JOURNAL OF APPLIED PHYSICS **97**, 10R504 (2005)

Quantitative measurement of magnetic moments with a torsional resonator: Proposal for an ultralow moment reference material

D. H. Min
National Institute of Standards and Technology, Boulder, Colorado 80305 and Colorado School of Mines, Golden, Colorado 80401

John Moreland[a)]
National Institute of Standards and Technology, Boulder, Colorado 80305

(Presented on 9 November 2004; published online 17 May 2005)

We present a method for defining the magnetic moment of a reference material based on a torsional resonator with a patterned magnetic film on its surface. Given accurate measurements of the magnitude of the applied field, the moment of inertia of the resonator, and the magnetic stiffening effect of the film on the resonance frequency of the resonator, we can obtain an absolute measurement of the anisotropy energy and magnetic moment of the film independently. With this approach, the anisotropy energy and the magnetic moment of the film are measured directly, eliminating the need for a detailed knowledge of the film's saturation magnetization or sample volume. In principle, all of the measurements needed for the reference material are traceable to an atomic clock frequency reference. We have performed preliminary measurements on $Ni_{0.8}Fe_{0.2}$ films patterned in circle of 4.17 mm diameter. Representative samples had measured magnetic moments of 5.04 ± 0.12 μA m^2 for nominal film thicknesses of 500 nm. [DOI: 10.1063/1.1853276]

I. INTRODUCTION

We propose a method for an absolute measurement of the magnetic moment, m, of a standard reference material (SRM) used to calibrate magnetometers. Dionne and Fitzgerald, and Weiss *et al.* have developed low-friction oscillating torque magnetometers for measuring the properties of relatively large planar films.[1,2] Recently, Morillo *et al.*[3] suggested a related method to measure the magnetic anisotropy of a thin film deposited on the head of a T-shaped silicon torsional resonator. The resonator was made by use of silicon micromachining techniques developed for microelectromechanical systems (MEMS). In this article we describe an extension of the work by Morillo *et al.* that is well suited for measuring thin films with total magnetic moments of less than 1 μA m^2 that would serve as reference materials for magnetometers in this measurement range. Currently, the lowest absolute moment reference material is 77 μA m^2 (NIST SRM 2853-YIG sphere). We give details of the operating principle of the torsional resonator optimized for SRM applications, the resonator chip fabrication process, and the apparatus for measuring the resonance frequency of the resonator using a laser interferometer, as well as a brief description of future plans to use frequency-based measurements to measure the critical parameters needed to determine m.

II. PRINCIPLE

Consider a simple MEMS sensor with a thin magnetic film deposited onto the surface of a micromachined torsional oscillator, as shown in Fig. 1. When the cantilever is excited resonantly by a small external ac torque field H_t, the direction of the film magnetization is rotated from its equilibrium direction during the course of the oscillation. An additional restoring torque due to a static applied bias field H_0 increases the resonance frequency of the oscillator. The resonance frequency f_0 of the torsional mode without the applied field H_0 is

$$f_0^2 = \frac{k_0}{4\pi^2 I}, \tag{1}$$

where I is the rotational moment of inertia of the cantilever and k_0 is the resonator's mechanical torsional stiffness. When the bias field is applied to the resonator paddle, the resonance frequency will be changed to a new value

$$f^2 = \frac{k_t}{4\pi^2 I}, \tag{2}$$

where $k_t = k_0 + k_m$, and k_m is the mechanical stiffness due to the magnetic field. From Eqs. (1) and (2), we solve for k_m as follows:

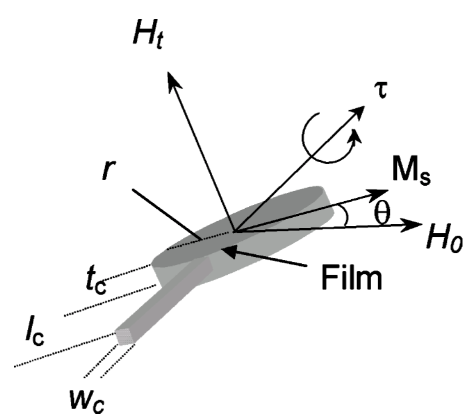

FIG. 1. Dimensions of resonator spring and paddle showing external bias field, torque field, and torque directions.

[a)]Electronic mail: moreland@boulder.nist.gov

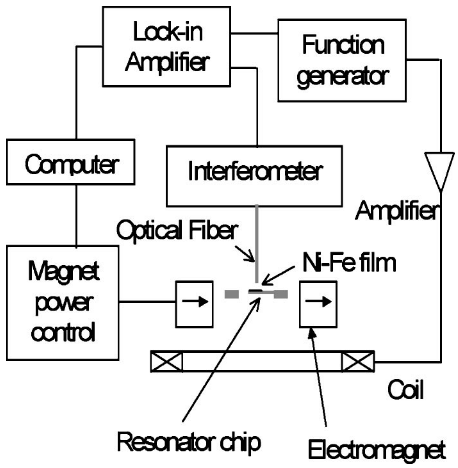

FIG. 2. Schematic diagram of experimental setup.

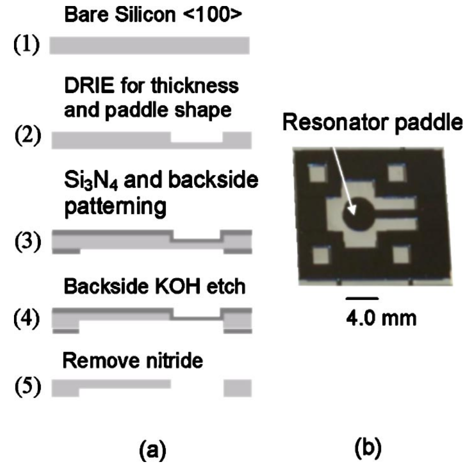

FIG. 3. Diagram of resonator paddle process steps: (a) fabrication process steps of the paddle; (b) photograph of finished paddle.

$$\frac{1}{k_m} = \frac{1}{4\pi^2 I(f^2 - f_0^2)}. \tag{3}$$

k_m can also be derived from the relation between the magnetic contribution to the restoring force and the potential energy per unit volume of the film[1] and can be written in the small-angle limit as

$$\frac{1}{k_m} = \frac{1}{2k_E a_f t_f} + \frac{1}{\mu_0 H_0 M_s a_f t_f}, \tag{4}$$

where k_E is the perpendicular magnetic anisotropy energy constant, a_f is the magnetic film area, M_s is the saturation magnetization of the film, t_f is the film thickness, and μ_0 is the permeability of free space. Combining Eqs. (3) and (4) relates resonance frequency to H_0:

$$\frac{1}{f^2 - f_0^2} = \frac{2\pi^2 I}{k_E a_f t_f} + \frac{4\pi^2 I}{\mu_0 M_s a_f t_f}\frac{1}{H_0} = \frac{2\pi^2 I}{k_E V_f} + \frac{4\pi^2 I}{\mu_0 m}\frac{1}{H_0} \tag{5}$$

Equation (5) is linear when plotting $1/(f^2-f_0^2)$ vs $1/H_0$, assuming k_E is constant as a function of H_0. We can determine the slope, and thus accurately calculate the magnetic moment of the film $m=M_s a_f t_f$ and the anisotropy energy $k_E V_f$ from the intercept. A detailed knowledge of small changes in k_E as a function of H_0 measured by independent means (such as FMR for example) would be necessary for further development of a reference material.

III. EXPERIMENT

The apparatus for detecting the resonance frequency shift with external bias field is shown in Fig. 2. The torsional resonator chip is located in the gap of an electromagnet. A sample holder fixes the resonator chip a few micrometers from the cleaved fiber end of the laser interferometer. The electromagnet generates a bias field H_0 at the oscillator chip that is greater than the saturation field and ranges from 52.0 to 86.0 kA/m, as measured with a Hall probe. The ac rms torque field H_t is 358 A/m. An optical fiber interferometer, similar to conventional optical fiber interferometers used in atomic force microscopy,[4,5] is used to measure the resonator's vibration and resonance frequency changes. H_0 is stepped in increments by adjusting the current through the electromagnet. At each field value, the resonance frequency is determined by measuring the resonator phase with a lock-in amplifier compared to a reference drive signal with a phase noise of −48 dB c. The uncertainty of the measurement is less than 1 mHz.

Standard MEMS and thin-film deposition processes were used to prepare the torsional resonator chip. Details of the process are shown in Fig. 3(a). We started with a buffered oxide etch to remove the native oxide from the double sided polished ⟨100⟩ Si wafer. Then, deep reactive ion etching was used to define the cantilevers. The cantilever thickness of 131 ± 10 μm was set by timing the DRIE process and measured with a profilometer. We deposited 0.4 μm Si_3N_4 on both sides of the wafer using low-pressure chemical vapor deposition. The backside nitride was patterned using reactive ion etching (RIE) to make the KOH etching windows. The cantilever release process is based on a KOH anisotropic etch (30% by weight with 2%–3% isopropyl alcohol, and an oxygen bubbler at 75 °C). After the KOH etch, the Si_3Ni_4 layer on the front side was removed by RIE before deposition of the magnetic film. The dimensions of oscillator and experimental parameters are described in Table I. Figure 3(b) is a photograph of the final structure of the resonator chip. The resonance frequency of the cantilever was 5824.6418 ± 0.0005 Hz. The Q value was measured by free decay of the resonator to be 2.50×10^5. To improve the resonator mechanical Q, the measurements were performed under a vacuum of 0.1 Pa.

TABLE I. Experimental parameters.

Symbol	Definition	Value
l_c	Cantilever length	5.000 ± 0.010 mm
w_c	Cantilever width	1.000 ± 0.010 mm
t_c	Cantilever thickness	0.130 ± 0.003 mm
r	Radius of paddle	2.084 ± 0.008 mm
ρ	Density of silicon	$2{,}330$ kg/m^3
I	Moment of inertia	$(4.680 \pm 0.115) \times 10^{-12}$ kg m^2
μ_o	Permeability of free space	$4\pi \times 10^{-7}$ H/m
H_0	External bias field	5.25–8.63×10^4 A/m

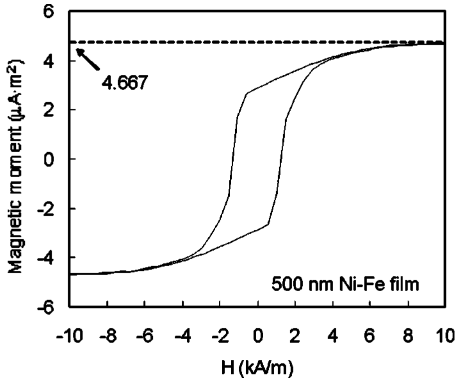

FIG. 4. Representative hysteresis loop measured with AGM of one of $Ni_{0.8}Fe_{0.2}$ samples.

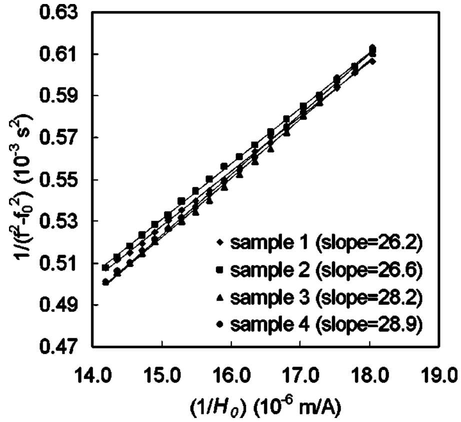

FIG. 5. Plots of $1/(f^2-f_0^2)$ vs $1/H_0$ for four samples of 500 nm of $Ni_{0.8}Fe_{0.2}$.

$Ni_{0.8}Fe_{0.2}$ films were deposited onto the cantilever paddle of the resonator chip. The deposition was done in a diffusion-pumped vacuum chamber with liquid nitrogen cold trap. The background vacuum pressure used was 2.6×10^{-4} Pa, during film deposition, the vacuum pressure was $5.3\times10^{-4\ \mathrm{Pa}}$. The films were evaporated from permalloy wire source material melted in alumina coated tungsten boats at a deposition rate of 6.5 nm/s. $Ni_{0.8}Fe_{0.2}$ films were deposited onto the circular paddles of resonator chips. We prepared four $Ni_{0.8}Fe_{0.2}$ samples of 500 nm thick with a 5 nm copper cap layer on the $Ni_{0.8}Fe_{0.2}$ film surfaces to avoid possible oxidation of the film. The dimensions of the resonator chip are described in Table I. The film thicknesses were measured with a profilometer to be 500 ± 1.75 nm.

IV. RESULTS AND DISCUSSION

Figure 4 shows the representative hysteresis loop of one of the samples as measured with an alternating gradient magnetometer (AGM). We determined the average magnetic moment for four samples to be 4.67 ± 0.46 μA m^2, after calibration with a Ni foil standard sample. The four plots of $1/(f^2-f_0^2)$ vs $1/H$ for the films are shown in Fig. 5. The magnetic moments of the films were estimated from the slope of plots of $1/(f^2-f_0^2)$ vs $1/H$, by use of Eq. (5). Thermal drift, apparent in the data that is probably due to the thermal sensitivity of the modulus of silicon, indicates the need for better temperature control in order to avoid phase drifting caused by the heat generated by the electromagnet. The average estimated magnetic moment is 5.04 ± 0.37 μA m^2 for all the samples, which is higher by 7.3% than the average of AGM measurements. The source of this apparent systematic difference will be investigated in future work. We estimated the anisotropy energy constant k_E was 143.7 ± 6.4 J/m^3, according to the intercept as $1/H_0\rightarrow0$ on the $1/(f^2-f_0^2)$ axis. k_E is dependent on deposition method, film thickness, stress, and composition. We have not performed static torque measurements or measured perpendicular hysteresis loops on our samples and therefore cannot comment on the source of the anisotropy in our films.

V. CONCLUSIONS

The MEMS chip described in this article provides a promising method for accurately measuring the magnetic moment of thin films. We conclude that torsional resonator measurements of m for reference samples with moments as small as 1 μA m^2 are valid warranting further development of a standard reference for ultra low moment calibration applications. Torsional resonators provide a simple accurate way to measure magnetic moment directly independently of M_s or k_E. The accuracy depends on how well we can measure the resonant frequency f_0 and the moment of inertia I of the silicon resonator, as well as the applied magnetic field H_0. The resonance frequency f_o can be measured to within an accuracy of a few microhertz using a rubidium atomic frequency reference, and the magnetic field can be measured accurately to within a few tenths of a milliampare per meter (a few micro-oersteds) with a frequency-based nuclear magnetic resonance magnetometer. The moment of inertia on the other hand represents an experimental challenge. The calculations of I for this article rely on accurate knowledge of the dimensions of the silicon resonator and the density of silicon. At best, we could hope for a confidence level of a few percent based on measurements of the dimensions of the resonator, as defined by microlithography accurate to within a few micrometers. Alternatively, the spring constant k_0 could be measured by independent means and then, using Eq. (1), combined with an accurate measurement of f_0 to determine I. In the future we will explore techniques to measure k_0 accurately to within 0.01%. One intriguing possibility is to look at the thermomechanical noise spectrum of the resonator.[6]

[1] G. F. Dionne and J. F. Fitzgerald, IEEE Trans. Magn. **21**, 1590 (1985).
[2] J. A. Weiss, D. R. LaFosse, B. S. Staveley, and G. F. Dionne, IEEE Trans. Magn. **27**, 4855 (1991).
[3] J. Morillo, Q. Su, B. Panchapakkesan, and M. Wuttig, Rev. Sci. Instrum. **69**, 3908 (1998).
[4] D. Sarid, *Scanning Force Microscopy with Applications to Electric, Magnetic, and Atomic Forces* (Oxford, New York, 1991), pp. 19–38.
[5] D. Rugar, H. J. Mamin, R Erlandsson, J. E. Stern, and B. D. Terris, Rev. Sci. Instrum. **59**, 2337 (1988).
[6] L. Hutter and J. Bechhoefer, Rev. Sci. Instrum. **64**, 1868 (1993).