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Influence of Magnetic Annealing on Properties of SmFe Thin Films

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Abstract: SmFe thin films were prepared by DC magnetron sputtering at room temperature and 300 $^{\circ}$ C. The influence of magnetic annealing temperature on the phase structure and magnetic properties was investigated. Results showed that thermal sputtering followed by a heat treatment process helped to obtain a structure with a relatively large fraction of SmFe₂. Residual phases observed were α -Fe, Sm₂O₃, and unknown phases. During the annealing treatment, the intrinsic compressive stress in SmFe films was relieved and could become tensile at higher annealing temperatures. The degree of in-plane anisotropy weakened, and furthermore, the anisotropy transformed into out-of-plane anisotropy.

Key words: magnetic annealing; anisotropy; compressive stress; SmFe thin films; rare earthsCLC number : TM271Document code: AArticle ID: 1002 - 0721(2007)04 - 0444 - 05

Giant magnetostrictive rare earth (RE)-Fe based allovs were developed by Clark, Belson^[1] and Koon et al., approximately one quarter of a century ago^[2]. Although many RE-Fe alloys are found to exhibit giant magnetostriction^[3], studies have mainly been directed towards Tb-Fe and Sm-Fe based alloys, because of their large magnetostriction at room temperature^[4]. TbFe₂-related compounds known as "Terfenol" have already been commercially used as powerful and fast actuators^[5,6]. Because of the prohibitively high material cost of Tb, their use has been mostly limited to military or high-end applications. For a broader range of applications in high magnetostriction materials, SmFe₂ with large saturation magnetostriction and a relatively low material cost will be a preferable choice. Furthermore, in contrast to the TbFe2-based compounds, which exhibit elongation under an applied magnetic field, SmFe₂ produces a negative magnetostricton that will be useful in applications requiring large instantaneous compressive stress/strain

on demand^[7]. With rising applications in microelectromechanical systems, SmFe₂ thin films could provide a fast-responding microactuator that can be remotely switched using a magnetic field^[8]. A large number of investigations have been performed in this field. Amorphous Sm-Fe thin films obtained by double-cathode DC sputtering have shown in-plane anisotropy, whereas, films obtained by opposite-target DC magnetron tend to show a tilted anisotropy^[9]. Lim S H et al.^[10], report that adding small amounts of B improved the magnetic and magnetostrictive properties of Sm-Fe based thin films. The giant magnetostrictive multilayers prepared by alternately depositing SmFe hard-magnetic films and NiFe soft-magnetic films can further reduce the saturation field^[11]. Stresses in this film also influence the magnetic anisotropy of the film with compressive stresses promoting in-plane easy magnetization^[12]. Field-sputtering under a magnetic field of 40 ~ 48 kA \cdot m⁻¹ was carried out to induce anisotropy^[13]. However, few fundamental studies

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have been undertaken on the influence of magnetic annealing on SmFe films. Further research on the magnetic properties of SmFe films under magnetic annealing will be beneficial for the technological applications of magnetostrictive material devices.

In this study, SmFe films were prepared by DC magnetron sputtering and annealed in a magnetic field. The influence of magnetic annealing was shown to depend on the stresses present in the film during annealing.

1 Experimental

SmFe films with the thickness of approximately 600 nm were deposited on a silicon substrate by DC magnetron sputtering. The temperature of the substrate was controlled at room temperature and 300 $^{\circ}$ C. A SmFe target (Φ 70 mm) with 45% (atom fraction) Sm and 55% of Fe was utilized. High-pure argon (99.999%) was employed as the sputtering gas and the sputtering pressure was 0.7 Pa. Additional sputtering conditions were used: 1×10^{-4} Pa base pressure, 60 mm target to substrate distance, and 30 W DC input power. The film composition determined by EDX was Sm₃₆Fe₆₄. The films were then given magnetic annealing treatments in high vacuum (below 2×10^{-4} Pa) with 48 kA \cdot m⁻¹ field applied in the plane. Annealing temperatures were applied at 225, 325, 425 and 550 ℃ for 30 min.

The crystal structure of the samples was examined by X-ray diffraction (XRD) [Rigaku, D/ max-RB] using Cu K α radiation. The surface morphology and composition of the films were characterized by using field emission scanning electronic microscopy (FE-SEM) [XL30S-FEG, Philips] and energy dispersive X-ray spectroscopy (EDX). Room temperature magnetic measurements were carried out in an ac grads sample magnetometer (AGM) [MicroMag' 2900, USA]. Samples were prepared by cutting with a diamond wafering saw. The properties measured include coercivity (H_c), saturation magnetization (M_s), and remanence (M_r). M_s and M_r depend on sample volume which may not be measured accurately. Therefore, the ratio of M_r/M_s was used to compare samples.

2 Results and Discussion

2.1 XRD characterizations

The XRD results provide more detailed information regarding the structure of the SmFe films. Fig. 1 shows the XRD patterns of SmFe films deposited at room temperature and annealed at different temperatures with a magnetic field. The XRD spectrum of the as-deposited sample reveals a very broad peak around 30° indicating that the sample is XRD amorphous. When the annealing temperature is $325 \ ^{\circ}C$ (Fig. 1(2)), a clear peak is evident, which can be attributed to the oxidation of Sm. In contrast, when the annealing temperature reaches $425 \ ^{\circ}C$ (Fig. 1 (3)), the diffraction peak of Sm₂O₃ intensifies. Elemental iron, SmFe₂ phase, and an unidentified phase are also detected.

Fig. 2 shows the results of the SmFe films deposited at 300 $^{\circ}$ C and magnetic annealed at different temperatures. The SmFe film prepared on a substrate and heated at 300 $^{\circ}$ C has crystallized. Portions of Sm atoms have been oxidized to Sm₂O₃ (Fig. 2 (1)). The peak of SmFe₂ {220} appears, but still shows low intensity. With the annealing temperature increasing, the diffraction peak of Sm₂O₃ has obviously intensified. At the same time, the peaks of SmFe₂ and Fe have al-



Fig. 1 XRD patterns for the SmFe films deposited at room temperature (1) and magnetic annealed at 325 °C (2), 425 °C (3)



Fig. 2 XRD patterns for the SmFe films deposited at 300 $^{\circ}$ C (1) and magnetic annealed at 550 $^{\circ}$ C (2)

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