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## Controlled rotation of the exchange-bias direction in IrMn/Cu/Co via ion irradiation

D. Schafer, J. Geshev,<sup>a)</sup> S. Nicolodi, L. G. Pereira, J. E. Schmidt, and P. L. Grande  
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Co/Cu/IrMn films were irradiated with 40 keV He<sup>+</sup> ions varying the fluence and the current, with magnetic field applied at 120° with respect to the original exchange-bias direction. The angular variations of the exchange-bias field of the irradiated samples were compared with those of the as-made and the thermally annealed films. Gradual deviation of the exchange-bias direction with the fluence increase was observed. Complete reorientation of the easy axes of both ferromagnet and antiferromagnet toward that of the field applied during irradiation was achieved for fluences higher than  $1 \times 10^{15}$  ions/cm<sup>2</sup>, accompanied with a significant enhancement of the exchange-bias field.

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Since the pioneering paper of Chappert *et al.*,<sup>1</sup> low energy ion irradiation has been employed to controllably tune the magnetic properties of various thin films and multilayers in order to engineer the behavior of small magnetologic devices (for recent reviews, see Refs. 2 and 3). One of the important magnetic phenomena that can be tuned is the exchange bias<sup>4</sup> (EB), which may even be initialized by ion bombardment. The most known EB manifestation is the hysteresis loop shift away from the zero-field axis, called EB field,  $H_{EB}$ , whose magnitude is related to the exchange coupling between the ferromagnet (FM) and uncompensated interfacial spins in the antiferromagnet (AF).  $H_{EB}$  is usually decreased or even fully suppressed after irradiation.<sup>5</sup> For certain AF materials, however, an increase in  $H_{EB}$  could be observed. If during the ion bombardment an external magnetic field,  $H_{ib}$ , antiparallel to the original EB direction is applied, the sign of  $H_{EB}$  could be changed from negative to positive.<sup>6,7</sup> It has been shown<sup>8</sup> that, in the very particular case of the extremely soft NiFe, it is possible to set the EB of a NiFe/IrMn film in an arbitrary in-plane direction by ion bombardment in the presence of  $H_{ib}$ .

The very low anisotropy of NiFe permitted us to reorient both FM and AF easy axes in NiFe/NiO also by thermal postannealing,<sup>9</sup> which is the common method to initialize the EB. Annealing Co/NiO in a field applied in a direction different from the original EB direction, however, has led to off-aligned FM and AF easy axes owing to the relatively high Co anisotropy.<sup>9,10</sup> Ion-irradiation-induced change in the FM easy axis<sup>11</sup> is not expected to occur in thin films other than amorphous alloys or NiFe.<sup>8,12</sup> The latter is so soft that even its remnant magnetization along any in-plane direction (equivalent to an effective anisotropy axis set by a small magnetic field at the starting annealing temperature) acts on the AF during the zero-field postcooling and is able to initialize the EB in this direction.<sup>13</sup>

In the present letter we demonstrate that a complete reorientation of both FM and AF easy axes toward the  $H_{ib}$  direction of a system with relatively high FM anisotropy could be achieved in a controlled manner under appropriate ion bombardment, accompanied by  $H_{EB}$  enhancement.

IrMn/Cu/Co films with variable Cu layer thicknesses were recently investigated.<sup>14</sup> Among them, the sample for

which thermal postannealing did not modify  $H_{EB}$  was chosen for the present study in order to probe if the effect can be enhanced by ion bombardment. Furthermore, the latter could insert substitutions from the Cu interlayer in the volume part of the IrMn layer. Such a dilution of the AF strongly influenced  $H_{EB}$  in other EB systems.<sup>15,16</sup> The Ru(15 nm)/IrMn(15 nm)/Cu(0.75 nm)/Co(5 nm)/Ru(3 nm) film was deposited onto a Si(100) substrate by magnetron sputtering with a base pressure of  $5.0 \times 10^{-8}$  mbar, Ar pressure of  $2.5 \times 10^{-3}$  mbar for the deposition of Ru, Cu, and Co, and  $1.0 \times 10^{-2}$  mbar for IrMn. A conventional x-ray diffraction analysis identified (111)-texture of the IrMn and Co layers, promoted by the Ru buffer. The shift of the Cu(111) peak toward a smaller angle as compared to its bulk value suggests a Cu lattice expansion, which also follows the IrMn fcc structure. The magnetic characterization was done at room temperature via an alternating gradient-field magnetometer, with the magnetic field,  $H$ , applied in the plane of the films.

After determining the easy magnetization direction of the film, induced by the stray field from the magnetron during deposition, it was cut into nine pieces. No other treatment was done on the first one (as-made sample, S1) prior to its magnetic characterization; the second piece (henceforth, annealed sample, S2) was kept for 15 min at 200 °C in Ar atmosphere with a magnetic field of 1.6 kOe applied along the EB direction in the plane of the film. The other pieces were irradiated with He<sup>+</sup> ions of 40 keV energy in a chamber specially designed, with  $H_{ib}$  of approximately 5 kOe applied at 120° with respect to the original EB axis. The beam was perpendicular to the samples' plane, and the used fluences were between  $5 \times 10^{13}$  and  $5 \times 10^{15}$  ions/cm<sup>2</sup> with a constant current of 100 nA/cm<sup>2</sup>. The current was also varied between 50 and 300 nA/cm<sup>2</sup> for a constant fluence of  $1 \times 10^{14}$  ions/cm<sup>2</sup>. The fluences and currents used as well as the denominations of these samples are given in Fig. 1. The He<sup>+</sup> penetration depth, calculated using the SRIM 2008 code,<sup>17</sup> exceeds the sample's thickness. The correspondingly estimated displacements per atom for Cu and IrMn at their interface are 0.20 and 0.19 for S9, respectively, 0.04 and 0.04 for S8, and much smaller for the other samples. Finally, after being characterized magnetically, S1 was irradiated without  $H_{ib}$  using the same fluence and current of the irradiated sample that showed the highest  $H_{EB}$ , i.e., S8.

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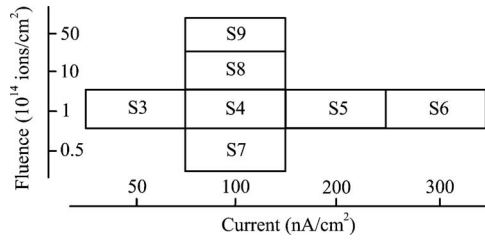


FIG. 1. Fluences and currents used and the respective denominations of the samples irradiated in magnetic field.

The magnetization curves for the as-made and the annealed films as well as for samples S6 and S8 are shown in Fig. 2, where the respective  $H_{EB}(\phi_H)$  variations are also plotted. Here,  $\phi_H=0$  corresponds to the magnetic field of the measurements  $\mathbf{H}$  parallel to the EB direction of the as-made sample. The  $H_{EB}(\phi_H)$  variations of the as-made and the annealed films practically coincide, while those of the samples irradiated with  $H_{ib}$  are shifted toward higher  $\phi_H$ , being the EB direction completely reoriented along the  $H_{ib}$  direction ( $\phi_H=120^\circ$ ) for samples S8 and S9. The maximum EB field  $H_{EB}^{\max}$ , i.e., that for  $\mathbf{H}$  applied along the EB direction, is  $\approx 30\%$  higher than that of the as-made and annealed films. All  $H_{EB}(\phi_H)$  curves show the typical pure cosine behavior for unidirectional anisotropy i.e., one minimum and one maximum. Also, the angular variations of the coercivity  $H_C$  (not shown) are very close to  $\cos 2\phi_H$ , with maxima coinciding with the extrema in  $H_{eb}(\phi_H)$ . These features indicate that the FM and AF easy axes coincide for all samples. Otherwise,  $H_{EB}(\phi_H)$  and  $H_C(\phi_H)$  should have much more complex shapes.<sup>9,18</sup>

Figure 3 shows  $H_{EB}^{\max}$  as a function of the current during the irradiation for constant fluence and  $H_{EB}^{\max}$  and  $H_C^{\max}$  as functions of the fluence for constant current. Since  $H_{EB}^{\max}$  is practically independent of the current, effects coming from a macroscopic heating of the samples during the irradiation or from the overlap of nuclear or electronic cascades in time can be ruled out. Therefore, the modifications of the coercivity as well as the EB field magnitude and direction studied here depend only on the ion fluence. They could be explained considering four mechanisms that may act during the ion bombardment, three increasing and one decreasing  $H_{EB}$ . The latter is AF/FM interface intermixing and interface defect creation by ion bombardment due to the nuclear energy loss by the impinging ions.<sup>5</sup> It works irrespectively whether

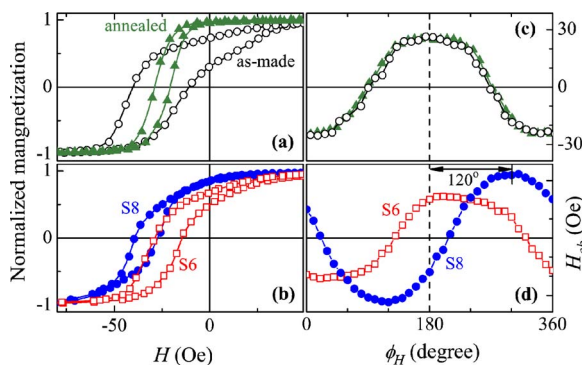


FIG. 2. (Color online) (a) EB direction hysteresis loops for the as-made and annealed films and (b) irradiated samples S6 and S8. The corresponding  $H_{EB}(\phi_H)$  variations are plotted in (c) and (d), respectively. The lines are guides to the eye.

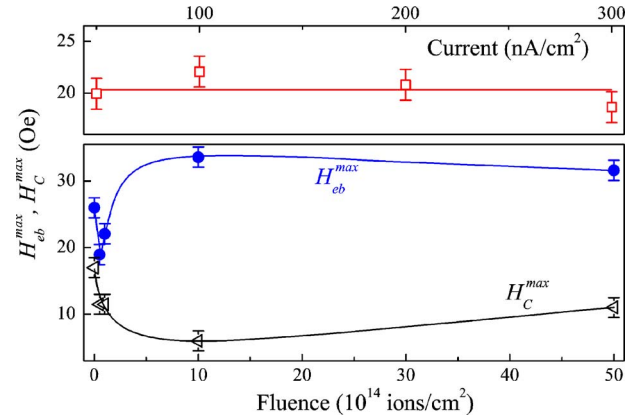


FIG. 3. (Color online)  $H_{EB}^{\max}$  vs the current during the irradiation for a constant fluence of  $1 \times 10^{14}$  ions/cm<sup>2</sup> (top);  $H_{EB}^{\max}$  and  $H_C^{\max}$  vs the fluence for a constant current of 100 nA/cm<sup>2</sup> (bottom). The lines are guides to the eye.

$H_{ib}$  is applied or not and depends only on the ion fluence. As a consequence, at high fluences it turns out to be more important and is responsible for the gradual decrease in  $H_{EB}^{\max}$  as a function of the fluence seen in Fig. 3.

The first of the mechanisms increasing  $H_{EB}$  due to ion irradiation is the local hyperthermal heating.<sup>6</sup> This mechanism is actually very similar to the common magnetic annealing and serves as a source of energy that leads to a rearrangement of the spins in order to lower the energy of the system. If, during the bombardment, sufficiently high  $H_{ib}$  is applied, the FM moments line up along its direction and, in their turn, align the adjacent interfacial AF spins. The latter, after irradiation, could at most deviate away toward their closest easy axes, which—for cubic anisotropy materials (normally the AFs used in EB systems are of this type)—are confined to a solid angle, the half-apex angle of which is  $\cos^{-1}(1/\sqrt{3}) \approx 55^\circ$ . Consequently, the direction of the effective AF moment is changed to one closer to that of  $H_{ib}$ , thus resulting in an enhancement of  $H_{EB}$  along the  $H_{ib}$  direction.

The second mechanism increasing  $H_{EB}$  is a dilution of the AF layer by nonmagnetic defects in its volume part away from the interface, supporting the formation of volume AF domains.<sup>15,16</sup> Here, the defects are Cu atoms inserted by means of ion bombardment from the spacer between Co and IrMn layers. Although  $H_{EB}$  decreases at the high fluence range due to intermixing and defect creation at the interface (see above), the decrease is not so abrupt as those found in the literature<sup>3</sup> due to the dilution of the IrMn layer by Cu atoms.

The third mechanism that could increase  $H_{EB}$  is defects in the bulk of the AF, leading either to a decrease in the AF anisotropy constant or to a reduction of the magnetically effective volume, both lowering the AF energy barrier. Therefore, some AF spins may thermally change their anisotropy axes, resulting in a slow increase in  $H_{EB}$  with time after the bombardment.<sup>8</sup> However, since none of our samples showed such an effect, this mechanism should be discarded here and the increase in  $H_{EB}$  up to a fluence of  $10^{15}$  ions/cm<sup>2</sup> should be attributed to the first two EB enhancing mechanisms.

The very initial decrease in  $H_{EB}^{\max}$  as a function of the fluence indicates that none of the last three mechanisms is important at very low fluences. This, together with the sharp initial decrease in  $H_C^{\max}$ , could be attributed to combined strain relaxation in the Co layer and interface roughening.<sup>19</sup>

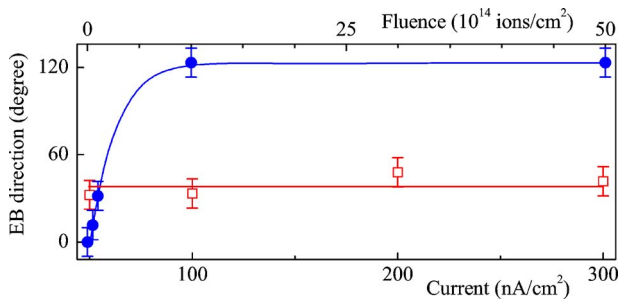


FIG. 4. (Color online) EB directions ( $0^\circ$  denotes that of the as-made sample) vs the current during the irradiation using a fluence of  $1 \times 10^{14}$  ions/cm<sup>2</sup> and vs the fluence using a current of 100 nA/cm<sup>2</sup>. The lines are guides to the eye.

The  $H_C^{\max}$  variation with the fluence (for constant current) for fluences higher than  $1 \times 10^{14}$  ions/cm<sup>2</sup> (Fig. 3) shows an initial decrease followed by a gradual increase for fluences higher than  $10^{15}$  ions/cm<sup>2</sup> and seems to be quite the opposite of the respective  $H_{EB}^{\max}$  variation. This is in excellent agreement with the role of the different in stability spins at the AF part of the interface.<sup>20,21</sup> AF spins with very small relaxation times behave superparamagnetically and do not contribute to either  $H_{EB}$  or  $H_C$ . Spins with intermediate relaxation times give a raise to  $H_C$  and not to  $H_{EB}$ , while those with relaxation time greater than the measurement one contribute to the EB only. When  $H_{EB}$  is enhanced by increasing the number of interfacial AF spins of the latter type at the expense of those with intermediate relaxation times,  $H_C$  is reduced and vice versa (see Fig. 3). This clearly points out that  $H_{EB}$  and  $H_C$  vary mainly due to interface modifications. Part of the gradual increase of  $H_C$  at high fluences could also be attributed to the dilution of the AF layer.<sup>16</sup>

The EB directions as functions of the current (at constant fluence) and fluence (at constant current) of the irradiation are given in Fig. 4, where  $0^\circ$  denotes the EB direction of the as-made sample. While an approximately  $40^\circ$  deviation was obtained for all currents at a constant fluence, a gradual deviation of the EB direction with the fluence is observed, reaching a complete reorientation toward the  $H_{ib}$  direction of  $120^\circ$  for fluences higher than  $10^{15}$  ions/cm<sup>2</sup>.

The film irradiated without  $H_{ib}$  showed no modification of its EB direction, the same  $H_C^{\max}$  as that of the corresponding field-irradiated sample, and  $H_{EB}^{\max}$  8% lower than that of the as-made sample. The latter and the significant  $H_{EB}^{\max}$  enhancement when irradiating with  $H_{ib}$  denote the importance of the local hyperthermal heating, which increases  $H_{EB}$  only when  $H_{ib}$  is applied. Otherwise, the intermixing and defect creation at the AF/FM interface decrease  $H_{EB}$ , though not very pronounced for our samples due to the competition with the other mechanism that increases the EB field, i.e., the dilution of the AF layer by nonmagnetic defects in its volume part.

The independence of  $H_C^{\max}$  on applying  $H_{ib}$  or not for high fluence points out that no phase transitions occur in the FM layer. It also indicates that the easy-axis reorientation of our FM with non-negligible anisotropy when  $H_{ib}$  is applied is apparently due to locally transferred energy of the incoming He<sup>+</sup> ions through electronic energy losses or phonon excitation. Since Co is a material with cubic magnetocrystalline anisotropy like IrMn, this mechanism acts in the FM in a manner very similar to that in the AF part of the interface discussed above. Such easy-axis reorientation of the FM is

not observed after field cooling, given that the energy received by thermal heating during annealing is much lower than the energy deposited via ion bombardment.<sup>22</sup>

It is worth noting that Ehresmann *et al.*,<sup>8</sup> after setting the EB by ion bombardment with  $H_{ib}$  applied along an arbitrary in-plane direction in a bilayer with very low FM anisotropy, measured smaller values of  $H_{EB}$  for any other  $H_{ib}$  direction. Although irradiating a system with rather higher FM anisotropy, we succeeded, along with the complete reorientation of both FM and AF easy axes, to significantly enhance  $H_{EB}$ . There are several possible reasons for these improvements, among which are the use of higher ion fluences, the stronger and rather uniform  $H_{ib}$ , as well as the dilution of the AF volume part by nonmagnetic defects.

In summary, we demonstrated that under appropriate conditions a complete reorientation of the easy axes of both FM and AF toward that of the magnetic field applied during the He<sup>+</sup> ion irradiation can be achieved on an exchange-coupled system with high magnetic anisotropy of the FM, accompanied with a significant exchange-bias field enhancement.

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