High-resolution nanowire atomic force microscope probe grown by a field-emission induced process

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A technique to grow a nanowire probe on an atomic force microscope (AFM) tip using a field-emission induced growth process has been developed. The simple and highly reproducible technique produces vertically aligned nanowire probes whose length is controlled by the growth duration. Using a cantilever clamping arrangement, nanowire probes can be grown on low-stiffness cantilevers. Experiments using the robust nanowire AFM probe demonstrate its ability to produce high-resolution tapping mode AFM images and improved profiling of structures with steep sidewalls due to its very sharp tip and high aspect ratio. No degradation in imaging performance was observed after a period of continuous scanning and storage. © 2004 American Institute of Physics. [DOI: 10.1063/1.1765202]

Atomic force microscopy $(AFM)^1$ is an important tool in surface analysis for both conducting and nonconducting materials. With integrated circuit devices getting smaller and the rise in importance of nanotechnology, surface studies of specimen features down to atomic levels are becoming more common. The need for higher resolution means that sharper tips with a smaller radius of curvature and a higher aspect ratio are required to obtain high-resolution AFM images and to minimize the distortion of images that depend on the shape of the tip. Several techniques to fabricate sharp tips have been reported. The techniques can be generally classified under three main approaches—by etching, deposition, or attachment. Anisotropic etching, isotropic etching, dry etching, oxidation sharpening and focused ion beam etching^{2,3} have all been used to microfabricate sharp tips.⁴⁻⁶ Techniques such as utilizing a sharp corner of the cantilevers for the probing tip,⁷ using a silicon nitride pyramid tip,⁵ and using a sharp single-crystal silicon tip⁴ have been able to achieve good results in resolving relatively flat samples with high resolution. Methods that are based on deposition include techniques using electron beam induced deposition⁸ and diamond-like film deposition.⁹ Another alternative is to attach a thin probe to an otherwise conventional tip, and examples include attaching a zinc oxide whisker to the cantilever end,^{10,11} attaching a carbon nanotube (CNT) or bundles of CNTs to an existing silicon etched tip¹²⁻²⁰ and attaching a single nanowire to an existing silicon etched tip.²¹ While CNT probes have so far shown great potential due to their high aspect ratio and small radius of curvature, as well as being chemically stable and mechanically robust, the difficult and low yield process of mounting a single SWNT on an existing probe remains a stumbling block. Methods that directly grow a single CNT by surface growth chemical vapor deposition (CVD) processes^{14,22,23} have low yields that can be as low as around 10% for individual CVD SWNT tips.²² In addition, CNT tips may also give rise to artifacts introduced by the probe tip structure.²

In this letter, we report the controlled growth and performance of tungsten nanowire probes on standard commercial

silicon etched AFM tips by a field-emission induced growth method²⁵ in the ambient of a suitable precursor gas, tungsten carbonyl. A technique in making sharp tungsten nanotip with a two-step field-emission induced growth process was previously reported.²⁶ The relatively long tungsten filament probes were of a thicker shank diameter to provide imaging stability and the method does not always yield vertically aligned probes, while the process damages the underlying silicon base tip due to vacuum arc formation. The current single-step growth method uses a proximal anode and operates in a lower field emission current regime where nanowire forking during growth is avoided, yielding much finer vertical nanometer probes that are self-aligned to the apex of the original AFM tip with excellent reproducibility. When a sufficiently high electric field is applied to the sharp AFM tip, field emission from the AFM tip occurs, initiating the growth of a nanowire at the tip. At the onset of field emission, the field-emission electrons dissociate the precursor gas molecules near the AFM tip and the ionized molecules are then attracted back to the AFM tip which results in a selfsustained growth of a tungsten nanowire with the sharp tip end of the formed nanowire taking over as the field-emission source. The use of a micromanipulator to hold down the cantilever allows the growth technique to be used on lowstiffness cantilevers typically used for soft-sample applications or for contact-mode AFM applications, which would otherwise not be possible due to cantilever deflection caused by electrostatic attraction to the anode.

The growth procedure is carried out in the high vacuum chamber of an environmental scanning electron microscope (XL-300 ESEM FEG, Philips), equipped with two three-axis micromanipulators (MM3A, Kleindiek Nanotechnik). The SEM provides high-resolution viewing capabilities to assist in the precise positioning of an electrochemically etched tungsten sharp tip (anode) and the AFM tip (cathode), as well as inspection of the tungsten nanowire that is grown. An etched tungsten wire tip is attached to each micromanipulator. One tip acts as the anode which is placed in close proximity ($\sim 3 \mu$ m) to and aligned to the axis of the AFM tip, while the other is used to hold down the AFM cantilever [Fig. 1(a)]. The latter is necessary for growth on low-

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5207

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FIG. 1. (a) SEM image showing the orientation of the AFM tip and the two etched tungsten sharp tips that are attached to the micromanipulators positioned for field-emission induced growth. (b) SEM image of a 200 nm long tungsten nanowire AFM tip. The tungsten nanowire appears much thicker than as-grown in the SEM image due to contamination during viewing.

stiffness cantilevers to prevent the AFM tip from contacting the anode when an anode bias is applied to initiate field emission. This clamping arrangement has been found to work well for cantilevers with stiffness values below 1 N/m. The sharp anode localizes the electric field with an axial alignment to the AFM tip so that a vertical nanowire will be grown. Previous attempts with distant anode positioning yielded poor reproducibility of the grown probe alignment, and often damage to the original tip as a result of highenergy vacuum arc formation.

After alignment and cantilever clamping, nanowire growth is carried out by first admitting tungsten hexacarbo $nyl [W(CO)_6]$ vapor through a leak valve and nozzle directed at the growth area. The local vapor pressure is estimated to be around 10^{-2} mbar. The anode bias is then applied and growth is carried out at a constant field-emission current of typically 100 nA. On uncoated silicon AFM tips, the silicon apex experiences localized melting upon field emission, which rounds off the tip in a controlled manner, but does not otherwise affect the nanowire growth. Initiation of field emission from a melted tip requires an anode bias of around 650-700 V, but the bias drops to between 80 and 150 V during steady-state nanowire growth. The nanowire length is controlled by the duration of growth cycle. Although lower field-emission currents give rise to tungsten nanowires of smaller diameter, very thin nanowires are not suitable for this application as such nanowires cannot be grown long and yet remain stiff enough to be used as AFM probes. A good compromise was achieved at a field-emission current of 100 nA for tungsten nanowires up to 1 μ m in length. The fieldemission induced growth duration is controlled in the range of 3-10 s, producing single tungsten nanowires between 200 nm and 1 μ m in length which are robust enough for AFM applications [Fig. 1(b)]. Transmission electron microscope (TEM) studies of the tungsten nanowires grown on a TEM grid with the field-emission induced growth method under similar growth current and pressure found that the polycrystalline tungsten nanowire is typically 5-10 nm in diameter depending on the exact growth conditions, comprising a tungsten core of 3-6 nm coated with a layer of low Z carbonaceous material. The sharp tip end of the tungsten nanowire has a smaller radius of curvature, typically around 1-2 nm, than its shank radius due to the nature of tungsten ion deposition at the tip. The nanowire is expected to be similar to that grown on the cantilever tip as the substrate is remote from the vicinity of the growth front between the nanowire tip and anode. Cobalt nanowire probes have also been grown from suitable precursors and demonstrate similar



FIG. 2. AFM tapping mode images of a platinum thin film on silicon substrate using (a) a standard tapping mode AFM tip (displayed height range =4 nm) and (b) a nanowire probe (displayed height range =5 nm). The scan area is 300 nm \times 300 nm.

imaging performance, as well as the ability to perform magnetic force microscopy.

The fabricated tungsten nanowire AFM probes are used in tapping mode AFM imaging on a JEOL JSPM-5200 SPM to assess their capabilities relative to commercial AFM tips. The standard tapping mode AFM tips used for comparison are BS-Tap300Al AFM tips (40 N/m, <10 nm tip radius) from BudgetSensors. The tungsten nanowire is also grown on these tips so as eliminate the possibility of any improved performance that may result from different specifications of the AFM cantilever.

Scans of a silicon substrate with a thin evaporated platinum film at a scan size of 300 nm \times 300 nm obtained using a tungsten nanowire probe (with a 400 nm long tungsten nanowire probe) are able to produce high resolution images that clearly show the individual platinum grains and the small gaps in between the platinum grains [Fig. 2(b)] that are not resolvable with a new standard AFM tip [Fig. 2(a)].

Scans of an evaporated gold surface at a scan size of $300 \text{ nm} \times 300 \text{ nm}$ were carried out using the tungsten nanowire probe continuously for 1 h. The initial AFM image obtained using the tungsten nanowire probe [Fig. 3(a)] is



FIG. 3. AFM tapping mode images of a gold thin film on silicon substrate using a tungsten nanowire probe. (a) Image obtained at the start of one hour of continuous scanning. (b) Image obtained at the end of one hour of continuous scanning. (c) Image obtained on day 4 after storing the tungsten nanowire probe for 3 days. Displayed height range =6 nm. The scan area is $300 \text{ nm} \times 300 \text{ nm}$.



FIG. 4. Single line measurements obtained from AFM tapping mode images of one side of the steep sidewall of the TGZ03 calibration grating using (a) a standard tapping mode AFM tip and (b) a long tungsten nanowire probe. The cantilever is parallel to the grating lines, while the scanning is carried out in a perpendicular direction. The scan length is 2 μ m.

able to produce high-resolution images showing the individual gold grains. After 1 h of continuous scanning, there is no noticeable loss in resolution in the AFM image obtained [Fig. 3(b)] with the individual gold grains still clearly visible. In addition, the same tungsten nanowire AFM tip is kept in an enclosed tip holder in a dry storage cabinet for 3 days before being used again to scan the same test sample. The tungsten nanowire probe is able to reproduce the same high resolution AFM image with no observable changes [Fig. 3(c)]. The results show that the tungsten nanowire probe is robust and is capable of producing consistent high-resolution images over long periods of scanning as well as after storage under proper dry conditions.

Scans of the vertical steep sidewall of a calibration grating consisting of one-dimensional arrays of rectangular silicon dioxide steps on silicon with a step height of about 500 nm (TGZ03 from MikroMasch) using a standard AFM tip and a tungsten nanowire probe (Fig. 4) show that the tungsten nanowire probe is able to profile the vertical sidewall better than the standard pyramidal tapping mode AFM tip. Coupled with the very sharp tip end of the tungsten nanowire, the tungsten nanowire probe will be able to perform much better than the standard tapping mode AFM tip in imaging profiles with deep trenches or holes.

In summary, we have demonstrated a controlled fieldemission induced growth technique in growing a single tungsten nanowire on existing tapping mode AFM tips. With the cantilever clamping arrangement, the nanowire probe can also be grown on low-stiffness cantilevers. The simple and consistent technique produces vertically aligned nanowire AFM probes with good reproducibility. The length of the nanowire is easily controlled by the growth duration and the aligned growth ensures that the tungsten nanowire is grown vertically at the apex of the AFM tip. The very sharp tungsten nanowire probes with a high aspect ratio are able to produce high-resolution tapping mode AFM images, have good stability under prolonged use and better performance in profiling structures with steep sidewalls. The metallic nature of the tungsten nanowire probe has great potential in broader applications such as conducting AFM and I-V measurements. Other metallic nanowire AFM probes, including ferromagnetic probes, can also be fabricated by using other different precursor gases during the field-emission induced growth process. Alternatively, the tip of the nanowire probe can be terminated with a different material by continuing the growth momentarily with a second precursor.

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