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Electron-Beam-Induced Deposition of Carbon Nanorod via Spot Mode as Highly Stable and Sensitive AFM Probe Tip

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Precise fabrication of high-aspect ratio and sharp carbon probe tips for atomic force microscopy (AFM) has been investigated by use of electron-beam-induced deposition (EBID) via spot mode, which can be easily produced in large quantities with high repeatability. The EBID process and subsequent SEM imaging were performed in an FEI Helios NanoLab 660. Selection of electron beam voltage, current, and deposition time is critical for determining the radius and height of the resulting tip, while optimal focus and astigmatism correction are crucial for precise and repeatable production of a sharp tip. Through systematic testing, an electron beam voltage of 18 kV and beam current of 50 pA has been proved to produce tips with the smallest radius-of-curvature (15±5 nm) via spot mode.

Fig. 1a-b shows SEM images of a commercial Olympus-TR400PB silicon nitride (SiN) cantilever (a) before and (b) after EBID of carbon nanorod by localizing the beam at a point on the tip apex of the cantilever. Compared with ~30 nm tip radius of a commercial probe, a high aspect-ratio and sharp tip is grown, the tip radius is sharpened to ~15 nm for the EBID-modified probe, with tip height of 200 nm while setting dwell time for 15 s. Fig. 1 c shows SEM images of a commercial Bruker MLCT-010 SiN tipless cantilever (probe F). Fig. 1 d and 1e shows the low and high magnification of the same cantilever (probe F) followed by EBID of the carbon nanorod tip with 2 μm height, while setting dwell time for 150 s. We noticed that adjustment of the dwell time can tailor the tip height, but does not increase its radius of curvature. As shown in Fig. 1f, the tip height is shortened to 1 μm while setting dwell time for 60 s. For the image of Fig 1c, the axis of probe is tilted at 0˚to the electron beam. All other five images, the axis of probe is tilted at 52˚to the electron beam.

Topographical AFM imaging of λ-DNA and highly ordered pyrolytic graphic (HOPG) were used to evaluate the imaging performance of the modified probes and to show that they are sufficiently robust for routine AFM imaging. All AFM images were recorded using an Asylum MFP3D Bio AFM in air, and was operated in AC mode to minimize the lateral and shear forces which is more occurred in contact mode, making the modified tips suitable for imaging soft biological samples. Fig. 2a-b shows AFM images of DNA on mica. The results indicated that modified carbon tips can produce sharper images of DNA molecules (~10 ±2 nm in width) than commercial SiN tips (~15 ±2 nm in width). We also note that the periodicity along the DNA chain depends on the orientation of the DNA molecule with respect to the direction of the tip movement (Fig. 2b). We noticed that probe height is an important factor while imaging two dimensional materials, such as fresh exfoliated atomic layers of HOPG. Fig. 2c-d shows that shorter tips of 1 μm can generate sharper and more sensitive images with monolayer thickness measurement (~3.5-3.6 Å) compared with a longer tip of 2 μm (~3.7-4.0 Å) which is probably due to the bending and oscillation of the nanorod tip during the tapping process.

We believe that the precise fabrication of high-aspect ratio and sharp carbon tips by EBID has great potential for improving the resolution of biological samples and membrane, as well as to image deep trench or small particles in AFM which is previously limited by the sidewall angles of the tip [4].

References:
[1] N. Wilson *et al*, Nature Nanotechnology **4** (2009), p. 483.
[2] C. Cheung *et al*, PNAS **97** (2000), p. 3809.
[3] F. Zenhausern *et al*, Journal of Applied Physics **73** (1993), p. 7232.
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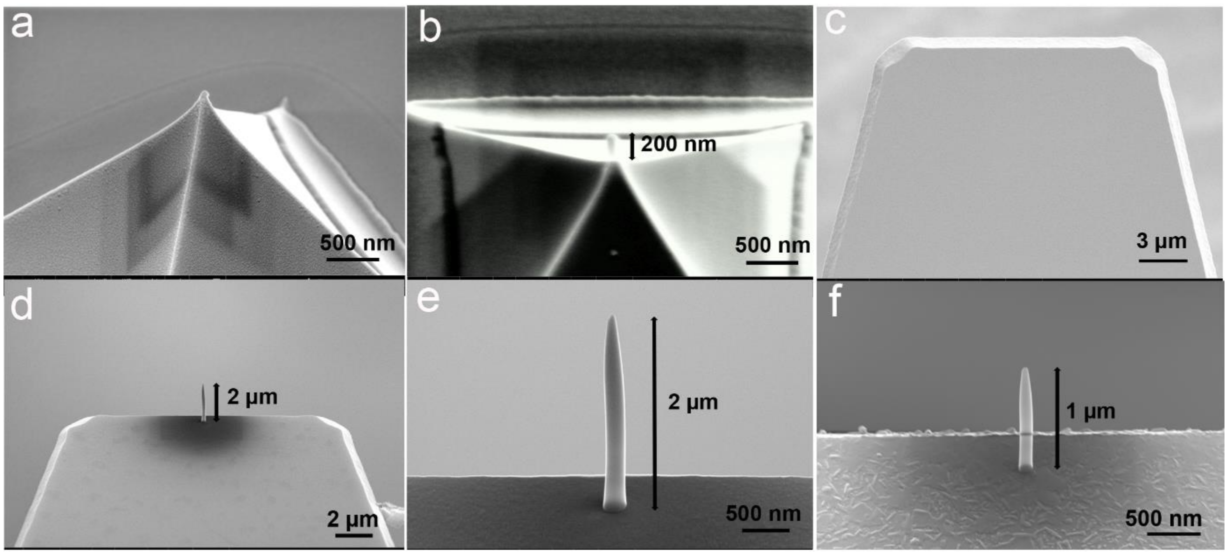

Figure 1. SEM images of (a) a commercial Olympus-TR400PB SiN cantilever and (b) the same cantilever followed by the EBID of carbon nanorod tip via spot mode with 200 nm height. (c) a commercial Bruker MLCT-010 SiN tipless cantilever (probe F); (d)-(e) low and high magnification of same cantilever (probe F) followed by EBID of carbon nanorod tip with 2 µm height via spot mode; (f) similar with (e) but with different tip height 1 µm. For the image of (c), the axis of probe is tilted at 0˚to the electron beam. All other five images the axis of probe is tilted at 52˚to the electron beam. All the deposited tip radius is between 10-20 nm.

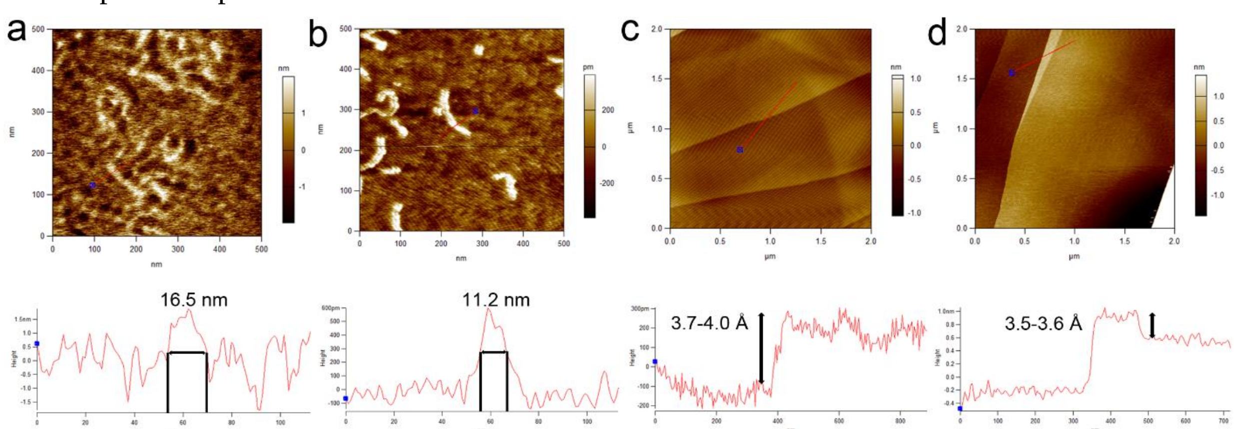

Figure 2. (a)-(b) AC mode AFM images of DNA on mica (a) by using commercial Olympus-TR400PB SiN probe tip, with cross section of the molecule (16.5 nm);, (b) by using the same cantilever followed by the EBID of carbon nanorod tip with 200 nm height via spot mode, with cross section of the molecule (11.2 nm); (c)-(d) fresh exfoliated HOPG surface by using commercial Bruker MLCT-010 SiN tipless cantilever followed by EBID of carbon nanorod tip (c) monolayer thickness measurement between 3.7-4.0 Å with tip height 2 µm and (d) monolayer thickness measurement between 3.5-3.6 Å with tip height 1 µm.

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