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Characterisation of evaporated and laser-ablated 3% silicon–iron

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Abstract

A novel rotating cryostat vacuum system originally designed to fabricate organic layers has been used for the first time to evaporate 3% silicon–iron magnetic materials. Results of all films deposited on glass and silicon exhibit isotropic magnetic behaviour but the films produced on kapton generally show an in-plane magnetic anisotropy within the film plane. Results were also confirmed with the laser-ablated films. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Magnetic thin films; Anisotropy; Coercivity

1. Introduction

Due to improved manufacturing techniques and a better understanding of factors that control the magnetic properties, the quality of electrical steels has recently improved vastly. Silicon–iron main use as electrical steels is the magnetic core materials of rotating machines and transformers [1,2]. The primary focus of the work was the deposition of silicon–iron films with compositions equivalent to that of conventional electrical steel in order to investigate a possible future to produce large amounts of silicon–iron as the magnetic core material for power transformers. Therefore, a novel rotating cryostat (RC) [3–5] has been used for the first time to evaporate silicon–iron magnetic materials. The analysis of magnetic properties of films evaporated on plastic kapton, rigid glass or silicon substrates using a resistively heated furnace placed around the RC is investigated. The cause of the anisotropy has been discussed in detail on kapton substrates.

2. Experimental methods

The main distinction of this system from other thin film deposition systems is the liquid nitrogen cooled cylindrical drum, 13 cm in diameter (80 cm long, 2 cm wide) on which substrates are mounted. Therefore, the biggest advantage of such a system is its ability to produce long strips of material. With multiple sources the RC system was evacuated using a conventional oil diffusion pump with liquid nitrogen trap, which could attain base pressures down to 2×10^{-6} mbars. Before each experiment commenced, the substrate was placed to the drum of the RC at ambient temperature, and then the RC was pumped down to a pressure of $\sim 10^{-6}$ mbar. The inner drum and outer drum were filled with liquid nitrogen. Deposition was then started. The target material used in this study was 97% (1–40 μm diameter powdered) iron and 3% silicon. The deposition was continued over a time-period of half an hour. Approximately, 100-nm-thick films have been successfully produced while the RC was stationary.

The deposition of silicon–iron film specimens was also achieved using a 308 nm wavelength pulsed laser beam produced from a Lambda Physik LPX 315I excimer laser which was focussed onto a target rotating at a frequency of 3 Hz. The target was a bulk of silicon–iron (25 mm diameter, 0.8 mm thick, 99.8% pure) cut from

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an commercial electrical strip. The laser beam was focussed onto the target using a 30 cm focal length lens to produce a spot size of 1 mm^2. The resultant plasma cloud of material condensed onto the substrate was positioned directly above the target at a distance of around 3 cm. Typical deposition parameters consisted of a laser pulse fluence of 4.5 J/cm^2, a pulse frequency in the range 10–40 Hz and a total number of pulses for one deposition of around 40,000–50,000. Typical deposition rates of 0.03 Å per pulse were achieved which corresponded to film thicknesses of around 100 nm. All depositions were carried out in a vacuum chamber evacuated to a base pressure of 10^{-7} mbars. Films were deposited on to the same three substrates for comparative analysis.

3. Results and discussion

Magnetic measurements were made on the films in the form of circular discs approximately 1 cm diameter reflective and mirror-like in appearance irrespective of the substrates used. The field was applied along the rotation direction, 0° and at 30°, 60° and 90° to the rotation direction in the film plane and also perpendicular to the film plane. Measurements were primarily carried out using magneto-optic kerr effect (MOKE) but comparisons were made with measurements performed using a vibrating sample magnetometer (VSM).

It has been found that the films evaporated on kapton exhibit an in-plane magnetic anisotropy as shown in Fig. 1. This figure shows the hyteresis loops obtained at various angles relative to the rotation direction of the kapton substrate placed on the inner drum of the RC. In the Stoner–Wohlfarth model of uniaxial anisotropy [6,7] when the field is applied along the easy axis of a single domain sample the loop is a square with a coercive field H_c equal to H_a, where H_a is the anisotropy field. When the field is perpendicular to the easy axis the magnetisation varies linearly from H_a to $-H_a$ without hysteresis. The discrepancies of the results with respect to this model (nearly square loop measured in the easy direction and existence of a hysteresis in the hard direction) indicate that the observed anisotropy is uniaxial, which resembles the Stoner–Wohlfarth model.

In Fig. 1, when the field is applied along the rotation direction, the loop exhibits a smaller coercivity, 2.15 kA/m, in comparison with anisotropy field, 18.2 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, 8.25 kA/m. Coercivity field is different from anisotropy field 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 evaporated films show that the coercivities decrease when the angle of measurement is rotated from 90° to 0° to the rotation direction. In the film plane when the applied field is perpendicular to the rotation direction, the loop is almost square, whereas with applied field parallel to the rotation direction the loops remanence ratio has decreased. The magnetisation loops for the easy directions (and all directions in the isotropic films) were very square in shape with a remanence ratio close to unity and a very rapid reversal of magnetisation at the coercive field. This indicates the possibility of strong exchange coupling between grains in these films.

Further in-plane anisotropy investigation was carried out by measuring typical coercivity ratios of the evaporated films on kapton, see Fig. 2 and typical remanence ratio of the evaporated films on the same substrate, see Fig. 3 for different directions of the applied field in the film plane. Films were rotated through 360° and magnetisation-field loops were recorded at 10° intervals. The resultant plots of remanence ratio versus angle and coercivity versus angle both give two maxima corresponding to the easy directions (parallel and anti-parallel directions) and two minima corresponding to the hard directions. This also confirms that the observed anisotropy is indeed uniaxial.

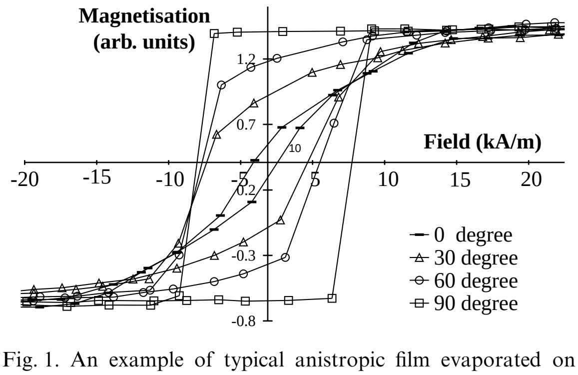

Fig. 1. An example of typical anistropic film evaporated on kapton measured using MOKE.

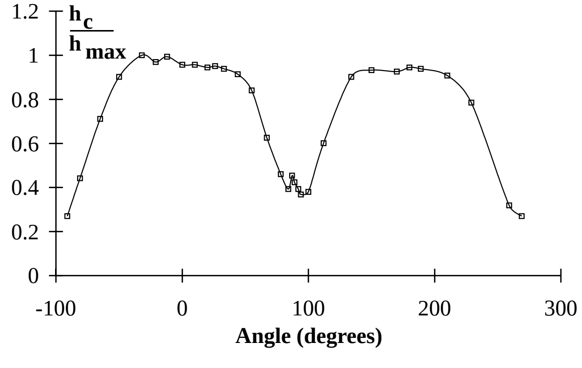

Fig. 2. Variation of coercivity field ratio versus magnetising angle of typical evaporated films on kapton.

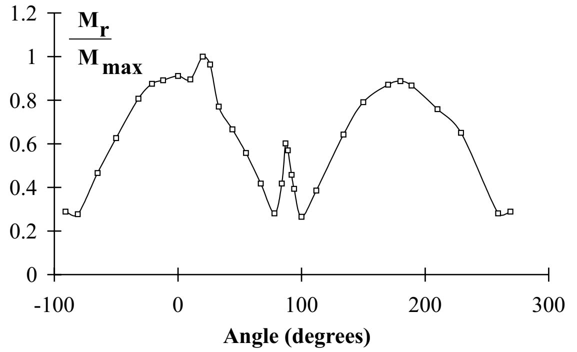

Fig. 3. Variation of remanent magnetisation with magnetising angle of typical evaporated films on kapton.

On the other hand, the films evaporated on glass substrate showed isotropic magnetic behaviour within the film plane. Although there was quite a slight variation between each loop with angle, the film does not show any sign of in-plane anisotropy. This is also seen for films deposited on silicon substrate.

The implication here is that glass or silicon substrates provide very smooth surfaces for film deposition compared with the surface features of kapton which can be observed microscopically and are seen in the film as well. Therefore, the kapton film specimens might induce stress in the film, which might be inherent in flexible kapton substrates. Stress effects are less likely to appear in rigid substrates like glass or silicon unless a significant thermal mismatch between film and substrate occurs. A number of mechanisms for in-plane anisotropy have been previously proposed [8]. Stress effects have 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.

In general the maximum coercivity is observed along the easy direction, whereas the minimum coercivity occurs when magnetising along the hard direction. Film depositions on kapton substrates were found generally to have higher coercivities than those deposited on glass. The coercivities measured on glass were in the range 0.1–4.8 kA/m, whereas on kapton the range was 0.6–6.2 kA/m. These values include measurements taken along both easy and hard directions and in between. The values obtained in this work are consistent with values quoted by other researchers [9,10], however, they are approximately 100 times larger than those measured in bulk electrical steel (12 A/m).

Scanning electron microscope was used to analyse the composition of the films. The average values indicate good compositional homogeneity across these films. The average variation of the silicon content measured in the films during this investigation was in the order of 1.1–3.5%.

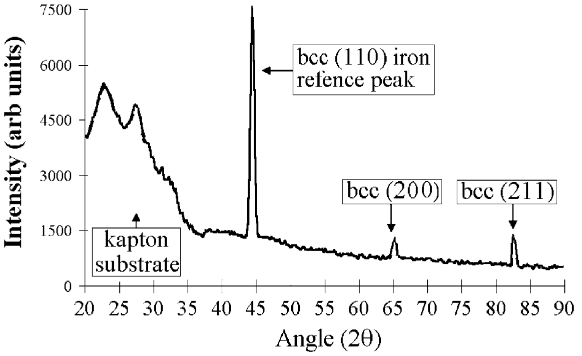

Fig. 4. Typical X-ray diffraction traces showing the BCC α-iron structure of evaporated silicon iron films on kapton.

In order to determine the structure of the films, X-ray diffraction measurements of typical evaporated silicon–iron films on kapton were carried out with Cu Kα radiation in the range of $10^\circ < 2\theta < 90^\circ$, where θ is Bragg's angle. Bragg reflections were obtained for $2\theta = 45^\circ, 65^\circ, 82.5^\circ$ as shown in Fig. 4. It can be clearly identified that the (1 1 0), (2 0 0) and (2 1 1) reflections of the body-centred cubic (BCC) structure of α-iron.

Separate VSM measurements made with the magnetising field applied parallel to and perpendicular to the film plane confirm that the film has in-plane magnetic anisotropy. 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. All film specimens studied during the course of this investigation showed the planar magnetisation loops of anisotropy.

The emphasis of this work has not only been placed on the investigation of evaporated films, but measurements have also been performed on laser-ablated films. As with evaporated films, laser-ablated samples have comparatively low coercivities of 2 kA/m or more with confirmation of isotropy and anisotropy cases obtained for evaporated films.

4. Conclusions

This investigation showed that silicon–iron films can be produced using the RC, and they exhibit very interesting magnetic properties especially in relation to their in-plane anisotropies. X-ray diffraction gave a strong peak at 2.03 Å which corresponded to Bragg reflections from a BCC α-iron structure. The factors effecting the in-plane anisotropy occurred in the films were found to be stress induced in the flexible substrates. Further measurements indicated that the films deposited on rigid substrates posses isotropic magnetic behaviour. The findings also demonstrate that with appropriate changes this method has technical advantages over other systems for producing thick single or layered magnetic strips. Relatively thin 40 cm × 2 cm magnetic strips have been deposited but this method has the potential for scaling up to produce thicker strips for the potential applications as power-transformer cores.

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