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著者	角田 匡清
journal or publication title	Applied Physics Letters
volume	84
number	25
page range	5222-5224
year	2004
URL	<a href="http://hdl.handle.net/10097/35036">http://hdl.handle.net/10097/35036</a>

doi: 10.1063/1.1765739

# Exchange anisotropy of polycrystalline Mn–Ir/Co–Fe bilayers enlarged by long-time annealing

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(Received 24 November 2003; accepted 29 April 2004; published online 10 June 2004)

The effect of long-time annealing on the exchange anisotropy of polycrystalline  $\text{Mn}_{75}\text{Ir}_{25}$   $d_{\text{AF}}/\text{Co}_{70}\text{Fe}_{30}$  4 nm bilayers was investigated to induce large unidirectional anisotropy constant,  $J_K$ , with very thin antiferromagnetic layer. As a notable result, extra large value of  $J_K=0.87$  erg/cm<sup>2</sup> was obtained in the bilayer with  $d_{\text{AF}}=5$  nm after 200 h annealing at 250 °C, which is larger than twice the maximum value of PtMn/Co–Fe system usually used in spin valves of hard disk drives. According to the single spin ensemble model, the enlargement of  $J_K$  by the long-time annealing is explained as a result of the change of the distribution of antiferromagnetic spin directions. © 2004 American Institute of Physics. [DOI: 10.1063/1.1765739]

Reduction of total thickness of spin valves (SVs) is one of the key issues to realize ultrahigh density hard disk drives (HDDs), because the shield gap for a reproducing head element becomes narrower and narrower with increasing the linear recording density. The SV-head-architectures with current-perpendicular-to-plane (CPP) configuration have been proposed to satisfy this crucial requirement.<sup>1,2</sup> Even when the CPP architecture is utilized, however, the antiferromagnetic (AFM) layer, whose thickness is dominant in the total thickness of SVs, should be thinner without degrading exchange biasing properties, in order to achieve higher recording density. Mn–Ir is a promising candidate for the AFM layer in forthcoming ultrahigh density HDDs, because of its very thin critical thickness,  $d_{\text{AF}}^{\text{cf}} < 5$  nm, above which the exchange anisotropy is induced in the adjacent ferromagnetic (FM) layer.<sup>3–6</sup> One of reasons for the practical use of PtMn in SVs of the present HDDs is a strength of exchange anisotropy. Mn–Ir has been believed to induce weaker exchange anisotropy than PtMn. However, as the authors demonstrated, a large unidirectional anisotropy constant,  $J_K$ , is inducible with Mn–Ir by changing a chemical composition of the FM layer.<sup>7</sup>  $J_K$  of Mn–Ir/Co<sub>70</sub>Fe<sub>30</sub> bilayer exceeds 0.5 erg/cm<sup>2</sup> after 300 °C annealing,<sup>7</sup> and is 25% greater than the values reported in PtMn/Co–Fe system.<sup>8,9</sup> In the present study, we investigated the effect of thermal annealing on the exchange anisotropy of the Mn–Ir/Co<sub>70</sub>Fe<sub>30</sub> bilayers in detail, in order to induce large exchange anisotropy with very thin AFM layer thickness. In particular, a long-time annealing at relatively low temperature was attempted to suppress interdiffusion at a hetero-interface.

Mn–Ir/Co–Fe bilayers were deposited on thermally oxidized Si wafers with the structure of sub./Ta 5 nm/Ni–Fe 2 nm/Cu 5 nm/Mn<sub>75</sub>Ir<sub>25</sub>  $d_{\text{AF}}/\text{Co}_{70}\text{Fe}_{30}$  4 nm/Cu 1 nm/Ta 2 nm under an ultraclean sputtering process.<sup>10</sup> The thickness of Mn–Ir layer,  $d_{\text{AF}}$ , was changed from 2.5 to 20 nm. A

magnetic field of 30 Oe was applied in the film plane during the deposition of bilayers. The bilayers were annealed at 100–400 °C in vacuum less than  $5 \times 10^{-6}$  Torr under a magnetic field of 1 kOe along the same direction of the applied field during the deposition. Structural analysis was performed by x-ray diffraction (XRD) and grazing incident x-ray diffraction (GID) with Cu  $K\alpha$  radiation source. Magnetization curves were measured with a vibrating sample magnetometer at room temperature. The unidirectional anisotropy constant,  $J_K$ , was calculated with an equation of  $J_K = M_s d_F H_{\text{ex}}$ , where  $M_s d_F$  is the areal saturation magnetization of Co<sub>70</sub>Fe<sub>30</sub> layer, and  $H_{\text{ex}}$  is the exchange biasing field determined as a shift of the center of the magnetization curve along the field axis.

Figure 1 shows the  $d_{\text{AF}}$  dependencies of  $J_K$  for the bilayers annealed at various temperatures,  $T_a$ . The annealing duration was fixed at 0.5 h. In as-deposited state,  $J_K$  shows relatively small value ( $\approx 0.1$  erg/cm<sup>2</sup>), regardless of  $d_{\text{AF}}$ . With increasing  $T_a$ ,  $J_K$  gradually increases. Below  $T_a$

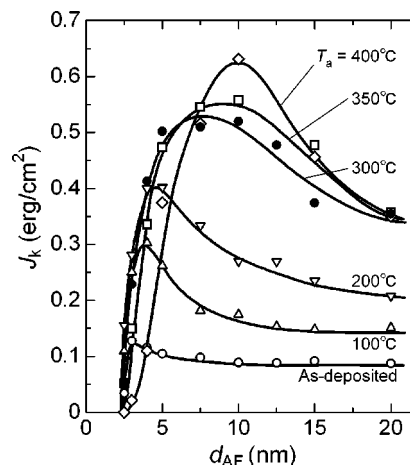


FIG. 1. Changes of  $J_K$  of  $\text{Mn}_{75}\text{Ir}_{25}$   $d_{\text{AF}}/\text{Co}_{70}\text{Fe}_{30}$  4 nm bilayers annealed at various temperature,  $T_a$ , as a function of the Mn–Ir layer thickness. Annealing duration was fixed at  $t_a=0.5$  h. Curves are guides for eye.

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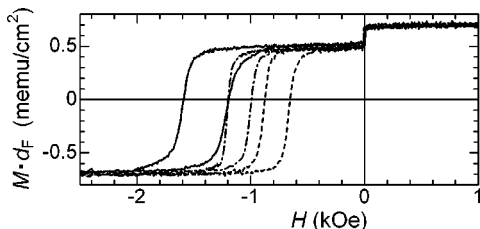


FIG. 2. Magnetization curves of Mn<sub>75</sub>Ir<sub>25</sub> 7.5 nm/Co<sub>70</sub>Fe<sub>30</sub> 4 nm bilayers annealed at 250 °C. Annealing duration,  $t_a$ , was 0.5 h (dashed), 20 h (dotted dashed), and 200 h (solid line).

=300 °C, a growing rate of  $J_K$  with increasing  $T_a$  is larger for the bilayers around  $d_{AF}=5$  nm than for the bilayers having thicker  $d_{AF}$ . When  $T_a=300$  °C,  $J_K$  shows a broad peak over 0.5 erg/cm<sup>2</sup> around  $d_{AF}=5-10$  nm. With further increasing  $T_a$  above 300 °C,  $J_K$  still increases in the bilayer with  $d_{AF}=10$  nm, while that in the bilayer with  $d_{AF} \leq 5$  nm remarkably decreases. This means a shift of the critical thickness of the AFM layer to higher value, and is not favorable from the application point of view. The increase of the critical thickness above  $T_a=300$  °C might be due to the interdiffusion at AFM/FM interface, resulting in a reduction of effective AFM layer thickness. We thus investigated the effect of long-time annealing under relatively low temperature on the exchange anisotropy of the Mn–Ir/Co<sub>70</sub>Fe<sub>30</sub> bilayers, in order to suppress the interdiffusion at the interface. We performed successive thermal annealing at  $T_a=250$  °C on the same bilayers to change the annealing duration. The cumulative annealing duration,  $t_a$ , was 0.5–200 h. Figure 2 shows the changes of magnetization curve of the bilayer with  $d_{AF}=7.5$  nm, for example. The vertical axis corresponds to the areal magnetization density. A small step observed at zero field corresponds to the magnetization reversal of the underlaid Ni–Fe layer. One can clearly see a shifting  $H_{ex}$  toward negative field direction with increasing  $t_a$ , while the saturation magnetization density is constant. Figure 3 shows the changes of  $J_K$  as a function of  $t_a$ . In the case of the bilayer with  $d_{AF}=3$  nm,  $J_K$  increases until  $t_a=5$  h and then decreases. For the cases of the bilayer with  $d_{AF} \geq 4$  nm, however,  $J_K$  monotonously increases with increasing  $t_a$ . After 200 h annealing, the extra large  $J_K$  of 0.87 erg/cm<sup>2</sup> is obtained in bilayers with  $d_{AF}=5-7.5$  nm. The value of 0.87 erg/cm<sup>2</sup> is larger than twice the maximum  $J_K$  reported for the PtMn/Co–Fe system, usually used in HDDs.

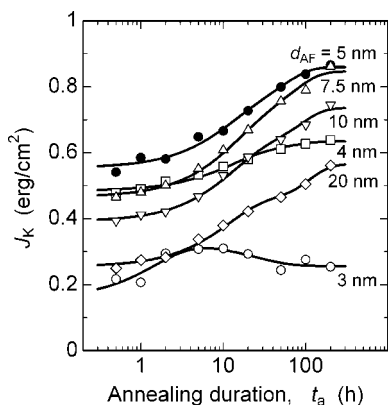


FIG. 3. Changes of  $J_K$  of Mn<sub>75</sub>Ir<sub>25</sub>  $d_{AF}$ /Co<sub>70</sub>Fe<sub>30</sub> 4 nm bilayers annealed at 250 °C as a function of cumulative annealing duration,  $t_a$ . Curves are the fitted calculation, based on the single spin ensemble model.

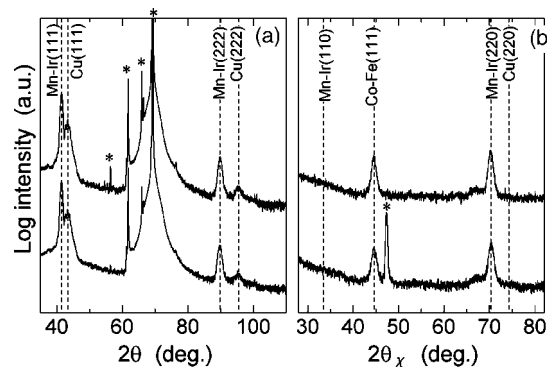


FIG. 4. Conventional x-ray diffraction profiles (a) and grazing incident x-ray diffraction profiles (b) of Mn<sub>75</sub>Ir<sub>25</sub> 10 nm/Co<sub>70</sub>Fe<sub>30</sub> 4nm bilayer, before (top) and after (bottom) thermal annealing at  $T_a=250$  °C,  $t_a=200$  h. Asterisks index the diffraction peaks from silicon substrate.

In order to clarify the cause of the enlargement of  $J_K$  by long-time annealing, structural analysis was carried out with x-ray diffraction technique. Figure 4 shows XRD (a) and GID (b) profiles of the bilayer with  $d_{AF}=10$  nm, before and after 200 h annealing at  $T_a=250$  °C. Remarkable differences within the accuracy of the present experiment are not recognized in the XRD profiles. The superlattice diffraction from Mn–Ir (110), expected for the ordered Mn<sub>3</sub>Ir, is not also detected in the GID profiles. Namely, no significant changes occurred in the microstructure of the bilayers by long-time thermal annealing, such as grain growth and formation of ordered phases. We otherwise confirmed from x-ray reflectivity measurements that the interfacial roughness between Mn–Ir and Co–Fe has not changed, too. We thus conclude that the cause of the enlargement of  $J_K$  is not the change of crystallographic structure but the change of the magnetic structure of bilayers.

We examined the single spin ensemble model (SSEM), which is an extended Meiklejohn and Bean model<sup>11</sup> for polycrystalline FM/AFM bilayers,<sup>12,13</sup> to elucidate the enlargement of  $J_K$ . In the SSEM, the AFM layer is regarded as an aggregation of the AFM grains. In as-deposited state of bilayers in the present study, directions of AFM spins are randomly orientated in the film plane, since the AFM layer was deposited first on the nonmagnetic Cu underlayer surface and no index was there to determine the direction of AFM spins. Figure 5 shows the schematic view of AFM spin directions of as-deposited bilayers. The “+state” grain corresponds to the AFM grain whose surface spin direction is close to the FM layer magnetization vector. The “+state” grain contributes for the unidirectional anisotropy along rightward direction in the figure. The “–state” grain indicates the AFM grain with opposite spin direction, and it contributes negatively for the unidirectional anisotropy.<sup>12</sup> As a result,  $J_K$  of the as-deposited bilayers are small, owing to the nearly random distribution of AFM spin direction. Once the thermal annealing is performed on the bilayers, the distribution of AFM spin directions changes. In Fig. 5, the total free energy of AFM grains,  $E_{AF}$ , is also shown as a function of the AFM spin direction.  $E_{AF}$  of “+state” grain is lower than that of “–state” grain owing to the interfacial coupling energy,  $J \cos(\alpha-\beta)$ , where  $J$  is the exchange coupling energy at the interface between the FM layer and the AFM grains,  $\alpha$  and  $\beta$  are the orientation angles for an AFM surface spin and a FM layer magnetization vector. The activation energy,  $E_a$ , corre-

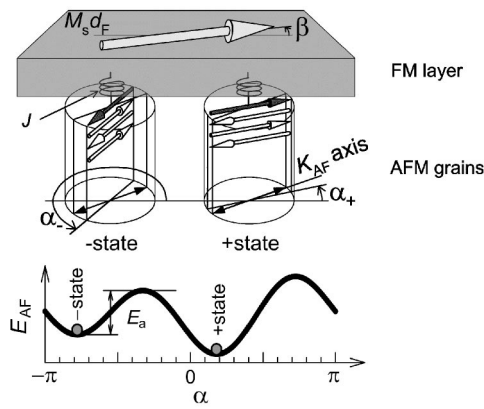


FIG. 5. Schematic illustration of the single spin ensemble model for the AFM spins in FM/AFM bilayer and total free energy of AFM grains as a function of the angle of the AFM spin direction. While the bilayer includes huge number of AFM grains, only two AFM grains were illustrated as cylinders, for example. Black colored single-headed arrows indicate the surface spin of AFM grains, facing to the FM layer. Small springs labeled  $J$  indicate the interfacial coupling strength. Double-headed arrows at the bottom of cylinders indicate the magnetic anisotropy axis of AFM grains,  $K_{AF}$ . The  $K_{AF}$  axis of each AFM grain is randomly oriented in the film plane.

sponding to the magnetic anisotropy energy of AFM grains, is needed for AFM spins to flip from “-state” to “+state.” The thermal annealing procedure supplies this energy to the AFM grains. To put it briefly, the enlargement of  $J_K$  with increasing annealing duration is regarded as the result of the change of the distribution of AFM spin directions.

We tried to fit the experimental data with the following equation, taking into account the enlarging  $J_K$  process by thermal activation and the degrading  $J_K$  process by interdiffusion:

$$J_K = J_K^{\max} \left\{ 1 - \exp \left[ -\frac{(t_a - t_0)}{\tau_{\text{act}}} \right] \right\} \times \left\{ (1 - \Delta) + \Delta \exp \left[ -\frac{t_a}{\tau_{\text{deg}}} \right] \right\}, \quad (1)$$

where  $J_K^{\max}$ ,  $\Delta$ ,  $t_0$ ,  $\tau_{\text{act}}$ , and  $\tau_{\text{deg}}$  are fitting parameters:  $J_K^{\max}$  is the achievable value of  $J_K$  after the infinite annealing duration without the degradation process,  $\Delta$  is degrading portion of  $J_K$  by interdiffusion,  $t_0$  gives an initial (as-deposited) state for the AFM spin distribution, and  $\tau_{\text{act}}$  and  $\tau_{\text{deg}}$  are the respective time constant for the enlarging and the degrading processes of  $J_K$ . The fitting parameters used are listed in

TABLE I. Respective values of the fitting parameters,  $J_K^{\max}$ ,  $\Delta$ ,  $t_0$ ,  $\tau_{\text{act}}$ , and  $\tau_{\text{deg}}$ , used for the calculation of the curves in Fig. 3.

$d_{AF}$ (nm)	$J_K^{\max}$ (erg/cm <sup>2</sup> )	$\Delta$ (erg/cm <sup>2</sup> )	$t_0$ (h)	$\tau_{\text{act}}$ (h)	$\tau_{\text{deg}}$ (h)
3	0.33	0.197	-0.78	1.2	20
4	1.02	0.469	-15.2	29.9	20
5	1.50	0.425	-14.4	31.4	20
7.5	1.50	0.439	-12.5	33.9	20
10	1.50	0.513	-10.8	36.3	20
20	1.50	0.628	-8.1	44.4	20

Table I.  $\tau_{\text{deg}}$  was fixed for all the bilayers, since the interdiffusion will progress independently on  $d_{AF}$ .  $J_K^{\max}$  was also fixed for the bilayers with  $d_{AF} \geq 5$  nm, in accordance with the SSEM, which deduces the saturating  $J_K$  beyond  $d_{AF}^{\text{cr}}$ . The calculated curves are shown in Fig. 3. They fairly fit the experimented data plots.

In conclusion, we induced, extra large  $J_K$  of 0.87 erg/cm<sup>2</sup> with very thin (=5 nm) AFM layer in Mn-Ir/Co<sub>70</sub>Fe<sub>30</sub> system, applying long-time annealing at relatively low temperature. It is a hopeful result to realize very-thin spin valves for the forthcoming ultrahigh density HDDs. The enlarging mechanism of  $J_K$  is suggested to be the change of distribution of AFM spin directions.

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