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Uniaxial in-plane magnetic anisotropy in silicon-iron films prepared using vacuum coating plant (VCP)

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Abstract. The novel VCP system is a mobile physical deposition method to deposit metallic/magnetic films using various source materials including powder, lump, pre-alloyed ingots and wires. The VCP system consists of a large deposition area of 960 cm^2 and has been used for the first time to prepare magnetic thin films of Si_3Fe_{97}. The source material evaporated by a resistively heated furnace, which was position right under the substrate within the VCP system, contains small pieces of conventional 3% silicon-iron steel as source materials. The magnetic analysis of the films was achieved by using a vibrating sample magnetometer (VSM). Observations indicate that the magnetic anisotropy and coercivity are dependent on the type of substrate and the deposition conditions. Results of all films deposited on flexible kapton^{TM} are anisotropic in the film plane whereas the films deposited on glass substrate indicate the less-well defined anisotropy in the film plane while the substrate holder of the VCP system was run at the speed of 100 rpm. In the case of stationary magnetic materials production, the films deposited on kapton and glass substrates show isotropic magnetic behaviour. All films showed planar magnetic anisotropy irrespective of type of substrate and the production conditions used. The findings are discussed in terms of scaling up the technique for the possible production of various shapes of circular, square or strip components with the compositions equivalent to that of conventional electrical steels in order to investigate a possible future to produce large scale of silicon-iron as the core materials for rotating machines and power transformers.

PACS. 75.30.Gw Magnetic anisotropy – 75.50.Bb Fe and its alloys – 75.70.Ak Magnetic properties of monolayers and thin films

1 Introduction

Development of new and traditional magnetic materials based on better understanding of magnetisation process will lead to significantly improved product performance, and new applications which would benefit most sectors of industry [1]. Better manufacturing techniques and improved understanding of the factors has improved the quality of electrical steels vastly [2,3]. Therefore, a novel Vacuum Coating Plant (VCP) system has been designed for the first time to evaporate silicon-iron magnetic materials formerly undertaken by the Rotating Cryostat (RC) system [4,5]. The main interest of this study was focused on the fundamental understanding of the process and the tools affecting the magnetic properties of silicon iron films. Approximately 100 nm thick silicon-iron films were deposited onto polyimide kapton and glass substrate under the conditions of stationary and rotated at the speed of 100 rpm and their magnetic analyses of the films are discussed in terms of magnetic anisotropy.

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2 Experimental

The VCP system has a large deposition area of 960 cm^2 compared to 80 cm^2 in Rotating Cryostat (RC) [4–8], and also a few cm^2 in static techniques. The target materials can be deposited on to the surface of a rotating (up to 100 rpm) liquid nitrogen-cooled substrate in an evacuated chamber (~10^{-7} mbar). Figure 1 shows a schematic diagram of the Vacuum Coating Plant (VCP) system. Films with good uniformity can be prepared using an adjustable source to substrate distance. By using up to six targets including evaporation and dc magnetron sputtering sources, many different materials can be deposited in any prescribed sequence; films of several millimetres thickness can be readily produced; making use of more than one target sources can generate magnetic multilayers. Furthermore, running costs of this technique are potentially cheap.

Table 1. Coercivity and remanence ratio of the evaporated 3% SiFe films.

Substrate	Rotation speed (rpm)	Coercivity (H_c) 0°	30°	60°	90°	Remanence ratio (M_r/M_s) 0°	30°	60°	90°	In-plane magnetic anisotropy
Kapton	100	3.35	4.29	4.48	4.78	0.64	0.82	0.87	0.97	Yes
Glass	100	2.05	2.23	2.43	2.68	0.73	0.79	0.86	0.97	Yes (less defined)
Kapton	Stationary	4.38	4.33	4.38	4.45	0.88	0.89	0.88	0.89	No
Glass	Stationary	1.93	1.95	1.90	1.98	0.79	0.80	0.80	0.80	No

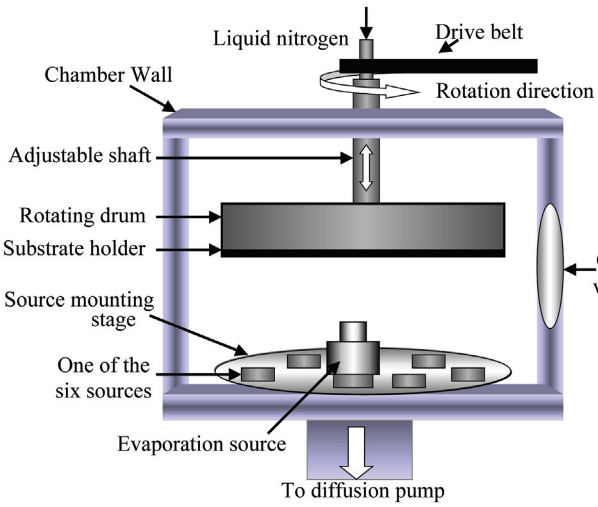

Fig. 1. A schematic diagram of vacuum coating plant (VCP) system.

In this study, silicon-iron films were produced onto a (10 cm × 30 cm) polyimide kapton and glass substrates, respectively. Before each experiment commenced, the substrates were attached on the surface of the substrate holder of the inner drum inside the VCP which was then pumped down to a pressure of $\sim 10^{-7}$ mbar with help of the liquid nitrogen. The purpose of using the liquid nitrogen was not only to help to lower the pressure in the camber but also to see if crystalline structure of the films shifted to an amorphous phase or not. The inner drum was then filled with liquid nitrogen before film deposition commenced. The system was run under two conditions. The substrate holder of the inner drum was run at the speed of 100 rpm in the first part of this investigation. Later the films were also evaporated on kapton and glass substrates at the same time while the system was stationary. Source material of commercially available 3% silicon electrical steel was vaporised from a resistively heated tungsten wire pouch. The graphite was required to prevent alloying between the molten iron and the tungsten. Future developments will involve the deposition of magnetic materials by using a second source such as dc sputtering source.

The films were structurally characterised using X-ray difractometer (XRD) and observed that they have bcc crystalline structure. It is seen that the use of liquid nitrogen has not an effect on crystalline structure of the films. Scanning electron microscope (SEM) was used to analyse the composition of the films. It was found that the films composition consistent with the source material of 3% silicon iron steel. The composition analysis results were also confirmed by using the inductively coupled plasma atomic emission spectrometry (ICP-AES). A vibrating magnetometer (VSM) was employed to determine the magnetic properties of the films.

3 Results and discussion

Magnetic measurements were carried out to examine in-plane and out-of-plane anisotropies at room temperature. In-plane hysteresis loops were measured at various angles using VSM. The total angular range of measurement in the film plane was 90°, with the 0° direction chosen arbitrarily. A circular film of 6 mm diameter was used to minimise demagnetising effects. In the first part of this investigation, silicon-iron films were evaporated on plastic kapton substrate while the substrate holder of inner drum in VCP system was rotated at the speed of 100 rpm. Figure 2 shows the hysteresis loops of a typical film measured in 0°, 30°, 60° and 90 directions in the film plane. In-plane hysteresis loops in this figure show that the sample has a well-defined uniaxial anisotropy. When the field is applied at 0° the loop exhibits a smaller coercivity, 3.35 kA/m, in comparison to anisotropy field, 15.13 kA/m, but not equal to zero. When the field is applied at 90°, the loop is almost square with a coercivity field of 4.78 kA/m that differs from the anisotropy field of 7.17 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. The ratios of remanent to saturation magnetisation (M_r/M_s) and coercivities obtained from the films produced during this study are summarised in Table 1. 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 was the easy axis in all films produced on kapton during this investigation under these conditions. The coercivity also decreases as the angle is rotated from 90° to 0°.

When the field applied perpendicular to the film plane, perpendicular anisotropy was found not to exist. Figure 3 indicates the hardest axis of the M-H loops is perpendicular to the film plane. As a result of demagnetising

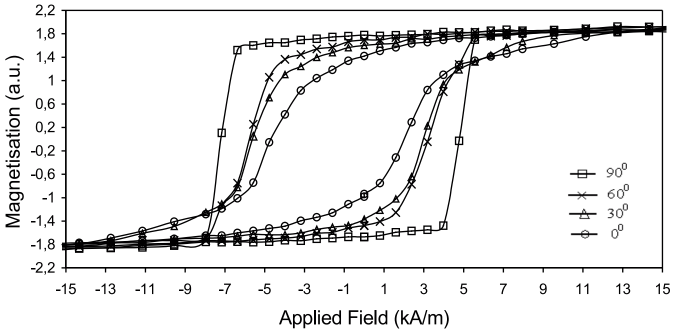

Fig. 2. An example of typical anisotropic silicon-iron film evaporated on kapton substrate (under the VCP rotation of 100 rpm) measured at various angles of 0° to 90°.

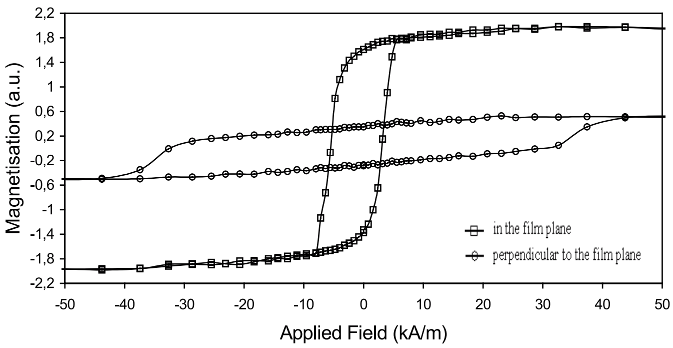

Fig. 3. Perpendicular anisotropy of evaporated silicon-iron film on kapton measured by using VSM.

effect, the film shape anisotropy dictates that specimens must have planar easy axis. This shows the planar magnetisation loops, typical of all polycrystalline film samples studied during this investigation.

The VCP system has produced the films with well-defined in-plane magnetic anisotropy without applying any ex situ treatment. Uniaxial anisotropy can be induced in many ways during deposition [9,10]. Anisotropy in some materials can be induced by applying a magnetic field during the deposition or an external stress before the deposition. In this study, the easy axis at 90° and is probably either the type of substrates used or the rotation of the sample, possibly introducing stress into sample during deposition. In order to distinguish between alternatives, further sets of films on glass substrate at the 100 rpm and also the films on both kapton and glass substrate under stationary conditions were evaporated and discussed in the second part of this study.

When the VCP is run at the speed of 100 rpm, an example of the hysteresis loops of films evaporated on glass is shown in Figure 4. An in-plane anisotropy remains, but the variation with angle of the hysteresis loops is weaker than those in Figure 2. In the film plane, the coercivities and remanence ratio decreases when the angle of measurement is rotated from 90° to 0° directions. The use of glass substrate makes the uniaxial magnetic anisotropy less well defined due to a smaller spread in coercivity values in Table 1. This means the substrate type has an effect on the observed uniaxial in-plane anisotropy.

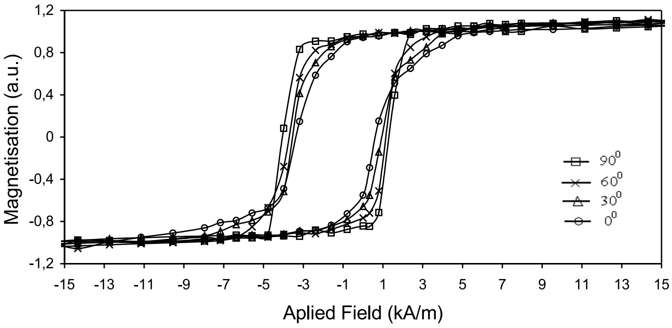

Fig. 4. In-plane hysteresis loops of the film deposited on glass substrate (under the VCP rotation of 100 rpm) as a function of angles of applied field.

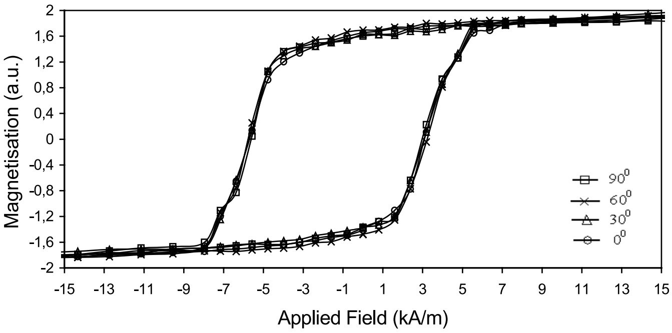

Fig. 5. In hysteresis loops of of typical film evaporated on kapton substrate under stationary condition.

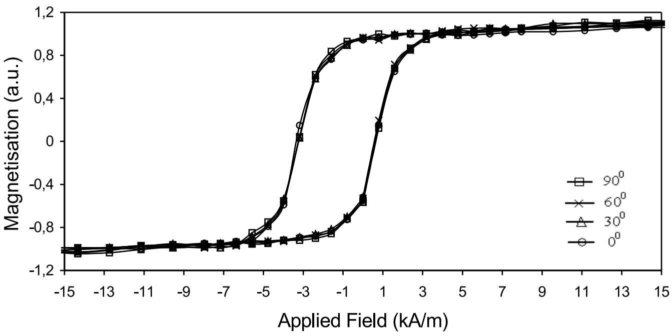

Fig. 6. In-plane hysteresis loops of evaporated silicon-iron film on glass substrate (under stationary condition) measured at arbitrary chosen angle of 0°, 90°.

The films deposited on kapton show higher degree of magnetic anisotropy compared to that of the glass substrate at the speed of 100 rpm. The reason for the difference is due to the type of the substrates, which causes the stress effects in the film plane. The implication here is that glass substrates provide very smooth surface features 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 film on kapton might induce further stress in the film, which might be inherent in flexible kapton substrates.

Figures 5 and 6 show the in-plane hysteresis loops measured at various angles of applied magnetic field for evaporated silicon-iron films on kapton and glass substrates under stationary conditions, respectively. The loops in these figures show that the films no longer possess a uniaxial anisotropy. Although there are quite slight variations between loops as the angle is varied, the film does not show any sign of an easy axis along the 90°. These experiments indicates that the stationary material production makes the samples posses an isotropic magnetic anisotropy since the coercivity values and remanence ratio in Table 1 were not varied as in the films in Figures 2 and 4.

In Table 1, maximum coercivity is observed along the easy direction, whereas the minimum coercivity occurs when magnetising along the hard direction. The reason for the shift in the centre of the hysteresis loops and at the point of $H = 0$ was due to the measurements system itself and has not an effect on the coercivity and magnetisation values because of symmetrical feature of hysteresis loops. 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 of 1.90 kA/m to 2.68 kA/m, whereas on kapton the range was 4.33 kA/m to 4.78 kA/m. Generally, smooth substrates result in softer films. An underlayer separating the magnetic film from the substrate has been shown to enhance soft magnetic properties. That could be the correlation between the coercivity and the type of substrate, as the glass providing much more smoother surfaces for film deposition compared to that of plastic kapton. The values obtained in this work are consistent with the values quoted in our previous study [4,5]. Other researchers [11,12] reported that the coercivities of the films were approximately 100 times larger than those measured in bulk electrical steel of 12 kA/m.

The presence of a uniaxial in-plane magnetic anisotropy at 90° in Figures 2 and 4 is clearly due to the rotation of the samples, which is a stressing effect, during the deposition process. The use of plastic kapton substrate combined with the rotation of the sample films leads to a better-defined uniaxial in-plane anisotropy. These evaporated films have demonstrated the feasibility of producing silicon-iron film samples with small thicknesses and sizes. Scaling up the technique to produce larger and thicker strips requires additional sources.

4 Conclusions

The silicon-iron films 100 nm thick can be deposited onto kapton and glass substrate using VCP system. They show very interesting magnetic properties in terms of magnetic anisotropy in the film plane. The factors affecting magnetic properties including coercivity and in-plane magnetic anisotropy were found to be stress induced in the films was due to the deposition method and substrate used. The reason for in-plane magnetic anisotropy was the rotation of the samples. The higher degree of anisotropy for the films deposited on kapton was further aided by its flexibility nature of plastic substrate. The VCP system has the capability for scaling up to deposit silicon-iron strips of comparable area to that of conventional electrical steel. Further work is needed to increase the number deposition sources to production of thicker substrate.

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