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In-situ characterization of microstructural changes in a carbon nanotube sheet under sustained load☆

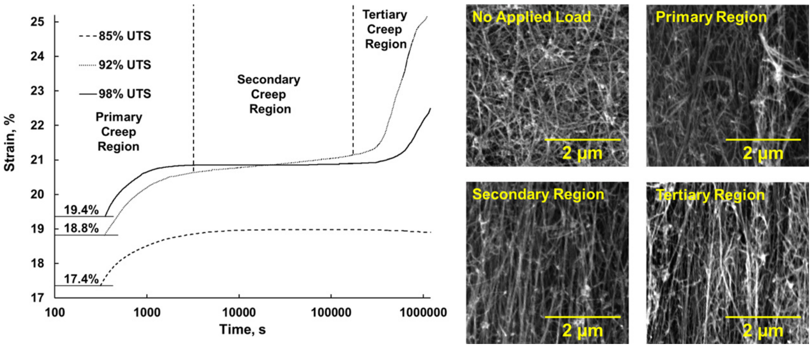
Gregory R. Cobb *, Abhendra K. Singh, Shankar Mall

Air Force Institute of Technology, Department of Aeronautics and Astronautics, 2950 Hobson Way, Wright-Patterson AFB, OH 45433-7765, USA

HIGHLIGHTS

- Carbon nanotube sheets were studied in-situ in a scanning electron microscope to characterize creep behavior.
- A computational pattern recognition program was created and used to characterize individual carbon nanotube alignment.
- The carbon nanotubes exhibited continuous alignment throughout the testing phase.
- The sheets did not fail after 1,000,000 s at high loads relative to the ultimate tensile strength.

GRAPHICAL ABSTRACT



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ABSTRACT

The present study characterizes the creep behavior of carbon nanotube (CNT) sheets under externally applied sustained loads. Creep loads at levels ranging from 85% to 98% of the ultimate tensile strength of the CNT sheet were applied using a tensile tester inside a scanning electron microscope chamber. The microscope enabled in-situ characterization of the microstructural changes in the CNT sheet under the influence of the applied load. The loads were sustained for 1,000,000 s or failure, whichever occurred first. A computational pattern recognition technique was also developed that enabled quantitative approximation of the time dependent changes in distribution of individual CNT orientation with reference to the loading direction. It was observed that the CNTs increasingly aligned along the loading direction during the initial loading phase when the CNT sheet was ramped up to the desired load level. Further microstructural changes by way of individual CNTs continuing to gradually align along the loading direction was observed after the loads were sustained at the desired levels. The pattern recognition computational results validated the experimental findings. Slight relaxation of the CNTs was also observed upon the removal of load once 1,000,000 s was reached.

1. Introduction

Carbon nanotubes (CNTs) are being readily considered as one of the leading materials for current and future applications due to their excellent mechanical, thermal, and electrical properties. These properties include lower density, higher stiffness and tensile strength, higher thermal and electrical conductivities, and a larger aspect ratio as compared to other materials [1–2]. These exceptional mechanical properties

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* Corresponding author.
E-mail address: gregory.cobb.ctr@afit.edu (G.R. Cobb).

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make them strong candidates to replace traditional materials from the perspective of strength and weight [3]. Their electrical properties also make them ideal candidates to improve electromechanical interference shielding for components used in the aeronautic or aerospace industries [4]. The fabrication of bulk CNTs in the form of a wire, tape or sheet has made it increasingly feasible to use the CNTs as a drop-in replacement for other conventional materials in actual applications. To this end, CNT sheets have already been successfully implemented in the manufacture of coaxial cables and antennas, as well as utilized in components such as tubes and sandwich panels in the Juno spacecraft [5–7]. The CNT sheets are likely to experience constant mechanical loads over prolonged periods of time during their actual usage. Therefore, a better understanding of the effects of long-term loading on these materials is very crucial to their efficient and cost-effective implementation and use.

While there are numerous studies on the mechanical properties of CNTs [8–10], a very limited number of studies have been performed in determining their durability when subjected to constant mechanical loads. Zhang et al. [11] observed that CNT yarns are resistant to creep and stress relaxation. They found that when a CNT yarn was held at a constant strain of 6% for 20 h, the stress of the yarn decreased (stress relaxation) by no more than 15%, occurring within the first 20 min in a viscoelastic fashion [11]. Xu et al. [12] found that bulk CNTs had similar creep and creep recovery properties to silicon rubbers, but they had superior thermal resistance when compared to silicon rubbers. They observed that the creep of the CNTs was very low ($<0.001\%\ \mathrm{min}^{-1}$) at 400 °C, and they determined computationally that the strain recovery process was driven by non-aligned and unbundled CNTs [12]. Misak et al. [13] reported that the amount of fibers in the CNT yarn inversely affected the creep rate. The single fiber yarn displayed a much lower steady-state creep rate than a yarn with 100 fibers [13]. Zhang et al. [14] studied the amount of viscous creep displayed by CNTs vertically-aligned in the out-of-plane direction. Their study shows that the CNTs strain rate sensitivity depends on the density of the CNT bundles, with more dense bundles resulting in lower creep rates [14]. A number of studies exist on the creep behavior of composite materials infused with CNTs [15–17]. However, the CNTs are simply introduced in the matrix of the composite materials as nanofillers and in this form fail to display their full structural potential. The authors are not aware of any previous studies that exclusively looked at the creep-rupture behavior of thin CNT sheets under long-term constant mechanical load. Further understanding on the effects of creep loading on the mechanical behavior of CNT sheets is crucial for applications with prolonged externally applied loads.

In the present study, CNT sheets are loaded in tension to a certain percentage of their ultimate tensile strength (UTS) and the loads are sustained while the microstructural changes in the CNT sheet are observed in-situ using a scanning electron microscope (SEM). The SEM images of the CNT sheets are processed using a pattern recognition code developed in-house, which enabled quantitative approximation of the time dependent distribution of the individual CNT orientation with reference to the loading direction. The computational results validated the experimental findings from the SEM images. This study provides useful insights into the microstructural behavior of the CNTs in a CNT sheet under sustained loads which will be very useful in design decisions for future structural applications of these CNT sheets.

2. Material and test methodology

2.1. Material

The CNT sheet used in this study were acquired from Nanocomp Technologies, Inc., based in New Hampshire, USA. The sheet is “untreated” (without acid treatment) and consists of multitudes of non-woven multi-walled CNTs that agglomerate together without the use of any matrix or binder. The CNTs have diameters that are on the 10 nm scale and can have average lengths up to the millimeter scale as specified on the manufacturer's website [18]. While the exact process used to create these sheets is proprietary, the company has stated that their CNT sheets are created via chemical vapor deposition using an iron catalyst, are acetone condensed and contain no bonding agents. In appearance, the CNT sheet looks like a thin membrane and has a nominal thickness and areal density of 40 μm and 10–15 g/m^2, respectively, as specified by the manufacturer. The thickness was verified using a Rex Material Thickness Gauge (Model MTG-DX2). The test coupons retrieved from the CNT sheet for the mechanical tests had a width of 5.60 ± 0.05 mm and an overall length of 50 mm. The specimen gage length for the mechanical tests however was 30 mm.

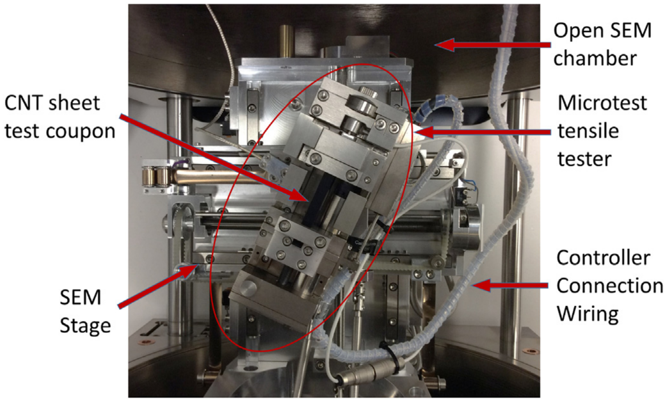

Fig. 1. Experimental test set-up showing a sample CNT sheet ready for testing using a tensile tester inside an SEM chamber. The tensile tester is circled in red. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

2.2. Experimental method

A Deben Microtest tensile tester with a 200 N load cell was used for the mechanical testing of the CNT sheets. The edge grips of the tensile tester were made of smooth finished stainless steel and were held by two screws on each side, which when tightened gripped the specimen in place. The specimen made direct contact with the edge grips without the need of any external adhesive to keep it in place. All tests were done at room temperature. Two monotonic tensile tests were first performed on the test coupons in order to determine the UTS of the CNT sheets. Three additional test coupons were then loaded to 85%, 92% and 98% of the UTS and the creep loads at these levels were sustained for 1,000,000 s. All the loadings were done at a ramp rate of 1 $\mathrm{mm\ min}^{-1}$ until the desired load levels were reached. Both the monotonic tensile and creep tests were performed inside a Quanta 450 SEM which was used to take SEM images in-situ at different times. An in-built port inside the SEM chamber enabled the Microtest tensile tester to be

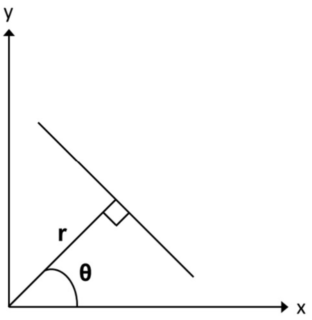

Fig. 2. Schematic of the relationship between Cartesian coordinates and Hough space coordinates. The equation of the line perpendicular to r can be determined if r and θ are known using Eq. (1).