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Perpendicular anisotropy in granular Co–Zr–O films

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Soft granular Co–M–O films have promise for application in high-frequency thin-film inductors. But granular Co-based films are often found with perpendicular anisotropy, which can result in stripe domains and poor hysteresis behavior. Film microstructure was analyzed for Co-rich Co–Zr–O granular films with and without stripe-domain behavior. It is suggested that perpendicular anisotropy originates from columnar structure with Co columns perpendicular to film plane. The appearance of columnar structure is determined by sputter pressure and oxygen content. © 2005 American Institute of Physics. [DOI: 10.1063/1.1851711]

I. INTRODUCTION

As a core material in thin-film inductors for high-frequency integrated dc–dc converters and similar applications, soft Co-based granular films of the form Co–M–O (M =nonmagnetic metal) with high saturation magnetization M_s , high electrical resistivity ρ , and high in-plane anisotropy field H_k should have great advantages over other metal-alloy soft magnetic materials. High resistivity and high anisotropy field are beneficial to obtain low loss and high power density in thin-film inductor applications,¹ and are usually understood to arise from the particular granular structure of films, in which nanoscale metallic magnetic grains are embedded in nonmagnetic insulating oxide.²

Co–M–O granular films are generally prepared using sputtering.^{2,3} Electrical resistivity and anisotropy field can be adjusted by controlling the ratio between cobalt and oxide. Decreasing the Co/oxide ratio usually increases the electrical resistivity. On the other hand, to keep high enough saturation magnetization M_s , a high fraction of cobalt is desired. Ohnuma *et al.*^{2,3} have also reported that favorable soft magnetic properties can only be obtained in a limited composition range. Outside of this range, a strong perpendicular component of anisotropy often occurs, which can result in stripe domains⁴ which increase coercivity and lower permeability. Although the coercivity will decrease to a very small value when the material with stripe-domain behavior is driven in unipolar field which is often the case in inductor application, the large residual magnetization B_r results in a reduced flux swing and thus in inferior performance.^{5,6} Stripe domains also result in reduced permeability; low permeability is beneficial in some inductor applications, but does not make up for the reduced flux swing.⁶ Additionally, we found that sputter pressure during film deposition also affects occurrence of stripe-domain behavior. In this paper, we analyze the microstructure of the Co–Zr–O films with good soft behavior and the Co–Zr–O films with stripe-domain behavior sputtered at different deposition conditions, and propose the origin of the perpendicular anisotropy effects which cause stripe-domain behavior.

II. EXPERIMENT

Co–Zr–O films, 1 μm thick, were deposited by dc magnetron sputtering on water-cooled glass slides and Si substrates in an Ar+O₂ atmosphere, using a CoZr alloy target (Co:Zr=85:15 atomic ratio). The base pressure of the chamber was better than 3×10^{-7} torr. Sputter power was set at 100 W; sputter pressure and oxygen introduction were varied. A Halbach magnet array providing a 40-mT in-plane field was used to induce in-plane anisotropy in the films. Magnetic properties and electrical resistivity were measured with a vibrating sample magnetometer (VSM) and the conventional four-point method, respectively. Film cross-sectional images were taken with a scanning electron microscope (SEM), and composition was characterized by energy dispersive spectroscopy (EDS). Surface topographies were observed with an atomic force microscope (AFM). X-ray diffraction analysis was also carried out.

III. RESULTS AND DISCUSSION

It was found that soft magnetic properties of as-deposited Co–Zr–O films are affected by sputter pressure and oxygen gas introduction. No significant difference was observed between films on glass or Si substrates. Good soft properties never occurred in films fabricated at sputter pressures higher than about 1.5 mtorr. At lower sputter pressures, Co–Zr–O films with oxygen concentration within a certain range showed good soft properties, which is consistent with the fact reported in reference.³ The lower the pressure, the wider the oxygen range over which favorable properties were found. Below we select four Co–Zr–O film samples sputtered at different pressures or oxygen partial pressures to examine the relationship between microstructure and magnetic properties, and then suggest the origin of perpendicular magnetic anisotropy.

Table I lists sputter pressure, composition, electrical, and magnetic properties of these samples. The hysteresis loop of films A, as shown in Fig. 1(a), is characteristic of stripe-domain behavior.⁷ Films C and D have similar loops as film A. The hysteresis loop of film B, shown in Fig. 1(b), is typical for soft magnetic films with large in-plane anisotropy. Comparing samples A and B, which have similar composition, we see that the film deposited at 1.2 mtorr sputter pres-

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TABLE I. Sputter pressure P_s , composition, electrical resistivity ρ , saturation magnetization M_s , and hard axis coercivity H_{ch} of four Co–Zr–O granular films. Type SD: hysteresis loop of materials with stripe-domain behavior. Type SF: hysteresis loop of soft magnetic materials.

| Sample | A | B | C | D |
|---------------------------|--------------------------------------------------------|--------------------------------------------------------|--------------------------------------------------------|--------------------------------------------------------|
| P_s (mtorr) | 5 | 1.2 | 1.2 | 1.2 |
| Composition | Co _{65.4} Zr _{6.7} O _{27.9} | Co _{65.5} Zr _{6.8} O _{27.7} | Co _{76.4} Zr _{7.5} O _{16.1} | Co _{65.5} Zr _{5.4} O _{29.1} |
| ρ ($\mu\Omega$ -cm) | 340 | 332 | 88 | 460 |
| M_s (T) | 1.09 | 1.19 | 1.41 | 1.13 |
| H_{ch} (Oe) | 70 | 2.5 | 55 | 45 |
| Loop type | type SD | type SF | type SD | type SD |

sure exhibits good soft behavior, while the film prepared at 5 mtorr has stripe-domain behavior. Samples B, C, and D were all sputtered at a pressure of 1.2 mtorr, but only sample B has good soft magnetic properties. This is because sample B was deposited with oxygen concentration in the middle of the range in which good soft magnetic properties result, whereas samples C and D were deposited with oxygen concentrations below and above this range, respectively.

The microstructure of the Co–Zr–O films was analyzed to deduce the mechanism of perpendicular anisotropy responsible for the stripe-domain behavior in films A, C, and D. First, these four samples have similar x-ray diffraction (XRD) patterns, as shown in Fig. 2, in which only cobalt peaks are visible, and cobalt hcp (002) is the strongest. Therefore, these films show hcp (002) preferred orientation to some degree, because the strongest peak is (101) in the

XRD pattern of hcp cobalt powder. Cobalt grain sizes are calculated to be 3–6 nm based on Debye–Scherrer method.

Cross sections were then analyzed using SEM, as shown in Fig. 3. From the cross-sectional images, noticeable difference in structure between films with and without stripe-domain behavior can be observed. In sample A with stripe-domain behavior, the film has obvious columnar structure, with columns perpendicular to the film plane. Samples C and D also show evidence of some columnar structure. In contrast, sample B with good soft properties does not show such structure. Based on the SEM cross section in Fig. 3(b) and the surface topography in the AFM image in Fig. 4, we conclude that the structure of sample B is likely to be three-dimensionally (3D) isotropic. All the samples have similar surface topography.

From above it has been noted that Co–Zr–O granular films of interest have similar grain sizes, crystalline phase, and orientation. What finally determines perpendicular anisotropy appears to be the columnar structure. The columns may be composed of nanosized cobalt granules separated by non-magnetic Zr–O and voids. Avoiding columnar structure helps to produce good soft magnetic properties and can be achieved by adjusting film deposition parameters. In particular, decreasing sputter pressure and keeping oxygen content in a narrow range have been found to decrease perpendicular anisotropy, resulting in favorable magnetic properties. The former approach is readily confirmed by a well-known fact that high sputter pressure promotes column growth by decreasing the Ar-ion bombardment.⁸ The influence of oxygen

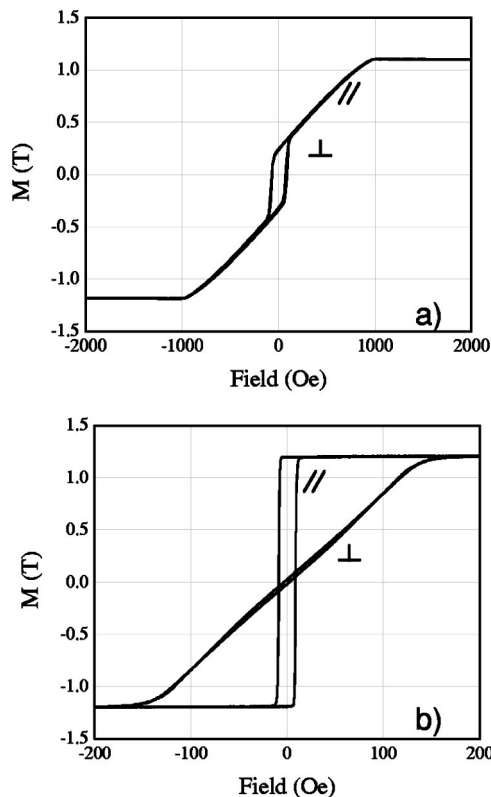


FIG. 1. Magnetization curves of Co–Zr–O films: (a) sample A, with stripe-domain behavior; and (b) sample B, with soft magnetic properties. Loops were measured in film plane. // and \perp represent directions parallel and perpendicular to the dc magnetic field applied during film deposition, respectively.

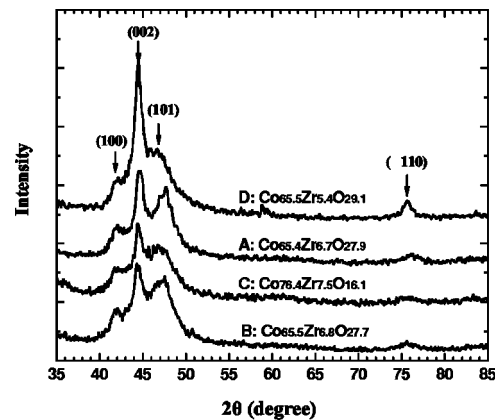


FIG. 2. X-ray diffraction patterns of Co–Zr–O granular films A, B, C, and D. All indicated peaks belong to hcp Co.

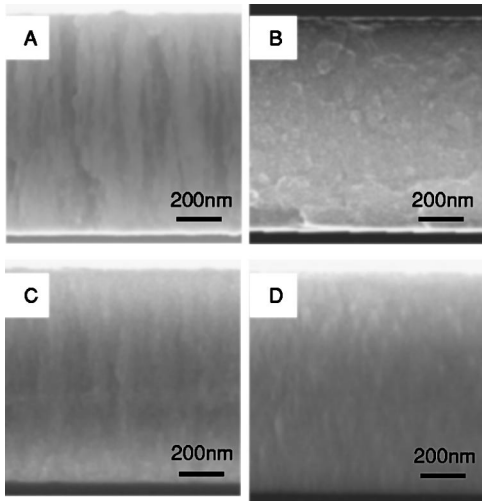


FIG. 3. SEM cross-sectional images of Co-Zr-O granular films A, B, C, and D.

on the structure is not fully understood and needs further study.

IV. SUMMARY

The influence of sputter pressure and oxygen content on magnetic properties of Co-Zr-O granular films has been observed:

- (1) Co-Zr-O films sputtered at high pressure have stripe-domain behavior, resulting from strong perpendicular magnetic anisotropy.
- (2) If Co-Zr-O films are deposited at relatively low sputter pressure, perpendicular anisotropy will also appear if the oxygen ratio in the films is outside of a particular region.

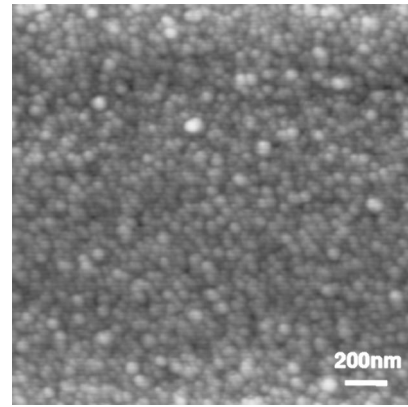


FIG. 4. AFM image shows the surface topography of Co-Zr-O granular film B.

The occurrence of strong perpendicular magnetic anisotropy is suggested to originate from columnar structure with columns perpendicular to film plane.

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