A torque magnetometer for thin films applications

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A R T I C L E   I N F O

Article history:
Received 6 September 2011
Received in revised form 28 November 2011
Available online 9 December 2011

Keywords:
Torque magnetometry
Magnetic anisotropy
Thin film

A B S T R A C T

We describe the development of an automatic torque magnetometer based on a torsion pendulum. The instrument uses the capacitive arrange developed by Randall D. Peters, Rev. Sci. Instr. 60 (8), 2789 (1989), as sample's angular position sensor. The instrument performance is illustrated by measuring the in plane magnetic anisotropy of Co thin films and systems with exchange-bias. It possesses a sensitivity of $10^{-10}$ Nm and is capable to determine anisotropy constants in magnetic films as thin as 3 nm. The instrument design and the measurement procedures are presented.

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1. Introduction

Torque magnetometry is known as a powerful tool to determine and analyze the magnetic anisotropy in magnetic materials. Weiss [1], Webster [2] among others have built torque magnetometers to study bulk magnetic materials. The torque data were collected manually and a full torque measurement usually required several hours. Later [3,4] the instruments incorporated some kind of automation, becoming faster, with higher precision and reliability. The torque technique was applied to the study of magnetic thin films by Boyd [5] and Humphrey [6]. The equipments required increased sensitivity in order to work with such low magnetic moments. Since then torsion cantilevers and piezoelectric cantilevers have been used in order to obtain a greater sensitivity [7-9]. Nowadays, the torque magnetometry is still used to determine both in plane and out of plane anisotropy constants in magnetic thin films [10,11], also at low temperatures [12], reaching sensibilities of $10^{-13}$ Nm as in the instrument described by Rossel and co-workers [8] based on a torsion cantilever. Systems based on piezoelectric sensors present very good sensitivities, but also temperature and magnetic field dependency, which must be regarded in the development of the instrument [8].

We have developed, and described in this work, an automated torque magnetometer based on a torsion pendulum, capable of investigating the magnetic torque and anisotropies on samples as thin as 3 nm. By providing an appropriate tension on the suspending wire, samples with larger magnetic moment can also be measured. In addition the instrument is capable of measuring out of plane and in plane anisotropies, by an appropriate positioning of the sample relative to the applied field. This feature permits the study of magnetic system such monocrystalline thin films or complex structures such as that with exchange-bias [13], which still present some controversy on the origin of the energy terms [14]. The sample's angular position sensor that was used is that developed by Peters [15], and presents a very low cost, simple design and high angular sensitivity. The sensitivity obtained was $10^{-10}$ Nm, which can be increased by reducing the environmental mechanical noise. This sensibility is comparable to the best instruments already developed based in torsion pendulum. The instrument described is capable of measuring the full angular torque range in magnetic films in a few minutes. To demonstrate the final performance of the instrument, we show planar torque measurements in thin Co films with thickness within the range from 5 to 50 nm and a film presenting exchange-bias.

2. Torquemeter basics

The torque magnetometry technique consists basically in applying a magnetic field in a known direction relative to the sample, and detecting the torque produced. If the sample is free to rotate, it will try to align its magnetization direction with the applied field direction. The torque is detected by measuring the restoring torque needed to keep the sample in the original position.

The system is based on a torsion balance, and a sketch is presented in Fig. 1. Helmholtz coils (item 1 in Fig. 1) generate a homogeneous magnetic field at the sample's position (item 2). It is worth to note that using these coils we can keep the sample always under an homogeneous magnetic field. The coils' base is free to rotate around the sample (item 3) and the direction of the
applied field is controlled by a stepping-motor (item 4). The field direction can be measured by a multi-turn potentiometer mechanically coupled to the coils' base (item 5). Item 6 in Fig. 1 encloses the sample's angle sensor, which is rigidly connected to the sample and the torsion balance.

The torsion balance is detailed in Fig. 2. The sample holder can be seen at the bottom of the figure (item a in Fig. 2). The sample can be mounted with its plane coplanar or orthogonal to the plane defined by the applied field direction when the Helmholtz coils are moved, for in plane or out of plane anisotropy investigation, respectively.

The sample holder is connected to the angular position sensor [15] (item b) through a Pyrex rod (item c) and the set is suspended by a non magnetic wire (item d), which contours the sample holder as a small coil. The suspending wire (diameter $= 50 \mu m$) can be tensioned by the screws at the top and at the bottom (item e). The ideal tension of the suspending wire depend on the measuring torque range, as higher is the wire's tension the higher the maximum torque that can be measured. An air damping system to prevent angular oscillations (item f) is attached to the Pyrex rod in order to minimize the stabilization time.

When a magnetic field ($\mathbf{H}$) is applied to the sample, the torque ($\tau$) works to turn the sample around the suspending axis. In order to determine the torque exerted, a current ($I$) is fed to the small restoring coil (through the suspending metallic wire), which will produce an additional torque that keeps the sample in its original position. This position is measured by the angular position sensor (item b in Fig. 2).

The torque can be determined by:

$$\tau = m \times \mu_0 H,$$

where $\mu_0$ is the vacuum permeability, $m$ is the magnetic moment of the restoring coil, related to the geometry (number of turns $N$ and cross section $A$) and applied current ($I$) by the expression:

$$m = NI/An$$

$n$ is a unit vector orthogonally aligned to the coils plane. In order to obtain the anisotropy constants, the measured torque curve must be fitted to the proper torque equation, according, for example, the sample's crystalline structure (see Ref. [16] for torque versus angle expressions of cubic magnetocrystalline anisotropies with the field applied at different planes).

A critical feature in determining the torque is to measure the angular position of the sample. To overcome this difficulty the angular position sensor developed by Peters [15] was used as depicted in the Fig. 3(a). The sensor is composed by three coaxial disks made of metallic coated circuit board as sketched in the figure. The central disk (item 2 in Fig. 3(a)) is attached to the Pyrex rod, being free to rotate with the sample. Disks 2 and 3 form a capacitive bridge. If the bridge is initially balanced, motion of the disk 2 relative to the 3 will unbalance the bridge, and this can be measured by a lock-in amplifier connected to disk 1. The equivalent electric circuit of the capacitive bridge is shown in Fig. 3(b). A signal generator supplies the reference signal to the capacitive bridge ($4 \text{ V at } 30 \text{ kHz}$) and a lock-in amplifier reads the output voltage, which is proportional to the angular position of the sample. As the sensitivity of the angular position sensor is inversely proportional to the distance between the disks, the central disk had its thickness reduced which permits an additional approximation between them.
saturation field $H_C$, $M = M_S$, and the torque increases linearly with the applied field, allowing one to obtain $M_S$.

Another kind of measurement requires the angle between $n$ and $H$ to be variable, and allows one to obtain, for example, the anisotropy constants in the plane of the film. At certain angles the counter-torque current needed to maintain the position of the sample might be too high. This would increase the temperature of the suspending wire, and would modify the separation between the plates of the angular position sensor due to thermal expansion of the wire. In cases like these, it is more convenient to let the sample rotate under the torque promoted by the applied field aligning itself to the equilibrium direction.

By knowing the torsion constant of the suspending wire and the angular dislocation of the sample, the torque can then be easily measured. This procedure requires calibration of both the wire’s torsion constant and the angle-to-voltage ratio of the angular position sensor. These can easily be made with the help of the torque produced by the restoring coil and by a slight rotation the magnetic field around the sample.

The position sensor is calibrated placing the sample plane aligned to the suspending wire. A magnetic field parallel to the sample plane, high enough to saturate the sample, is applied. As the sample’s magnetization and the magnetic field are aligned, no torque is produced. As the magnetic field is slightly dislocated from its initial position, the sample will rotate accompanying the field. As the restoring torque from the torsion wire is much smaller than the magnetic torque, the angular displacement of the field and the sample can be considered the same. By measuring the output voltage of the position sensor ($V_{sensor}$) at several angles relative to the initial position, a linear relation can be verified, and it is used as the angle-to-voltage ratio of the angular position sensor.

To calibrate the wire’s torsion constant ($k$), a two step process is needed. Without a sample in the sample holder and with a known current flowing through the restoring coil a known magnetic moment, $m_m$, is generated. When $H$ and $m_m$ are aligned, no angular displacement is observed when the magnetic field intensity is changed. Next, $H$ is rotated by a known angle ($\theta_0$), which will produce a torque over $m_m$, and the new equilibrium position is established when the magnetic torque is counter-balanced by the torsion of the suspending wire or

$$m_mC_H \sin(\theta_H - \theta_m) = k \theta_{max} = aV_{sensor},$$

where $a$ is the proportionality constant between torque and $V_{sensor}$, $\theta_m$ is the angular displacement of $m_m$, which is known from the anterior calibration procedure. So the torque over the counter torque coil can be determined and related to $V_{sensor}$.

At this point it is important to point out that the measurable maximum torque it is associated to the $k$ parameter, which is proportional to the suspending wire’s stress, and it must be adjusted to retain the Eq. (3) in the linear regime for the desired torque range and better resolution. The angular resolution obtained was of $10^{-7}$ rad.

Fig. 5 presents a measurement of the planar torque obtained from a circular Co sample 5 nm thick and 6 mm in diameter. Open circles are the experimental data corrected for the sample rotation (the original experimental data, before correction for the sample rotation, is shown as a dashed blue line). The solid red curve was obtained fitting the first three even terms from a Fourier series expansion, used to obtain the anisotropy constants. It can be seen from the figure that even for this very thin sample, the noise (roughly the size of the symbols) is much smaller than the signal from the measured torque. It is worth to note that the studied samples are textured with the c-axis orthogonal to the sample’s plane; also the measured effective anisotropy is an
in-plane one induced during the sample’s growth by the field of the magnetron sputtering.

The fitting of a Fourier series to the data shows a strong component from a uniaxial anisotropy (2nd order), and additional terms of 4th and 6th orders. As the thickness of the Co film is increased from 5 to 50 nm, a gradual reduction of the higher order terms is observed, as shown in Figs. 5, 6 and Table 1. Just the term relative to the uniaxial anisotropy is present for films 50 nm and thicker.

Another example of application of torque magnetometer requiring high sensitivity in function of the reduced magnetic moment is the study of systems with exchange bias. Fig. 7 presents the torque curves obtained from a NiFe/FeMn sample at several applied fields. It can be seen from the figure an evolution of torque curves from a behavior predominantly unidirectional to another predominantly uniaxial.

4. Conclusions

A torque magnetometer to study the magnetic properties of thin films has been built. One of the most important features of the instrument is the possibility of self-calibration. The instrument’s sensitivity is high enough to measure the planar torque of thin films Co samples as thin as 5 nm, and bilayers presenting exchange bias. The smallest torque that can be usefully measured is approximately $10^{-10}$ Nm. The ultimate sensitivity could be improved further by reduction of the mechanical noise from the surrounding environment.

Acknowledgments

We would like to thanks to Prof. Lucio S. Dorneles for valuable suggestions and the critical reading the manuscript. This work was supported by the Brazilian agencies CNPq, CAPES and FAPERGS.

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