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# Ion irradiation effects on the exchange bias in IrMn/Co films

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The present work reports on the influence of ion irradiation in exchange-coupled bilayers. Magnetron-sputtered IrMn<sub>4</sub>/Co films were irradiated with 40 keV He<sup>+</sup> ions and the dependence of their magnetic properties was studied as function of ion fluence and current used during the irradiations. The effects of ion damage and electronic excitation were also studied through additional irradiations with H<sup>+</sup> and Ne<sup>+</sup> ions. The results show a clear dependence of the exchange-bias field on the defects caused by the ion bombardment. No correlations with other irradiation effects were observed. © 2011 American Institute of Physics. [doi:10.1063/1.3532044]

## I. INTRODUCTION

The magnetic exchange bias (EB) effect refers to the coupling between a ferromagnet (FM) and an antiferromagnet (AF) when placed in atomic contact and it has been extensively studied since its discovery by Meiklejohn and Bean<sup>1</sup> more than 50 years ago. In a system that presents the effect, the FM magnetization is pinned by that of the AF at their interface which is manifested as a shift in the hysteresis loop away from the zero field-axis, i.e., the exchange-bias field  $H_{EB}$ . EB can be induced by several ways: during the growth of the materials in the presence of a magnetic field, through magnetic annealing and also via ion bombardment, also with magnetic field,  $H_{ib}$ . The last type of postdeposition treatment has been extensively studied since the pioneer work of Chappert *et al.*<sup>2</sup> In particular, low-energy ion irradiation is capable to reduce and even suppress the EB in certain systems.<sup>3</sup> Otherwise, with  $H_{ib}$  applied during the ion bombardment, an enhancement of the effect is obtained.<sup>4-7</sup>

Recently, an increase in the EB effect through He<sup>+</sup> irradiation accompanied with a complete reorientation of the EB direction with the fluence in an IrMn/Cu/Co system has been reported.<sup>7</sup> There are four mechanisms that are usually pointed out as responsible for EB modifications induced by ion irradiation. (i) Interfacial intermixing and defect creation due nuclear energy loss of the impinging ions has been related to EB reduction.<sup>3,8</sup> (ii) Modifications on the bulk of the AF could lead to an enhancement of the EB; in this case, the AF barrier could be lowered if the effective magnetic volume of the AF grains or the AF anisotropy constant is decreased.<sup>9</sup> (iii) Hyperthermal heating triggered by electronic excitations has also been related to the EB increase since it is the electronic stopping power that predominates for such light ions' energy. (iv) Defects created in the bulk AF due to insertion of nonmagnetic atoms could support the formation of domain walls leading to an increase in  $H_{EB}$ .<sup>10-12</sup>

In this work, we investigate the effects caused by ion irradiation on the EB of an IrMn/Co system. Results on samples without a spacer between the IrMn and Co layers are compared with those previously obtained on films with Cu

spacer.<sup>7</sup> The absence of a nonmagnetic spacer led to a great variation in  $H_{EB}$  in samples annealed and irradiated as compared with the as-made one,<sup>13</sup> making possible a more accurate investigation of the irradiation effects. Here, the effects of the ion fluence and current on  $H_{EB}$  were investigated independently, and the results were compared with those obtained on the sample with Cu spacer. Irradiations with different ions were also used to study the influence of the production of defects, phonons and electronic excitation on the magnetic properties of the system.

## II. EXPERIMENTAL

Ru(15 nm)/IrMn<sub>4</sub>(15 nm)/Co(5 nm)/Ru(3 nm) films were deposited by magnetron sputtering with base pressure  $5.0 \times 10^{-8}$  mbar, Ar pressure  $2.5 \times 10^{-3}$  mbar for the deposition of Ru and Co, and  $1.0 \times 10^{-2}$  mbar for IrMn, onto Si(100) substrate. Samples with the same nominal composition with additional 0.75 nm thick Cu spacer layer between the IrMn and Co ones, produced at the same conditions, have been studied in a previous work of ours.<sup>7</sup> The magnetic characterization was done at room temperature by using an alternating gradient-field magnetometer with the magnetic field,  $H$ , applied in the plane of the films.

After the deposition, the EB direction (induced by the stray field from the magnetron during the deposition) of each film was determined from the magnetic measurements and then the samples were cut in pieces. The magnetic annealing was made at 200 °C during 15 min in an Ar atmosphere with an annealing magnetic field,  $H_{ann}$ , of 1.6 kOe applied at 120° away from the EB direction. This particular angle was used in order to compare the present results with those reported in the previous work of ours.<sup>7</sup> There, the orientation of the in-plane field applied upon postdeposition treatments has been arbitrarily chosen, the only restraint being that this must not coincide with the original EB direction. The ion bombardments were made in a 500 kV HVEE linear accelerator with a specially designed chamber providing  $H_{ib}=5.5$  kOe. The beam incidence direction was perpendicular to the film's plane for all samples and, in order to compare the variation

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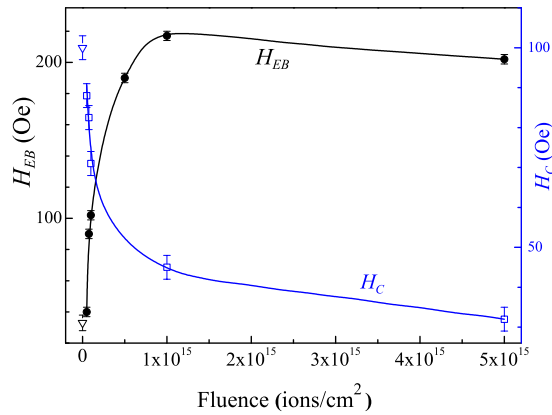


FIG. 1. (Color online)  $H_{EB}$  (full circles) and  $H_C$  (empty squares) vs the He<sup>+</sup> fluence (the current during the irradiation was 100 nA/cm<sup>2</sup>). Data for the as-made sample are represented by the empty triangles. The lines are guides to the eyes.

in the magnetic parameters with those previously obtained on IrMn/Cu/Co,  $H_{ib}$  was applied at 120° away from the EB direction.

The as-made samples were irradiated with 40 keV He<sup>+</sup> ions, where the fluences used were between  $5.0 \times 10^{13}$  and  $5.0 \times 10^{15}$  ions/cm<sup>2</sup> for a current of 100 nA/cm<sup>2</sup>, and the used currents were between 50 and 600 nA/cm<sup>2</sup> for a constant fluence of  $1.0 \times 10^{14}$  ions/cm<sup>2</sup>. Identical treatments were performed on samples with Cu spacer in the previous work of ours.<sup>7</sup>

### III. RESULTS AND DISCUSSION

Figure 1 shows the variations in  $H_{EB}$  and  $H_C$ , derived from the hysteresis loops traced for  $H$  applied along the EB direction, versus the fluence used in the irradiations. A gradual increase in  $H_{EB}$  up to a fluence of  $1.0 \times 10^{15}$  ions/cm<sup>2</sup> is seen as well as a reduction in  $H_C$ . The same tendency was observed for samples with Cu spacer;<sup>7</sup> however,  $H_{EB}$  of the sample without a spacer always increases while the sample with Cu spacer shows a reduction in  $H_{EB}$  for low fluences, followed by an increase with the fluence applied. The treatment with the highest fluence has led to a certain  $H_{EB}$  reduction. The same effect has been also observed for the case with Cu spacer and should be again attributed to the AF/FM interface intermixing and interface defect creation due to the nuclear energy loss by the impinging ions caused by the ion bombardment.<sup>3</sup>

A reorientation of the EB direction along  $H_{ib}$ , i.e., 120°, is clearly seen in Fig. 2. This reorientation is very abrupt for the film with no spacer (a change from 0° for a  $5.0 \times 10^{13}$  ions/cm<sup>2</sup> fluence to ≈120° for a fluence of  $7.5 \times 10^{13}$  ions/cm<sup>2</sup>) while it is continuous for the sample with Cu spacer.<sup>7</sup> Although the EB direction of the annealed sample was also reoriented along the direction of  $H_{ann}$ , the increase in  $H_{EB}$  is much weaker than that of the irradiated samples. Indeed,  $H_{EB}$  of the film irradiated with a fluence of  $1.0 \times 10^{15}$  ions/cm<sup>2</sup> (which is the sample that showed the largest increase in the EB field) is approximately 50% higher than that of the annealed sample.

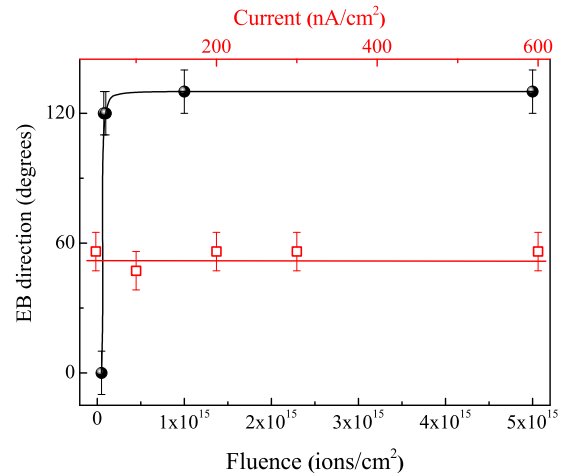


FIG. 2. (Color online) Shift in the EB direction as a function of the fluence for constant current (full circles) and current at a fixed fluence (empty squares) in the He<sup>+</sup> irradiated samples. The lines are guides to the eyes.

The influence of the current used during the irradiations can be seen in Figs. 2 and 3. One notes in Fig. 2 that the direction of  $H_{EB}$  was not affected by the current. In fact, for this fluence applied ( $1.0 \times 10^{14}$  ions/cm<sup>2</sup>), the EB direction is already set along  $H_{ib}$ , so one should not expect any further reorientation due to the current. The samples with Cu spacer showed no variation in the EB direction with the current where, however, the EB direction's reorientation has not been accomplished for a fluence of  $1.0 \times 10^{14}$  ions/cm<sup>2</sup>. In the present work,  $H_{EB}$  is affected by the current used during the irradiations as seen in Fig. 3, differently from the EB orientation. A gradual increase in  $H_{EB}$  is observed for fluences up to 300 nA/cm<sup>2</sup>, where  $H_{EB}$  shows a maximum.

Dilution of the AF has been pointed out previously<sup>7</sup> as possibly responsible for the enhancement observed in  $H_{EB}$  of IrMn/Cu/Co systems. This effect could be now better evaluated by comparing the results of samples with and without Cu spacer.  $H_{EB}$  in irradiated samples at fluences of  $1.0 \times 10^{15}$  ions/cm<sup>2</sup> is ≈30% larger than those of the as-made and annealed samples with Cu spacer and about 600% and 50% higher for the as-made and annealed samples without spacer, respectively. Hence, the effects of diluting the AF layer by insertion of Cu atoms (via recoil implantation) seem

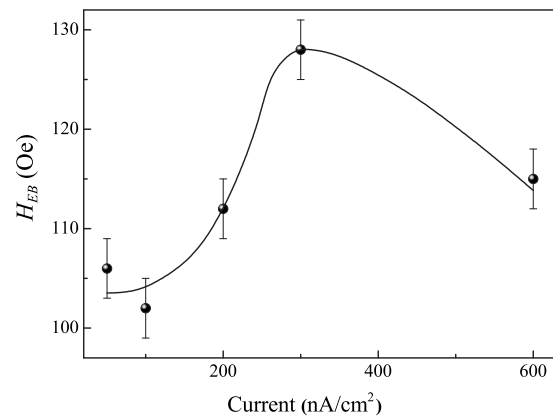


FIG. 3.  $H_{EB}^{max}$  as function of the current applied during the irradiations at a fluence of  $1.0 \times 10^{14}$  ions/cm<sup>2</sup>. The line is a guide to the eyes.

to have no importance in this system since the increase in  $H_{EB}$  of the IrMn/Co sample is larger than that of the IrMn/Cu/Co film. In order to produce an increase in  $H_{EB}$ , Cu atoms should be inserted in the volume part of the AF away of the interface.<sup>10</sup> However, SRIM simulations<sup>14</sup> give Cu atoms displacements of at most three atomic layers.

Although interfacial intermixing may occur in samples with and without Cu spacer, an initial drop of  $H_{EB}$  with the fluence is only observed for samples with a Cu spacer.<sup>7</sup> The reorientation of the EB direction seen in Fig. 2 is gradual for samples with spacer and abrupt for the films with no Cu layer. Since one would expect one and the same AF structure for all samples provided that the IrMn depositions were made under exactly the same conditions, overcoming of the AF energy barriers for low fluences, if any, must occur for both types of samples. The gradual reorientation of the EB direction of the samples with spacer layer could indicate that the number of pinning sites changes according to the fluence and saturates at  $\approx 1.0 \times 10^{15}$  ions/cm<sup>2</sup>. Thus, the initial reduction in  $H_{EB}$  for samples that contain nonmagnetic spacer could be explained in terms of interfacial structure modifications that could decrease the FM/AF coupling. This effect may also occur in samples without spacer; however, it is practically undetectable since the very significant increase in  $H_{EB}$  due to the initial improvement of the uncompensated AF interface magnetic structure masks the above discussed decrease due to structural changes at the interface.

In order to investigate the physical processes that occur during ion bombardment, additional irradiations with 40 keV H<sup>+</sup> and 200 keV Ne<sup>+</sup> ions were performed of samples without Cu spacer, where the enhancement of EB is much larger. The used current was 100 nA/cm<sup>2</sup> for all samples. The results of 40 keV He<sup>+</sup> irradiations (corresponding to the series of samples without spacer, for constant current and varying fluence) were also used for comparison. Full TRIM cascade<sup>15</sup> simulations were performed to obtain the production of phonons and interstitial atoms for all irradiations. For electronic excitation, we simply used the electronic stopping power ( $S_E$ ) times the fluence, where the values of  $S_E$  were obtained from the SRIM code.<sup>14</sup> All quantities were calculated for the AF material. Note that although the irradiation energies used here are not at the high energy regime, they lie below the electronic stopping maximum. Nevertheless, the ratio between the electronic and nuclear stopping powers, in electronvolt per angstrom units each, is about 20 for the case of He<sup>+</sup> (17.66/0.82), almost 200 for H<sup>+</sup> (13.38/0.073), and 3 (48.08/17.42) for the case of Ne<sup>+</sup>.

Figure 4 shows  $H_{EB}/H_{EB}^{max}$  and  $H_C/H_C^{max}$  ( $H_{EB}^{max}$  and  $H_C^{max}$  are the corresponding maximum EB and coercive fields measured) versus the normalized electronic excitation, production of phonons, and interstitial atoms. In the top and middle panels of this figure, one cannot find definite trends for the variation in  $H_{EB}$  (and  $H_C$ ) as a function of the electronic excitation or phonons, i.e., no simple correlation can be established from these data. Nevertheless,  $H_{EB}/H_{EB}^{max}$  as a function of the normalized interstitial atoms' number, see the bottom panel of Fig. 4, increases steadily up to 0.2 (corresponding to 0.06 displacements per atoms) and then starts to decrease;  $H_C/H_C^{max}$  shows a general trend of reduc-

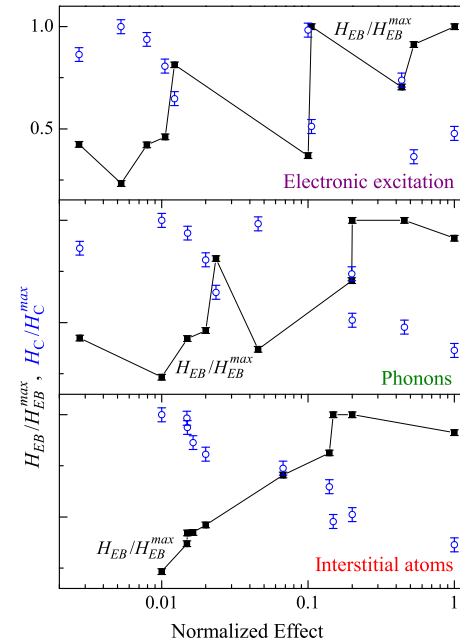


FIG. 4. (Color online)  $H_{EB}/H_{EB}^{max}$  (full squares) and  $H_C/H_C^{max}$  (empty circles) as function of the electronic excitation (top), phonons (middle), and interstitial atoms (bottom), normalized by the respective maximum values. The lines are guides to the eyes.

tion with the increase in the number of interstitial atoms. One notes that these variations are qualitatively identical to those of Fig. 1.

Therefore, point defects (interstitial and vacancies) produced by the ion bombardment in the bulk AF lead to an increase in  $H_{EB}$  independently on other ion irradiation effects, as long as the sample is not too damaged. This could be explained considering the work of Vallejo-Fernandez *et al.*<sup>16</sup> where the AF grain-size distribution of their IrMn/CoFe samples obeys a lognormal function and three regimes of sizes can be distinguished. This is schematically shown in Fig. 5, illustrating two possible AF energy barrier distributions to the reversal, before and after ion irradiation: (i) grains with volume,  $V$ , smaller than a critical volume,  $V_C$ , below which thermal instabilities overcome the energy barrier providing no contribution to EB; (ii) grains with volumes larger than a certain setting volume,  $V_{set}$ , above which ordinary procedures are incapable to reorient the AF moments with the adjacent FM ones, and (iii) grains with volumes

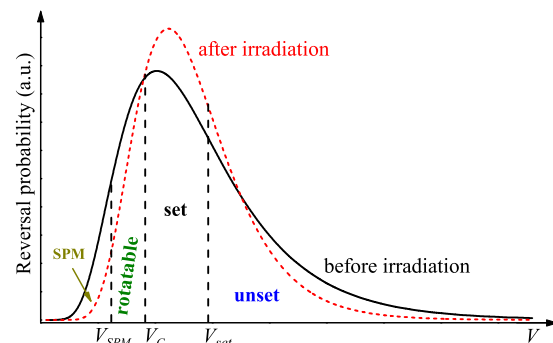


FIG. 5. (Color online) Schematic of energy barrier distributions to the reversal before and after ion irradiation.

between  $V_C$  and  $V_{set}$ , subject to reorientation along the direction of the postdeposition treatment field ( $H_{ann}$  or  $H_{ib}$ ). In such a case,  $H_{EB}$  is proportional to the integral of the grain size distribution between  $V_C$  and  $V_{set}$ . In the present work, we further divided the  $V < V_C$  region into two subregions by considering even smaller, superparamagnetic (SPM) grains, which are thermally unstable during the measurement and do not contribute to either  $H_{EB}$  or  $H_C$ .

Defects created by ion irradiation could modify the AF grain-size distribution, as illustrated by the dashed curve in Fig. 5. The increase in  $H_{EB}$  with the number of interstitial atoms and vacancies may indicate that the irradiation transforms grains with  $V$  greater than  $V_{set}$  to grains with  $V_C < V < V_{set}$  immediately after finishing the collision cascades. Postcollision effects such a relaxation of defects produced by the bombardment could also transform grains with  $V < V_C$  to ones with  $V_C < V < V_{set}$  and, simultaneously, grains with  $V > V_C$  might be transformed to ones with  $V_{SPM} < V < V_C$ . Since the coercivity decreases monotonously with the fluence and the interstitial atoms' number, it seems that the augment rate of the number of SPM grains is higher than that of grains with  $V_{SPM} < V < V_C$ , thus explaining the gradual decrease in  $H_C$ . An increase in the number of grains with  $V < V_C$ , expected for higher number of defects, explains the reduction in  $H_{EB}$  at very high fluences as well.

The change in  $H_{EB}$  with the ion current (observed for the sample without spacer) is also consistent with the above scenario. The overlap of nonrelaxed defects produced by collision cascades would increase the number of defects thus enhancing  $H_{EB}$ . On the other hand, dynamical annealing during high-flux irradiation<sup>17</sup> reduces the number of defects created explaining the decrease in  $H_{EB}$  observed for a current of 600 nA/cm<sup>2</sup> (see Fig. 3). A current dependence was not detected in the sample with Cu spacer probably because the variation is in the range of the measurement errors.

In conclusion, we demonstrate that the increase in  $H_{EB}$  and the reorientation of the EB direction through ion irradiation is related to defect creation in the AF material. This

conclusion is supported by data of irradiations with H, He, and Ne ions, at different fluences and currents.

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