Enhancement of exchange bias in Mn-Ir/Co-Fe based spin valves with an ultrathin Cu underlayer and in situ Mn-Ir surface modification

doi: 10.1063/1.1357146

Enhancement of exchange bias in Mn–Ir/Co–Fe based spin valves with an ultrathin Cu underlayer and *in situ* Mn–Ir surface modification

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Enhancement of exchange bias induced at the interface of the antiferromagnetic (AF)/ferromagnetic (F) layers was studied using the bottom ''spin-valve films'' (SVs) with the Mn–Ir/Co–Fe exchange coupled films. Exchange bias increased using an ultrathin Cu underlayer. Meanwhile, both exchange bias field, H_{ex}, and blocking temperature, T_B, increased intensively by heating specimens after depositing Mn–Ir film in a high vacuum. These two enhancement effects worked in an additive. As a result, an unidirectional anisotropy constant, J_K, of 0.39 erg/cm^2 (H_{ex} of 1.3 kOe) and T_B of ~325 °C were obtained for the bottom SVs with a total thickness of 233 Å including an AF layer of 68 Å $Mn_{74}Ir_{26}$ and a pinned layer of 20 Å $Co_{90}Fe_{10}$, where the SVs were field annealed at 320 °C. A microstructural analysis using x-ray diffraction revealed that H_{ex} did not depend on the diffraction intensity from Mn–Ir (111) for the SVs with various underlayers, and no remarkable changes occurred in the microstructure of the SVs with the heating treatment in a vacuum. Therefore, the enhancement effects might result from some changes in the microstructure and/or the morphology of the interface of AF/F layers. © *2001 American Institute of Physics.* [DOI: 10.1063/1.1357146]

I. INTRODUCTION

Enlarging the biasing field of the pinned layers in spin valves (SVs) is one of the most important factors in putting them to practical use. The synthetic ferrimagnetic (Sy-ferri) structure has been proposed for pinned layers in order to apparently satisfy this requirement.[1–3] Exchange anisotropy of antiferromagnetic (AF)/ferromagnetic (F) bilayers is, however, essentially important even in the case of using Sy-ferri structure, because the direction of magnetization in one of the F layers in the Sy-ferri pinned layer should be fixed enough with an AF layer against the external applied field. It is also necessary to reduce the total thickness of SVs to achieve high recording density, while maintaining excellent heat durability to ensure the dynamic properties at high temperature. A Mn–Ir/Co–Fe system shows promise of obtaining strong exchange bias with an ultrathin AF layer.[4–7]

The microstructures of the AF layer and the interface between AF and F layers are the most important factors to be controlled, because the reduction of the AF grain size lowers the blocking temperature (T_B),[8–10] and the exchange anisotropy is essentially derived from the magnetic coupling at the interface. The microstructure of the AF layer is generally controlled with underlayers.[6,11–13] Pakala and co-workers[6] for example, suggested using $(Cu/Ru)_n$ multilayers as the underlayers of Mn–Ir/Co–Fe based SVs, and achieved large exchange bias (0.3 erg/cm^2 in the unidirectional anisotropy constant). The suggested underlayers, however, are difficult to use in SVs because of the large number of films and their total thickness when stacked. To date, the microstructure of AF/F layers has been generally controlled indirectly using the existing underlayers. In such a way, incidents other than the interface structure may influence simultaneously, and make it difficult to clarify the effect of the microstructural change of the interface on the exchange anisotropy.

In the present study, we fabricated Mn–Ir/Co–Fe based SVs under an extremely clean process,[14] a superior method of controlling the microstructure of thin films. We then investigated the dependence of ultrathin underlayers on the exchange bias field (H_{ex}). In order to intentionally modify the surface structure of AF layers, we heated the specimens on wafers in ultrahigh vacuum pressure after depositing Mn–Ir layers.

II. EXPERIMENTAL PROCEDURE

SVs were deposited on thermally oxidized Si wafers in the sequence of sub/Ta 50 Å/underlayer d_{UL}/$Mn_{74}Ir_{26}$68 Å/$Co_{90}Fe_{10}$20 Å/Cu25 Å/$Co_{90}Fe_{10}$20 Å/Cu10Å/Ta20 Å using a magnetron sputtering method with ~30 Oe of dc magnetic field in the film plane. The base pressure of the sputtering chambers was in the range of 10^{-11} Torr. Ultraclean Ar (9 N) was used as the process gas. SVs were postannealed at 280 °C–320 °C for 1 h, then cooled to room temperature in a magnetic field of 0.7 kOe along the same direction of the field applied during deposition. The annealing temperature was determined from the study of T_B mentioned below. The underlayers were fabricated in single or dual layers, using Ni–Fe, Cu, and Co–Fe. The thickness of the underlayers varied from 0 through 50 Å.

All the measurements were carried out for the annealed specimens. Magnetoresistance (MR) curves were measured using a dc four-probe method. $M-H$ curves were measured with a vibrating sample magnetometer. H_{ex} was estimated as the shift of the center of a hysteresis loop for a pinned layer from the zero field in MR or $M-H$ curves. Unidirectional anisotropy constant (J_K) was calculated using the equation of $J_K = H_{ex} M_P d_P$, where M_P is saturation magnetization and d_P is the thickness of a pinned layer. Structural analysis was carried out with an x-ray diffractometer (XRD).

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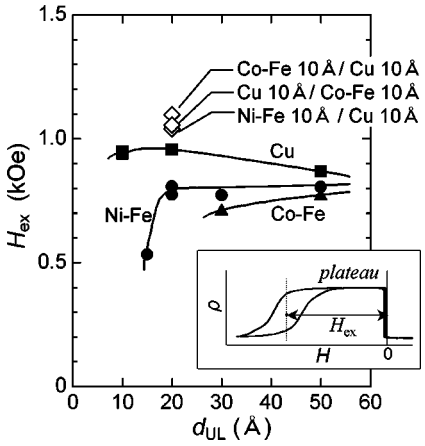

FIG. 1. Changes of exchange bias field, H_{ex}, as a function of the thickness of various underlayers, d_{UL}. A typical MR curve to explain a plateau and H_{ex} is shown in an inset.

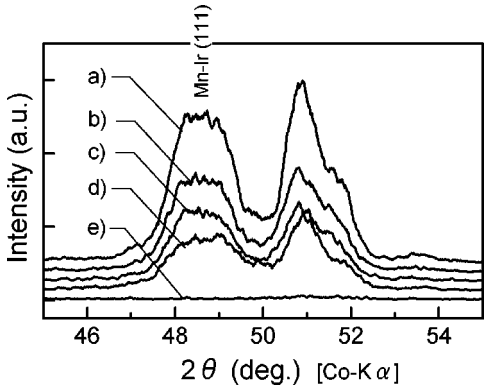

FIG. 2. XRD profiles for SVs with various underlayers (a) Ni–Fe 20 Å, (b) Co–Fe 10 Å/Cu 10 Å, (c) Ni–Fe 10 Å/Cu 10 Å, (d) Cu 10 Å/Co–Fe 10 Å, and (e) Ni–Fe 10 Å/Co–Fe 10 Å.

III. RESULTS AND DISCUSSION

A. Enhancement of J_K using ultrathin Cu underlayers

Figure 1 shows the changes of H_{ex} as a function of the thickness of various underlayers (d_{UL}). H_{ex} was estimated only when a plateau with good reproducibility appeared in a MR curve. Here, a plateau is the area where an antiparallel alignment is achieved between magnetization of pinned and free layers. (see an inset of Fig. 1). In the case of $d_{UL}=0$, without any underlayer, a MR curve was collapsed and H_{ex} could not be determined.

When Ni–Fe was used as an underlayer, H_{ex} was an almost constant value of 0.8 kOe($J_K=0.24\,\mathrm{erg/cm^2}$) above $d_{UL}=20$ Å and H_{ex} dropped below $d_{UL}=20$ Å. A plateau of a MR curve did not appear below $d_{UL}=10$ Å with sufficient reproducibility. When Co–Fe was used as an underlayer, H_{ex} gradually decreased with decreasing d_{UL} and showed 0.7 kOe at $d_{UL}=30$ Å. A plateau did not appear below $d_{UL}=20$ Å. In contrast, when Cu was used as an underlayer, H_{ex} maintained its large value of 0.95 kOe even with the ultrathin underlayer ($d_{UL}=10$ Å), which is favorable for the transport properties, avoiding large shunting current.

In the case of the dual underlayers of 20 Å in total thickness, including a Cu 10 Å layer, H_{ex} was larger than in the case of a Cu single underlayer. When Co–Fe 10 Å/Cu 10 Å were used as underlayers, for example, H_{ex} took the value of 1.1 kOe ($J_K=0.3\,\mathrm{erg/cm^2}$). In the case where the Cu layer was not used, Ni–Fe 10 Å/Co–Fe 10 Å, exchange bias was hardly induced and a plateau collapsed in a MR curve.

Figure 2 shows conventional θ–2θ scanned XRD profiles (Co Kα source) for SVs with various dual underlayers [Figs. 2(b)–2(e)]. An XRD profile for the SV with a Ni–Fe 20 Å single underlayer is also shown in Fig. 2(a). The profiles did not change from the respective profiles for as-deposited SVs, meaning that the postannealing procedure did not influence the microstructure of the SVs. The broad peaks around $2\theta=48°-49°$ correspond to the diffraction from Mn–Ir (111). The peaks around $2\theta=50°-52°$ result from the interference of XRD mainly caused by (111) of Co–Fe, Cu, and Ni–Fe. Diffraction peaks from planes other than (111) were not observed in the XRD profiles for any SVs.

In the case of dual underlayers with a Cu 10 Å layer [Figs. 2(b)–2(d)], which induced large H_{ex}, the diffraction peaks due to Mn–Ir (111) and other face-centered-cubic (fcc) (111) clearly appeared. The intensity of the peaks was about half of that in the case of a Ni–Fe 20 Å single underlayer [Fig. 2(a)]. On the other hand, in the case of Ni–Fe 10 Å/Co–Fe 10 Å [Fig. 2(e)], which barely induced exchange bias, the diffraction peaks from (111)s hardly appeared. From these results, it may be difficult to find a correlation between the intensity of Mn–Ir (111) diffraction peaks and H_{ex}. This is not consistent with the previous reports by Mao,[4] Pakala,[6] Anderson,[7] Nakatani,[11] each with their respective co-workers. One can only say that the grain size of SVs should be large enough to produce the diffraction peaks from (111) to induce exchange bias, since the reduction of grain size of AF films reduces T_B.

Consequently, the essential incident to enhance the exchange bias is not the changes in the crystallographic texture of the AF layers, but the changes in the microstructure of the interface of the AF/F layers. A direct modification for surface of AF (Mn–Ir) layers was then examined.

B. Enhancement of J_K and T_B by heat treatment

In order to modify the surface of the Mn–Ir films intentionally, specimens were heated after the deposition of Mn–Ir film under ultrahigh vacuum pressure.

The specimens were heated by IR irradiation from outside of the sputtering chamber through the α-Al_2O_3 window. The irradiation was controlled with current supplied to an IR lamp. Pressure of the chamber was in the range of 10^{-11} Torr before the heat treatment to prevent the surfaces of the Mn–Ir films from contamination due to impurity gasses. Temperature of specimens (Ta/UL/Mn–Ir films on the wafer) was varied between 70 °C–180 °C, which was estimated from the temperature of the sample stage holding a wafer on it. Holding time at maximum temperature was 20 min–1 h. After the specimens were then cooled to ~40 °C, a pinned layer (Co–Fe) and remaining layers were further deposited. An influence of the heat treatment on H_{ex} and T_B was investigated *ex situ*.

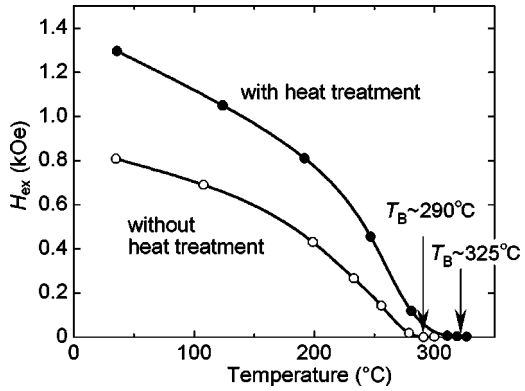

FIG. 3. Dependence of H_{ex} of SVs with a Ni–Fe 20 Å single underlayer on measuring temperature with and without the heat treatment (180 °C in 20 min) in a vacuum.

In the case of a SV with a Ni–Fe 20 Å single underlayer, having H_{ex} of 0.8 kOe without heat treatment, H_{ex} was enhanced up to 1.0 kOe using the heat treatment at 110 °C in 1 h. Furthermore, H_{ex} was enhanced up to 1.3 kOe using the heat treatment at 180 °C in 20 min. The value of $H_{ex} = 1.3$ kOe corresponds to 0.39 erg/cm^2 in J_K, which is comparable to values found in ordered AF/F layers, such as NiMn/Ni–Fe (Ref. 15) and PtMn/Co (Ref. 16).

In the case of a SV with Cu 10 Å/Co–Fe 10 Å dual underlayer, the heat treatment at 110 °C in 1 h enhanced H_{ex} up to 1.28 kOe. This value is larger than that for the SV with a 20 Å thick Ni–Fe single underlayer under the same heat treatment. It means that the enhancement of H_{ex} using the *in situ* heat treatment and using an ultrathin Cu underlayer works independently.

Figure 3 shows the dependence of H_{ex} of a SV with a Ni–Fe 20 Å single underlayer on measuring temperature with and without the heat treatment (180 °C in 20 min) in a vacuum. One can clearly find the enhancement of T_B from ~290 °C to ~325 °C after the *in situ* heat treatment.

In order to clarify the mechanism of enhancement of H_{ex} and T_B using the *in situ* heat treatment, a microstructural analysis was carried out with XRD (Cu Kα source). Figure 4(a) shows x-ray reflective profiles ($2\theta = 0° - 10°$) for the SVs with and without the heat treatment. Remarkable differences within the accuracy of this experiment were not recognized in the two profiles, meaning that no significant changes occurred in the interfacial roughness and the thickness of the Mn–Ir film. Figure 4(b) shows conventional θ–2θ scanned XRD profiles for the same SVs. The peaks around $2\theta = 41° - 42°$ and 43°–45° correspond to the diffraction from Mn–Ir (111) and (111) of other fcc materials, respectively. Remarkable differences were not recognized either between the two profiles. In order to examine the formation of ordered phase in Mn–Ir films, known as Mn_3Ir, in-plane XRD measurement was carried out for the same SVs using a grazing incidence angle XRD. Although a super lattice diffraction from Mn–Ir (110) was expected, only a fundamental diffraction from Mn–Ir (220) was detected.

In conclusion, some structural changes in the very surface of a Mn–Ir film, undetectable with XRD, occurred by heating a Mn–Ir film in a vacuum, and resulted in the enhancement of H_{ex} and T_B. As for surface changes, atoms in the very surface of a Mn–Ir film may evaporate when heated in high vacuum pressure, and thus the surface composition or the surface morphology of a Mn–Ir film may be mainly changed.

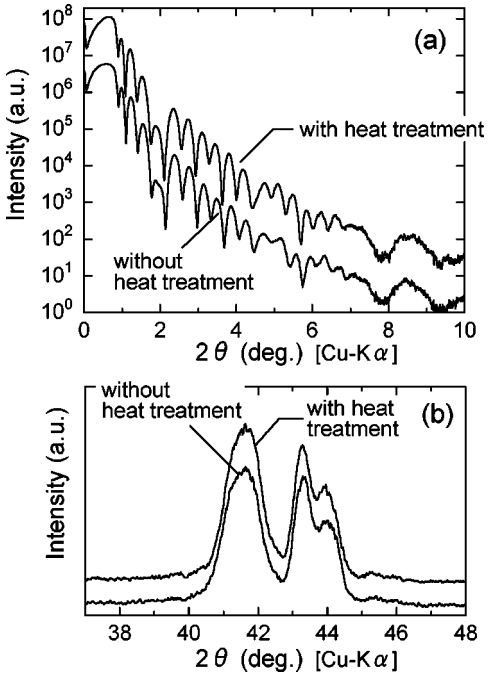

FIG. 4. (a) X-ray reflective profiles and (b) conventional θ–2θ scanned XRD profiles for SVs with a Ni–Fe 20 Å single underlayer, with and without the heat treatment (180 °C in 20 min) in a vacuum.

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