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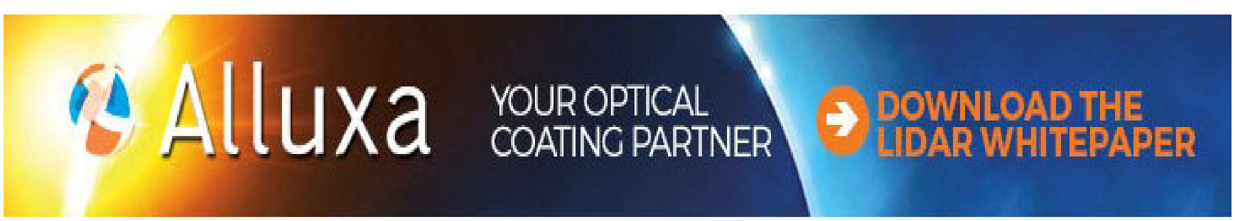

The growth of nanoscale structured iron films by glancing angle deposition

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Chiral nanostructured thin films can be produced through precise control of the angle of incidence of a vapor flux concurrent with substrate rotation. The technique has been employed to create unique porous iron thin film structures on Si(100) with a columnar microstructure. Scanning electron microscopy images illustrate columnar iron films produced with azimuthal rotation during sample growth with the incident flux at an angle of 75° with respect to the surface normal. The columns were found to be well isolated with a narrow distribution of diameters, resulting in aspect ratios of approximately 8 to 1. Hysteresis loops reveal the columnar growth induced a large magnetic shape anisotropy relative to that observed for an iron film grown with normal incidence. The evolution of the columnar microstructure was followed from simple oblique deposition (no substrate rotation), giving a fibrous slanted microstructure, to high-speed rotation where a broad size distribution of highly faceted columnar structures was observed. The measured microstructure is related to the observed magnetic properties. [S0021-8979(99)63308-4]

I. INTRODUCTION

The technique of glancing angle deposition was first reported in 1959.[1,2] For oblique incidence, deposited films show a columnar morphology. The columns are inclined from the substrate normal toward the vapor incidence direction. This morphology introduces a magnetic anisotropy.[3] The formation mechanism, microstructure, texture, and magnetic anisotropy of films evaporated at oblique incidence have been investigated.[4–11] The combination of glancing angle deposition with rotation of the substrate dramatically changes columnar morphologies and produces porous films consisting of isolated, evenly spaced columns.[12–15] The morphologies of the columns can be controlled precisely to form C-shape, S-shape, zigzag, and helical shapes by changing substrate rotation speed and substrate position relative to evaporation source. Thin films develop columnar morphologies generally under low adatom mobility conditions, thus column formation is due to a self-shadowing mechanism. Previous studies have focused on the effect of these complex morphologies on the optical and electrical properties and variation of morphologies by controlling the angular relation of the substrate surface with respect to the incoming vapor direction. Here, a magnetic material, iron, was chosen as the film material. This article reports the relation between microstructure and magnetic properties for a variety of Fe films produced by the technique of glancing angle deposition.

II. EXPERIMENTAL PROCEDURE

The Fe films were prepared by e-beam evaporation in a vacuum chamber evacuated to 5×10^{-8} Torr with a 550 ℓ/s compound turbomolecular pump and a 200 ℓ/s ion pump. The evaporation source material was 1–2 mm chips of 99.98% Fe in a 15 cc crucible. The e-beam source uses electron beam sweeping in order to obtain uniform evaporation characteristics. The source-to-substrate distance is 10 in. The metal flux is measured by a quartz crystal microbalance placed near the substrate and adjusted to 20 Å/s. A stepper motor that rotates the substrate during film deposition drives the substrate holder. The Fe films were deposited onto silicon wafers. After film deposition, the samples were cleaved to produce a clean edge for observation. The thin film thickness and column shapes and sizes were measured by a Philips XL 30 scanning electron microscope. Magnetic measurements were made with an alternating gradient magnetometer and vibrating sample magnetometer.

III. RESULTS

Thin Fe films with a variety of morphologies were made by changing deposition angle and rotation speed of the substrate. Figure 1 shows the five Fe films that were produced for this study (films a–e). Cross-sectional views are presented in the left column and hysteresis loops are shown in the right column. Film a, shown in Fig. 1(a), which was produced with no rotation and perpendicular incident flux, is shown for comparison. A closely packed microstructure is observed for this film since shadowing of atoms by growing columns does not occur. The magnetic measurements show that the easy axis is parallel to the thin film plane and the hard axis perpendicular to the thin film plane, which is the expected behavior.

Film b shown in Fig. 1(b) with the matchstick-like structure was made by simply tilting the substrate 75°. This gives a 75° angle between incident flux and substrate surface normal. The matchstick-like structures are closely packed and composed of slanted columns with an aspect ratio of 12:1. The angle between the substrate surface normal and the long axis of the columns is 59°. The hysteresis loops for this film reveal that the hard axis is perpendicular to the film plane. In addition, there is a large in-plane anisotropy with the in-plane easy axis parallel to the column growth direction and

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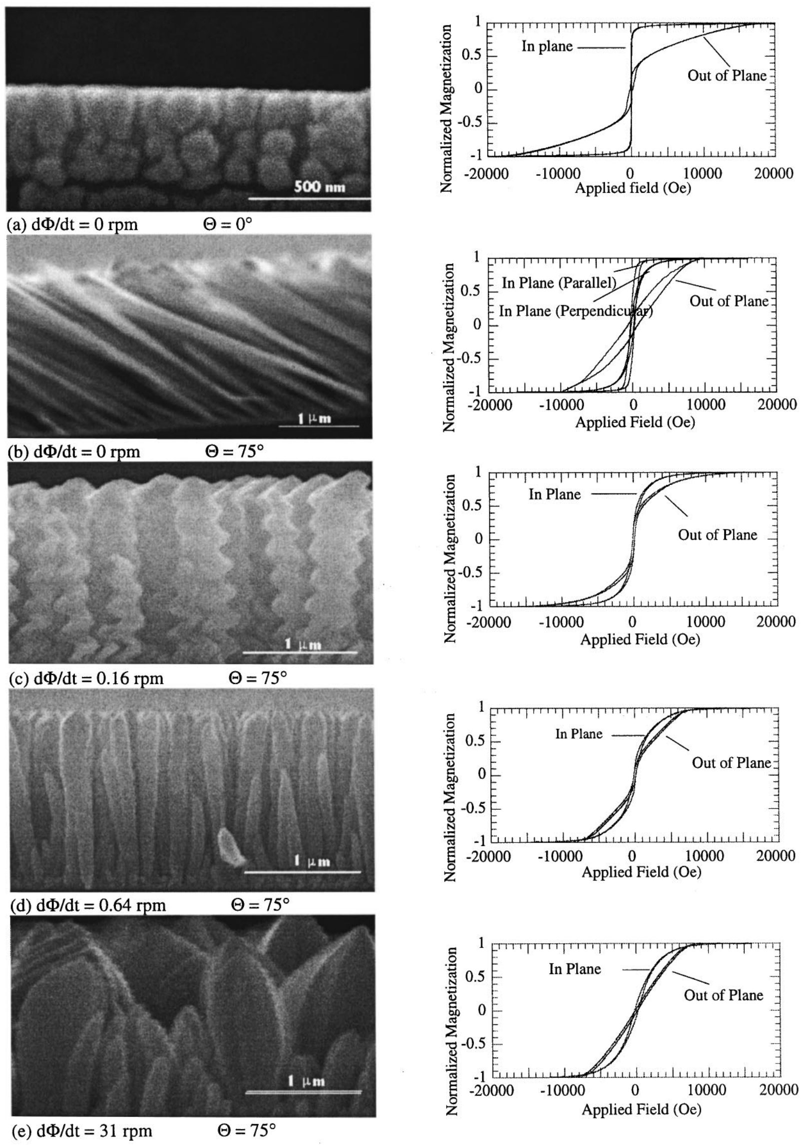

FIG. 1. The left column shows scanning electron microscope micrographs of Fe films with varying deposition conditions. Normal incidence flux (a), oblique flux at a polar angle of 75° and azimuthal rotation speed of 0 rpm in (b), 0.16 in (c), 0.64 in (d), and 30 rpm in (e), respectively. The measured hysteresis loops in the right column correspond to each specimen.

the in-plane hard axis perpendicular to the column growth direction.

Figure 1(c) shows the microstructure and magnetic behavior for a film deposited at a polar angle of 75° with an azimuthal rotation speed of 0.16 rpm. For this set of deposition conditions, helical columns are produced. The aspect ratio of the columns is approximately 5:1, and the packing fraction is 0.35. The packing fraction was determined by measuring the area occupied by the columns on a top view micrograph. By increasing the rotation speed of substrate to 0.64 rpm, slimmer columns are produced as shown in Fig. 1(d). The aspect ratio of the columns is approximately 8:1, and the packing fraction is 0.5. The ratio between out-of-plane and in-plane coercivities for films c and d are 0.88 and 1.02, respectively. Comparing results for films c with d shows that increasing the substrate rotation speed increases the aspect ratio and perpendicular anisotropy. Film d has a large variation in column sizes with shorter columns comprising about half of the columns. The easy axis is not well defined for this film. It is expected that this film would have a high perpendicular anisotropy because of the high aspect ratio and the low packing fraction. Eliminating the initial magnetic layer could produce better easy axis behavior since the particles are coupled through the initial magnetic layer. Experiments are underway to study the initial nucleation phase and control the thickness of the initial layer or to replace it by a nonmagnetic material.

To investigate the possibility of producing films with an even greater perpendicular anisotropy, we increased the rotation speed of substrate to 30 rpm. The result is shown in Fig. 1(e). Film e consists of leaf-like columns. This sudden transition to a different growth mode was not expected. This film has a large variation in the size of the columns and a much lower perpendicular anisotropy with a ratio between the out-of-plane and in-plane coercivities of 0.6. This shows that the distribution of sizes and shapes of the structures gives a magnetic behavior similar to a film with the hard axis perpendicular to the film plane.

IV. CONCLUSIONS

Varying deposition angle and substrate rotation speed can dramatically change morphologies of thin films. Increasing the substrate rotation speed increases the aspect ratio of columns and anisotropy for films. However, if the rotation speed of the substrate is too high, a very different morphology and anisotropy is observed. These preliminary results indicate that both the morphology and magnetic properties can be controlled within a limited range by this new film production technique.

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