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Research Article

Effects of Annealing Conditions on Magnetic Properties of $Ni_{81}Fe_{19}/Ir_{20}Mn_{80}$ Exchange-Biased Thin Films

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Abstract: The $Ni_{81}Fe_{19}/Ir_{20}Mn_{80}$ bilayer was grown using DC sputtering. After magnetic field cooling under an external magnetic field of 50 kOe, the room-temperature magnetic properties were investigated using resonance-vibrating sample magnetometry. The exchange bias field and coercive field were determined from hysteresis loops, and no training effect was observed during the remagnetization process. Effects of annealing conditions on magnetic properties of the exchange-biased thin film were investigated. The exchange bias field decreases with increasing annealing temperature, while the saturation magnetic moment is rather constant before dramatically decreasing at a critical annealing temperature. Saturation magnetic moment and exchange bias field of the exchange-biased thin film strongly depend on phase transformation of the ferromagnetic layer during the annealing process.

Keywords: Annealing, Exchange bias effect, Hysteresis loop, Mn diffusion

1. Introduction

The exchange bias effect was discovered in 1956 by Meiklejohn and Bean when studying Co particles embedded in antiferromagnetic (AFM) CoO [1]. It is one of the phenomena associated with the exchange anisotropy created at the interface between a ferromagnetic (FM) and an antiferromagnetic (AFM) material. The critical temperatures of materials should satisfy the condition: $T_C > T_N$, where T_C is the Curie temperature of the FM layer and T_N is the Neel temperature of the AFM layer. Exchange bias can be induced by the field cooling process.

After the field cooling process, the magnetic moment of the AFM near the AFM-FM interface is considered to be rigid in the field cooling direction. The AFM moments at the interface exert a microscopic torque on the FM moments and keep them in their original position. In other words, both the AFM and the FM are in a single domain state during the magnetization process. A stronger external field is needed to overcome this coupling and to rotate the ferromagnetic moments, and the center of the magnetization curve is displaced at negative values of the applied field. To achieve the exchange bias coupling, a minimum thickness (t_{cr}) of the AFM layer is required. With increasing AFM thickness, the exchange bias field increases and reaches a maximum value at a particular thickness. However, when the thickness of the FM layer increases, the critical AFM thickness for the exchange bias to occur also increases [2].

For magnetic materials, the annealing-dependent magnetic properties of materials can be explained by stress relief [3], grain modification [4], phase transition [5], and elementary diffusion [6]. The temperature dependence of exchange bias has been reported [7-9]. The increase in the value of the exchange bias field (H_{eb}) as measurement temperature decreases has been explained as the temperature dependence of the number of grains contributing to Heb. The effects of annealing temperature on exchange-biased systems have been investigated by Kim *et al.* [6]. The compositional depth profile at the NiFe/FeMn/CoFe interfaces obtained from X-ray photoelectron spectroscopy (XPS) results revealed the asymmetric compositional depth profiles of the Mn and Fe atoms throughout the FeMn layer at the different annealing temperature. The annealing temperature dependence of the exchange bias fields is related to compositional depth profiles of the Mn and Fe atoms throughout the FeMn layer.

In the exchange-biased systems, the anisotropies of the FM can affect the shape of the hysteresis loop and exchange bias field when exchange bias coupling at the FM/AFM interface is low and comparable to the coercive field of the FM layer. To eliminate these contributions, the high anisotropy AFM layer and low anisotropy FM layer were used to study the exchange bias effect. The Ir_xMn_{1-x} alloys were used widely in exchange-biased systems due to a large exchange bias field and good thermal stability [10]. The Néel temperature of bulk IrMn ranges from 690-1000 K [11-13]. For InMn/FM, the Néel temperature (498-550 K) of the thin IrMn layer is much lower than that of the bulk IrMn [14,15] due to the structural degradation induced by the lattice mismatch at the IrMn/FM interface. The coercivity and anisotropy field of $Ni_{81}Fe_{19}$ thin film with a thickness higher than 12.5 Å are low and seem to independent on the layer thickness [16]. Therefore, IrMn and NiFe are suitable for use as AFM and FM respectively for studying exchange-biased systems.

In hard-disk drive manufacturing, the read/write head patterns were grown on the Si wafer. Then, the wafers are cut into individual die pieces, etched, and cleaned. The dies were annealed to improve the stability of the read/write head. In this work, effects of annealing temperature, annealing time, annealing atmosphere, and heating rate on magnetic properties of exchange-biased thin films were investigated.

2. Materials and methods

The studied exchange-biased thin films were grown by DC magnetron sputtering. Initially, a Ruthenium (Ru) layer with a thickness of 10 Å was deposited onto a 6-inch (150mm) Si(100)/SiO_2 wafer and used as a seed layer. A heterostructural thin film of $Ni_{81}Fe_{19}$ (255 Å)/$Ir_{20}Mn_{80}$ (500 Å) was then deposited onto Si(100)/SiO_2/Ru. To induce exchange coupling, the films were heated to 560 K in an Ar atmosphere before being cooled to room temperature in the presence of an in-plane magnetic field of 50 kOe. Finally, the thin films were coated with a photoresist layer to protect them from oxidation. Afterward, the effects of annealing conditions on the magnetic properties of exchange-biased thin films were studied. The samples with width and length of about 3-4 mm were cut from the wafer, and after removal of the photoresist by acetone wiping, the samples were annealed using a tube furnace with the desired annealing conditions shown in Table 1. The room-temperature magnetic properties of the as-prepared and annealed samples were measured using an in-house developed resonance-vibrating sample magnetometer (R-VSM) [17]. To distinguish between the effects of annealing condition and the training effect on magnetic properties, variation in the hysteresis loop of the as-prepared exchange-biased sample due to repetition of the external cyclic magnetic field was also investigated.

Table 1 Annealing conditions of the exchange-biased thin films

Parameter	Annealing conditions			
	Temperature (Temperature step) (°C)	Time (h)	Atmosphere	Heating rate (°C/min)
Annealing temperature	150–300 (50) 300–500 (100)	2	Ar (30 lb/in^2) Air	5
Annealing time	150–250 (50)	0.5–48	Ar (30 lb/in^2)	5
Heating rate	200	2	Ar (30 lb/in^2)	1–20

3. Results and discussion

Figure 1 shows m-H curves with one to six external cyclic magnetic fields of the as-prepared $Ni_{81}Fe_{19}/Ir_{20}Mn_{80}$ exchange-biased thin film sample (see also Table 2). The superposition of m-H curves indicates that no training effect was involved during the remagnetization process of the sample. Coercive field H_c and exchange bias field H_{eb} of the exchange-biased magnetic materials can be calculated from the following equations:

$$H_c = \frac{(-H_{c1} + H_{c2})}{2} \quad (1)$$

$$H_{eb} = \frac{(H_{c1} + H_{c2})}{2} \quad (2)$$

where H_{c1} and H_{c2} are the applied fields at zero magnetization when the applied field changes from maximum value H_{max} to $-H_{max}$, and $-H_{max}$ to H_{max}, respectively. It is noted from the result that the exchange bias coupling at the AFM-FM interface of the sample is well induced.

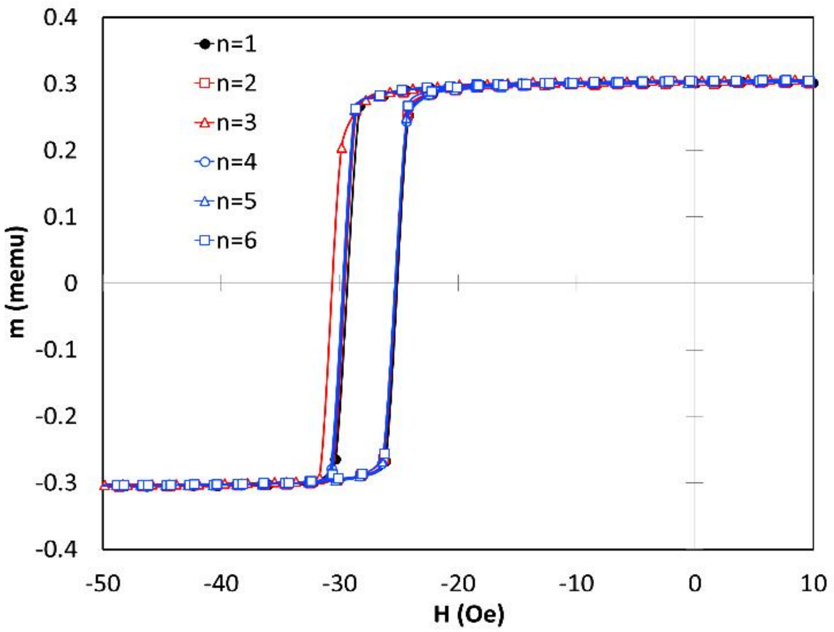

Figure 1 nth m-H curves of the $Ni_{81}Fe_{19}/Ir_{20}Mn_{80}$ exchange-biased thin films, where n=1 to 6.

Table 2 Exchange bias field (H_{eb}), coercive field (H_c), and saturation magnetic moment (m_s) obtained from nth m-H curves of the as-prepared $Ni_{81}Fe_{19}/Ir_{20}Mn_{80}$ exchange-biased thin films, where n=1 to 6

nth curve	H_{eb} (Oe)	H_c (Oe)	m_s (memu)
1	27.225	2.105	0.3055
2	27.415	2.155	0.3053
3	27.955	2.655	0.3052
4	27.495	2.155	0.3054
5	27.550	2.200	0.3053
6	27.440	2.180	0.3052

3.1 Effect of annealing temperature and annealing atmosphere

Hysteresis curves of the $Ir_{20}Mn_{80}/Ni_{81}Fe_{29}$ exchange-biased thin films before and after annealing at 300 °C for 2 h with a heating rate of 5 °C/min in argon atmosphere are compared in Figure 2. It can be observed that the hysteresis loop of the annealed sample shifts closer to the origin and the exchange bias field decreases, while saturation magnetic moment is comparable to that of the sample before annealing.

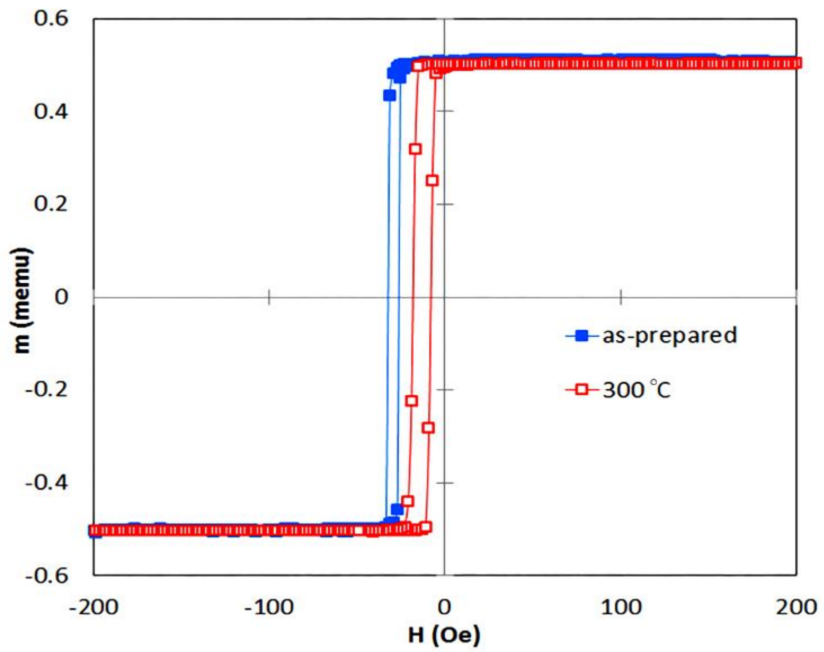

Figure 2 m-H curves of the sample annealed at 300 °C in argon atmosphere for 2 h with a heating rate of 5 °C/min compared with that of the as-prepared sample.

In Figure 3, the decrease in saturation magnetic moment (m_s) percentage of the samples annealed at different temperatures in the argon atmosphere is compared with that of the samples annealed in the air atmosphere. The drastic change of saturation magnetic moment starts earlier for the samples annealed in the air atmosphere. Since the magnetic moment of exchange-biased thin films is mainly due to magnetic ordering of the ferromagnetic NiFe layer, the decrease in m_s indicates the deterioration of this layer and may be a result of Mn diffusion from the antiferromagnetic IrMn layer to the ferromagnetic NiFe layer [6]. The formation of the NiFeMn antiferromagnetic phase causes a decrease in the net magnetic moment of the sample. In addition to Mn diffusion, the formation of iron oxides is accelerated during annealing, and the decrease in the magnetic moment of the samples annealed in the air atmosphere, therefore, occurs at a temperature lower than that of the samples annealed in the argon atmosphere.

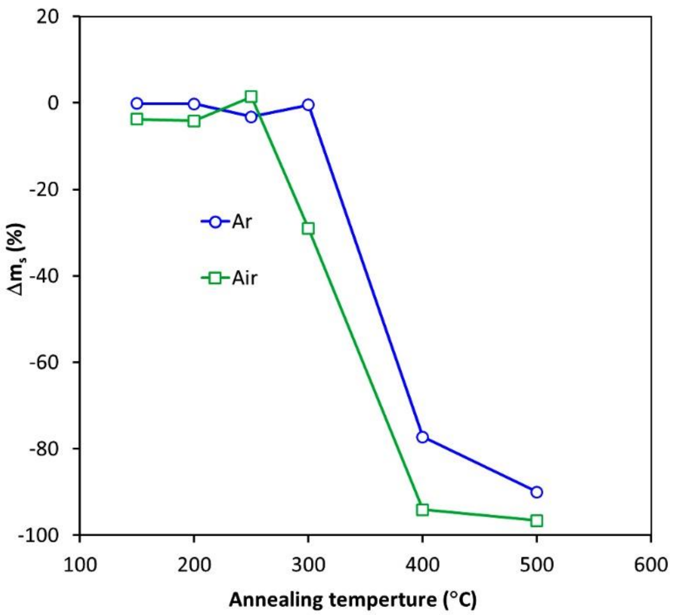

Figure 3 Percentage decrease of the saturation magnetic moment (m_s) of the samples annealed at different temperatures in argon atmosphere compared with those of the samples annealed in air atmosphere.

Exchange bias fields are plotted against annealing temperature, as shown in Figure 4. In the annealing temperature range lower than magnetic phase transformation temperature (250 °C and 300 °C for the samples annealed in air and argon atmosphere, respectively), the magnitude of the exchange bias field decreases rather linearly with annealing temperature. The decrease in the magnitude of the exchange bias field indicates the decrease in AFM-FM exchange bias coupling. According to the ideal Meiklejohm-Bean model [1], the exchange bias field of the exchange-biased thin film can be expressed as:

$$H_{eb} = -\frac{J_{eb}}{\mu_0 M_F t_F} \tag{3}$$

where μ_0 is the permeability of free space, M_F is the magnetization of the FM layer, J_{eb} is the interfacial exchange energy per unit area, and t_F is the thickness of the FM layer. Since m_s of the samples annealed at temperature 150–300 °C in argon atmosphere (and 150–250 °C in air atmosphere) is rather constant as shown in Figure 3, it is implied that MF and t_F of those samples were not affected by the annealing process. The decrease in J_{eb} is therefore the main factor contributing to the reduction of Heb. IrMn with Ir content of 20 at.% induces the highest exchange field in the NiFe/IrMn exchange-biased bilayer [18]. Since the Mn diffusion from the antiferromagnetic layer to the ferromagnetic NiFe layer, the at.% of Ir in the antiferromagnetic layer increase. According to the spin-glass model, the exchange bias can be reduced linearly as the function of interface disorder [19]. Variation of Mn content and increasing of disorder at the interface by thermal annealing may result in the decrease of J_{eb} and move the m-H curve closer to the origin. Annealing in air atmosphere, oxidation of Mn in antiferromagnetic layer to MnO_x can be more easily induced [20], and the decrease in the exchange bias field can be therefore accelerated, as shown in Figure 4.

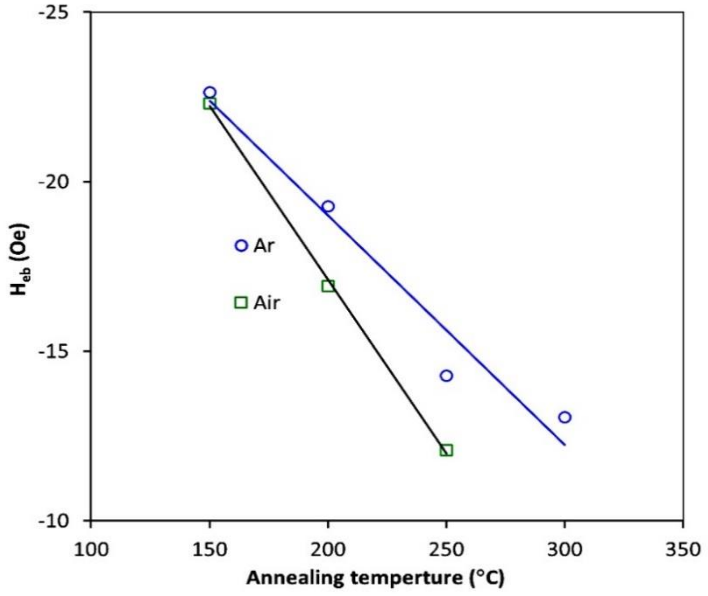

Figure 4 Variation of exchange bias field of the samples annealed at different temperatures in argon atmosphere compared with those of the samples annealed in air atmosphere.

3.2 Effect of annealing time

The effect of annealing time was studied using an argon atmosphere. The saturation magnetic moment of the samples annealed at 150–250 °C for 0.5 to 48 h is comparable to that of the samples before annealing. The exchange bias field is rather insensitive to the annealing time, but decreases with increasing annealing temperature, as shown in Figure 5 and Figure 6. It is noted that the annealing time of 30 min is sufficiently long to make the exchange-biased thin films system to reach a stable final state.

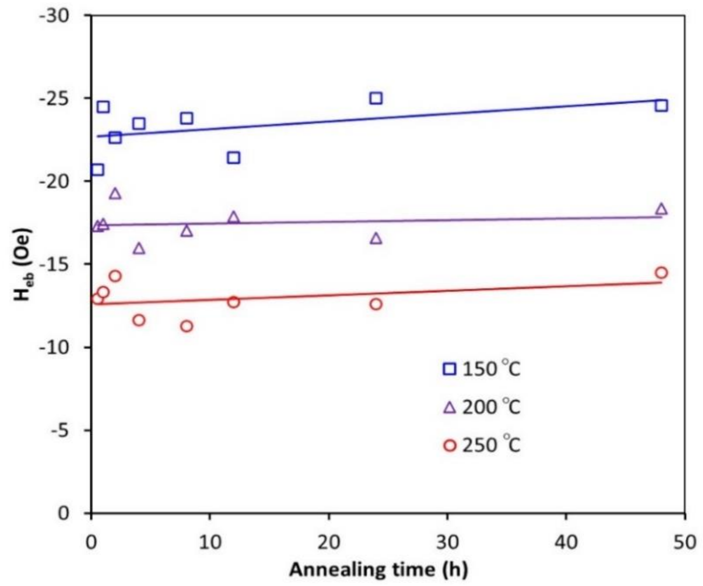

Figure 5 Exchange bias field of the samples annealed with different annealing times in argon atmosphere.

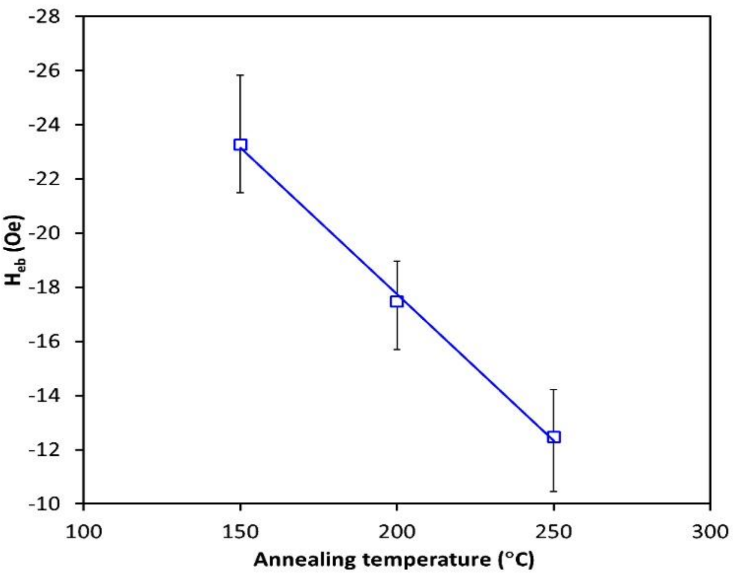

Figure 6 Average exchange bias field of the samples annealed at different temperatures in an argon atmosphere.

3.3 Effects of heating rate

In Figure 7, the exchange bias field of the samples annealed at 200 °C in an argon atmosphere is plotted against the heating rate. The exchange bias field gradually decreases with increasing heating rate. With increasing heating rate, the overshooting temperature (the difference between the set temperature and highest temperature) of the PID-controlled furnace increases. Since the exchange bias field is very sensitive to the temperature as shown in Figure 4 and Figure 6, the increase in overshooting temperature with increasing heating rate results in a decrease in the exchange bias field of the exchange-biased thin film system.

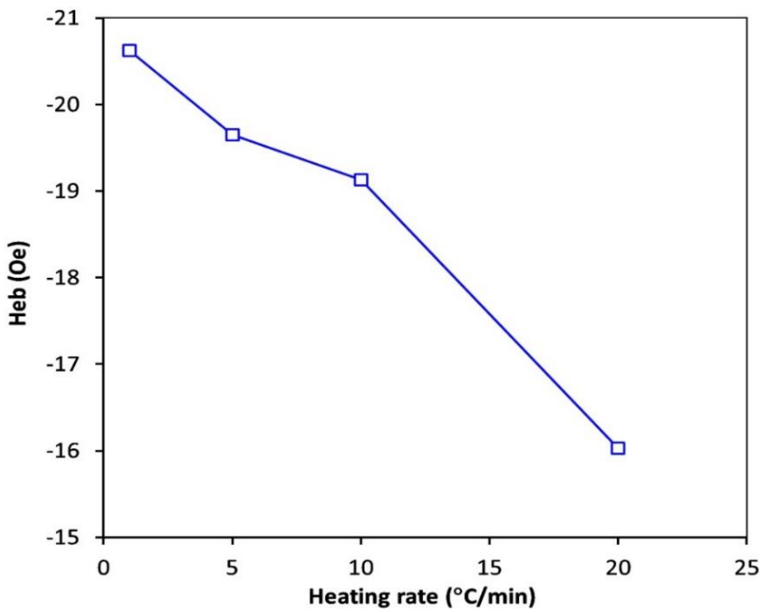

Figure 7 Exchange bias field of the samples annealed at 200 °C in an argon atmosphere with different heating rates.

4. Conclusions

Magnetic properties of exchange-biased thin films strongly depend on the annealing conditions. Annealing time of 30 min can be used to sufficiently control the magnetic properties to the stable final state for each annealing temperature. Monotonic decay in the exchange bias field indicates the decrease in AFM-FM exchange bias coupling with increasing annealing temperature. A drop of the saturation magnetic moment of the exchange-biased sample starts at a lower annealing temperature when annealing in the air atmosphere. At a higher heating rate, overshooting temperature increases and affects the decrease in the exchange bias field.

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