

Magnetic properties of dc magnetron sputtered and evaporated amorphous films

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Abstract. Compositions of $\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$, $\text{Fe}_{67}\text{Co}_{18}\text{Si}_1\text{B}_{14}$ and $\text{Fe}_{5.85}\text{Co}_{72.15}\text{Mo}_2\text{B}_{15}\text{Si}_5$ were deposited on to rigid and flexible substrates for the first time using a dc sputtering source as part of a novel Rotating Cryostat (RC). The films sputtered on silicon and glass show only isotropic magnetisation, whereas those sputtered on a polyimide (KaptonTM) substrate exhibited either isotropic or anisotropic magnetisation depending upon composition. Similar findings were obtained for equivalent evaporated films.

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

1 Introduction

In general amorphous magnetic materials are produced in the form of ribbons, wires or films and are manufactured from various alloys that exhibit a range of magnetic properties. Amorphous materials have generally a number of superior properties over crystalline materials such as higher electrical resistivity, flexibility without loss of hardness, high tensile strength and good corrosion resistance. The iron-based alloys such as $\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$ and $\text{Fe}_{67}\text{Co}_{18}\text{Si}_1\text{B}_{14}$ combine high saturation induction, very high permeability, and relatively high magnetostriction with low hysteresis loss. The cobalt-based alloys such as $\text{Fe}_{5.85}\text{Co}_{72.15}\text{Mo}_2\text{B}_{15}\text{Si}_5$ have even higher permeability, low loss and have near zero magnetostriction [1, 2]. Magnetostrictive films are sensitive to stress, depositing such a magnetic film on a pliable substrate of suitable dimensions may therefore enable it to be used as a magnetoelastic sensor. A novel rotating cryostat (RC) vacuum system originally designed to fabricate organic layers [3] has been developed to produce magnetic material. The thickness dependence of the produced film has been published previously [4]. In this paper, the effect of production parameters on the magnetic anisotropy of the films produced by dc magnetron sputtering and evaporation sources, which is implemented in RC, is investigated and presented.

2 Experimental

In contrast to the static techniques, the RC system can deposit material onto the surface of a rapidly rotating (up

to 2000 rpm) liquid nitrogen cooled substrate (13 cm diameter) under vacuum. The system has a large deposition area of 80 cm² (40 cm in length and 2 cm wide), and up to ten target sources can be placed around the RC. Making use of multiple target sources can therefore produce various magnetic material compositions and multilayers. Before each experiment commenced, the substrate was placed onto the inner drum surface inside the RC which was then pumped down to a pressure of $\sim 10^{-6}$ mbar. The inner and outer drums were then filled with liquid nitrogen before film deposition was started. Amorphous ribbons of atomic composition $\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$, $\text{Fe}_{67}\text{Co}_{18}\text{Si}_1\text{B}_{14}$ or $\text{Fe}_{5.85}\text{Co}_{72.15}\text{Mo}_2\text{B}_{15}\text{Si}_5$ were used as the source material. Sputtered and evaporated films were deposited onto stationary and rotating substrates respectively. 100 nm thick films were successfully produced on silicon, glass and KaptonTM substrates [5].

Magnetic investigations were carried out primarily using a magneto-optical Kerr effect instrument (MOKE) but comparative measurements were also made using a vibrating sample magnetometer (VSM). The MOKE system is illustrated in Figure 1 and utilises the transverse Kerr effect to measure loops proportional to the M - H loops. The system consists of a diode laser of wavelength 670 nm that is modulated at 500 Hz. The laser beam is split into two separate beams one of which is reflected off the film surface and the other that is used as a reference. The intensity of the two beams is equalised prior to taking measurements and fed into a lock-in amplifier. The output from the lock-in amplifier is digitised and sent to a computer together with a voltage signal, which is proportional to the applied magnetising field. The data can then be interpreted in the form of a magnetic hysteresis loop. The MOKE system has a sample mounting stage that allows the sample to be

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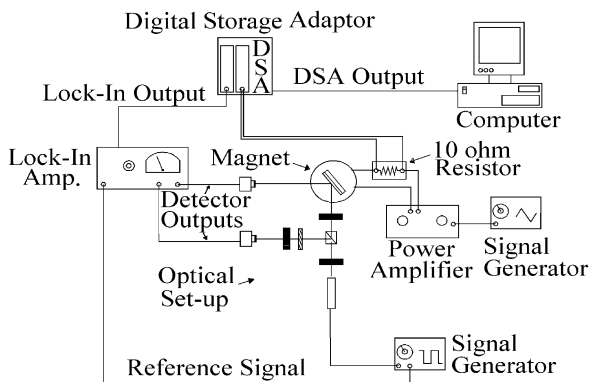


Fig. 1. The Magneto-Optic Kerr Effect loop plotter.

rotated about a horizontal axis with a precision of 1 degree. The sample can therefore be magnetised in the range of ± 60 kA/m in different directions within the film plane enabling an investigation of in-plane magnetic anisotropy. A circular film of diameter 1 cm was used for magnetic analysis to minimise demagnetising effects.

Physical structure was investigated using a Philips PW1820 X-ray diffractometer. The output as shown in Figure 2 is the intensity of diffracted X-rays plotted against twice the Bragg angle.

3 Results and discussion

A magnetic field was applied at different angles within the plane of the sample using MOKE and also perpendicular to the film plane using VSM at room temperature after the films have been taken from the RC system and put into the measuring units. A MOKE system was used to measure the properties of the produced thin films. During deposition the film plane was perpendicular to the horizontal plane. In-plane hysteresis loops were then measured at angles of 0° , 45° and 90° with respect to the horizontal plane. All films were highly reflective and mirror-like in appearance irrespective of the substrate used.

The amorphous nature of these film samples was confirmed by X-ray diffraction measurements. Figure 2 shows the X-ray diffraction trace for a $\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$ sputtered film on Kapton. To check the validity of the measurement an iron reference film was also measured along with an uncoated Kapton substrate. The iron reference gives a strong Bragg diffraction peak confirming the presence of a polycrystalline α -Fe phase. The amorphous film produces an almost identical trace to that of the blank Kapton substrate indicating the absence of crystallisation. Compositional analysis obtained using scanning electron microscopy indicated only small variations of film composition from the original amorphous ribbon source material.

All sputtered films on glass substrates exhibited isotropic magnetic behaviour within the film plane. An example of this is shown in Figure 3 for a film produced from a $\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$ ribbon target. This shows a number of

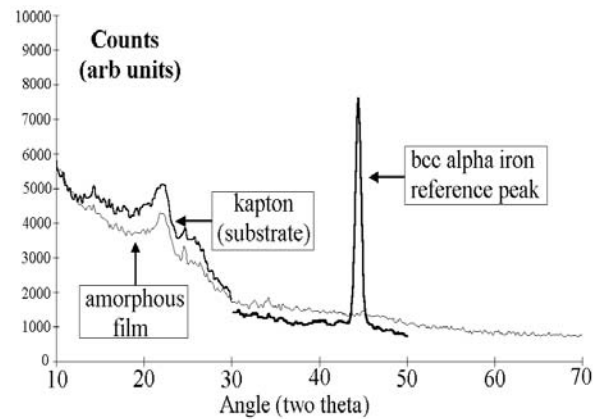


Fig. 2. X-ray diffraction pattern for an amorphous film deposited using $\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$ ribbon as the source material.

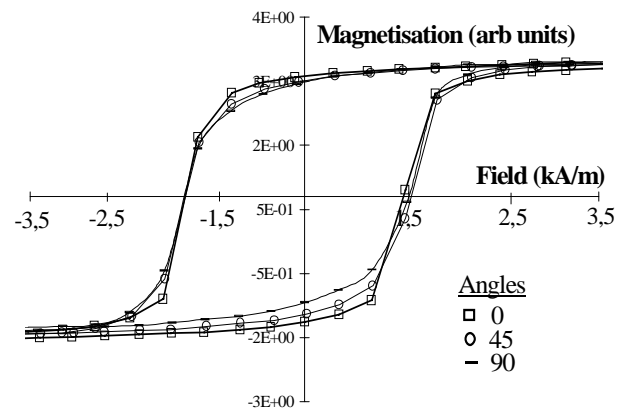


Fig. 3. M - H loops measured, using MOKE, for different directions in the film plane for a film specimen produced on glass from $\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$ ribbon.

M - H loops measured in various directions within the film plane. The angles represent directions with respect to the horizontal plane during deposition. The coercivity of these films is very high, over 2000 times greater, compared to those of the equivalent bulk material. This is also observed for films deposited on silicon substrates.

In the case where Kapton substrates have been used, it has been found that certain film compositions exhibit in-plane magnetic anisotropy. Three different film compositions have been investigated using targets made from $\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$, $\text{Fe}_{67}\text{Co}_{18}\text{Si}_1\text{B}_{14}$ or $\text{Fe}_{5.85}\text{Co}_{72.15}\text{Mo}_2\text{B}_{15}\text{Si}_5$ ribbons. The latter composition is cobalt based and is characterised by having almost zero magnetostriction, they produced films on Kapton with isotropic magnetic behaviour. The opposite was true for the films produced from the relatively high magnetostrictive materials, which exhibited in-plane magnetic anisotropy as shown for $\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$ in Figure 4. The implications here is that the relatively high magnetostrictive film specimens are sensitive to stress effects, which develop as a result of the flexible nature of the Kapton substrates. Stress effects are less likely to appear in rigid

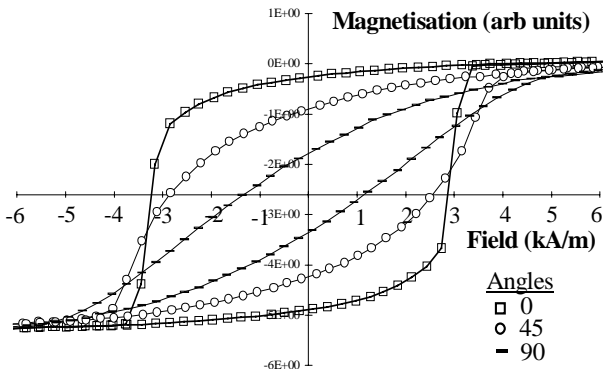


Fig. 4. M - H loops measured, using MOKE, for different directions in the film plane for a film specimen produced on Kapton from $\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$ ribbon.

substrates like silicon or glass unless a significant thermal mismatch between film and substrate occurs during deposition.

A number of mechanisms for in-plane anisotropy have been previously proposed [6]. These include shape anisotropy; due to tilted columnar or curve grain structures formed because of the oblique angle of incidence of the deposition material, or magneto-crystalline anisotropy when film texturing occurs. Stress effects have also been convincingly put forward as a cause of in-plane anisotropy in magnetic film media and it seems the most likely explanation for the observations made in this investigation.

An explanation for the high coercivity values observed in amorphous films is not easy to find. Other researchers have observed very high coercivity, greater than 400 A/m, and large in-plane anisotropy fields in sputtered cobalt rich amorphous films when compared to similar ribbon material [7]. The strain theory of domain wall pinning predicts that increase in coercivity is proportional to the increase in the number of sites of inhomogeneous microstrain [8]. Through magnetoelastic coupling domain wall motion is impeded when they interact with these localised regions of stress. Such regions are usually associated with structural deformities such as crystal dislocations. In this investigation amorphous structures were investigated which are expected to be of high quality due to the method of dc magnetron sputtering. The coercivity values summarised in Table 1 show that the highly magnetostrictive films do not generally exhibit higher coercivity than the non-magnetostrictive films. This is contrary to expectation if the magnetoelastic coupling is a dominant factor. Clarification of the significance of stress effects will only be possible once these films have been annealed in order to remove inhomogeneous strains.

The influence of substrate on coercivity is inconclusive as can be seen from the data in the Table 1. Generally smooth substrates result in softer films. An under-layer separating the magnetic film from the substrate has been shown to enhance soft magnetic properties [9]. Silicon and glass provide very smooth surfaces for film deposition whereas surface features on Kapton can be observed

Table 1. Magnetic characteristics of sputtered films on various substrates.

Target material (amorphous ribbon)	Substrate	Coercivity (kA/m)	In-plane magnetic isotropy
$\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$	silicon	4.55	yes
$\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$	Kapton	1.25 to 3.10	no
$\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$	Kapton	1.85 to 2.95	no
$\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$	glass	3.85	yes
$\text{Fe}_{67}\text{Co}_{18}\text{Si}_1\text{B}_{14}$	silicon	2.65	yes
$\text{Fe}_{67}\text{Co}_{18}\text{Si}_1\text{B}_{14}$	glass	5.70	yes
$\text{Fe}_{67}\text{Co}_{18}\text{Si}_1\text{B}_{14}$	Kapton	2.25 to 3.15	no
$\text{Fe}_{67}\text{Co}_{18}\text{Si}_1\text{B}_{14}$	Kapton	1.25 to 3.10	no
$\text{Fe}_{5.85}\text{Co}_{72.15}\text{Mo}_2\text{B}_{15}\text{Si}_5$	glass	4.45	yes
$\text{Fe}_{5.85}\text{Co}_{72.15}\text{Mo}_2\text{B}_{15}\text{Si}_5$	silicon	5.80	yes
$\text{Fe}_{5.85}\text{Co}_{72.15}\text{Mo}_2\text{B}_{15}\text{Si}_5$	Kapton	9.05	yes

microscopically and are seen in the film as well. However in this investigation there was no definite correlation of coercivity with the type of substrate used.

As expected, magnetisation was achieved most easily in the film plane due to the smaller demagnetising factor. Magnetisation loops obtained by the 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. The investigation of stress sensitivity using the VSM was conducted by inducing a bending stress in a $\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$ film sample on Kapton. Only small changes in the M - H characteristics were observed. It is thought that the film is stress biased after deposition and further increases in stress has little effect on magnetisation.

The emphasis of this work has been placed on the investigation of dc magnetron sputtered films, however measurements have also been performed on films evaporated from amorphous ribbons. As with sputter deposited films, evaporated samples have comparatively high coercivity of 1 kA/m or more. Initial observations on the amorphous evaporated films suggest a stress dependence on magnetisation when measured in the MOKE system using different sample fixation pressures. Further investigations of amorphous films is expected to reveal the factors affecting magnetic softness leading to improved properties suitable for sensor applications.

4 Conclusions

Amorphous magnetic film compositions have been successfully deposited using a d.c. magnetron sputtering and a resistively heated furnace in a RC vacuum system on

a range of substrates. These films have been found to exhibit relatively hard magnetic properties compared to those achievable in amorphous ribbon (in some cases the coercivity was over 2000 times larger than their ribbon counterparts). It is likely that no single parameter is responsible for this magnetic hardness. It is also vitally important that the origin of the in-plane anisotropy observed in $\text{Fe}_{81}\text{B}_{13.5}\text{Si}_{3.5}\text{C}_2$ and $\text{Fe}_{67}\text{Co}_{18}\text{Si}_1\text{B}_{14}$ compositions is resolved when considering magnetoelastic sensor applications.

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