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Anisotropy and magnetostriction constants of nanostructured Fe₅₀Co₅₀ films

N. A. MORLEY*, S. RIGBY, M. R. J. GIBBS

Dept. of Engineering Materials, University of Sheffield, Mappin Street Sheffield, S1 3JD, UK

In this study Fe₅₀Co₅₀ films were grown using two different deposition systems: DC and RF sputtering. The DC system allowed substrate rotation during fabrication, and had the facility to heat the substrate up to temperatures above 1100 K. The RF system had stationary substrates and no substrate heating. The thickness range of the Fe₅₀Co₅₀ films was from 10 nm up to 100 nm. For each film, the anisotropy fields were determined from the magnetisation loops measured using a magneto-optic Kerr effect magnetometer to establish the symmetry of any anisotropy. Measurements were taken as a function of the direction of the magnetic field with respect to the film edge. It was found that the rotated films were isotropic in-plane, while the non-rotated films showed uniaxial anisotropy. The magnetostriction constants were measured using the Villari technique. X-ray diffraction (XRD) was used to determine the texture of the films, and the grain size was found using the Scherrer equation. It was determined that to achieve highly magnetostrictive Fe₅₀Co₅₀ films with uniform thickness, the films have to be rotated during growth at elevated temperatures.

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1. Introduction

Bulk single crystal Fe₅₀Co₅₀, in the disordered phase, has a maximum magnetostriction constant of $\lambda_{100} = 150$ ppm, with a low first anisotropy constant of 10^4 J.m^{-3} [1]. If this performance can be matched in thin films, application to strain sensors and MagMEMS [2] is possible. Thin films (< 100 nm) produced by sputter deposition will be polycrystalline aggregates, although there may be strong texture. Detailed control of the fabrication processes of the film may influence both order and texture, and the *net* saturation magnetostriction constant in the film plane. This paper investigates how different growth conditions vary the magnetic and microstructural properties of thin Fe₅₀Co₅₀ films.

Previous work has studied the effect of underlayers [3,4], the substrate material [5] and the rate of deposition [6] on the magnetic properties of Fe_{1-x}Co_x films (where x ranges from 35 to 65). Jung *et al* studied 50 nm Fe₆₅Co₃₅ films grown on glass substrates and on thin Cu underlayers [4]. They found that the Cu underlayer induced a uniaxial anisotropy into the films compared to the isotropic films grown just on glass. The Cu underlayer also reduced the coercive field and the stress in the films. The magnetostriction remained around ~ 50 ppm, although the structure changed from <200> texture with no Cu layer to <110> texture with the Cu underlayer. Jung *et al* also studied other metallic underlayers [3], and found that NiFe, Ru and Ta/NiFe layers also induced uniaxial anisotropy in the FeCo films, while reducing the film stress. They determined this was due to the underlayer changing the texture of the film from <200> on glass to <110>. Vopsaroiu *et al* determined that the slower the

sputtering rate for 20 nm isotropic CoFe films (no composition given), the smaller the grain size and the lower the coercive field [6]. Cooke *et al* found that the substrate and the annealing temperature of 300 nm RF sputtered Fe₅₀Co₅₀ films varied the anisotropy and coercive fields [5]. In this paper, we present our work on thin Fe₅₀Co₅₀ films grown in two different deposition systems, using different fabrication parameters, and study how each one varies the magnetic and structural properties of the films. In this thickness range, it might be expected that the magnetostriction constants of the films would differ from the bulk polycrystalline value [7]. The variation of magnetostriction constant with thickness is usually inversely dependant and given by [8]:

$$\lambda_s = \lambda_v + \frac{\lambda_i}{t} \quad (1)$$

where λ_v and λ_i are the contributions to the magnetostriction constant from the volume and the surface/interface of the film and t is the film thickness.

2. Experimental Set-up

The Fe₅₀Co₅₀ films studied in this work were grown on silicon substrates with native oxide in either a DC or RF magnetron sputter system. The RF sputtered films were grown at 300 K, at 6.6×10^{-3} mbar Ar pressure and at a rate of 5 nm/min. The substrates were stationary during growth. Three different sets of films were grown in the DC system, at 7×10^{-3} mbar and at a rate of 5.2 nm/min. This

pressure was chosen as the films grown were isotropic and had the smallest anisotropy field on the pressure-distance (pd) plot for the deposition system [9]. The first film set was grown at 300 K and the substrates were rotated at 0.1 rps. The second set was grown at 300 K and the substrates were stationary during growth. The third set was grown at a substrate temperature of 600 K, with the substrates being rotated at 0.1 rps. The thickness range of the films was between 10 nm and 100 nm.

The films were characterised to determine how the magnetic and microstructural properties varied between the different sets. The magnetisation hysteresis loops were measured on a magneto-optic Kerr effect (MOKE) magnetometer. The loops were normalised to the saturation magnetisation. For each film, the magnetisation loops were taken as a function of angle with respect to one of the edges of the film. From each loop, the anisotropy fields, H_K , were determined and used to establish the symmetry of any anisotropy in the films.

The magnetostriction constant (λ_s) of each film was determined using the Villari method. This involved bending each film over five known bend radii ($R = 600$ mm – 200 mm) and measuring the magnetisation loop on the MOKE magnetometer [10]. The experimental magnetostriction constants were determined by plotting the anisotropy field as a function of the inverse bend radius, and using the equation [11]:

$$\lambda_s = \frac{dH_K}{d \frac{1}{R}} \frac{2\mu_o M_s (1-\nu^2)}{3\tau Y} \quad (2)$$

where H_K is the anisotropy field, R is the bend radius, ν is the Poisson ratio, τ is the thickness of the substrate and Y is the Young's modulus of the substrate.

The film's thickness, uniformity and surface roughness were measured on a Digital Instruments DI3000 atomic force microscope (AFM). On the same AFM, the magnetic structure of the films was measured using a CoCr coated MESP magnetic tip (magnetic force microscopy, MFM mode). Scanning parameters, particularly lift height, were chosen to ensure that there was no AFM breakthrough in to the MFM images. The MFM images were taken at a lift-height of 50 nm. X-ray diffraction (XRD) using a Cu source (wavelength = 0.154 nm) was used to determine the texture and the lattice constant of the films. The lattice constant, a , was determined from the $\langle 110 \rangle$ peak position. The average grain size, D , in the films was determined using Scherrer equation:

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (3)$$

where β is the full width at half maximum of the $\langle 110 \rangle$ peak.

3. Results and discussion

The anisotropy symmetry of each film was determined from the anisotropy fields as a function of applied field angle. For the 300 K DC rotated films the magnetisation loops were isotropic in-plane (Fig. 1a), similarly the 600 K DC rotated films were isotropic. For the non-rotated films (DC and RF), uniaxial anisotropy was observed, which was strong in the DC films, but weak in the RF films. One possible cause of the uniaxial anisotropy is the stray field from the magnetron gun. For the DC system, the stray field at the substrate was 80 Am^{-1} and for the RF system the stray field at the substrate was 2200 Am^{-1} . For the stray field to have a significant effect it must be capable of saturating the sample. In the DC case the stray field is much lower than the measured anisotropy field, but is the same order of magnitude for the RF films. This suggests that the DC non-rotated films uniaxial anisotropy was not caused by the stray field. Rotating the substrate during growth should also counteract the stray field from the magnetron gun, hence other mechanisms such as stress and shape anisotropy will cause the uniaxial anisotropy.

The anisotropy fields of the rotated DC films linearly decreased with increasing film thickness (Fig. 1b), while the anisotropy fields of the non-rotated films (both DC and RF) increased with increasing film thickness. Rotating the films in the DC system has reduced the anisotropy field by up to a factor of seven compared to the non-rotated DC films. Growing at elevated temperatures has reduced the anisotropy field by a further factor of two. The coercive fields of the 600 K DC rotated films and the RF non-rotated films were similar ($H_c \sim 2 \text{ kAm}^{-1}$) and were the smallest for the different film sets. The 300 K DC rotated film's coercive fields linearly decreased with increasing thickness, but were still at least a factor five larger than the 600 K DC rotated films. The DC non-rotated films had the largest coercive fields ($H_c \sim 20 \text{ kAm}^{-1}$).

From Fig. 2, it is observed that the deposition parameters effect the magnetostriction constant. For the DC magnetron sputtered films at 600 K substrate temperature and with rotation of the substrate during growth, and the RF non-rotated films, it is seen that the magnetostriction constants as a function of thickness do obey equation (1). For the 600 K film, the volume and interface magnetostriction constants are $\lambda_v = 63 \pm 1 \text{ ppm}$ and $\lambda_i = -1199 \pm 25 \text{ ppm.m}$, and for the RF film, $\lambda_v = 53 \pm 3 \text{ ppm}$ and $\lambda_i = -669 \pm 75 \text{ ppm.m}$. Thus the bulk magnetostriction constant of the 600 K films is within error of the bulk isotropic polycrystalline value of 66 ppm for $\text{Fe}_{50}\text{Co}_{50}$ calculated from the data in [1]. For the non-rotated DC films, it is observed that the magnetostriction constant increases linearly with film thickness, hence not obeying equation (1). For the DC magnetron sputtered at room temperature with rotation of the substrate, the magnetostriction constant is $11 \pm 3 \text{ ppm}$ for all thicknesses.

From Fig. 3a, the XRD scans of the different Fe₅₀Co₅₀ films are observed. In all cases only the {110} reflection was observed. For the DC rotated films, the <110> peak is observed in all thicknesses down to 30 nm, while for the non-rotated films (DC and RF), the <110> peak is not observed until over 35 nm of film has been deposited, but this may be an instrumental effect. Thus all films were textured, with the <110> direction out of the plane of the film.

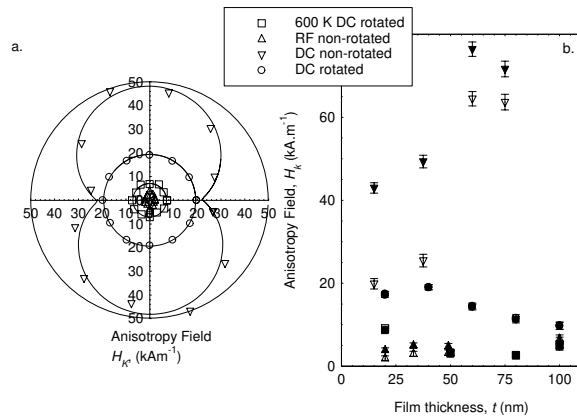


Fig. 1. (a) Anisotropy fields as a function of magnetic field direction with respect to the edge of the film. The solid lines are a guide for the eye. (b) Anisotropy field as a function of film thickness for each film set. The open shapes represent the easy axis and the closed shapes represent the hard axis.

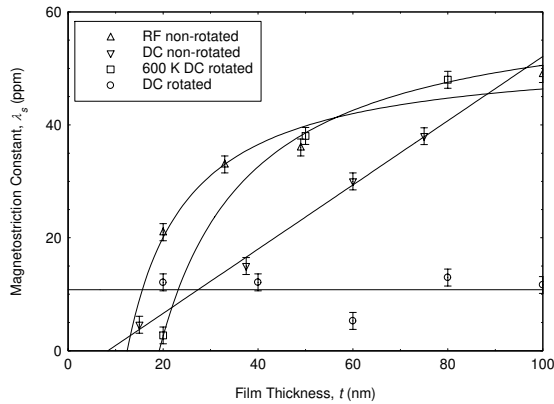


Fig. 2. Magnetostriction constant as a function of film thickness for each film set. The lines are a guide for the eye.

For bulk Fe₅₀Co₅₀, the <110> peak occurs at 44.97° with Cu radiation [12]. It is observed that for all the films the <110> peak has its centroid above 45°. This may be due to stresses in the films or the sample height displacement. Assuming that the shift in the peak was due to stresses in the films, the lattice constants were also determined (Fig. 3b). For all the films the lattice constants

were smaller than the bulk value. Taking a Young's modulus for a 2 μm thick FeCo film on Si as $Y = 165$ GPa [13], and the average strain given by the change in lattice parameter depicted in Fig. 3b, gives a compressive stress of $\sigma = 1.4$ GPa on the film. This is not an unreasonable value as taking,

$$K_\sigma = \frac{3}{2} \lambda_{\text{net}} \sigma \quad (4)$$

with $\lambda_{\text{net}} = 30$ ppm, yields a stress anisotropy constant of $K_\sigma \sim 6.3 \times 10^3$ J m⁻³. This in turn yields an anisotropy field

$$H_K = \frac{2K_\sigma}{\mu_0 M_s} \quad (5)$$

of 53 kA m⁻¹ using a saturation induction ($\mu_0 M_s$) of 2.4 T, which is of the order of the measured values (60 nm DC non-rotated film). The RF non-rotated and 600 K DC rotated films had lattice constants closest to the bulk value suggesting a lower film stress than in other films. It is these films that most closely follow equation (1). This may imply that the surface/interface magnetostriction does not dominate in films having high stress. Further investigation is required, starting with a simple post-deposition anneal of these films to reduce the residual stress. This work is ongoing.

The average grain size for each film was determined from the XRD data and Scherrer equation (equation (3)). We have ascribed all peaks broadening to grain size effects, rather than inhomogeneous strain, making the grain sizes quoted a lower bound estimate. For all the films grown in the DC sputter system, it is observed that the grain size was almost constant with thickness within the error of the experiment (Fig. 4), while the RF non-rotated films grain size decreased with decreasing film thickness. The grain size of the 600 K films was 25 nm, which was double that of the DC 300 K films ($D \sim 14$ nm) and the non-rotated 300 K films ($D \sim 12$ nm).

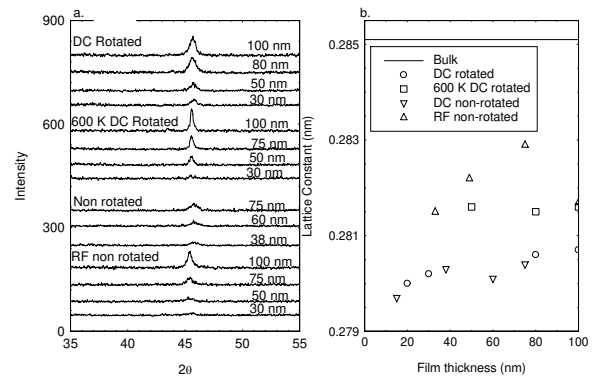


Fig. 3. (a) XRD data as a function of film thickness for all film sets. (b) the lattice constants determined from the XRD data.

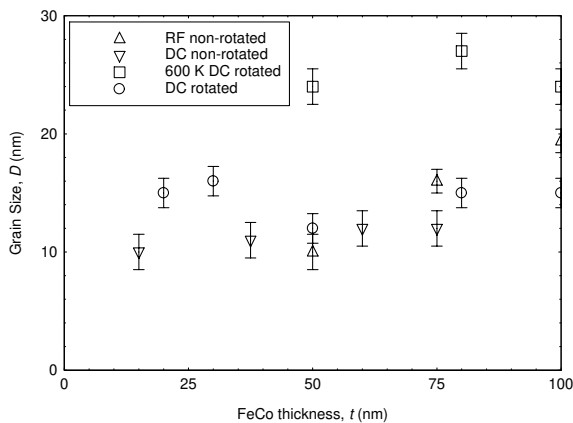


Fig. 4. Grain size as a function of film thickness for each film set.

For all the films, the surface roughness measured using the AFM was < 1 nm, which is observed as the featureless images in Figs. 5a & c. The film uniformity was also measured on the AFM. It was found that the DC rotated films were uniform thickness across the whole sample, while the DC non-rotated films were wedge shape across the sample. This is because the DC magnetron gun is at an angle to the substrate in the system, which means uneven coverage occurs when the substrates are not rotated. The RF non-rotated films were uniform at small thicknesses, while at larger thicknesses a convex surface was observed.

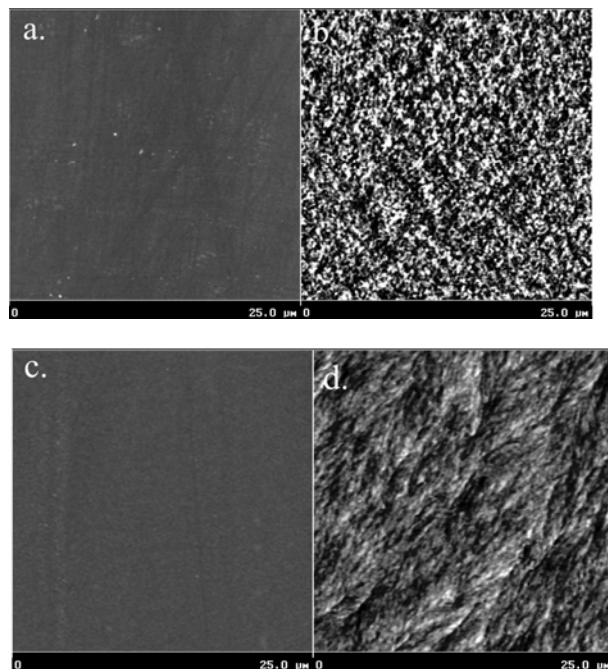


Fig. 5. The AFM/MFM image pairs of the 50 nm 300 K (a and b) and the 50 nm 600 K (c and d) DC rotated films.

The MFM images (Figs. 5b & d) show a difference in the magnetic phase contrast between the 300 K DC rotated film (Fig. 5b) and the 600 K DC rotated film (Fig. 5d). For the 300 K film, high magnetic phase contrast was observed. A high level of contrast implies strong and variable magnetic field gradients above the surface. This further suggests biaxial compression in the film (for a sample of positive magnetostriction, with the magnetisation pointing out of the sample plane), and is consistent with the earlier discussion. For the 600 K film, the MFM image shows little magnetic phase contrast, which means that the magnetisation is in the plane of the film. This means that the annealing of the film, which occurred during deposition and/or during cool down has reduced the stress in the film, allowing shape anisotropy to be the dominant anisotropy.

Although the DC rotated films were grown at the pressure which should have given minimum stress, they still had biaxial compressive stress in them, which was strong enough to force the magnetisation out of plane. With a single easy axis from the mechanism, we would expect to see the measured isotropy in the film plane. By growing films at elevated temperatures, the biaxial stress was reduced, which meant shape anisotropy was dominant in the films, thus the magnetisation was in the plane of the film. Elevated temperature growth also decreased the anisotropy fields and increased the grain size in the films, but the films were still isotropic in plane. By reducing the stress in the films, the coercive field was also reduced. The DC non-rotated films had a strong uniaxial anisotropy and magnetostriction constants with a linear dependence on thickness. The reason for this uniaxial anisotropy is probably the non-uniformity of the film thickness rather than the stray magnetron fields, as they were not strong enough. The RF non-rotated films had weak uniaxial anisotropy, which was probably due to the stray magnetron fields during growth. The magnetostriction constants had the same thickness dependence as the 600 K DC rotated films, and were similar order of magnitude. The disadvantage of these films is that at larger thicknesses, the film thickness becomes non-uniform as the substrate is stationary during growth.

4. Conclusions

The DC and RF films grown under no rotation had magnetostriction constants which depended on the film thickness and tended towards the bulk polycrystalline value. The main problem with them was that the film thicknesses were not uniform across the film and the anisotropy was uniaxial. Also the DC non-rotated film had large anisotropy fields and coercive fields in comparison to the other film sets. Rotating the substrate during growth improved the thickness uniformity, reduced the anisotropy fields, and produced isotropic films but suppressed the magnetostriction constants thickness dependence and reduced their magnitude; hence they were the same value at all thicknesses. By growing the films at elevated temperatures, with substrate rotation, the internal stresses

were reduced in the films. This meant that the magnetostriction constants became depended on the thickness again and were of similar magnitude to the non-rotated films plus the film thicknesses were uniform, the anisotropy fields were reduced and the films were still isotropic.

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*Corresponding author: N. A. Morley@sheffield.ac.uk