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Phase formation and magnetic properties of Co-rare earth magnetic films

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Co-Sm and Co-Pr films were deposited by dc magnetron sputtering. Transmission electron microscopy and magnetic measurement were used to study the microstructure and magnetic property relationship. The nanostructure of the as-deposited Co 19 at. % Sm films consists of two phases: the amorphous phase and the crystallite phase. Upon annealing at 600 °C, the Co₅Sm phase with the Cu₅Ca structure, having grain size of about 20 nm, is obtained along with high coercivity (45 kOe). The as-deposited Co 22 at. % Sm films also have nanostructure similar to the Co 19 at. % films except the volume fraction of the crystallite is reduced. This is related to the concentration of Sm which promotes the formation of the amorphous phase. A new metastable phase Co₃Sm is formed upon annealing of the Co 22 at. % Sm film at 500 °C. This phase has the DO₁₉ structure in which the Sm atoms take ordered positions of a triangular pattern in the close-packed planes. A relatively high coercivity value of 29 kOe was obtained from this phase. The as-deposited Co-Pr films show mainly an amorphous phase. Upon annealing at 500 °C for 20 min, Co₂Pr with the Mg₂Cu-type structure was identified in the Co 35 at. % Pr film. Two phases were identified in the Co 16 at. % Pr films. Coercivities up to 3.1 kOe were achieved in these films. © 1998 American Institute of Physics. [S0021-8979(98)30611-8]

I. INTRODUCTION

Hard and semihard Co rare earth films are of increasing interest for magneto-electronic and magnetic-recording applications. The Co-Sm and Co-Pr systems also have high T_c suitable for high temperature magnets. A number of articles on the Co-Sm and Co-Pr systems have been presented.¹⁻⁵ The magnetic properties of materials can be divided into intrinsic properties and extrinsic properties. The intrinsic properties such as magnetization are related to the crystal structure of the magnetic phase while the extrinsic properties such as coercivity are affected by the microstructure. Development of new magnetic materials involves the search for new magnetic phases and the design of microstructure. In this article we report our detailed study on phase formation, microstructure, and magnetic properties relationship in films based on the Co-Sm system and Co-Pr system heat treated at different temperatures.

II. EXPERIMENTAL PROCEDURE

For the Co-Sm system, the films were deposited by dc magnetron sputtering. Two compositions near the Co₅Sm and Co₇Sm₂ were selected. All films have a Cr underlayer of about 90 nm except the one with the composition of Co 19 at. % Sm annealed at 600 °C which is deposited on a quartz substrate. All the films have a Cr cover layer of about 10 nm. For the Co-Pr system multilayers of Co-Pr/Co films were deposited using a multiple-gun dc and rf sputtering chamber. The multilayer microstructure is designed to promote magnetic hard phase and soft phase coupling in order to gain maximum energy product. A 50 nm underlayer and a 10 nm

cover layer of Cr were used for film seeding and protection. Plan-view transmission electron microscopy (TEM) samples were prepared by dimpling and ion milling process. TEM study was conducted using a JEOL 2010 transmission electron microscope.

III. RESULTS AND DISCUSSION

The deposition parameters, film thickness, coercivity, and phase identification results are summarized in Table I.

FIG. 1. Magnetization loop of the Co₅Sm film annealed at 600 °C.

TABLE I. Composition, deposition condition, nanostructure, and magnetic properties relation in Co–Sm and Co–Pr films. C indicates crystallite phase and A amorphous phase. Vc is the volume fraction of the crystallite phase against the amorphous phase.

Film com. (at. %)	Ar pressure (mTorr)	Film thickness (nm)	Phases	Vc (%)	Grain size (nm)	Coercivity (kOe)
Co 19 at. % Sm	5	24	C+A	91	5	0.61
Co 19 at. % Sm	12	24	C+A	65	5	2.58
Co 19 at. % Sm	30	24	C+A	54	5	0.92
Co 19 at. % Sm Annealed at 600 °C	20	360	Co ₅ Sm (Cu ₅ Ca)	100	20	45
Co 22 at. % Sm	5	30	C+A	81	5	1.2
Co 22 at. % Sm	17	30	C+A	57	5	4.1
Co 22 at. % Sm	30	30	C+A	48	5	3.4
Co 22 at. % Sm Annealed at 500 °C		394	DO ₁₉	100	15	29

FIG. 2. (a) Comparison of SAD pattern and calculated intensity distribution for the film described in Fig. 1. (b) TEM image of the microstructure.

FIG. 3. (a) Comparison of SAD pattern and calculated intensity distribution for the Co 22 at. % Sm film. (b) TEM image of the microstructure.

The nanostructure of the as-deposited Co 19 at. % Sm films has been studied in earlier work¹ and is included here for comparison. Upon annealing at 600 °C, the equilibrium phase Co₅Sm with the Cu₅Ca structure is obtained along with high coercivity. Figure 1 shows the magnetization loop. Coercivities up to 45 kOe were achieved. Figure 2 shows the selected area diffraction (SAD) pattern and TEM micrograph. The diffraction shows perfect match with the calculated intensity. The grain size is about 20 nm.

The Co 22 at. % Sm films also have a nanostructure similar to the Co 19 at. % films except the volume fraction of the crystalline against amorphous phase is reduced. This is related to the concentration of Sm which promotes the formation of the amorphous phase. Figure 3 shows the SAD pattern and the TEM image of the Co 22 at. % Sm film annealed at 500 °C. A new phase Co₃Sm is formed as indicated by the matching of the TEM pattern and calculation. This phase has the DO₁₉ structure in which the Sm atoms taking the ordering positions of a triangular pattern in the close-packed planes. The close-packed planes are stacked by the sequence of ABAB in the *c* direction. A relatively high coercivity value of 29 kOe was obtained from this phase.

The appearance of the DO₁₉ structure is not a surprise but rather easy to comprehend. In the as-deposited Co–Sm films, the structure of the crystallite is the close-packed structure with Sm atoms randomly distributed in the close-packed planes and short range packing order in the *c* direction. Upon annealing, two things happened: one is that the Sm atoms within each close-packed plane form a triangular ordering pattern, and the second is that the packing in the *c* direction takes the long range ABAB packing. The lattice parameters deduced from this phase are $a = 0.256$ nm $c = 0.419$ nm compared to the lattice parameters of Co $a = 0.2505$ nm, 0.4065 nm. It is interesting to note that the lattice parameters between the DO₁₉ phase and the Co phase are close to each other, suggesting a low energy state at the interphase. It is suggested that two-phase structure composed of Co and DO₁₉ phase could be stable up to 500 °C.

The as-deposited Co–Pr films show largely amorphous phase. Upon annealing at 500 °C for 20 min, Co₂Pr of the Mg₂Cu-type structure was identified in the Co 35 at. % Pr film. Two phases were identified in the Co 16 at. % Pr films. Coercivities up to 3.1 kOe were achieved in these films.

IV. CONCLUSIONS

Three metastable phases were found in the sputtered Co–Sm films: the amorphous phase, the close-packed hexagonal phase with different stacking mode in the as-deposited film, and the DO₁₉ structure phase in the film annealed at 500 °C.

Corresponding to the different phases and microstructure, the coercivities change from about 1–42 kOe. Maximum coercivity was achieved from the Co₅Sm phase with the Cu₅Ca structure. The new metastable phase of DO₁₉ structure found in the Co 22 at. % Sm film also showed relatively high coercivity of 29 kOe.

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¹Y. Liu, B. W. Robertson, Z. S. Shan, S. Malhotra, M. J. Yu, S. K. Renukunta, S. H. Liou, and D. J. Sellmyer, *IEEE Trans. Magn.* **6**, 4035 (1994).

²Y. Liu, D. J. Sellmyer, B. W. Robertson, Z. S. Shan, and S. H. Liou, *IEEE Trans. Magn.* **31**, 2740 (1995).

³S. S. Malhotra, Y. Liu, Z. S. Shan, S. H. Liou, D. C. Stanford, and D. J. Sellmyer, *J. Magn. Mater.* **161**, 316 (1996).

⁴E. M. T. Velu and D. N. Lambeth, *IEEE Trans. Magn.* **28**, 3249 (1992).

⁵K. Chen, H. Hegde, S. U. Jen, and F. J. Cadieu, *J. Appl. Phys.* **73**, 5923 (1993).