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Effect of tensile offset angles on micro/nanoscale tensile testing

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For one-dimensional (1D) structures such as tubes, wires, and beams, tensile testing is a simple and reliable methodology for measuring their mechanical properties. The tensile offset angle effect on mechanical property measurement has long been ignored. In this study, theoretical and finite-element analysis (FEA) models for analyzing the tensile offset angle effect have been established. It is found that longitudinal stress decreases with increasing offset angles. The theoretically calculated elastic modulus relative errors reach 4.45% at the offset angle of 10°, whereas the experimentally measured elastic modulus relative errors are 45.4% at the offset angle of 15°. The difference in elastic modulus relative errors between the theoretical analysis and the experimental results is discussed with reference to the sensing system in the experimental instrumentation. To accurately measure the mechanical properties using the tensile testing technique, perfect alignment with a zero or small offset angle less than 5° is needed. A calibration methodology for aligning specimens has been developed. © *2005 American Institute of Physics.* [DOI: 10.1063/1.1865732]

I. INTRODUCTION

Recent developments in science and engineering have advanced our capability to fabricate and control materials/structures on the scale of micro/nanometers, and have brought problems of material behavior on the micro/nanometer scale into the domain of science and engineering.[1–5] Studies have revealed that material properties are size dependent. For example, the bending strength of silicon beams shows a clear specimen size dependence, with nanoscale numbers being twice as large as numbers reported for large-scale specimens.[4] Material properties of micro/nanostructures cannot necessarily be predicted via extrapolation from existing theories used for larger structures. A precise characterization of the mechanical properties of micro/nanostructures is required to use them as structural elements in devices.[1,4]

The small dimensions of micro/nanostructures impose a tremendous challenge for the experimental study of their mechanical properties. For one-dimensional (1D) structures such as tubes, wires, and beams, tensile testing is a simple and reliable methodology for measuring their mechanical properties, but seems rather difficult to implement at the micro/nanoscale. There have been few experimental reports on tensile testing of 1D micro/nanostructures.[6–10] An example is the study of mechanical properties of carbon nanotubes with a nanotensile stage operated within a scanning electron microscope (SEM). To date, however, no agreement has been reached among these publications regarding the mechanical properties of carbon nanotubes; in particular, the elastic modulus. Although various micro/nanoscale tensile testing instruments have been developed, micro/nanomechanics theories are still left behind. In nanotensile testing with the nanotensile stage, as shown in Fig. 1, the top stiff cantilever was driven upward while the bottom flexible cantilever with a known stiffness was bent upward by a distance l_s. The nanotube was stretched from its initial length of l_0 to $l_0+l_h-l_s$, as shown in Fig. 1(a).[6] However, the most critical problem, which has been ignored, is the tensile offset angle effect. When the top stiff cantilever was driven upward, it did not shift instantaneously to the right to keep the nanotube well aligned with zero tensile offset angle, as shown in Fig. 1(b). In fact, the nanotensile tester was operated such that the tensile offset angle increased with an increase in tensile load. The actual elongation of the nanotube [Fig. 1(b)] would be $\Delta l=[(l_0+l_h+l_s)^2+(L_s-\sqrt{L_s^2-l_s^2})^2]^{1/2}-l_0$, rather than $\Delta l=l_h-l_s$, where L_s is the effective length of the lower cantilever.

The second concern regarding the tensile offset angle effect comes from the SEM imaging system. The nanotensile stage can be manipulated in real time such that the nanotube can be well aligned in the tensile loading direction from the front view of the SEM image; the side view, however, is behind the scene, where there is no way to know whether the nanotube is aligned to the tensile loading direction. An even worse situation is that the sample is well aligned from the front view, but in fact, remains slack with an offset angle in the side view, as shown in Fig. 2. This could happen easily in real micro/nanoscale tensile testing.

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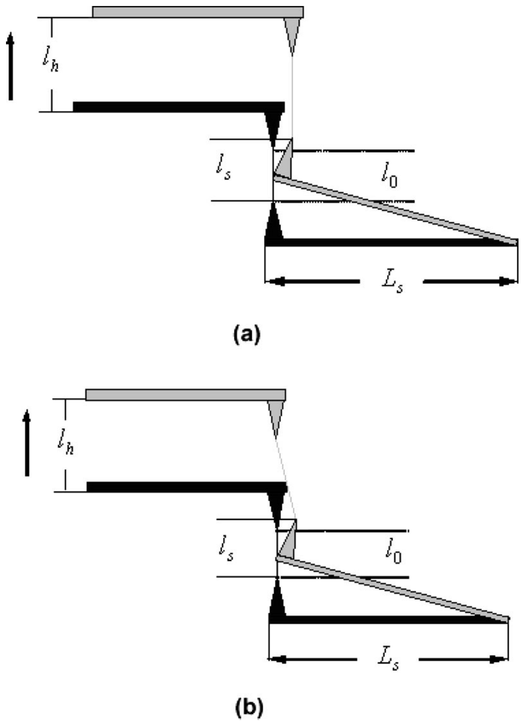

FIG. 1. Schematics of a cantilever-based nanotensile stage. (a) The upper cantilever shifts instantaneously to the right to keep the nanotube well aligned with zero tensile offset angle during tensile testing. (b) The upper cantilever moves vertically without instantaneously shifting to the right, inducing a varying offset angle during tensile testing.

Hence, the critical issue that needs to be addressed is how much the offset angle can affect the mechanical property measurement in micro/nanoscale tensile testing. This article presents the effect of tensile offset angles on the stress, strain, and elastic modulus in micro/nanoscale tensile testing through mechanics theory, finite-element analysis (FEA), and micro/nanoscale tensile experiments performed on polypropylene microfibers using a nanoscale tensile tester. A calibration technique for aligning specimen has been developed.

II. EXPERIMENT

Micro/nanoscale tensile tests were performed using a nanoscale tensile tester—the MTS Nano Bionix® testing system (Nano Innovation Center, MTS Corp., TN). This system is capable of applying up to 500 mN tensile load while maintaining its load resolution of 50 nN. The system accommodates elongations up to 150 mm with a 35 nm displacement resolution. The longitudinal strain or extension is measured automatically by the extensometer. The engineering stress-strain curves were obtained from various alignment offset angle tests.

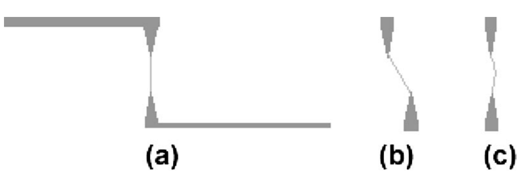

FIG. 2. (a) The specimen is well aligned from the front view. (b) The sepcimen is straight, with an offset angle from the side view. (c) The specimen remains slack with an offset angle from the side view.

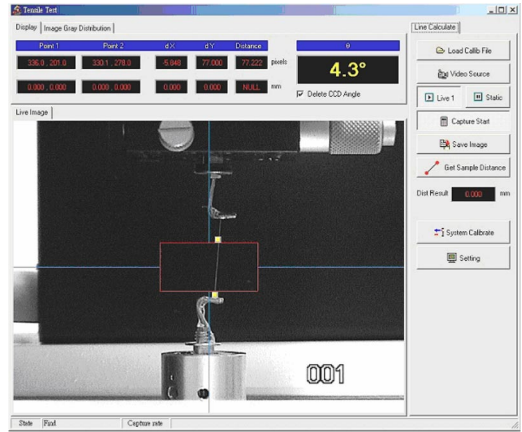

FIG. 3. (Color online) Digital imaging system used to calibrate the specimen alignment from both front view and side view. The real-time gage length and offset angle can be measured and recorded through the whole process of tensile testing.

Two CCD imaging systems were used to calibrate the sample alignment from both front view and side view. The real-time gage length and offset angle were measured and recorded through the whole process of tensile testing, as shown in Fig. 3.

Polypropylene fibers with a diameter of 110 μm were used for tensile testing. The fiber diameter was measured using SEM before tensile testing, as shown in Fig. 4. For each test with a different offset angle, the starting distance between the upper and lower hooks is set to be 14.00 mm, and the distance that the upper hook travels is 0.14 mm, which ensures the tests to be in the elastic region of the polypropylene fiber. The elastic modulus was calculated based on this initial small portion to avoid a gradual stiffening tendency. An appropriate amount of Zap-A-Gap® glue was put on the lower hook, and one end of the fiber was bonded on it. The other end of the fiber was stretched straight gently and held onto the upper hook to a degree that was close to the desired offset angle from the front view (moni-

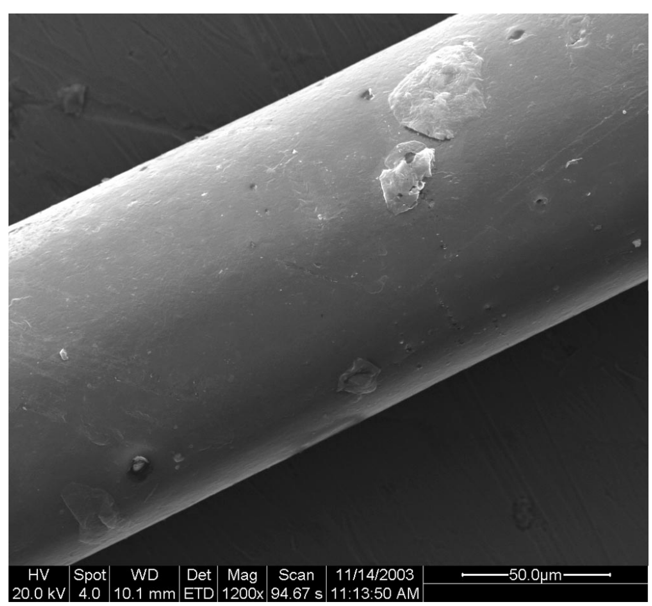

FIG. 4. SEM image of the polypropylene fiber used in this study.

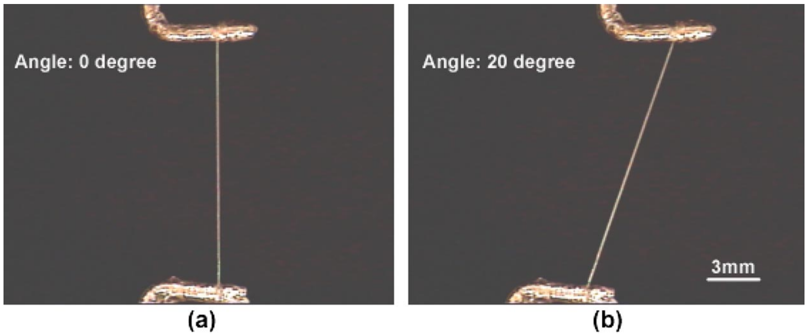

FIG. 5. (Color online) Alignment of the polypropylene fiber at an offset angle ranging from 0° to 20° before tension.

tored by CCD001), and to a degree close to zero from the side view (monitored by CCD002). The position of the upper hook could be adjusted by adjustment stage such that the fiber had no offset angle from the side view but the exact expected offset angle from the front view before the test. The fiber was then wound on the upper hook and glued. By this means, the fiber could be aligned straight without being prestressed before the test. Figures 5(a) and 5(b) show the polypropylene fiber aligned at an offset angle ranging from 0° to 20° before tension.

III. RESULTS AND DISCUSSION

For an ideal tensile test, a specimen is under pure tension and can be treated as a "two-force element," as shown in Fig. 6.

In the present study, a polypropylene fiber was fixed onto the upper and lower hooks; thereby, the fiber had a bending moment. Figure 7(a) shows the schematic diagram of an applied force on the polypropylene fiber with an initial offset angle of θ. It can be seen that the resultant force F_p given by the upper hook can be decomposed into two perpendicular components: F_h and F_v. The relation between F_v and F_p is described as follows:

$$F_v = F_p \cos\theta. \tag{1}$$

Figure 7(b) shows the difference in extension between the tensile tests with zero offset angle and θ initial offset angle. In the case of zero offset angle, l_v is the vertical length and Δl_v is the vertical displacement. For the θ initial offset angle, the actual displacement is given by

$$\Delta l_a = \sqrt{(\Delta l_v + l_v)^2 + (l_v \tan\theta)^2} - l_a, \tag{2}$$

where the actual original length $l_a = l_v \sec\theta$.

The stress induced by F_p is given by

$$\sigma_p = E\Delta l_a / l_a = E[\sqrt{(\varepsilon_v^2 + 2\varepsilon_v)\cos^2\theta + 1} - 1], \tag{3}$$

where $\varepsilon_v = \Delta l_v / l_v$ is the engineering strain when the offset angle is zero. α is a change in offset angle during tensile loading. α can be derived as

$$\alpha = \cos^{-1}\left(\frac{(l_a + \Delta l_a)^2 + l_a^2 - \Delta l_v^2}{2(l_a + \Delta l_a) l_a}\right). \tag{4}$$

Therefore, the relation between the stresses induced by F_v and by F_p can be described as follows:

$$\sigma_v = \sigma_p \cos(\theta - \alpha). \tag{5}$$

In the FEA, the bending effect was taken into account. Figure 8 shows the stress distribution along a polypropylene fiber with various offset angles. A decrease in von Mises stress is observed when the offset angle is increased. The bending effect is observed near the ends of the fiber. As the offset angle increases, the stress near the ends decreases due to the bending effect. For high aspect ratio (length/diameter) specimens, the bending effect can be ignored.

It should be noted that the nanotensile tester measures F_v rather than F_p. In addition, the nanotensile tester measures l_v instead of l_a, resulting in additional errors in the tensile testing. Figure 9 shows the engineering stresses as a function of

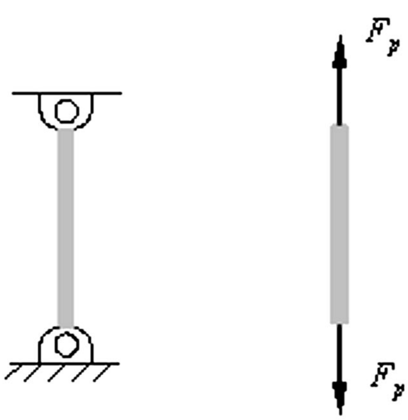

FIG. 6. Schematic of an ideal tensile test in which a specimen is under pure tension.

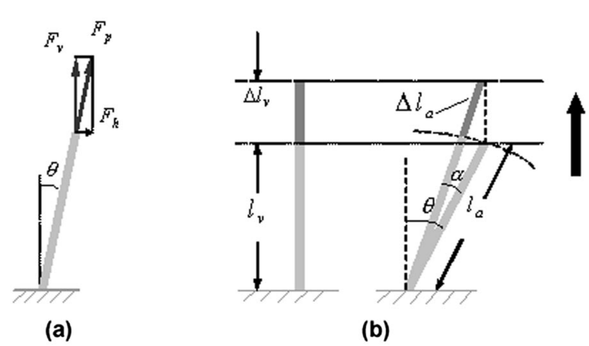

FIG. 7. Schematics of (a) an applied force on the polypropylene fiber with an offset angle of θ and (b) difference in extension between the tensile tests with zero offset angle and θ offset angle.

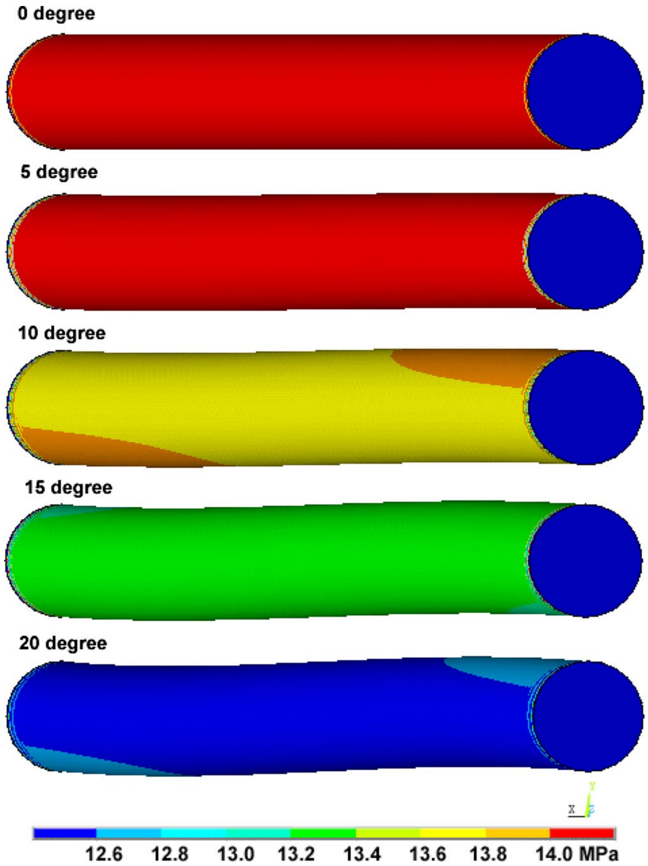

FIG. 8. (Color online) Pulling stress distribution along a polypropylene fiber with various offset angles.

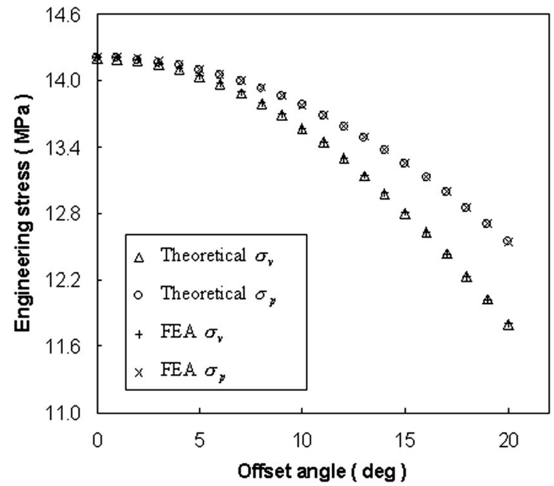

FIG. 9. Engineering stress as a function of offset angle obtained from both theoretical and FEA analyses.

offset angle obtained from both theoretical and FEA analyses. It can be seen that the theoretical σ_p and σ_v are in good agreement with the FEA results. This indicates that for a long fiber specimen with high aspect ratio, the bending effect at the fixed ends on the tensile testing can be ignored. It can be seen from Fig. 9 that with increasing offset angle, the deviation of σ_v and σ_p increases.

From the foregoing theoretical and FEA analyses, the theoretical error in calculating elastic modulus of the sample includes (1) error of using σ_v rather than σ_p and (2) error of using ε_v rather than ε_a, which is the actual engineering strain. Figure 10(a) shows the theoretically calculated engineering stress-strain curves with various offset angles. Figure 10(b) shows the engineering stress-strain curves of polypropylene fibers with various offset angles obtained using the MTS Nano Bionix® testing system. Comparison of Figs. 10(a) and 10(b) shows that below 10°, the experimental stress-strain curves coincide with the theoretical stress-strain curves. At and above 10°, the experimental stress-strain curves are far below the theoretical ones, showing a dramatic drop of slope.

Based on σ_v and ε_v, the theoretically calculated elastic modulus as a function of offset angle is normalized and presented in Fig. 11. It can be seen that the elastic modulus decreases with increasing offset angle.

Figure 12 summarizes the elastic modulus relative errors with various offset angles. The theoretical errors are comparable to the experimental results with a small range of offset angles. The experimentally measured elastic modulus exhibits approximately 3.63 times bigger errors than the theoretical values when the offset angle reaches 15°. This is because at a large offset angle, the center plate of the capacitive displacement sensor in the nanotensile tester is tilted, resulting in a lateral force and an inaccurate measurement of force and displacement.

From the theoretical, FEA, and experimental analyses presented, we can see that tensile offset angle is an important

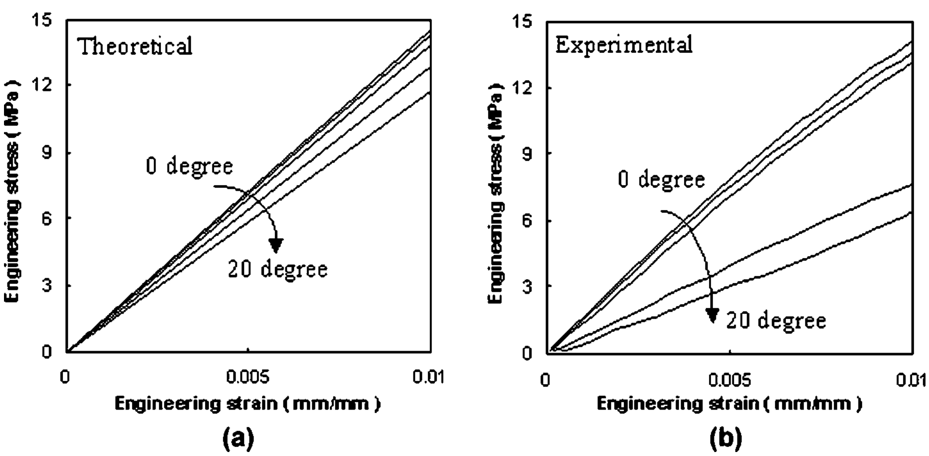

FIG. 10. (a) Theoretically calculated engineering stress-strain curves with various offset angles. (b) Engineering stress-strain curves of the polypropylene fiber with various offset angles obtained using the MTS Nano Bionix testing system.

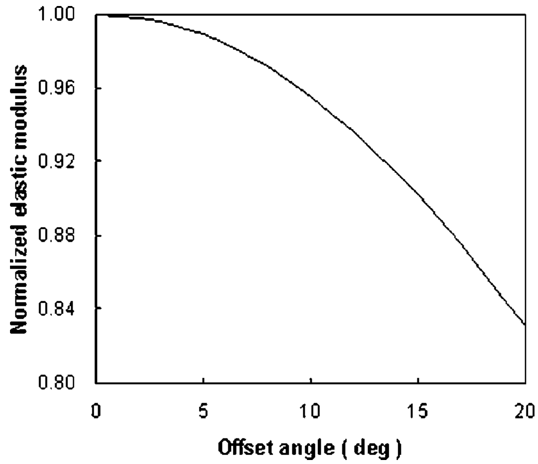

FIG. 11. Based on σ_v and ε_v, normalized theoretical elastic modulus as a function of offset angle.

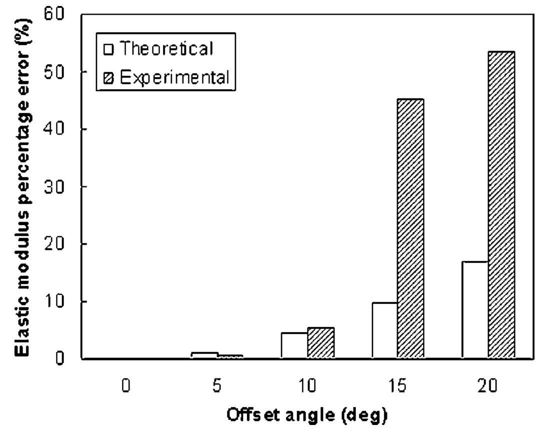

FIG. 12. Bar charts summarizing the elastic modulus relative errors with various offset angles.

issue that causes errors in micro/nanoscale tensile testing. Aligning the specimen with zero or a small offset angle is needed. A digital tensile specimen alignment system has been developed that can be integrated into the current micro/nanoscale tensile tester to enhance the measurement accuracy.

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