Variable temperature magnetic force microscopy with piezoelectric quartz tuning forks as probes optimized using *Q*-control

F. D. Callaghan,[a] X. Yu,[b] and C. J. Mellor
School of Physics and Astronomy, University of Nottingham, University Park, Nottingham NG7 2RD, United Kingdom

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We have performed magnetic force microscopy at various temperatures utilizing piezoelectric quartz tuning forks as probes. Due to their large force constants ($\sim 10^4$ N/m), quartz tuning forks are intrinsically less sensitive to force gradients than conventional cantilevers. However, we demonstrate that the technique of *Q*-control can be used to increase their sensitivity, making their use as probes for variable temperature magnetic force microscopy a viable option. © *2005 American Institute of Physics*. [DOI: 10.1063/1.2132525]

We report the use of piezoelectric quartz tuning forks as probes for variable temperature magnetic force microscopy (MFM). In particular, the effect of probe quality factor on the sensitivity to magnetic force gradients is discussed. Low temperature MFM with both conventional[1–3] and piezoresistive[4] cantilevers is now an established technique. Room temperature MFM with tuning fork probes has also been reported.[5–7] Extension of this technique to low temperatures was recently reported,[8] and is a natural progression due to the intrinsic advantages that tuning forks provide. Their self-sensing capability removes the need for optics inside the cryostat, and problems associated with misalignment of components on cooling down are eliminated.

Our microscope is based on a commercial instrument,[9] modified to incorporate tuning fork sensors. To enable detection of magnetic force gradients, a conventional MFM cantilever[10] is attached, using epoxy adhesive, to the end of one tine of the fork. The fork is driven with a sinusoidal voltage and the resulting piezoelectric current (which is proportional to the fork velocity, and therefore to the fork amplitude if driven at constant frequency) is measured. The MFM cantilever oscillates perpendicular to the surface of the sample and so the measurements reported here are more sensitive to the perpendicular component of the stray field than the in-plane component. The other tine is immobilized for reasons previously discussed.[11] The tines of the quartz tuning forks used in this work were 3.1 mm long, 0.24 mm wide, and 0.38 mm thick. The resulting force constant c and resonant frequency f_0 are ~ 8.7 kN/m and 32 kHz, respectively. Calculations predict the response of such forks to be ~ 1.4 nA/nm.[12]

Due to their large force constants, c, tuning forks are intrinsically less sensitive to force gradients than conventional cantilevers. This can be seen from the expression

$$\frac{\delta f}{f_0} \cong -\frac{1}{2c}\frac{\partial F}{\partial z}, \tag{1}$$

where δf is the shift in resonant frequency f_0 of a probe with force constant c in the presence of a tip-sample force gradient $\partial F/\partial z$, where z is the coordinate in the direction perpendicular to the sample surface. Typical force gradients detected by MFM are in the range 10^{-4}–10^{-2} N/m.[13] For a typical tuning fork, the corresponding frequency shifts are 0.2–20 mHz. This small frequency shift is difficult to measure. By increasing the effective quality factor Q of the tuning fork, a given frequency shift corresponds to a larger phase shift so decreasing the smallest detectable force gradient. It should be emphasized that Eq. (1) is an approximation and is valid only when the probe oscillation amplitude is small compared to the tip-sample distance. An accurate analytical expression relating force to frequency shift, which is valid for arbitrary oscillation amplitudes, has recently been proposed.[14]

The intrinsic lack of sensitivity of tuning forks has motivated us to boost the quality factors of our probes using the technique of *Q*-control. It is well known that for a system at the thermal noise limit the minimum detectable force gradient is proportional to $1/\sqrt{Q}$.[15] Grober *et al.* have derived an expression for the signal-to-noise ratio (SNR) obtainable with quartz tuning forks.[16] In particular, they show that an increased quality factor leads to an increased SNR. The exact result depends on whether the thermal noise or the electronic noise from the feedback resistor in the amplifier is dominant. In our case the dominant noise is of a nonthermal origin, but we show that we can still benefit from effective quality factors increased using *Q*-control. Briefly, *Q*-control allows one to electronically increase or decrease the probe quality factor. The technique has recently been treated theoretically.[17,18] An increase in effective quality factor can result in greater sensitivity to tip-sample interactions and has been used to reduce the tip-sample interaction force while imaging soft (biological) samples, thereby improving image resolution.[19] A decrease in effective quality factor, on the other hand, may be used to increase scanning speed by reducing the mechanical settling time of the cantilever.[20] In a previous letter[11] we demonstrated the ability to vary the quality factors of tuning forks over a range of two orders of magnitude. MFM images acquired with a range of probe quality factors are presented here.

Even without *Q*-control, the quality factors of tuning forks (10^3–10^5) are intrinsically greater than those of conventional cantilevers (10–100). However, the difference is not great enough to compensate for the lack of sensitivity

[a]Present address: Department of Physics, Simon Fraser University, Burnaby BC V5A 1S6, Canada; electronic mail: fcallagh@sfu.ca
[b]Present address: Department of Physics, Fudan University, 220 Handan Road, Shanghai, China 200433.

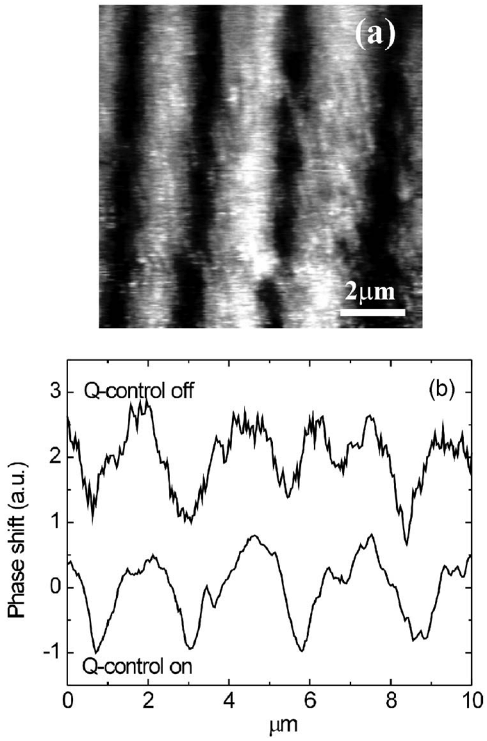

FIG. 1. (a) 10 μm $\times$ 10 μm MFM image of magnetic recording disk acquired in air at room temperature using Q-control (Q=9200). (b) Cross sections of MFM images acquired with Q-control off (Q=1980, upper trace) and with Q-control on (Q=9200, lower trace).

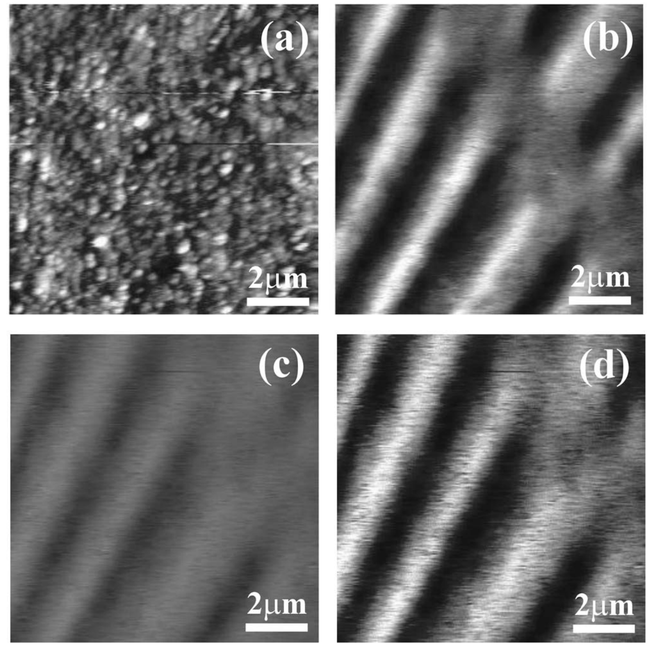

FIG. 2. (a) 10 μm $\times$ 10 μm topographic image of magnetic recording disk acquired in vacuum at T=136 K(Q=8400). (b) MFM image acquired during acquisition of topographic image in Fig. 2(a). (c) MFM image of same area with Q=1800. (d) As Fig. 2(c), but with z-scale expanded to emphasize decreased SNR due to lower Q.

due to increased stiffness. A disadvantage of large quality factors is that slow scanning is necessitated as the mechanical settling time of the probe is given by $\tau \sim Q/\omega_0$. For a typical tuning fork in air, at room temperature with one time immobilized, we find that Q is approximately 2000 and f_0=32 kHz; this gives a mechanical settling time of 10 ms, which would require a scan speed of around 20 ms/pixel (i.e., ~5 s/line) for optimum image resolution. To overcome this problem it is common practice to implement a phase-locked loop to actively track the probe resonant frequency while scanning.[5] However, the frequency shifts due to the magnetic force gradients are so small that we have obtained better performance by driving the fork at a constant frequency and measuring changes in the phase of the fork response. Although force gradient-induced shifts in f_0 are Q independent, for small shifts δf in resonant frequency, the associated phase shift $\delta\varphi$ increases with Q. This therefore enables increased sensitivity with standard phase detection in conjunction with Q-control.

For the acquisition of topographic data, a feedback loop is used to keep the amplitude of oscillation constant as the tip is scanned over the sample surface. The amplitude set point is typically 95% of the free amplitude. After acquiring the topographic line profile, magnetic force gradients are detected by retracing the topography at a fixed height above the surface and measuring phase shifts in the tuning fork response. The lift height is typically in the range 50–150 nm, which is greater than that usually employed in conventional MFM. The width of the forks is much greater than that of conventional cantilevers and leads to increased hydrodynamic "squeeze damping" between fork and sample[21–23] and also to changes in the effective probe mass.[11,22] This is due to increasing confinement of the fluid medium between fork and sample and leads to increased topographic contributions to the MFM image at lift heights less than 100 nm. This problem could be avoided by operating in vacuum or by employing a longer tip (e.g., an etched wire of length several hundred microns). Measurements reported below were all made on the same instrument. Room temperature measurements were made in ambient conditions. Low temperature measurements were performed in a few mbar of helium exchange gas.

Figure 1(a) shows a 10 μm $\times$ 10 μm room temperature MFM image of a magnetic recording disk acquired at a lift height of 150 nm and with a fork current of 46 nA rms and a scanning speed of 2 μm/s. The stripes in the image correspond to bits of information stored on the disk. Q-control was used to increase the probe quality factor from 1980 to 9200. For such a high quality factor, the optimum scan speed would be about four times slower than 2 μm/s. However, in this case the lift height is the limiting factor in determining the lateral resolution, and so the scan speed can be increased. Figure 1(b) shows cross sections of two images of the same area of the sample; one acquired with Q-control on (Q=9200), and the other with Q-control off (Q=1980). The signal levels have been normalised in the plots, so that the difference in SNR is clear. Measurements of $\partial\varphi/\partial f$ in conjunction with equation (1) showed that the measured phase shifts in Fig. 1 correspond to force gradients of order 10^{-3} N/m.

For low temperature imaging, the microscope is located in the variable temperature insert of a ^{4}HE cryostat. The sample is cooled by cold helium gas flowing through the sample space. Figures 2(a) and 2(b) are 10 μm $\times$ 10 μm topographic and MFM images, respectively, of a magnetic recording disk acquired in vacuum at T=136 K. The images were acquired with a lift height of 150 nm, a scan speed of 2 μm/s, and a fork amplitude of 50 nm rms. The unmodified fork quality factor was $Q_H=8400$. The phase contrast is $\sim 0.1°$. Figure 2(c) is a MFM image of the same area acquired under the same conditions, but with a probe quality factor of $Q_L=1800$. The image is plotted with the same z scale as Fig. 2(b) to emphasize the difference in contrast. In Fig. 2(d) the z scale is expanded, showing clearly that the signal-to-noise ratio is decreased compared to that of the high-Q image.

This use of Q-control to lower the quality factor serves two purposes. It allows us to further demonstrate the effect of Q on signal-to-noise ratio and it also demonstrates that we can tune the probe quality factor in order to reproduce the sensitivity we had at room temperature without Q-control. This is potentially very useful when performing a series of experiments under different experimental conditions. For example, the quality factor is affected by pressure and temperature[24] and varies from probe to probe. Q-control therefore allows the experimenter to reproduce the same sensitivity under different conditions by compensating for the effect of the environment on the damping of the probe.

The data acquired at room temperature and discussed earlier have been analyzed to quantify the effect of Q-control. First, a two-dimensional cross correlation was used to determine the positional shift between the images. Then further analysis was carried out using high and low Q images of the *same* region of the sample. The SNR was estimated using the method proposed by Sijbers *et al.*[25] They define an amplitude SNR as $\sqrt{(\sigma_S^2/\sigma_N^2)}$ where σ_S and σ_N are the standard deviations of the signal and noise, respectively. Assuming the noise is uncorrelated and additive an estimate of the SNR can be made by measuring the same image twice as the signal should be unchanged whereas the two noise signals should be uncorrelated. In our case adjacent lift-mode scan lines are separated by around 40 nm, a distance smaller than the lift height. As the magnetic force is long range, adjacent scan lines are strongly correlated. Therefore we carry out the analysis detailed in Ref. 25 using pairs of adjacent lines to estimate the SNR of a scan line. This minimizes the chance that changes in tip or sample, from one complete scan to the next, will affect the results. The overall SNR of an image is estimated using the mean and standard deviation of the SNRs of the scan lines that make up an image. The phase response of the cantilever increases as Q increases. To quantify this we have again used the area of the sample common to both images and we have used periodograms to calculate the change in rms signal level observed for low (natural) and high Q (Q-control) conditions.

We find that the amplitude SNRs of the low- and high-Q images are 3.03 ± 0.59 and 5.1 ± 1.0, respectively. The SNR changes by a factor of 1.6 ± 0.28, which is less than a $\sqrt{Q}$ dependence, as $\sqrt{(Q_H/Q_L)}=2.2$. The rms level of the signal increased by a factor of 6.21 ± 0.66 from the low to the high-Q image, which is greater than Q_H/Q_L (=4.6). An increase in rms level is to be expected, as the measured phase shift is expected to scale with Q for small frequency shifts. That the observed gain is higher than expected is not understood, but may be due to a slight phase error in the Q-control feedback signal leading to a steeper phase vs. frequency characteristic. The fact that the SNR increases by a factor smaller than $\sqrt{Q}$ suggests that the Q-control circuit itself is the cause of a small amount of noise in the response.

As the measured phase shifts near f_0 are small, they are proportional to the corresponding shifts in f_0. As discussed, the shift in resonant frequency induced by a force gradient is Q independent, so that in terms of frequency shift the signal is the same for both high and low Q images. Therefore the increase in SNR corresponds to a decrease in measured frequency noise. From this point of view, the Q-controlled quartz tuning fork system essentially acts as a tuned band-pass filter/amplifier, with a bandwidth that varies as $1/Q$, centered at f_0. However, as noted earlier the Q-control feedback itself may inject noise into the system limiting the increase in SNR.

In summary, we have demonstrated that piezoelectric quartz tuning forks can be used as probes for variable temperature magnetic force microscopy. Although intrinsically less sensitive to magnetic force gradients than conventional MFM cantilevers, their quality factors can be boosted using the technique of Q-control. This results in increased sensitivity to long-range force gradients, whether the noise level is greater than or at the thermal noise limit. It also allows the normalization of probe quality factors when acquiring images under different experimental conditions. These are important developments as they allow the advantages of tuning forks for low temperature work to be applied to variable temperature magnetic force microscopy.

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