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### Detailed investigation on elastoplastic deformation and failure of carbon nanotube fibers by monotonic and cyclic tensile experiments



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#### ABSTRACT

Monotonic and cyclic tensile loading experiments were conducted for analyzing elastoplastic deformation and failure of carbon nanotube (CNT) fibers. The deformation stages of the CNT fibers under tensile loads consist of a short elastic stage and a long-lasting plastic stage, while the elastic stage of the CNT fibers can be generally divided into two parts: a perfect-elastic stage and an elastic-like stage. Microstructural evolution for each deformation stage was interpreted by means of scanning electron microscope photos. An elastic limit and an offset yield point were discovered through the cyclic loading experiments. The elastic strain limit of the CNT fibers was determined at about 0.35% and the offset yield strain at about 1.2% for quasi-static loading. The effect of strain rates on the deformation and failure mode of CNT fibers was investigated as well.

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#### 1. Introduction

Carbon nanotubes (CNTs) have been widely investigated since their discovery in 1991 due to their excellent mechanical and electrical properties. For the further industrial applications, CNTs will be assembled into varieties of macrostructures, such as CNT films [1], CNT fibers [2], and CNT blocks [3]. Among those macrostructures, CNT fibers are highly-expected as a new kind of high-performance fiber after glass fiber [4,5] and carbon fiber [6,7]. However, in contrast to glass and carbon fibers, the mechanisms of elastoplastic deformation and fracture of the CNT fibers are very difficult to be clarified because of their complicated microstructures.

It has been demonstrated that macroscopic mechanical properties of the CNT fibers are strongly dependent on their fabrication methods, including wet spinning method [8,9] (strength is about 0.15 GPa and elongation is about 2%), dry spinning method [10,11] (strength is about 0.46 GPa and elongation is about 10%) and direct spinning method [12,13] (strength is about 1.46–9 GPa and elongation is about 5–10%). However, experimental results have shown that most of these CNT fibers have not reached their highest properties, as expected in [14]. Since the CNT fibers consist of millions of separated CNTs and their self-assembled bundles, the

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orientation distributions of them can considerably influence the mechanical properties of the CNT fibers. Previous experimental results by Li et al. [15,16] indicated that tensile stress-strain curves of the CNT fibers include three stages: elastic stage, strengthen stage and damage-fracture stage. They also pointed out that the self-assembled CNT bundles in the CNT fibers are the main load-carrying parts, and their slipping can lead to plastic deformation of the CNTs fibers. Zhang et al. [17] observed failure modes of the CNT fibers at different strain rates by means of scanning electron microscope (SEM) micrographs. They found that slipping of CNTs is the major failure mode at low strain rates, while breakage of CNTs due to their imperfect alignment is the major failure mode at high strain rates. On the basis of above observations, CNT fibers with high strength should be assembly of aligned thin and long CNTs [18]. Liu et al. [19] applied MD simulation to indicate that plastic deformation of the CNT fiber generates after short elastic-like deformation under tensile loading, which depends on the applied strain rate. Moreover, the surface angle of the CNT fibers is also one of the main influence factors of the fiber strength. Sugimoto et al. [20] indicated that the tensile modulus and Poisson's ratio of the CNT fibers decrease with the increase of the twist angle. However, the maximum tensile strength of the CNT fiber appears as the twist angle of the fiber is 25°. Fang et al. [21] showed that a peak tensile strength of 340 MPa is achievable with 20° helix angle for a 20 μm-diameter CNT fiber.

Although CNTs are now a well-established area of interest, there is a dearth of knowledge relating to CNT-reinforcement of

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how they may be used to enhance composite materials. Experimentally, there is little study on the CNT-reinforcement in composite development, and limited work has been documented relating to how CNT fibers may be of benefit to composite structures. Hence, this work will address this issue by studying the elastoplastic deformation and failure of CNT fibers using monotonic and cyclic tensile experiments. In this study, monotonic and cyclic loading experiments were performed at several strain rates to analyze detailed deformation and failure process of the CNT fibers. The elastoplastic deformation and fracture mechanisms of the CNT fibers were investigated at multi-scale levels. The effects of the strain rates of monotonic and cyclic loadings on the mechanical properties of the CNT fibers were examined as well, which shows potential applications of CNT fibers.

#### 2. Experimental method

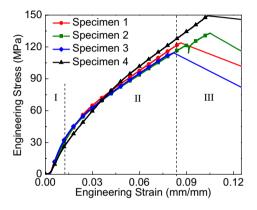
The CNT fibers adopted in this study were produced by Jiangsu Jiedi Tech Co. Ltd. The mechanical properties of the CNT fibers are significantly different based on their fabrication methods, and only the CNT fibers produced by direct spinning method would be studied here. The multi-walled carbon nanotubes (MWNTs) in the CNT fibers have a diameter of 6–12 nm, a strength of 270–310 MPa, a modulus of 4–6 GPa and a elongation of 15–25%. The SEM image of an entangled structure of the CNT fiber is shown in Fig. 1(a), while a zoom-in SEM image of a loose structure of the CNT fiber is shown in Fig. 1(b), where CNT bundles (diameter is about 100–550 nm) and CNT threads (diameter is about 6–50 nm) can be clearly identified.

Tensile experiments of the CNT fibers were performed by using a nano-tensile tester (Agilent UTM T150) which has a maximum force of 450 mN. The specimens with a length of 10 mm were prepared by fixing the fiber segment on a paper holder with instant epoxy adhesive. An initial force of 750 μN was applied on the specimens to ensure that the fibers maintain straight before the tensile tests. The specimens were tested at stretch rates of  $2 \times 10^{-4}$  mm/s,  $2 \times 10^{-3}$  mm/s, and  $2 \times 10^{-2}$  mm/s, which correspond to strain rates of  $2 \times 10^{-5}$ /s,  $2 \times 10^{-4}$ /s and  $2 \times 10^{-3}$ /s, respectively. The diameter of each specimen is 55 µm measured by using an optical microscope. The experimental data was collected by the testing system automatically. Thus, the stress can be calculated by tensile load divided by the cross-sectional area. In the cyclic tensile tests, the applied maximum strains were increased by a constant value, for example, 0.5% or 1%, for each reloading step in the present experiments. For the SEM images used in Fig. 4, the CNT fibers were first stretched to a certain tensile strain and then were taken out from the tensile tester. Next, the CNT fibers were fixed on the test bed of the SEM equipment. Finally, the SEM images were obtained as shown in the manuscript. And all SEM images present the microstructure of the CNT fiber which has been relaxed for 24 h.

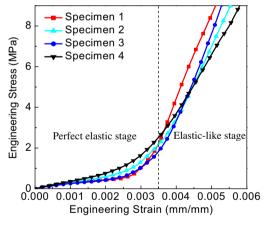
#### 3. Monotonic loading experiments

3.1. Mechanisms of elastoplastic deformation and fracture of CNT fibers

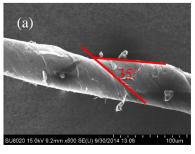
Fig. 2 reveals typical tensile stress–strain relation of the CNT fiber under monotonic loading at a low strain rate  $2\times 10^{-5}/s$  which can be viewed as a quasi-static loading. The consistency of the curves from four repeated tests ensures that a stable mechanical performance of the CNT fiber can be obtained. As displayed in Fig. 2, the stress–strain curves can be divided into three stages, namely elastic deformation stage (stage I), plastic deformation

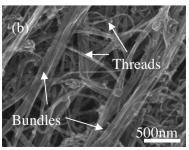


**Fig. 2.** Tensile stress–strain curves of CNT fibers. (A color version of this figure can be viewed online.)



**Fig. 3.** Stage I of stress-strain curves of CNT fibers. (A color version of this figure can be viewed online.)





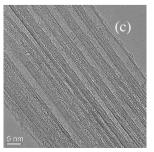


Fig. 1. SEM photos of CNT fiber, (a) macroscopic, (b) microscopic morphology and (c) MWNTs. (A color version of this figure can be viewed online.)

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