# **Cover sheet**

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# In-situ Si/Al Composite Produced by Semisolid Metal Processing

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Hypereutectic aluminium-silicon alloy can be considered as an *in-situ* composite with primary silicon acting as the reinforcing phase. In this paper, we use semisolid metal processing technique to produce such *in-situ* Si/Al composite. Hypereutectic Al-Si alloy, AlSi17Cu5Mg0.55, was cast into a permanent mould with controlled solidification conditions. In the resulted composite, primary Si particles were observed to co-exist with primary  $\alpha$ -Al particles, uniformly dispersing in Al-Si eutectic matrix. The structure of the Si particle can be controlled by AlP refinement and thermal refinement, and the  $\alpha$ -Al particle can be controlled by semisolid partial remelting.

Keywords: Aluminium-silicon alloy; Hypereutectic aluminium alloy; *in-situ* aluminium-based composite, Semisolid metal processing; AlP refinement.

### Introduction

Hypereutectic Al-Si alloys can be considered as in-situ natural metal matrix composites with primary silicon acting as the reinforcing phase. They combine the beneficial properties of the metal matrix, such as good ductility, good thermal and electrical conductivity, and high toughness, with the properties of the silicon reinforcing phase, such as high wear resistance, high yield strength and low thermal expansion. The materials have attracted increasing interest in automotive applications, especially in heavy wear components, such as pistons, cylinder blocks, pump bodies and compressors [1,2]. However, their use has always been tempered by several difficulties, in particular, their high latent heat and consequent long solidification time resulting in die wear, and difficulty in controlling the size and distribution of the primary Si phase [3]. In hypereutectic Al-Si alloys, the Si crystal, once nucleated, can grow to a large and irregular size.

Semisolid metal processing provides an attractive way to process metal matrix composites, in which the reinforcement can be pre-controlled in the semisolid feedstock production. During the subsequent semisolid casting processes, the Al matrix is remelted while the Si particle remain as solid, therefore the initial Si structure is retained in the final semisolid castings [4,5]. Moreover, using semisolid casting, the casting temperature and heat content are very much reduced, which results to less die wear and total shrinkage. Production of hypereutectic Al-Si alloy with small primary silicon particles has been reported using electromagnetic stirring [2], mechanical stirring [6] and ultrasonic treatment [7]. This paper presents the results of producing *in-situ* Si/Al composite by semisolid metal processing.

## Experimental

Hypereutectic Al-Si alloy with a normal composition of AlSi17Cu5Mg0.65 was prepared using commercial purity Al, Si, Mg and Cu. The materials were charged in an induction furnace. The melt was raised to 750°C, then degassed for 10 min with argon bubbled through a graphite lance. Aluminium phosphide was used to refine the primary silicon phase at P level of 75 ppm by adding master alloy ALCUP rod. ALCUP is AlP ready master alloy. The melts with and without AlP were cast into steel moulds specially designed for rapid heat extraction. One mould had a cylindrical cavity of 50 mm in diameter and 70 mm deep to obtain a lower cooling rate

and the other with a cylindrical cavity of 20 mm in diameter and 200 mm deep for a higher cooling rate. The pouring temperature was 770°C and the mould temperature was 200°C.

Small cylindrical samples (20 mm in diameter and 10 mm in height) were cut from the castings for semisolid partial remelting, which was conducted in a molten salt-bath. The salt bath was used for rapid heating (about 15°C/s) and good thermal contact. The samples were isothermally held in semisolid region at 570°C for 10 min, and then quenched in iced for microstructural examination. water Metallographic samples were prepared using a standard procedure with a final polishing stage of 0.05 µm colloidal silica. The microstructures of the samples were examined using optical microscopy.

#### **Results**

#### As-cast microstructure

Figure 1 shows the as-cast microstructures of the materials with and without AlP refinement, and solidified at two different sizes of the mould, which refers to two different cooling rates. The microstructure consists of primary Si particles, primary dendritic  $\alpha$ -Al particles and Al-Si eutectic. The coexistence of primary Si and primary  $\alpha$ -Al phase was not expected from the phase diagram. The special designed moulds had a large heat extraction rate during solidification, which avoids the "explosive" growth of silicon phase [3].

AlP is very effective in refining the primary Si phase. Without the AlP refinement, the size of the Si particle was about 100  $\mu$ m and the distribution was not uniform (Fig. 1a and b). After the AlP addition, the size of the Si particles was significantly reduced to about 40-50  $\mu$ m with a very uniform distribution (Fig. 1c). High cooling rate further reduced the particle size to 20  $\mu$ m.

In terms of  $\alpha$ -Al particles, AlP addition has less significant influence. It seems that the  $\alpha$ -Al particles became less clearly distinguishable from the eutectic in the AlP-containing samples. It is unclear whether this reflects a decrease in the fraction of primary Al formed or is just an apparent effect caused by different growth characteristics of the eutectic Si in the presence of P. High cooling rate also resulted in a slightly smaller Al dendrites.

When the AIP refinement was combined with high cooling rate, a very fine and uniform composite structure was obtained, with silicon particles at an average size of less than 20  $\mu$ m being uniformly dispersed in the aluminium matrix, as shown in Fig. 1d.



Figure 1.—As-cast microstructures of the samples without (a and b) and with (c and d) AlP refinement, (a and c) were with low cooling rate; and (b and d) were with high cooling rate.

#### Semisolid microstructures

The as-cast samples were partially remelted and isothermally held in the semisolid region at 570°C for 10 min. Figure 2 shows the semisolid microstructures of the Al-Si alloy with and without AIP refinement after isothermal holding. The microstructure suggests that the materials were about 40% solid at the holding temperature and the rest was liquid which had been remelted from the Al-Si eutectic. Both primary Si and primary Al co-existed as the solid and they were uniformly distributed in the liquid Al-Si eutectic. The primary Si structure did not change much from the as-cast condition. The particles were at a similar size to the as-cast materials, and the morphology remained polyhedral but slightly rounded. The primary Al dendrites, on the other hand, underwent significant structural evolution. The dendritic morphology transformed to a globular one in order to reduce the solid-liquid interface area. Most of the Al-Si eutectic became liquid, although the presence of mid-size Si, particularly in Fig. 2a suggested some coarsening of eutectic Si still remained solid.

For the AlP-free alloys (Fig 2a and b), the Si particles remained at 100  $\mu$ m in size at low cooling rate and 60  $\mu$ m at high cooling rate. The primary Al particles became globular with a size of 80  $\mu$ m and 60  $\mu$ m respectively. The AlP-refined samples showed a similar response to semisolid holding as that observed with non-AlP samples. The silicon particles had no significant changes in both size and morphology, 40  $\mu$ m for the lower cooling rate (Fig. 2c) and 20  $\mu$ m for



Figure 2.—Semisolid microstructures after isothermal holding at  $570^{\circ}$ C for 10 min. (a) and (b) were AlP free, and (c) and (d) were AlP refined. (a) and (c) were cast at the low cooling rate, and (b) and (d) were cast at the high cooling rate.

the higher cooling rate (Fig. 2d), while the primary  $\alpha$ -Al phase became very globular.

#### Solidification sequence

better understand In order to the solidification sequence, cooling curves of the alloy were used to identify phase-change temperatures and samples were then quenched from various points along the curve, as shown in Figure 3. The cooling rate in the fully liquid state was about 2°C/s, which is significantly slower than for any of the prepared microstructures, but allowed more accurate location of the quench point and a more efficient quench. The quenched microstructures are shown in Figure 4.

The cooling curve shows a recalescence between A and B, indicating the nucleation and growth of the primary Al. The primary Al phase nucleated in the Al-enriched zone adjacent to the primary Si crystals, then grew into the melt. At point C the Al-Si eutectic reaction was well under way, with no recalescence to mark where it began. This probably occurred soon after point B. The recalescence at around 500°C was presumed to relate to the appearance of  $Al_2Cu$  in the eutectic.



Figure 3.—Cooling curve illustrating the locations at which samples in Fig. 4 were quenched.

#### Discussion

Solidification of the hypereutectic Al-Si alloy begins with the precipitation of primary silicon at about 654°C. From the equilibrium phase diagram, the next phase should be Al-Si eutectic phase, and the final microstructure should consist of primary silicon and Al-Si eutectic, plus some complex eutectic phases formed due to the existence the minor elements.



Figure 4.—Quenched microstructures from points defined in Fig. 3. (a) point A, before primary aluminium precipitation, (b) point B: during growth of primary aluminium precipitation, and (c) point c, during the Al-Si eutectic growth.

In this work, it was observed that both primary silicon and primary aluminium coexist in the ascast microstructure (Fig. 1 and 2). The precipitation of primary aluminium phase is not uncommon in Si-rich alloys and is usually explained in terms of eutectic growth switching from the liquidus extension on one side of the eutectic coupled zone to the other, driven by nucleation difficulties and solute build-up. The coexistence of both silicon and aluminium phases in a primary form has also been reported by Arnberg et al [8] and Kapranos et al [2].

During the partial remelting and isothermal holding, the primary silicon particles remain solid, so the size and morphology of silicon particle need to be carefully controlled in the ascast stage. In hypereutectic Al-Si alloys, without heterogenous nuclei, primary Si nucleates only after a deep undercooling. If it is nucleated with high undercooling, the crystal can grow very quickly to a large size [3]. AlP refinement and thermal refinement are two effective methods to control the primary silicon size. Fig. 1 indicated that AlP addition more than halved the size of primary Si crystals. AlP acts as an effective nucleus for the Si phase because AlP has basically the same crystal structure as silicon with very similar lattice parameters. The refined samples retain a fine silicon structure after the semisolid isothermal holding.

Thermal refinement is another method for controlling the size and morphology of the primary silicon phase. Higher cooling rate results in smaller silicon particle size. This was observed to some extent in AlP-free samples but seems more effective when combined with AlP refinement. With the combination of AlP refinement and thermal refinement, a very fine and uniform composite structure has been achieved. The size of the Si reinforcement particles was about 20  $\mu$ m. The fine silicon structure remains unchanged in the semisolid status (Fig. 2d).

During semisolid reheating, the isothermal temperature was above the apparent eutectic temperature (565°C, Fig. 3), and it was above the temperature at which the primary Al formed. However the presence of solid Si with Al in the partially remelted material means that the holding temperature must have been below the equilibrium eutectic temperature for that

composition. The apparent discrepancies must reflect the relatively high undercoolings that normal solidification entails for this alloy.

If this was a pure binary alloy then the eutectic temperature would be an invariant and it would not be possible to have this semisolid structure during isothermal holding. The shape of the fraction-solid vs temperature curve in this region is determined entirely by the minor element concentrations and allows the formation of this semisolid microstructure. This is fortunate, because the amount of primary Si by itself is insufficient to permit ready utilisation in semisolid forming. One drawback, on the other hand, is that the fraction solid in this region is more sensitive to temperature errors and composition variations than, for example, Al-7Si alloys.

The primary aluminium dendrites undergo a significant microstructure evolution. As the globular structure is more compact than a dendritic structure, the final size of the globular aluminium particles is smaller, at size of about 60-80  $\mu$ m. Some of the "primary" Al may well have remelted, assisting the globularisation process. The as-cast primary Si often has a halo of Al, no doubt from the layer rich in Al that would have been built up during initial growth of the Si. This appears to break up during isothermal holding, perhaps driven by a relatively high surface energy between solid Al and solid Si. This has helped the particle size remain small.

## Conclusions

1. *In-situ* Si/Al composite structure can be produced in hypereutectic Al-Si alloys using semisolid metal processing. The structure consists of very fine silicon particles and globular aluminium particles being uniformly dispersed in the Al-Si eutectic matrix.

2. The silicon particles can be controlled using AlP refinement and thermal refinement. A combination of the two produced the smallest Si particles, with a size of 20  $\mu$ m. The silicon structure remains almost unchanged during semisolid remelting.

3. The  $\alpha$ -Al particle has a dendritic form at as-cast condition. After partial remelting, the dendritic structure evolves to a globular

structure. Uniform globular structure of  $\alpha$  phase with an average particle size of 60  $\mu$ m was achieved.

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