Atomic force microscopy of nickel dot arrays with tuning fork and nanotube probe

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The advantages of tuning fork scanning force microscopy are combined with the unique properties of carbon nanotubes to improve the spatial resolution of atomic force microscopy (AFM) images of nickel dot arrays. These arrays have high relief features that prevent the resolution of the actual dot size and shape with a regular cantilever tip. The modification of two key parts of a commercial AFM, the force sensor, and the probe allowed the acquisition of true data on feature size and shape. AFM images of Ni dot arrays were taken using conventional optical detection of a commercial cantilever, tuning fork with attached commercial cantilever tip, and tuning fork with attached multiwalled carbon nanotube. The resolutions of the AFM images were compared, and it was shown that probing with a carbon nanotube provided a 30% improvement of lateral resolution compared to a conventional AFM tip. © *2003 American Vacuum Society.* [DOI: 10.1116/1.1539066]

In atomic force microscopy (AFM), researchers have tried to achieve higher-resolution images by improving force sensor properties and tip characteristics. Optical detection of cantilever deflection is the most common method of force detection employed by commercial instruments.[1] This technique has proved to be very reliable and applicable at ambient conditions for most samples. However, scientists often want to apply AFM techniques in environments or for objects where the currently available possibilities are not enough. Recently, a technique based on the properties of piezoelectric tuning fork quartz oscillators has been used in scanning force microscopy (SFM).[2] This technique exploits the very high mechanical quality factor $Q(10^3-10^5)$ of the tuning fork and offers high sensitivity to pN forces when the sensor is driven at its mechanical resonance frequency.[3–6] In addition, tuning forks are small and can operate in an UHV and at low temperatures with fully electronic control that significantly simplifies the design and technical requirements of the experimental setup.

In all of the applications where tuning forks are used as a force sensor, an appropriate probe is attached to one of the tuning fork prongs. For example, etched wires and the tips of commercial cantilevers have been used as tips on tuning fork transducers. For optimal resolution, the radius of the curvature and aspect ratio are most critical characteristics of the AFM tips. The use of probes with small radii of curvature and large aspect ratios provides the best spatial resolution. Commercially available Si or Si_3N_4 tips of a pyramidal or cone shaped have a radii of curvature of about 20 nm, and aspect ratios of 3:1. These characteristics however, might not be good enough in the case of samples with high relief, when the tip cannot probe the area between high individual features. The shape and size of the imaged features can reflect the tip profile rather than the features themselves. To enable a precise measurement of the force between the tip and a deep valley in the sample, we need a tip that is both extremely sharp and long. The first candidate for such a tip is a carbon nanotube, in particular, a single-walled carbon nanotube. The fabrication and advantages of using nanotube tips in different SFM applications are well described.[7,8] In the present article, we report on AFM studies of Ni dot arrays, and compare resolutions of AFM images taken with regular cantilever tips, and tips consisting of multiwalled carbon nanotubes.

In our experiments, we used a ThermoMicroscopes Explorer AFM equipped with a 2 μm tube scanner that allows one to acquire topography images with minimum distortion. The detection method was based on either laser beam deflection from the back of a commercial AFM cantilever, or a home-made tuning fork detector interfaced to the electronics and software of the Explorer; the details of the latter technique have already been described elsewhere.[9] For the tuning fork transducers, we employed commercial tuning forks[10] with a resonance frequency of 2^{15} Hz, and a spring constant of 3700 N/m. An ultrasharp cantilever tip from commercially available chips[11] was glued to the end of the tuning fork face surface with cyanoacrylate glue.[9] The silicon tip had a radius of curvature of less than 10 nm, a cone angle of 20°, and a height of 20 μm. The small size of the cantilever tip minimized the modification of the tuning fork characteristics.[9] For the nanotips, a multiwalled carbon nanotube was mounted on the end of a commercial cantilever tip by

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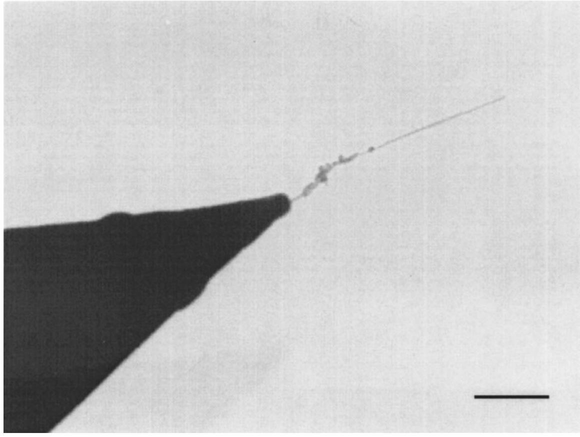

FIG. 1. TEM image of carbon nanotube attached to the end of ultrasharp commercial Si tip. The size bar corresponds to 1 μm.

electron-beam welding, using a special three-dimensional nanomanipulator within a transmission electron microscope (TEM) chamber. The process will be described in detail elsewhere.[12] Figure 1 shows a scanning electron micrograph of the resulting commercial cantilever with a mounted carbon nanotube.

The samples that were imaged consisted of arrays of Ni dots of different diameters and interparticle spacing. These were fabricated by electron-beam lithography for ferromagnetic resonance studies.[13] The results discussed next were obtained from a sample having the following parameters: dot diameter 45 nm, dot height 60 nm, and spacing between dots 150 nm. Figure 2 shows a scanning electron microscope (SEM) image of the pattern and a sketch of the structure profile. For samples with such high relief, it is difficult to distinguish individual features using commercial cantilever tips, as the characteristics of the tip shape are convoluted with the sample features. As an example of the strong effect that the tip shape has on topography data, we show in Fig. 3(a) an AFM image of a part of the array shown in Fig. 2, taken by conventional optical detection techniques in noncontact AFM mode. The profile and dimensions of each dot clearly represent the pyramidal shape of the probe, which was a three-face pyramid-shaped Si tip with the radius of its apex about 25 nm.

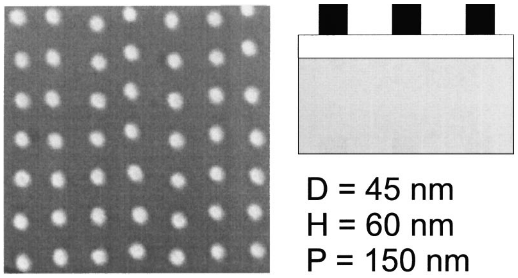

FIG. 2. SEM image of Ni dot array and sketch of feature geometrical characteristics. *D* is the diameter of the particles, *H* is the height, and *P* is the spacing between particles.

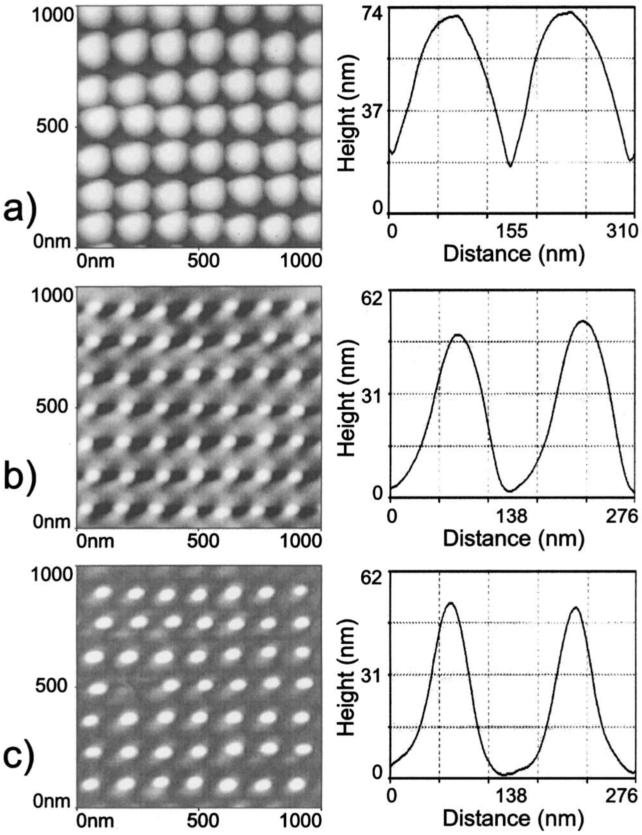

FIG. 3. Topography images of the dot arrays and cross sections of the features observed in AFM studies by using (a) conventional optical detection technique and pyramid-shaped tip, (b) tuning fork with attached commercial ultrasharp AFM cantilever, and (c) tuning fork with attached carbon nanotube.

There are two main factors that restrict the resolution of AFM systems in noncontact operation mode: The characteristics of the tip itself, and the tip–sample interaction. The effect of the tip profile has just been discussed. With regard to the tip–sample interaction, commercial cantilevers have relatively small spring constants (42 N/m), which restrict the minimum distance above the sample at which they can be operated. At smaller distances, the strong tip–surface interaction may pull the tip into the surface. To overcome the latter problem, we used a high spring-constant tuning fork as the detection mechanism. A larger spring constant allows operation under stronger tip–sample interaction forces without the possibility of the tip crashing into the surface due to short-range attractive van der Waal's interactions. This means one can drive the probe very close to the sample surface, improving the resolution of the image. Figure 3(b), which is an image taken with a tuning fork with a commercial cantilever tip, illustrates the higher resolution that can be achieved with a tuning fork. The spatial resolution of the features achieved with the tuning fork is similar to the SEM data. However, as can be seen from the image cross section in Fig. 3, the lateral resolution of the shape and dimensions of the dot is somewhat different compared to the lithography pattern. The ability to distinguish the shape and size of the dots is limited by the probe characteristics. In the noncontact mode of tuning fork operation, attractive van der Waal's forces are detected, as the probe gets closer to the sample surface. The resulting force is a sum of forces affecting the tip in the normal and lateral directions. The lateral force is associated with interaction between the tip and the side of each dot. In the case of structures with high relief, the lateral forces can be sufficient to damp the piezoelectric signal in normal mode. In order to overcome this problem, one can employ a narrow tip with a very high aspect ratio.

Toward this end, a multiwalled carbon nanotube of 5 nm in diameter was mounted to the end of a commercial tip and used in the same way as a regular cantilever tip with a tuning fork. The result of AFM scanning with this carbon nanotube is shown in Fig. 3(c).[14] On the basis of the obtained images, x, y, and z resolutions were found in each case. An analysis of the cross section shows that dot diameters estimated from AFM imaging using a tuning fork with a commercial cantilever tip attached and a tuning fork with a carbon nanotube attached are 65.1 ± 3.17 nm and 48.2 ± 2.25 nm, respectively, based on seven measures of the width at half of the full height of the dot. The latter result is in good agreement with the SEM data. Comparing the results, we can conclude that imaging with a carbon nanotube provides a 26% improvement in spatial resolution with respect to the results obtained with a conventional cantilever tip.

We have not discussed the influence of scan speed and response time on the AFM image resolution. A fast response of the feedback control system is essential to obtain high-resolution images. However, we found that conventional proportional (P) and integral (I) feedback control is sufficient for most uses of tuning forks to provide stable control during the measurements. In our experiments, the control system was optimized by relative magnitudes of P, I, and scan rates that were 1, 0.02, and 15 lines per minute, respectively.

In conclusion, a combination of a tuning fork and a carbon nanotube mounted to the end of commercial cantilever tip provides 26% better resolution compared to regular commercial cantilevers. We show that the use of a tuning fork and carbon nanotube opens new possibilities in the study of objects in different environments.

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[14] The image was obtained using the following parameters: tuning fork drive voltage V 0.02 V. The set point was equal to 25% of the current generated by the freely oscillating prongs (120 nA). The scanning rate was 15 lines per minute. The corresponding mechanical amplitude of prong oscillations was 15 nm calculated according to R. D. Grober, J. Acimovic, J. Schuck, D. Hessman, P. J. Kindlemann, J. Hespanha, A. S. Morse, K. Karrai, I. Tiemann, and S. Manus, Rev. Sci. Instrum. **71**, 2776 (2000).