki University

Environmental problems such as global warming and exhaustion of fossil fuels have been growing into the serious problems. On this background, fuel cell vehicles using hydrogen as a clean energy have been paid attention to. In the fuel cell vehicles, high-pressure hydrogen gas is stored in a cylinder composed of aluminum alloy liner and shell of carbon fiber reinforced plastic. Only 6061-T6 is approved among aluminum alloys for the liner in the current Japanese standard. Application of other alloys having higher strength is considered since higher maximum filling pressure is demanded to achieve longer mileage per fuel filling. However, hydrogen embrittlement has been reported to occur in some aluminum alloys. To guarantee the safety of the fuel cell vehicles, hydrogen embrittlement through the investigation on the behavior of hydrogen is needed. Hydrogen microprint technique (HMPT) has been known as an effective method to investigate the hydrogen behavior, in which the reaction between silver bromide emulsion and atomic hydrogen emitted from the sample is used. In the

present study, the behavior of electrolitically charged hydrogen in aluminum with 99.99% purity has been investigated by means of HMPT.

L7 Effectiveness of Ion-Plating on the Detection of Hydrogen in Pure Aluminum during Tensile Deformation: Goroh Itoh<sup>1</sup>; *Hideki Iwahashi*<sup>1</sup>; Takahiro Shikagawa<sup>2</sup>; <sup>1</sup>Ibaraki University, Department of Mechanical Engineering; <sup>2</sup>Ibaraki University

In recent years, fuel cell vehicles (FCVs) are attracting attention as a way of resolving global environment problems. In the FCVs, high-pressure (35MPa) hydrogen is contained in type-3 cylinder composed of 6061-aluminum-alloy liner and surrounding carbon-fiber-reinforced-plastic layer. The mileage per a filling of FCVs is not as long as that of gasoline vehicles. Thus, replacing the 6061 alloy with a higher-strength aluminum alloy is demanded to increase the mileage by raising the initial pressure to 70MPa. In order to guarantee the safety of the hydrogen cylinder, investigation on the hydrogen behavior as well as on the hydrogen assisted embrittlement is required. Although hydrogen microprint technique (HMPT) has been known to be effective to investigate the hydrogen behavior, the low detection efficiency of hydrogen was reported. Ion-plating has been reported to increase the detection efficiency of HMPT in 6061 and 7075 alloys. However, the mechanism for the increase in the detection efficiency has not been elucidated yet. In this study, aluminum plates of 99.99% purity were ion-plated with Sn, plastically deformed by about 5 per cent and then increased to HMPT. It has been confirmed that the detection efficiency of the impurity hydrogen was markedly increased by the ion-plating.

#### **L8 Evaluation of Hydrogen in Liquid Aluminum by Means of Thermal Analysis**: Julathep Kajornchaiyakul<sup>1</sup>; *Sompob Petcrie*<sup>1</sup>; Witthaya Samit<sup>1</sup>; <sup>1</sup>National Metal and Materials Technology Center

Thermal analysis technique may be used to predict the degree of grain refinement of hypoeutectic aluminum-silicon alloy. This technique is based upon an analysis and interpretation of cooling curve characteristics observed over the solidification of the alloy. Interestingly, it is observed that dissolved hydrogen appears to affect characteristics of the cooling curve. In order to gain insight whether such a relationship really exists, the present work investigates how the dissolved hydrogen in liquid aluminum alloy may affect the cooling curve characteristics. Casting trials were carried out using a hypoeutectic aluminumsilicon alloy. Emphasis was placed on different amounts of the dissolved hydrogen in the liquid alloy. During the trials samples of the liquid alloy were carefully controlled to minimize variations, such as composition, pouring temperature, cooling rate, which may undesirably confuse the thermal analysis. Regarding certain conditions addressed in the present study, it was found that the dissolved hydrogen appears to affect degree of undercooling of the cooling curve. An attempt toward establishment of an empirical relationship between the dissolved hydrogen and the degree of undercooling is presented. Plausible mechanisms underlying this finding are noted.

## L9 Observation of Hydrogen Behavior in a Eutectic Mg-Ni Alloy by the Silver Decoration Method: *Hideyuki Saitoh*<sup>1</sup>; Misato Shimpo<sup>1</sup>; <sup>1</sup>Muroran Institute of Technology

Silver decoration method was applied to the Mg-11.3mol%Ni eutectic alloy to investigate hydrogen behavior in it. The plate-like specimens 1 mm thickness were electrochemically hydrogen charged using NaOH aqueous solution at ambient temperature. Then the specimens were immersed in the silver decoration solution of KOH aqueous solution of pH 11 containing 4.3 mol/m<sup>3</sup> K[Ag(CN)<sub>2</sub>] to visualize hydrogen position as the location of silver grain. Prior to the observation, effect of Pt coating on the decoration process was examined. It is shown that the Pt coating drastically decreases the background silver grains caused by the direct reaction between the specimen and the decoration solution. The observed hydrogen location on the hydrogen input side of the plate specimen is not the Mg phase but the Mg<sub>2</sub>Ni phase in the eutectic structure. The hydrogen location on the hydrogen is preferentially absorbed in the Mg<sub>2</sub>Ni phase and penetrates into the specimen through the Mg,Ni phase.

#### L10 Cr-Doped Carbon Films on 316L Stainless Steel as Bipolar Plates for Proton Exchange Membrane Fuel Cell: *Guoqiang Lin*<sup>1</sup>; Wu Bo<sup>1</sup>; <sup>1</sup>Dalian University of Technology, MMLab

A series of Cr-doped carbon films on 316L stainless steel are prepared as bipolar plates for proton exchange membrane fuel cell (PEMFC) by pulsed bias arc ion plating (PBAIP). The modified films are identified using scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), X-ray diffractometry (XRD). The results indicate that the films are integral and dense, the chromium content of the films varies from 0.04 to 0.23, and the deposited films are mainly amorphous with some chromium phases. The interfacial contact resistance and anticorrosion property are also measured. Compared with the bare stainless steel plates, the interfacial contact electric conductivity and anticorrosion property are significantly improved. A minimum value of 2.79 mO cm<sup>2</sup> is obtained



under 1.2 MPa compaction force. The lowest corrosive current density of 9.14  $\times$  10<sup>-8</sup> A cm<sup>-2</sup> is obtained at 0.6 V (vs. SCE) by the potentiodynamic test in a simulated corrosive circumstance of the PEMFC under 25°C.

L11 Effect of Annealing on the Hydrogen Permeation and Mechanical Behaviour of Nb-Ni-Zr Alloy Membranes: *Timothy Wong*<sup>1</sup>; Zhengrong Yu<sup>1</sup>; Kiyonori Suzuki<sup>1</sup>; Mark Gibson<sup>2</sup>; Kazuhiro Ishikawa<sup>3</sup>; Kiyoshi Aoki<sup>3</sup>; <sup>1</sup>Monash University; <sup>2</sup>CSIRO; <sup>3</sup>Kitami Institute of Technology

Nb-Ni-Zr composite alloy membranes have been reported to have a hydrogen permeability higher than that of pure Pd. Since the hydrogen permeation behaviour in these composite alloys is highly microstructure sensitive, then hydrogen permeability is likely to depend on annealing conditions used to generate different microstructures. This work has looked into the effect of annealing treatment on the hydrogen permeability of as-cast Nb-Ni-Zr alloys with the goal of helping in the advancement of Nb-based alloy membranes as cost-effective alternatives to the Pd-based alloy membranes used for hydrogen purification. Nb-Ni-Zr alloy ingots of different compositions were prepared by argon arc-melting. The samples were vacuum sealed in quartz tubes and annealed isochronally for 1 h between 500°C and 900°C. It was found that the samples annealed at 900°C exhibit higher hydrogen permeability than the as-cast samples. However, these samples were found to be less resistant to hydrogen embrittlement and the membranes exhibited cracks after the permeation test. The main mechanical failure mechanism was due to intragranular cracking for the alloys with high Nb content, while the mechanism was by intergranular cracking for those with lower Nb-content. The relationship between dendrite ripening and the improved hydrogen permeability is also discussed.

#### L12 Effects of Dissolved Hydrogen on the SCC Susceptibility SUS316L Stainless Steel: *Katsuhito Nakagawa*<sup>1</sup>; Masahiro Nono<sup>1</sup>; Akihiko Kimura<sup>2</sup>; <sup>1</sup>Graduate School of Energy Science,Kyoto University; <sup>2</sup>Institute of Advanced Energy,Kyoto University

TGSCC was observed in the welded region of core shroud of boiling water reactors in Japan. The TGSCC was accompanied by hardening at the shroud surface, and the SCC fracture mode changed from transgranular to intergranular at the softened region. SSRT were carried out to evaluate the susceptibility to SCC, but the brittle fracture mode has been considered to be influenced by strain rate, dissolved hydrogen and oxygen content. In the previous our work, it was observed that the SCC of SUS316L stainless steel (SS) in hydrogenated pressurized water was IGSCC. The objective of this work is to clarify the effect of sensitization on the SCC fracture mode in hydrogenated water. SSRT have been performed on 316L-SS in high temperature pressurized water environments at 288 °C under a pressure of 7.8 MPa. The water chemistry was controlled with DO (0.2 ppm) or DH (0.4, 0.1 ppm) or DO and DH (0 ppm). The SCC fracture mode of the specimen surface has been clarified by EBSD. No SCC was observed in the non-sensitized 316L-SS tested in the DO condition. In contrast, SCC occurred in the non-sensitized 316L-SS in the DH conditions. Effects of sensitization on the SCC behavior will be shown.

#### L13 Enhanced Conversion Efficiency of Solar Cells by Nano-Sized Anti-Reflection Layer Fabricated Using Nano-Imprint Lithography: Kang-Soo Han<sup>1</sup>; Ju-Hyeon Shin<sup>1</sup>; Woo-Young Yoon<sup>1</sup>; Heon Lee<sup>1</sup>; <sup>1</sup>Korea University

As an effective method of increasing conversion efficiency of solar cells and modules, a hydrophobic SAM (self assembled monolayer) coated motheye layer was fabricated on various surfaces of solar cells and modules. This moth-eye structure serves as an anti-reflective layer, due to its gradual refractiveindex change. To form nano-structures on the thermoplastic plate and glass plate for solar modules, hot-embossing method and nano-imprinting method were used, respectively. After forming the structure, hydrophobic SAM coating was performed to add self-cleaning functionality. Finally, these were installed on solar modules to protect solar cells from external damages. Moreover, the motheye structure, the surface reflectance of patterned layers was decreased. As a result, solar cells with moth-eye layers exhibited higher quantum efficiency and total conversion efficiency than solar cells without moth-eye layers.

#### L14 Fabrication of Nanocrystalline TiO<sub>2</sub> Films by Aerosol Deposition Method for Dye-Sensitized Solar Cells: *Da-Long Cheng*<sup>1</sup>; Chia-Yi Lin<sup>1</sup>; Chih-Sheng Liu<sup>1</sup>; Kuo-Sheng Kao<sup>1</sup>; Chih-Ming Wang<sup>2</sup>; <sup>1</sup>Shu-Te University; <sup>2</sup>Cheng Shiu University

This work present a low resistance dye-sensitized solar cell (DSSC) based on metal substrates. Different kinds of metal meshes are used to fabricate nanocrystalline TiO<sub>2</sub> electrode and counter-electrode. The nanoporous TiO<sub>2</sub> films were fabricated via an aerosol deposition method. The TiO<sub>2</sub> colloid was produced by dissolving P25 powder in ethanol solution and atomized using an ultrasonic nebulizer. The flexible electrodes can be sintered under 450–550 °C and exhibit high-temperature sinterability. The tuning of TiO<sub>2</sub> film thickness coated on metal mesh towards optimization of solar energy conversion efficiency had been investigated. Experimental results show that the excellent electric conductivities improve the fill factor and conversion efficiency for the large-size DSSCs due to the reduced internal resistance of the cell. These flexible metal substrates are a promising approach especially from the viewpoint of large-scale, cost effective industrial manufacturing of solar cells.

L15 Evaluation of Irradiation Hardening of Fe-Ion Irradiated F82H by Nano-Indentation Techniques: *Yoshiyuki Takayama*<sup>1</sup>; Ryuta Kasada<sup>1</sup>; Kiyohiro Yabuuchi<sup>1</sup>; Akihiko Kimura<sup>1</sup>; Dai Hamaguchi<sup>2</sup>; Masami Ando<sup>2</sup>; Hiroyasu Tanigawa<sup>2</sup>; <sup>1</sup>Kyoto University; <sup>2</sup>Japan Atomic Energy Agency

The F82H, reduced activation ferritic/martensitic steel, is one of the candidates for the fusion blanket structural material. Precise evaluation and prediction of the material degradation such as swelling and irradiation hardening/embrittlement has been strongly demanded for designing the future DEMO reactors. Massive production of F82H may suffer the impurity contamination of Cu and Ni elements during processing because of the reuse of steel-scraps for the construction of blanket modules. These elements have been considered to affect the irradiation hardening or embrittlement of the ferritic steels, but the detail of the effects is not clear. Ion irradiations with 6.4 MeV Fe3+ were carried out at 563K up to damage levels of 10 dpa in DuET facility, Kyoto University. Nano-indentation hardeness tests were carried out using Elyonix ENT-1100a and MTS Nano-indentor G200. Irradiation induced hardness change was observed for all materials (F82H, F82H-1at.% Ni, F82H-2at.%Ni, F82H-0.2at.%Cu, F82H-0.5at.%Cu) irrespective to the chemical composition of the steel. Enhancement of irradiation hardening, however, was observed for the Ni-added F82H but not for the Cu-added F82H.

L16 Thermoelectric Properties of Nanostructured Bismuth Telluride Prepared by Mechano-Chemical Process: *Kyung Tae Kim*<sup>1</sup>; Gil-Geun Lee<sup>2</sup>; Gook Hyun Ha<sup>1</sup>; <sup>1</sup>Korea Institute of Materials Science (KIMS); <sup>2</sup>Pukyong National University

Nanostructured bismuth telluride materials have fabricated by consolidating Bi-Te nanopowders obtained from mechano-chemical process (MCP) which employs a chemical and mechanical reaction among the oxide constituents. XRD and microstructural analysis of synthesized nanopowders clearly show p-type phase consisting of Bi-Sb-Te and polycrystalline characteristics, respectively. The powders are sintered into bulk Bi-Te materials with 100nm-sized nanograins, which can be effectively used in scattering phonons. Thermoelectric properties of nanostructured Bi-Te materials reveal that nano-structuring produces decreased thermal conductivities compared to micro-grained bismuth telluride at 293K, resulting in enhanced thermoelectric performance, ZT value. As results of these, it is concluded that the developed MCP is very useful to prepare thermoelectric nanopowders as raw materials for nanograined bismuth telluride.

#### L17 Study of Hydrogen Storage Properties of LiBH<sub>4</sub>-MgH<sub>2</sub>-Al and LiBH<sub>4</sub>-MgH<sub>2</sub>-MAIH<sub>4</sub> (M=Li, K) Systems: *Seito Niwa*<sup>1</sup>; Tatsuya Higuchi<sup>1</sup>; Hiroyuki Takeshita<sup>1</sup>; <sup>1</sup>Kansai University

Recently, Vajo et al. proposed the method of mixing MgH<sub>2</sub> to improve the reversibility and thermodynamics of the dehydrogenation of LiBH<sub>4</sub>. The LiBH<sub>4</sub>-MgH<sub>2</sub> system with 11.5 mass% in storage capacity releases hydrogen by the following reaction, LiBH<sub>4</sub> + 1/2MgH<sub>2</sub> = LiH + 1/2MgB<sub>2</sub> + 2H<sub>2</sub> and the rehydrogenation can be achieved at 573K and 5MPa of H<sub>2</sub>[2]. But the reversible reaction becomes difficult to occur due to the following reaction. LiBH<sub>4</sub> +  $1/2MgH_2 \rightarrow LiH + 1/2Mg + B + 2H_2$ , if the dehydrogenation is performed below 0.3MPa of H. Li in our laboratory reported that Al addition was effective to improve hydrogen desorption temperature and reversibility of LiBH<sub>4</sub>-MgH<sub>2</sub> system. In the LiBH<sub>4</sub>-MgH<sub>2</sub>-Al system, Mg<sub>1-x</sub>Al<sub>x</sub>B<sub>2</sub> was formed instead of MgB<sub>2</sub> even under no hydrogen atmosphere, which contributes to the improvement of reversibility. But the addition of Al leads to decrease in gravimetric hydrogen storage capacity of the system. In this paper, we focus on MAlH<sub>4</sub> (M = Li, K) in place of Al, in order to improve hydrogen storage capacity.

#### L18 The Effects of Cryogenic Milling and Catalytic Additives on the Hydrogen Desorption Behaviour of Nanostructured MgH<sub>2</sub>: Chan Seo<sup>1</sup>; *Xiaodong Wu*<sup>1</sup>; Kiyonori Suzuki<sup>1</sup>; <sup>1</sup>Monash University

 $MgH_2$  is one of the most attractive metal-hydrides as a candidate for hydrogen storage applications. However, the hydrogen sorption kinetics of Mg or Mg-based alloys is sluggish and this remains a challenging problem yet to be resolved. Hence, the focus of alloy development in Mg-based hydrogen storage alloys is to improve the absorption and desorption kinetics. Promising approaches to this problem reported to date include additions of catalytic metal oxides and nanoscale microstructural refinement by ball milling. However, the vast majority of previous reports on ball milled MgH<sub>2</sub> have employed room temperature milling processes despite that fact that the nanocrystallite size after milling can be reduced by lowering the milling temperature. In this report, we have prepared nanostructured MgH<sub>2</sub> powders by ball milling at room and cryogenic temperatures and the hydrogen desorption behaviours of milled powders have been investigated. The mean grain size of MgH<sub>2</sub> shows a dramatic decrease after milling for 100 h. The



onset of the endothermic reaction shows a systematic decrease with increasing milling time. The desorption temperature of ball milled  $MgH_2$  was found to be reduced by  $Ba_3$  ( $Ce_{1+x}Nb_{2,x}$ ) $O_{9-d}$  (BCN) addition. The effect of cryogenic milling on the hydrogen desorption behaviour of  $MgH_2$  is also presented.

## L19 Different Bi<sub>2</sub>Se<sub>3</sub> Thermoelectric Nanostructures Prepared by the Solvent Thermal Method: Lina Cheng<sup>1</sup>; Zhigang Chen<sup>1</sup>; *Jin Zou*<sup>1</sup>; <sup>1</sup>University of Queensland

Developing efficient thermoelectric materials has attracted much attention recently due to their applications in solid state cooling and power generation from waste heat. The current strategy to increase figure of merit ZT=(S2 ó / K)T), which evaluates the quality of thermoelectric materials, is focusing on the development of thermoelectric materials with low dimensional nanostructure owing to the great reduction of thermal conductivity caused by the increased interfaces to scatter phonons more effectively. In order to achieve its optimum ZT values, we synthesized a series of Bi<sub>2</sub>Se<sub>2</sub> thermoelectric materials with different nanostructures by chemical methods. It has been found that the nanostructure of Bi<sub>2</sub>Se<sub>3</sub> can be controlled by adjusting the PH value of reactants. Besides, Bi<sub>2</sub>Se<sub>3</sub> nanostructures with wire-like morphology can be formed without any sodium hydroxide in the reactants, and while, the production of Bi<sub>2</sub>Se<sub>3</sub> hexagonal slices can be achieved when the concentration of sodium hydroxide reaches 0.5mol/L. The morphology of these two Bi<sub>2</sub>Se<sub>3</sub> productions can play a very important role in their thermoelectric properties. For understanding the relationship between the structure, property and controlling the morphology and size completely, their growth mechanics are explored.

#### L20 Effects of Chemical Composition on the Impact Properties of A533B Steels: *Byung Jun Kim*<sup>1</sup>; Ryuta Kasada<sup>1</sup>; Akihiko Kimura<sup>1</sup>; <sup>1</sup>Kyoto University

Small specimen test technique (SSTT) for the evaluation of irradiation embrittlement of reactor pressure vessel steel (RPVS) has been considered to be essential to operate light water reactors of extended lifetime. In this research, specimen size effects were investigated for RPVS to apply small specimen test technique to surveillance test method. All specimens used in this study were machined from welded A533B plate materials, which are standard, low Mn, high Cu, high P, and high Cu and high P steels. Different size of specimens, Standard-, CVN-1/2, CVN-1/3 and CVN-1.5 mm were fabricated from weld bond. Charpy tests were carried out at temperatures from 100K to 500K. Test results were discussed in view of the differences in specimen size and compositions of elements of Cu, Mn, and P. The ductile-to-brittle transition temperature (DBTT) and upper shelf energy (USE) were reduced by decreasing specimen size. It was found that the amount of Cu, Mn and P affect the DBTT and USE. The effects of notch position and chemical compositions on DBTT are independent of specimen size. This indicates that small specimen test technique is applicable and effective to surveillance test of RPVS of extended operation period.

L21 Electrochromic Properties of WO<sub>3</sub> Thin Films Prepared by Electron Beam Evaporation: Chih-Ming Wang<sup>1</sup>; *Shih-Yuan Lin*<sup>2</sup>; Chih-Yu Wen<sup>1</sup>; Ying-Chung Chen<sup>2</sup>; Hsu-Ting Hsiao<sup>1</sup>; Hsien-Hung Tang<sup>1</sup>; <sup>1</sup>Cheng Shiu University; <sup>2</sup>National Sun Yat-Sen University

Thin film of tungsten oxide (WO<sub>3</sub>) has been extensively studied as an electrochromic material and has numerous applications in electrochromic devices, smart windows, gas sensors and optical windows. WO<sub>3</sub> thin films were deposited on ITO/Glass substrates by electron beam evaporation technique. Electrochromic properties of WO<sub>3</sub> thin films were investigated using cyclic voltammograms (CVs) and in situ transmittance measurements, which were carried out on WO<sub>3</sub> thin films were found to show reversible blue coloration upon Li<sup>+</sup> ion intercalation in this study. Experimental results reveal that the processing parameters will influence the electrochromic properties of WO<sub>3</sub> thin films such as transmittance, CVs, inserted charge, optical density change, coloration efficiency and insertion coefficient.

#### L22 Influence of Temperature on Self-Discharge and High-Rate Discharge Characteristics of La-Rich AB5-Based MH Alloy Electrode: *Huai-Ying Zhou*<sup>1</sup>; Pei-Pei Wang<sup>1</sup>; Zhong-Min Wang<sup>1</sup>; Ruiping Zou<sup>2</sup>; Cheng-Yuan Ni<sup>1</sup>; <sup>1</sup>Guilin University of Electronic Technology; <sup>2</sup>University of New South Wales

The influence of temperature on self-discharge and high-rate discharge characteristics of  $MmNi_{3.65}Co_{0.75}Mn_{0.6}$  alloy electrode are investigated using simulated battery tests. Self-discharge behaviors of the MH electrode are measured by two methods: the continuous mode self-discharge and the step mode self-discharge. The results indicate that both reversible and irreversible capacity loss of the MH electrode are mainly affected by temperature with the capacity loss after storage for 4 days being 30.88% at 323K, 15.02% at 273K, and 20.09% at 303K. Furthermore, SEM analysis shows that some needle corrosion products would be formed on the surface of the MH electrode, especially after storage at high temperatures. For the high rate discharge behavior, the MH electrode performs better at 303K, compared with the ones at 273K and 323K. In addition,

the high-rate discharge capacities are found to decrease with the increase of discharge current. However, an extra low-current discharge process (0.2C) would ultimately discharge the retained capacity, resulting in a decline in cycle life.

### **L23 Irradiation Hardening Behavior of Fe Based Binary Alloys Irradiated by Neutron**: *Kiyohiro Yabuuchi*<sup>1</sup>; Masashi Saito<sup>1</sup>; Ryuta Kasada<sup>1</sup>; Akihiko Kimura<sup>1</sup>; <sup>1</sup>Kyoto University

Irradiation hardening of Reactor Pressure Vessel steels (RPVs) is one of the essential degradation issues. It is well know that the irradiation hardening due to Cu-rich precipitates (CRP) at lower irradiation dose. However nature of matrix defects, which have been recognized to affect irradiation hardening at higher dose, has not been clarified yet. Especially studies about effects of each element in RPVs on irradiation hardening at high dose were limited. This study was motivated by extracting effects of each element in RPVs on irradiation hardening under high dose irradiation. Pure-Fe, Fe-1Cr, Fe-1Mn, Fe-1Ni, Fe-1Cu and Fe-1Mo were used as specimens. Neutron irradiation was carried out under various irradiation conditions from 0.3 to  $8.5 \times 10^{19}$  n/cm<sup>2</sup> at 290 °C. A significant irradiation hardening in Fe-1Mn as well as a typical irradiation hardening in Fe-1Cu was observed. The irradiation hardening of Fe-1Cu considered to be due to CRP was saturated at lower dose and that agrees with previous studies. On the other hand that of Fe-1Mn was not saturated but showed rapidly increase in its irradiation hardening at around  $5\times10^{19}\,n/cm^2$ . As for other binary alloys, significant irradiation hardening was not observed.

L24 Joining of ODS steels and Tungsten for Fusion Applications: Sanghoon Noh<sup>1</sup>; Ryuta Kasada<sup>1</sup>; Nakao Oono<sup>1</sup>; Takuya Nagasaka<sup>2</sup>; Akihiko Kimura<sup>1</sup>; <sup>1</sup>Kyoto University; <sup>2</sup>National Institute for Fusion Science

Oxide dispersion strengthened (ODS) steels and tungsten (W) are considered as promising candidate materials for structural and plasma facing materials of the first wall and divertor components in DEMO fusion reactor. ODS steels shows excellent elevated temperature strength, corrosion resistance, radiation resistance and W has high resistance against sputtering and low tritium retention in fusion environment. It is considered that the joining of ODS steels and W is one of essential issues for the development of fusion application. However, ODS steels and W have significant differences in their physical properties, particularly the mismatch of coefficients of thermal expansion (CTE). Thus, suitable joining process and joint strength evaluation method need to be developed. In this study, diffusion bonding between ODS steel and W was carried out and its joint strength was investigated. High-Cr ODS ferritic steel block and W plate were diffusion bonded at various temperatures for 1h with/without Ti interlayer using uni-axial high vacuum hot press. Cross sectional microstructures of joint region were observed by scanning electron microscope. To evaluate mechanical properties of the joint region, cross sectional hardness profiles measurements and miniaturized torsion tests were carried out.

L25 Microstructural Evaluation of Dy-Ni-Al Grain-Boundary-Diffusion (GBD) Treatment on Sintered Nd-Fe-B Magnet: *Naoko Oono*<sup>1</sup>; Masato Sagawa<sup>2</sup>; Ryuta Kasada<sup>1</sup>; Hideki Matsui<sup>1</sup>; Akihiko Kimura<sup>1</sup>; <sup>1</sup>Kyoto University; <sup>2</sup>Intermetallics Co., Ltd.

GBD treatmet with Dy-Ni-Al eutectic alloy powder enhanced the coercivity of sintered Nd-Fe-B magnet plate as thick as 5mm to 22kOe. EELS image indicated that this industrially epoch-making treatment spread Dy, which is coercivity enhancing element, from surface to centre of the magnet through Nd-rich phase. We also confirmed by STEM-EDS analysis that Dy diffused into grain interior of the magnet. It is considered that Ni and Al, which are melting point depressants of Nd, melt not only Nd-rich phase but also the grain regions adjacent to grain boundaries during GBD treatment.

L26 Microstructure Control of Rapidly Solidified Si-Ni Base Alloys by the Optimization of Melt Spinning and Heat Treatment Processes: *Sung-Min Jeon*<sup>1</sup>; Jong-jin Song<sup>1</sup>; Hyoun-wook Han<sup>1</sup>; Keun Yong Sohn<sup>1</sup>; Won-wook Park<sup>1</sup>; <sup>1</sup>Inju University

Anode composite materials have been studied to improve the performance and the durability for Li-ion secondary batteries in our laboratory. Rapidly solidified Si-Ni-Al and Si-Ni-Ti-Cu alloy ribbons were fabricated by optimizing the melt spinning at the cooling rate of about 106°C/sec, which show good potentials to replace the conventional anode materials mostly consisted of graphite powders. The thin ribbons of ~20µm in thickness possessed the amorphous and nanocrystalline structures, and the nano-scale Si particles were crystallized in the matrix by heat treatment at 400~600°C. At the wheel side of the ribbon, 50nm size of Si particles were formed as a nucleation-type; whereas at the air side Si particles were more coarsened. Microstructures of the ribbon were investigated using SEM equipped with EDS, X-ray diffractometer (XRD) and TEM. The precipitated phases in Si-Ni base alloy were NiAl, SiTiNi, Si<sub>2</sub>NiTi, and Si. Finally, the ribbon was pulverized to make the powder for the anode material of secondary batteries, which showed the excellent capacity and stability for energy storage compared to conventional powder materials.

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L27 Study on Decomposition Process of Sodium Alanate by Means of Transmission Electron Microscope: *Hao Yao*<sup>1</sup>; Shigehito Isobe<sup>1</sup>; Yongming Wang<sup>1</sup>; Naoyuki Hashimoto<sup>1</sup>; Somei Ohnuki<sup>1</sup>; <sup>1</sup>Hokkaido University

Sodium alanate (NaAlH<sub>4</sub>), which has a total hydrogen capacity of 5.6 wt.%, is considered as a promising candidate for the application of the onboard vehicles because of its high hydrogen weight capacity and low cost. For improving its hydrogen storage properties, it is important to clarify the mechanism of the hydrogenation/dehydrogenation and catalysis, especially with Ti-additives. In this study, after preparing sodium alanate with and without catalyst treated in a glove box under inert gas and transferred to microscope without exposure to air, decomposition of NaAlH<sub>4</sub> were observed by in-situ and ex-situ TEM. An in-situ electron beam diffraction showed NaAlH<sub>4</sub> decomposed to Na<sub>3</sub>AlH<sub>6</sub> and Al, and then NaH and Al during heating to 150-200°C. Additionally, porous structures appeared during heating, indicating that structural defects or cavities were formed due to volume changing of the phases. Simultaneously, irregular distribution of Al crystals and Na<sub>3</sub>AlH<sub>6</sub> particles was exhibited. Further, it is indicated that porous structure could be one of the special features for some hydrogen storage materials composed of light elements because of their high chemical activities.

#### L28 The Study of Recycling Technology for the Silicon Wafer Backgrinding Sludges: *Man Sik Kong*<sup>1</sup>; Hang-Chul Jung<sup>1</sup>; Hyun Seon Hong; Gu Sung Kim<sup>2</sup>; <sup>1</sup>IAE: <sup>2</sup>EPworks Co. Ltd

Recently the price of photovoltaic generation system is highly depended on silicon material cost of 50%. Silicon material shortage makes effects to photovoltaic generation industrial and which is expected to continue. So development of recycling technology for silicon waste, like waste sludge of wafer backgrinding process, is one of the most important one for demands of silicon material. A lot of silicon waste sludge is produced in the wafer backgrinding process. In addition, because of the thickness of the semiconductor wafer is being thinner rapidly, the amounts of silicon waste sludge have been continually increased. In this study, the recycling technology and wafer consolidation process of silicon material from wafer backgrinding sludge is studied. Previously, the properties of silicon sludge was analyzed and applied to the wet classifier system for pre-processing of recycling. Finaly the silicon powder was consolidated by hot press. The separation efficiency, powder purity, consolidation properties of silicon material are investigated.

## **L29** Variation of Heat Dissipation Properties of LED Packages with Thermal Vias: *Shin Hyeong-won*<sup>1</sup>; Hyo-Soo Lee<sup>1</sup>; Seung Boo Jung; <sup>1</sup>KITECH/Foundry Technology Service Center

Light Emitting Diode is largely used in industry of consumer electronics such as cell-phones, PDAs, and computers. Since all light sources convert electric power into radiant energy and heat, LED also does the same. However, it only converts 15~25% of electric power into visible light; the rest of the power, 75~85% is converted into heat. This excess heat should be conducted away from the LED die to circuit boards or heat sinks since heat directly affects performance of the LED. As a short term effect, which is reversible, it will bring color shift and reduced light output. Furthermore, lifecycle of the LED will shorten nonreversibly if the problem continues. In order to prevent LED from these negative effects, low thermal resistance path needs to be achieved so that heat conducts from the LED to underlying circuit board. Thus, thermal-via optimization study is performed through experiment. 1W and 3W LED assembled printed circuit board (PCB) with 16 different via design is set up to measure its temperature for 4 hours in a real time. Via design is differed by number, diameter, and pitch of vias. For 1W LED assembled PCB, 350mA was given; and for 3W LED assembled PCB, 700mA was given.


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Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T	, J121, 1 hia, H harjee, D1 I	133 .71 .80 175 .76 .50
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M	, J	133 .71 .80 175 .76 .50 38
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingije	, J	133 .71 .80 175 .76 .50 38 26
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyie,	, J	133 .71 .80 175 .76 .50 38 .26 26
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Bingyi, Bingyi,	, J	<ol> <li>133</li> <li>.71</li> <li>.80</li> <li>175</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.26</li> <li>.07</li> </ol>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Bingyi,	, J	<ol> <li>133</li> <li>.71</li> <li>.80</li> <li>175</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.26</li> <li>107</li> <li>47</li> </ol>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis Blejde,	, J	133 .71 .80 175 .76 .50 38 .26 .26 107 .47
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis, Blejde, Bo, W	, J	133 .71 .80 175 .76 .50 38 .26 .26 107 .47
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis, Blejde, Bo, W Bocciar	, J	<ol> <li>133</li> <li>.71</li> <li>.80</li> <li>175</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.26</li> <li>.26</li> <li>.07</li> <li>.47</li> <li>.92</li> <li>.37</li> </ol>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis Blejde, Bo, W Bocciar Bohlen,	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>175</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.26</li> <li>.26</li> <li>.26</li> <li>.47</li> <li>.92</li> <li>.37</li> <li>.75</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot	, J	133 .71 .80 175 .76 .50 38 .26 .26 107 .47 192 137 .75 .61
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>.75</li> <li>.76</li> <li>.50</li> <li>.38</li> <li>.26</li> <li>.26</li> <li>.26</li> <li>.07</li> <li>.47</li> <li>.92</li> <li>.37</li> <li>.75</li> <li>.61</li> <li>.66</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>175</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.26</li> <li>.26</li> <li>.07</li> <li>.47</li> <li>.92</li> <li>.47</li> <li>.92</li> <li>.37</li> <li>.61</li> <li>.66</li> <li>.88</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boz, U	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>175</li> <li>.76</li> <li>.50</li> <li>.38</li> <li>.26</li> <li>.26</li> <li>.26</li> <li>.07</li> <li>.47</li> <li>.92</li> <li>.37</li> <li>.75</li> <li>.61</li> <li>.66</li> <li>.88</li> <li>.61</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>175</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.26</li> <li>.26</li> <li>.47</li> <li>.92</li> <li>.37</li> <li>.61</li> <li>.66</li> <li>.88</li> <li>.61</li> <li>.66</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonma	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>175</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.26</li> <li>.26</li> <li>.47</li> <li>.92</li> <li>.37</li> <li>.61</li> <li>.66</li> <li>.68</li> <li>.61</li> <li>.90</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonma	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>175</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.26</li> <li>.07</li> <li>.47</li> <li>.92</li> <li>.37</li> <li>.75</li> <li>.61</li> <li>.66</li> <li>.88</li> <li>.61</li> <li>.90</li> <li>.82</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonma Boorea, O	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>175</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.26</li> <li>.26</li> <li>.07</li> <li>.47</li> <li>.92</li> <li>.37</li> <li>.75</li> <li>.61</li> <li>.66</li> <li>.88</li> <li>.61</li> <li>.90</li> <li>.82</li> <li>.24</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonpra Borca, ( Borek, N	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>175</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.26</li> <li>107</li> <li>.47</li> <li>192</li> <li>137</li> <li>.75</li> <li>.61</li> <li>.66</li> <li>188</li> <li>161</li> <li>190</li> <li>182</li> <li>124</li> <li>152</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingjie, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonma Booner, Borca, C Borce, Y	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>175</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.26</li> <li>107</li> <li>.47</li> <li>192</li> <li>137</li> <li>.75</li> <li>.61</li> <li>.66</li> <li>188</li> <li>161</li> <li>190</li> <li>182</li> <li>124</li> <li>152</li> <li>173</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Birbilis Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonma Borca, C Borck, T Borca, C	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>.75</li> <li>.76</li> <li>.50</li> <li>.38</li> <li>.26</li> <li>.26</li></ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boores, Borca, C Bores, Y	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>.75</li> <li>.76</li> <li>.50</li> <li>.38</li> <li>.26</li> <li>.26</li> <li>.07</li> <li>.47</li> <li>.92</li> <li>.37</li> <li>.61</li> <li>.66</li> <li>.66</li> <li>.66</li> <li>.61</li> <li>.68</li> <li>.68</li> <li>.68</li> <li>.68</li> <li>.68</li> <li>.68</li> <li>.68</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonma Borca, C Borek, Y Borozna Boswel Botor, J	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>175</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.26</li> <li>107</li> <li>.47</li> <li>192</li> <li>137</li> <li>.75</li> <li>.61</li> <li>.66</li> <li>.88</li> <li>.61</li> <li>.90</li> <li>.82</li> <li>.24</li> <li>.68</li> <li>.68</li> <li>.68</li> <li>.68</li> <li>.68</li> <li>.68</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingjie, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonpra Borca, C Borek, Y Borozna Boswel Botor, J Bouaziz	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>.75</li> <li>.76</li> <li>.50</li> <li>.38</li> <li>.26</li> <li>.26</li> <li>.26</li> <li>.07</li> <li>.47</li> <li>.92</li> <li>.37</li> <li>.61</li> <li>.66</li> <li>.88</li> <li>.61</li> <li>.90</li> <li>.82</li> <li>.24</li> <li>.68</li> <li>.82</li> <li>.80</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingjie, Bingjie, Bingjie, Bingji, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonma Boorca, C Borca, C Borca, J Bouaziz Bouzy,	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>175</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.26</li> <li>.26</li> <li>.07</li> <li>.47</li> <li>.47</li> <li>.492</li> <li>.47</li> <li>.61</li> <li>.66</li> <li>.88</li> <li>.61</li> <li>.90</li> <li>.82</li> <li>.80</li> <li>.87</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Birbilis Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonma Borca, C Borca, C Borca, C Borca, J Borozna Boswell Botor, J Bouzziz Bouzzy, Bo You	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>.75</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.26</li> <li>.007</li> <li>.47</li> <li>.92</li> <li>.37</li> <li>.61</li> <li>.66</li> <li>.88</li> <li>.61</li> <li>.90</li> <li>.82</li> <li>.40</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boorna Borca, C Borek, Y Borozna Boswel Botor, J Botor, J Bouaziz Bouzy, Bo You: Bradbul	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>.75</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.26</li> <li>.07</li> <li>.47</li> <li>.92</li> <li>.37</li> <li>.61</li> <li>.66</li> <li>.88</li> <li>.61</li> <li>.90</li> <li>.82</li> <li>.73</li> <li>.68</li> <li>.82</li> <li>.80</li> <li>.82</li> <li>.80</li> <li>.82</li> <li>.80</li> <li>.82</li> <li>.80</li> <li>.82</li> <li>.80</li> <li>.81</li> <li>.82</li> <li>.83</li> <li>.83</li> <li>.84</li> <li>.84</li> <li>.84</li> <li>.84</li> <li>.84</li> <li>.84</li> <li>.84</li> <li>.85</li> &lt;</ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boorek, Borek, Borek, Borozna Borek, Borozna Boswel Botor, J Bouaziz Bouzy, Bo Yout Bradbul Brandt	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>.75</li> <li>.76</li> <li>.50</li> <li>38</li> <li>.26</li> <li>.07</li> <li>.47</li> <li>.92</li> <li>.37</li> <li>.61</li> <li>.66</li> <li>.88</li> <li>.61</li> <li>.90</li> <li>.82</li> <li>.24</li> <li>.152</li> <li>.61</li> <li>.68</li> <li>.82</li> <li>.80</li> <li>.82</li> <li>.82</li> <li>.80</li> <li>.82</li> <li>.81</li> <li>.82</li> <li>.82</li> <li>.81</li> <li>.82</li> <li>.82</li> <li>.83</li> <li>.84</li> <li>.82</li> <li>.81</li> <li>.82</li> <li>.83</li> <li>.84</li> <li>.84</li> <li>.84</li> <li>.84</li> <li>.84</li> <li>.84</li> <li>.85</li> <li>.85</li> <li>.86</li> <li>.86</li> <li>.87</li> <li>.40</li> <li>.38</li> <li>.20</li> </ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingjie, Bingji, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonma Borek, Borozna Borek, Borozna Borek, Botor, J Bouaziz Bouzy, Bo You Bradbu Brandt, Brandt,	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>.75</li> <li>.76</li> <li>.50</li> <li>.38</li> <li>.26</li> <li>.26</li> <li>.07</li> <li>.47</li> <li>.92</li> <li>.37</li> <li>.61</li> <li>.66</li> <li>.88</li> <li>.61</li> <li>.62</li> <li>.61</li> <li>.66</li> <li>.61</li> <li>.66</li> <li>.61</li> <li>.62</li> <li>.61</li> <li>.66</li> <li>.61</li> <li>.66</li> <li>.61</li> <li>.66</li> <li>.61</li> <li>.66</li> <li>.61</li> <li>.66</li> <li>.61</li> <li>.62</li> <li>.61</li> <li>.62</li> <li>.61</li> <li>.62</li> <li>.61</li> <li>.62</li> <li>.61</li> <li>.62</li> <li>.61</li> <li>.62</li> <li>.64</li> <li>.64</li> <li>.64</li> <li>.65</li> <li>.64</li> <li>.64</li> <li>.65</li> <li>.65</li></ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingjie, Bingjie, Bingjie, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonma Booner, Borca, C Borca, C Borca, J Bouaziz Bouzy, Bo You Bradbul Brandt, Bravo, A	, J	133           .71           .80           .75           .76           .77           .92           .37           .75           .61           .66           .88           .61           .62           .82           .80           .82           .80           .82           .80           .82           .83           .82           .83           .82           .83           .82           .83           .83           .83           .84           .94
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Birbilis Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonma Booner, Borca, C Borca, C Borca, C Borozna Botaziz Bouzy, Bo You Bradbul Brandt, Bravo, A Brechet	, J	<ul> <li>133</li> <li>.71</li> <li>.80</li> <li>.75</li> <li>.76</li> <li>.76</li> <li>.26</li> <li>.26</li> <li>.007</li> <li>.47</li> <li>.92</li> <li>.37</li> <li>.61</li> <li>.66</li> <li>.88</li> <li>.61</li> <li>.90</li> <li>.82</li> <li>.80</li> <li>.81</li> <li>.81</li> <li>.82</li> <li>.80</li> <li>.82</li> <li>.80</li> <li>.81</li> <li>.81</li> <li>.82</li> <li>.81</li> <li>.82</li> <li>.81</li> <li>.82</li> <li>.81</li> <li>.82</li> <li>.81</li> <li>.82</li> <li>.81</li> <li>.81</li> <li>.81</li> <li>.81</li> <li>.81</li> <li>.81</li> <li>.82</li> <li>.81</li> <li>.81</li></ul>
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Birbilis. Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boorar Borca, C Borek, Y Borozna Borozna Borozna Borozna Botor, J Bouzziz Bouzy, Bo You Bradbu Brandt, Bravo, A	, J	133         .71         .80         175         .76         .50         .82         .26         .007         .92         .37         .75         .61         .68         .61         .90         .82         .73         .68         .82         .80         .82         .83         .84         .82         .83         .84         .85
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingyi, Birbilis Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonar Borca, C Borek, Y Borozna Boswel Botor, J Botor, J Botor, J Bouaziz Bouzy, Bo You Bradbul Brandt, Bravo, A Brechet Breen, A	, J	133         .71         .80         .77         .76         .50         .76         .75         .61         .66         .88         .61         .90         .82         .73         .68         .82         .80         .82         .82         .82         .83         .74
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bilek, M Bingjie, Bingjie, Bingji, Birbilis, Blejde, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonma Borek, Borozna Borek, Borozna Borek, Botor, J Bouaziz Bouzy, Bo You Bradbun Brandt, Brandt, Brandt, Brech, A Brice, C Brinckn	, J	133         .71         .80         175         .76         .50         38         .26         .07         .92         .37         .75         .61         .68         .61         .68         .61         .68         .61         .62         .63         .64         .65         .61         .68         .61         .62         .63         .64         .64         .65         .82         .80         .82         .80         .82         .82         .82         .80         .82         .83         .74         .80
Beynon Bhadesl Bhattac Bian, J Bian, M Bigg, T Bilek, M Bingjie, Bingjie, Bingjie, Bingjie, Bingjie, Bo, W Bocciar Bohlen, Boidot, Boland, Bonek, Boo-Hy Boonma Booner, Borca, C Borek, Y Borozna Boswel Botor, J Bouaziz Bouzy, Bo You Bradbul Brandt, Bravo, A Brechet Breen, A Brice, C Brinckn Brown,	, J	133         .71         .80         .75         .76         .50         .38         .26         .007         .47         .92         .37         .75         .61         .66         .88         .61         .62         .75         .61         .62         .63         .64         .82         .80         .82         .80         .82         .80         .82         .83         .840         .22         .88         .80         .82         .880         .82         .880         .82         .880         .82         .880         .82         .880         .82         .880         .840         .840          .840          .841          .852          .8
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