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A NEW METHOD FOR MEASURING M-H HYSTERESIS LOOPS OF UNIAXIALLY MAGNETIC MATERIALS

Jeen Hur and Sung-Chul Shin
Department of Physics, Korea Advanced Institute of Science and Technology, Kusung-Dong, Yusung-Gu, Taejon 305-701, Korea

Abstract–We have developed a new method for measuring the M-H hysteresis loop of a spheroid-shape magnetic material having a uniaxial anisotropy and discussed its accuracy at fields near the coercivity. Our torque magnetometric method simultaneously gives the saturation magnetization and the remnant magnetization. Furthermore, the coercivity depending on the applied field orientation is accurately measured by this simple technique. An accuracy of the present method is negligibly affected even at fields near the coercivity, where the magnetization is not uniform. The technique makes a torque magnetometer an extremely high sensitive tool for measuring M-H hysteresis loop.

I. INTRODUCTION

Measuring the M-H hysteresis loop is essential to understand and develop magnetic materials. The vibrating sample magnetometer (VSM), the Faraday balance (FB), the superconducting quantum interference device (SQUID) magnetometer, and the vibrating reed magnetometer (VRM) are well known tools for measuring the M-H hysteresis loop. The VSM is a very robust and versatile one widely used to measure the magnetic moment down to 10^{-5} emu [1]. The FB has a higher sensitivity by a factor of 10 [2]. However, the FB is not proper for measuring the absolute value of the magnetic moment because of the difficulty of determining the applied field and its gradient at the position of a sample. The SQUID magnetometer has been a most sensitive instrument during decades [3]. It has a high sensitivity of about 10^{-8} emu. The VRM was developed for measuring the magnetic moments of microscopic particles [4]. More improved variations of VRM, today, compete the SQUID magnetometer in the sensitivity [5-7]. Despite of high sensitivities of the SQUID magnetometer and the VRM, they are not popularly used. This is partially due to lacking of versatility: the SQUID magnetometer gives the M-H hysteresis loop only by point-by-point measurements which are time consuming, because a superconducting shield should be removed whenever an applied field is varied. In addition, it requires very expensive liquid He even at an elevated temperature. The VRM could not show a high sensitivity in measuring the magnetic moment of a large sample such as a thin film on the substrate. The mass and the surface area of a millimeter-sized substrate are large enough to drastically degrade the sensitivity of the VRM.

While, the torque magnetometer which measures a torque on the sample in the applied field is commonly used for investigating the saturation magnetization, the coercivity, and the rotational hysteresis loss as well as the anisotropy energy [8]. After R. F. Penoyer's null-type apparatus [9], the sensitivity of the torque magnetometer is greatly improved to be 10^{-6} dyn·cm [10], which is high enough to measure the saturated magnetic moment of a monolayer Ni film. For example, the saturated magnetic moments of Ni films varying in thickness from 3 to 200 Å were measured by C. A. Neugebauer [11].

A highly sensitive method has been recently developed for measuring the M-H hysteresis loop of the uniaxially magnetic material having a high anisotropy field intensity. This method enabled a torque magnetometer having a torque sensitivity of 2×10^{-3} dyn·cm to yield a high sensitivity of 10^{-6} emu in measuring the magnetic moment [12]. This implies that a torque magnetometer having a torque sensitivity of 10^{-6} dyn·cm can give an extremely high sensitivity of 10^{-9} emu in measuring the magnetic moment by the method. In spite of this remarkable high sensitivity, the inaccuracy of approximation used in the method linearly depends on the ratio of the applied field intensity to the anisotropy field intensity. This forces the method to be applicable only for measuring the M-H hysteresis loop of the sample having the high anisotropy field intensity. In this paper, we report a simple and versatile torque magnetometric method to measure the M-H hysteresis loops of spheroid-shape magnetic materials at any applied field orientation and discuss its accuracy at the fields near the coercivity.

II. PRINCIPLES

A. Theoretical Background

A spheroid-shape magnetic material is assumed to have a uniform magnetization. For simplicity, we consider only the first order magnetic anisotropy constant for representing the intrinsic uniaxial anisotropy whose orientation is parallel or perpendicular to a long axis of the spheroid as illustrated in Fig. 1. M, H, and K_i are the magnetization intensity, the applied field intensity, and the first order intrinsic anisotropy constant, respectively. θ and ϕ are the magnetization orientation and the applied field orientation, respectively. All orientation are measured counterclockwise from the long axis. Because of the shape, the shape anisotropy strength is also considered as $\frac{1}{2}NM^2$, where N is the difference between two demagnetization factors along the directions parallel and perpendicular to the long axis. For examples, N equals to 4π for the thin film and 2π for the long cylinder [13,14]. Under the applied field, the total energy of this spheroid E is expressed by

$$E = K\sin^2(\theta - \theta_o) - MH\cos(\phi - \theta) , \quad (1)$$

where K is an effective anisotropy strength defined by $|\frac{1}{2}NM^2 \pm K_i|$ [15]. Here, the plus or the minus sign is taken when the intrinsic anisotropy orientation is parallel or perpendicular to the long axis, respectively. θ_o represents the easy axis orientation, which will be 0 or $\frac{\pi}{2}$. For the example of the thin film having the intrinsic anisotropy perpendicular to the long axis(the film plane), θ_o will be 0 (in-plane easy axis) when $K_i < \frac{1}{2}NM^2$ or $\frac{\pi}{2}$(perpendicular easy axis) when $K_i > \frac{1}{2}NM^2$. Since the magnetization will be oriented to minimize E given by Eq. (1), an equilibrium condition $(\partial E/\partial\theta) = 0$ gives

$$MH\sin(\phi - \theta) = K\sin 2(\theta - \theta_o) . \quad (2)$$

Because the torque τ equals to the vector product of the magnetization by the applied field, τ becomes

$$\tau = MH\sin(\phi - \theta) , \quad (3)$$

where we take the positive sign of torque to exert counterclockwise on the spheroid [16]. From Eq. (2) and (3), one can obtain the magnetization expressed by

$$M = \frac{\tau}{H\sin\left(\phi - \theta_o - \frac{1}{2}\sin^{-1}\left(\frac{\tau}{K}\right)\right)} . \quad (4)$$

Hence, knowing K and θ_o one can obtain the M-H hysteresis loop from the τ-H hysteresis loop at any applied field orientation. It should be emphasized that the accuracy of our method does not changed even in measuring the magnetic moment of the sample having a low anisotropy field intensity.

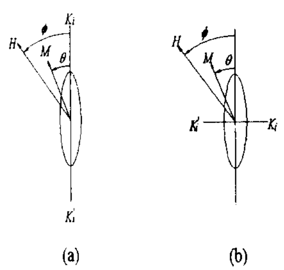

FIG. 1 Coordinates in this torque magnetometry, where K_i-K_i' represents the intrinsic anisotropy orientation (a) when it is parallel to the long axis and (b) perpendicular to the long axis.

B. Measurement Procedure

The standard procedure for measuring the M-H hysteresis loop is as follows: first, measure the easy axis orientation θ_o and the effective anisotropy strength K from plots of the torque vs the applied field orientation as illustrated in Fig. 2. Then, measure the τ-H hysteresis loop after setting ϕ to a desired orientation. Finally one can obtain the M-H hysteresis loop from the τ-H hysteresis loop, θ_o, and K. It should be noted that θ_o can be measured by the applied field orientation ϕ_o where $\tau = 0$ and $(\partial\tau/\partial\phi) > 0$ in the τ-ϕ curve. K can also be measured from τ_p that is defined in Fig. 2 when $H > (H_k/\sqrt{2})$ [17]. Here, the effective anisotropy field intensity is defined by $H_k \equiv (2K/M)$. Then, Eq. (4) can be simply expressed by

$$M = \frac{\tau}{H\sin\left(\phi - \phi_o - \frac{1}{2}\sin^{-1}\left(\frac{\tau}{\tau_p}\right)\right)} \quad (5)$$

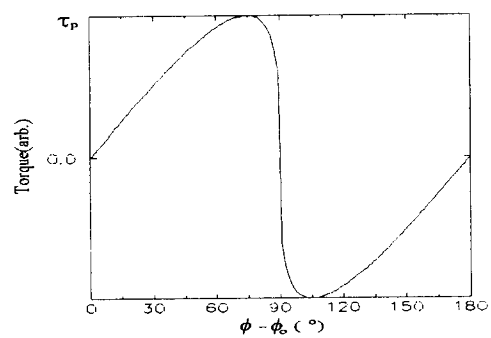

FIG. 2 A typical τ-ϕ curve. Here, ϕ_o equals to the easy axis orientation and τ_p measures the anisotropy strength of a uniaxially magnetic material. Note that τ_p is always positive in our notations.

III. RESULTS AND DISCUSSION

Our method was applied to measure the M-H hysteresis loops of 300-Å-thick Co/Pd multilayer thin films to investigate the sensitivity and of a polycrystalline Ni(99.9%) wire to confirm the accuracy of this method. Details of the preparation technique for Co/Pd multilayer thin films have been published elsewhere [18]. The Ni wire having a diameter of 0.0127 cm and a length of 0.488 cm. A homemade torque magnetometer [19], having 0.002-dyn·cm and 0.02-dyn·cm resolutions in the measurement ranges of 5-dyn·cm and 50-dyn·cm, respectively, has been used.

A. Sensitivity and Resolution

A higher anisotropy field intensity of the sample yielded a higher sensitivity in the M-H hysteresis loop measurement. For example, the M-H hysteresis loop of the (2-Å Co/9-Å Pd)$_{27}$ sample measured at $(\phi - \phi_o) = \frac{\pi}{20}$ is depicted in Fig. 3. The saturation magnetization and the coercivity were 301 emu/cm^3 and 3120 Oe, respectively. Compared to the M-H hysteresis loop shown in the inset in Fig. 3 measured by conventional VSM, our method clearly shows a higher sensitivity and less noise in measuring the magnetic moment. A typical sensitivity of 10^{-6} emu could be achieved by our method. The anomalous peaks near the zero applied field intensity can be eliminated by accurately measuring the applied field intensity or simply shifting the origin of the applied field intensity. The effective anisotropy strength of $K = 1.80 \times 10^6$ erg/cm^3 and the easy axis orientation of $\theta_o = \frac{\pi}{2}$ were obtained from the experimental τ-ϕ curve of (2-Å Co/9-Å Pd)$_{27}$ sample. The effective anisotropy strength and the saturation magnetization gave the anisotropy field intensity of 11700 Oe.

A higher applied field intensity achieved a higher resolution in the measurement of the magnetic moment without any sacrifice of an accuracy. The less noise in the measurement can be seen from Fig. 3 at the higher applied field intensity. This is a remarkable advantage compared to the methods utilizing the torque magnetometers [20–22] and the other magnetometers [1,3,4]. It should be mentioned that this method, with a small value of $(\phi - \phi_o)$, can works well even if the anisotropy field intensity is too high to accurately measure K from τ_p [12].

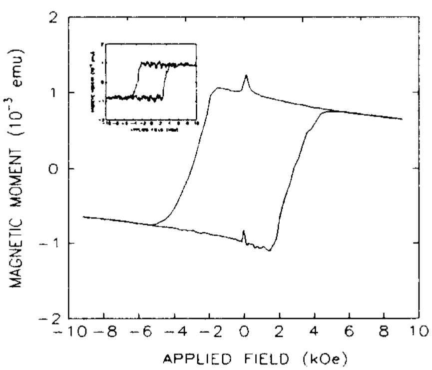

FIG. 3 **The M-H hysteresis loop of the (2-Å Co/9-Å Pd)$_{27}$ multilayer. Inset: the M-H hysteresis loop of the same sample measured by a VSM having a sensitivity of 10^{-5} emu.**

B. Accuracy

$\phi_o = 0$ and $\tau_p = 7.63 \times 10^5$ erg/cm^3 were measured from the τ-ϕ curve of the Ni wire. The M-H hysteresis loop of the Ni wire was obtained from the τ-H hysteresis loop measured at $(\phi - \phi_o) = 7.2°$ as depicted by solid in Fig. 4. The saturation magnetization, the remnant magnetization, and the coercivity were 493, 295 emu/cm^3, and 213 Oe, respectively. Note that K_i of the Ni wire is zero within an experimental error because the value of τ_p is same as that of the shape anisotropy strength.

The accuracy of our method were not degraded even at the fields where the magnetization was not uniform. The present method could have somewhat ambiguity when the magnetization is not uniform. It is mathematically reasonable that the accuracy in the measurement of M becomes low if the value of $\left|\frac{1}{2}NM^2 \pm K_i\right|$ severely varies from τ_p during the measurement of τ-H hysteresis loop. This implies that the worst accuracy is expected when the sample having a zero intrinsic anisotropy constant is measured. However, an inaccuracy was found to be negligibly small in measuring the M-H hysteresis loop of the Ni wire whose intrinsic anisotropy constant is very small.

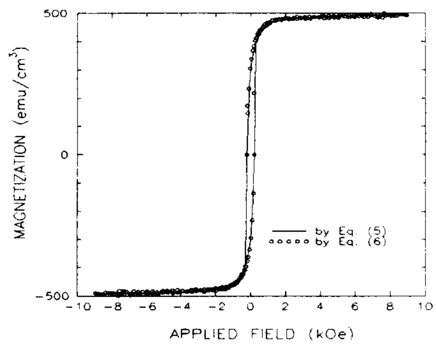

FIG. 4 The M-H hysteresis loops of the same Ni wire.

In order to investigate the accuracy, we consider the case of $K_i = 0$. From the previous mathematical definitions of K and H_k, H_k becomes $2\pi M$. Putting $2\pi M$ instead of H_k into Eq. (4) in the Ref. [12], one can obtain an approximate equation expressed by

$$M = \frac{\tau}{H \sin\left(\phi \frac{2\pi M}{H + 2\pi M}\right)}, \qquad (6)$$

where $(\phi - \phi_o)$ is less than $\frac{\pi}{12}$. It should be noted that the inaccuracy of the approximate Eq. (6) is less than 1% even at the fields where the magnetization is not uniform. One can obtain the M-H hysteresis loop of a sample having $K_i = 0$ from the τ-H hysteresis loop by numerically solving M in Eq. (6). The M-H hysteresis loop of the Ni wire obtained by the present approximation is also depicted by circles in Fig. 4. The nearly same shapes of two M-H hysteresis loops in Fig. 4 clearly show that the accuracy of our method is negligibly affected even at the coercivity. This was also confirmed by comparing the M-H hysteresis loops measured by our method and by a VSM.

IV. CONCLUSION

We have developed a new torque magnetometric technique to obtain the M-H hysteresis loop of the uniaxially magnetic material at any applied field orientation. The sensitivity of this method in measuring the magnetic moment depends on the anisotropy field intensity of the sample as well as the torque sensitivity. The accuracy is negligibly affected even at the fields near the coercivity, where the magnetization is not uniform. Utilizing this method, a torque magnetometer having a torque sensitivity of 10^{-6} dyn·cm could yield an extremely high sensitivity of 10^{-9} emu in the measurement of the M-H hysteresis loop.

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