

Fast, high-resolution atomic force microscopy using a quartz tuning fork as actuator and sensor

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We report a new method of achieving tip-sample distance regulation in an atomic force microscope (AFM). A piezoelectric quartz tuning fork serves as both actuator and sensor of tip-sample interactions, allowing tip-sample distance regulation without the use of a diode laser or dither piezo. Such a tuning fork has a high spring constant so a dither amplitude of only 0.1 nm may be used to perform AFM measurements. Tuning-fork feedback is shown to operate at a noise level as low as that of a cantilever-based AFM. Using phase-locked-loop control to track excursions in the resonant frequency of a 32 kHz tuning fork, images are acquired at scan rates which are fast enough for routine AFM measurements. Magnetic force microscopy using tuning-fork feedback is demonstrated. © 1997 American Institute of Physics. [S0021-8979(97)03615-3]

I. INTRODUCTION

Commercially available atomic force microscopes (AFM) presently employ a diode laser whose beam is reflected from the back of a micromachined Si cantilever to monitor the tip-sample interaction. Unfortunately, the use of a diode laser in AFM introduces several problems. First, for many types of measurement, illumination from the diode laser is a detriment. Second, the diode laser introduces drift into the measurement from sources such as thermal mode hopping and can cause an apparent noise of several nm in AFM images,¹ orders of magnitude greater than the nominal noise floor. Finally, interaction of the diode-laser illumination and the sample topography causes spurious modifications to the measured tip-sample interaction, causing imaging artifacts and preventing the reliable imaging of low-amplitude corrugations near tall steps.

In alternating-current (AC) AFM-related techniques, the cantilever is driven at or near its resonant frequency. Due to the low spring constant of Si cantilevers, AC AFM-related techniques require a large oscillation amplitude, typically tens to hundreds of nm. For instance, magnetic force microscopy (MFM) relies upon a micromachined Si cantilever coated with a magnetic thin film. Due to the large oscillation amplitude, the magnetic field is averaged over a large volume when performing MFM. At best, this distorts the measured profile; at worst, it obscures structure. Similar arguments can be made for other AC AFM-related techniques such as Kelvin-probe force microscopy. A small oscillation amplitude should improve the spatial resolution obtainable with AC AFM-related techniques.

Workers in near-field scanning optical microscopy (NSOM) have explored nonoptical means of sensing the tip-sample interaction because stray illumination is a major detriment to NSOM measurements.²⁻⁵ Most schemes rely upon a miniature oscillator whose mechanical properties such as

resonant frequency and Q depend upon the tip-sample interactions.²⁻⁴ In these studies, the probe tip oscillates parallel to the sample surface, in a shearing motion. For instance, several workers^{3,4} attach the probe tip to a quartz tuning fork in which the tines are oriented perpendicular to the sample surface. However, most high-resolution AFM work uses AC techniques in which the tip oscillates perpendicularly to the surface in order to minimize damage to the probe tip,⁶ as in the well-known TappingMode.⁷

In this article, we describe an implementation of a tuning-fork sensor suitable for high-resolution AFM imaging.⁸ As shown in Fig. 1, we mount the AFM tip perpendicular to the tuning-fork tine so that the tip oscillates normally to the sample surface, minimizing tip wear. Furthermore, the high spring constant of the tuning-fork tines allows the use of small oscillation amplitudes as small 0.1 nm, offering potentially improved spatial resolution for AC AFM-related techniques such as MFM.

Due to the high mechanical Q of quartz tuning forks, there is a delay of tens to hundreds of ms before the tuning-fork oscillations reach their steady-state condition. As a result, traditional, "open-loop," methods of AC AFM⁶ are not appropriate for tuning-fork feedback, since an AFM could not track a surface at a scan rate faster than 0.1 Hz with a 100 ms lag in its sensor. We use a phase-locked-loop (PLL) circuit to overcome this limitation by actively tracking the tuning-fork resonant frequency. With PLL control of a tuning fork, we demonstrate for the first time that high-resolution AFM imaging may be performed at a rate of 1 Hz, a scanning rate comparable to those employed for cantilever-based AFM scanning.

PLL tracking of a resonant frequency has been used to perform cantilever-based MFM in vacuum⁹ and to perform NSOM with a tuning-fork sensor.⁴ The latter demonstrated a noise level of under 3 nm, much higher than the vibrational noise floor for most cantilever-based AFM instruments, which is typically under 0.05 nm. In this article, we present

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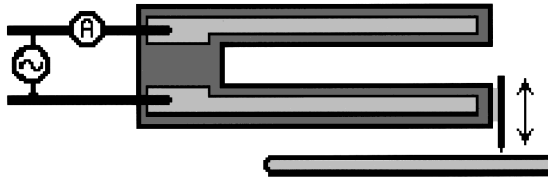


FIG. 1. Configuration of the piezoelectric quartz tuning-fork sensor/actuator. A sharpened 0.001 in. wire is fixed to one tine of a quartz tuning fork. In this configuration, the probe tip oscillates perpendicular to the sample surface. The motion is actuated by driving one electrode of the tuning fork by an AC voltage near the tuning-fork resonant frequency. The tuning-fork oscillations are sensed by monitoring the current from the other electrode.

the first demonstration of AFM imaging using a tuning-fork sensor in which the noise level is as low as in cantilever-based AFM. Finally, we demonstrate MFM using a tuning-fork sensor for the first time.

II. AC DETECTION IN ATOMIC-FORCE MICROSCOPY

AC AFM imaging was developed to reduce destructive interactions between tip and sample.^{6,7} The cantilever is mechanically driven near its resonant frequency so that the tip oscillates vertically, moving toward and away from the sample. The oscillation amplitude of the cantilever is monitored by lockin detection, and its value is maintained constant by the feedback loop controlling the Z piezo in order to track the sample surface.

In AC AFM imaging, the amplitude or phase of cantilever oscillations is monitored and used by the AFM control electronics as a measure of tip-sample interactions, allowing tip-sample distance regulation. We refer to this mode of sensing cantilever oscillations as “open-loop” control.

If the cantilever employed has a high mechanical Q , the mechanical settling time $t_{\text{ring}} = 2Q/\omega_0$ (Ref. 9) is too long for open-loop detection. This is the case for a tuning fork, for which Q can attain values of several thousand and the resonant frequency is 32 kHz, so that t_{ring} can be tens to hundreds of ms, whereas typical AFM scan rates require a settling time of a few ms or less. Thus, it is useful to consider control methods which do not rely upon the long-time behavior of a mechanical oscillator.

If a force gradient dF/dz were applied suddenly to the tip by the sample, the effect would be the same as if the cantilever’s spring constant were modified to $k \rightarrow k + dF/dz$ (Ref. 10). This causes a shift in the cantilever resonant frequency since $\omega_0 = \sqrt{k/m}$. The time lag between this shift and the application of the force gradient is the time it takes sound to traverse the cantilever, about $t_{\text{sound}} \approx 1/\omega_0$. Because the cantilever equation of motion depends upon ω_0 , the cantilever response to the drive signal begins to change after a time t_{sound} , even though its behavior will not match the long-time forms in Ref. 9 until a time t_{ring} has passed. In other words, while it takes t_{ring} for the amplitude and phase relative to the drive signal of a cantilever to settle into its steady-state condition, the cantilever begins to respond to a change in tip-sample force gradient a factor of $2Q$ faster.

Now suppose that, instead of maintaining a fixed value, the drive frequency can be changed in response to the varia-

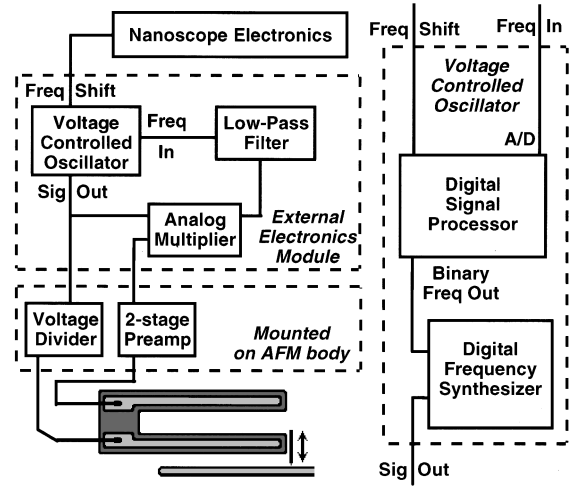


FIG. 2. Schematic of PLL used to track the tuning-fork resonant frequency. Left: VCO drives one electrode of the tuning fork to excite resonance. Current from other electrode is monitored to sense mechanical response of tuning fork. Analog multiplier and low-pass filter detect the phase difference between drive and sense signals, which in turn controls VCO frequency. Right: VCO comprises a DSP chip controlling the frequency output of a digital frequency synthesizer.

tions in the cantilever’s response to the drive signal. This type of control scheme is referred to as “closed loop,” since a feedback loop has been closed between the drive frequency and the detected cantilever response. The most straightforward quantity to track in this closed loop is the phase difference between the drive signal and the response, which can be measured using a phase-sensitive detector (PSD). When the phase difference between drive and response is held constant in a closed loop by servoing the frequency, a PLL is formed.

In practice, PLL control of a mechanical oscillator may be performed in several ways. One is to use the mechanical oscillator as the frequency-determining element of an oscillatory electrical circuit.⁹ FM-demodulation techniques may be applied to extract the variations of this circuit’s oscillation frequency with time.⁹ This approach also has been applied to tuning-fork feedback in NSOM.⁴

Our approach is to use a separate electrical oscillator, a digital frequency synthesizer, whose frequency is actively controlled by a digital signal processor (DSP) in order to track the resonant frequency of the tuning fork. As shown in Fig. 2, the DSP monitors the phase shift between the drive signal and the tuning-fork response through the “Freq In” input, and servoes the applied drive frequency (“Binary Freq Out”) in order to maintain this phase shift at a fixed value. This approach ensures the synchronization of the digital frequency synthesizer with the tuning fork. The use of a DSP allows us to record quantitatively the actual frequency excursions undergone by the tuning fork in response to applied force gradients. It also allows sophisticated control schemes to be employed in the frequency tracking PLL by software.

III. PREPARATION OF TUNING-FORK TIPS

We will next describe the techniques we use to produce sharp tips on tuning forks, with a light-weight electrical lead to the tip. Tips are electrochemically sharpened by immer-

sion in a meniscus of etchant suspended on a small loop of wire which serves as a counter-electrode. We have found it most convenient (and controllable) to clamp the tip and move it with the X-Y stage of the microscope, and to move the wire loop using a single-joystick micromanipulator. Similarly, in the mounting of tips on tuning forks, we have evolved techniques, and this evolution has been driven, not by simplicity but by convenience considerations. It is clearly possible to do the entire mounting process by hand, but it is much more convenient to use various tools (available from Custom Probes Unlimited, Gaithersburg, MD).

We have thus far been working with 25 μm diam W wire, and the methods are suitable for wires of other materials as well. First, we cut a length of wire and put a right-angle bend in it such that one section is about 1 cm long, to later function as the light-weight electrical lead, and the other section is about 5–8 mm long. We will refer to these as the lead and the tip sections. Next, we grasp and clamp the wire, using mechanically assisted tweezers, somewhere in the lead section, such that the tip section is forward of the tweezers tips.

The tip section is then reduced in length and sharpened. This is accomplished by mounting the tip-in-tweezers on a manipulator and positioning it under an optical microscope, at 75 \times , such that the tip section is clearly visible. Then the wire loop, containing some electrolyte (KOH in the present case), is moved, using another micromanipulator, so that the tip section goes through the liquid, and the tip section is reduced to a length of about 1 mm, and sharpened. Next, the tip is inspected at 600 \times magnification, and electropolished further, if needed, to produce a very sharp tip.¹¹ Finally, while still in the tweezers, the tip is rinsed in water.

A small dab of epoxy is then applied near the end of one of the tuning fork tines, observing the process under a 3 \times binocular microscope. We position the tuning fork, held in the output arm of a one-dimensional micromanipulator, or other holding device, in the field of view, and then add the epoxy¹² to the tine assisted by a single-joystick micromanipulator.

Finally, still using the binocular microscope, the tweezers with tip is attached to the output arm of the three-dimensional manipulator, and the tip is gently put into the epoxy on the tine, so that the tip section is perpendicular, coplanar with the tines and protruding about 0.3–0.5 mm from the outer edge of the tine to which it is attached. The entire tip wire is parallel with the tines at this point. Then the tip wire is released from the tweezers and the tweezers is removed from the scene. Gravity causes the tip wire to move such that the lead section drops to a stable vertical position hanging between the tines.

IV. OPEN-LOOP TUNING-FORK IMAGING

Our first experiments with tuning-fork feedback used open-loop detection of tuning-fork oscillations as shown in Fig. 1. A signal generator drives one electrode of the tuning fork on resonance (32.768 kHz) with a small amplitude, typically around 1 mV. According to Ref. 3, where similar tuning forks were used, this results in an oscillation amplitude of around 0.1 nm. The tuning-fork oscillation amplitude is

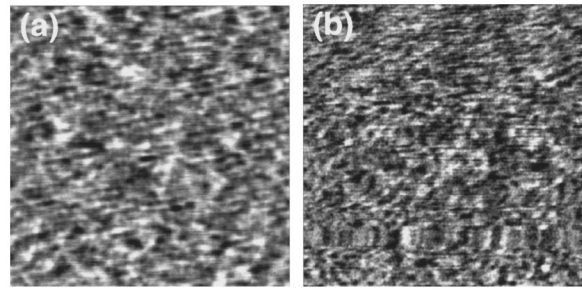


FIG. 3. ($1\ \mu\text{m}$)² images of a polished, flat Si wafer. (a) Typical image taking using cantilever-based AFM with a 1 Hz scan rate. (b) Image acquired using an open-loop controlled tuning-fork sensor with a 0.05 Hz scan rate. Root mean square roughness of both images is 0.1 nm, typical of polished Si wafers. Textured appearance is also typical of polished Si wafers, indicating a noise floor in each case of under 0.05 nm.

monitored by measuring the current flowing through the other electrode. Using a lockin amplifier to monitor this current component and feeding it back into the AFM-control electronics, we obtained the image in Fig. 3(b). The sample is a flat, polished Si wafer.

The surface of a polished Si wafer is covered by a native oxide with a shallow but distinct texture as displayed in a TappingMode image.⁷ The typical lateral scale of this texture is a few tens of nm, and the rms roughness of AFM images of a polished Si wafer is generally around 0.1 nm. The image shown in Fig. 3(a) is a typical TappingMode image of such a sample, with these attributes. Figure 3(b) shows these attributes as well, but was obtained with open-loop controlled tuning-fork feedback.

Polished Si wafers serve as a useful benchmark for measuring the noise floor of AFM systems. If an AFM system can measure the 0.1 nm roughness of a polished Si wafer, it will easily measure the 0.3 nm steps on the surface of an epitaxial Si film, for instance. Thus, the successful imaging of a polished Si wafer using tuning-fork feedback indicates that tuning-fork feedback does not hurt the noise floor of a typical AFM. Specifically, the apparent rms noise in the image must be under 0.05 nm to view the surface texture of the polished Si wafer surface. This is a noise floor nearly two orders of magnitude lower than was demonstrated for the shear-mode NSOM described in Refs. 3 and 4.

Although our initial open-loop controlled tuning-fork experiments showed promise due to their low noise level, the imaging speed was very slow. The TappingMode image in Fig. 3(a) was acquired with a 1 Hz scan rate; the 512 lines were acquired in under 10 min. The image in Fig. 3(b) was acquired at a 0.05 Hz scan rate—its acquisition took an entire afternoon. It is clear that open-loop control is not appropriate for tuning-fork feedback.

V. DESIGN OF PLL CONTROL ELECTRONICS

To overcome the speed limitation imposed by open-loop control, we developed a DSP-based PLL control circuit to track the resonant frequency of our tuning-fork tips. The design of PLL has been treated in detail elsewhere,¹³ we will

simply point out the unique features of our design as well as the differences between our application and more traditional PLL applications.

A PLL comprises a voltage-controlled oscillator (VCO), a PSD, and a loop filter.¹³ A VCO produces a repetitive output waveform at a frequency which is controlled by the DC level of an input signal. In our case, as shown in Fig. 2, the VCO includes a DSP module which supplies a 32-bit frequency word to a digital frequency synthesizer. An analog input on the DSP samples the output of the PSD; to maintain a fixed input level, the DSP program raises or lowers the frequency it provides to the digital frequency synthesizer. The DSP also uses one of its analog outputs to produce a voltage (“Freq Shift”) proportional to the excursions of the drive frequency from the tuning-fork free-oscillation resonant frequency (as measured with the tip far from the sample). Since a PLL is formed, this drive frequency tracks the resonant frequency of the tuning fork. As discussed above, these frequency excursions are thus proportional to the force gradient between tip and sample. This frequency-excursion output is fed into the Nanoscope⁷ electronics for use in tip-sample distance regulation, or for the MFM signal as discussed below.

For PSD, we used an analog multiplier. If the drive signal is given by $A_{\text{drive}} \sin(\omega t)$ and the tuning-fork response by $A_{\text{response}} \sin(\omega t + \varphi)$, then their product is

$$\begin{aligned} & A_{\text{drive}} \sin \omega t A_{\text{response}} \sin(\omega t + \varphi) \\ &= A_{\text{drive}} A_{\text{response}} (\sin^2 \omega t \cos \varphi + 1/2 \sin 2\omega t \sin \varphi) \\ &= 1/2 A_{\text{drive}} A_{\text{response}} \cos \varphi, \end{aligned}$$

where we arrive at the second equality after averaging over several periods of the drive signal (as happens with the loop filter, below), so that the second term cancels and the time dependence of the fit term gives a factor of 1/2. However, even with several periods of averaging, the second term is not entirely eliminated, so for loop filter, we used a 6 kHz, 8-pole low-pass filter. This filter eliminates the frequency-doubled component generated in the analog multiplier, so that the frequency input to the DSP sees only a DC signal related to the phase difference between drive and response.

As shown on the left side of Fig. 2, a small signal-conditioning module mounted on the AFM body includes a voltage divider for the drive signal and a current-to-voltage preamplifier to sense the tuning-fork response. The voltage divider takes the 1 V amplitude drive signal from the external electronics module and scales it to 1 mV, ensuring a 0.1 nm oscillation amplitude as discussed in Ref. 3. The current preamplifier takes the nA-range tuning-fork response and amplifies it to the volt range to improve the signal-to-noise ratio of the PSD.

Not shown in Fig. 2 is the rms-to-DC converter which provides the DSP with the amplitude of the tuning-fork oscillation. This signal is used in the initial frequency sweep which the DSP performs to find the resonance frequency of the tuning fork.

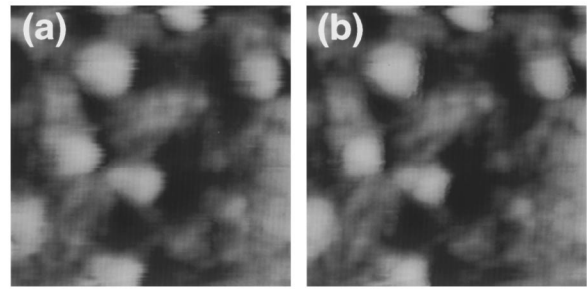


FIG. 4. Fast ($1 \mu\text{m}$)² images obtained using PLL-controlled tuning fork sensor. (a) Trace and (b) retrace images match in detail. Scan rate is 1 Hz and vertical scale is 25 nm.

VI. PLL CONTROLLED TUNING-FORK IMAGING

Tip-sample distance regulation using tuning-fork feedback is accomplished as follows. The tuning-fork resonant frequency is initially determined by a frequency sweep taken with the tip far from the sample. As shown in Fig. 2, the DSP outputs an analog signal (“Freq Shift”) proportional to the difference between the tuning-fork resonant frequency and this free-oscillation frequency. A variable DC offset is subtracted from this signal using a variable voltage supply and a differential amplifier. The resulting analog signal is fed into the feedback-signal input in the AFM-control electronics. The Nanoscope control electronics⁷ for our Digital Instruments Dimension 3000 AFM system⁷ serves the Z piezo drive to minimize this analog signal. In other words, the Nanoscope⁷ electronics pushes the tuning-fork tip towards the sample until the frequency-shift signal equals the voltage setpoint added by the variable voltage supply. We typically used a few tenths of a volt, which corresponds in our apparatus to a frequency shift of a few tenths of a Hz, a frequency variation of one part in 10^5 .

In this configuration, our PLL tuning-fork control circuit performs constant-frequency-shift tip-sample distance regulation. Since a force gradient causes a frequency shift in the tuning fork as discussed above, our PLL control circuit really allows us to perform constant-force-gradient AFM imaging. Open-loop control, on the other hand, mixes information about energy dissipation (through changes in Q) in the tip-sample junction with force-gradient information (which causes the position on the resonance curve to change).

The first test of our PLL-controlled tuning-fork sensor was to attempt fast scanning. The results of this test are shown in Fig. 4, where the sample is a video tape. Figures 4(a) and 4(b) show the images obtained on the trace and retrace of the fast scan; the topography matches in detail, indicating a good tip-sample tracking. This image was acquired at a scan rate of 1 Hz, which is comparable to those used in cantilever-based AFM measurements. The feedback loop was able to track a heavily corrugated surface.

Furthermore, a video tape sample is fairly soft; for instance, contact-mode measurement of this sample would create damage visible in the features of the image. This indi-

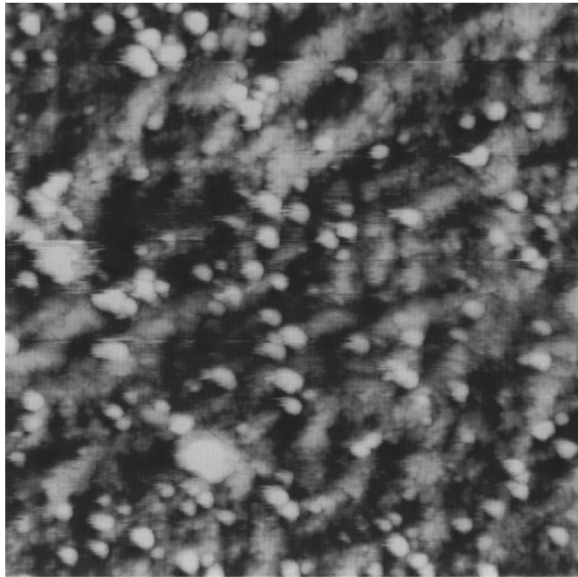


FIG. 5. $(5\ \mu\text{m})^2$ image acquired at 0.5 Hz scan rate, showing very detailed structure. Vertical scale is 25 nm.

icates that tuning-fork feedback is a nondestructive imaging technique for soft samples, consistent with the arguments of Karrai and Grober³ that the force–gradient sensitivity of a tuning-fork sensor is comparable to that of a Si cantilever used for TappingMode.⁷ Figure 5 shows a larger scan of the same video tape. Note the large amount of detail visible in Fig. 5, obtained at a scan rate of 0.5 Hz. These images show that PLL control improves the imaging speed by an order of magnitude over open-loop control while maintaining the ability to track a heavily-corrugated surface.

VII. MFM WITH PLL-CONTROLLED TUNING-FORK FEEDBACK

The above discussion indicates that PLL control of a tuning fork measures excursions in its resonant frequency. This is the basis for a popular method of performing MFM,⁹ in which the magnetic field due to the sample exerts a force gradient on a magnetic tip, causing a shift in the cantilever resonant frequency. To perform MFM, we use LiftMode,⁷ in which an “interlace” scan is added between

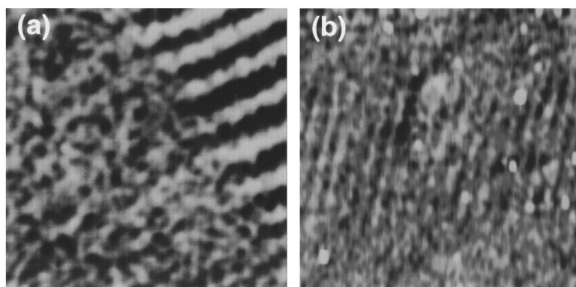


FIG. 6. Magnetic force microscopy images of a video tape. (a) Typical $(5\ \mu\text{m})^2$ MFM image of video tape using a commercially available MFM tip. (b) $(10\ \mu\text{m})^2$ MFM image of a video tape acquired using a PLL-controlled tuning-fork sensor. The frequency-shift scale in (b) is a few hundredths of a Hz, or a variation in resonant frequency of one part in 10^6 .

regular scans. On each interlace scan, the tip is lifted and traced over the contour found on the previous scan. FM detection is used during the interleave scans to track shifts in the cantilever resonant frequency due to the nondissipative magnetic force. Figure 6(a) is a typical MFM image of a video tape sample obtained using a commercially available MFM cantilever.⁷ The stripes running across the top-right corner of the image are due to the magnetization of the video tape.

Next, we tried MFM imaging using tuning-fork feedback. The Nanoscope electronics monitored the frequency-shift error signal “Freq Shift” from our PLL circuit, as shown in Fig. 2. Figure 6(b) shows the first MFM image obtained using a tuning-fork sensor. The magnetic tip in this case is a piece of 0.002 in. diam Fe wire. The tip was formed by cutting the wire with a blade. No special steps were taken to magnetize the Fe wire, so its magnetization is expected to be quite weak.

The vertical stripes running in a nearly horizontal band across the middle of the image in Fig. 6(b) are the tracks of the video tape. The spacing of these tracks is identical to that of Fig. 6(a), although they appear half as big due to the larger scan size.

VIII. CONCLUSIONS

We have used a quartz tuning fork to demonstrate non-optical monitoring of AFM tip–sample interactions for a geometry in which the tip oscillates perpendicularly to the sample, similar to ordinary AC-mode AFM imaging. Using open-loop detection to track the tuning-fork oscillation amplitude, we were able to obtain images of a polished Si wafer with 0.1 nm roughness, indicating that AFM with a tuning-fork sensor can attain a noise floor comparable to cantilever-based AFM instruments. Using a novel form of PLL detection to maintain a constant resonant–frequency shift in the tuning fork, we demonstrated that PLL tuning-fork feedback can track sample topography with a scan rate comparable to that used in TappingMode. Finally, we demonstrated magnetic-force imaging of a video tape sample for the first time using a tuning-fork sensor.

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⁷TappingMode, TESP, NanoScope, and LiftMode are trademarks of Digital Instruments, Inc., Santa Barbara, CA.

⁸The results contained in this article were previously presented by Hal Edwards and Walter Duncan in an oral presentation in session NS-MoM at American Vacuum Society National Symposium, Philadelphia, PA, October 14, 1996.

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