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Substrate type and their rotation speed controlled magnetic anisotropy of iron films evaporated by a newly designed vacuum coating system

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A newly designed Vacuum Coating System (VCS) system has been used for the first time to prepare magnetic iron thin films on plastic kapton and rigid glass substrates at different rotation speeds (0 rpm, 30 rpm, 60 rpm, and 90 rpm). The powdered iron as a source material was evaporated by a resistively heated furnace positioned right under the substrate within the VCS system. Magnetic measurements showed that an in-plane magnetic anisotropy exist in all films. The films deposited on flexible kapton show that the degree of uniaxial magnetic anisotropy and coercivity decreased with decreasing rotational speed. For glass substrates used at low speeds (0 rpm and 50 rpm) magnetic isotropy was observed and coercivity was found to be almost constant while a slight anisotropy and a small change of coercivity was observed at 90 rpm. Coercivity of the films deposited on kapton substrates was found to be higher than that of the films on glass substrates. The estimation of in-plane uniaxial anisotropy was obtained from the hysteresis loops of the films confirmed the experimental in-plane magnetic anisotropy findings. Observations indicate that the uniaxial in-plane magnetic anisotropy and coercivity are dependent on the type of substrate and their rotation speeds.

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1. Introduction

In recent years, important developments have taken place concerning the production of magnetic thin films in order to utilise their unique magnetic and physical properties over a wide range of applications, especially in the sensor and storage technology sectors [1,2]. Therefore, this has provided the motivation for the development of new fabrication methods and improvements in existing production techniques [3-11]. A novel Vacuum Coating System (VCS) system has been designed to produce magnetic materials for the potential sensor and transformer core applications. In order to understand the magnetic characteristics of the VCS system, the effect of the substrate type and its rotation speed on magnetic anisotropy and coercivity of the films was studied and presented here. For this purpose, a series of samples was evaporated onto flexible and rigid rotating substrates under different rotation speeds using a resistively heated furnace situated in the VCS system. Estimation of magnetic uniaxial anisotropy obtained from hysteresis loops were found to be consisted with the experimental results.

2. Experimental procedure

In the VCS system, the target materials can be deposited on to substrates rotating up to 100 rpm at ~10^{-6} mbar pressure. In this study, 100 nm iron films were evaporated by depositing iron on to both flexible kapton and rigid glass substrates. Before each experiment commenced, the substrates were placed inside the VCS which was then pumped down to a pressure of ~10^{-6} mbar. This was followed by the rotation of the substrate holder at one of the following speeds: 0 rpm, 30 rpm, 60 rpm and 90 rpm. Powdered iron (purity 99.0 % and 1-450 micron in diameter supplied by Goodfellow) was vaporized from a resistively heated tungsten pouch, and the substrate holder was at room temperature. Film thickness was measured using a Maxtek TM100 thickness monitor.

A purpose built Magneto Optic Loop Plotter (MOKE) system was primarily used to obtain hysteresis loops of the samples at 0^0, 30^0, 60^0 and 90^0 for each pre-set rotational speed value. These are arbitrary angles selected so that the 0^0 and 90^0 equal the easy and hard axis direction for anisotropic films, respectively. Comparative measurements and perpendicular hysteresis loops were carried out using a Vibrating Sample Magnetometer, VSM (The DMS Model EV9, ADE Technologies). The composition of the source material and the produced films were analysed using Inductively Coupled Plasma Atomic Emission Spectrometry, ICP-AES (Perkin Elmer Optima 3100 XL) at room temperature.

3. Results and discussion

3.1 Experimental characterisation of magnetic anisotropy

Under study, kapton substrates were placed in the centre of the sample holder on the inner drum of the VCS system and rotated at a speed of 90 rpm, 50 rpm and zero rpm, respectively.For each speed value, Fig. 1 shows the normalised remanence ratio, M_r/M_s obtained from the hysteresis loops of evaporated iron films on kapton substrates measured at angles of 0^0, 30^0, 60^0 and 90^0 in the film plane. The coercivities obtained from the loops are summarised on the left hand side of Table 1. In Fig. 1, the films deposited on kapton at 90 rpm show well defined uniaxial in-plane anisotropy whereas the films produced at 50 rpm have a less well-defined anisotropy. When the substrate on the drum was stationary (zero rpm) during the deposition, M_r/M_s values indicate a slight in-plane uniaxial anisotropy compared to the film produced at 90 rpm and 50 rpm. For the films on kapton, the decrease in rotation speed makes the uniaxial magnetic anisotropy less well defined, also there is a smaller spread in coercivities for each direction, see Table 1. Uniaxial anisotropy in some materials can be induced by applying a magnetic field during the deposition process [12, 13]. In this investigation, the easy axis is at 0^0 and its origin is probably due to both the rotating substrate and the type of substrate used which may have introduced stress into the sample during deposition. Therefore, in order to differentiate this effect further sets of films were deposited on rigid glass substrates at speeds of 90 rpm, 50 rpm and 0 rpm.

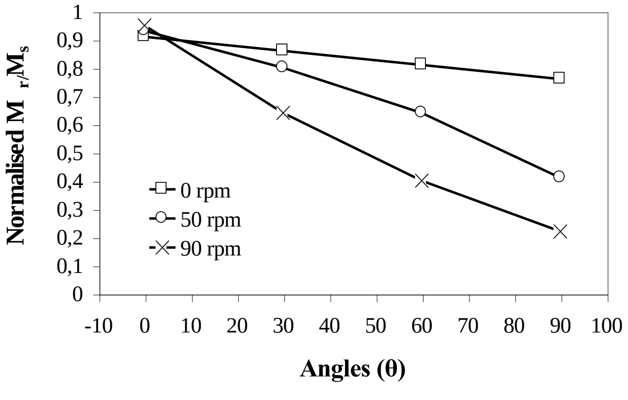

Fig. 1. The remanence ratio, M_r/M_s versus angles ($\theta=0^0$, 30^0, 60^0 and 90^0) at the rotating speed of 90 rpm, 50 rpm and stationary (0 rpm) for iron films evaporated on kapton substrate.

Fig. 2 shows the normalised M_r/M_s obtained from the hysteresis loops of evaporated iron films on glass substrates. The films deposited on glass substrates at 90 rpm exhibited a slight in-plane magnetic anisotropy. Although the features characteristic of in-plane anisotropy was observed, the M_r/M_s are different from that of the films deposited on kapton substrate at 90 rpm. A smaller spread in coercivities for each direction also confirms slight uniaxial magnetic anisotropy. Furthermore, although there are slight variations between each loop with angles, the films produced on glass substrates at 50 rpm and 0 rpm (stationary) does not show any sign of in-plane anisotropy. These results indicate that the substrate has an effect on the observed uniaxial anisotropy.

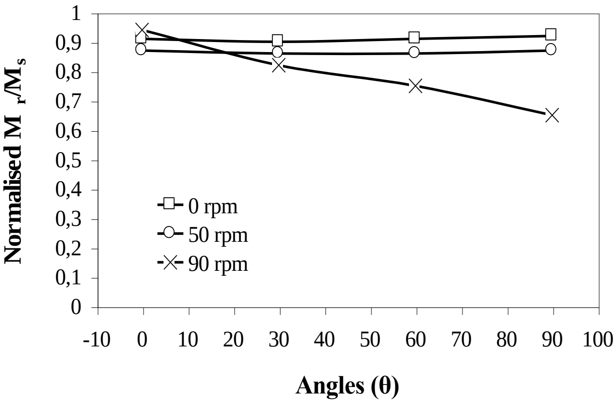

Fig. 2. The remanence ratio, M_r/M_s of iron films evaporated on glass substrate and measured at the angles of 0^0, 30^0, 60^0 and 90^0 for the rotating speed of 90 rpm, 50 rpm and stationary (0 rpm).

In some cases, the measured coercivities of the films were up to 12.25 kA/m. Dionisio et al [13] reported that bcc iron films have coercively values of around 6 kA/m. The coercivities of the films reported here have similar values to those found in reports [12-14]. The effect of substrate type on coercivity is clear from Table 1. In general, films on kapton had coercivities greater compared to those films deposited on glass under identical conditions. The rough texture of flexible kapton compared with very smooth surface of rigid glass substrates is likely the cause of increase in coercivity.

The results showed that the main reason for the anisotropy of the films produced on kapton substrates are that the rotation of the substrates, which might cause the stress effects in the plane of the films. The higher degree of anisotropy for the films deposited on kapton could be further aided by the flexible nature of plastic substrates. The implication here is that glass substrates may provide a very smooth surface for film deposition compared with the surface features of kapton, which can be observed microscopically and are seen in the film as well. Therefore, kapton film might induce further stress in the film, which might be inherent in flexible kapton substrates.

The films produced at the speeds of 70 rpm and 25 rpm fills the gap for the data confirming the results obtained at the speeds of 90 rpm, 50 rpm and zero rpm. The M_r/M_s and coercivity values obtained from magnetisation loops were also confirmed by VSM measurement, which have produced very similar hysteresis loops. Perpendicular anisotropy measurements were carried out using VSM. As a result of the demagnetising effect the film shape anisotropy dictates that specimens must have a planar easy axis. Normalized data from ICP-AES analysis confirmed that elemental concentrations had almost the same composition as the source material.

3.2 Theoretical estimation of magnetic anisotropy

In order to verify the results obtained by now, an estimation of in-plane anisotropy can be made of the data obtained from the hysteresis loops of the films deposited on kapton and glass substrates, respectively. The anisotropy field, H_a was taken as the magnetic field required saturating the film specimen along the hard axis, i.e. 90^0. The intrinsic uniaxial anisotropy, K_u can be estimated from equation 1[10], assuming the saturation magnetisation (M_s= 1.71 × 10^6 A/m) for extrapolation in hysteresis loops to find the H_a.

$$K_u = \frac{1}{2}\mu_0 H_a M_s \qquad (1)$$

This equation was only applied to those films with clearly defined easy and hard directions in their hysteresis loops. Therefore, the calculated K_u values are shown in Table 1 for the rotation speeds of 90 rpm, 50 rpm and zero rpm, respectively. 25.20 kJm^{-3} was obtained from the loops of the iron films evaporated on to kapton whereas the films on glass yielded a value of 10.80 kJm^{-3} at the same speed of 90 rpm. The magnetostriction coefficient of randomly oriented polycrystalline cubic iron has been calculated to be a small negative magnetostriction of -4.2 × 10^{-6} from two independent magnetostriction constants λ_{100} (21 × 10^{-6}) and λ_{111} (-21 × 10^{-6}) [15]. Stress induced magnetic anisotropy refers to the dependence of the anisotropy energy on the state of strain of the lattice or microstructure. This dependence occurs as a result of the magnetostrictive nature of a material [15]. For a material with isotropic magnetostriction the anisotropy contribution is given by;

$$\sigma = \frac{2K_u(\sigma)}{3\lambda} \qquad (2)$$

Table 1. Values of the coercivity and the computed anisotropy values, H_a, K_u and σ of iron films evaporated on kapton and glass substrates measured at 0, 30, 60 and 90 degree for the rotation speed of 90 rpm, 50 rpm and 0 rpm.

		Coercivity (kA/m)				**Properties of Uniaxial In-plane Magnetic Anisotropy**			
Rotation Speed	Substrate	90°	60°	30°	0°	Uniaxial Anisotropy	H_a (kA/m)	K_u (kJm^{-3})	σ (Gpa)
90 rpm	Kapton	2.05	6.65	9.75	12.25	Yes	23.45	25.20	4.00
	Glass	3.00	3.35	3.95	4.20	Slight	10.05	10.80	1.71
50 rpm	Kapton	2.20	4.95	6.25	7.90	Yes	17.35	18.65	2.96
	Glass	4.05	3.95	4.25	4.10	No	-	-	-
0 rpm	Kapton	3.95	4.30	4.85	5.05	Slight	11.55	12.41	1.97
	Glass	3.25	3.15	3.00	3.20	No	-	-	-

The stress induced in the films as a result of the rotation speeds of the flexible type of the substrate used was calculated using equation 2 and is shown in Table 1. 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. In Table 1, the induced stresses are 4.00 GPa and 1.71 GPa for the films on kapton and glass substrates at the speed of 90 rpm, respectively. The stresses in the samples are quite large. It should be noted that the in-plane anisotropy was estimated from the loops to give a value for induced stress during deposition. The stress values for the films on kapton were found to be larger than the films on glass substrates and have decreased with the decrease of the rotational speed, which can be deduced by looking at Table 1.

4. Conclusions

Magnetic iron films have been produced at various speeds of rotating substrates using the newly designed VCS system. Magnetic measurements showed that the films produced on flexible substrates exhibit an in-plane uniaxial magnetic anisotropy and the degree of the anisotropy of the films on kapton increased with the increase of the rotational speed. The films on rigid substrates showed a slight in-plane uniaxial anisotropy at the speed of 90 rpm unlike those deposited at 50 rpm and zero rpm. The rotation of the sample combined with the flexible nature of the substrate leads to a well-defined in-plane uniaxial anisotropy. Estimations of anisotropy values also verified the findings of magnetic anisotropy. Future developments will involve the deposition of various materials in the form of alloys and multilayers by using additional sources when considering potential applications of sensors and recording media materials.

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References

[1] C. Chappert, A. Fert, F. N .V. Dau, Nature Materials **6**, 813 (2007).
[2] J. Liu, Z. Guo, F. Meng, Y. Jia, J. Liu, J. Phys. Chem. C, **112**(15), 6119 (2008).
[3] J. E. Mahan, Physical Vapor Deposition of Thin Films, (Wiley-VCH, January 2000).
[4] D. Ruiz, T. Ros-Yanez, R. E. Vandenberghe, E. De Grave, Y. Houbaert, Revista de Metalurgia, **40**, 374 (2004).
[5] T. Meydan, H. Kockar, European Physical Journal-B, **24 (**4), 457 (2001).
[6] H. Kockar, T. Meydan, Journal of Magnetism and Magnetic Materials, **242-245P1**, 187 (2002).
[7] H. Kockar, T. Meydan, Physica-B, **321**, 124 (2002).
[8] T. Meydan, H. Kockar, P. I. Williams, Journal of Magnetism and Magnetic Materials, **254–255**, 91 (2003).
[9] T. Meydan, H. Kockar, J. Optoelectron. Adv. Mater. **6**(2), 633 (2004).
[10] H. Kockar, Journal of Superconductivity, **17**(4), 531 (2004).
[11] J. Bydzovsky, L. Kraus, P. Svec, M. Pasquale, M. kollar, Sensor Actuat A-Phys, **110**, 82 (2004).
[12] G. Suran, K. Ounadjela, F. Machizaud, J. Appl. Phys. **61**, 3658 (1987).
[13] P. H. Dionisio, Thin Solid Films, **217**,152 (1992).
[14] M. Rivas, J. F. Calleja, M. C. Contreras, J. Magn. Magn. Mater. **166**, 53 (1997).
[15] D. Jiles, Introduction to Magnetism and Magnetic Materials, (Chapman and Hall, London, 1991).

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