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Dynamic mechanical characterization for nonlinear behavior of single carbon fibers

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In the work presented here, small amplitude in-situ dynamic loads are superimposed onto quasi-static tensile tests, to obtain storage and loss modulus as a function of global strain for deforming single PAN carbon fibers. This technique allows for the measurement of carbon fiber modulus at unprecedented resolution without the need for compliance correction, which is difficult to implement as carbon fibers have a non-linear modulus dependency with strain. The relationship of the elastic modulus with strain is quantified to evaluate the single fiber nonlinearity during extension. Fibers with initially higher modulus are shown to have greater nonlinear dependency on strain amplitude, even for fibers taken from the identical tow. The strong correlation between tensile modulus with strain and its initial modulus indicates that the measured mechanical properties describe a fundamental structure of carbon fibers.

1. Introduction

The unique mechanical behavior of high performance carbon fibers is their nonlinear, elastic stress/strain relationship, whereby the ratio of axial stress to strain (Young's modulus) increases with increasing stress/strain amplitude [1–8]. The phenomenon has been observed for multiple specimen configurations including unidirectional resin-impregnated fiber tow, unidirectional composite coupon fiber mat layup, and single filament tensile test. It is widely accepted that the nonlinearity results from a recoverable reorientation of carbon planes (alignment of carbon planes to the fiber axis) with applied axial stress or strain prior to reaching failure stress, within the "elastic" regime [1,5,9]. Due to the highly anisotropic nature of the graphitic composition, the apparent modulus increases as these structural units reorient. Hence, the initial modulus and the change in modulus with applied stress/strain are directly related to material micro-structure, depending in part on the orientation of carbon planes with the fiber axis. Experimental data indicate that the tensile modulus increases with axial strain according to the following relationship:

$$E(\varepsilon) = E_0[1 + \gamma\varepsilon] \tag{1}$$

where E is the tangential modulus, E_0 is the initial tangential modulus, and γ is the coefficient describing the modulus dependency on strain, ε, for a given fiber type [4]. Accurately measuring carbon fiber modulus and the modulus dependency with strain are fundamental to understanding the mechanical behavior of high-performance carbon composites, specifically in the critical areas of interface load transfer mechanism and the development of high performance fibers [10]. Measurement of the empirical parameter γ depends greatly on the experimental approach used to evaluate tensile stress–strain behavior (single fiber or tow based testing). Thus, a wide variation in γ has been reported from past literature for many fibers, and in some cases, little, if any, nonlinearity was observed [11]. This indicates the mechanical response reported in the majority of the published literature is dependent on the testing protocol rather than the material microstructure. In the case of obtaining stress–strain behavior of carbon fibers by a resin impregnated tow of fibers (bundle), varied amounts of inter-fiber effects, e.g. fiber alignment, length distribution, and interactions, can dominate the measured stress–strain relationship, inhibiting the ability to infer true stress–strain behavior of single carbon fibers as a result of carbon plane realignment during axial extension. Fig. 1 compares the stress–strain behavior and modulus from the authors 25 mm T700 single carbon fiber tensile test with a 100 mm 12 k T700 impregnated carbon fiber tow from Zhou et al. [11]. Clearly these tests, although on the same material, demonstrate obvious differences in mechanical behavior and hence, variation of coefficients from Eq. (1). Although easier to prepare and apply on-specimen techniques for strain, resin-impregnated tows consistently underestimate axial moduli and its dependency on strain due to the inter-fiber effects. For example, Hughes has measured the dimensionless, nonlinear coefficient, γ, of Toray T300 fibers to be 15 by tow

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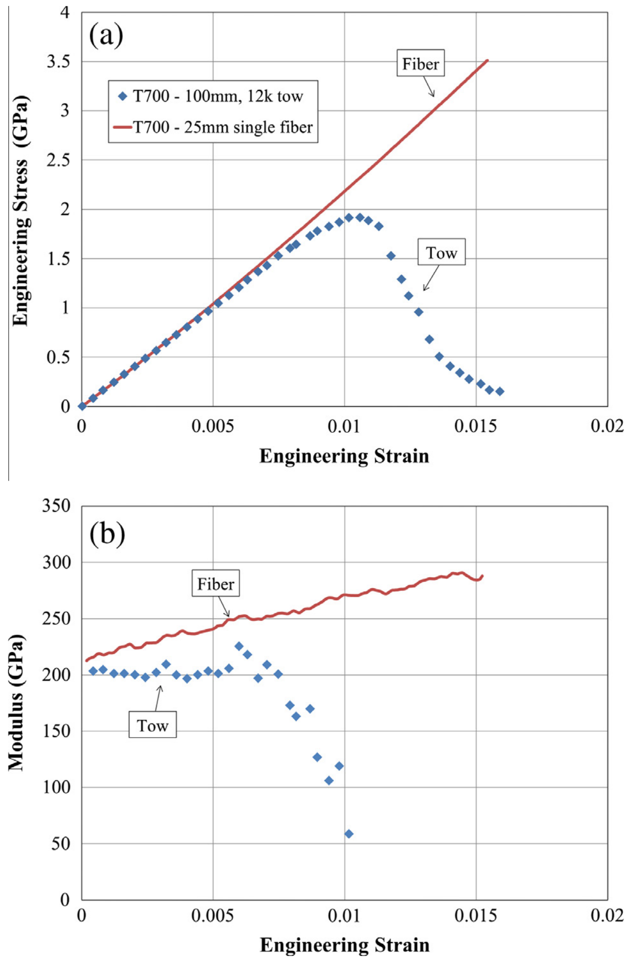

Fig. 1. Comparison of (a) stress/strain behavior and (b) modulus of T700 carbon fibers obtained by single fiber and fiber tow tensile test, demonstrating that tow based tensile testing does not replicate the mechanical response of a single fiber [11]. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

method, while Djordjević et al. reported γ to be 22.65 based on single fiber tensile test [4,7]. Furthermore, it is likely for tow based testing that the measured modulus does not involve the entire volume fraction of reinforcing constituents. R'mili tested 6 k T300 tows, which when compared with single fiber moduli were estimated to consistently engage only 4000 fibers, suggesting the rest were broken during sample preparation and hence, did not contribute to the load bearing capacity of the tow during extension [12].

A significant advantage of the resin impregnated tow test is the ability to apply on specimen strain techniques to avoid grip compliance issues associated with engineering strain. An extensometer is typically used for on-specimen strain, but rigidly attaching this device results in stress concentrations at the attachment locations and potential premature failure. Optical strain techniques are possible, although challenging due to the initial and changing surface topography when surface tracking is required. Thus, it is not uncommon in industry for separate samples to be created for obtaining failure stress and modulus, leaving the relationship between tow failure and modulus unknown.

To obtain mechanical properties of fibers, the single-fiber tensile test is preferred [4]. However, accurately measuring strain and initial fiber diameter along the gage region presents a significant challenge, in addition to suitably gripping a single fiber. Commonly, single-fiber specimens are glued to either paper or plastic templates to reduce stress concentrations at the fiber ends during tensile testing as per ASTM and ISO standards [13,14]. The additional material—glue, template and embedded fiber—introduces compliance into the load path that must be subtracted from the total displacement measurement. Current single fiber tensile testing standards apply a single compliance correction to the entire strain domain, independent of sample loading. However, due to progressive interface delamination within the grip region, system compliance will increase during a tensile test, decreasing the measured modulus.

To overcome the challenges and limitations described above for obtaining stress–strain behavior of carbon fibers, a novel in-situ dynamic method for single fiber tensile testing is implemented in this study to determine axial mechanical properties with exceptional measurement resolution in axial load (~nN) and displacement (~nm). Most importantly, sample stiffness is determined independent of total specimen displacement or engineering strain, making the measurement of moduli unaffected by system compliance [2]. Herein, nonlinear, elastic properties for different types of Toray PAN carbon fibers are presented using this dynamic approach. Due to the nature of nano-tensile testing precision, it is hypothesized a significant increase in the modulus dependency with strain will be observed compared to what was reported from conventional single fiber and tow based tensile testing techniques in the past published literature. The objective of this study is to evaluate the 'γ' parameter accurately for a given fiber to relate carbon fiber synthesis techniques to mechanical properties in a unified formulation.

2. Experimental approach

2.1. Single-fiber tensile test

Individual polyacrylonitrile (PAN) carbon fibers were mounted on tabs for tensile testing following techniques described previously [13–15]. Six types of Toray PAN carbon fibers were used in this study selected to represent low, intermediate, and high modulus with designations T300, T400, T700, T800, M40J, and M50J. The fiber cross-sections were assumed round, and the diameters were acquired from manufacturer's specifications. Diameter measurements obtained by scanning electron microscope on 30, T700 fibers demonstrate the average diameter of samples tested here, 6.95 ± 0.29 μm, are in close agreement with that of the manufacturer's specification of 7.0 μm.

Tensile tests on single fibers were performed using the MTS Nano Bionix UTM® Universal Testing System (Fig. 2). The load sensing apparatus of the Nano UTM is the nano-mechanical actuating transducer (NMAT). The NMAT column has three main components; an electromagnet, a capacitance gage, and supporting leaf springs, as shown schematically in Fig. 2. A deforming sample is attached to the NMAT at one end and is extended at the other by an optically encoded stepper motor with 35 nm resolution in displacement positions. The capacitance gage measures the vertical displacement of the NMAT column, while the electromagnet applies load, either tension or compression. The testing system uses a PID feedback loop to obtain the load on the sample by applying the necessary force to maintain a zero displacement condition in the NMAT's capacitance gage when the sample is extended in tension via the extension axis at the top. Hence, deformation is applied through the extension axis at the top of the fiber, while the control system applies load through the electro-magnet at the bottom of the fiber, necessary to keep the NMAT in a fixed position. Maximum load is limited by the current allowed to the electromagnetic coil, approximately ±500 mN. When the NMAT shaft is not engaged with a sample, an upward, vertical force of approximately 250 mN is needed to lift the weight of the column using