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Influence of temperature on thermal relaxation of exchange bias field in CoFe/Cu/CoFe/IrMn spin valve*

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A multilayered spin valve film with a structure of Ta (5 nm)/$Co_{75}Fe_{25}$(5 nm)/Cu(2.5 nm)/$Co_{75}Fe_{25}$(5 nm)/$Ir_{20}Mn_{80}$ (12 nm)/Ta(8 nm) is prepared by the high-vacuum direct current (DC) magnetron sputtering. The effect of temperature on the spin valve structure and the magnetic properties are studied by x-ray diffraction (XRD), atomic force microscopy (AFM), and vibrating sample magnetometry. The effect of temperature on the exchange bias field thermomagnetic properties of multilayered spin valve is studied by the residence time of samples in a reverse saturation field. The results show that as the temperature increases, the IrMn (111) texture weakens, surface/interface roughness increases, and the exchange bias field decreases. Below 200 °C, the exchange bias field decreases with the residence time increasing, and at the beginning of the negative saturation field, the exchange bias field H_{ex} decreases first quickly and then slowly gradually. When the temperature is greater than 200 °C, the exchange bias field is unchanged with the residence time increasing.

Keywords: exchange bias field, spin valves, temperature, thermal relaxation

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1. Introduction

In 1986, Grunberg *et al.*[1] found that in the Fe/Cr/Fe sandwiched structure, exchange coupling between Fe layers occurs through the Cr layer, and when the Cr layer thickness is appropriate value, antiferromagnetic coupling is present between the two Fe layers. In 1988, Baibich *et al.*[2] reported that at a low temperature (4.2 K) with an outfield of 20 kOe (1 Oe = 79.5775 $A{\cdot}m^{-1}$), the resistance of Fe (3.0 nm)/Cr (0.9 nm) multilayered film prepared by molecular beam epitaxy is increased by 50%, far exceeding the anisotropic magnetoresistance (about 0.2%) of the pure Fe layer. This is called the giant magnetoresistance (GMR) effect, which has attracted attention because of its wide application prospects in high-density readout heads and magnetic storage devices (such as hard disks).[3–6] Therefore, the recording density of computer hard disks and the data reading speed of magnetic heads are greatly increased, which allows the hard disk capacity to be increased while their sizes are reduced. Because the exchange bias principle in ferromagnetic/antiferromagnetic systems has important applications in GMR heads, non-volatile memory, and magnetic transducers for high-density magnetic recording, materials with exchange coupling effects, such as spin valves and tunnel junctions, have been extensively investigated.[7–11]

The magnetic electronic devices,when being used, must be able to withstand high temperatures or magnetic fields, which reduces the size of the exchange bias field of the devices and also restricts their performances. The thermal stability of an exchange bias greatly influences the reliability of magnetic electronic device. For example, after being treated at 250 °C, the FeMn nailing spin valve multilayered film loses the nailing effect of its antiferromagnetic layer, and the GMR may even disappear.[12] In the IrMn nailing spin valve multilayered film, after about 100-°C temperature treatment, it was found that the nailing effect of IrMn antiferromagnetic layer is weakened, and exchange bias field is reduced.[13] Therefore, a study of the thermal–magnetic stability of the exchange bias will help design and use magnetic electronic devices. In addition, the study of thermal relaxation of an exchange bias field will reveal the nature of the exchange bias.

This paper focuses mainly on the influence of temperature on the structure and magnetic properties of spin valve. The relationship between temperature and exchange bias field is analyzed through modeling. At the same time, the characteristics of thermal relaxation of spin valve multilayered film are studied, and the thermal relaxation phenomenon is reasonably analyzed.

2. Experimental process

CoFe/Cu/CoFe/IrMn spin valve was prepared on a silicon substrate by high-vacuum magnetron sputtering (Japan

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vacuum MPS-4000-HC2). The structure and thickness of the spin valve was Ta(5 nm)/$Co_{75}Fe_{25}$(5 nm)/$Ir_{20}Mn_{80}$(12 nm)/Ta(8 nm). To prevent the sample from being oxidized in the air, an 8-nm-thick Ta layer was deposited on the IrMn layer as a protective layer. During sputtering, the background vacuum degree was higher than 5×10^{-7} Pa. High-purity argon gas was used for sputtering, the air pressure was 7×10^{-2} Pa, the sputtering power was 120 W, and the growth rate was controlled at 0.03 nm/s–0.12 nm/s. During deposition, an external magnetic field of 100 Oe was applied parallel to the film surface to induce an easy magnetization direction.

An HH10 VSM was used to measure hysteresis loops at room temperature. The magnetic field was applied parallel to the film surface, and the positive field direction was parallel to the external field direction added during deposition. The magnetic field scanning range was from −800 Oe to 800 Oe, and the scanning speed was 3 Oe/s. The texture of the film was studied with a D8-Advance XRD, and the interface roughness was studied by AFM.

During the thermal relaxation of spin valve, the magnetic field was swept from the forward saturation field to the reverse saturation field, and then remained under the reverse magnetic field with an absolute value greater than 200 Oe–300 Oe. After holding for a certain time, the magnetic field was swept from the reverse saturation field to the forward saturation field. During magnetic field conversion, the back branch curve of the hysteresis loop can be measured. When there is no hold time in the forward saturation field, the front hysteresis loop can be measured by sweeping directly from the forward field to the reverse field. A second residence was then performed at a magnetic field in a range of 200 Oe–300 Oe in the reverse saturation field. The effect of temperature on the thermomagnetic properties of the exchange bias field of the spin valves was studied by the time accumulation method.

3. Results and discussion

3.1. Effect of temperature on structure of spin valves

Figure 1 shows the x-ray diffraction (XRD) pattern of the CoFe/Cu/CoFe/IrMn spin valve film at room temperature, 150 °C, and 275 °C and IrMn has a good (111) direction texture. When the temperature increases to 150 °C, the peak intensity of IrMn (111) decreases (curve *b* and curve *c* in Fig. 1). When the temperature reaches 275 °C, the diffraction peak shifts towards a higher 2θ value, possibly because of chemical diffusion and strain-relief.[14,15]

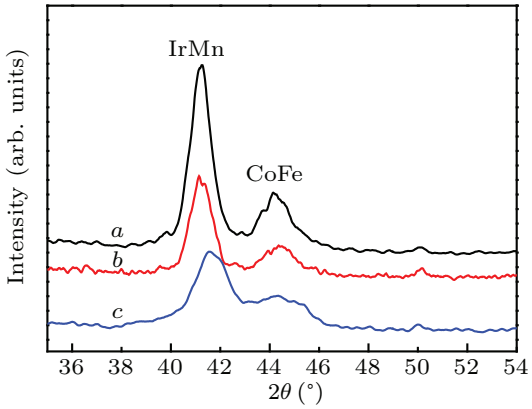


Fig. 1. XRD patterns of spin valves at room temperature (curve *a*), 150 °C (curve *b*), and 275 °C (curve *c*).

To further investigate the effect of temperature on the structure of the spin valve multilayered film, the surface/interface morphologies are studied by using AFM. It is generally believed that multilayered film deposited by magnetron sputtering is continuous and coherent at a certain thickness when it is deposited continuously at the same pressure. In this way, when the interface is not far away from the surface, the surface morphology is approximately the same as the interface morphology.[16] The ferromagnetic/antiferromagnetic interface in the bilayer film studied in this paper is 20 nm away from the surface. Thus, it is likely that the surface roughness of the sample can indirectly reflect the interfacial roughness between IrMn and CoFe layers.

The AFM images are shown in Fig. 2, where figures 2(a), 2(b), and 2(c) show the three-dimensional topograph of the sample at room temperature, 150 °C, and 275 °C, respectively. The corresponding R_{rms} roughness of the sample is 0.542 nm, 0.791 nm, and 1.078 nm, respectively. The surface/interface R_{rms} increases with the temperature rising after the spin valve has been heated. This is consistent with the conclusion obtained by Chen *et al.*,[17] in which the roughness of NiFe/FeMn bilayer increases with the temperature rising.

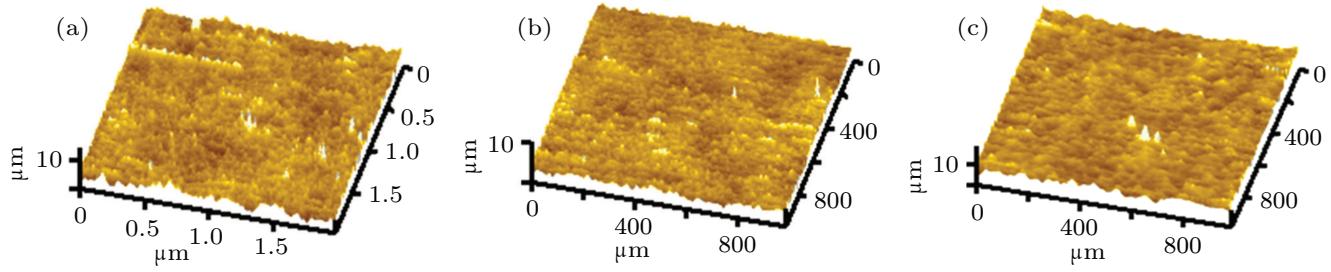


Fig. 2. AFM images of spin valve at (a) room temperature, (b) 150 °C, and (c) 275 °C.

3.2. Effect of temperature on magnetic properties of multilayer spin valve film

Figure 3 shows that the shape of the hysteresis loop of the CoFe/Cu/CoFe/IrMn spin valve changes greatly with the increase of temperature. $H_{ex} = (H_{c1} + H_{c2})/2$ (H_{c1} is the value of the forward branch of the hysteresis loop deviating from the zero field, and H_{c2} is the value of the backward branch of the hysteresis loop deviating from the zero field.) shows that the exchange bias field decreases as the temperature increases (specific data are given in Table 1). The coercive force of the pinned layer and the free layer can be obtained from the formula, $H_c = (H_{c1} - H_{c2})/2$. The coercivity of the nailed layer decreases at higher temperatures, while the coercivity of the free layer increases as the temperature increases (specific data are listed in Table 1).

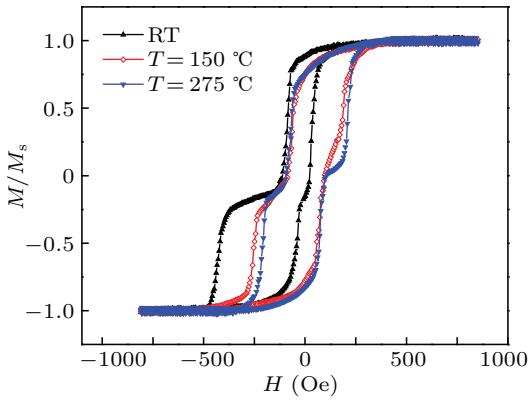


Fig. 3. Hysteresis loops of samples at T_m = room temperature (RT), 150 °C, and 275 °C.

Table 1. Values of spin valve exchange bias field, pinning layer, and free layer coercivity at three different temperatures.

Temperature (°C)	H_{ex} (Oe)	H_{cp} (Oe)	H_{cf} (Oe)
25	234	195	69
150	110	166	124
275	70	127	139

According to the XRD pattern (Fig. 1), IrMn has a good (111) directional texture after it has been heated at different temperatures, showing that the multilayered spin valve experiences exchange bias after having been heated. However, upon increasing the heating temperature, the IrMn (111) directional texture is weakened, which reduces the pinning effect of IrMn and the spin valve exchange bias field as well.

Roughness is also an important factor that affects exchange bias.[17–19] Generally, the exchange bias field decreases upon increasing the roughness;[20,21] therefore, the increased surface/interface roughness after heating at 150 °C and 275 °C is another major reason for the reduction in the multilayer exchange bias field of the spin valves.

The relationship between the temperature T_m and exchange bias field H_{ex} for the spin valve is shown in Fig. 4, where H_{ex} is normalized. H_{ex} decreases monotonically with temperature increasing, which has already been found in many thin films of exchange coupled systems, such as CoFe/IrMn exchange-coupled bilayer[22] and (CoFe, Co, and NiFe)/Cu/CoFe/IrMn spin valves.[23] The cutoff temperature of thin film can be determined by the linear extrapolation of the exchange bias and temperature plot, and this method shows that the cutoff temperature of the CoFe/Cu/CoFe/IrMn spin valve is about 340 °C.

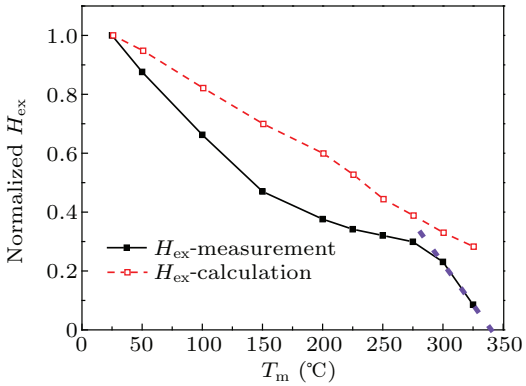


Fig. 4. Measured and calculated variation of exchange bias field (H_{ex}) with temperature of pinning layer in a spin valve.

In general, when the temperature is room temperature (T_m), the relation of the exchange bias field with the exchange coupling constant and the saturated magnetic moment can be written as[24]

$$H_{ex}(T_m) = \frac{J(T_m)}{M_{sp}(T_m) \cdot t_F}, \tag{1}$$

where $H_{ex}(T_m)$ is the exchange bias field at T_m, $J(T_m)$ is the interface exchange coupling constant at T_m, $M_{sp}(T_m)$ is the saturation magnetic moment of the pinned ferromagnetic layer at T_m, and t_F is the thickness of the pinned ferromagnetic layer of the spin valve.

The relationship between J and T_m can be written as[25,26]

$$J(T_m) = J(0)(1 - T_m/T_N)^{1/2}, \tag{2}$$

where $J(0)$ is the exchange bias constant between ferromagnetism and antiferromagnetism at 0 K, and T_N is the Nell temperature.

According to Eqs. (1) and (2), the equation representing the effect of the size of H_{ex} becomes

$$H_{ex}(T_m) = \frac{J(0)(1 - T_m/T_N)^{1/2}}{M_{sp}(T_m) \cdot t_F}. \tag{3}$$

According to formula (3), the relation between H_{ex} and T_m is fitted as shown by the dotted line in Fig. 4, which indicates that there is a slight deviation between the fitted data and the experimental data. This is mainly because changes in the microstructure, such as grain size and texture, are not considered in the fitting formula. However, in the actual processes, increasing the temperature will affect the microstructure (Fig. 1).

Figure 5 shows the coercivity of the free layer H_{cf} *versus* temperature and the saturation magnetic moment M_{sp} *versus*

temperature of the spin valve multilayered film. The change in the coercivity with T_m of the free layer in spin valve is non-monotonic, and the maximum value appears at $T_m = 225$ °C. This phenomenon has also been observed in exchange-coupled NiFe[27] and CoFe films.[28,29]

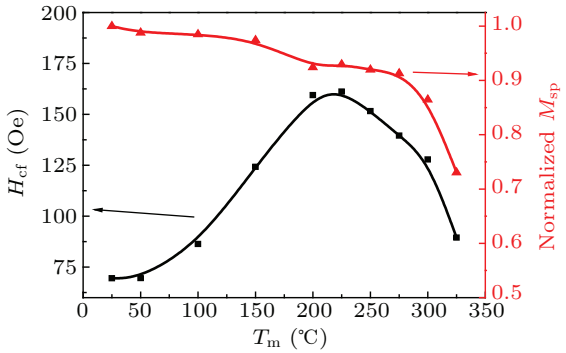


Fig. 5. Coercivity of the free layer (H_{cf}) and saturation magnetic moment (M_s) *versus* temperature of the spin valve multilayered film.

When the temperature is lower than 225 °C, both T_m and H_{cf} increase, mainly because of the phase transition of the CoFe free layer. In general, when CoFe is deposited on the Cu layer, an fcc structure forms easily.[30] In this study, the CoFe free layer deposited on the Ta seed layer may form a partial bcc structure. As the temperature increases, some CoFe with a bcc structure changes into an fcc structure, which increases the fcc phase in the CoFe free layer. Because fcc phase is harder than bcc phase, H_{cf} increases with T_m increasing when the temperature is lower than 225 °C.

When the temperature is higher than 225 °C, T_m increases and H_{cf} decreases. This is mainly due to the decline in the texture direction of CoFe(111) in the free layer at elevated T_m (Fig. 1), resulting in a less-anisotropic material.[31] In addition, the transition from a bcc structure to an fcc structure of the CoFe free layer may terminate at about 225 °C. For these two reasons, H_{cf} decreases as T_m increase at temperatures greater than 225 °C.

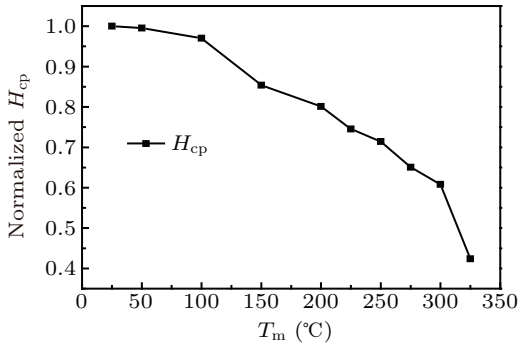


Fig. 6. Normalized coercivity H_{cp} varying with temperature in nailed layer.

Figure 6 shows the variation in the coercivity of the nailing layer H_{cp} with temperature T_m. The normalized coercivity H_{cp} of the nailed layer decreases monotonically with the temperature increasing, which is consistent with the Lam Y *et al.*'s result,[32] who studied the influence of temperature on the coercivity of NiMn/CoFe/NiMn film.

3.3. Effect of temperature on thermal relaxation of multilayer spin valve film

The effect of temperature on the thermal relaxation of the spin valve is studied by placing the samples in a reverse saturated field for different times. Figure 7 shows the variations of multilayered film exchange bias field with residence time in the reverse saturation field of the spin valves at different temperatures. As can be seen from Fig. 7, when the temperature is lower than 200 °C the exchange bias field decreases as the residence time increases At the beginning of the negative saturation field, the exchange bias field decreases first faster, and then slowly gradually. When the temperature is higher than 200 °C, the exchange bias field remains unchanged as the residence time increases.

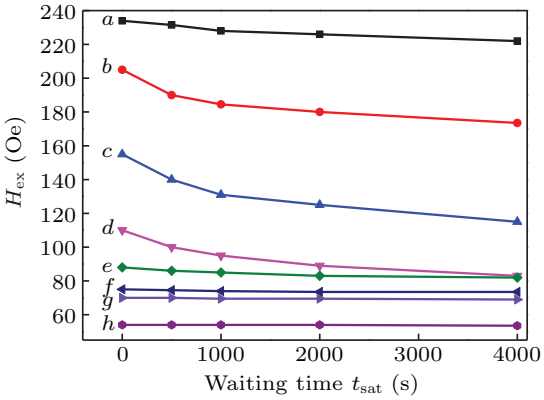


Fig. 7. Curves of spin valve multilayered film exchange bias field (H_{ex}) *versus* residence time in reverse saturation field (t_{sat}) at (curve *a*) room temperature, (curve *b*) 50 °C, (curve *c*) 100 °C, (curve *d*) 150 °C, (curve *e*) 200 °C, (curve *f*) 250 °C, (curve *g*) 275 °C, and (curve *g*) 300 °C.

When the temperature is lower than 200 °C, the exchange bias field of the spin valves decreases with the residence time increasing because the magnetic moments in some regions of the antiferromagnetic layer are reversed due to the thermal activation across the energy barrier.[33–35] When the applied magnetic field is scanned from the forward saturation to the reverse saturation, the magnetic moment direction of the ferromagnetic layer is parallel to the direction of the external magnetic field, for the Zeeman energy produced by the external magnetic field is greater than the exchange coupling energy between ferromagnetism and antiferromagnetism. However, the anisotropy of the antiferromagnetic layer is greater than that of the Zeeman energy generated by the external magnetic field. Therefore, the magnetic moment of the antiferromagnetic layer does not change with the direction of the external magnetic field but remains in its original direction. At this time, the system is in a metastable state. When the CoFe/Cu/CoFe/IrMn spin valve multilayered film is still in the reverse saturation field, the magnetic moment of antiferromagnetism begins to reverse due to exchange coupling between

strong ferromagnetism and strong antiferromagnetism. The anisotropy energy of the antiferromagnetic layer decreases with the reversal of the magnetic moment, which weakens the pinning effect of the antiferromagnetic layer. With the increase of the residence time of CoFe/Cu/CoFe/IrMn in the reverse saturation field, more magnetic moments in the antiferromagnetic layer are reversed by thermal activation across the energy barrier distribution. Finally, the multilayer film exchange bias field of the spin valve decreases with the increase of the residence time in the reverse saturation field.

At each temperature below 200 °C, at the beginning of the negative saturation field, H_{ex} decreases first faster and then slowly gradually because the smaller antiferromagnetic (AF) domain barrier is lower. The smaller AF domains are reversed more easily than the larger ones.[36] The number of small domains with low energy barriers in the antiferromagnetic layer decreases with residence time increasing at each temperature. In addition, magnetic domain nucleation and domain growth occur during inversion. Under a negative saturation field, vacancies and crystal defects are generated in the thin film, and the magnetic domain nucleation process is more likely to occur near crystal defects.[37] With the film's residence time in the negative saturation field increasing, the number of defects available for nucleation gradually decreases in comparison with the number of initial nucleation defects. Thus, at the beginning of the residence in a negative saturation field, at each temperature, H_{ex} began to decrease first faster and then slowly gradually.

When the temperature is higher than 200 °C, the exchange bias field does not change with the residence time increasing, which is mainly because the high temperature increases the energy barrier of magnetic moment inversion in some regions of the antiferromagnetic layer and narrows the overall energy barrier distribution. During residence in a reverse saturation field, the exchange coupling energy between the ferromagnetic layer and the antiferromagnetic layer cannot overcome the energy barrier of the antiferromagnetic moment reversal, because the energy barrier of the moment reversal of each part of the antiferromagnetic layer is relatively large. That is to say, the magnetic moment of the antiferromagnetic layer does not flip when the residence time in the reverse saturation field increases. Therefore, when the temperature is greater than 200 °C, the exchange bias field of the spin valve does not change at a longer residence time.

It can be seen from Fig. 7 that temperature speeds up the thermal relaxation of H_{ex} in the exchange bias field, that is, the increase of temperature quickens the decrease of H_{ex}. The distribution of the energy barrier determines AF domain inversion, and the mechanism of AF domain inversion is different at different temperatures.[38] When the temperature is relatively low, the magnetic domain of AF layer is reversed uniformly. The energy barrier is fairly wide. To achieve a reversal, all domains must simultaneously cross the highest energy barrier. This reversal pattern must therefore be powerful enough. As the temperature increases, the reversal mechanism changes from uniform inversion to the inversion in the helical domain form. The energy barrier distribution of the inversion in the spiral domain form is relatively narrow, so it is easier to realize the inversion. As the temperature increases, the number of reversals increases due to the helical domains formed by magnetic domains.[36,38] So this speeds up the decrease of H_{ex}. Therefore, the temperature increase can quicken the thermal relaxation of the spin valve exchange bias field H_{ex}.

4. Conclusions

The effect of temperature on the thermal relaxation of CoFe/Cu/CoFe/IrMn spin valve in an exchange bias field is studied. The results show that the temperature affects the structure and magnetic properties of CoFe/Cu/CoFe/IrMn. As the temperature increases, the texture of IrMn (111) decreases, the surface roughness and interface roughness increase, and the exchange bias field decreases. When the temperature is lower than 200 °C, the exchange bias field decreases with residence time increasing, and when the temperature is greater than 200 °C, the exchange bias field is basically unchanged though the residence time increases.

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