Nanotube manipulation with focused ion beam

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We demonstrate the ability to straighten and align metal-coated carbon nanotubes with a focused ion beam. The metal-coated nanotubes align toward the source of the ion beam allowing their orientation to be changed at precise angles. By this technique, metal-coated nanotube tips that are several micrometers in length are prepared for scanning probe microscopy. We image high-aspect-ratio structures on the surface of a cell using these tips. © *2006 American Institute of Physics*. [DOI: 10.1063/1.2161395]

Probes equipped with carbon nanotube tips have been proposed to improve the resolution and capabilities of atomic force microscopy (AFM) (Refs. 1–5) and various other scanning probe force microscopy (SPM) techniques.[6–10] Among other advantages, the long and thin cylindrical shape of the nanotube reduces the tip convolution effects compared to pyramidal tips made of etched crystal silicon. To accurately map the forces between the sample and the nanotube tip in these applications, straight, well-aligned, and stiff nanotubes on AFM cantilevers are required.[3,11,12]

In this letter, we present a fast and reliable method of making straight metal-coated single-walled carbon nanotubes aligned in a precisely controlled direction. Metal coating on single-walled carbon nanotubes has been shown to be valuable for functionalization, for example, for magnetizing the nanotube tips for magnetic force microscopy.[10] Here, we show that metallization has additional advantages: It makes the nanotube stiffer[13] and also enables its reproducible manipulation with a focus ion beam (FIB). We demonstrate the application of these tips to image high-aspect-ratio topographic features.

The nanotubes are grown by chemical vapor deposition[14] using a thin film of iron catalyst on a wafer of commercial AFM cantilevers[15] (type Tap 150 from Nanodevices).[16] The cantilevers are shaped like diving boards with sharp pyramids at the free end. We inspected more than 200 cantilevers with scanning electron microscopy (SEM). 24% of the cantilevers had a single nanotube pointing straight up from the apex of the pyramid within 35° or better. An additional 36% of the cantilevers had a curving nanotube pointing back toward the pyramid, with the end either free or touching the pyramid. We coated the nanotube tips with a sticking layer of Ti, followed by another metal such as Co, Ni, or Pt in an electron-beam evaporator, sometimes followed by a Ti or Au cap layer to prevent oxidation.[17] The crystal thickness monitor in the evaporator measures the nominal thickness of the coating. The thicknesses of metal coating on the nanotube tips in the figures are summarized in Table I. The directions of the nanotubes with one free end often change during the coating process.

We use a dual-beam FIB machine with SEM capability.[18] The ion beam consists of Ga^{+2} ions accelerated at 30 kV. For the alignment process, we apply repeated single scans of the imaging mode of the ion beam, while we track the progress of each alignment by taking a SEM picture between ion beam scans. In each of our scans in the ion beam-imaging mode, the SEM beam is blanked while the ion beam scans the whole imaged area once. The sample is hit by the ion beam at as many points as the number of pixels in the image, distributed evenly over the area of the image. The ion

TABLE I. The materials and thicknesses of the coating on each tip shown in the figures.

Tip in:	Layer 1	Layer 2	Layer 3
Figure 1(a)	Ti, 3 nm	Co, 7 nm	Ti, 3 nm
Figure 1(b)	Ti, 8 nm	Pt, 80 nm	⋯
Figure 2	Ti, nm	Ni, 35 nm	Pt, 20 nm
Figure 3	Ti, 5 nm	Ni, 25 nm	Pt, 20 nm

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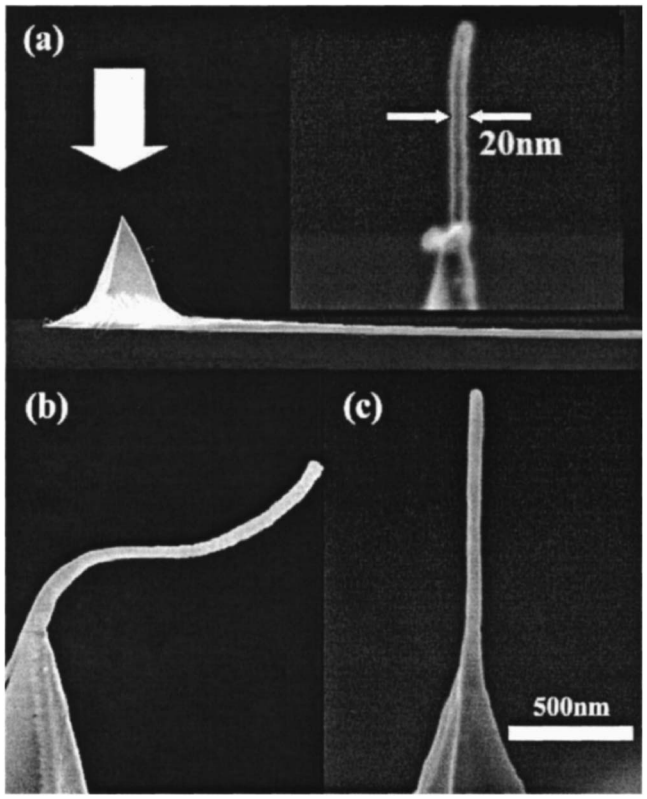

FIG. 1. (a) SEM image of an AFM cantilever with a metal-coated nanotube on the pyramid. The arrow shows the direction of the FIB and the metal coating. Inset: Image of the metal-coated nanotube at the apex of the pyramid. (b) and (c) A metal-coated nanotube tip before (b) and after (c) alignment with FIB. It is 1.2 μm long and has a diameter of 45 nm, as determined from the SEM image, which gives it an aspect ratio of 27.

beam is 7 nm in diameter, and stays focused on each point for typically a few microseconds in our application before moving to the next point. We do not want to extensively damage the nanotubes, so we avoid continuously scanning our sample with the ion beam. We also avoid using the drilling mode of the ion beam, where the ion beam scans a specified narrow area on the sample for typically several seconds or longer. The ion beam is directed as shown in Fig. 1(a). The metal-coated nanotube aligns parallel to the direction and opposite to the flux of the ion beam. We speculate that the alignment could be due to changes in the surface energy of the metal coating during the exposure to the ion beam. The final alignment of the nanotube is verified by taking SEM scans from two different angles. Alignment of the nanotubes is stable over time; they do not bend back when inspected again, even after weeks.

Figure 2 shows how this technique can be used to straighten a twisted metal-coated nanotube in a step-by-step process. Figure 2(a) shows the initial shape of the nanotube. As seen from Figs. 2(a)–2(e), the nanotube becomes increasingly straighter after several 6.35 s high-resolution single ion beam scans applied between each SEM scan. The magnification of the ion beam scans ranges between 10 kx and 50 kx, and the maximum dose of ions delivered at an individual scan is 4.0×10^6 ions μm^2. We use a 10 pA aperture, and the current of the ion beam is 15–16 pA. Figure 2(f) is taken from a different angle than Fig. 2(e) to verify the resulting alignment of the metal-coated nanotube.

Precise control over the orientation of a metal-coated nanotube using a FIB is further illustrated in Fig. 3. We first

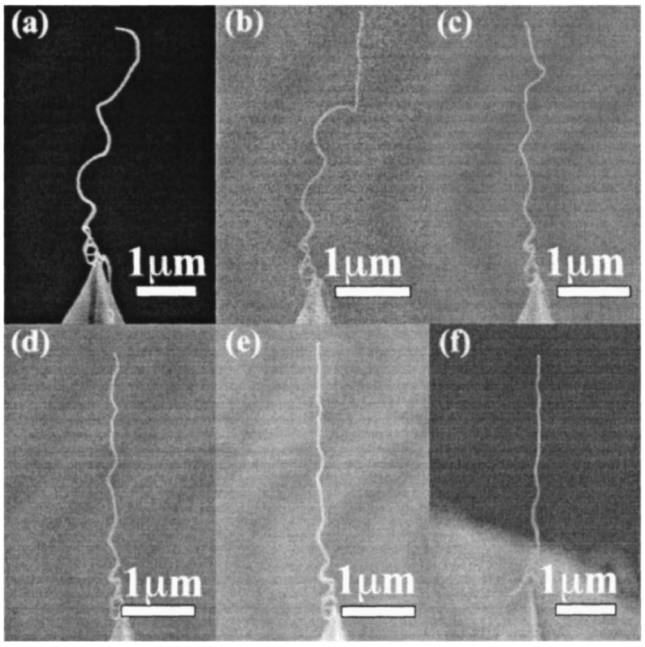

FIG. 2. (a) to (e) SEM images of a metal-coated nanotube all acquired from the same angle. A dose of ion beam was applied between each of the images. (f) SEM image of the same tip in (e) without additional dose of ion beam, after the sample stage is rotated 90°.

tilt the sample stage at the maximum allowed angle of 52°, and then align the metal-coated nanotube with respect to the axis of the pyramid of the AFM tip as seen in Fig. 3(a). Next, we scan the tip with the ion beam incident at precise angles with respect to the initial orientation as shown on each image. The coated nanotube bends incrementally sideways as seen in Figs. 3(a)–3(e). Then, we bend it to the other side and bring it back into vertical position in Fig. 3(f).

Micrometers-long straight cylindrical tips are ideal for SPM imaging of samples with high-aspect-ratio topography, such as cells or large molecules. The sample we choose in order to test our tips aligned by FIB are microvilli on Madin Darby Canine Kidney (MDCK) cells as seen in the SEM image in Fig. 4(a). MDCK cells are often used in studies of cell polarity, including questions of how cells set up and maintain distinct subcellular domains, and how cells form and maintain cell-cell junctions.[19–21] We are able to image

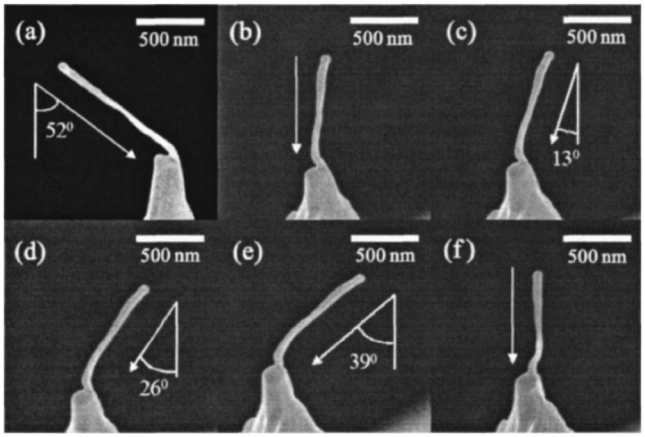

FIG. 3. (a) SEM image of a metal-coated nanotube aligned at 52° with respect to the axis of the pyramidal tip. (b) to (f) SEM images of the same tip after being exposed to the ion beam incident along the direction of the arrow drawn in each image.

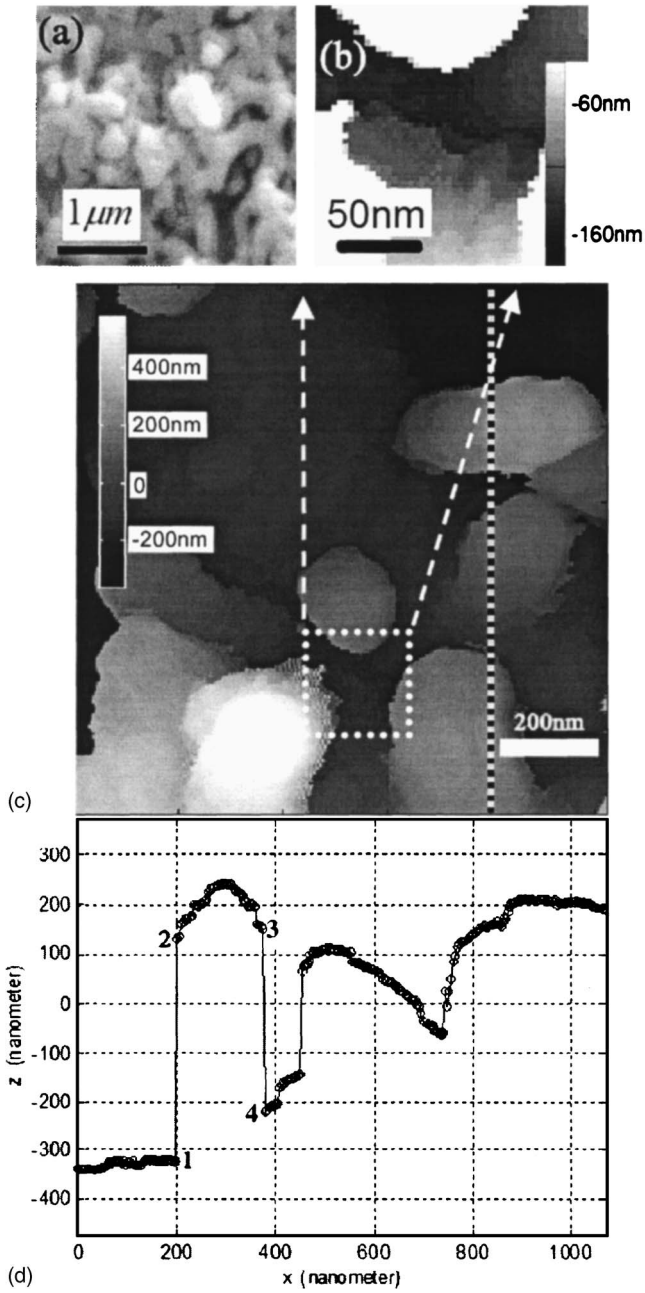

FIG. 4. (a) A SEM image of microvilli on cell surface coated with 5 nm Pt for better contrast. AFM image of these microvilli, taken with the tip in Fig. 1(c), is shown in (c). Zoomed in image of the features around the base of the microvilli inside the dashed square in (c) is shown in (b). Cross section of topography along the vertical dashed line in (c) is given in (d). Points 1 to 4 outline an almost vertical microvillus.

tall structures standing in various orientations with respect to the sample surface [Fig. 4(c)] using the tip in Fig. 1(c). In the particular case of a vertically standing villus, our metal-coated nanotube tip resolves between two points separated by more than 400 nm vertically and less than 5 nm horizontally [between Points 1 and 2 in Fig. 4(d)]. Moreover, features on the surface of the cell around the base of the microvilli are resolved, as seen in Fig. 4(b), which are generally beyond the reach of pyramid-shaped tips.

In conclusion, the ability to straighten and align individual metal-coated nanotubes using the imaging mode of an FIB has been demonstrated. Future work will include modeling the interaction between ion beams and coated nanotubes to fully understand the manipulation of metal-coated nanotubes by ion beams. This alignment technique may prove to be useful with carbon nanotubes coated with other materials, such as dielectrics. The tips prepared with this method significantly reduce the convolution effects from the shape of the tip during topography imaging. Micrometers-long metal-coated carbon nanotube tips not only remarkably improve high-aspect-ratio topography imaging, but they are also potentially useful for magnetic force microscopy, electrostatic force microscopy, scanning gate microscopy, and other SPM techniques.

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