Ferromagnetic resonance spectroscopy with a micromechanical calorimeter sensor

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We describe a new type of ferromagnetic resonance (FMR) spectroscopy that is based on a calorimeter sensor. We use an atomic force microscopy cantilever coated with a ferromagnetic thin film as a bimaterial sensor to measure absorption of microwaves at 9.17 GHz. The spectra show a peak in the cantilever deflection as a function of applied magnetic field corresponding to a peak in the absorbed microwave power that occurs at the FMR resonance of the ferromagnetic film. The saturation magnetization M_{eff} and the damping factor α were determined from the FMR microwave absorption spectra for Co, NiFe, and Ni thin films. The data correlate well with conventional FMR spectra taken with a tuned cavity spectrometer. Our instrument can detect magnetic moments as small as $1.3\times10^{-12}\,\mathrm{A\,m^2}$ (1.3×10^{-9} emu) with prospects for sensitivity improvements to the $1\times10^{-16}\,\mathrm{A\,m^2}$ (1×10^{-12} emu) level. The technique provides a potentially superior way to make quantitative measurements of saturation magnetization of thin-film samples with very small total magnetic moments. [S0034-6748(00)02908-7]

I. INTRODUCTION

In this article we discuss the principles of calorimeter detection of microwave absorption with a micromechanical bimaterial sensor as applied to ferromagnetic resonance (FMR) in magnetic films. Calorimeter sensors have been demonstrated to be viable detectors for studying nuclear magnetic resonance[1] and electron spin resonance.[2] The sensitivity of these instruments was demonstrated to be superior to conventional magnetic resonance probes when measurements were performed at 4 K. In these experiments thermal absorption is typically measured with a small resistance bolometer attached to a small sample in order to minimize thermal masses and maximize thermal response.

More recently, thermal properties of small samples have also been measured with micromechanical calorimeter sensors. In particular, Barnes *et al.*[3] demonstrated picowatt photothermal absorption spectroscopy with a bimaterial cantilever sensor in air at room temperature. The picowatt sensitivity of this instrument depends on atomic force microscopy (AFM) technology for detecting cantilever deflections at the picometer level.

The work reported in this article is the demonstration of FMR spectroscopy with a calorimeter sensor and the application of a bimaterial cantilever sensor for detecting FMR. We describe our instrument, compare our microwave absorption FMR spectra to conventional tuned-cavity FMR spectra of the same samples, review the principles of calorimeter spectroscopy with a bimaterial micromachined sensor, and, finally, discuss the ultimate sensitivity of this type of instrument and relevant applications.

II. EXPERIMENT

Figure 1 shows the experimental configuration. A commercial Si cantilever coated on one side with a thin ferromagnetic film is positioned about 200–300 μm above a microstrip resonator. The resonant frequency of the resonator is 9.17 GHz. The resonator was made from a commercially available epoxy-ceramic composite 0.635 mm thick with a 20-μm-thick copper cladding on both sides. The substrate material has a dielectric constant of 9.7 and a loss tangent of 0.003 at 10 GHz, as specified by the manufacturer. The copper cladding was patterned with photolithography and subsequently etched in an $FeCl_2$ etching solution to form a microstrip resonator 0.5 mm wide and 6 mm long. Microwaves are coupled into the resonator through a 30 μm gap from an adjacent microstripline. The cantilever and the resonator are mounted on the kinematic stage of a commercially available AFM. The AFM head is, in turn, mounted in a precision electromagnet capable of sweeping the field up to 1.2 T. The field is swept at a rate of 0.2 mT/s. The figure also shows the orientations of the microwave field and the sweep field. The magnetic film sample is saturated by the sweep field H_0 oriented in the plane of the film at 90° to the long axis of the cantilever. The microwave field H_1 is not constant throughout the sample, changing direction and magnitude as a function of position along the film, as shown in the figure. However, H_1 has the proper orientation for FMR, perpendicular to H_0 throughout the sample.

The detection electronics are similar to those typical of optical chopping methods developed for photo absorption

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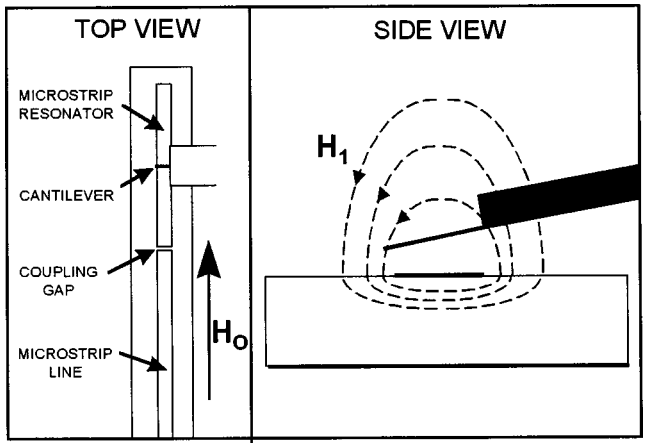

FIG. 1. Top view and side view of the experimental configuration for FMR thermal absorption spectroscopy. Note that the magnetic film samples are deposited on the upper side of the cantilever.

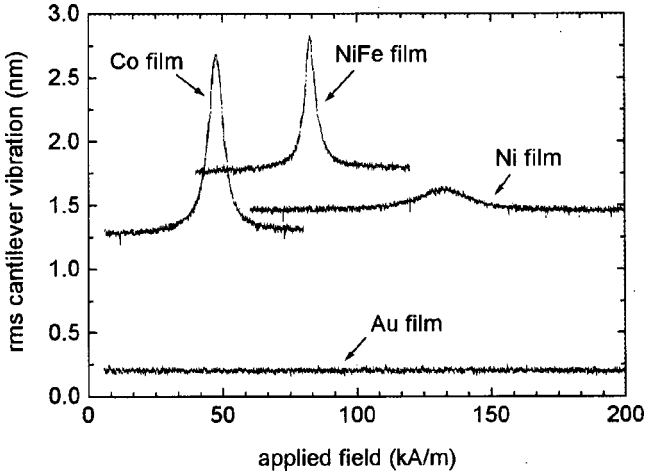

FIG. 3. Cantilever vibration vs applied field showing microwave absorption in Co, NiFe, Ni, and Au thin film samples.

experiments. The schematic diagram is shown in Fig. 2. We monitor the deflections of the cantilever with a laser beam-bounce method for measuring cantilever vibration. A diode laser source is focused onto the cantilever and reflected onto a split photodiode detector. This system is commonly found in commercial AFM instruments and is capable of detecting 10 pm vibrations under ambient conditions. The microwave output from the sweeper is amplitude modulated by a 1 kHz square wave. The square wave also serves as the reference for a lock-in amplifier that measures the difference output from the split photodiode detector. The lock-in time constant is set to 100 ms. The reflected wave from the microstrip resonator is motored with the tuning scope. The frequency is adjusted to obtain a minimum reflected wave amplitude as measured by the radio frequency detector, indicating a maximum coupling of microwave power into the micostrip resonator.

We prepared samples by depositing 30 nm films of either Co, NiFe alloy (81% Ni), Ni, or Au onto the flat sides of commercially available 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 deposition rate of 0.15 nm/s. The cantilever dimensions were $2.5\,\mu\text{m}\times49\,\mu\text{m}\times449\,\mu\text{m}$ with a spring constant of 0.35 N/m and a resonant frequency of 17 kHz.

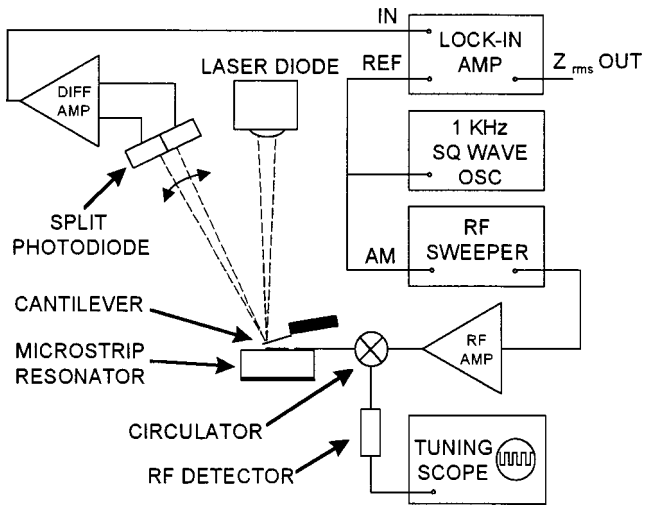

FIG. 2. Detection electronics for FMR thermal absorption spectroscopy.

III. RESULTS AND DISCUSSION

Figure 3 shows the FMR microwave absorption spectra of Co, NiFe, and Ni. We determined the peak locations and widths by fitting the data to Lorentzian absorption lines. The effective saturation magnetization M_{eff} (including anisotropy terms), the damping factor α, and the imaginary part of the susceptibility χ'' (at resonance), can be calculated using the following FMR relationships for a thin-film sample geometry in SI units:[4]

$$M_{\text{eff}}=\frac{1}{H_r}\left(\frac{\omega^2}{\gamma^2}-H_r^2\right), \tag{1}$$

$$\alpha=\frac{\gamma}{2\omega}\Delta H, \tag{2}$$

and

$$\chi''=\frac{M_s}{\alpha}\frac{\gamma}{\omega}\left(\frac{M_{\text{eff}}+H_r}{M_{\text{eff}}+2H_r}\right). \tag{3}$$

Here, H_r is the resonant field, γ is the gyromagnetic constant, ΔH is the width of the resonance peak at half maximum as determined by the Lorentzian fit, and $\omega=2\pi f$ is the microwave frequency. The results are shown in Table I. We have also included a summary of the FMR data obtained for the same samples with a conventional resonant cavity spectrometer in Table I. Note that $M_{\text{eff}}=M_s-8\pi K_s/M_s$ where M_s is the saturation magnetization and K_s is the uniaxial surface anisotropy energy density parameter.[5] The uniaxial surface anisotropy term $8\pi K_s/M_s$ is about 20 kA/m for a 30 nm ferromagnetic film and negligible to first order compared to M_s for these measurements. We therefore assume $M_{\text{eff}}=M_s$. Generally, the M_{eff} and α values agree for the two types of FMR measurements, but with some differences possibly due to the different detection methods. The resonant

TABLE I. Comparison of FMR data—microwave absorption vs tuned cavity detection. Note: $\gamma = 2.31 \times 10^8$ rad/s (kA/m)$^{-1}$.

Sample	H_r (kA/m)	ΔH (kA/m)	f (GHz)	M_{eff} (kA/m)	α	χ''
Co[a]	47.6	6.8	9.17	1260	0.014	348
Co[b]	60.6	10.4	9.88	1130	0.021	205
NiFe[a]	82.7	5.1	9.17	672	0.010	242
NiFe[b]	93.9	5.7	9.88	676	0.011	219
Ni[a]	132.9	20.0	9.17	334	0.040	26
Ni[b]	154.1	27.4	9.88	326	0.055	18

[a]Microwave absorption.
[b]Resonant cavity.

cavity FMR spectrometer measures the derivative of the absorption line as a function of field as opposed to the microwave absorption spectrometer described here which measures the absorption line directly. In addition, the cantilever chip has several small indentations so that some portions of the magnetic films are at odd angles relative to the applied field. This also tends to broaden the FMR line as observed with the resonant cavity instrument.

A strong background signal is present in the FMR microwave absorption spectra (see Fig. 3). We have observed similar differences in offset levels in three different sets of samples and therefore conclude that the offsets are related to material-specific properties. In particular, we believe that the background levels are different for different materials due to their specific microwave absorption coefficients off-resonance and their specific elastic properties as discussed below.

Figure 4 shows several FMR microwave absorption spectra for a 30 nm Co film at different microwave input power levels. Figure 5 shows the peak and the offset microwave absorption determined by fitting the data to Lorentzian line shapes. Both peak and offset cantilever vibration levels follow a linear dependence on microwave input power with an intercept close to zero. We observe a slight upward shift in H_r from 47.5 kA/m for power levels above 5 dB. At 14 dB H_r shifts to 48.0 kA/m. This may be an indication of nonlinear FMR effects in the film at higher power levels.

The earlier data can be understood within the mathematical framework developed for bimaterial thermal sensors. Consider a rectangular beam fixed at one end comprised of two layers that have different thermal properties. Barnes *et al.*[4] solve the heat equation for this configuration and show that the deflection at the free end of the beam is

$$z = \alpha \frac{E_1}{E_2} \frac{t_1^2 l^3}{t_2^3 w} \left(\frac{\gamma_1 - \gamma_2}{\lambda_1 t_1 + \lambda_2 t_2} \right) P, \tag{4}$$

where γ, λ, t, w, l, and E are, respectively, the thermal expansion coefficient, thermal conductivity, thickness, width, length, and Young's modulus of the beam layers (subscripts refer to the different materials) and P is the absorbed power. Equation (4) applies only in the limit $t_1 \ll t_2$ (t_1 is thickness of the magnetic film and t_2 is the thickness of the silicon cantilever). Also, Barnes *et al.* assume that the temperature is constant over any cross section along the axis of the cantilever—this is a good approximation if t_1, $t_2 \ll l$. The constant α ranges from a value of 2, if power is absorbed near the end of the beam, to a value of 1.25, if power is absorbed uniformly along the beam.

Let us first consider the significant ''off-resonance'' microwave absorption background discussed earlier. The canti-

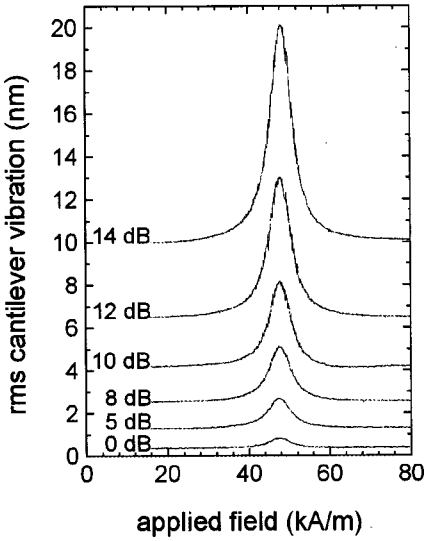

FIG. 4. Cantilever vibration vs applied field for a 30 nm Co film at different relative input microwave power levels.

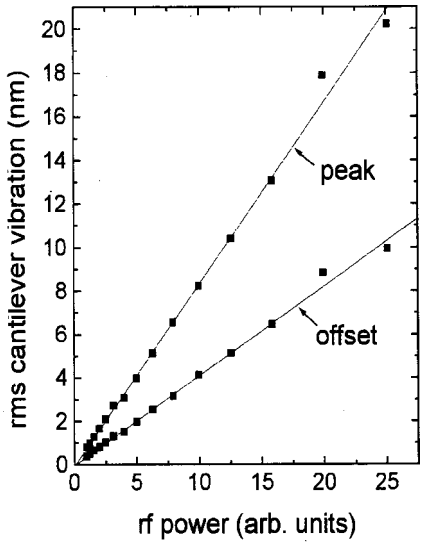

FIG. 5. Lorentzian peak and offset cantilever vibration vs input power for a 30 nm Co film (derived from the data in Fig. 4).

lever geometry for each sample is nearly the same (all cantilevers came from the same batch). Upon inspection of Eq. (4) we see that the differences in microwave absorption background levels should stem primarily from differences in P, E_1, and γ_1. Presumably, differences in P stem from differences in off-resonance heating of the ferromagnetic films that have a different resistivity ρ. In these experiments the skin depth $\delta=(2\rho/\mu\omega)^{1/2}$ is about 1 μm (μ is the permeability of the film; note that for saturated magnetic films $\mu=\mu_0$, the permeability of vacuum). The alternating current (ac) power loss for a thin metal film scales with ρ when $t_1<\delta$ as is the case here.[6] For Co, NiFe, and Ni, the literature bulk values for ρ are 6.2, 17 ± 4, and 7.1 $\mu\Omega$ cm, respectively. γ_1 is $13\pm1\times10^{-6}\,\mathrm{K}^{-1}$ and E_1 is 200 ± 10 GPa for all three metals. We also measured the background thermal absorption level for a 30-nm-thick Au film (see Fig. 3). Au has a similar γ_1 of $14\times10^{-6}\,\mathrm{K}^{-1}$ compared to the magnetic film samples but ρ and E_1 are significantly lower with values of 2.5 $\mu\Omega$ cm and 78 GPa. Under these conditions we expect that the offsets of the absorbtion peaks observed in Fig. 3 for these materials should scale approximately as ρE_1. This scaling trend is generally satisfied with the exception that the offset level for the NiFe film is somewhat lower than expected.

The power absorbed at resonance can be derived from the peak cantilever vibration value using Eq. (4) and compared to the theoretical absorbed power determined from our experimentally determined χ''. An estimate of the average magnitude of H_1 at a given input power level can be made based on the off-resonance cantilever vibration values given the resistivity of the magnetic film. In this way we can check the data for self-consistency. For Co film measurements at a power level of 10 dB we find that $P_{\mathrm{offset}}=1.28\times10^{-4}$ W and $P_{\mathrm{peak}}=1.29\times10^{-4}$ W given the parameters $z_{\mathrm{offset}}=4.10\times10^{-9}$ m, $z_{\mathrm{peak}}=4.08\times10^{-9}$ m, $a=\frac{1}{2}(1.25+2)=1.6$, $E_1=209\times10^9$ Pa, $E_2=47\times10^9$ Pa, $t_1=30\times10^{-9}$ m, $t_2=2.5\times10^{-6}$ m, $I=449\times10^{-6}$ m, $w=49\times10^{-6}$ m, $\gamma_1=13\times10^{-6}\,\mathrm{K}^{-1}$, $\gamma_2=2.6\times10^{-6}\,\mathrm{K}^{-1}$, $\lambda_1=100\,\mathrm{W\,m}^{-1}\,\mathrm{K}^{-1}$, and $\lambda_2=150\,\mathrm{W\,m}^{-1}\,\mathrm{K}^{-1}$. Assuming $P_{\mathrm{offset}}=P_{\mathrm{ac\ loss}}=A\rho/t_1$, H_1^2 for a metal film with $t_1\ll\delta$,[6] we estimate that the average $H_1=53$ A/m, given the following parameters for the Co film sample: $A=2.2\times10^{-8}\,\mathrm{m}^2$, $\rho=6.2\times10^{-8}\,\Omega$ m, and $\mu=\mu_0=4\pi\times10^{-7}$ H/m. By definition, $P_{\mathrm{FMR}}=\mu_0\omega A t_1\chi'' H_1^2=4.7\times10^{-5}$ W, given $H_1=53$ A/m. P_{FMR} is about about one third of P_{peak}. This is within reasonable agreement given (1) the lack of a detailed description of the vector field H_1 in the vicinity of the cantilever, (2) our assumptions regarding microwave absorption in thin metallic films, and (3) the use of bulk Co parameters to describe the characteristics of thin film samples.

We estimate the magnetic moment sensitivity of our instrument to be $\Delta m=At_1M\,\Delta s/s$, where M is the sample magnetization from Table I and $\Delta s/s$ is the experimentally determined signal-to-noise ratio (SNR) at the 1σ level. Given $M=1\times10^5$ A/m and $\Delta s/s=0.020$, then $\Delta m=1.3\times10^{-12}\,\mathrm{A\,m}^2$. This value compares favorably to resonant cavity FMR measurements with reported sensitivities of $1\times10^{-7}\,\mathrm{A\,m}^2$ and the best reported FMR sensitivity based on inductive detection of $1\times10^{-13}\,\mathrm{A\,m}^2$ for the coplanar waveguide transmission measurements described by Zhang *et al.*[7] More recently Koch *et al.*[8] and Russek *et al.*[9] have measured magnetization dynamics using sensors based on spin-polarized tunneling with demonstrated sensitivities of $1\times10^{-15}\,\mathrm{A\,m}^2$.

The potential for improving the sensitivity of FMR microwave absorption spectroscopy as described here lies in both optimizing the cantilever geometry and modulating the microwave field at the cantilever resonance frequency. Some important issues to consider are as follows. The thermal relaxation time constant τ of the sensor can be estimated from the ratio of excess energy stored in the cantilever divided by thermal conduction to the chip[6]

$$\tau=\frac{l^2}{2}\left(\frac{\rho_1C_1t_1+\rho_2C_2t_2}{\lambda_1t_1+\lambda_2t_2}\right). \tag{5}$$

Here, ρ is the density and C is the heat capacity of each of the beam layers (subscripts refer to different materials). For Co $\rho_1=8.9\times10^3\,\mathrm{kg/m}^3$ and $C_1=420\,\mathrm{J\,K}^{-1}\,\mathrm{kg}^{-1}$, and for Si $\rho_2=2.3\times10^3\,\mathrm{kg/m}^3$ and $C_2=714\,\mathrm{J\,K}^{-1}\,\mathrm{kg}^{-1}$, and we find $\tau=1.1\times10^{-3}$ s. This is consistent with our observation of significant rolloff in the cantilever vibration signal at 1 kHz microwave power modulation rate. τ puts significant constraints on cantilever geometry since we would like to operate at the resonance frequency ω_c of the cantilever to take advantage of the potentially high mechanical Q of a micromechanical resonator for improved SNR. SNR $\propto(\omega_cQ/k)^{1/2}$ for a mechanical resonator with resonant frequency ω_c and spring constant k.[10] For a rectangular cantilever $\omega_c\approx(t/t^2)(E/\rho)^{1/2}$ and $k\approx Ewt^3/4l^3$.[11] Given that the thermal conductivities and heat capacities of the two materials in the cantilever are similar we see from Eq. (5) that τ is proportional to l^2 and more or less independent of t_1 and t_2. In addition, the signal for a given microwave power level is proportional to l^3 [see Eq. (4)]. We therefore conclude that, to improve sensitivity we must accept the low τ limitation and develop very thin cantilevers with a lower ω_c in the 1 kHz range. This will also lower k and thus further increase the SNR as discussed earlier.

The Brownian motion of the cantilever fundamentally limits its ultimate sensitivity. However, there are several other sources of noise that must be reduced before the Brownian motion limit can be achieved. In particular, we have observed substantial noise contributions from the laser diode, the photodiode detector, the microwave source, room vibration, acoustic coupling, and air convection. Operating in vacuum would not only reduce viscous damping and thus increase Q but help decrease acoustic and convection noise sources as well. The Q for bare, single-crystal, silicon cantilevers can be well over 10^4. If the Brownian motion limit can be achieved and we can fabricate coated cantilevers with a Q of 10^4 then it should be possible to realize sensitivities of 1 pW at room temperature as discussed by Barnes *et al.*[3] Given the definitions $P_{\mathrm{FMR}}=\mu_0\omega At_1\chi'' H_1^2$ and $m=At_1M$, then $m=P_{\mathrm{FMR}}M/(\mu_0\omega\chi'' H_1^2)$. For a Co film sample with a H_1 of 53 A/m and a power sensitivity of 1 pW we conclude that it should be possible to measure magnetic moments as small as $2.5\times10^{-16}\,\mathrm{A\,m}^2$ with thermal absorption FMR.

This corresponds to 30-nm-thick Co sample with an area of $A = 2.6 \times 10^{-16}\,\mathrm{m}^2$ (16 nm×16 nm).

IV. FUTURE PLANS

We have demonstrated FMR spectroscpy with a micromechanical calorimeter sensor for the first time. The instrument currently has a magnetic moment sensitivity of $1.3 \times 10^{-12}\,\mathrm{A\,m}^2$ with the potential for substantial sensitivity gains at room temperature by operating in vacuum and improving cantilever geometry. In the future we plan to combine microtorque magnetometry with FMR thermal absorption spectroscopy in one apparatus. In this way we will be able to perform $M-H$ loops on nanoscale samples and calibrate the saturation magnetization using FMR.

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