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Investigation into microstructure of carbon nanotube multi-yarn ☆

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ABSTRACT

Structural analysis at the nano and micro scale was performed on a carbon nanotube (CNT) multi-yarn. The yarns were made by a process of drawing CNTs into a ribbon and twisting the ribbon into a yarn. Scanning electron microscopy (SEM) was used to view the exterior of the yarn. Polarized microscopy was used to examine details of the 1-yarn, and it also identified ribbon–ribbon boundaries. Further examination of interior structure was done by NanoCT scans which showed that folding of the ribbons had occurred causing complicated structures. The interior folding was found by milling into the yarn with a focus ion beam gun (FIB) and imaging with SEM. These different methods thus provided various microstructural details (structure, ribbon–ribbon boundary, folding and void fraction) of CNT multi-yarn which could be used to compare with other yarns fabricated with different procedures/sources as well to provide parameters for analytical tools. Further, these microstructural details can be related to macro mechanical and physical properties.

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1. Introduction

Carbon nanotubes (CNTs) have impressive mechanical and physical properties, therefore the development of CNT based products are currently under active consideration for commercial, industrial, and military applications. One such product is a long CNT based yarn [1]. The production of CNT yarns made by drawing CNTs from a substrate was first reported in 2002 [2]. Feng et al. described a process in which double-walled carbon nanotube thin films were produced by rolling these ribbons into a yarn that were then wrapped like a “roll of paper” [3]. Ma et al. showed that the tensile strength of the CNT yarn diminished with an increase in diameter [4]. Sabelkin et al. also found that the tensile strength of the multi-yarn decreased by increasing the number of yarns [5]. A possible explanation of why strength is reduced with an increase in size is due to the difference in tensile and compressive stiffness of CNTs. This can result in a flex induced kink-band failure mechanism in which separation of yarns occurs in the form of buckling and kinks at the boundary of tensile and compressive regions. Flex kink-bands are typically seen in polymeric fibers that have low compressive strength such as polyester and polyamide [6]. The CNT-yarns consist of aligned CNTs with limited lateral bonding, thus low

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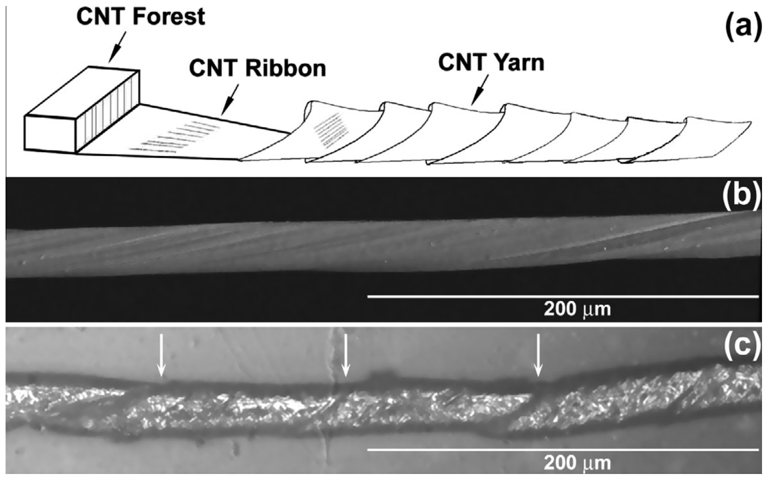

Fig. 1 – (a) Simplified schematic of a CNT-yarn formed from CNT forest, CNT 1-Yarn image by (b) SEM and (c) polarized light microscopy.

compressive strength is expected. However, CNTs are very resistant to failure in bending and are fully reversible up to 120° [7], and CNT-yarns can recover after creep [8]. Also, the tightness of the yarns is a concern; loose yarns can become torn or ripped when unevenly strained at a stress concentrator [9]. By fatiguing the multi-yarn, the yarn can be stretched in increments which will slowly tighten the loose yarns resulting in an increase in density, conductivity, and strength [10,11]. Still, this interaction between the multi-yarns as "rolled up ribbons" is still not well understood, and to date there still lacks an understanding of how these ribbons of CNTs are rolled into a yarn and what are the ramifications of twisting of outer yarns around the center yarns. In this study, polarized light microscopy, scanning electron microscope (SEM) and NanoCT scans were used to characterize the microstructure of the yarn. This information is useful in understanding of physical and mechanical properties as well as failure mechanisms of multi-yarn CNT wires/conductors.

2. Methods

Two types of CNT yarns, consisting of 1-yarn or 100-yarn, were procured from the Nanocomp Technologies, Inc., Concord, New Hampshire, USA. The CNT 1-yarn has a linear density of 0.98 ± 0.10 tex and tenacity of 65 ± 08 cN/tex [5]. The CNT 100-yarn has a linear density of 253 ± 5.3 tex and tenacity of 29 ± 0.4 cN/tex [5]. A Quanta 200 3D focus ion beam (FIB) SEM was used to mill holes in the yarn exposing internal information. The FIB emitted Ga^+ ions at an accelerating voltage of 30 kV at normal incidence to the sample surface. An ion beam current of 1.3 nA was used. A Quanta 450 SEM set at 2 kV acceleration voltages with a spot size of 2.5 was used to image the CNT-yarns. Zeiss Axio Observer was used to do the polarized light microscopy. A CNT 1-yarn was intentionally bent to simulate normal handling of the yarn to observe the resultant defects. NanoCT scans were done at Cornell University BRC CT Imaging Facility with 2 cm long as-received CNT 100-yarn. NanoCT uses X-rays to create cross-sections of density that can be assembled to recreate 3D structures at the nano scale.

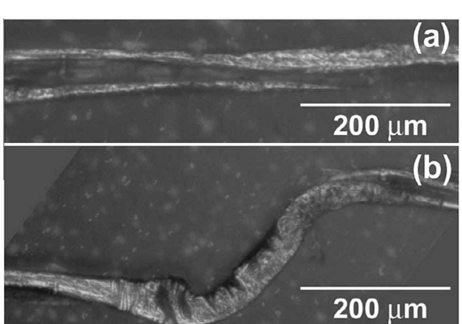

Fig. 2 – Defects in 1-yarn; (a) opening-up, and (b) buckling.

3. Results

3.1. *Single yarn*

CNT yarn can be formed by drawing CNTs off a substrate in the form of a ribbon and then twisting. Fig. 1a is a simplified schematic of this process. This process forms a long yarn of looped ribbons that take on the appearance of a solid material. Fig. 1b shows a SEM image of the yarn which appears as a solid material. However, utilizing polarized light microscopy, the looped ribbons within the yarn can be observed as shown in Fig. 1c. The arrows point out the edges of the ribbons, and confirm the accuracy of the schematic shown in Fig. 1a. As the yarn is made out of looped ribbons, it is possible for the yarn to untwist upon damage. Fig. 2 shows a few defects that can occur to the yarn: (a) opening-up, (b) buckling etc. These defects can cause stress concentrators that would significantly reduce the mechanical properties. Further, this can complicate the production of multi-yarn since the single yarns will be rubbed, pressed, and stretched against each other during the fabrication.

3.2. *Multi-yarn*

The as-received 100-yarn consists of CNT yarns that are twisted into strands. These strands have been further twisted