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The rotation and clamping effect on the magnetic properties of iron films deposited onto a rotating substrate

Hakan Kockar[a,*], Turgut Meydan[b]

[a] *Physics Department, Science and Literature Faculty, Balikesir University, 10100 Balikesir, Turkey*
[b] *Wolfson Centre for Magnetics Technology, School of Engineering, Cardiff University, Cardiff, UK*

Abstract

We have investigated the effect of fabrication conditions on the magnetic anisotropy of iron films produced by a novel rotating cryostat (RC) system. Clamped films exhibited uniaxial in-plane anisotropy with the easy axis in the film plane and perpendicular to the rotation direction of the RC system. An unclamped one showed no uniaxial magnetic anisotropy. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Anistropy; Rotating cryostat; Magnetism; Iron films

1. Introduction

Magnetic thin film science and technology is changing rapidly, and there is great interest in new ways to produce materials for applications such as magnetic recording heads, recording media, sensors, etc. [1]. This study reports results from a technique that might have the ability to produce new magnetic materials for applications, a novel rotating cryostat (RC) system. The new system consists of a rapidly rotating, liquid nitrogen cooled drum, around which a substrate can be wrapped. The possibility of using multi-port sources gives the system-wide capabilities for producing new materials [2]. The aim of the present study is to prepare and characterise films of a standard magnetic material, Fe, and to understand how the magnetic properties of the films depend upon the details of the production process, especially upon whether or not the substrate is clamped to the rotating drum.

2. Experimental

The RC system utilises a physical deposition method in which the substrate is rotated. The target material is deposited onto a substrate wrapped around the circumference of a rapidly rotating, liquid nitrogen cooled, stainless-steel drum (13 cm diameter) (see Fig. 1) that can be rotated at speeds up to 2000 rpm in an evacuated chamber ($\sim 10^{-6}$ mbar). The deposition area of 80 cm^2 (2 cm wide) is much larger than the typical few centimetre square of static techniques. Up to 10 target sources can potentially be positioned on the periphery of the drum and hence many different materials may be continuously deposited in any prescribed sequence. In this investigation,

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*Corresponding author. Fax: +90-266-245-6366.
E-mail address: hkockar@balikesir.edu.tr (H. Kockar).

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a DC magnetron sputtering source and a resistively heated furnace were positioned next to the rotating surface of the drum, and 100 nm thick iron films were produced at a rotation speed of 1300 rpm.

Sputtered films were made from an iron disk (25 mm diameter, 0.8 mm thick, 99.8% pure) on to a 75 μm thick polymide (kapton™) substrate for magnetic analysis, or a potassium bromide substrate for transmission electron microscopy (TEM) analysis. The substrates and the target material were first prepared and placed inside the RC and the system was pumped down to $\sim 10^{-6}$ mbar. Before the deposition started, the inner and outer drum were filled with liquid nitrogen and then rotated at the speed of 1300 rpm. Sputtering was carried out in a background gas consisting of 99.98% pure argon at $\sim 10^{-2}$ mbar. The Fe was deposited for half an hour. For evaporated films, the target material was 99.0% pure, 1–40μm diameter, powdered iron.

To calibrate the deposition rate, the film thickness was measured after the fact with a MAXTEK thickness monitor.

Anisotropy measurements were made on 6 mm outer diameter specimens at room temperature, using a vibrating sample magnetometer (VSM) (NUVO, Molspin Ltd.). The applied magnetic fields extended up to 50 kA/m and were applied perpendicular and parallel to the film specimen at various angles to the rotation direction. The crystal structure of the iron films was investigated with a JOEL TEM.

3. Results and discussion

Magnetisation measurements were performed to examine in-plane and out-of-plane anisotropies. In samples designated by case 1 in Table 1, Figs. 2

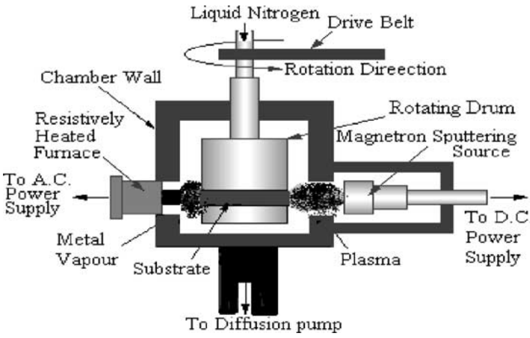

Fig. 1. The schematic diagram of the RC system showing the general principal of the system.

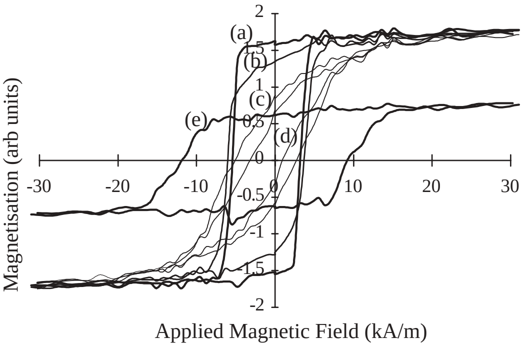

Fig. 2. Typical hysteresis loops of sputtered films with different angles ((a) 90°, (b) 60°, (c) 30°, (d) 0°, and (e) perpendicular to the film plane) between the applied magnetic field and the rotation direction of the RC (case 1).

Table 1
Results from VSM measurements of coercivity and M_r/M_s

Case	Production technique	Coercivity (kA/m)				M_r/M_s				In-plane magnetic anisotropy
		0°	30°	60°	90°	0°	30°	60°	90°	
1	Sputtering	2.05	3.90	4.90	5.20	0.28	0.41	0.74	0.89	Yes
1	Evaporation	1.10	7.40	11.30	12.60	0.11	0.50	0.84	0.96	Yes
2	Sputtering	2.60	3.00	3.25	3.70	0.44	0.62	0.75	0.90	Yes (less defined)
3	Sputtering	5.20	5.03	4.83	5.15	0.85	0.89	0.86	0.86	No

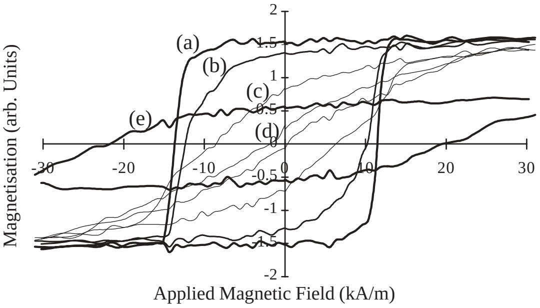

Fig. 3. Typical hysteresis loops of evaporated films with different angles ((a) 90°, (b) 60°, (c) 30°, (d) 0°, and (e) perpendicular to the film plane) between the applied magnetic field and the rotation direction of the RC (case 1).

and 3, the substrate was clamped onto the drum and rotated at 1300 rpm. An in-plane uniaxial anisotropy was produced in the samples with an easy axis 90° to the rotation direction of the cryostat.

Fig. 2 shows the hysteresis loops of a typical sputtered iron film with the field in the sample plane and at 0°, 30°, 60° and 90° to the rotation direction, and also perpendicular to the film plane. The loops become less square, with the remanent magnetisation M_r deviating further from the saturation magnetisation M_s, as the applied field direction is varied from 90° to 0°. Ninety degree to the rotation direction was the easy axis in all films produced during this investigation. This means that the samples have a well-defined uniaxial in-plane anisotropy. The coercivity also decreases as the angle is rotated from 90° to 0°. The coercivities and ratios of remanent to saturation magnetization (M_r/M_s) obtained from the films produced during this work are summarised in Table 1. When the field is applied at 90° to the rotation direction, the loop is almost square with a coercivity field H_c that differs from anisotropy field H_a. A comparison of the coercivity and anisotropy field was carried out with the extrapolation of the hysteresis loops, because the anisotropy fields between 1400 and 1700 kA/m were considerably larger than the standard laboratory fields of around 800 to −800 kA/m. This difference means that when the field is applied along the easy axis, the magnetisation does not rotate uniformly but there is nucleation and propagation of domain walls. Our measured coercivities ranged from 2.05 to 5.20 kA/m, similar to results on iron films in the literature [3,4].

When the field is applied perpendicular to the film plane, the saturation magnetization decreases and the coercive field increases. This indicates that the hardest axis is perpendicular to the film plane, as expected from the demagnetising effect. A perpendicular hard axis was found in all of our samples.

Fig. 3 shows the hysteresis loops of a typical evaporated iron film produced and measured under identical conditions to those used for the sputtered films. The data confirm the findings of in-plane magnetic anisotropy found for sputtered films.

The RC has produced films with well-defined in-plane magnetic anisotropy without applying any ex situ treatment. Special conditions of deposition or appropriate ex situ treatments can give rise to an in-plane magnetic anisotropy. In some materials, e.g., a uniaxial anisotropy can be induced by applying a magnetic field during the deposition process [4] or an external stress before the deposition [5]. In the present case, the easy axis is at 90° to the rotation direction of the rotating drum and is probably due to either the rotation of the drum or the clamping of the substrate onto the drum surface using a metal clamp, possibly introducing stress into the sample during deposition. To distinguish between these alternatives, films were deposited with clamping and a stationary cryostat (called case 2) and without clamping and a stationary cryostat (case 3).

Fig. 4 shows typical hysteresis loops of sputtered iron films for case 2. An in-plane anisotropy remains, but the variation with angle of the $M-H$ loops is weaker than those in Figs. 2 and 3. The stationary material production makes the uniaxial magnetic anisotropy less well defined due to closer coercivity values and M_r/M_s ratios for each direction (see Table 1).

Fig. 5 shows loops for case 3. The film no longer possesses a uniaxial magnetic anisotropy. Although there are slight variations between loops as the angle is varied, the film does not show any sign of an easy axis 90° to the rotation

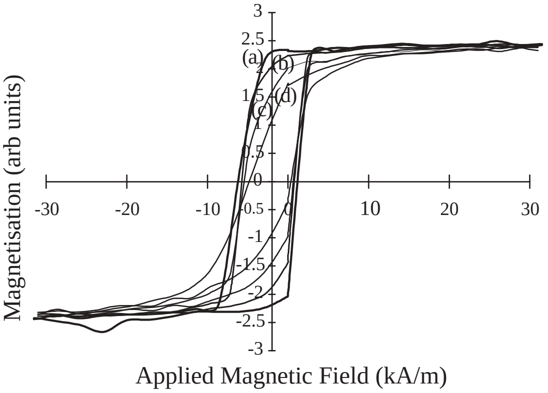

Fig. 4. Typical hysteresis loops of sputtered films with different angles ((a) 90°, (b) 60°, (c) 30°, (d) 0°, and (e) perpendicular to the film plane) between the applied magnetic field and the rotation direction of the RC (case 2).

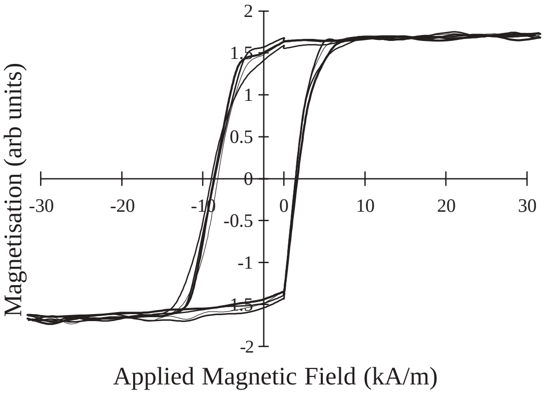

Fig. 5. Typical hysteresis loops of sputtered films with different angles (90°, 60°, 30°, 0°) and between the applied magnetic field and the rotation direction of the RC (case 3).

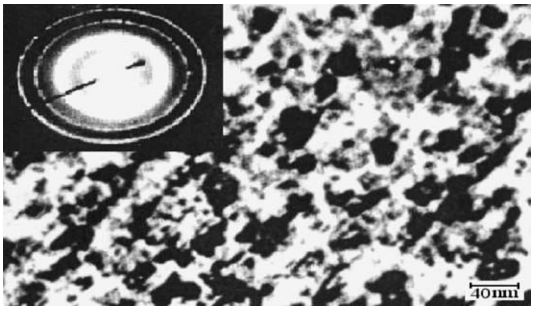

Fig. 6. A typical example of TEM micrographs showing the diffraction pattern produced by a sputtered film together with the crystalline microstructure of the sputtered film responsible for the diffraction pattern.

direction. The loops in Fig. 5 and the data presented in Table 1 indicate that these films are nearly isotropic.

The presence of a uniaxial magnetic anisotropy at 90° to the rotation direction in both cases 1 and 2 films is clearly due to the clamping, which is a stressing effect, during the deposition process. Rotation of the samples combined with clamping leads to a better-defined uniaxial in-plane anisotropy.

The structures of the thin films were examined directly by TEM. A diffraction micrograph and microstructure of typical sputtered films are shown in Fig. 6. The rings of sputtered samples corresponded with reflections from BCC α-iron. The films had a polycrystalline structure consisting of small randomly distributed crystallites with sizes in the range of 5–50 nm and a higher number of large crystals. The TEM micrograph of the sputtered film shows morphological features that indicate an inhomogeneous structure. XRD measurements also confirmed the BCC structure of α-iron.

4. Conclusion

The RC system has been used to produce thin films of magnetic iron. Clamped and/or rotated films had an in-plane magnetic anisotropy, presumably due to the stress induced by clamping the substrate onto the drum of the RC system, aided by rotation of the substrates during deposition. Rotation of the RC system makes it possible to produce long strips of materials, hence paving the way to production of long magnetic multilayer films using multiple sources.

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