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Surface effects on elastic properties of silver nanowires: Contact atomic-force microscopy

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Silver nanowires with different diameters were synthesized by a hydrothermal chemical method. The elastic properties of the nanowires with outer diameters ranging from 20 to 140 nm were measured using contact atomic force microscopy. The apparent Young modulus of the nanowires is found to decrease with the increase of the diameter. When the diameter of the silver nanowires is larger than 100 nm, the Young modulus approaches a constant value. The size dependence of the apparent Young modulus of the silver nanowires is attributed to the surface effect, which includes the effects of the surface stress, the oxidation layer, and the surface roughness. Thus, a theoretical analysis is presented to explain the size dependence. This analysis is different from the previous models in that both the surface stress and the surface moduli are included in it. We also show that the apparent surface modulus and the surface stress of the silver nanowires can be experimentally determined.

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I. INTRODUCTION

The study of the variation of the properties of materials with their geometrical feature size has a long history because of its importance in many fields, e.g., in physics and chemistry, solid state physics, and materials science. The interest has been heightened recently at the nanoscale because nanostructures are pervasive in nature[1,2] and in modern industry,[3–8] and the large ratio of surface atoms to the bulk can have a profound effect on their properties.[3,4,9–11] Recently, scanning probe microscopy (SPM) was proved to be a powerful means in manipulating and characterizing the properties of nanostructures. For example, contact atomic force microscopy (C-AFM) was employed to study the mechanical properties of the one-dimensional (1D) nanomaterials.[12–16] The strength and stiffness of SiO_2 nanowires, ZnO nanobelts, polypyrroles, and MoS_2 nanotubes have been widely studied and measured in experiments.[17–26] These experiments showed that the elastic moduli of these nanostructures varied with the diameter of the nanowires or the thickness of the plates.

The mechanical behavior of materials at the nanoscale is different from that at the macroscopic scale due to the increasing ratio of the surface to the volume. For the nanostructural materials with a large ratio of the surface area to the bulk, the surface effect can be substantial.[27] Recently, many attempts have been made to reveal the influence of surface properties on the elastic properties of nanobeams, nanowires, nanoplates, etc. The works of Miller and Shenoy,[3] Cuenot *et al.*,[19] Zhou and Huang,[9] and Duan *et al.*[28,29] showed that the elastic moduli of homogeneous and heterogeneous materials vary with their characteristic size due to the surface effect. Moreover, Zhang *et al.*[30,31] studied the surface effect on the nano-indentation and showed that the apparent surface stress plays an important role in the depth-dependent hardness. In their papers, the apparent surface stress included two components: one was the surface stress, and the other was the pseudo-surface-stress induced by friction and plastic deformation occurring at the surface.[30,31] Atomistic simulations of the uniaxial tensile and compressive responses of nanostructures, e.g., carbon nanotubes,[32–34] nanoplates, nanobeams, and nanowires have also been completed.[3,9,35,36] The linear and nonlinear elastic properties of the nanoplates and nanowires of silicon, copper, and tungsten obtained from atomistic simulations by Miller and Shenoy,[3] Zhou and Huang,[9] and Villain *et al.*[35] are dependent on their characteristic size.

The role of surface stresses has been recognized by researchers to play an important role when the characteristic length scales become comparable with the atomistic scale. For example, Cammarata[37] studied the effect of surface stress on the critical thickness during the growth of a thin film. A classical continuum model to explain the surface effect on the elastic properties of nanostructures originated from Gurtin and Murdoch.[38] Later, it was further developed by many researchers[3,10,28,29,39] to analyze the elastic properties of nanostructured materials. Miller and Shenoy[3,39] compared the results obtained by the classical continuum model with those obtained by the atomistic simulations for nanobeams and nanowires, and found that the two methods almost lead to the same results. In essence, the classical continuum model assumes that a nanostructure=bulk+surface,[39] and the surface modulus of the nanostructure is different from that of the bulk.

Cuenot *et al.*[19] analyzed the surface effect on the elastic moduli of silver and lead nanowires both experimentally and theoretically, and they attributed the size dependence of the elastic moduli to the surface tension. In this paper, the elastic properties of the nanowires with outer diameters ranging from 20 to 140 nm were measured using contact atomic force microscopy (C-AFM). The apparent Young modulus of the nanowires is found to decrease with the increase of the diameter. The size dependence of the apparent Young modulus of the silver nanowires is attributed to the surface effect, which includes the effects of the surface stress, the oxidation layer, and the surface roughness. A theoretical analysis is presented to depict the size dependence of the elastic moduli.

It is different from the previous theoretical analysis of Cuenot *et al.*[19] in that both the constant surface stress and the surface moduli are included in the present analysis. We also show that the apparent surface modulus and constant surface stress of the silver nanowires may be experimentally determined. Unlike the previous works in the literature, we emphasize that caution should be exercised in explaining the size-dependent variation of the apparent elastic constants using the concepts of the surface stress and surface modulus. The experimentally obtained apparent surface modulus using synthesized nanowires with complicated surface morphology and composition may not be comparable with that obtained from the atomistic simulations or similar approaches.

II. EXPERIMENT

The elastic modulus of silver nanowires is measured by performing nanoscale three-point bending tests on nanowires suspended over etched holes in a silicon wafer. The AFM (Seiko Instruments Inc., SPA300-HV) cantilever tip is used to apply a small force at the middle point along its suspended length. In our prior work, the modulus of an individual silicon nitride nanobelt was measured by this SPM system.[40] The cantilever here with calibrated resonance frequency 33 KHz and spring constant about 2.7 nN/nm was used in the test.

Silver nanowires were synthesized using a simple hydrothermal method, and the diameter is controlled by the synthesizing time. This synthesis method was depicted in detail elsewhere.[41,42] A series of diameters ranging from 20 to 140 nm were successfully synthesized. Wafer silicon substrates with holes of different diameters etched by focused ion-beam (Dual-beam FIB, start 235, FEI) were used in the experiment. A suspension of silver nanowires in absolute ethyl alcohol was prepared by ultrasonic dispersion. Several drops of the silver suspension were dispersed onto the substrate and dried at room temperature to obtain the suspended nanowires over the holes.

Before measurement with AFM, scanning electron microscopy (SEM) images were obtained to determine the positions of the suspended nanowires over the holes. Most of the nanowires were lying on the substrate but only some of them were suspended over the holes and could be used to measure their mechanical properties (Fig. 1). Then the substrate was put into the chamber of the AFM system. Contaminants and lubricants affected the measurement, as did the thin layer of water that was often present when operating an AFM in air, so we performed the measurement in vacuum of 4×10^{-7} Torr. The AFM images of the sample were taken first at low magnification (20 μm $\times$ 20 μm) in order to visualize and to select an individual nanowire of interest suspended over one hole. Once a suspended nanowire was selected, an AFM image at higher magnification was carefully scanned. The suspended length L of the nanowire could be determined in the AFM image; the diameter D of the nanowire was measured in the SEM image. The AFM tip was then moved to the midpoint of the selected nanowire. A schematic diagram of a nanowire with midpoint deflected by an AFM tip is shown in the inset of Fig. 1. The adhesion between the nanowire and the silicon substrate was found to satisfy the assumption that both ends of the nanowires are clamped as the nanowire ends are found to remain in place even after the test is conducted.

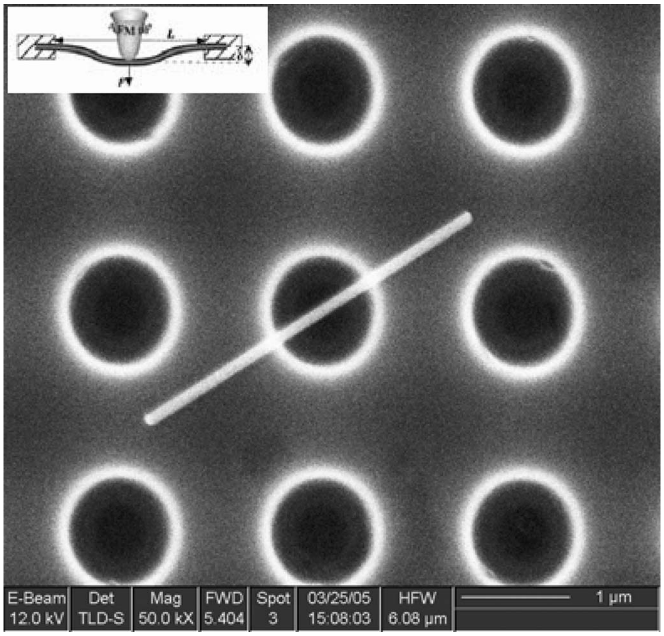

FIG. 1. SEM image of a typical suspended nanowire (diameter 79 nm). Schematic diagram of a nanowire with midpoint deflected by an AFM tip is shown in the inset.

Assuming that a force F is applied at the beam midpoint and induces a deflection δ, we measured both the force-deflection curves from the silicon substrate and the midpoint of the nanowire suspended, because both the nanowire and the cantilever of the AFM would bend when a piezoelectric actuator approached to the tip. The sample was brought to contact with the tip by a piezoelectric actuator, resulting in both cantilever deflection and the bending of the suspended nanowire. The deflection versus the applied force showed that the nanowire response was linear and elastic for the range of applied forces. No permanent deformation of the nanowires was detected. The slope of the applied force F versus the deflection δ (Fig. 2) gives the contact stiffness k_e. The sensitivity of the AFM detector was first calibrated by measuring a F-δ curve on the silicon substrate, and the slope

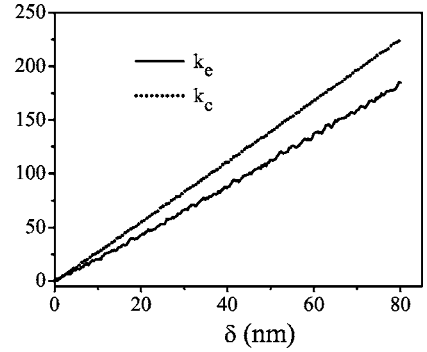

FIG. 2. Typical F-δ curves of the substrate and of a nanowire located on the substrate (diameter 79 nm).

of the linear portion of the curve (i.e., the stiffness k_c of the cantilever) was obtained (Fig. 2). Thus, the stiffness of the nanowire k_s could be derived from the following formula:

$$\frac{1}{k_e} = \frac{1}{k_c} + \frac{1}{k_s}. \tag{1}$$

In the general case, the deflection of the nanowires involves both bending and shear deformations. The superposition principle implies that the total deflection, δ, is the sum of the deflection due to bending, δ_B, and to shear δ_S. Using the unit-load method for a concentration load F, the deflection at the middle of the beam becomes[43]

$$\delta_B = \frac{FL^3}{192EI}, \quad \delta_S = \frac{f_s FL}{4\mu A}, \tag{2}$$

where f_s is a coefficient related to the shape of the sample's cross section ($f_s = 10/9$ for a beam with a circular cross section), and E and μ are the Young and the shear moduli of the considered materials, respectively, A is the area of cross section and I is the moment of inertia. For the beam with the circular cross section,

$$I = \frac{\pi D^4}{64}, \quad A = \frac{\pi}{4} D^2, \tag{3}$$

Eqs. (1)–(3) lead to

$$\frac{\delta_S}{\delta_B} = \frac{20}{3}(1+\nu)\left(\frac{D}{L}\right)^2. \tag{4}$$

If we take $\nu = 0.3$, when $D/L < 1/16$, then $\delta_S/\delta_B < 0.034$. In this case, the effect of the deflection δ_S due to shear can be neglected.[43] Thus, in order to reduce the shear influence, D/L should be smaller than $1/16$. Therefore, according to the theory for a three-point bending of a beam with two ends clamped and the constraint of the geometrical condition $D/L < 1/16$, the Young modulus E of the silver nanowire can be calculated using the following formula:[43]

$$E = L^3 k_s/(3\pi D^4), \tag{5}$$

where the stiffness k_s of the nanowire is obtained by Eq. (1). Using Eq. (5) and the measurement, the elastic moduli of a series of silver nanowires with different diameters can be obtained. The relationship between the Young modulus and the diameters of the nanowires is shown in Fig. 3. For large diameters, the measured values are almost independent of the diameter and are close to a constant value, which is lower than that reported in the literature for the bulk material [i.e., 76 GPa for Ag (Ref. 44)]. When D decreases down to 20 nm, the measured Young modulus increases continuously. For the silver nanowires with the smaller diameters ($D = 20$ nm), the measured Young modulus is approximately two or three times that of the bulk material. Similar behavior was previously observed for silver and lead nanowires.[19] Moreover, Cuenot *et al.*[19] pointed out that such an increase in stiffness of the nanowires cannot be explained by structural modifications of the materials at the nanoscale. It is noted that the errors of the Young moduli shown in Fig. 4 mainly came from the diameter D and the length suspended L of the silver nanowires, and they can be precisely measured by SEM and AFM with accuracies of the order of 3% and 4%, respectively.

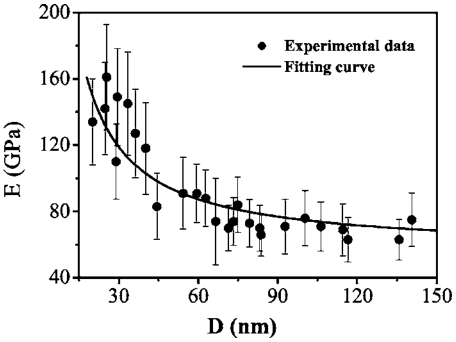

FIG. 3. Variation of the Young modulus as a function of the diameters of silver nanowires.

III. THEORETICAL ANALYSIS

In this paper, the theoretical analysis of the elastic property of the nanowires is carried out using the classical continuum model with consideration of the surface effect. As mentioned before, in this framework, nanostructures are regarded as being made up of bulk and a bounding surface in a continuum theory.[3,10,28,29,38,39] As the magnitude of the deflection of a nanowire was always small compared to its diameter, the theory of small deflections of beams is applied to evaluate the contribution of surface effects on the stiffness. The boundary conditions of the suspended nanowires were previously determined as clamped ends.

We begin the theoretical analysis with the image of a silver nanowire. From the images of silver nanowires with different diameters, it can be seen that an oxidation layer, thick or thin, always exists on the surface of the silver nanowire. Figure 4 shows the image of a typical silver nanowire with a core diameter $D = 46.6$ nm, and the thickness of the oxidation layer is $t = 3.8$ nm. This kind of core-layer structure, consisting of a silver core and oxidation layer, can be

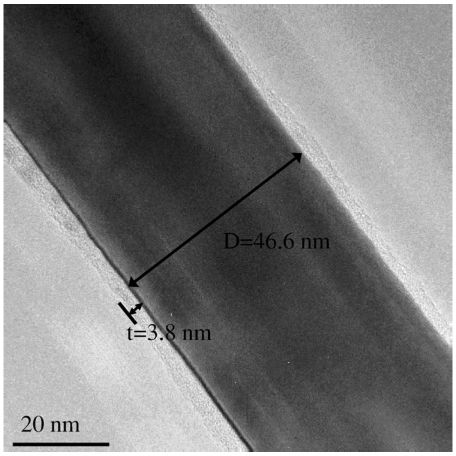

FIG. 4. Image of a silver nanowire (with core diameter 46.6 nm). The surface of the wire is covered with an oxidation layer (about 3.8 nm).

found in all samples of the silver nanowires with different diameters. According to the analysis of the property and image, it is found that the composition of the oxidation layer is different from that of the pure silver, and is also less compact and much softer than the pure silver. Therefore, the silver nanowire used for experimental measurement is assumed to be a composite coaxial nanowire, which consists of a silver core and an outermost oxidation layer.

The effective flexural rigidity $E_b I_b$ of a composite nanowire is

$$E_b I_b = E_{\mathrm{Ag}} I_{\mathrm{Ag}} + E_c I_c . \tag{6}$$

Here and in the following, the subscripts "Ag," "c," and "b" denote the core of the silver nanowire, the oxidation layer, and the bulk material without the surface effect, respectively. Thus, the Young modulus E_b of a nanowire with a circular cross section is given by

$$E_b = f^2 E_{\mathrm{Ag}} + (1 - f^2) E_c , \tag{7}$$

where $f = D_{\mathrm{Ag}}^2 / D^2$ is the volume fraction of the core of the silver nanowire, D_{Ag} is the diameter of the silver nanowire (core), and D is the outermost diameter of the oxidation layer.

As stated above, the large ratio of surface atoms to the bulk can have a profound effect on the properties of nanostructures, and this effect can be described by the classical continuum model when considering the surface effect.[39] When the elastic properties are concerned, the surface effect can be depicted by the surface stress. The surface stress is regarded as an excess of the bulk stress in the material near the surface, which is essentially caused by the difference between the property of a material near the surface and that of the material away from the surface.[45–47] Therefore, for the silver nanowires studied in the present paper, we introduce a surface stress to simulate the surface effect.

The surface stress generally varies with the strain. For infinitesimal deformation, the stress-strain relation can be approximated by a linear constitutive law.[3,28] When a nanowire is under bending, its surface is primarily subjected to tensile and compressive deformations in the axial direction of the nanowire. Thus, as in the works of Streitz *et al.*,[48] Wu *et al.*,[49] and Müller and Saúl,[47] the relation between the surface stress τ and the strain ε in the axial direction can be simulated by the 1D linear function

$$\tau = \tau_0 + S\varepsilon , \tag{8}$$

where τ_0 is the surface stress (excess quantity) at $\varepsilon = 0$ and S is the surface modulus.[47]

Assuming that a force F is applied at the beam midpoint and induces a deflection δ, if we consider the tension and bending at the same time, the Young modulus of the nanowire can be obtained by the energy competition between the surface and the bulk. The bending of a beam with both ends clamped results in an extension of its length. As a first approximation, it is assumed that the usual deflection curve of a clamped beam is not affected by the surface contribution. Thus, for clamped boundary conditions, the beam extension ΔL can be easily calculated and expressed as[43]

$$\Delta L = \frac{12}{5} \frac{\delta^2}{L} . \tag{9}$$

Then, the strain ε_0 of the neutro-axis is

$$\varepsilon_0 = \frac{\Delta L}{L} = \frac{12 \delta^2}{5 L^2} . \tag{10}$$

The strain $\varepsilon_{\theta r}$ along the axial direction of the nanowire at a point with the angle θ (measured from the neutral axis) and the radius r is (where $r - \theta$ constitute a polar-coordinate system in the circular cross section of the nanowire)

$$\varepsilon_{\theta r} = \varepsilon_0 + \frac{r \sin \theta}{\rho} , \tag{11}$$

where ρ is the radius of the curvature of the bending beam,

$$\rho = \left| \frac{L^3}{24 \delta (L - 4x)} \right| , \tag{12}$$

where x is the distance from the midpoint of the beam.

In terms of Streitz *et al.*[4] and Müller and Saúl,[47] the strain energy of a material with the surface stress effect can be separated into two parts: the work done against the surface stress and that required to strain the volume of the materials. Therefore, for a nanowire under consideration, according to Eqs. (8) and (10)–(12), the strain energy U_s corresponding to the surface stress effect is

$$\begin{aligned} U_s &= D \int_0^{L/2} \int_0^{2\pi} \left(\tau_0 \varepsilon_{\theta D} + \frac{S \varepsilon_{\theta D}^2}{2} \right) d\theta dx \\ &= DL\pi \varepsilon_0 \tau_0 + \left(\frac{12\pi}{L^3} D^3 \delta^2 + \frac{\pi}{2} DL \varepsilon_0^2 \right) S , \end{aligned} \tag{13}$$

where $\varepsilon_{\theta D}$ can be obtained by Eq. (11) with $r = D/2$.

The elastic energy U_b in a nanowire without the surface effect can be expressed as

$$\begin{aligned} U_b &= 2 \int_0^{L/2} \int_0^{D/2} \int_0^{2\pi} \frac{1}{2} E_b \varepsilon_{\theta r}^2 r d\theta dr dx \\ &= \frac{3\pi}{2L^3} D^4 \delta^2 E_b + \frac{\pi}{8} L D^2 \varepsilon_0^2 E_b , \end{aligned} \tag{14}$$

where E_b is the Young modulus of the silver nanowire without the surface effect, and is given in Eq. (7).

If the nanowire is regarded as a homogeneous material with an apparent Young modulus E, then the total elastic energy U can be expressed as

$$\begin{aligned} U &= 2 \int_0^{L/2} \int_0^{D/2} \int_0^{2\pi} \frac{1}{2} E \varepsilon_{\theta r}^2 r d\theta dr dx \\ &= \frac{3\pi}{2L^3} D^4 \delta^2 E + \frac{\pi}{8} L D^2 \varepsilon_0^2 E . \end{aligned} \tag{15}$$

According to the energy equivalency relation, $U = U_s + U_b$, the apparent Young modulus E (i.e., the Young modulus with the surface effect) can be obtained,

$$E = E_b + \frac{8S}{D} - \frac{12S\frac{\delta^2}{D^2}}{D\left(\frac{25}{4} + \frac{3\delta^2}{D^2}\right)} + \frac{10L^2\tau_0}{D^3\left(\frac{25}{4} + \frac{3\delta^2}{D^2}\right)}. \quad (16)$$

Generally, $\delta/D \ll 1$. Therefore, the apparent Young modulus can be obtained from Eq. (16),

$$\frac{E - E_b}{E_b} \approx \frac{8}{D}\frac{S}{E_b} + \frac{8L^2}{5D^3}\frac{\tau_0}{E_b}. \quad (17)$$

As far as the authors know, this formula has not been seen in the literature.

In particular, if the tension of the neutro-axis is ignored, namely, let $\varepsilon_0 = 0$ in Eqs. (13)–(15), the apparent Young modulus E is the same as that obtained from pure bending,[3]

$$\frac{E - E_b}{E_b} = \frac{8}{D}\frac{S}{E_b}. \quad (18)$$

If the surface stress related to the surface strain is ignored [i.e., $S=0$ in Eq. (13)] and $\varepsilon_0 = 0$ in Eqs. (14) and (15), the apparent Young modulus E can be obtained as

$$\frac{E - E_b}{E_b} = \frac{8L^2}{5D^3}\frac{\tau_0}{E_b}. \quad (19)$$

Equation (9) in the paper of Cuenot *et al.*[19] is similar to Eq. (19), and it can be obtained by multiplying $(1-\nu)$ on the right-hand side of Eq. (19).

As stated above, the silver nanowires used for the experimental measurement consist of silver cores and the outermost oxidation layers. Thus the parameters S and τ_0 in Eq. (17) are for the *composite silver nanowire*, instead of a nanowire of *pure silver*. The surface image of the oxidation layer of a silver nanowire is very rough. As the tip of AFM touches the surface of the nanowire, we postulate that a rough surface may consume more energy than a smooth surface during the deformation. Therefore, it should be noted that the surface stress in the above analysis includes the excess of the bulk stress in the *composite nanowire* caused by two factors. The first is the excess caused by the atoms at the outmost surface, as the atoms at the surface experience a different local environment than atoms in the interior of the material, and the equilibrium position and energy of these atoms will, in general, be different from those of the atoms in the interior. This part of the surface stress is usually due to several layers of the atoms. This is the usual meaning of the surface stress in the literature. The second factor is the surface roughness. This argument is similar to the analysis of the surface effect on the nano-indentation by Zhang and Xu.[30] They introduced the concept of an apparent surface stress, which includes two components: one is the surface stress, and the other is the pseudo-surface-stress induced by friction and plastic deformation occurring at the surface.

Following the above observation and arguments, we emphasize that the surface stress in this paper for the composite silver nanowires is also an *apparent surface stress*, and thus the obtained S and τ_0 are the *apparent quantities* of the composite silver nanowires. Equation (17) is used to fit the experiment results, and the fitting curve is also shown in Fig. 3. It can be seen that the tendency of the Young modulus versus the diameter of the silver nanowires described by Eq. (17) fits reasonably well with the experimental data. It is found that the optimized curve fitting yields the values for the three parameters, $E_b = 56$ GPa, $S = 8.7$ N/m, and $\tau_0 = 5.8$ N/m. The obtained S and τ_0 of the composite silver nanowires are in the same order 1–10 N/m of pure silver,[3,39] and the obtained E_b for the composite silver nanowires is smaller than that of pure silver, which is well described by Eq. (7).

IV. CONCLUSION

The apparent elastic modulus of silver nanowires with diameters ranging from 20 to 140 nm is measured using the C-AFM. It is found that the apparent Young modulus increases remarkably with the decrease of the diameter. This phenomenon is theoretically analyzed through considering the surface effect (surface stress, oxidation layer, and surface roughness). However, we emphasize that caution should be exercised in explaining the size-dependent variation of the apparent moduli of nanoscale materials using the concepts of the surface stress and surface modulus. The experimentally obtained surface stress and modulus with complicated surface morphology and composition may not be comparable with those obtained from atomistic simulations or similar approaches. Nonetheless, the surface effect can still be attributed to the energy competition between the surface and bulk. Therefore, in essence, many physical properties related to surface effects follow the similar variations with the characteristic sizes to that of the elastic moduli of the nanowires studied in this paper.

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