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Micromechanical torque magnetometer for *in situ* thin-film measurements

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Abstract-- We describe a new type of magnetometer based on a microelectromechanical system (MEMS) for *in situ* monitoring of magnetic film moment during the film deposition process. The magnetometer measures mechanical torque on a film as it is deposited onto a microscopic flexible silicon cantilever. The cantilever is excited by an external ac magnetic field and its angular displacement is proportional to the magnetic moment of the film. The instrument has a magnetic moment sensitivity of 1×10^{-12} A m^2 / $\sqrt{Hz}$ corresponding to a torque sensitivity of 4×10^{-16} N m / $\sqrt{Hz}$. We were able to detect the moments of Fe films as thin as 3 nm. For thicker films (above 9 nm) we can detect thickness changes as small as 0.3 nm, corresponding to the instrument's moment sensitivity limit.

***Index Terms*-- torque magnetometer, microelectomechanical systems (MEMS), atomic force microscope (AFM), micro-cantilever**

I. Introduction

THE characterization of nanometer-scale magnetic multilayer films, patterned recording media, and magnetic devices has proven to be a challenge for conventional magnetometers. The limitation on sensitivity stems from low sample-energy/sensor-energy ratios typical of conventional magnetometers. Sensitivity can be improved by integrating samples with the measurement transducer using microfabrication methods. In particular, we are developing a new class of magnetometer based on microelectromechanical systems (MEMS) for measuring magnetic forces and torques on samples deposited onto microscopic flexible structures.

As an example, consider the simple MEMS sensor having a thin magnetic film deposited onto a micromachined torsional oscillator. When the oscillator is excited by an external ac magnetic field, its angular displacement is proportional to the magnetic moment of the film. In principle, a torque as small as 10^{-20} N·m / $\sqrt{Hz}$ can be detected at room temperature by operating high Q, low spring-constant micromechanical oscillators at resonance [1]. In contrast, the best torque sensitivity for conventional instruments is 10^{-10} N m / $\sqrt{Hz}$. Several groups have investigated MEMS-based magnetometers over the last ten years [2]-[11]. MEMS magnetometers allow accurate measurements of isolated thin-film samples with nanometer dimensions under ambient conditions. In addition, batch-fabricated MEMS magnetometers have the potential to be a cost-effective method for measuring the magnetic properties of small samples and devices.

The growth conditions for making ultra-thin films and thin-film multilayers are critical to maintain product uniformity. Typically, film deposition is monitored *ex situ* with B-H loopers that determine the $M_s t_f$ product for the film, where M_s and t_f are the saturation magnetization and the thickness of the film, respectively. We present here a method based on micromechanical sensors for making *in situ* measurements during film deposition. In particular, we show results for Fe films deposited onto silicon micro cantilevers. Ultimately, our goal is to develop disposable active substrates for quantitative measurements of films as they are deposited with sub-monolayer magnetic moment sensitivity.

II. Apparatus

The micromechanical torque magnetometer system studied here is shown schematically in Fig.1. An atomic force microscope (AFM) detector head equipped with a beam-bounce detection scheme having a split photodiode detector is used to measure the magnetic torque on an AFM cantilever coated with a magnetic film. A small solenoid close to the cantilever provides the necessary ac torque field H_T of 300 A/m rms at 10 kHz. An oscillator combined with a low-noise power amplifier supplies the current to the solenoid. The oscillator signal is also used as the reference for a lock-in amplifier. The cantilever deflection signal is fed to a lock-in amplifier and the output from the lock-in is proportional to the magnetic moment as discussed below. The lock-in is operated with a 200 ms time constant. The AFM head is placed between a pair of SmCo permanent magnets that supply a bias field H_0 of 10.8 kA/m. Under these conditions, the film should be fully saturated in the plane. The orientation of the cantilever relative to the $\boldsymbol{H_T}$ and $\boldsymbol{H_0}$ is shown in Fig. 2. $\boldsymbol{H_T}$ is nominally perpendicular to the surface of the magnetic film deposited onto the cantilever. The AFM head is tilted at 7° and an evaporation mask is used to avoid coating the optics during deposition.

Fe films (99.9%) were deposited onto commercial single-crystal silicon cantilevers. Depositions were done in a diffusion-pump vacuum chamber with a liquid nitrogen cold trap. The background pressure was 2.66×10^{-4} Pa. The films were evaporated from alumina coated tungsten boats at a

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deposition rate ranging from 0.1 to 0.3 nm/s. Cantilevers had a length l_c of 449 μm, a width w_c of 49 μm, a thickness t_c of 2.5μm, a spring constant of 0.35 N/m, and a resonant frequency of 14 to 17 kHz.

In the case of a uniformly magnetized thin film with a strong in-plane anisotropy [12], the torque on the film is $\boldsymbol{T_M} = \mu_o |\boldsymbol{m} \times \boldsymbol{H_T}| = \mu_o m H_T$, assuming a 90º angle between H_T and the total magnetic moment m [13]. In the configuration as described in Fig. 2, the torque acts as a uniform bending moment along the cantilever. By integrating the bending moment along the cantilever we find the displacement z at the end of the cantilever to be [14]

$$z = \frac{6\mu_o M_s t_f w_c H_T}{Ewt_c^3}\int_0^{l_c}(l_c^2 - x^2)dx \qquad (1)$$

$$= \frac{4\mu_o m H_T l_c^2}{Ew_c t_c^3} \qquad (2)$$

$$= \mu_o M_s t_f \times \frac{4H_T l_c}{Et_c^3}. \qquad (3)$$

For a 40 nm thick iron film (m = 8.9 × 10^{-10} A m^2 from Ref. [9]) with an H_T of 300 A/m rms we calculate z = 3 nm rms given a Young's Modulus E for silicon of 1.3 x 10^{11} N/m^2. This number is in reasonable agreement with our experimental measurement of z = 2.7 V rms (lock-in signal) × 50 mV/10V (lock-in sensitivity) × 344 nm/V (AFM sensitivity) = 4.6 nm rms, given uncertainties regarding the cantilever geometry (particularly thickness) and the value of H_T at the cantilever.

III. Results and Discusion

We perform magnetic moment measurements as a function of film thickness in the following way. First, 2 nm of Fe is deposited onto a bare cantilever. The system is allowed to equilibrate and the rms deflection of the cantilever is determined. During deposition the cantilever heats and bends and it is necessary to let it cool to take a stable measurement. Equilibration takes about one minute. Subsequently, 2 nm increments of are added to the film and deflection measurements are made after each deposition. In addition, as the film thickness exceeds 10 nm, the position of the photodiode detector must be repositioned due to residual stress in the Fe film causing the cantilever to bend out of the linear range of the system. The Fe film and the silicon cantilever form a bimaterial couple that bends when heated due to the differences in thermal expansion coefficients. Film thickness is determined with a quartz crystal microbalance to within an accuracy of 0.1 nm. Figure 3 shows the magnetic moment of the film as a function time during the incremental deposition process. Note that the magnetic moment is disproportionably small until the film thickness reaches 9 nm.

Figure 4 shows the averaged values of the magnetic moment after each deposition increment as a function of the corresponding film thickness. The trend is for the m/t_f ratios to be smaller for films less than 50 nm, becoming vanishingly small for a thickness below 9 nm. It is not unexpected that the moment should be reduced for films a few nanometers thick because dead layers can form during the initial stages of deposition. In addition, the film may be forming oxides or clusters. Further study is required to determine film microstructure in order to differentiate these possibilities. Finally, the film anisotropy may be quite low and thus there would then be little torque transferred to the cantilever (as discussed in Ref. [11]). The fact that the data for large thickness appears to extrapolate to zero supports the low anisotropy argument.

The data in Fig.s 3 and 4 indicate that we are able to detect the moments of films as thin as 3 nm. For thicker films (above 9 nm) we can detect thickness changes smaller than 0.3 nm corresponding to the instrument's moment sensitivity limit of 1 × 10^{-12} A m^2 / $\sqrt{Hz}$. Based on the relationship $T_M = \mu_o m H_T$, discussed above, this corresponds to a torque sensitivity of 4 × 10^{-16} N m / $\sqrt{Hz}$.

IV. Future Work

It would be desirable to measure film magnetic moment continuously during the deposition, but we found this to be difficult due to the cantilever bending due to heating effects. In addition, the heating also makes resonant operation of the cantilever difficult since the resonant frequency changes with temperature. In the future, we plan to develop torsional oscillators that suffer less from this problem, potentially allowing for resonant operation of the oscillator to enhance the signal-to-noise ratio, which is limited by the thermal excitation of the cantilever. The equivalent noise is

$$T_{noise} = \sqrt{\frac{2k_s k_B T}{Q f_o}}. \qquad (4)$$

where k_s is the torsional spring constant, Q is the mechanical quality factor, f_o is the resonant frequency, and $k_B T$ is the thermal energy.

With a Q of 10^4 we expect a factor of 100 reduction in thermal noise. Yasumara *et al*. [15] have performed a systematic study of these effects on the Q for cantilever deflection modes, however, a similar study on torsional modes has not yet been done. If coated cantilevers with such a high Q can be realized along with reductions in the k_s / f_o ratio then it should be possible to achieve a sensitivity of 10^{-18} A m^2 / $\sqrt{Hz}$ at room temperature with specialized micromechanical oscillators. This sensitivity is sufficient for measuring equivalent magnetic moments well below the monolayer level during film growth.

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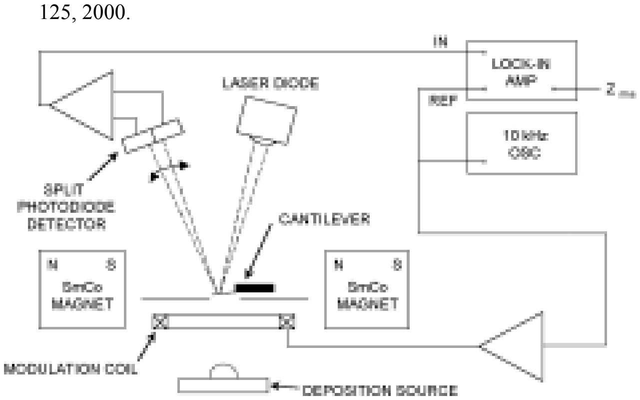

Fig. 1. Schematic diagram of the experimental setup showing the micromechanical torsional oscillator system including the laser beam-bounce detector, micro-cantilever, and the modulation coil. The experiment was performed in a vacuum bell jar with the torsional oscillator system suspended above the deposition source.

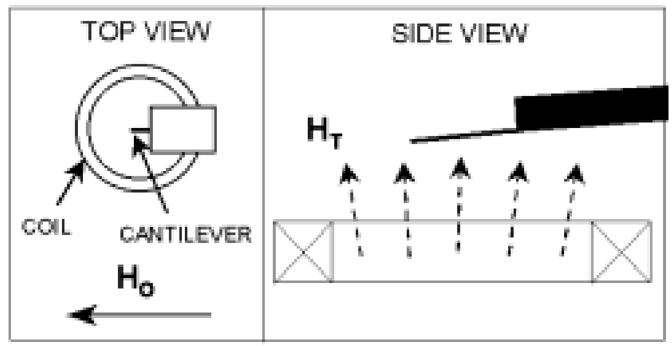

Fig. 2. Diagram showing the orientation of the magnetic fields relative to the cantilever during deposition.

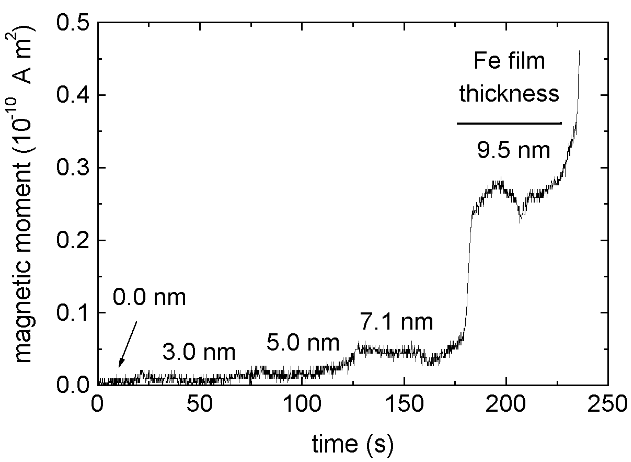

Fig. 3. Magnetic moment of the Fe film measured with the micromechanical torsion magnetometer over time. The film was deposited in 2 nm steps. After each step the magnetic moment was measured.

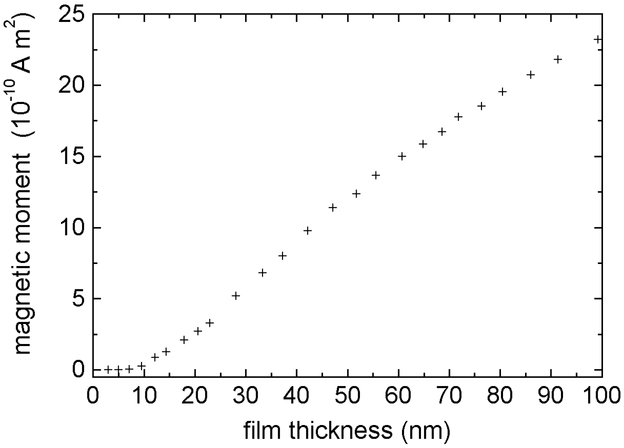

Fig. 4. Magnetic moment measured with the micromechanical torsion magnetometer versus Fe film thickness measured with a quartz crystal microbalance.