Development of a versatile SMOKE system with electrochemical applications

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We describe the design, construction, and implementation of a simple and inexpensive, yet versatile surface magneto-optic Kerr effect (SMOKE) setup designed to operate in conjunction with the electrodeposition of magnetic layers both *in situ* and *ex situ*. The system is based on a homemade electromagnet and commercially available components. The sensitivity of the system is demonstrated by measuring *ex situ* SMOKE hysteresis loops of Co thin films (down to three monolayers thick) electrodeposited onto a Au(111) electrode substrate. © *2002 American Institute of Physics.* [DOI: 10.1063/1.1490416]

I. INTRODUCTION

The current general thrust in nanotechnology and nanostructures has generated a great deal of interest in the development of techniques capable of producing and characterizing such structures. The case of magnetic thin films and layered structures is an especially challenging one, which is economically and technologically important as the recording media industry shows.[1]

One of the most frequently used techniques for the analysis of thin ferromagnetic samples is based on the Kerr effect, described by the Reverend John Kerr in 1877.[2] This consists of the rotation of the polarization plane of a light beam when reflected from a magnetized surface—the interaction between the incident light and the magnetized surface rotates the polarization plane of the incident light by an amount that is proportional to the magnetization of the sample.[3] The practical method to measure this effect, and thus the magnetization state of the sample, is relatively simple; laser light is reflected from the sample of interest, and two crossed polarizers, before and after the sample, cancel the light intensity in the detector at zero magnetic field. Any subsequent change in the intensity represents rotation of the polarization plane of the light due to the magnetization of the sample induced by an applied external magnetic field.

This effect has been extensively used to study the magnetism of a great variety of samples and the technique was named MOKE (magneto-optic Kerr effect).[4,5] The penetration depth of the light beam in the sample lies in the range[6] of 200 Å, which allows a good sampling of the material under study. The potential for the surface sensitivity of this technique (surface MOKE or SMOKE) was first explored in the mid 1980s by the preparation of thin films on top of nonferromagnetic materials.[7] Thus, the rotation of the polarization plane is dominated by the interaction with the ferromagnetic overlayer.

A theoretical description of the magneto-optic Kerr effect was achieved as early as 1932,[8] and more complete quantum mechanical approaches have been proposed by Kittel in 1951[9] and Argyres in 1955.[4] (See also Ref. 10.) In the eighties, the increased interest in thin films and low dimensionality structures induced the development of the SMOKE technique[7] and some years later theoretical models were proposed.[6] Since then, as the importance of reduced dimensionality in magnetism has grown, the number of applications of the surface magneto-optic Kerr effect has increased.[3,11,12]

The three different geometries—transverse or equatorial, longitudinal, and polar—that can be used in order to study the orientation of the magnetic moments in a sample are depicted in Fig. 1. The longitudinal and polar geometries are usually employed to study magnetizations parallel and perpendicular to the surface of the sample, respectively. The transverse Kerr effect is generally smaller in magnitude than the longitudinal and polar cases, but it was often used in the past to study ferromagnetic metals.[3,13]

In this article we describe a simple and inexpensive, yet versatile setup to perform SMOKE experiments. The system was tested on thin cobalt samples prepared by electrochemical deposition techniques and analyzed *ex situ*, but *in situ* measurements, in an electrochemical cell or an ultrahigh vacuum (UHV) chamber, are also possible. In such *in situ* measurements, careful consideration is needed of potential birefringent effects introduced by additional interfaces.

II. EXPERIMENTAL SETUP

A schematic of our SMOKE setup is shown in Fig. 2. The light source *L* is a 5 mW, 635 nm diode laser (Coherent, model 31-0128) which is very stable in both intensity and polarization plane. The experimental determination of these variations (light intensity measured at the detector after passing through a polarizer) showed a total variation of the order of 0.5% of the total intensity. The manufacturer's specifications for this laser indicate a maximum variation in the intensity of 0.06% and a negligible rotation in the polarization plane.

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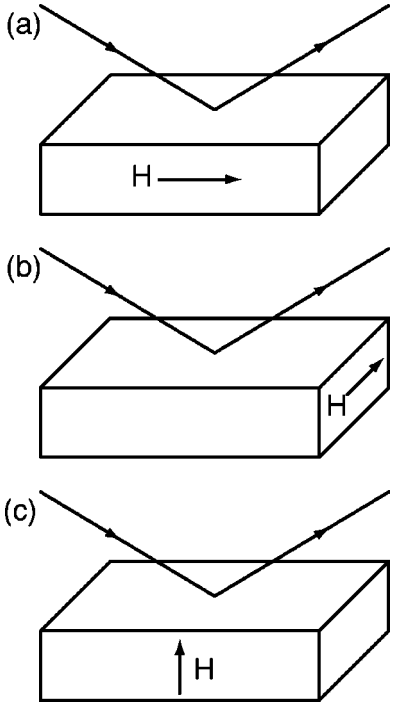

FIG. 1. A schematic of the three possible geometries in a SMOKE experiment: (a) longitudinal, (b) transverse, and (c) polar.

After passing through the first polarizer $P1$ the light is reflected off the sample, passes through the second polarizer $P2$ and is detected by a radiometer/photometer, D (EG&G Electro-optics, model 450-1). The two photographic polarizers (Tiffen, 49 mm in diameter) are mounted on rotation stages, $RS1$ and $RS2$ (Huber, model 410 with 10:1 gear reducers), allowing for fine control and reproducibility in positioning. The quality of these polarizers is measured by the extinction ratio,[14] which in this case is $\sim 1 \times 10^{-4}$.

The magnetic field is generated by a homebuilt electromagnet (M in Fig. 2) as shown in Fig. 3. Copper magnet wire with a thin insulating layer (Rea, 18 gauge HTAIH) was wound on a 95 mm long aluminum spool with a 117 mm

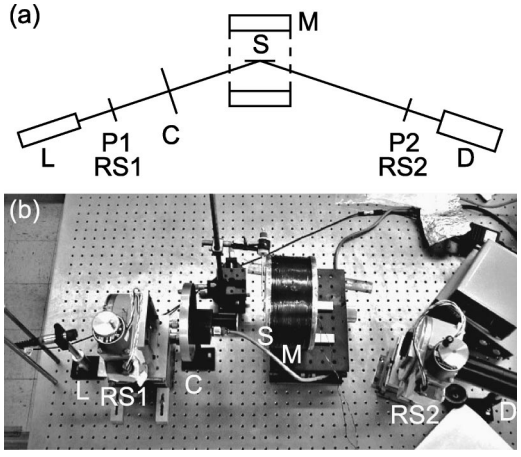

FIG. 2. (a) A schematic of the SMOKE setup in the longitudinal geometry. (b) A photograph of the experimental setup. L=laser; $P1$, $P2$=polarizers; $RS1$, $RS2$=rotation stages for the polarizers; C=chopper; D=detector; M=electromagnet; and S=sample. The polarizers P are mounted on the rotation stages RS.

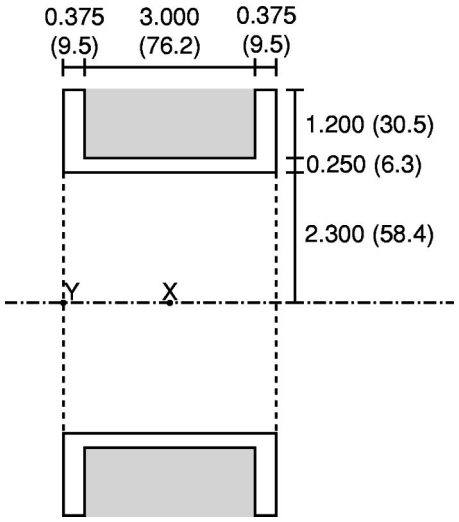

FIG. 3. A schematic of the homebuilt electromagnet, which is cylindrically symmetric about the dash–dotted line. The shaded areas indicate where the magnet wire was wound around the spool. The maximum magnetic fields (when 4 A are driven through the coil) are about 500 Oe at point X and 400 Oe at Y. The dimensions are given in inches (millimeters).

center bore, yielding a coil with a total resistance of $\sim 20\ \Omega$. To drive current in it, a bipolar, programmable power supply (Kepco, model BOP 100-4M) with a maximum current of 4 A at 100 V is used. The relationship between the current driven in the coil and the magnetic field produced on the axis was calibrated using a Hall probe (F. W. Bell Gauss/Tesla meter, model 4048) at several distances from the edge of the coil. The maximum achievable field on the axis of the coil was about 500 Oe in the center of the magnet (point X in Fig. 3) and about 400 Oe at the edge (point Y in Fig. 3).

Its size and geometry make this electromagnet quite versatile. Figure 2 shows the longitudinal SMOKE geometry, but the polar and transverse geometries can be realized as well. In addition, the magnet can be attached to a standard 4.5 in. flange of an UHV chamber for *in situ* vacuum measurements.

To facilitate signal detection, the laser intensity is modulated by a mechanical chopper (Laser Precision Corporation, model CTX-534; C in Fig. 2) at $\sim$500 Hz. The output of the detector is fed into a lock-in amplifier (EG&G-Princeton Applied Research, model 5209). The power supply and the lock-in amplifier are controlled with a computer via a National Instruments GPIB card and LabView software, both to sweep the magnetic field and to record the data. For the data reported in this article, every complete hysteresis loop took between 10 and 20 s to measure. Replicate measurements (between 2 and 25) were carried out to achieve appropriate signal-to-noise ratios.

III. MEASUREMENT PROCEDURE

It is well known that when linearly polarized light is reflected from any surface, it becomes elliptically polarized. However, this effect is eliminated when the incident light is purely *s* or *p* polarized. In those cases, the light maintains its polarization state upon reflection.[15]

To achieve either of these geometries with our setup for a particular measurement, the first polarizer was nominally set to the desired polarization, and then the two polarizers were adjusted iteratively to produce the minimum intensity of reflected light at the detector. The ratio of the light intensity in this extinction condition to the intensity with the analyzing polarizer parallel to the first polarizer typically ranged from 5×10^{-5} to 5×10^{-4}. This compares favorably to the extinction ratio for these polarizers, which indicates that, within the accuracy of the measurement, the light incident on the sample is purely *s* or *p* polarized.

Following Qui and Bader,[12] in order to quantify the measured Kerr signals, the analyzing polarizer was rotated by a fixed angle δ (e.g., 5° or 8°) from the extinction position for each measurement. In this situation, the Kerr rotation ϕ' is related to the measured light intensity (lock-in amplifier voltage) I via

$$\phi' = \frac{\delta}{2}\frac{I-I_0}{I_0}, \tag{1}$$

where I_0 is the average intensity in a hysteresis loop (the measured intensity for $\phi'=0$ or no Kerr rotation) and δ is assumed to be small. This procedure has the added advantage of increasing the signal-to-noise ratio in the measurement. For a given Kerr rotation, the measured intensity change with respect to the average I_0 is

$$I-I_0 = 2|E|^2\delta\phi'. \tag{2}$$

In this equation, $|E|^2$ represents the intensity of the reflected light in the original polarization state. I_0 is also the fraction of this light transmitted through the analyzing polarizer, rotated from extinction by an angle δ, or $I_0=|E|^2\delta^2$. Thus, for a constant noise source, the signal-to-noise ratio increases with δ.

IV. TESTING OF EXPERIMENTAL SETUP

The first tests were performed on vapor deposited Co films on quartz substrates. The films were about 100 Å thick as determined from a quartz crystal thickness monitor.

All three different geometries were investigated—longitudinal, polar, and transverse—with the laser at near normal incidence. The results are presented in Fig. 4. Hysteresis loops were obtained in all cases except in the polar geometry, indicating that the magnetization in these samples does not occur perpendicular to the surface layer. The high sensitivity of our system as constructed is apparent by the observation of the measurable hysteresis loops in the longitudinal case, which is a smaller effect.[3,13]

Using our SMOKE setup, *ex situ* measurements were performed on several Co films electrochemically deposited on a Au substrate. The nominally Au(111) sample was formed, oriented, and cut using the Clavilier method,[16] yielding a sample with a diameter of about 2.3 mm. Before each experiment, the sample was flame annealed and checked for cleanliness by cyclic voltammetry in a 0.1 M NaOH solution. The Co films were then deposited on the sample from either 0.01 M or 0.1 M $CoCl_2$ with no additional supporting electrolyte. The potential on the Au electrode was swept down to

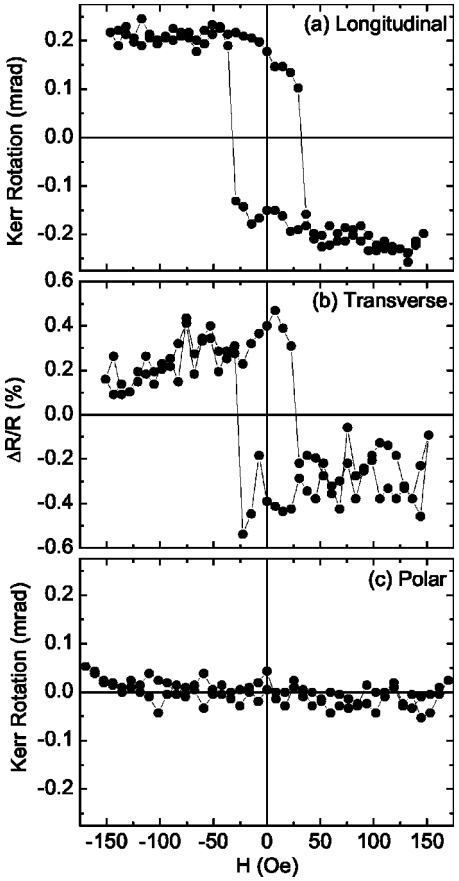

FIG. 4. Near normal incidence SMOKE hysteresis loops for a Co sample vapor deposited on a quartz substrate in the (a) longitudinal, (b) transverse, and (c) polar geometries with the incident light initially *p* polarized.

−0.9 V vs Ag/AgCl and held there for a predetermined amount of time *t* depending on the desired coverage. Afterwards, the sample was removed from the electrolyte while the potential was maintained and transferred to the SMOKE setup.

The system was arranged in the longitudinal SMOKE geometry with the laser light incident on the sample approximately 77° from the surface normal. SMOKE hysteresis loops were recorded for both *s*- and *p*-polarized light and for $\delta=5°$ and 8°. Afterwards, the sample was transferred back to an electrochemical cell containing 0.1 M H_2SO_4 as an electrolyte, and the potential was swept up from −0.7 V vs Ag/AgCl to strip the Co film from the Au substrate. From the charge associated with this stripping, the average Co film thickness could be calculated, assuming a surface atom density equal to that of Au(111).

Figure 5 shows the SMOKE data obtained from three different Co films. The stripping measurements showed that the films were 80, 18, and 6 ML thick. The 6 and 80 ML films were deposited from 0.01 M $CoCl_2$ and 0.1 M $CoCl_2$, respectively, in both cases with a deposition time of 30 s. The difference in film thickness is consistent with the difference in concentration, assuming mass-transport limited deposition. The 18 ML film was deposited from the 0.01 M $CoCl_2$ solution with a deposition time of 60 s. The data shown were analyzed using Eq. (1), and the results for $\delta=5°$ and $\delta=8°$ were averaged. The saturation values compare favorably

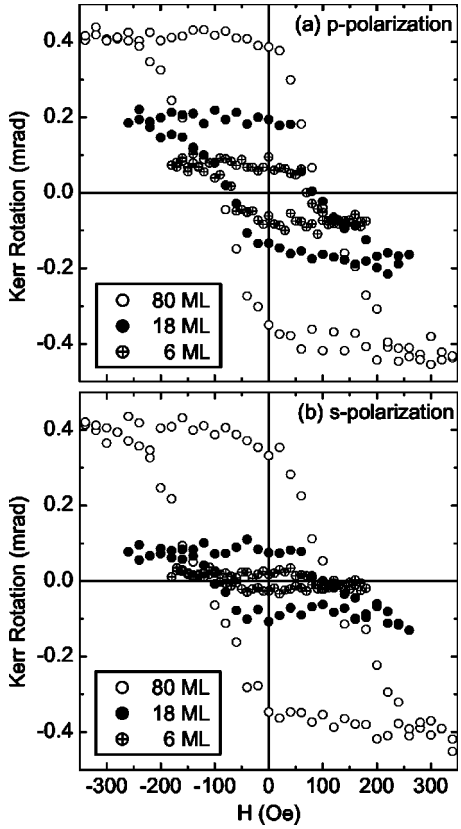

FIG. 5. Longitudinal SMOKE hysteresis loops of three Co thin films (6, 18, and 80 monolayers) electrochemically deposited on a Au(111) substrate taken with the incident light initially (a) *p* polarized and (b) *s* polarized.

with theoretical calculations by Zak and co-workers[17] (also available on the internet[18]). These data show that the system, as constructed, is sensitive to magnetic films that are only a few monolayers thick.

In summary, we have assembled a flexible and straightforward SMOKE setup. Using a homemade electromagnet and commercially available components, a simple system has been constructed which is sensitive to a few monolayers of Co on Au. It is sensitive enough to detect transverse SMOKE signals.

ACKNOWLEDGMENTS

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