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High Coercivity SmFeAlC Thin Films Fabricated by Multilayer Sputtering*

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Abstract—Multilayer sputtering of SmFe/C(Al) plus appropriate thermal processing has been used to fabricate SmFeC thin films with Al substitution for Fe. It was found that the SmFeAlC thin films exhibited exceptionally high coercivities. A coercivity of 28 kOe was realized for the thin film with structure of Ta(1000Å)/[SmFe(40Å)/ C(Al)(5Å)]×50/Ta(200Å) with subsequent annealing at 650°C.

I. INTRODUCTION

SmFeN and SmFeC intermetallic compounds based on the rhombohedral Th₂ Zn_{12} -type structure have been attracting much attention as potential candidates for permanent-magnet materials owing to their excellent magnetic properties[1,2]. For example, a room temperature coercivity of 15 kOe, saturation magnetization of 92.4 emu/g, and Curie temperature of 624K were reported for $Sm_2Fe_{14}Ga_3 C_{1.5}[3]$. Much effort has been devoted to the improvement of the intrinsic magnetic properties, such as the various elemental substitutions for Fe in the SmFeC materials. Recently, it has been shown that the substitution of Ga, Al or Si for Fe can help only the formation of rhombohedral Th_2Zn_{17} -type rare-earth iron compounds, but also is very effective in raising the value of the anisotropy field H_A [3-5]. For instance, the SmFeC compound with A1 substitution for Fe has $H_A=110$ kOe, which compares with 53 kOe for the SmFeC compound[5]. Also, in the optimum case, a coercivity of 9.4 kOe was obtained in a bulk specimen of $Sm_2Fe_{15}Al_2C_{15}$ compound[6]. It is of interest, therefore, to see whether similar coercivities can be realized in SmFeAIC thin films, which is required for modern magnetoelectronic device applications.

II. EXPERIMENTS

SmFeAlC thin films were prepared in a DC magnetron sputtering system using a composite Sm_2Fe_{17} alloy target and a composite graphite(Al) target. The overall composition of the target is estimated to be $Sm_2Fe_5Al_iC_{1.5}$. In order to compensate for the loss of Sm during sputtering, several extra pieces of Sm were mounted on the SmFe target. Thin films were prepared in an argon pressure of 5 mTorr with base pressure better than 3×10^{-7} Torr. The deposition rates were about 5Å/sec for SmFe and 2Å/sec for C(Al), respectively. First, multilayered SmFe/C(Al) thin films with the structure of Si/Ta(X)/[Sm(40Å)/ (C(Al)(YÅ)] × N/Ta(200Å) were prepared

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at ambient temperature, where X is the thickness of Ta underlayer, Y is the thickness of C(Al) layer, and N is the number of SmFe/C(Al) bilayers, respectively. The films were then thermal annealed at various temperatures in a vacuum of 5×10^{-7} Torr for 5 min.

The composition of the thin films was checked by energy dispersive x-ray analysis (EDX) attached to a scanning electron microscope (SEM) and x-ray fluorescence (XRF). The result for a film with high coercivity (28 kOe) indicated a stoichiometry of $Sm_2Fe_{5,2}Al_{0.8}C_x$. The structure was analyzed by x-ray diffraction and HRTEM. The magnetic properties were measured with an alternating gradient force magnetometer (AGFM) and SQUID magnetometer.

III. RESULTS

Figure 1 shows the coercivity H_c of SmFeAlC thin films as a function of the thickness of the C(Al) layer. It is seen that the





coercivity H_c of the SmFeAlC thin films strongly depends on the thickness of the C(Al) layer and annealing condition. Depending on the thicknesses of the C(Al) layer and annealing temperature, three types of behaviors are seen in Fig.1: (1) H_c shows a peak with the variation of the C(Al) layers. This behavior was also observed earlier in our reported SmFeSiC thin films[7]. (2)The peaks of H_c move toward larger thicknesses of the C(Al) layer with an increase of the annealing temperature. As seen in Fig.1, the maximum coercivity of the thin films appears at 15Å C(Al) layer when thin films were annealed at 750°C, whereas this maximum moves to 20Å C(Al) layer when the annealing temperature was increased to 775°C. (3)The maximum coercivities decrease with an increase of the annealing temperature. When the thin film was annealed at 650° C, a coercivity of 28 kOe was obtained for the thin film with structure of Ta(1000Å)/[SmFe(40Å)/C(Al)(5Å)]× 50/Ta (200Å). However, when the thin film was annealed at 775°C, a coercivity of the thin film with the structure of Ta(1000Å)/[SmFe(40Å)/C(Al)(20Å)]×50/Ta(200Å) is 13.5 kOe. These results indicate that magnetic properties of SmFeAlC thin films are highly sensitive to the ratio of SmFe to C(Al) and annealing temperature.

It is well known that a favorable microstructure is essential to obtain high coercivity in magnetic materials, because the grain size and shape, the defect type and distribution, and the grain boundary structure affect the domain structure and its wall movement, and thus control the coercivity of a magnetic material. In addition, it was reported that in magnetic thin films grain size and texture of magnetic layer were closely related to its underlayer[8]. For example, magnetic properties of CoCrPt[9] and SmCo[10,11] thin films were improved by the presence of a Cr underlayer[9]. By using Ta underlayer, similar behaviors have been observed in SmFeSiC thin films[7]. Based on the same idea, we deposited the SmFeAIC thin films on Si substrates with Ta underlayer. Our results show that magnetic properties are improved by the presence of the Ta underlayer. Figure 2 shows a dependence of H_c and M_c of



Fig.2. Dependence of H_{e} and M_{s} of SmFeAlC thin films on the thickness of Ta underlayer.

SmFeAlC thin films on the Ta underlayer thickness. It is seen that both H_c and M_c increase with increasing the thickness of the Ta underlayer up to about 400 Å, and then remain nearly constant with the Ta underlayer thickness increasing further. TEM images show grain sizes in the range of 15 nanometers. Figure 3 shows a bright field TEM micrograph of a SmFeAlC thin film. Attempts at structural analysis of SmFeAlC thin films by x-ray diffraction have not been successful because no characteristic crystalline peaks were observed on the diffraction pattern except peaks of <002> texture of Ta and <111> texture of Si. This result is consistent with other observations of SmCo and SmFeSiC thin films by Shan et al. [8] and Zhang et al. [7]. Probably this is attributed to small SmFe crystallites normally obtained with these sputter-deposited rare-earth thin films, which are in the so-called x-ray amorphous states. Further efforts for analyzing crystal structure of the magnetic phase are in progress by convergent beam electron diffraction and HRTEM.



Fig.3.Bright field TEM micrograph of Si/Ta(1000Å)/ [SmFe(40Å)/ C(A1)(5Å)] \times 50/Ta(200Å) thin film after annealing at 650°C for 5 min. The grain size is about 150Å.

Figure 4 shows a coercivity of SmFeAlC thin film as a function of the number of SmFe/C(Al) bilayers, that is, the relationship between coercivities and the total thickness of films. It can be seen that the coercivities of the thin film increase when the total thicknesses of the thin films increase from 500Å to 1500Å, and then remain nearly constant with further increase of the film thickness. This result suggests that the hard magnetic phase does not appear until the thickness of the SmFeAlC layer is larger than 1000 - 1500Å.



Fig.4 Coercivity of Si/Ta(1000Å)/[SmFe(40Å)/C(Al) (10Å)]×N/Ta(200Å) thin films on the number of SmFe/C(Al) bilayer N, that is, the relationship between coercivities and the total thickness of the SmFeAlC thin films. Thin films after annealing at 750 °C for 5 min.

A typical hysteresis loop of SmFeAlC thin film is shown in Fig. 5. Loops were measured at room temperature by the SQUID with a maximum applied field of 55 kOe. an in-plane coercivity as high as 28 kOe was realized in the SmFeAlC thin films. The saturation magnetization is about 340 emu/cc. Loops for both in-plane and perpendicular directions are nearly the same, which means that the SmFeAlC thin films prepared by this method are approximately isotropic. The kink near zero field suggests either the presence of a second soft phase or a low field nucleation phenomena that initiates magnetization reversal.



Fig.5 Hysteresis loop of the SmFeAlC thin film with the structure of $Si/Ta(1000 Å)/[SmFe(40 Å)/C(Al) (5Å)] \times 50/Ta(200 Å)$. Thin film after annealing at 650°C. (a) Applied field is in-plane; (b) Applied field is perpendicular to the thin film plane.

To the best of our knowledge, the exceptionally high coercivity of 28 kOe found for the first time here in SmFeAIC thin films is comparable to that of SmCo thin films (about 31 kOe)[12], and is higher than that of SmFeSiC thin films (about 7.2 kOe)[7] and SmFeAIC compounds (9.2 kOe)[6]. The reason for the higher coercivity in SmFeAlC thin films is probably associated with the specific nanostructure and/or defects obtained with these sputter-deposited thin films. The saturation magnetization of SmFeAlC thin films, however, is somewhat lower, although it is the same order of the magnitude to that of SmCo thin films. The reason for this is that the replacement of Fe by Al, which is nonmagnetic, leads to a considerable reduction of saturation magnetization. The effort to adjust the composition of SmFeAlC thin films is in progress in order to improve the saturation magnetization. The dependence of high H_c on SmFeAl composition is unclear now but it has been shown that the H, strongly depends on the Al content. Further studies of the effect of Al addition on the magnetic properties are under way.

IV. CONCLUSION

Thin films of SmFeAlC having interesting magnetic properties can be successfully fabricated using SmFe/C(Al) multilayer sputtering plus thermal processing. Thin film with structure of Si/Ta(1000Å)/[SmFe(40Å)/C(Al)(5Å)]×50/Ta(200Å), which was subsequently annealed at 650°C, has a coercivity of 28 kOe. This exceptionally high value is considerably higher than the value obtained with bulk

specimens of SmFeAlC compound, but these could be an altogether different composition and structure. However, the relationships between higher coercivity and the composition of the optimal thin films, and the crystal structure of a magnetic phase need to be further investigated in detail.

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