Microcantilever torque magnetometry of thin magnetic films

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We have developed a microcantilever torque magnetometer based on a torsion-mode atomic force microscope. Thin magnetic films are deposited directly onto micromachined silicon cantilevers. We have measured hysteresis loops of iron thin films with thicknesses ranging from 1 to 40 nm and total magnetic volumes ranging from 2.2×10^{-11} to $8.8\times10^{-10}\,\mathrm{cm}^3$. The magnetic moment sensitivity is estimated to be $1.3\times10^{-12}\,\mathrm{A\,m^2/Hz^{1/2}}$ at room temperature and ambient conditions. We expect that by operating at the cantilever torsion resonance frequency and at higher torque fields sensitivity will be improved by a factor of 100–1000. [S0021-8979(00)17508-5]

There are many standard techniques, including vibrating sample magnetometry (VSM), alternating gradient magnetometry (AGM),[1,2] torque magnetometry,[3] and superconducting quantum interference device (SQUID) magnetometry, that have been successfully employed to study magnetic properties of materials.[4] With the development of magnetic thin-film sensors[5] and actuators with nanometer dimensions the needs for improving sensitivity and spatial resolution in magnetometry are paramount.

Microcantilever torque magnetometry (MTM) based on a torsion-mode atomic force microscope (AFM)[6] offers several advantages over current magnetometers. In this article we describe a sensitive magnetometer for measuring micromagnetic properties of thin magnetic films under ambient conditions. The gain in sensitivity is obtained by integrating the micrometer sized sample directly with an AFM microcantilever. The picometer deflection sensitivity that is typical for AFM instruments allows the measurement of very small torques with cantilevers having sufficiently small torsion constants.

The standard AFM head is equipped with a beam bounce detection scheme[7] having a four-quadrant diode detector array capable of measuring both torsion and deflection of a micromachined cantilever. A small solenoid close to the cantilever provides the necessary ac torque field. The 100 Hz ac current to the solenoid is supplied by an oscillator combined with a power amplifier. The oscillator signal is also used as reference for the two lock-in amplifiers. The cantilever deflection signal is fed into lock-in amplifier (1), whereas the cantilever torque signal is fed into lock-in amplifier (2). This configuration enables us to detect both the deflection and the torque signals at the same time. Torque coil currents of 1–50 mA generated fields of 0.01–0.57 mT. The head of the AFM is nonmagnetic and fits into a pair of Helmholtz coils for field measurements up to 7 mT.

The orientation of the cantilever versus the torque field B_T and sweep field B_0 is shown in Fig. 1. The torque-field orientation is perpendicular to the magnetization M of the magnetic thin film deposited onto the flat side of the cantilevers. In order to measure hysteresis loops of thin magnetic films the sweep field provided by the pair of Helmholtz coils is applied in the plane of the film and parallel to the short side of the cantilever. The sweep rate was 0.25 mT/s for a lock-in time constant of 300 ms.

The magnetic films were deposited onto the flat side of the silicon microcantilever. Mica substrates 6 mm×6 mm were placed alongside the cantilever samples. These codeposited samples were used for comparative AGM measurements.

The cantilevers have a length of $l=449\,\mu\mathrm{m}$, a width of $w=49\,\mu\mathrm{m}$, and a thickness of $t=2.5\,\mu\mathrm{m}$.

A series of thin iron films were prepared by thermal evaporation. The background pressure in the vacuum chamber during the deposition was 4×10^{-4} Pa, and the evaporation rate was 0.15 nm/s. In order to prevent the iron thin films from oxidizing rapidly, we covered the films with a 5 nm thick Au cap layer.

Figures 2(a), 2(c), and 2(d) show hysteresis loops for iron films with different film thicknesses taken with the MTM. In this experiment, we ramped up the sweep field B_0 to the maximum negative value of 7 mT before recording the data. The torque field B_T provided by the solenoid was kept constant at a level of 0.1 mT for the measurement. The torque L of the cantilever (see Fig. 1) as measured by lock-in amplifier (2) was plotted as a function of B_0. Several interesting observations were made: The torque signals of the MTM sensors (cantilever plus magnetic film) decreased lin-

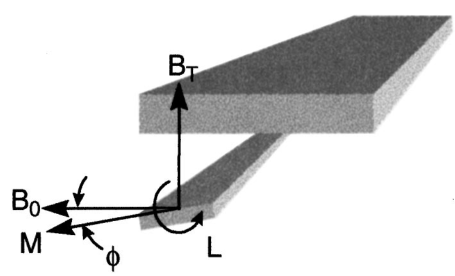

FIG. 1. Experimental configuration: A cantilever experiences a torque L when the magnetization M of the deposited magnetic film is aligned with the applied field B_0 and an additional torque field B_T is present. For small values of the cantilever twist angle ϕ, the torque is proportional to ϕ.

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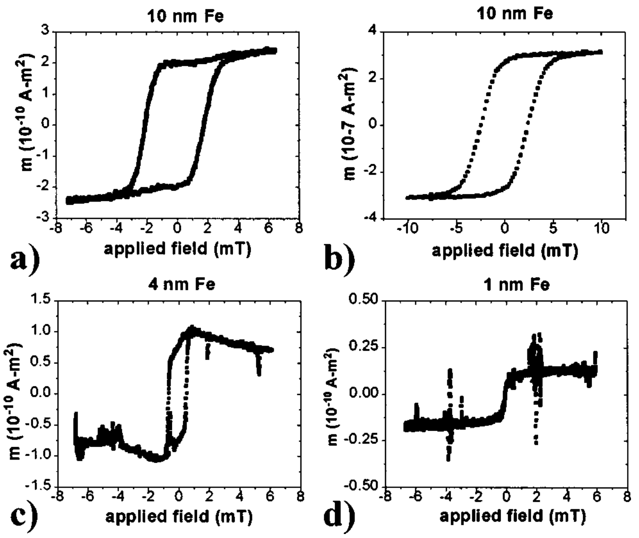

FIG. 2. (a), (c), (d) Hysteresis loops of integrated m vs B_0 for iron films measured with the MTM. (b) A typical hysteresis loop of integrated m vs B_0 for iron films measured with the AGM.

early with decreasing film thickness, i.e., magnetic volume. The magnetic moment m decreased from $8.9\times10^{-10}\,\mathrm{A\,m^2}$ for a 40 nm thick iron film to $1.8\times10^{-11}\,\mathrm{A\,m^2}$ for a nominally 1 nm thick iron film [Fig. 2(d)]. For iron film thicknesses from 40 to 4 nm the hysteresis loop was open, as expected, for an easy-axis hysteresis loop of a ferromagnetic film.

These results agree with our AGM measurements performed on co-deposited samples [see Fig. 2(b)] on 6 mm $\times$6 mm mica substrates. Generally, the characteristic features of the thin iron films are apparent in both sets of data.

An increased noise was observed in our MTM measurements beginning from an iron film thickness of less than 4 nm. This noise pattern is not random, but shows a correlation with the applied sweep field value. We believe that this noise is related to changes in the domain structure or to movements of domain walls within the iron films at certain field values. One would expect the formation of domain structures, since the iron films are patterned structures with the dimensions of the silicon microcantilever.[8] We conclude from the data, that the signal to noise ratio (SNR) of our room-temperature MTM allows measurements of iron films with a nominal 1 nm thickness. Figure 2(d) shows the hysteresis loop of the 1 nm iron film. The behavior of the film is still ferromagnetic. The total magnetic volume of this sample was calculated to be $2.2\times10^{-11}\,\mathrm{cm^3}$. A direct correlation of the measured loop with an AGM measurement was not possible, since the sensitivity of the available instrument was not sufficient to detect any ferromagnetic signals from iron films with a thickness less than 4 nm. However, we have used the AGM to calibrate our MTM for iron films thicker than 4 nm. The summary of the measured magnetic properties is given in Table I. The magnetization M values determined from AGM measurements for the thicker iron films are in the range of 1000–1100 kA/m at 6 mT. It is known that prepared thin iron films could have smaller magnetization values due to structural changes.[9] This leads to a smaller effective magnetic volume, and therefore to a smaller calculated magnetization of the iron films. The magnetic moment for each iron film at 60 mT which was calculated from $m = V^{*}_{\mathrm{film}} M_{60\ \mathrm{mT}}$, is given also in Table I.

TABLE I. Magnetic properties of iron films.

Iron film thickness (nm)	Magnetic volume V (cm^3)	Magnetic moment m (A m^2)	Magnetization $M_{60\ \mathrm{mT}}$ (10^3 A/m)
40[a]	1.0×10^{-6}	1.0×10^{-6}	1008
40[b]	0.88×10^{-9}	8.9×10^{-10}	1008
20[a]	0.58×10^{-6}	6.4×10^{-7}	1103
20[b]	0.44×10^{-9}	4.9×10^{-10}	1103
10[a]	0.28×10^{-6}	3.1×10^{-7}	1092
10[b]	0.22×10^{-9}	2.4×10^{-10}	1092
4[a]	0.10×10^{-6}	8.3×10^{-8}	807
4[b]	0.88×10^{-10}	7.1×10^{-11}	807
2.5[b]	0.55×10^{-10}	4.4×10^{-11}	807[c]
1[b]	0.22×10^{-10}	1.8×10^{-11}	807[c]

[a]Alternating gradient magnetometer (AGM) measurement.
[b]Microcantilever torque magnetometer measurement.
[c]Estimated value based on AGM measurements of thicker films.

The dependence of the torque signal on the applied torque field for the 10 nm iron film is shown in Fig. 3. The torque signal increases linearly with the applied torque field, which scales as expected with the current in the coil. We could not observe any saturation of the torque signal at higher torque fields that could be assigned to canting or out-of-plane rotation of the magnetization. We estimate that our torque fields are in the range of 0.01–0.57 mT. Even for a torque field as small as 0.1 mT, a SNR of 40 is obtained with our MTM instrument.

We can define the limit for the applied torque field B_T as the field strength at which the thin film's in-plane magnetization will be rotated 5° into the out-of-plane orientation. Below this limit we estimate the MTM in-plane magnetic moment is within 1% of its true value. This field strength is given by the in-plane and out-of-plane anisotropy fields for the given ferromagnetic material. Given that only the demagnetization field keeps the magnetization in plane, we find that for polycrystalline iron films thicker than 10 nm field values on the order of 800 kA/m would be required to rotate the

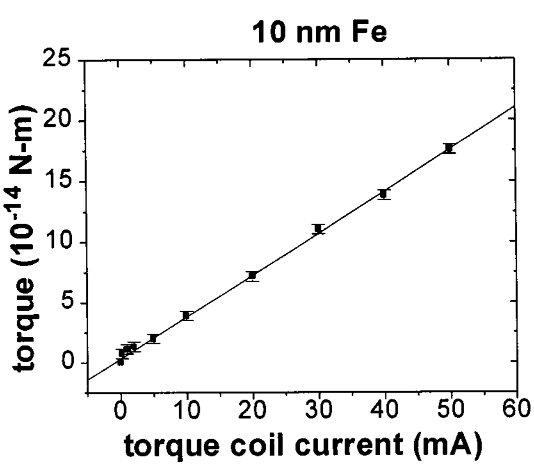

FIG. 3. Torque vs torque coil current for an iron film thickness of 10 nm.

magnetization 5° out of plane. For thinner films this field value is reduced due to an increase in out-of-plane anisotropy.[10] First-order calculations show that, for a 1 nm iron film, torque fields of 80 kA/m would be required to tilt the magnetization 5° out of plane. Theoretically we could increase our torque sensitivity by a factor of 800 for thin films (<2.5 nm) and by a factor of 8000 for thicker films (>10 nm). In the case of a uniform magnetized thin film with a strong in-plane anisotropy, we can calculate the torque on the microcantilever as $L_M = |\mathbf{m} \times \mathbf{B_T}| = m B_T \sin \varphi$, with φ the angle between the torque field and the magnetization.[11] In our case the angle between the MTM sensor and the torque field φ is 90°; therefore the torque is given by $L_M = m \mu_0 H_T$. For the 10 nm thick iron film ($m = 2.4 \times 10^{-10}$ A m^2) at a torque field of 90 A/m we calculate $L_M = 2.7 \times 10^{-14}$ N m. To check this calculation we can compute the torque from the geometrical parameters of the microcantilever, given the fact that we know the torsion angle. The torsion angle of a beam with uniform torque and with $t \ll w$ can be calculated from elastic theory to be[12]

$$\phi = \frac{6(1+n)l}{E(wt^3)} * L. \tag{1}$$

Here, E is the Young's modulus ($E = 1.3 \times 10^{11}$ N/m^2), n is the Poisson ratio ($n = 0.28$), and l is the length of the microlever ($l = 449\ \mu$m). The torsion angle ϕ is calculated to be 1.35×10^{-6}rad, since the four-quadrant diode detector has a sensitivity of 344 nm/V and the torque signal of the 10 nm iron film was 1.76 mV. The corresponding torque L_G calculated from geometrical parameters is $L_G = 3.9 \times 10^{-14}$ N m. The torque values derived from the magnetic and geometrical calculations are very close, $L_M = 2.7 \times 10^{-14}$ compared to $L_G = 3.9 \times 10^{-14}$ N m, respectively. The small difference may be attributed to eddy currents generated in the metallic parts close to the cantilever; the eddy currents change the local torque field.

Several cantilever-based magnetometers[13–15] rely on high-Q resonators.[16] The SNR is limited ultimately by the Brownian motion of the cantilever. Generally the MTM sensitivity can be improved by operating at the resonance frequency, in high vacuum and at low temperature.[17] However, we point out that our instrument, operating at room temperature under ambient conditions and well below the cantilever resonance frequency, has a magnetic-moment sensitivity on the order of 10^{-12} A m^2.

A similar instrument has been used to measure out-of-plane anisotropy K of thin magnetic films, described by Morillo *et al.*[18] In this mode of operation the shifts in the torsion resonance frequency caused by the magnetic stiffening of a torsion oscillator are measured as a function of applied field. Future prospects include combining $M-H$ and K measurements into one instrument.

One of the authors (M.L.) has been supported by the Deutscher Akademischer Austauschdienst (DAAD), Die Arbeit wurde im Rahmen des Gemeinsamen Hochschulsonderprogramms III von Bund and Ländern über den DAAD ermöglicht. The authors acknowledge Tom Silva and Ron Goldfarb for helpful discussions and insight.

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