Improving the Tensile Strength of Carbon Nanotube Spun Yarns using a modified spinning process

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Abstract.

A modified process for the dry spinning of carbon nanotube (CNT) yarn is reported. The approach gives an improved structure of CNT bundles in the web drawn from the CNT forest and in the yarn produced from the twisted web leading to improved mechanical properties of the yarn. The process enables many different mechanical and physical treatments to be applied to the individual stages of the pure CNT spinning system, and may allow potential for the development of complex spinning processes such as polymer-CNT based composite yarns. The tensile strength and yarn/web structure of yarn spun using this approach have been investigated and evaluated using standard tensile testing methods along with scanning electron microscopy. The experimental results show that the tensile properties were significantly improved. The effect of heat treatments and other yarn constructions on the tensile properties are also reported.

1. Introduction

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Research studies on CNTs have revealed a unique atomic structure with interesting related properties such as high tensile strength and Young's modulus, high aspect ratio, and good electrical and thermal conductivities [1-4]. These unique properties open up various novel application areas, for example, in nano-mechanics [5], advanced electronics [6], and bio-technology [7].

Significant efforts have been carried out to fabricate macroscopic structures to make use of the CNT's unique properties. Many promising techniques and results have been published [8-13]. In addition blended polymer fibre-CNT composites with improved physical and mechanical properties have been studied [14].

The first publication on spinning continuous CNT yarn from Professor Fan's group at Tsinghua University [8] resulted in many other researchers being attracted to the field of improving the properties of CNT based yarns and films using a variety of techniques. More recently the Commonwealth Scientific and Industrial Research Organisation (CSIRO, Australia) has collaborated with the University of Texas at Dallas (USA) to develop a dry-spinning process to produce CNT yarns from substrate-based 'forests' of multi wall CNTs (MWCNTs) [10]. Like some other groups [8, 15], this CSIRO work was based on traditional textile spinning principles. Twist was applied to the web as it was drawn from the forest and the applied twist imparted tensile strength to the yarn without any chemical binders.

To date, two distinct methods have been utilized to improve the properties of CNT yarns, firstly the development of longer and better aligned CNT forests on wafers [13, 15-17] and secondly, techniques to improve the spinning performance of CNT yarns and in this spirit this paper reports on further developments to the CNT yarn spinning process. This spinning development aims to significantly improve the mechanical and physical properties of CNT-based macro-structures and products.

2. Review of the yarn formation and associated interactions in CNT yarn structure and dry spinning process

2.1 *Formation of yarn and interactions of CNTs in yarn structure*

At the micro-structural level, within the yarn, there is a hierarchical structure containing two levels: (i) individual CNTs at the fundamental level and (ii) bundles of aggregated CNTs (so called secondary fibres). These bundles form a continuous network, called the web, with a preferred orientation along the yarn axis.

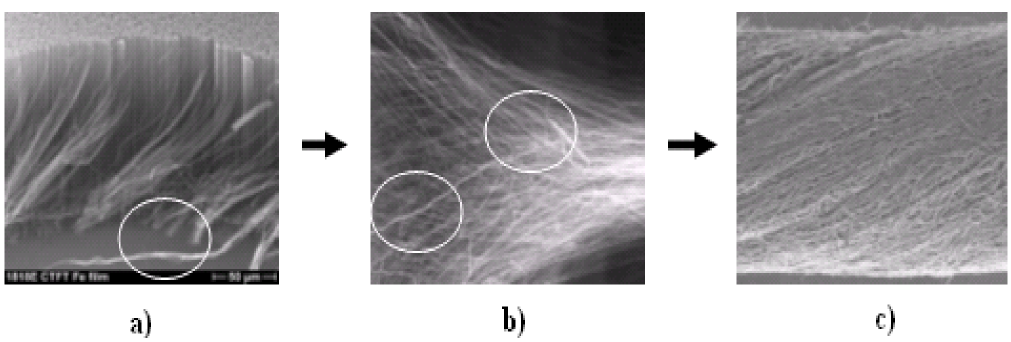

Figure1. The structure of the successive stages of the CNT spinning process: a) CNT forest structure; b) web structure and c) yarn structure. The middle picture

(b) is borrowed from a video clip of the dry spinning CNT process. (Courtesy of NanoTech Institute, University of Texas at Dallas)

The micrographs (Figures 1 a,b) show that the CNTs are not individualised in the web, instead they tend to be stripped from the forest wall in a range of bundle sizes. According to recent publications [18, 19], CNTs usually form into bundles containing up 100 parallel CNTs and these have been described as nano-ropes. The schematic cross-sections of three and seven CNT bundles are given in Figure 2 in which r is the CNT diameter and d is the gap between CNTs. The CNT interaction forces which cause bundles to be formed are based on van der Waals forces. The cohesive energy for the formation of a bundle over retaining a free-standing tube depends on tube diameter [20].

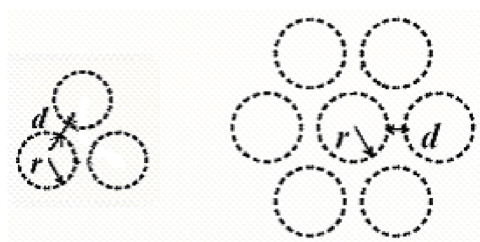

Figure 2 Schematic of the cross-section of three and seven CNT bundles.

This interactive energy of CNT bundles have been approximated by summing up the inter-CNT atom–atom pair energies Φ_i, using a Lennard Jones potential as follows [21]

$$\phi_i = \frac{\mathbf{A}}{\sigma^6}\left[\frac{1}{2}\mathbf{y}_o^6 \frac{1}{\left(\frac{\mathbf{r}_i}{\sigma}\right)^{12}} - \frac{1}{\left(\frac{\mathbf{r}_i}{\sigma}\right)^{6}}\right] \quad (1)$$

where r_i is the distance between the i^{th} atom pair, σ is the C–C bond length and A and y_0 are constants. The parameters for the Lennard Jones potential (A, y_o) have been further described by Girifalco and Lad [22].

The computational modelling [23] and the experiments of Ajayan [24] reported that the breaking force for bundles was from the sliding of CNTs rather than breakage of individual CNTs. Furthermore the sliding of CNTs along the axial direction caused a corrugation effect. Therefore the breaking force of a bundle can be quantified by the length of contact (*l*) between neighbouring CNTs and it consists of two components (i) the sliding force (F_1) and the force to overcome the corrugation effect (F_2) [23, 25-27] as follows

$$F = F_1 + F_2 = F_1 + lb \quad (2)$$

where b is a constant value. While F_1 depends on the interactive energy of CNT bundles described above, Qian *et al*. [23] notes that once the diameter of the tube is assigned, F_2 scales linearly with the contact length.

The mechanical properties of yarn depend not only on the interaction of CNTs in bundles but also on the degree of condensation (or packing) of CNT bundles in the yarn structure. Since the inter-tube interactions in CNT bundles is very weak [28], to improve the mechanical properties, there is a need to use mechanical or chemical techniques to increase the packing density of the bundles in the structure, for example by (i) inserting twist [10] or (ii) absorbing volatile liquids, such as acetone [29].

2.2 *Dry spinning process*

This paper focuses on the dry spinning process as it avoids the use of volatile liquids which introduce additional hazards and operating costs. Zhang *et. al.* [10] described a dry spinning process to spin CNT yarns (Figure 1c) from substrate-based 'forests' of MWCNTs (Figure 1a).

The success of the dry spinning process was attributed in part to the degree of twist applied during spinning. The role of twist was to reduce the inter-CNT distance whilst fixing the relaxed cross-section shape for individual CNTs in the bundle and also increasing the van der Waals forces (Eq. 1). It can be seen in Figure 3 that twist (Ω) applied to the CNT web (2) produces the required inter-bundle lateral cohesion after the web was pulled out from forest wall on the wafer (7). The current dry spinning process schematically described in Figure 3 has some disadvantages as follows:

- The CNT web pulled out of the wafer is poorly orientated (see Figure 1.b). Since the CNT web is twisted immediately after being pulled out from the forest this arrangement is locked in place. Thus it is difficult for the CNT bundles to be further aligned in order to improve the structure of yarn.

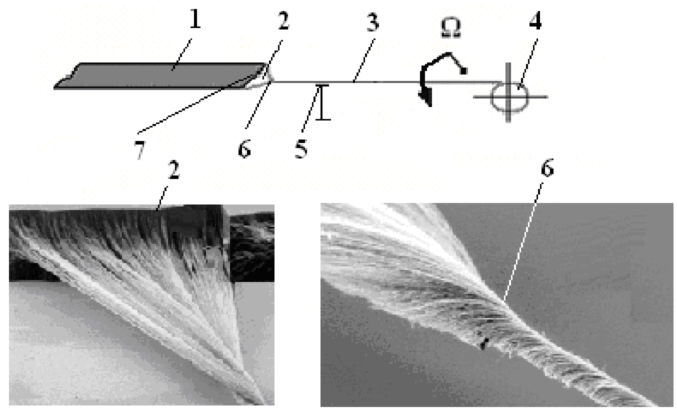

Figure 3: The schematic of a dry CNT yarn spinning

- The geometry (length) of the web is changeable and not controllable during spinning (The length of the web is defined as the distance from the forest wall where CNTs were pulled out to the convergence point on web/yarn). This causes heterogeneity in the tension of the web. Furthermore, the uniformity of the web tension depends on the ratio of the length to the width of the web strip. When the ratio is smaller (shorter triangle) the heterogeneity of web tension is higher. The experimental work showed that for the current dry spinning process, the length of triangle web is very short and the convergence point (6) (see Figure 3) tends to move close to the wall of CNT forest (7).

- Since there is no separation between the different stages (eg. web formation and twist insertion) of the current spinning process, processing web and yarn is either very difficult or not efficient.

In order to overcome these disadvantages, a modified dry spinning method is proposed and presented in the next section.

3. Modified dry-spinning process

The initial web of CNT bundles influences the packing structure of the yarn. For example, a web with a poorly oriented structure (Fig. 1b) results in yarns with less than optimal packing of the bundles because a large proportion of the CNT bundles are not aligned in the direction of the yarn. This produces yarns with inferior properties to those predicted from the properties of individual CNTs, that is, generally the tensile strength of CNT yarn spun by either the wet or dry-state spinning method is much lower than the strength of individual CNTs [9, 10].

We postulate that the dry spinning of CNTs brings together the characteristics of both traditional staple spun yarn and synthetic filament spinning, in which the web and yarn structures are the key factors affecting the yarn's physical and mechanical properties. In addition, the bundles of CNTs in web/yarn have similarities to the staple fibres of a spun yarn. Based on this idea and the interactions of CNTs in bundles described in Section 2.1, this work proposes a modified spinning system which seeks to enhance the CNT and bundle interaction by increasing the drawing and alignment of the CNTs in the yarn direction.

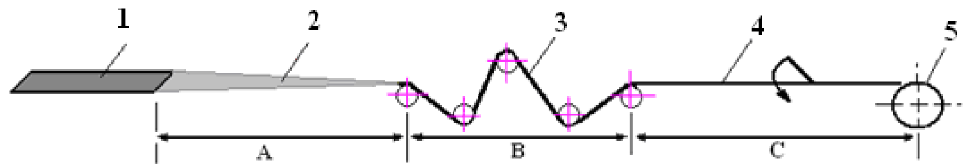

Figure 4: The schematic of modified CNT yarn spinning.

For the modified spinning process, the system was partitioned into three distinct zones (see Figure 4): web formation zone (A), tensioning/drafting zone (B), and twisting zone (C). The following description is general in nature and detailed calculations of our new process will be represented in the future publication.

3.1 *Web formation*

In the web formed from the forest, the bundles are oriented along the yarn axis in a variety of ways, some of them are bundles of parallel CNTs and others are poorly aligned or coiled (Figures 1b, 5b). It is desirable to have an aligned network of bundles of CNTs in the web structure as this is expected to improve the properties of CNT yarns and derived products. In order to improve the web structure, a web needs to be kept stable during the spinning process. With a CNT forest of given parameters (forest density, CNT length and diameter), web stability is improved by constant length of the web and uniform tension. The geometrical and kinetic model of the web can be obtained via the governing equation of the web formation with the following parameters: the tension T_w, the torque Ω caused by twisting and the combination force f_s of the CNT interaction, CNT-wafer interaction and the geometry of web (see Figure 5a). This will be represented in the future publication.

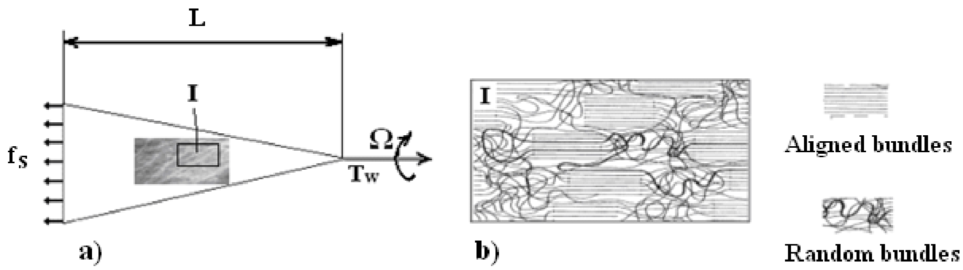

Figure 5 A model of CNT web structure. L is the length of CNT web.

After being pulled out from a wafer, the web was stabilized via the regulation of the tension and torque of a tensioning rod system described in the next section (Figure 6a). Since the web structure is only weakly bonded, this stabilization and isolation from the later spinning stage was important for allowing further treatments to be performed on the web (for example heat treatment) during the spinning process.

3.2 *Tensioning rod system*

In spite of the uniform tension, there are still random bundles as well as parallel ones in the web. As yarn passes through a capstan effect rod system (CERS) (Figure 6), the increased tension extends and aligns the bundles. Provided that the breaking extension of the yarn was not reached, some strain can occur. The change in tension as yarn passes around a rod (described in Figure 6) and the corresponding radial pressure on the yarn are given respectively as follows [30, 31]:

$$T_1 = T_W e^{\mu\theta} \quad (3a)$$

$$N_1 = \frac{T_1}{\mu}\left(e^{\mu\theta_1} - 1\right) \quad (3b)$$

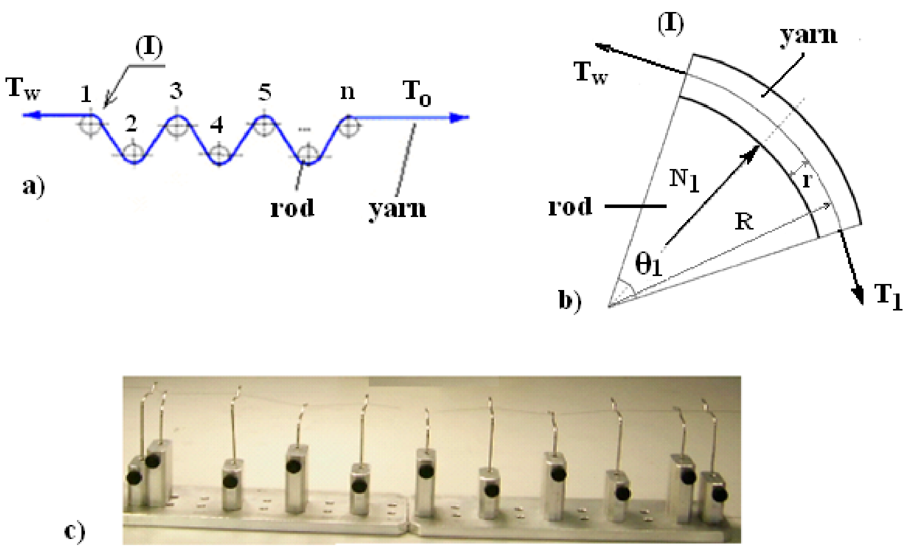

Figure 6.a) A schematic of the tensioning/drafting system using a capstan effect rod system (CERS); b) the change of tension and radial pressure in a yarn as it passes around a rod (first rod) and c) a photo of the experimental CERS. R and r are the rod and yarn diameters, respectively.

where μ, T_w, T_1, θ_1 and N_1 are the friction coefficient of yarn on the rods, the web (incoming) tension, outgoing tension, contact angle and radial pressure of yarn on the rod 1, respectively. The change in tension at the last rod of the CERS is given by

$$\mathbf{T_o} = \mathbf{T_w}\mathbf{e}^{\mu\sum_{i=1}^{n}\theta_i} \quad (5)$$

where T_o is the tension of CNT yarn rolled up on the bobbin; n is the number of rods and θ_i is the contact angle between yarn and i^{th} rod.

Although the yarn was subjected to gradually increasing tension ($T_w < T_1 < T_2 <\ldots< To$) through the CERS (Equation 5), the alignment and slip effects are observed to occur principally at the first rods, where CNT sliver (defined as bundles compacted of low density and without twist) is running through the CERS. The radial pressure imposed on the yarn at the rods, predominantly the later ones causes an increased CNT interaction in bundles and yarn. It was assumed that this process consists of two effects: (i) alignment of nanotubes in the fibres during the initial tensioning and (ii) the condensing or closer packing of the nanotubes together. The former effect increases the contact length between bundles and the latter one decreases the distance between tubes. Both effects are important to increase the tensile properties of the yarn, as the more aligned the fibre bundles, the higher will be the yarn compaction. The net effect being an increase in the tensile strength of yarns produced using this process compared to the process described in Section 2.1.

3.3 *Twisting zone*

As indicated earlier, twist is imparted to reduce the inter-CNT distance and then increase the van der Waals interaction force to produce the required inter-bundle lateral cohesion of the yarn. In the modified spinning process, the total twist reaches from the final rod of the CERS to the bobbin (Figure 7). A lower twist can be imposed on the yarn when running through the CERS by the adjustment of radial pressure of the yarn on rods via the contact angles (Equation 3b). This technique allows the influence of twist to occur after the CNT sliver has been stretched, as well as protecting the yarn from exceeding its breaking force.

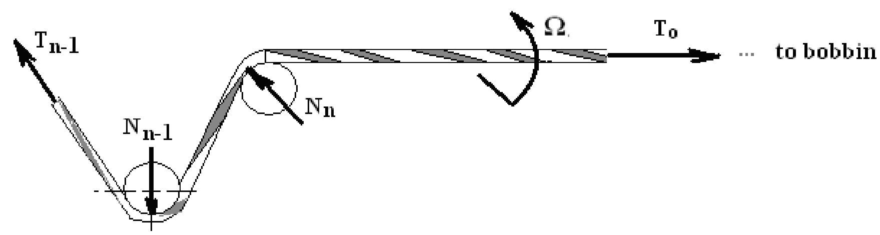

Figure 7 A schematic of the twist zone and final part of CERS showing the twist insertion.

Results show that the tenacity of a CNT yarn depends on stress transfer in shear mode between neighbouring CNT bundles. To achieve a higher tensile strength, the CNTs and bundles need not only to have a minimum length, but also they need to share maximum contact area.

4. Experimental results and discussion

It should be noted that the tenacity of a yarn depends not only on the spinning process but also on the quality and characteristics of the drawable forest, for example the CNT diameter and length and the density of the CNT forest. In fact, whilst the longer carbon nanotubes will increase inter-tube contact areas and therefore yield higher tensile strength, decreasing the CNT diameter may also increase the yarn tensile strength [12, 13, 15, 16]. In this work, the performance of the modified process has been investigated by comparing experimental results of tensile strength of CNT yarns produced using both methods.

Although the optimization of structural and engineering parameters of the modified process has still to be fully resolved, a significant improvement in the quality of CNT yarn has been obtained, especially enhancements in load transfer (the tensile strength) and the electrical conductivity which will be discussed in a later publication.

4.1 *The improvement of yarn tensile strength*

In this work, the experimental plan involved using MWCNT drawable forests grown on silicon wafers using Chemical Vapour Deposition (CVD). The CNT's length and outer diameter are approximately 300-400 μm and 7.5-8.5 nm, respectively. In order to remove the variability of the specification of the CNT forests from this investigation, the comparative results have been developed using MWCNT forest from the same wafer.

The modified system for spinning continuous twisted CNT yarn allows significant improvement in the alignment of bundles in the yarn. The Figures 8 b,c show a typical segment of web and sliver. The micrographs indicate that the bundles in web and sliver structure obtained by the modified process are parallel and highly aligned in the direction of the long axis when compared with the web in Figure 8a produced using the previous process as showed in Figure 3.

The scanning electron microscopy (SEM) images also depict the structural difference of sliver located between rod 1 and rod 2 of the CERS using the modified process without heat treatment (Figure 8c) and with heat treatment (Figure 8d) on the web.

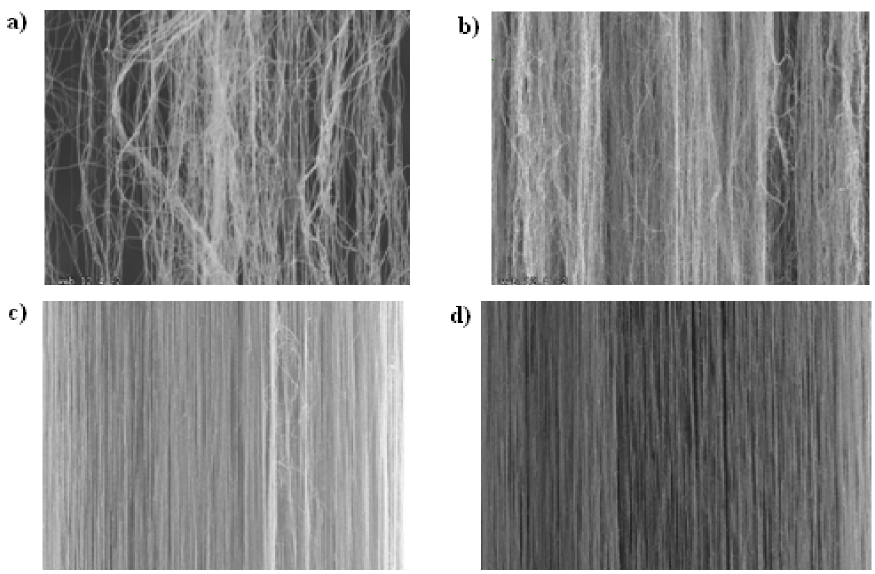

Figure 8. SEM image showing the microstructure of the bundles in webs; (a) web image using the previous process; b) web microstructure using the modified process; (c) web image under high tension between two initial rods (1) & (2) on the CERS (see Figure 6.a) and (d) web image under tension between two initial rods (1) & (2) with heat treatment.

Although the significance of the effect of CERS on the surface of CNT yarns has not yet been ascertained, the micrographs (Figure 9) show the surface layer of MWCNT yarns spun using the modified process and twisted 15000 turns per meter (TPM) with a heat treatment of 200^{o}C and 400^{o}C on the web.

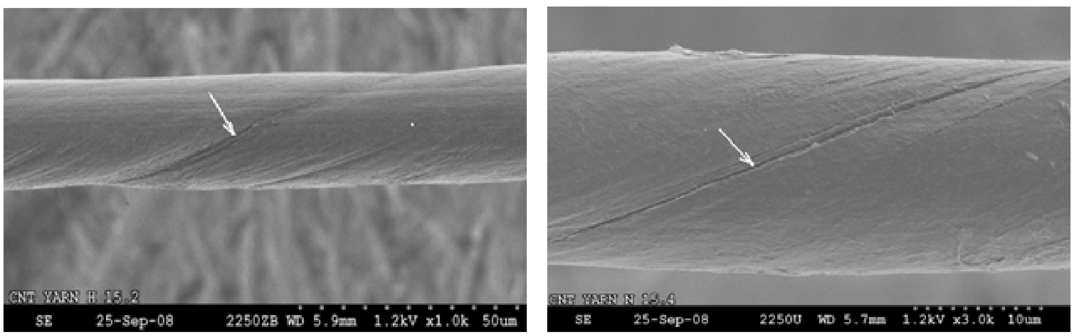

Figure 9. Micrograph (SEM images) of the surface layer for MWCNT yarn using the modified process: (a) yarn from web treated at 200^{o}C and (b) yarn from web treated at 400^{o}C. Arrow on the images shows the twist angle of yarn.

Twisting can enhance stress transfer between CNT bundles under tensile forces [24, 32]. However, the highly twisted CNT yarn tends to snarl or coil when unconstrained [33]. Figure 10 is a SEM image of a micro-snarling on a CNT yarn with 17000 TPM. The snarling occurs due to the imbalance between torque (caused by twisting) and the yarn tension.

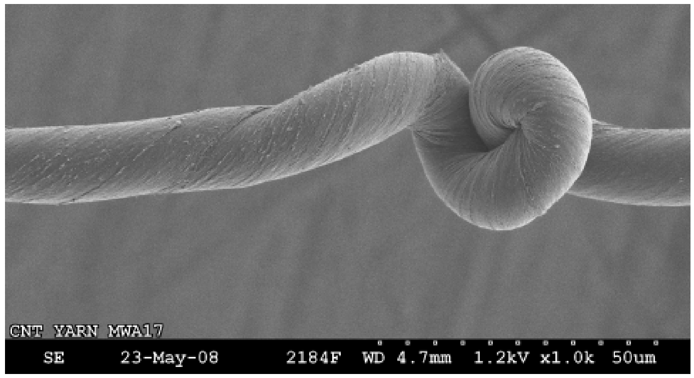

Figure 10. Micrograph of highly twisted yarn with densification: a snarl of yarn.

In order to investigate the tensile strength of CNT spun yarn, four drawable wafers of the same batch have been used to spin CNT yarns using the former process described in Section 2 and the modified process. All are spun using the same spinning parameters. Twist factor of 15000 TPM and the take-up speed of 6m/hour were used. The diameters of yarn samples were determined before testing their breaking force and extension on the tensile tester. The gauge length was 10mm. The results of 12 typical samples shown in Figure 11 depict that the tensile strength of the yarn from the new approach has increased significantly when compared with tensile results using the former process. The tensile strength and strain data covers a range from 970MPa to 1.4GPa and 5%-8%, respectively. The median tensile strength of 12 typical samples approaches 1.2GPa. The stress-strain curves also depicted that the new process produces yarn with reduced breaking elongation and increased Young Modulus. From the work reported earlier [12, 13, 15, 16, 17] the use of high quality CNT forests (longer and smaller diameter CNTs and cleaner and more aligned CNT forests) in our modified spinning process should produce correspondingly higher tensile strength yarns.

The improvement in the yarn structure was obtained by stretching CNT bundles and enabling more effective yarn compaction. This densification treatment was not only from the twist but also by the radial pressure through the CERS.

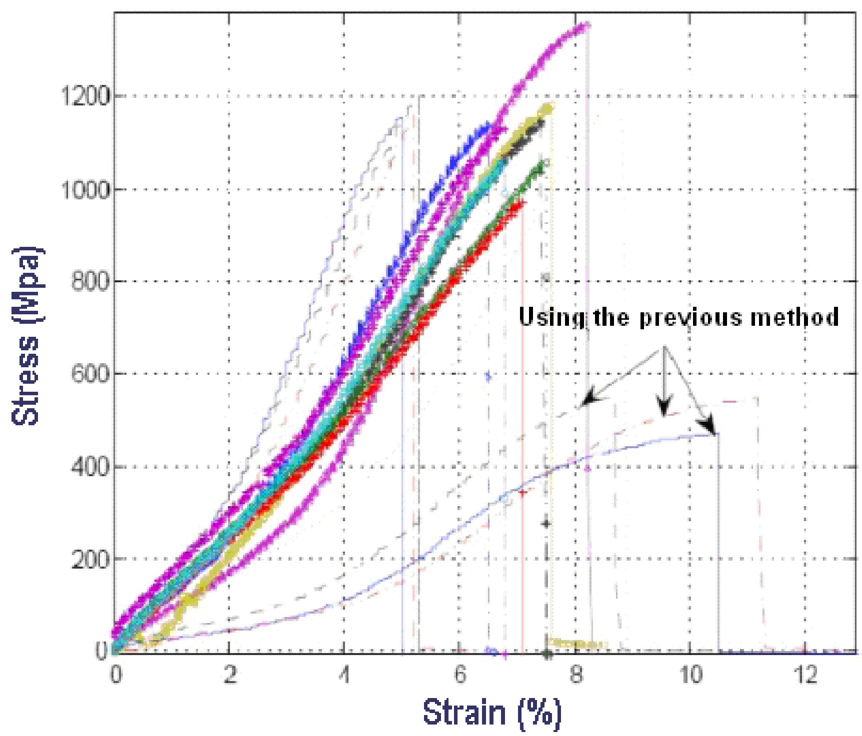

Figure 11 Stress-Strain curves of CNT yarn spun from the same drawable forest using the previous and modified processes: A comparison of the tensile strength of the CNT yarn samples spun from two methods.

4.2 *Several treatments on the individual stages of the modified process*

The separation of the different stages of distinctive mechanical and structural features of web and yarn allows for different treatments to be performed at individual stages of the spinning process. Several treatments have been carried out such as, heat treatment on the web, multi-web spinning from multi-wafers (Figure12) or single wafer (Figure13), and yarn annealing. Some of these have been represented in the next sections.

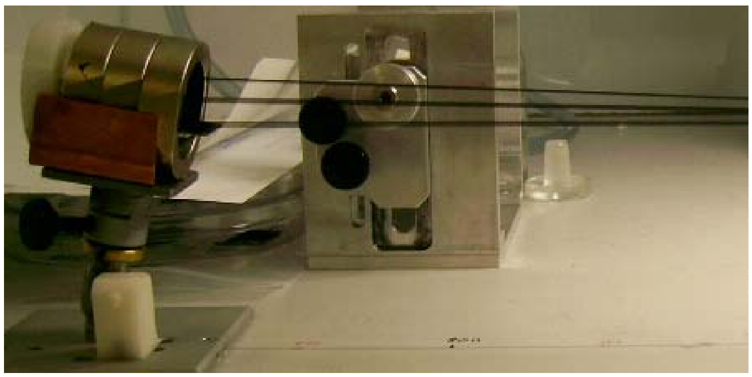

Figure 12 CNT Multi-web spinning using the modified process.

4.2.1 *Multi-web spinning*

Based on the principles of Sirospun [34] and multi-roving single yarn (Tran and Phillips, 2008)[1], CNT yarn has been spun from two-webs split on one single

[1] Tran CD, Phillips DG. The Fiber Society Conference, ENSISA Uni., Mulhouse, France, 2008; CD Proceedings.

wafer (Tran and Smith, 2008)[2]. The convergent point is located at the first rod of the CERS. Although it depends on many factors (the distance between the strips and position of the convergent point), the results of many different test samples on different wafers showed that the tensile strength was improved dramatically in comparison with one strip of web. Figure 13 depicts a typical result.

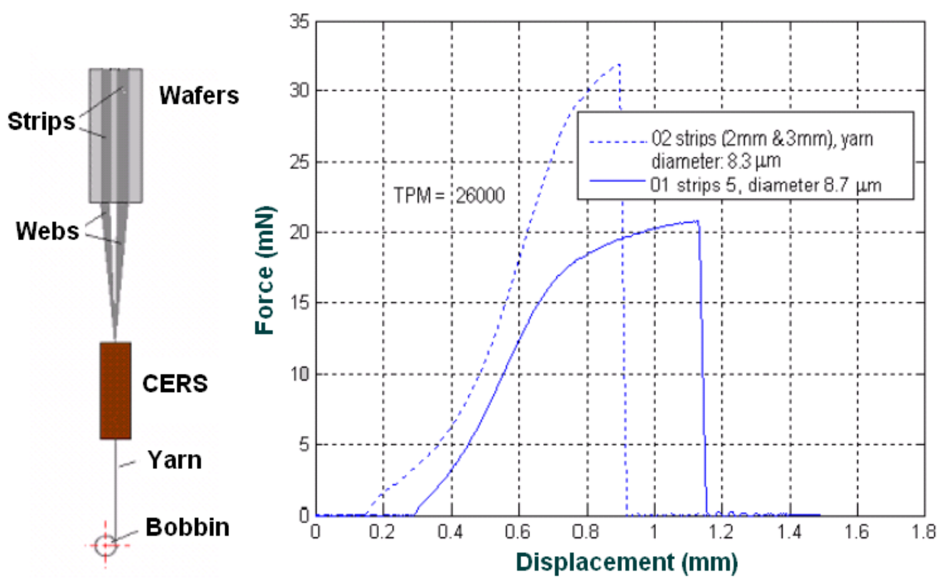

Figure. 13 Force-displacement curves of two typical samples of CNT yarn. The solid line is from one strip of 5mm and the dotted line is from two strips of 2mm and 3mm, respectively.

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[2] Tran CD, Smith SM. CSIRO Future Manufacturing Flagship Retreat & Science Symposium, 15-16 May 2008, Melbourne, Australia.

4.2.2 *Heat treatment of web*

It has been shown that the van der Waals force causes the inter-tubular CNT interactions related to the bundle and web formation and yarn compaction and that the magnitude of the force depends on thermal load and temperature [35-37]. Hence, temperature can be used as a method to control the CNT interaction during the CNT spinning process. In this section, heat treatment of the web has been considered as a factor in decreasing the van der Waals forces as the web is drafted through the CERS, thus increasing the efficiency of the CERS (Figure 14).

Figure 14 Heat treatment on web carried out at CSIRO

After extraction from the forest, the CNT web goes through a mini furnace whose temperature is adjustable from 200^{o}C to 600°C.

The results in Figure 15 showed that the temperature affects the mechanical properties of yarn via its effect on the van der Waals interaction between CNTs. Although the stretch of bundles and alignment level of CNTs depends on other factors such as tension, take-up speed and torque on the web and yarn, a reduction of inter-CNT interaction allows bundles to align more easily under the tension (from the CERS) and then the yarn is more compact under radial pressures. In Figure 15, the vertical bars describe the variation of the tensile strength of yarn samples at the different temperatures of heat treatment on the web. The dotted line represents the average values of tensile strength of six groups of yarn samples. Although further investigations are required with an optimisation of the CERS, the preliminary data has shown that the tensile strength reaches the highest value, at a web heat treatment temperature of 200^{o}C and then it tends to decrease as the temperature is further increased.

It is important to appreciate that the process of web heat treatment is carried out to improve the drafting properties of the CNT web. Web heat treatment can, but is not directly related to, strength improvements gained through the yarn annealing process. Yarn annealing has the objective of increasing the compaction of CNT yarn mentioned in section 4.2, but not detailed by the authors as an objective of this work.

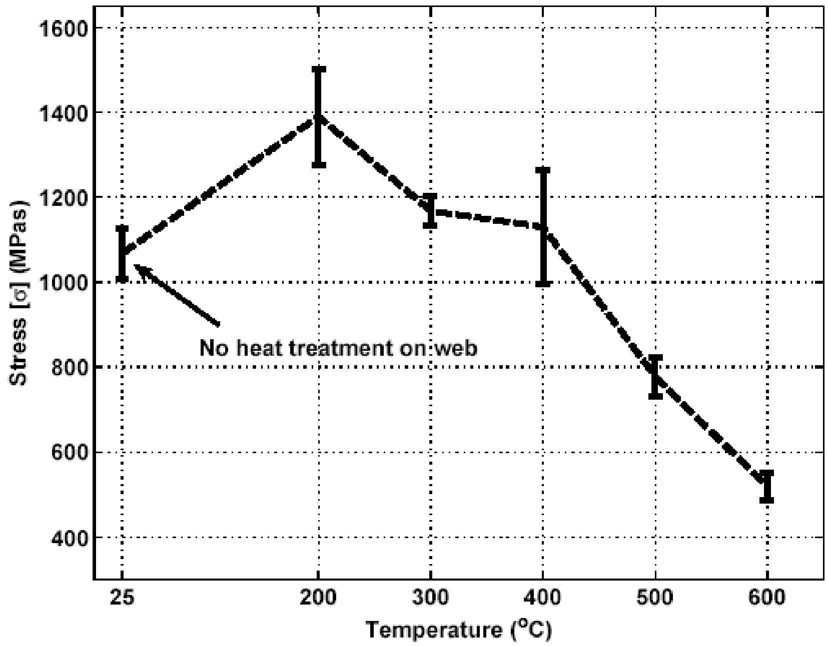

Figure15 Tensile strength of yarn samples plotted against the temperature of heat treatment on the web before going through the CERS (the dotted line represents the average values of tensile strength). The ambient temperature of the laboratory is 25°C.

5. Conclusions

A modified process for dry-spinning a web of CNTs into a yarn has been evaluated. The spinning system introduced a zone that allows some controlled tensioning of the CNT web and this significantly improved the alignment of the CNT bundles in the web/sliver and was transformed into a highly compacted yarn by twisting. This method led to a significant improvement of mechanical properties of yarn with the tensile strengths about double that of earlier studies.

The present modified process also allows different treatments in the individual zones, during the spinning, and the effect of heating of the web prior to twisting was evaluated and showed an optimum effect on the tensile strength at 200^{o}C. These preliminary results demonstrate that further refinement of this approach may contribute to the clarification of how the specific properties of drawable CNTs translate to the bulk properties of manufactured macroscopic structures. The optimization of the process parameters requires further investigation. The partitioning of the spinning system into separate zones has given new insights into the mechanisms involved and may allow further development of other types of CNT yarns, such as CNT composite yarns.

Acknowledgements

This work was supported by the CSIRO Future Manufacturing Flagship (Project R-306-01) and a grant of computing time from Australia Partnership for Advanced Computing (APAC) National Facility. The authors also note the technical assistance of Jill McDonnell, Jeff Baum and the Workshop (CMSE, Geelong). All of this support is gratefully acknowledged. The authors would like to thank Professor Ray Baughman, University of Texas at Dallas and Dr. David G. Phillips for their helpful discussion and the referees for their helpful comments.

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