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Magnetometry of monoatomic layers and spin electronics elements

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Two magnetometers: resonance vibrating sample magnetometer (R-VSM) and magnetooptical Kerr effect magnetometer (MOKE) designed for the measurements of hysteresis loops of ultra-thin films and spintronics elements are described. Both instruments have been built by the first author of this paper in the Department of Electronics. The measuring head of R-VSM is universal and can work in the Helmholtz coils as well as in an electromagnet. The frequency of R-VSM is about 75 Hz. Test measurements on MgO (001)/Fe (4, 3, and 2 ML)/50 Å Cr samples were performed and the sensitivity was estimated as 10^{-5} emu. The MOKE system with a differential amplifier (in contrast to lock-in detection technique) is especially recommended for rapid measurements up to 1.5 kHz. The calibration of the Kerr rotation angle was performed on the standard Fe-sputtered films (with thickness from 2 nm to 50 nm) and Fe-wedge sample, prepared by MBE-technique, within the range of thickness from 1 ML to 50 ML of Fe. Based on the measurement of 2, 3, 4, 5, 7 ML of Fe, the angle resolution of Kerr rotation was estimated as 0.001 min. The example measurement of hysteresis loop on the spin valve structure Si (100) Ta (52 Å)/Co(44 Å)/Cu (22 Å)/Co (44 Å)/FeMn (100 Å)/Ta (52 Å) is demonstrated.

Key words: *magnetometry; vibration sample magnetometer (VSM); magnetooptical Kerr effect (MOKE); magnetic monoatomic layers; spin electronics*

1. Introduction

Multilayers in the form of artificial superlattices in the range of thickness of several monoatomic layers consisting of different magnetic materials (e.g., soft or hard ferromagnets, antiferromagnets, semiconductors, insulators) form spin-nanosystems which may be applied as magnetoelectronic devices: cells of magnetic random access memory (M-RAM) [1], sensors [2] or read heads in hard disk drives [3]. Such very thin films deposited onto diamagnetic or paramagnetic substrate, where the volume of the ferromagnetic material is much smaller than the volume of the substrate, require

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very sensitive instruments and precise methods for measurement of hysteresis loops. In this paper, we demonstrate the construction and test measurements of two very highly sensitive magnetometers: resonance vibrating sample magnetometer (R-VSM) [4] and magnetooptical Kerr effect (MOKE) magnetometer [5]. Both arrangements, which were recently built in the Department of Electronics of the University of Mining and Metallurgy, are characterized by low-cost and sophisticated electronics solutions. We also show that for obtaining full information on magnetization reversal process in magnetic multilayers, both magnetometers should be used, because the methods are complementary. R-VSM measurements deliver information about averaged magnetization process from the whole volume of the sample, whereas magnetooptical information from MOKE magnetometer is local, limited by light-beam spot and penetration depth (for metals it is about 200 Å with an exponential decay).

2. Magnetometer R-VSM

The idea of R-VSM is based on the Faraday induction law and the original Foner solution [6] adopted for measurements of ultra-thin films. There are the following main advantages of R-VSM in relation to the Foner's VSM: the sample oscillates (with a maximum amplitude of 1 mm) parallel to the direction of external magnetic field, therefore it is always in the region of homogeneous field (in the Foner's VSM, the sample vibrates perpendicularly to the direction of external magnetic field), a configuration of pick-up coils in the form of small Smith coils is more favourable [7] than two pairs of pick-up coils connected adversely as in conventional Foner's VSM.

The block diagram of R-VSM is shown in Fig. 1. The magnetic thin film is attached to the end of a glass pipe-rod oscillating with the mechanical resonance frequency of the rod–sample system. The oscillations are forced by a piezoelectric transducer attached to the head (A). The piezoelectric transducer works far from its own resonance, in our case with the frequency equal approximately to 75 Hz. The sample placed in the magnetic field, produced by Helmholtz coils or electromagnet (B), oscillates between pick-up coils (C) inducing a signal which is transferred to the differential input of the lock-in amplifier. The capacity sensor (for electronics details see e.g. [8] or contact directly the first author) of the sample position (E) delivers the signal proportional to the amplitude of sample oscillations and supplies the voltage to the electronic system which generates a reference signal, further provided to the reference input of the lock-in amplifier. The output of digital-to-analog converter ($\mathrm{DAC_{1out}}$) and digital output ($\mathrm{D_{1out}}$) of the lock-in amplifier control the current flowing through the electromagnet and its direction, respectively.

The teslameter (Htm-11n), using a Hall sensor, measures magnetic field and the voltmeter measures the amplitude of the reference signal. The signal from the pick-up coils, detected by the lock-in amplifier, is measured as a function of the magnetic field and allows one to obtain the hysteresis loop of the examined sample.

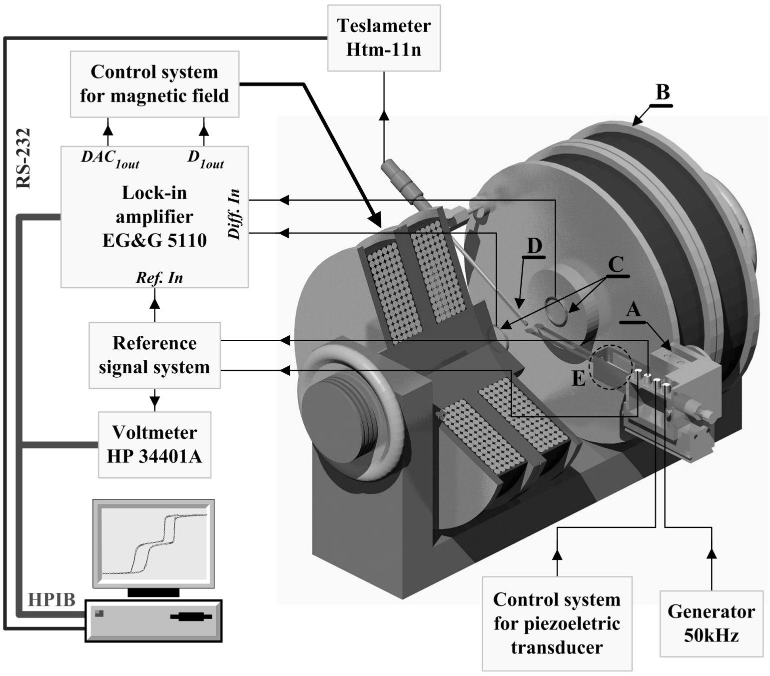

Fig. 1. Block diagram of R-VSM; A – head, B – electromagnet, C – pick-up coils, D – Hall sensor, E – capacity sensor of position

For harmonic oscillations of the sample, the signal *e* induced in the pick-up coils is proportional to the amplitude of oscillations *K*, frequency of the sample oscillations ω and magnetic moment of the measured sample *m*:

$$e = \mu_0 K \omega m g \cos(\omega t)$$

The geometry of the applied pick-up coils and dimensions of the investigated sample are taken into account by a geometrical factor *g* [9], which depends on the sample position measured between pick-up coils. Designed pick-up coils should be characterized by a large factor *g* which should be independent of the position of the moving sample. In R-VSM, the Smith geometry was applied [10] for pick-up coils because of the largest value of the geometrical factor and the flattest dependence of $g(z/a_0)$, in the wide range of sample positions [7] (Fig. 2). From the analysis of *g* as a function of pick-up coils size (under the assumption that the sample has a form of a dipole), we can see that the largest *g* is for $z_0/a_0 = 0.5$, where $2z_0$ is the distance between pick-up coils, and a_0 is the radius of the coils applied. For $z_0/a_0 = 0.866$ and $z_0/a_0 = 0.9244$, relative changes of *g* do not exceed 1%, hence these values are recognized as optimal. The calibration of magnetometer was carried out by examining the geometrical factor *g* for different shapes of flat samples made of high-purity nickel foil. The main purpose of using R-VSM was to make measurements of the hysteresis loop, saturation magnetization, coercive force and anisotropy field.

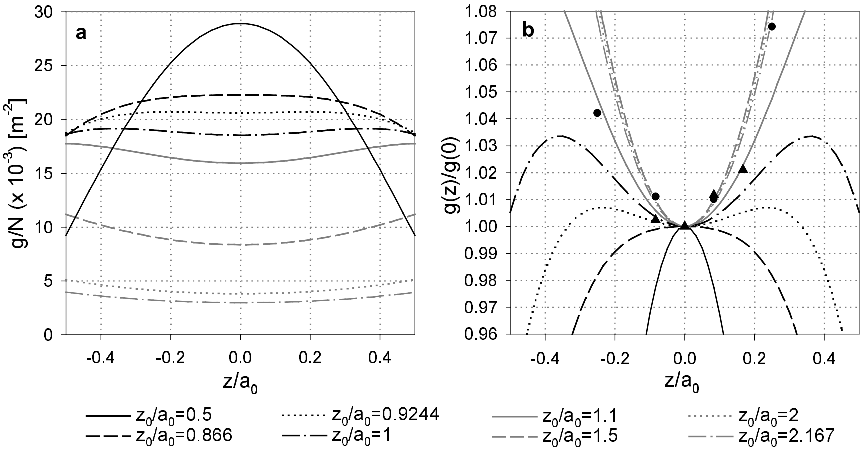

Fig. 2. Geometrical factor for one turn of coils (for dipole sample) (a) and relative change of geometrical factor for dipole (b): • – experimental data for Ni foil with diameter of 0.2 mm and thickness of 20 μm for $z_0/a_0 = 2.167$ and ▲ – for $z_0/a_0 = 244$)

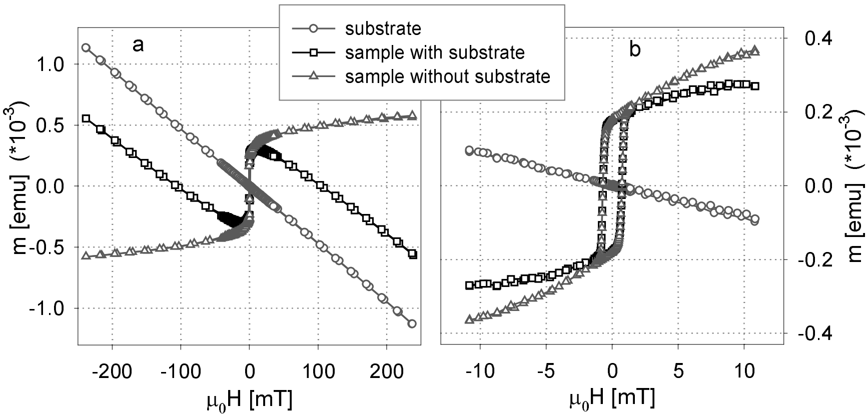

Fig. 3. High-field (a) and low-field (b) measurements of the MgO (001)/Fe (4 ML)/Cr (50 Å) sample oriented epitaxially

In the case of very thin magnetic films, a signal from the substrate is comparable to the signal from the thin layer as shown in Fig. 3a. Test measurements were performed on monolayers of Fe, with variable thickness: 4 ML, 3 ML and 2 ML (1 ML of Fe = 1.45 Å), deposited by the molecular beam epitaxy (MBE) technique on the MgO (001) substrate and protected against oxidation by a 50 Å thick Cr layer. The volume of the substrate was by seven orders of magnitude higher than that of iron layer (the volume of MgO substrate – 10^{-7} m^3 and volume of 5.8 Å Fe layer – $5.8\cdot10^{-14}$ m^3). The signal from the diamagnetic MgO substrate is negative and varies linearly with magnetic field (Fig. 3a). The total signal from substrate and Fe (4 ML = 5.8Å)/Cr (50 Å) was measured in the electromagnet in high magnetic field (Fig. 3a) and in the Helmholtz coils in low field (Fig. 3b), then the diamagnetic signal from the substrate was subtracted. Finally, the hysteresis loop in the hard direction was obtained (the easy axis is the [001] direction for Fe). A signal very weak, but sufficient to register the magnetic hysteresis, was obtained from 3 ML and 2 ML of Fe samples (Fig. 4).

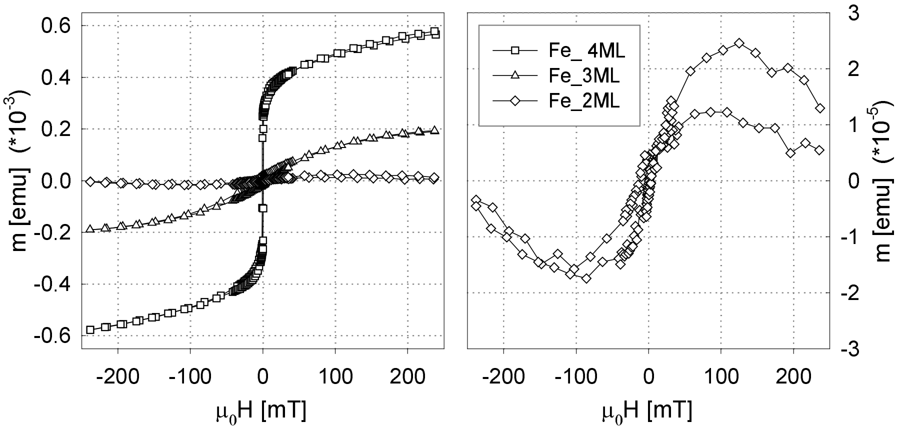

Fig. 4. Hysteresis loops for 2, 3 and 4 ML of Fe after subtracting the signal from the substrate

Based on these measurements, the sensitivity of R-VSM was estimated as 10^{-5} emu, but the magnetic moment resolution was equal to 10^{-6} emu. The resolution of magnetic moment measurement results from digital resolution of the signal measured by the lock-in amplifier.

3. Kerr magnetometer

A high-sensitivity magnetooptical Kerr magnetometer based on the differential amplifier, in contrast to lock-in detection technique, is especially recommended for rapid measurements up to 1.5 kHz. The arrangement for measurements of magnetooptical hysteresis loops is shown in Fig. 5. The coherent light from the He-Ne laser (Z), with 5 mW power and wavelength λ = 633 nm, is polarized by the Glan–Thompson prism polarizer (P) and is then reflected by the surface of the sample (S) placed in the centre of the Helmholtz coils (H) or an electromagnet. Next, it passes through the Wollaston prism (W) which splits the beam into two beams polarized linearly in mutually orthogonal planes. These beams are focused by the lens (L) on the photodiodes of differential amplifier (A) (for electronics details see [8]). The amplifier delivers both the common (U_C) and the differential (U_D) signals, proportional to the sum and difference of the light intensities of the beams, respectively. These signals are measured by a high-resolution (16 bit) and rapid (200 kHz) AD converter in PCI 6035 computer card. The computer controls the voltage of a programmable bipolar current source (KEPCO BOP 36-12M) which is used to drive the current through the Helmholtz coils (or the electromagnet) to provide a sweeping holtz coils (or the electromagnet) to provide a sweeping magnetic field. The magnetic field is accurately calibrated before measurements of the hysteresis loop using a teslameter (Htm-11n).

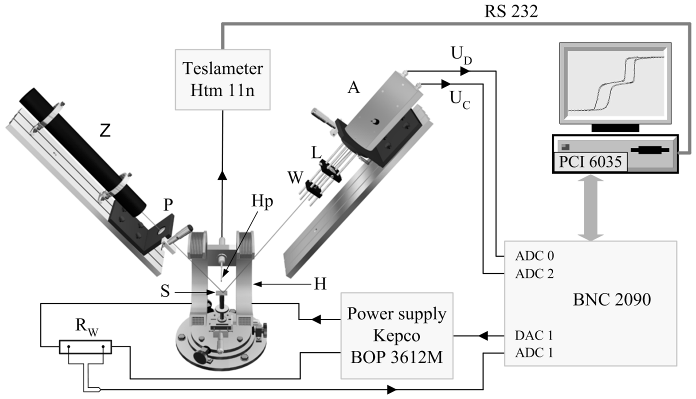

Fig. 5. Schematic layout of MOKE system arrangement: Z – laser, P – polariser, Hp – Hall probe, W – Wollaston prism, L – lens, A – differential amplifier, H – Helmholtz coils, E – capacity sensor of the sample position

The MOKE system is programmed using the LabView programming environment. The differential-to-common signal ratio measured as a function of the magnetic field gives hysteresis loop at the point of sample illuminated by the laser beam. For determination of the absolute value of the Kerr rotation angle, a calibration curve of

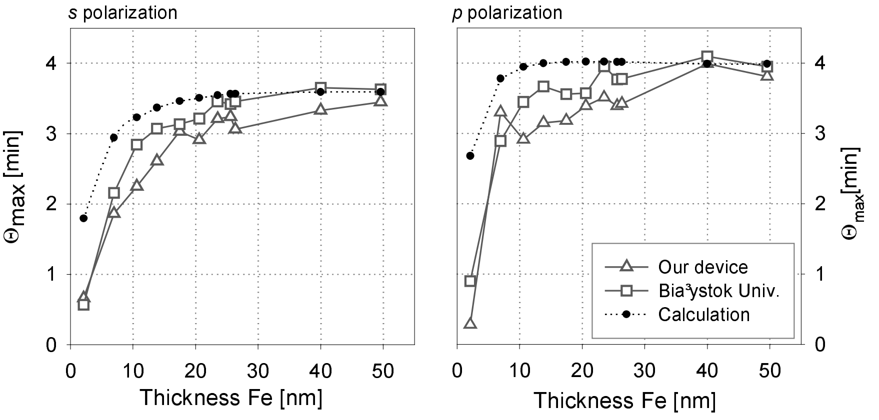

Fig. 6. Kerr rotation angle of Fe-sputtered films measured in saturation field (0.5 T) vs. thickness of Fe

differential-to-common signal ratio as a function of the rotation angle of the polarization plane $U_D/U_C = f(\Theta_K)$ was recorded. The calibration was done by rotation of a holder in which the Wollaston prism, the lens and the amplifier with photodiodes are mounted together. Additionally, the calibration of the Kerr rotation angle was performed on standard sputtered Fe films with the thickness from 2 nm to 50 nm, using a Kerr spectrometer (in the Institute of Physics, University of Białystok) based

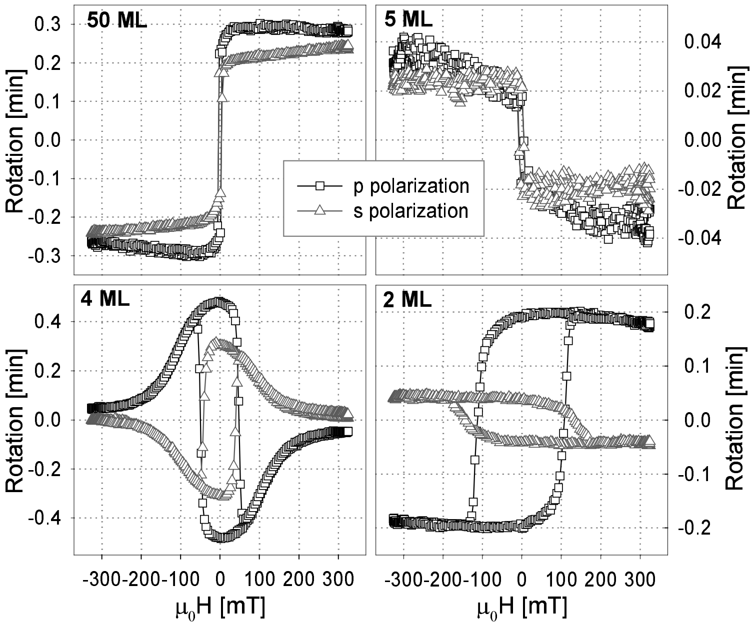

Fig. 7. Kerr rotation hysteresis loops
of 50, 4, 3, 2 ML of Fe for *s* and *p* polarization

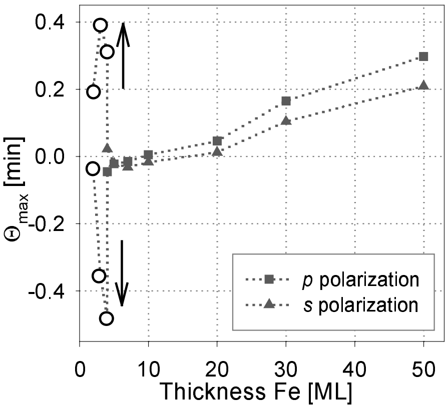

Fig. 8. Kerr rotation angle of Fe-wedge sample prepared by MBE
vs. thickness for *p* and *s* polarization. The arrows indicate
the transition from in-plane to perpendicular anisotropy

on the polarization modulation technique. Figure 6 shows the experimental and calculated data [5]. We can see that above the thickness of 25 nm, the experimental and calculated data agree very well but below that value the difference between them increases with decreasing thickness of Fe due to the presence of a nonstoichiometric iron-oxide layer about 2 nm thick [8, 11]. The sensitivity of the magnetometer designed was determined on the basis of measurement of ultra-thin Fe wedge film, prepared by MBE technique on the 170 Å thick buffer layer of Au (001) grown on MgO single crystal substrate. The thickness of Fe ranged between 1 ML and 50 ML. The sample was capped with a 30 Å thick Au layer to prevent oxidation. Examples of Kerr rotation hysteresis loops for *p* and *s* polarization for 50, 5, 4, 2 ML are shown in Fig. 7. The longitudinal Kerr effect measurement proved the presence of the in-plane magnetic anisotropy for Fe thickness between 50 and 5 ML, showing a decrease of the Kerr rotation with decreasing Fe thickness (Fig. 8). Below the critical thickness (5 ML), a drastic increase of the Kerr rotation was observed due to the change of the easy magnetization axis in the direction perpendicular to the layer. This change was accompanied by the appearance of the polar contribution to the measured signal [12]. The measurements on Fe wedge sample prove that 2 ML Fe is still ferromagnetic at a room temperature (Fig. 7). On the basis of these measurements the angle resolution of the Kerr rotation was estimated as 0.001 min.

4. Test measurements using spintronic elements

The measurements of the hysteresis loops were performed on selected spin valve structures and on an array of magnetic dots [8]. The spin valve Ta (52 Å)/Co (44 Å) /Cu (22 Å)/Co (44 Å)/FeMn (100 Å)/Ta (52 Å) structure sputtered on Si(100) is demonstrated here as an example (Fig. 9). In contrast to the magnetization of R-VSM measurement, the signal of the magnetooptical hysteresis loop is higher for pinned layer (lying at the top of multilayer system) due to light absorption effect.

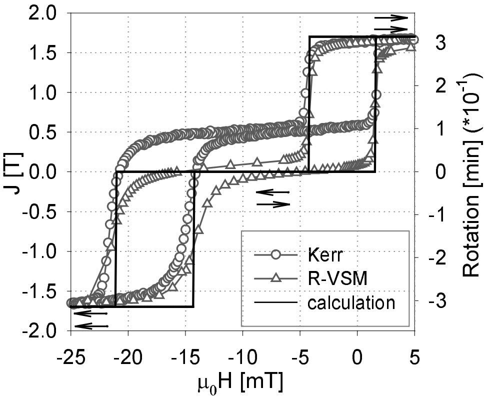

Fig. 9. Comparison of the spin valve hysteresis loops: magnetization (R-VSM), Kerr rotation angle and calculated values

Nevertheless, the switching fields determined from magnetization and Kerr effect measurements are almost the same. The calculations of magnetization hysteresis loop agree very well with the measurements performed by means of R-VSM. The fitting parameters are the following: interlayer exchange energy $J_1 = 7.9\cdot10^{-6}$ J/m^2, exchange biased energy $E_{EB} = 113\cdot10^{-6}$ J/m^2 and uniaxial anisotropy of a free and the pinned layers are $K_{u1} = 2.05\cdot10^3$ J/m^3 and $K_{u2} = 2.4\cdot10^3$ J/m^3, respectively.

5. Conclusions

The magnetometers designed are highly sensitive and able to measure very thin films down to 2 ML of Fe. Due to simple operation methods, both instruments are recommended for a fast characterization of fundamental magnetic parameters of monoatomic layers, multilayers and spintronics elements. They are simple from the electronic point of view as well as from the mechanical one, the cost of constructing the two magnetometers being very low in relation to commercial devices.

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