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In-plane anisotropy and stress detection of films deposited by RC technique

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Abstract. A Novel Rotating Cryostat (RC) vacuum system originally designed to fabricate organic layers has been developed in order to prepare magnetic materials for specific applications such as sensors. The RC sputtering system has a rotating drum (substrate holder) and the possibility of using multi-port deposition sources. The source material sputtered by a dc magnetron, which was positioned one of the ports around the RC, was an iron disk (25 mm diameter, 0.8 mm thick, 99.8% pure). Results show that films have exhibited isotropic and anisotropic magnetisation at various running conditions of the RC using a Magneto-Optic Loop Plotter (MOKE) and a Vibrating Sample Magnetometer (VSM). Estimation of magnetic anisotropy confirms in-plane anisotropy in the films. Moreover, when these films are subjected to a bending stress within the VSM, they show chances in their hysteresis loops. These findings indicate a possible future for this technique to produce sensing devices for stress detection.

PACS. 75.30.Gw Magnetic anisotropy – 75.50.Bb Fe and its alloys

1 Introduction

In recent years magnetic thin film science and technology continues to change at a rapid rate. The electromagnetic devices can be improved either by improving the properties of existing materials or developing a new class of magnetic materials [1]. For this reason, in order to prepare magnetic materials for specific applications such as sensors a Novel RC system has been developed. The first step of this paper was to produce and then analyse the magnetic properties of thin iron films in order to understand the features of the RC system. A dc magnetron sputtering source was positioned adjacent to the rotating surface of the drum and 100 nm thick films were successfully produced, and also the magnetic and physical analysis have been made.

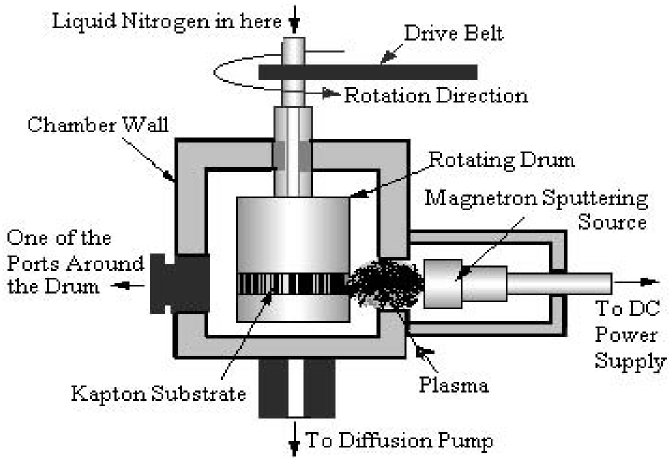

Fig. 1. The schematic diagram of the RC system showing the general principle of the RC sputtering system.

2 Experimental procedure

2.1 Rotating cryostat (RC) sputtering technique

The RC system utilises a mobile physical deposition method to produce films. In contrast to static deposition systems, the target material can be deposited onto the circumference of a rapidly (up to 2000 rpm) rotating liquid nitrogen cooled substrate (13 cm diameter) in an evacuated chamber ($\sim 10^{-6}$ mbar), see Figure 1. The system has a large deposition area of 82 cm^2 (2 cm wide) compared to a few cm^2 in static techniques; up to ten target sources, such as resistively heated furnaces, sputtering sources or electron guns. Therefore, making use of multiple target sources can produce various compositions of magnetic materials and multilayers [2,3].

The substrate and the target material were prepared and placed inside the RC, and the system was pumped down to a pressure of $\sim 10^{-6}$ mbar. Prior to depositing, the inner and outer drum was filled with liquid nitrogen and then the inner drum was rotated at a speed of 1300 rpm. All depositions were carried out in a background gas consisting of 99.98% purity argon at a pressure of $\sim 10^{-2}$ mbar. Thin iron films were sputtered from an iron disk (25 mm diameters, 0.8 mm thick, 99.8% pure)

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onto a 75 μm thick polymide (kapton^{TM}) substrate using a dc magnetron sputtering, which was positioned adjacent to the rotating surface of the drum. The deposition was continued over a time-period of half an hour, while the drum was rotating.

2.2 Characterisation techniques

The anisotropy investigations were carried out using primarily MOKE but comparative measurements were also made using VSM systems that magnetised samples in different directions with respect to the rotation. The rotation direction refers to the direction of inner drum of the RC system, see Figure 1. Anisotropy measurements were carried out on 6 mm diameter specimens at room temperature.

The MOKE system utilises the transverse Kerr-effect to measure loops proportional to the *M*-*H* loops. The systems consists of a diode laser of wavelength 670 nm, which is modulated at 500 Hz. Magnetising field of ±50 kA/m were used and the time taken to measure one hysteresis loop was approximately 300 seconds. A commercial VSM from Molspin Ltd was used to produce DC measured *M*-*H* loops. The samples were vibrated at 75 Hz in the fields of 800 kA/m. The MOKE set-up is essentially a surface technique, which sense only the magnetisation to a depth of 10 nm to 20 nm in the material. By using the VSM, which is a bulk measurement technique it is possible to determine if the surface magnetisation is similar to the bulk of the film. The physical structure was investigated using the standard technique of X-ray diffraction.

3 Results and discussion

3.1 Magnetic characteristics

The magnetic field was applied at different angles in the plane of the sample and also perpendicular to the film plane. The substrates of the films for samples A were tightly wrapped on the drum and rotated at the speed of 1300 rpm. Figures 2a and b show the hysteresis loops of typical sputtered iron films measured along the rotation direction, 0° and at 30°, 60° and 90° to the rotation direction in the film plane. Hysteresis loops in Figures 2a and b show that the sample has a well- defined uniaxial in-plane anisotropy. In Figure 2a, when the field is applied along the rotation direction, the loop exhibits a smaller coercivity, 1.85 kA/m, in comparison to anisotropy field, H_a, 19.50 kA/m, but not equal to zero. When the field is applied at 90° to the rotation direction, the loop is almost square with a coercivity field H_c, 5.90 kA/m. H_c is different from H_a which means that when the field is applied along the easy axis, the magnetisation does not rotate uniformly but there is a nucleation and propagation of domain walls. The coercivities obtained from sputtered films are summarised in Table 1 which shows that the coercivities decreases when the angle of measurement is rotated from 90° to 0° to the rotation direction. Dionisio *et al.* [4]

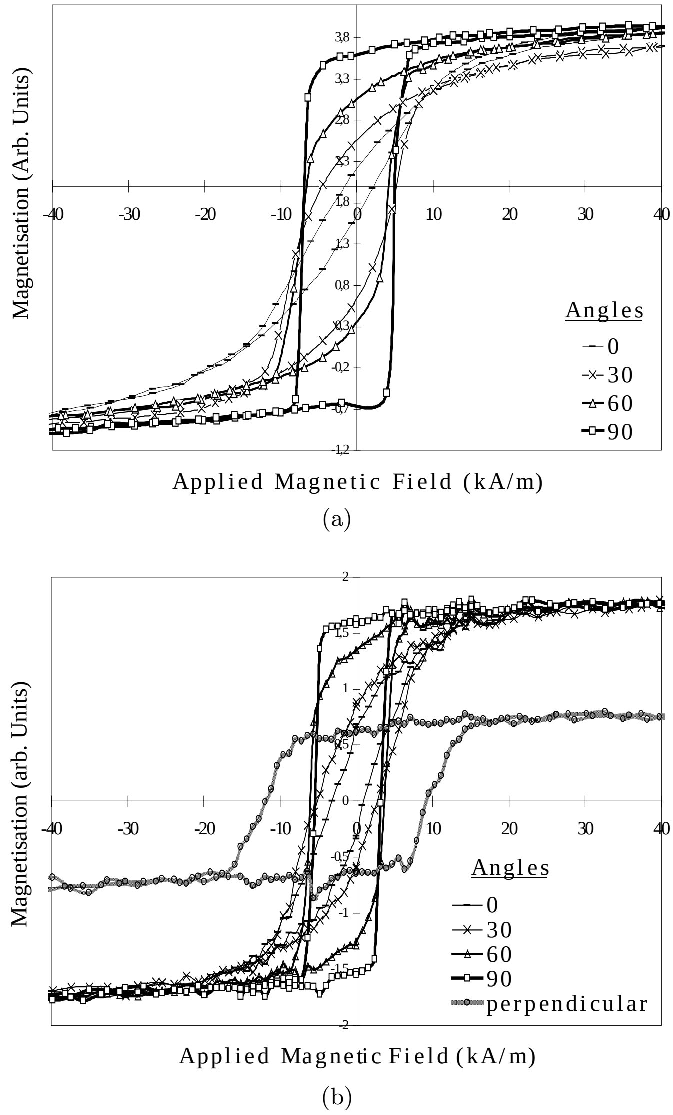

Fig. 2. Hysteresis loops measured by (a) MOKE and (b) VSM on the iron film (Sample A) with different angles (90°, 60 °, 30°, 0°, and perpendicular to the film plane) between the applied magnetic field and the rotation direction.

reported having dc magnetron sputtered bcc α-iron films has the coercivity values of around 6 kA/m. The coercivities of the sputtered films reported here have similar coercivity values to those found in the literature [4,5]. Even if the MOKE and VSM measurements indicate different coercivity values, they both confirm the uniaxial in-plane anisotropy in the specimens.

When the field is applied perpendicular to the film plane, perpendicular anisotropy was found not to be exist. This indicates the hardest axis of the *M*-*H* loops is the perpendicular direction. As a result of the demagnetising effect the film shape anisotropy dictates that specimens must have a planar easy axis. This is illustrated in Figure 2b, which shows the planar magnetisation loops typical of all polycrystalline film specimens studied during the course of this investigation. These values also show

Table 1. The summary of the coercivity (H_c) values and isotropy of the sputtered films obtained from MOKE and VSM.

	Coercivity, H_c (kA/m)				Explanation for Uniaxial Anisotropy
	0°	30°	60°	90°	
Sample	MOKE				
A	1.85	4.85	5.40	5.90	Anisotropic
B	2.75	3.30	3.75	4.20	Slight anisotropy
C	5.85	5.75	5.85	5.90	Isotropic
Sample	VSM				
A	2.05	3.90	4.90	5.20	Anisotropic
B	2.60	3.00	3.25	3.70	Slight anisotropy
C	5.20	5.03	4.83	5.15	Isotropic

that a tensile stress along the rotation direction is likely to make domain magnetisation perpendicular to this direction due to its negative magnetostriction.

The RC has produced films with well-defined in-plane magnetic anisotropy. In some materials, a uniaxial anisotropy can be induced by applying a magnetic field during the deposition process [5,6]. In this investigation, the easy axis is at 90° to the rotation direction of the rotating drum and its origin is probably due to either the rotation of the drum or to the tightly wraping of the substrate on the surface of the drum using a metal clamp which may have introduced stress into the sample during deposition. Therefore, further sets of the films (samples B) were deposited with tightly wrapping and stationary substrates and also the films (samples C) without wrapping and stationary substrates were produced.

Figure 3 shows the hysteresis loops of sputtered iron film of samples B measured at various angles relative to the direction of substrate rotation. Although the features characteristic of in-plane anisotropy was observed, the M-H loops corresponding to the various angles of the field are different from the previous M-H loops in Figure 2a and Figure 2b. In the film plane, when H field is perpendicular to the rotation direction the loop is almost square whereas with H parallel to the rotation direction the loops remanence ratio has decreased. The stationary material production makes the uniaxial magnetic anisotropy less well defined due to a smaller spread in coercivity values for each direction, see Table 1. The rotation of the substrate has only a small effect on the observed uniaxial anisotropy.

Figure 4 shows the hysteresis loops obtained at various angles to the rotation direction for a sputtered iron film (samples C). The substrate was not wrapped and the drum was stationary during the deposition. These loops show that samples C does not possess a uniaxial magnetic anisotropy. Although there are slight variations between each loop with angle, the film does not show any sign of an easy axis along the 90° to direction. The loops shown in Figure 4 and the data presented in Table 1 indicate that the samples C are isotropic in the directions in which the measurements took place.

These measurements show that the tight wrapping of the substrate was the origin of the induced tensile stress.

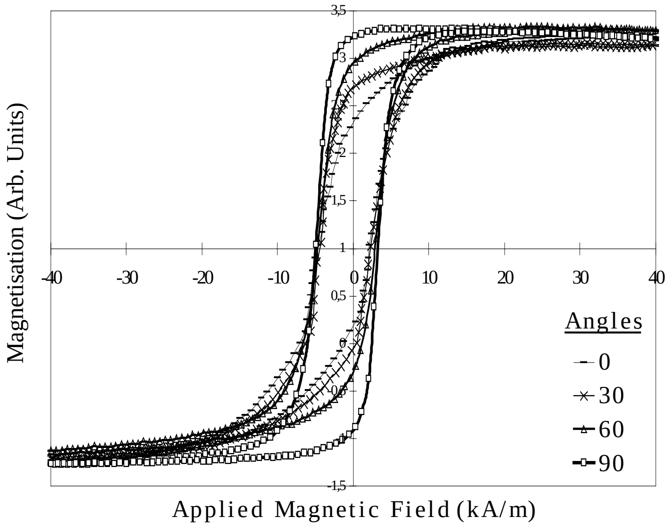

Fig. 3. Hysteresis loops measured by MOKE on iron film (Sample B) with different angles (90°, 60°, 30°, 0°) between the applied magnetic field and the rotation direction.

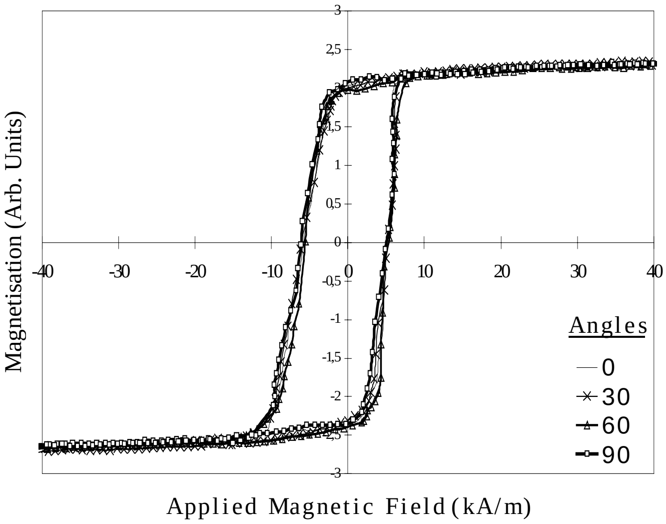

Fig. 4. Hysteresis loops measured by MOKE on iron film (Sample C) with different angles (0°, 30°, 60°, 90°) between the applied magnetic field and the rotation direction of the substrate.

Magnetisation loops obtained by VSM and MOKE systems have produced very similar hysteresis loops confirming that the surface magnetisation measured by the MOKE system is representative of the whole film. Further investigation of the stress sensitivity using the VSM was performed by inducing a bending stress in the films in order to observe weather the films was stress sensitive or not. A slight chance of the magnetisation values in the hysteresis loops was observed.

3.2 Estimation of the magnetic anisotropy in the films

The origin of the in-plane anisotropy obtained from experimental study is now discussed in detail by doing that the estimations as follows. An estimation of the in-plane anisotropy has been obtained from the *M-H* loops. It should be pointed out that these films exhibited very large uniaxial anisotropies with anisotropy fields between 1400 kA/m and 1700 kA/m. To obtain anisotropy field, H_a to do the estimations, the perpendicular magnetisation loop was extrapolated back to the saturation magnetisation level (to the film plane). The anisotropy field H_a was taken, as the magnetic field required saturating the film specimen along the hard axis, *i.e.* the direction of substrate rotation. The saturation magnetisation (M_s) of the films assumed to be 1.71×10^7 A/m [7]. The intrinsic uniaxial anisotropy, K_u can then be estimated from the following equation:

$$H_a = \frac{2K_u}{\mu_0 M_s} \cdot \tag{2}$$

The assumption when using this equation is that the anisotropy field is the field required to turn the magnetisation through 90° away from the hard axis. This equation was only applied to those films with clearly defined easy and hard directions in their *M-H* loops. The calculated K_u values are shown in Table 2. Values of 20.95 kJm^{-3} and 23.10 kJm^{-3} using MOKE, and 14.51 kJm^{-3} and 18.80 kJm^{-3} using VSM were obtained from the *M-H* loops of the iron films. Magnetostriction coefficients of randomly oriented polycrystalline cubic iron can be calculated using equation (3)

$$\lambda_s = \frac{2}{5}\lambda_{100} + \frac{3}{5}\lambda_{111} \tag{3}$$

where λ_s is measured along the field direction. For cubic iron there are two independent magnetostriction constants λ_{100} (21×10^{-6}) and λ_{111} (-21×10^{-6}) [7]. The theoretical value for iron is a small negative magnetostriction of -4.2×10^{-6}. Stress induced magnetic anisotropy refers to the dependence of the anisotropy energy on the state of the strain of the lattice or microstructure. This dependence occurs as a result of the magnetostrictive nature of a material [8]. For a material with isotropic magnetostriction the anisotropy contribution is given by

$$K_u(\sigma) = \frac{3}{2}\lambda\sigma. \tag{4}$$

The stress induced in the films as a result of tightly wrapping the substrate was calculated using equation (3) and is shown in Table 2. The associated strain arises from a reorientation of the atoms in the material, which consequently causes a rearrangement of the atomic magnetic moments. The magnitude of the anisotropy depends upon the magnitude of the stress and on the value of magnetostriction. The induced stresses are 3.33 GPa and 3.67 Gpa for MOKE measurements and 2.30 GPa and 2.98 Gpa for VSM measurements. The stresses in the samples are quite large. It should note that the in-plane anisotropy was estimated from the *M-H* loops to give a value for induced stress. The stresses in sample B values for sample B were found to be in the same range as sample A. The rotation of the substrate has only a small effect on the observed uniaxial anisotropy. A tensile stress along the rotation direction of the substrate is likely to make domain magnetisation perpendicular to this direction for iron because of its negative magnetostriction. This gives the main contribution to the uniaxial magnetic anisotropy in the films. This shows that the tight wrapping of the substrate was the origin of the induced tensile stress. The samples retained this induced stress while being measured in the VSM and the MOKE system.

Table 2. The H_a, K_u and σ and strain values of the sputtered A, B films calculated from the data obtained using MOKE and VSM.

	MOKE			VSM		
Sample	H_a kA/m	K_u kJm^{-3}	σ GPa	H_a kA/m	K_u kJm^{-3}	σ GPa
A	19.50	20.9 5	3.33	13.50	14.5 1	12.30
B	21.50	23.1 0	3.67	17.50	18.8 0	2.98

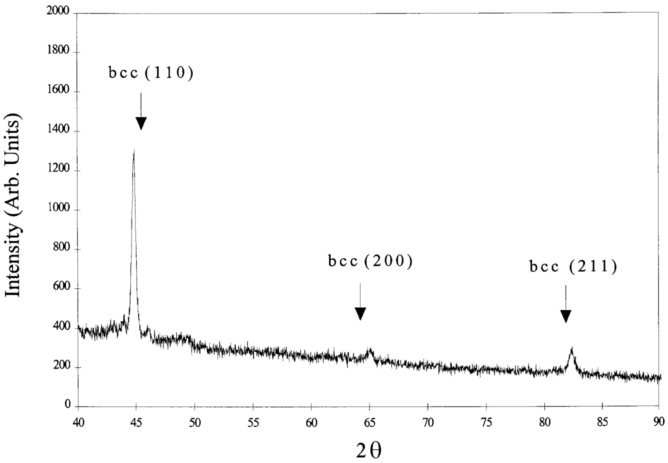

Fig. 5. A typical X-ray diffraction trace showing diffraction peaks due to the bcc α-iron structure in films produced using RC.

Magnetic measurements demonstrated that the cause of the uniaxial in-plane magnetic anisotropy was due to the RC technique. Further investigations using VSM and MOKE measurements suggest that the uniaxial anisotropy shifts to isotropic behaviour when producing materials on stationary substrates and with no wrapping.

3.3 Physical analysis

The structural characteristics of the films were determined using XRD (X-ray diffraction). Bragg reflections of the samples were obtained for $2\theta = 45°, 65°, 82.5°$ as shown in Figure 5. These angles represents the (110), (200), and (211) reflections of a body-centred cubic (bcc) structure for α-iron. TEM analysis also confirmed bcc α-iron phase.

4 Conclusions

The feasibility of a novel RC system for the preparation of magnetic films has been demonstrated. Results showed that films produced in the RC system generally exhibit an in-plane magnetic anisotropy which is very close to being uniaxial in nature with the tendency for the easy axis to lie close to the direction in which the film plane lies during the deposition. Further measurements and estimations showed that the cause of the anisotropy is due to the stress induced by tightly wrapping the film in the RC system, which means a tensile stress component in the film parallel to its length. The wrapping mechanism combined with the rotation of samples leads to a better-defined uniaxial in-plane anisotropy. On the other hand, initial observations of the applied stress on the films suggest a stress dependence on magnetisation when measured using VSM system. Therefore, it is proposed to deposit stress sensitive magnetic materials structures paving the way to a magnetoelastic sensor applications.

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References

1. B. Heirrich, J.A.C. Bland, *Ultrathin Magnetic Structures*, Vol. 1 (Springer-Werlay Berlin, 1994).
2. H. Kockar, D. Atkinson, T. Meydan, B. Mile, A.J. Moses, P. Silman, Nonlinear Electromagn. Systems **10**, 458 (1996).
3. T. Meydan, H. Kockar, Nonlinear Electromagn. Systems **13**, 491 (1998).
4. P.H. Dionisio, Thin Solid Films **217**, 152 (1992).
5. G. Suran, *et al.*, J. Appl. Phys. **8**, 61 (1987).
6. M. Rivas, *et al.*, J. Magn. and Magn. Mater. **166**, 53 (1997).
7. D. Jiles, *Introduction to Magnetism and Magnetic Materials* (Chapman and Hall, London, 1991).