Mechanical Properties of Sharpened Carbon Nanotube Tips

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Mechanical Properties of Sharpened Carbon Nanotube Tips

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We investigate the mechanical properties of sharpened nanotube tips for use in scanning probe microscopy in terms of tip bending under a force acting on the side of the tip. The sharpened nanotube probe fabricated by means of a modified electrical breakdown process effectively acts as a probe with high lateral resolution not only in the topographic measurement but also in the potential distribution measurement. Based on molecular mechanics calculations for a sharpened triple-walled nanotube probe, although the interlayer van-der-Waals interaction weakens the probe stiffness expected on the basis of the continuum model, the stiffness of the tapered nanotube is confirmed to be 10 times higher than that for a single-walled nanotube with the same tip radius and the same length. [DOI: 10.1143/JJAP.44.1637]

KEYWORDS: carbon nanotube, nano-mechanics, electrical breakdown process, scanning probe microscope tip, stiffness, bending modulus

1. Introduction

Carbon nanotube (CNT) probes¹–³ for use in scanning probe microscopy (SPM) are now widely applied in high-resolution imaging. The stiffness of the nanotube is crucial to obtaining an image with high stability. Insufficient stiffness of the nanotube probe causes image modification because of bending or vibration of the nanotube tip.⁴ The easiest way to prevent these effects is to use thick nanotube probes. However, the use of thick nanotube probes leads to the degradation of the image resolution. In order to improve the image resolution and retain a high stability, we have to use a shorter nanotube with a smaller diameter. In this case, however, the high aspect ratio, which is one of the features of the nanotube probe, is reduced. Consequently, a nanotube which has a sharp tip and a sufficiently thick body is appropriate for use in the SPM probe tip. The processing of the individual nanotube using an electrical breakdown has been reported.⁵ This process allows the shell of the multiwall nanotube to be peeled off layer by layer. Recently, we have modified this process to involve cutting the nanotube and then sharpening the tip to adjust the length of the nanotube probe, and the electrical breakdown process was performed in a scanning electron microscope (SEM) manipulator, termed the nanofactory.⁶ In this study, we investigate the mechanical stiffness of the sharpened nanotube tip for use in SPM. We also demonstrate the molecular mechanics calculations for analysis of the bending behavior of the sharpened nanotube tip.

2. Experiments

CNTS, which were synthesized using a DC arc discharge with a water-cooled coil,⁷ were aligned on a Pt-coated knife edge by an ac electrophoresis method. Pt-coated Si tips were used for the base of the nanotube tip. The attachment of a nanotube onto the Si tip and the processing of the nanotube were performed in the nanofactory in vacuum at less than 5 × 10⁻⁵ Pa. While the nanotube was processed, the current passing through the nanotube was monitored. When a certain voltage is applied to the nanotube, the electrical breakdown is induced and the current decreases stepwise, indicating the sequential destruction of individual nanotube shells. Continuing the stepwise decrease of the current cuts the nanotube and sharpens its tip.⁶ Single-walled nanotubes synthesized by catalytic chemical vapor deposition⁸ on Si substrates were used for the samples for the SPM measurement. The SPM measurements using the FM detection system were performed in vacuum at ~10⁻⁴ Pa, at room temperature. The FM detection system enables us to obtain a stable potential image by means of Kelvin force microscopy (KFM) under the vacuum condition.

3. Scanning Probe Microscopy Using Sharpened Nanotube Probe

Figure 1 shows a TEM image of an example of a sharpened nanotube probe. The nanotube was successfully sharpened and tapered. The tip diameter and the length of the nanotube are ~5 nm and ~280 nm, respectively. The diameter of the original nanotube was measured to be 12 nm from near the base in the TEM image. Thus, we have successfully fabricated a sharpened nanotube probe tip using a well-controlled electrical breakdown process.

Figures 2(a) and 2(b) show the topographic and potential images of the nanotube on Si. The cross-sectional profiles are also shown in Figs. 2(c) and 2(d). The diameter of the nanotube on the Si, d, is estimated from the height of the nanotube to be 2.6 nm. The width of the profile, Wcnt, is 9.4 nm. Assuming that the tip of the nanotube probe is hemispherical and the nanotube shape on the Si is a perfect cylinder, the tip diameter, D, is estimated to be 8.5 nm from the relation D = Wcnt/4d. This value is comparable to the tip diameter estimated from the TEM image. This indicates that the tip of the nanotube probe is sufficiently close to the sample surface in the noncontact mode and that no

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Fig. 1. TEM image of sharpened nanotube probe.
deformation of the nanotube probe occurred during SPM imaging. Consequently, the sharpened tip of the nanotube probe is sufficiently stiff to obtain a stable image.

The potential distribution image shown in Fig. 2(b) is very clear in comparison with the images obtained by means of conventional KFM performed in air in the amplitude modulation (AM) mode, and the contact potential difference (CPD) obtained in the vacuum is ~10 times larger than that obtained in the air. This is because the effect of an adsorbate such as a water layer, which creates a dipole to compensate for the CPD, is effectively reduced under the vacuum condition. Furthermore, the width of the image for the nanotube of 2.6 nm diameter is measured to be ~25 nm from the cross-sectional profile of the potential image shown in Fig. 2(d). The AM detection performed in air using the nanotube probe gives ~100 nm for the width of a similar nanotube. The improvement of the resolution of the potential image is due to the reduction of the tip-sample distance, which was achieved by using the FM-detection under the vacuum condition.

### 4. Calculation of Bending Behaviors

In order to clarify the mechanical properties of the sharpened CNT tip, we investigate the bending behavior of the sharpened nanotube tip on the basis of the continuum model and the molecular mechanics calculations. The stiffness of the sharpened nanotube tip is compared with that of a single-walled nanotube (SWNT), which has the same diameter as the tip of the sharpened nanotube and the same total length. Here, we examined the spring constant for the tapered shape and the triple-walled nanotube (TWNT), as illustrated in the continuum model shown in Fig. 3. Molecular mechanics calculations were also performed for a sharpened TWNT consisting of (5,5)–(10,10)–(15,15) nanotubes and a (5,5) SWNT, where an empirical potential field of MM3 was used for the intra-nanotube and the van-der-Waals (vdW) interaction with a Lennard–Jones type potential was used for the inter-nanotube, respectively.

Under the force, $W$, acting on the side of the nanotube cantilever beam for the continuum model, we solve a shape formula given by

$$y \frac{\pi r^4(x)}{4} \frac{d^2y}{dx^2} = W(L - x),$$

Fig. 3. Nanotube models for calculations: (a) taper, (b) TWNT for continuum model, (c) SWNT for molecular mechanics calculations, (d) DWNT for molecular mechanics calculations. Coordinates for the calculations are also shown.
under the boundary condition of \( \frac{dy}{dx} |_{x=0} = 0 \), \( y(0) = 0 \), where \( r(x) \) is the radius of the nanotube at position \( x \) along the tube axis, \( y \) is the position of the tube center at \( x \), and \( L \) is the length of the nanotube. For the tapered shape shown in Fig. 3(a), the relation of \( r(x) = \left( r_0 - R \right) x / L + R \) is used for this calculation. On the basis of these relations, the spring constant of the tapered nanotube is given by

\[
 k = \frac{3Y \pi (r_0 R^3 - r_i^3)}{L^3} \quad (2)
\]

where \( r_0, r_i \) and \( R \) are the outer and the inner radii at the tip and the radius at the base of the sharpened nanotube, respectively. When the nanotube has the relation of \( 3r_0 = R \), i.e., the TWNT, the spring constant is \( \sim 27 \) times higher than that for the SWNT. For the TWNT shown in Fig. 3(b), the relation \( r(x) \), equals \( r_0, 2r_0 \) and \( 3r_0 \) at the corresponding position \( x \). In this model, the spring constant for the TWNT is \( \sim 22 \) times higher than that for the SWNT. According to these calculations, the tapered nanotube is much stiffer than the SWNT with the same tip diameter and length. In these cases, however, the vdW interactions between the interlayers are neglected.

Figure 4 shows the deflection dependence of the force acting on the side of the nanotube tip for the SWNT and the TWNT with \( L = 7.48 \) nm as obtained from the molecular mechanics calculations.

Figure 4. Deflection dependence of the force acting on the side of the nanotube tip for the SWNT and the TWNT with \( L = 7.48 \) nm as obtained from the molecular mechanics calculations.

Figure 5. Log-log plot of length dependence of the spring constant calculated on the basis of the molecular mechanics calculations for SWNT, (a), and TWNT, (b).

Figure 6. Length dependence of the ratio of the spring constant for the sharpened TWNT and the SWNT of the same length.

Figure 6 summarizes the length dependence of the ratio of the spring constant for the sharpened TWNT and the SWNT of the same length. The ratio increases with increasing length. It is noted that the spring constant of the protruded SWNT is proportional to \( L^{-3} \) and the vdW interaction is proportional to the total length. As a result, the effect of the interlayer vdW interaction becomes weak in comparison with the intralayer interaction, and thus the mechanical
property of the TWNT becomes closer to that in the continuum model. As shown in this figure, the ratio exceeds unity for all lengths, and thus the sharpened nanotube tip has a higher stiffness for the SWNT even with the occurrence of the interlayer vdW interaction. It is noted that the cone angle of the nanotube tip shown in Fig. 1 corresponds to that of the TWNT model with the length of ~50 nm in Fig. 6, and thus the expected stiffness of the tapered nanotube probe becomes ~10 times higher than that for the SWNT with the diameter of 5 nm.

5. Conclusions
We have investigated the mechanical stiffness of the sharpened nanotube probe used for SPM measurements. The modified electrical breakdown process of the nanotube has been utilized for the fabrication of the sharpened nanotube probe. The sharpened nanotube effectively acts as a probe with a high lateral resolution not only in the topographic measurement but also in the KFM measurement. The molecular mechanics calculation reveals that the stiffness of the sharpened nanotube probe is sufficiently higher than that of the SWNT probe with the same length even with the occurrence of the interlayer vdW interactions.

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