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Al-Omari, I.A.; Cunningham, N. J.; and Sellmyer, David J., "Magnetic and structural properties of Sm₂Fe₁₀Co₇/M_xC_y thin films" (1997). *David Sellmyer Publications*. 74. https://digitalcommons.unl.edu/physicssellmyer/74

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MAGNETIC AND STRUCTURAL PROPERTIES OF Sm₂Fe₁₀Co₇/M_xC_y THIN FILMS

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Abstract-The structural and magnetic properties of $Sm_2Fe_{10}Co_7/M_xC_y$ (M=Al and Si; $x \ge 0$ and $y \ge 0$) multilayer films with Ta underlayers and overlayers before and after annealing at 700 °C for 5-12 min have been fabricated and studied. Structural studies show evidence of layer diffusion upon annealing. X-ray diffraction shows that the samples after annealing consist of a soft phase, α -Fe, and a hard phase with the 2-17-type structure. The samples studied have in-plane anisotropy with single hysteresis loops indicating that the two phases are strongly exchange coupled. After annealing, the coercivity of samples with Al_xC_y is found to increase with increasing Al_xC_y composition to a maximum of 3.3 kOe. The energy products for these samples are found to increase from 1.4 MGOe for SmFeCo to 8 MGOe for SmFeCo with Al_xC_y .

I. Introduction

Enhancement of the energy product by remanence enhancement has been the subject of many theoretical [1]-[5] and experimental [6]-[7] studies. These enhancements were found in materials with strongly exchange coupled hard and soft phases; several experimental studies have been published on alloyed systems such as Nd₂Fe₁₄B with Fe₃B by Coehoorn, De Mooij, and DeWaard [6], in which they found a maximum energy product of 12 MGOe. Withanawasam, Hadjipanayis, and Krause [7] obtained a (BH)_{max} of 14 MGOe for a finegrained mixture of Nd₂Fe₁₄B and α -Fe prepared by the meltspinning method. Recently, we have studied this effect for the first time in thin films of SmCo/FeCo [8]. In that study we found that there is enhancement in the energy product by changing the layer thickness; a maximum energy product of 6 MGOe was found in these films.

Katter *et al.* [9] found that $\text{Sm}_2\text{Fe}_{17-x}\text{Co}_x$ reaches its maximum magnetization at $x \approx 7$. In this study we use M and/or M_xC_y to increase the coercivity of $\text{Sm}_2\text{Fe}_{10}\text{Co}_7$ which has small coercivity, ≈ 0.7 kOe, and to substitute M for Fe to produce a composite with soft, α -Fe, and hard, 2-17-type structure, phases.

In this paper, we present the effect of annealing and various

Manuscript received January 20, 1997.

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layer thicknesses and number of bilayers on the magnetic and structural properties of $\text{Sm}_2\text{Fe}_{10}\text{Co}_7/M_x\text{C}_y$ films with Ta underlayers and overlayers, where $\text{Sm}_2(\text{Fe},\text{Co},\text{MC})_{17}$ is the hard phase and α -Fe is the soft phase. The conditions for preparing the Ta underlayer were chosen to give the hard phase in-plane anisotropy and the highest achievable coercivity.

II. Experimental Procedure

Multilayer films of $Sm_2Fe_{10}Co_7$ and M_xC_y (M=Al and Si; and $y \ge 0$) were sputtered, in a multiple-gun sputtering system, onto Si substrates with Ta underlayers of 1000 Å by dc gun and Ta overlayers of 200 Å. SmFeCo, prepared by dc sputtering, layer thicknesses vary from film to film, ranging from 6 to 28.2 Å, while $M_x C_y$, prepared by rf sputtering, layer thicknesses ranged from to 0.0 to 36.8 Å. The SmFeCo target was made by pressing the Fe powder and Sm₂Co₇ particles together and then sintering in vacuum ($\approx 10^{-6}$ Torr) at a temperature of 1050 °C for 0.5 h. The Ta, Al and Si targets were commercially obtained and had 99.99% or more purity, four C chips of about 3 mm x 3 mm were put on the Al or Si targets to get the $\ M_x C_\nu$ layer. The base pressure of the sputtering system was 1.5-4x10⁻⁷ Torr and Ar pressure during sputtering was optimized, 15-20 mTorr, to give the highest coercivity. The sputtering rates and powers were 6-8 Å /sec and 75 W for Ta, 8-10 Å/sec and 20-60 W for SmFeCo, and 2-4 Å /sec and 50 W for $M_x C_y$. In each vacuum run 12 samples were made.

Half of the samples had total multilayer thicknesses of 200-300 Å (9-29 bilayers), while the other half had thicknesses of 1980-3000 Å (60-222 bilayers). Samples of each of the films were annealed at 700 °C for 5-12 minutes in a vacuum of $\approx 4 \times 10^{-7}$ Torr. X-ray diffraction (Cu-K α) was used to study the structure. The magnetic properties of the films were studied using an alternating gradient force magnetometer with a maximum field of 14 kOe.

III. Results and Discussion

Large-angle x-ray diffraction shows no peaks before annealing and after annealing it shows α -Fe and 2-17-type structure, and a very small amount of unknown phase. Small angle x-ray diffraction for unannealed samples shows multiple

Research supported by DOE Grants DE-FG02-86ER45262, University of Chicago 95-47DH-007, and Jordan University of Science and Technology.

peaks, which are attributed to the multilayer structure; after annealing the films, low-angle measurements show no peaks which suggests that the multilayers have indeed diffused.

Fig. 1 shows typical magnetization loops for some of the annealed samples studied. Magnetization loops parallel and perpendicular to the film plane, Fig.1 (a), show that these films have simple single loops with in-plane anisotropy; this indicates that there is a strong exchange coupling between the soft phase, α -Fe, and the hard phase, of 2-17-type. Figures 1(b) and 1[©] show the initial curve, hysteresis loop, and minor loops. These Figures display features of particle rotation as suggested by [10]-[13] for other materials , which suggests that particle rotation is the dominant coercivity mechanism in these films. Films with different SmFeCo and M-C thicknesses showed results similar to those of SmFeCo of 25 Å thickness.

Films with thicknesses of 200-300 Å were found to have small coercivity, <<1 kOe, before and after annealing. Before annealing, samples with thickness of about 2000 Å were found to have small coercivity; after annealing samples with Al_xC_y were found to have a coercivity higher than that of SmFeCo, (≈ 0.7 kOe) as seen in Fig. 2, while samples with Si_xC_y were found to have smaller coercivity than that of SmFeCo; therefore this paper is concerned chiefly with the thick multilayer films. Any generalized statement concerning the films studied refers only to the thick multilayer films. Substituting A1 or introducing A1-C in the 2-17 compounds



Fig. 1. Typical magnetization curves for Si//Ta(1000Å):[SmFeCo(25Å)/Al(x Å)]₈₀:Ta(200Å) (a) parallel and perpendicular loops (b) initial loop and hysteresis loop (c)minor hysteresis loops.



Fig. 2. Dependance of the coercivity for Si//Ta(1000Å):[SmFeCo(25Å)/M(X Å)]_{80}:Ta(200Å) on X.

was found to increase the coercivity, so the increase in the coercivity for small Al_xC_y thicknesses is attributed to this reason and the decrease in the coercivity for large Al_xC_y thicknesses is due to the increase in the percentage of the soft phase in the films. The coercivity for films with Al_xC_y thickness larger than 10 Å was found to be less than 0.3 kOe.

The magnetization in these films was found to decrease by increasing the M_xC_y thickness, which is due to the increase in the percentage of the non magnetic materials (Al and C) and this is in agreement with other observations for bulk compounds studied by Katter *et al.* [9]. Fig. 3 shows the energy product (BH)_{max} for films of SmFeCo and Al C; the enhancement in the energy product is due the enhancement in the coercivity because of the exchange coupling between the hard and soft phases. The decrease in (BH)_{max} for large thicknesses is due to the large increase in the percentage of the soft phase. Energy product for films with Al_xC_y thickness larger than 10 Å was found to be less than 1 MGOe.



Fig. 3. Dependance of the energy product for $Si//Ta(1000~{\rm \AA})$:[SmFeCo(25 Å)/M(X Å)]_{80}:Ta(200Å) on X.

The scatter in the data in figures 2 and 3 is due to the error in the layer thickness and the error in measuring the area of the sample which gives an error in the volume of the sample.

The magnetic interactions in these films were studied by measuring the isothermal remanence $M_r(H)$ and the dc demagnetization remanence $M_d(H)$; where $M_r(H)$ is measured by a progressive magnetization of an initially ac-demagnetized sample and $M_d(H)$ is measured by a progressive demagnetization from a previously saturated state [14]-[18]. The technique of Hankel plots [19] (M_d versus M_r) to study the magnetic interactions was developed by Kelly *et al.* [14], where by plotting the interaction-based deviation parameter $\Delta I(H)$ versus the applied field, the type of interaction can be investigated. The relation between $\Delta I(H)$ and I_r and I_d is given by the following equation:

$$\Delta I(H) = I_d(H) - [1 - 2I_r(H)]$$

where I_r and I_d are the remanences M_r and M_d normalized to the saturation values. Fig. 4 shows $\Delta I(H)$ versus the applied field (H) for various films. From this Figure we see that ΔI is predominantly negative for small Al and Al_xC_y layer thicknesses, which indicate a dipole interaction; while for large layer thicknesses ΔI is predominantly positive, which indicate a positive exchange coupling, or a ferromagnetic interaction. The change in the position of the peak is due to the change in the coercivity of the films.



Fig. 4. Dependance of $\Delta I(H)$ for Si//Ta(1000Å):[SmFeCo(25Å)/Al(X Å)]₈₀: Ta(200Å) on X.

IV. Conclusions

Samples of the form Si//Ta(1000 Å):[SmFeCo(X \dot{A} /M_xC_y(Y \dot{A})]_n:Ta(200Å) with different layer thickness (X and Y) and different bilayer number (n), M = A1 and Si; $x \ge 0$ and $y \ge 0$, were prepared and studied before and after annealing. All the samples studied were found to have in-plane anisotropy and simple single loops which indicate a strong exchange coupling between the hard and soft phases. After annealing, the coercivity of samples with Al_xC_y is found to increase with increasing Al_xC_y composition from 0.7 kOe for Y=0 to a maximum of 3.3 kOe for Al_xC_y . The energy products for these samples are found to increase from 1.4 MGOe for SmFeCo to 8 MGOe for SmFeCo with Al_xC_y . In these films, particle rotation is the dominant coercivity mechanism. The magnetic interactions are found to change from dipole for small Al_xC_y layer thicknesses to exchange coupling for large Al_xC_y layer thicknesses. Such samples with relatively low rare-earth concentration and in-plane anisotropy might be used in devices that require relatively small (BH)_{max}.

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