# Low-temperature high-resolution magnetic force microscopy using a quartz tuning fork

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We have developed a low-temperature high resolution magnetic force microscope (MFM) using a quartz tuning fork that can operate in a magnetic field. A tuning fork with a spring constant of 1300 N/m mounted with a commercial MFM cantilever tip was used. We have obtained high-resolution images of the stray magnetic fields exerted from grains with a spatial resolution of 15 nm and force resolution of 2 pN at 4.2 K. Tuning fork-based magnetic force microscopes have the potential to be used at millikelvin temperatures due to their low power dissipation and high force sensitivity. © *2005 American Institute of Physics*. [DOI: 10.1063/1.2037852]

Since the possibility of a force microscope to detect magnetic stray fields was first demonstrated by Martin and Wickramashinghe,[1] magnetic force microscopy (MFM) has been developed as a high-resolution magnetic imaging tool.[2] In a conventional MFM, optical detection is used to measure the vibrational amplitude and frequency of a cantilever. These microfabricated cantilevers have small spring constants (0.1–10 N/m) leading to a high force sensitivity ($\sim$1 pN), comparable to the magnetic force between a tip and sample. However, cantilever-based MFM has some weak points: (i) Inconvenient optical alignment, (ii) a tendency for the tip to crash into the surface due to the low stiffness of the cantilever, (iii) high-resolution MFM is difficult because the dithering amplitude is large ($\gtrsim$10 nm), and (iv) the sample is exposed to laser light that may be detrimental for some applications.

One alternative method of optical detection that has been used for low-temperature MFM is fiber optic interferometry.[3,4] However, one of the disadvantages of the fiber-optic method is that the design of the MFM scan head is necessarily complex as it requires more than two coarse approach mechanisms in order to align the fiber optics and the cantilever. To reduce the complexity of the MFM design, piezoresistive cantilever detection has been used.[5,6] While piezoresistive cantilever detection has advantages, such as simple design and electrical detection without optics, it has serious drawbacks such as low sensitivity, poor spatial resolution, and excessive heat dissipation.

Using a tuning fork as a force transducer in a MFM overcomes many of the drawbacks of other designs. (i) Because the tuning fork sensor is stiff, the tip mounted on the tuning fork is not as easily pulled to the sample surface by attractive forces. (ii) Since the spectral noise density of the tuning fork is $\sim$100 fm/$\sqrt{\text{Hz}}$,[7] the minimum dithering amplitude is much smaller than that of a cantilever, allowing high-resolution imaging. (iii) As the tuning fork is a self-dithering and self-sensing device, no optics are required and it is simple and small. (iv) Because no light source is necessary and the dissipated power can be reduced down to $\sim$1 pW,[7] tuning fork-based MFM can be operated in the dark and in low-temperature conditions.

Edwards *et al.*[8] first demonstrated a MFM using a tuning fork with a piece of cut Fe wire. Todorovic and Schultz[9] employed an etched nickel wire as a tip in a similar geometry. They used a miniature tuning fork having a stiffness $k$ of 2000 N/m and they obtained MFM images of a hard disk. Both of these groups[8,9] achieved reasonable spatial resolution. However, the image contrast was not satisfactory compared to that obtained with cantilever-based MFM. While the resonance frequency $f$ of a tuning fork is similar to that of the cantilever, the quality factor $Q$ and the spring constant $k$ of a tuning fork are typically $10^2$ and $10^4$ times larger than those of the cantilever, respectively. Because the sensitivity of MFM is proportional to $\sqrt{Qf/k}$ in terms of thermal noise, the tuning fork-based MFM is less sensitive than the cantilever based MFM by a factor of 10.[9] This has been the most serious limitation of the tuning fork-based MFM.

In this letter, we report on high-resolution and high sensitivity MFM images that were obtained using a tuning fork-based MFM. The increased resolution and sensitivity are due to the use of tuning forks with smaller spring constants, operation at low temperatures which increases the $Q$ of the tuning fork and improves the stability of the instrument, and to our technique of attaching commercial cantilever tips to the tuning fork, which minimizes the loading of the tuning fork, leading to a high $Q$ value and a concomitant increase in the sensitivity.

Each prong of the tuning fork we used is 2.2 mm long, 190 $\mu$m thick, and 100 $\mu$m wide. This geometry of the tuning fork corresponds to a value of the spring constant $k \simeq 1300$ N/m. The technique for attaching the tip is similar to that described by Rozhok *et al.*[10] A tip with height 15–20 $\mu$m on a commercial MFM cantilever (Micromasch, NSC36/Co–Cr) was used. The cantilever was cut from its Si chip and glued at the end of a tuning fork prong with the tip facing outward using a home-made micromanipulator. The tip was magnetized by an electromagnet. After the tip was mounted on the tuning fork, the resonance frequency decrease, $\Delta f$, was about 20 Hz. According to the mass loading effect of the quartz crystal microbalance technique,[11] the added mass, $\Delta m$, can be estimated by $\Delta f/f_0 \simeq \Delta m/m_0$, where $m_0$ is the mass of the tuning fork and the original frequency of the tuning fork, $f_0$, is 32.768 kHz. This gives a total added mass including the epoxy, cantilever, and tip of just 0.1 $\mu$g.

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After the tip was mounted, the resonance full width at half maximum was about 5 Hz in air (1 Hz in vacuum). Its corresponding $Q$ value was about $10^4$ in air ($3\times10^4$ in a vacuum). These $Q$ values were almost the same as those before the tip was mounted. We attribute the remarkably small change in the $Q$ values to the very small amount of glue used. Because the $Q$ value was roughly 100 times larger than that of a cantilever, it compensated for the high stiffness of the tuning fork in terms of the sensitivity.

For the coarse approach mechanism, we modified the walker designed by Gupta and Ng[12] by replacing some materials in their original design for operation at low temperatures. The housing material (stainless steel in their design) was replaced by machinable ceramic (Macor) and polished alumina plates were attached to the housing surface to minimize the friction between the sapphire disks attached to the walker tube and the walker housing. With other choices of the contact materials (sapphire/glass or sapphire/brass), the walker invariably froze on cooling even to liquid-nitrogen temperatures.

The entire scan head was mounted on the end of an insert that could be cooled to 77 K and 4.2 K by dipping into liquid nitrogen and liquid helium, respectively. A magnetic field could also be applied by fitting the insert into a dewar equipped with a two-axis magnet capable of fields of 3 T in the axial direction and 1 T in the transverse direction. The resonance frequency shift and phase shift were measured by commercial phase-locked-loop (PLL) electronics (easyPLL from Nanosurf). We did not use any low-temperature preamplifier. The tuning fork signal was passed through a coaxial cable 1 m long and was fed into a current-voltage amplifier located outside the insert. The current-voltage amplifier consists of a 5 MΩ load resistor and × 100 gain instrumentation amplifier. The typical dithering amplitude was 5 nm (10 nm) at a drive voltage of 3.5 mV (10 mV) at 4.2 K (77 K).

Figures 1(a) and 1(b) show representative topographic and MFM images obtained simultaneously by the tuning fork probe at 77 K. The sample was a piece of a commercial hard disk in which the magnetic recording layer is a cobalt film. The size of the images is $3\times3\ \mu m^2$. For MFM scanning, constant height mode was employed with a lift height 50 nm. The corrugation seen in Fig. 1(a) is due to the grain structure of the thin film. The stripe pattern in the MFM image [Fig. 1(b)] is due to magnetically recorded bits written by normal operation of the hard disk. As there is little correlation between the two images, the MFM contrast indeed comes from the magnetic stray field.

Since the dynamic MFM contrast represents the spatial second derivative of the stray field or the force gradient, the frequency shift in MFM corresponds to the gradient of the magnetic force[13]

$$\Delta f \simeq \frac{1}{2}\frac{f}{k}\frac{\partial F}{\partial x}, \qquad (1)$$

where $f$ is the resonance frequency (33 kHz), $k$ is the spring constant of the tuning fork (1300 N/m), and $F$ is the magnetic force between the tip and the sample. The experimental noise level of the phase measurement was less than 0.5°, which corresponds to a frequency resolution of 1 mHz and a experimental force gradient resolution of $10^{-4}$ N/m. Considering that the dithering amplitude of the tuning fork was 10 nm, the experimental force resolution was 2 pN.

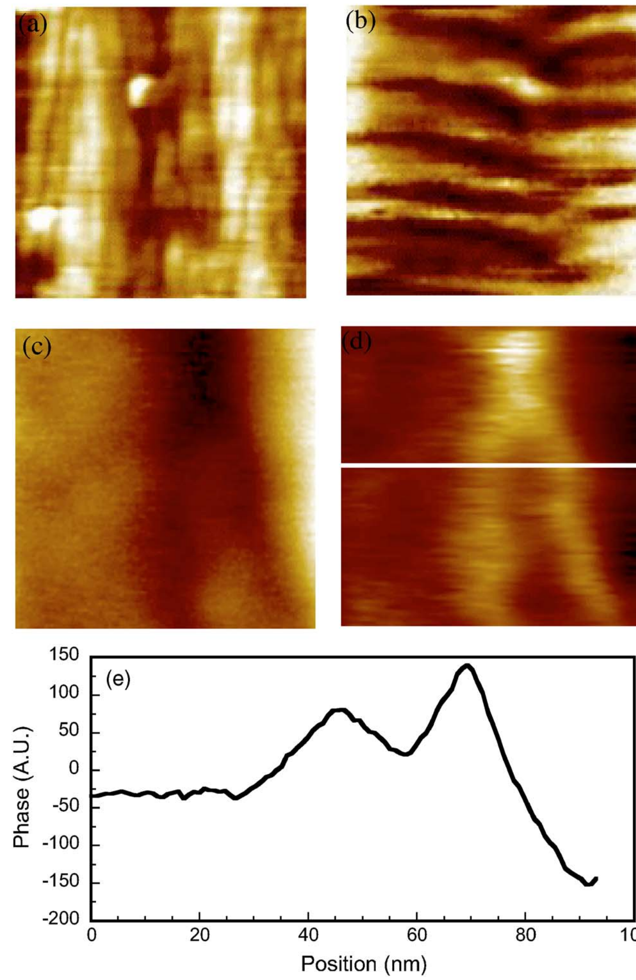


FIG. 1. Representative topographic (a) and MFM (b) images obtained simultaneously by a tuning fork probe at 77 K. The sample is a piece of a commercial hard disk. The scale of the images is $3\ \mu m\times3\ \mu m$. The structure seen in the topographic image arises from the grain structure of the film, while the stripes seen in the MFM image arise from magnetic domains on the hard disk. The two images are clearly uncorrelated. (c) and (d) Higher-resolution ($94\ nm\times94\ nm$) simultaneous topographic (c) and MFM (d) images of the same sample taken at 4.2 K. (e) The line profile across the white line shown in (d).

In order to evaluate the spatial resolution, the same hard disk sample was scanned over smaller areas with a 3.5 mV excitation at 4.2 K, corresponding to a dithering amplitude of 5 nm. Figures 1(c) and 1(d) show topographical and MFM images obtained simultaneously over an area of 94 nm × 94 nm. Lift mode scanning was applied for the MFM imaging by using the topographic profile to raise the tip 20 nm over the device. Assuming the sample has uniform magnetization on this size scale, the stray field above the surface depends on the topography. Bright parts in the MFM image correspond to valleys in the topography where the magnetic field flux is denser than that at the peaks. Due to low thermal noise and drift, a very high-resolution topographic image was obtained. The maximum height difference in Fig. 1(c) is 4 nm. The line profile between the arrows on the MFM image is shown in Fig. 1(e). Considering the narrowest width of peaks, the spatial resolution of our microscope is about 15 nm. This resolution is the same as the best resolution obtained with the conventional cantilever based MFM (Refs. 14

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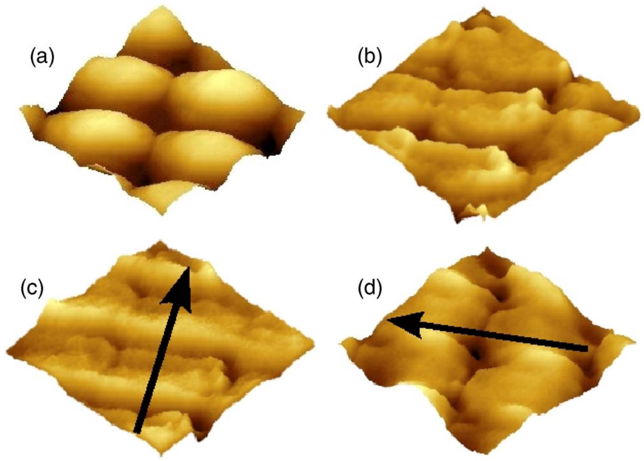


FIG. 2. Three-dimensional representations of simultaneous topographic (a) and MFM (b) images of an array of elliptical permalloy particles at 4.2 K, with magnetic field $H=0$. (c), (d) MFM images of the same sample at the same scale at 4.2 K with an applied field of 2.5 kG. The magnetic field was applied in the direction shown by the arrows in the figures. The scale of all images is 1 $\mu$m$\times$1 $\mu$m.

and 15) and much better than that of piezoresistive cantilever-based MFM.[5,6]

In order to demonstrate MFM imaging in an external magnetic field, a permalloy (NiFe) sample consisting of an array of elliptical particles fabricated by electron-beam lithography was imaged at 4.2 K with the external field applied in the plane of the permalloy film. Each elliptical particle has a long axis of 600 nm and short axis of 300 nm based on scanning electron micrograph images. A three-dimensional representation of the topographic image of the sample is shown in Fig. 2(a). The scanned area was 1 $\times 1$ $\mu$m$^2$ and the excitation voltage was 7 mV. Figure 2(b) shows the MFM image of the same region without magnetic field. From the height differences in the three-dimensional representation, it appears that each particle has its magnetization aligned along its long axis, although the height contrast is not pronounced. Dramatic changes of the MFM images occurred when a horizontal magnetic field ($H$ $=2.5$ kG) was applied. This field is large enough to align the magnetization of the sample as well as the tip. Figures 2(c) and 2(d) show the resulting MFM images with the direction of magnetic field denoted by the arrows. Both images have stripe patterns running at right angles to the magnetization. This is due to the attractive and repulsive interaction between the tip and sample that depend on the relative location of the tip.

In conclusion, we have demonstrated a low-temperature high-resolution MFM using a quartz tuning fork. We have obtained clear images with a spatial resolution of 15 nm at 4.2 K. Tuning fork-based MFM is a very promising candidate for millikelvin temperature MFM because of its low power dissipation, simple design, and high spatial resolution.

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