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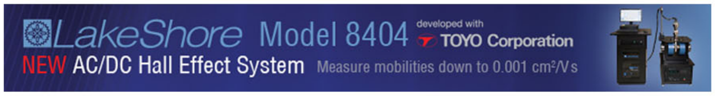

Exchange bias in a ferromagnet/antiferromagnet system with $T_C \ll T_N$

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This paper reports experimental results obtained on an unconventional exchange bias (EB) system where the ferromagnetic layer, $Ni_{0.75}Cu_{0.25}$, has lower ordering temperature (T_C) than that (T_N) of the antiferromagnetic one, NiO, with emphasis on modifying EB through either magnetic annealing or light-ion irradiation. Samples were cooled from temperatures higher than T_N or in between T_C and T_N to room temperature with magnetic field applied in different in-plane directions. Upon ion irradiation, magnetic fields, parallel or antiparallel to the orientation of the field present during the films deposition, were applied to explore different effects on EB. We found that the EB direction can be completely reversed by means of either annealing or ion bombardment; however, both postdeposition treatments provide very little variation of the EB field value over that produced during the film's growth. The importance of the annealing field strength was also discussed. The results were interpreted based on a mechanism which assumes that the interfacial moments adjacent to the antiferromagnetic layer are responsible for establishing the exchange biasing in the paramagnetic state. © *2011 American Institute of Physics*. [doi:10.1063/1.3572258]

I. INTRODUCTION

In a magnetization hysteresis loop measurement, the exchange bias (EB) phenomenon manifests itself as a shift of the loop to either the negative[1–3] or positive[4,5] direction of the magnetic field axis. This effect is usually observed in systems composed of a ferromagnet (FM) exchange-coupled to an antiferromagnet (AF), where the Curie temperature (T_C) of the FM is higher than the Néel temperature (T_N) of the AF. The well-established $T_C > T_N$ relation comes from the supposition that when initializing the effect via, e.g., magnetic-field cooling through T_N, the FM should be magnetically ordered as the magnetic order and exchange coupling of the AF are being established. Nevertheless, new and interesting effects and features related to EB in systems where T_N is higher or close to T_C were observed by Wu and Chien[6] in 1998 in α-$(Fe_{0.1}Ni_{0.9})_{80}B_{20}$/CoO and later in α-$Fe_4Ni_{76}B_{20}$/CoO,[7] FeMn/$Ni_{0.55}Cu_{0.45}$,[8] Cr/Gd/Cr,[9] as well as in AF nanoparticles with ferrimagnetic shells.[10] Despite these motivating results and the potential applicability of such systems in electronic devices, very few works have been reported since the publication of the pioneering paper,[6] most likely because of the difficulty in initializing (setting) and/or manipulating the EB effect.

In a conventional EB system with $T_C > T_N$, the EB direction can be set, and the effect established, using several distinct procedures: (i) by applying a sufficiently strong magnetic field during the sample's production; (ii) through appropriate postannealing, or (iii) via ion bombardment,[11–13] both in the presence of a magnetic field; (iv) applying sufficiently large magnetic field at a temperature lower than T_N may also set the effect along the field direction;[14,15] (v) even the FM remnant magnetization, acquired prior to cooling, is able to set the effect.[16] However, at least in theory, when $T_C < T_N$ none of these methods seems to be efficient to initialize the EB due to, as already mentioned, lack of ferromagnetic order of the FM, which is in the paramagnetic (PM) state when the AF order is established. Blamire *et al*.[8] have induced unidirectional anisotropy in their FeMn/$Ni_{0.55}Cu_{0.45}$ system with $T_C < T_N$ by growing FeMn on another magnetically saturated FM layer with very high T_C value. These authors have also found that the FM with a low T_C value has only a negligible influence on the AF spin state for $T > T_C$.

The present work reports experimental results obtained on an AF/FM system with $T_C \ll T_N$, namely a magnetron-sputtered (in a presence of magnetic field) bilayer of NiO/$Ni_{0.75}Cu_{0.25}$, already exhibiting EB in the as-deposited state. Annealing with a magnetic field of different strengths, applied in distinct in-plane directions and starting from different temperatures, as well as ion irradiation, also in a presence of an in-plane magnetic field, were employed to set/modify the EB effect. It was found that the EB direction can be completely reversed by means of any of these postdeposition treatments. The results reported here seem to support the mechanism which assumes that the interfacial moments adjacent to the antiferromagnetic layer are likely to be responsible for the exchange biasing in the paramagnetic state.[7]

II. EXPERIMENT

NiO(50 nm)/$Ni_{0.75}Cu_{0.25}$ (30 nm) film was produced by dc and rf magnetron sputtering (using AJA ORION 8 UHV Sputtering System) from a Ni and Cu targets in a 2 mTorr Ar atmosphere and from a NiO target in a 3 mTorr Ar atmosphere. Au was employed as a 10-nm-thick buffer layer in order to ensure that the NiO formed the (111) texture required for bias,[17] and as a 3-nm-thick cap layer. The film was deposited on a rotating glass substrate at room temperature (RT) under a stationary in-plane magnetic field with a magnitude of 130 Oe provided by a permanent magnet coupled to the

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substrate holder. The base pressure prior to deposition was less than 1×10^{-7} mbar and during deposition the pressure was 4×10^{-2} mbar. The layers' thicknesses were controlled by measuring the deposition rates using a calibrated quartz microbalance. The deposition rates of Ni and Cu were carefully chosen in order to produce a $Ni_{0.75}Cu_{0.25}$ alloy by codeposition of the elements. The T_C value of NiCu alloys can be tuned within a wide range of values by changing the relative composition of Ni and Cu. In this work, a Ni/Cu ratio of 3/1 was employed to obtain a NiCu layer with $T_C \ll T_N$, where the aim was to obtain a FM with a T_C between RT and 100 °C, which is the high temperature limit of our magnetization measurement facilities.

A thick (30 nm) NiCu layer was used in the present work in order to ensure a sufficiently strong magnetic response when measuring in the vicinity of T_C. The as-deposited sample was characterized structurally and magnetically prior to ion bombardment or annealing. The crystallographic structure of the film was studied by means of conventional x-ray diffractometry (XRD) using a Philips X'Pert MRD machine employing Cu $K\alpha$ radiation. The T_C value of the NiCu layer was verified by analyzing the magnetization temperature dependence carried out by superconducting quantum interference device (SQUID). The RT magnetic measurements were performed using an alternating gradient-field magnetometer with measuring magnetic field, H, applied in the plane of the films. Special care has been taken to ensure that the value of the maximum field is sufficient for effective magnetic saturation, thus avoiding possible overestimation of the EB effect due to minor loop effects.[18–20] Also, as the state of order in the AF layer could change during a measurement making some of the EB parameters nonreproducible,[21] prior to any definitive magnetization curve trace a series of eight sequential magnetization curves has been acquired in order to ensure the reproducibility of the measurements. None of the samples showed appreciable training effects given that these curves practically coincide.

After the structural and magnetic characterization of the as-made film, it was cut into pieces that were divided into two groups. The first group of samples was submitted to annealing at 110, 150, or 300 °C for 5 min in Ar atmosphere in a magnetic field, $\mathbf{H}_{\mathbf{AN}}$, of 4 kOe applied along one of three different in-plane directions, namely that parallel to the easy axis induced by the magnetron field (given by the angle $\varphi_{AN} = 0°$), the orthogonal ($\varphi_{AN} = 90°$), and the antiparallel ($\varphi_{AN} = 180°$) directions, respectively. Annealing at the same conditions but in zero field was also performed.

The samples of the second group were subjected to 50 keV He^+ irradiation at a current density of 100 nA/cm^2 at RT. Fluences of 5×10^{13}, 5×10^{14}, 1×10^{15}, and 5×10^{15} ions/cm^2 were used. During the ion bombardment, an external magnetic field, $\mathbf{H}_{\mathbf{IB}}$, with a magnitude of 5.5 kOe, was applied parallel ($\varphi_{IB} = 0°$) or antiparallel $\varphi_{IB} = 180°$) to the original EB direction.

III. RESULTS

A. X-ray diffraction

The XRD spectrum, obtained at RT on the as-deposited $NiO/Ni_{0.75}Cu_{0.25}$ film, given in Fig. 1, shows that the deposited layers are polycrystalline. Apparently, both NiO and NiCu layers exhibit (111) texture with comparable peak intensities; note that the (111) texture of the NiO layer seems to be essential for establishing exchange biasing.[17]

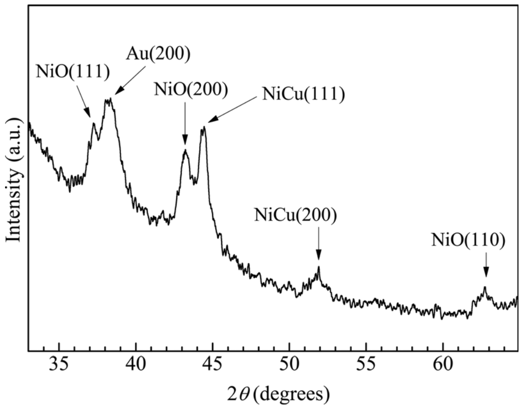

FIG. 1. X-ray diffraction spectrum of the as-deposited $NiO/Ni_{0.75}Cu_{0.25}$ film. The contribution coming from the glass substrate has not been subtracted.

B. SQUID measurements on the as-deposited film

The left-hand panel of Fig. 2 gives the zero-field-cooled (ZFC) and field-cooled (FC), under the in-plane field of 100 Oe, thermomagnetic curves of the as-deposited film. From Fig. 2, the estimated T_C value of 67(±2) °C is in a very good agreement with the literature data[22] for this Ni/Cu ratio. The respective T_N value of the 50-nm-thick NiO layer is certainly higher than 182 °C, i.e., the blocking temperature, T_B, value of a much thinner NiO film,[23] which is much higher than the T_C value of the here-studied FM layer. Consequently, the NiO/NiCu bilayer studied here is characterized by $T_C \ll T_N$, like the few systems previously reported.[6–10]

Hysteresis loops, traced on the as-deposited film at temperatures higher than RT and lower (57 °C), approximately equal to (67 °C), or higher (82 and 92 °C) than T_C of the FM

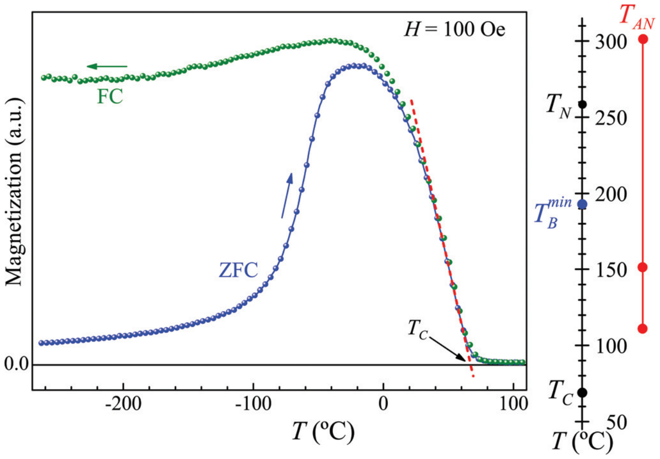

FIG. 2. (Color online). (Left) ZFC and FC curves of the as-deposited $NiO/Ni_{0.75}Cu_{0.25}$ film measured in a constant magnetic field of 100 Oe applied along the original EB direction, i.e., $\varphi_H = 0°$. The lines are only guides to the eyes. (Right) The annealing temperatures used along with T_C, T_N, and the value for the blocking temperature, referred to here as $T_B^{\min}$, estimated for a 4-nm-thick NiO layer.

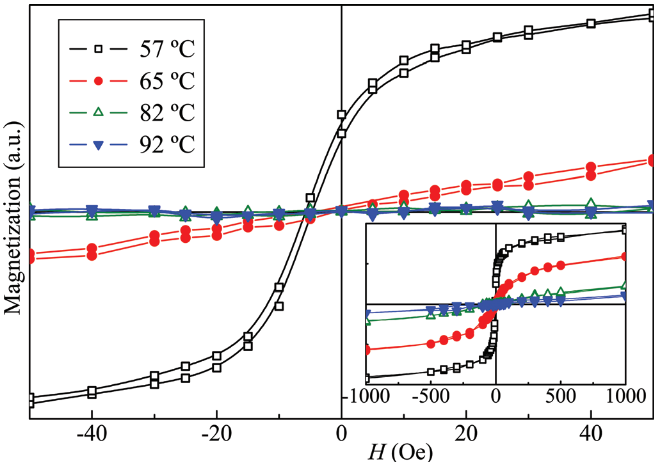

FIG. 3. (Color online). Hysteresis loops traced via SQUID magnetometer at different temperatures on as-deposited NiO/$Ni_{0.75}Cu_{0.25}$. The lines are guides to the eyes.

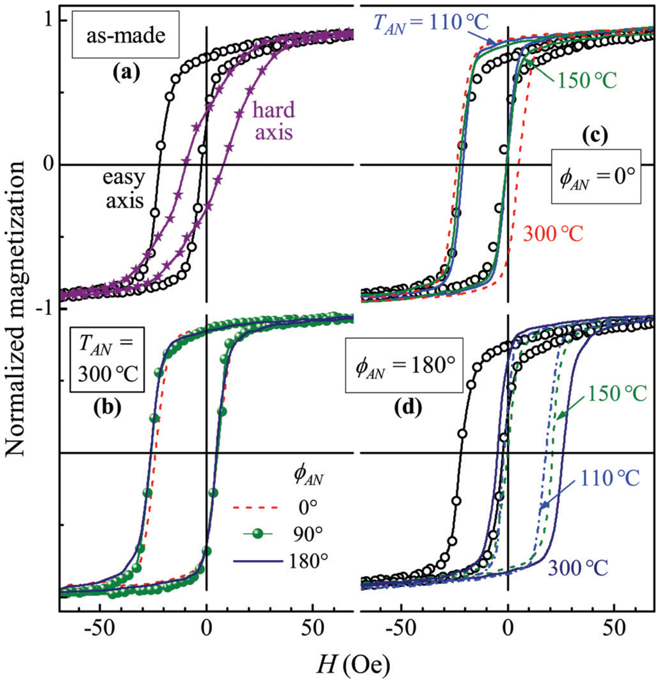

FIG. 4. (Color online). Easy-axis hysteresis loops traced on the as-made (open symbols) and annealed at different temperatures NiO/$Ni_{0.75}Cu_{0.25}$ samples; In (a), the hard-axis curve (stars) is also given. (b) Curves traced after annealing at 300 °C with $\varphi_{AN} = 0°$, 90°, or 180°; (c) Loops measured after annealing at different T_{AN} with $\mathbf{H_{AN}}$ parallel ($\varphi_{AN} = 0°$) to the original EB direction. Note that each annealing field orientation was converted into the respective new EB direction. (d) Loops measured on samples annealed at different T_{AN} with $\varphi_{AN} = 180°$. For comparison purposes, the loop of the as-made sample is also plotted in (c) and (d).

are shown in Fig. 3. EB was observed at $T \le T_C$ in the NiO/$Ni_{0.75}Cu_{0.25}$ bilayer, being the EB field $H_{EB} \le 5.5$ and 3.0 Oe at 57 and 67 °C, respectively. The characteristics of the loops measured at $T_C < T < T_N$ indicate that the NiCu layer is in the paramagnetic state, i.e., zero coercivity and magnetization at $H = 0$ and thus no field shift, differently from previous results on films of a $Fe_4Ni_{76}B_{20}$,[7] where a nonzero EB field has been measured at $T > T_C$. The other difference is that the film, sputtered in a presence of an in-plane magnetic field, shows EB in the as-deposited state.

C. Magnetic annealing

Easy and hard axis magnetization curves traced on the as-made film with **H** parallel to the original EB direction (i.e., $\varphi_H = 0°$) induced during deposition are plotted in Fig. 4(a). The easy-axis loop presents H_{EB}, defined as $H_{EB} = |H_{C2} + H_{C1}|/2$ of ≈ 10 Oe, where H_{C1} and H_{C2} are the coercive fields of the descending and ascending branches of a hysteresis loop, respectively, and $H_C \approx 10$ Oe $[H_C = (H_{C2} + H_{C1})/2]$. The value of H_C of the (unbiased) hard-axis loop is slightly smaller, ≈8.5 Oe.

Conventional magnetic annealing was employed in order to check if the unidirectional anisotropy can be optimized despite the lack of ferromagnetic ordering of the NiCu layer at the starting temperature of the annealing. Figures 4(b)–4(d) give easy-axis hysteresis loops traced on samples annealed at different temperatures with $\mathbf{H_{AN}}$ applied along distinct in-plane directions. In Figs. 4(c) and 4(d), curves corresponding to different T_{AN} are plotted where, for comparison purposes, the loop of the as-made film is also shown. Note that, as displayed in the right-hand panel of Fig. 2, the maximum T_{AN} of 300 °C used here is higher than T_N, and the two other annealing temperatures, although higher than T_C, are lower than the blocking temperature estimated for a more thinner NiO layer.[23]

The influence of the orientation of $\mathbf{H_{AN}}$ for $T_{AN} = 300$ °C can be seen in panel (b) where loops measured after annealing with $\varphi_{AN} = 0°$, 90°, or 180° are shown. Note that each $\mathbf{H_{AN}}$ orientation has been converted into a new EB direction, and the three loops are practically the same.

When $\mathbf{H_{AN}}$ is applied along the EB direction already set during the film's growth, the value of T_{AN} does not seem to influence the descending branch of the loops measured at RT, as seen in Fig. 4(c). The samples annealed at $T_{AN} = 110$ and 150 °C present H_C and H_{EB} identical with those of the as-made film being the shape of their magnetization curves very similar as well. Thus, as modification of H_{C2} of the film annealed at 300 °C is observed only, where a field strength of more than 5 Oe was needed to reverse the magnetization from a negative to a positive state, one may infer that treatments with $T_{AN} < T_B$ do not affect the magnetic state of the samples. However, as clearly seen in Fig. 4(d), when $\mathbf{H_{AN}}$ is antiparallel to the original EB direction, all the annealing procedures used affect the magnetic configuration being able to virtually reverse the EB direction. The loop traced at $\varphi_H = 0°$ on the sample treated at 300 °C with $\varphi_{AN} = 180°$ is practically identical to that corresponding to $\varphi_H = 180°$ and $\varphi_{AN} = 0°$, see Fig. 4(b). The curves obtained after annealing with $\varphi_{AN} = 180°$ and $T_{AN} = 110$ and 150 °C, however, have lower H_{C1} (here, due to the antiparallel configuration, H_{C1} corresponds to the negative-to-positive magnetization transition) than those of Fig. 4(c).

Figure 5 shows the effect of the cooling field strength on the easy-axis hysteresis loops' characteristics and the correspondingly-exctracted from them EB field in the NiO/NiCu bilayer. Again, the value of H_{C2} is changed more

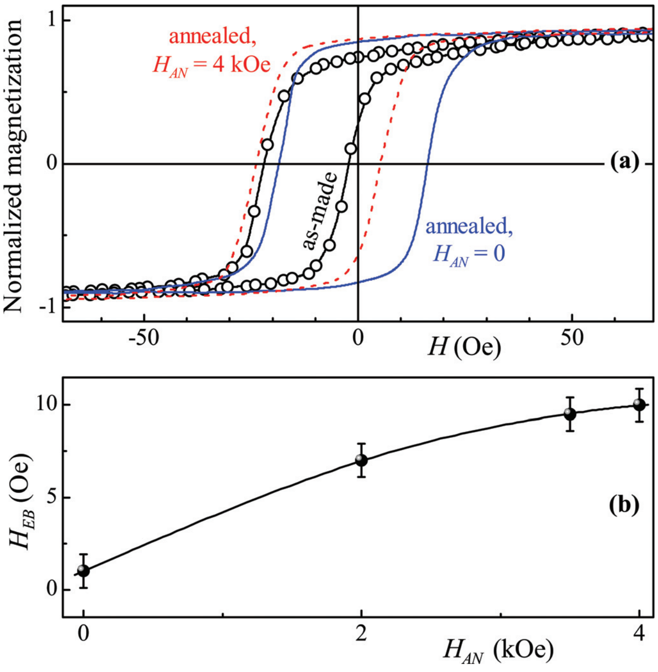

FIG. 5. (Color online). (a) Easy-axis hysteresis loops traced on the as-made NiO/$Ni_{0.75}Cu_{0.25}$ film (open symbols) and samples annealed at 300 °C with $H_{AN} = 4$ kOe (dashed line) and zero-field cooled, starting from 300 °C (solid line), respectively. (b) H_{EB} as a function of the cooling field.

significantly than the H_{C1} one. The curve traced on the zero-field-cooled from 300 °C sample presents slightly higher coercivity than that of the respective FC sample and is practically unbiased ($H_{EB} \approx 1$ Oe). It is worth noting that the latter sample is still anisotropic as the magnetization curve measured along the perpendicular direction (not shown) has lower coercivity and remnant magnetization and is more rounded than the easy-axis one. This indicates that even cooling from $T > T_N$ is not able to remove the anisotropy induced during the sample's deposition. Although H_{EB} gradually increases with H_{AN}, a tendency for saturation could be inferred in Fig. 5(b).

D. Ion irradiation

Figure 6 gives the easy-axis hysteresis loops traced under the same conditions as those shown in Figs. 4(c) and 4(d) but obtained after ion bombardment using different He^+ fluences instead of annealing. Similarly to annealing with $\varphi_H = 0°$ starting from temperatures lower than T_N, the magnetization curve traced after irradiation with $\varphi_{IB} = 0°$ using the lowest fluence of 5×10^{13} ions/cm^2 is practically identical to that of the as-made sample. Higher fluence irradiations modify the descending branch of the loops only, decreasing by 5 Oe the value of H_{C1}. It is worth noting that such a coercivity modification upon irradiation is opposite to that obtained by annealing, where H_{C1} remained constant and only the value of H_{C2} was increased after annealing with $T_{AN} > T_B$.

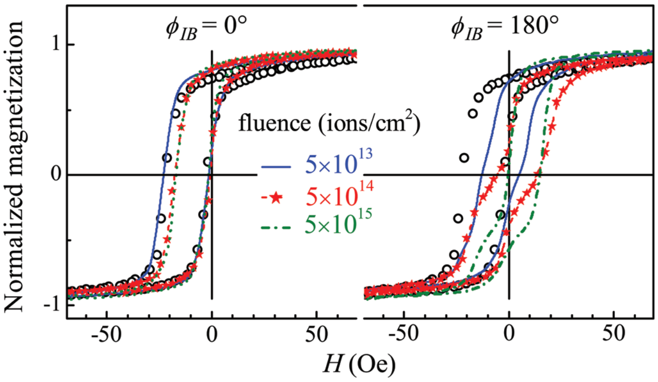

FIG. 6. (Color online). Easy-axis loops of the as-made film (open symbols) and samples irradiated using different ion fluences (lines). (Left) $\mathbf{H_{IB}}$ parallel and (right) antiparallel to the original EB direction.

Distinct results were obtained for $\mathbf{H_{IB}}$ antiparallel ($\varphi_{IB} = 180°$) to the original EB direction, where two features of the loops traced for $\varphi_H = 0°$ deserve to be highlighted. First, each loop looks composed of two magnetic phases, i.e., there appears a pattern of two subloops, which gradually shift to the positive direction with the raise of the fluence. The upper subloop is positively shifted for fluences higher than 5×10^{13} ions/cm^2, being the field shift (≈9.5 Oe) equal to that of the analogous pieces irradiated with $\varphi_{IB} = 0°$, and the relative weight of the other progressively decreases with the fluence.

IV. DISCUSSION

The EB manifestation of the as-made NiO/NiCu bilayer could be intuitively explained assuming that the field present during the film's growth has induced a preferential in-plane orientation of both AF and FM spins. The torque exerted by the spins of the FM atoms first deposited on top of the AF layer, from its turn, might be able to change the order of some of the still "hot" interface AF spins from the original state to the reverse orientation, thus, adding up to the partially uncompensated AF state at the interface with a net polarization in the field direction, necessary for the appearance of EB.

The mechanism responsible for establishing EB in NiO/NiCu through annealing in a field from above T_N differs from that setting the effect in conventional EB systems. Previous studies[6,7,10] on EB systems characterized by $T_C \ll T_N$ have pointed out that the FM spins at the FM/AF interface are likely to be responsible for establishing the exchange coupling in the PM state of the FM. Due to the proximity with the already ordered AF (as it is cooled from above T_N, its uncompensated spins are frozen with a net polarization along $\mathbf{H_{AN}}$), these interface moments are aligned prior to the remaining PM moments, which provides, in addition to $\mathbf{H_{AN}}$, a strong effective field.

Figure 4(b) shows that loops measured after annealing the film with $\mathbf{H_{AN}}$ applied at different in-plane directions starting from 300 °C are practically the same, which indicates that either annealing at fields ≥ 4 kOe is able to set practically all uncompensated AF interfacial spins for any direction of $\mathbf{H_{AN}}$, or this could be a result of isotropically distributed orientations of the still nonactivated uncompensated spins. However, the curves obtained after annealing with $\varphi_{AN} = 180°$ and $T_{AN} = 110$ or 150 °C, see Fig. 4(d), have lower H_{C1} values (here, due to the antiparallel configuration, H_{C1}

corresponds to negative-to-positive magnetization transition) than those measured after annealing with $\varphi_{\rm AN} = 0°$, see Fig. 4(c). This implies that when $T_{\rm AN} < T_B$, at least for $H_{\rm AN} \leq 4$ kOe, the annealing is not capable to set all uncompensated AF interface spins in this EB system and $H_{\rm EB}$ is reduced as more of these spins are frozen in random directions.[10]

The value of the cooling field is expected to play a crucial role in unconventional EB systems characterized by $T_C \ll T_N$. As discussed previously, the induced magnetization at the PM state of the NiCu should play a similar role as that of a FM layer when pinning an AF. The induced PM magnetization is proportional to the value of $H_{\rm AN}$ for relatively weak cooling fields (like these used here), i.e., it is expected that PM with a small induced PM moment cannot achieve the full strength of the exchange biasing as a fully aligned FM state is certainly not achieved. However, contrary to what is estimated from all theoretical models, Cai *et al.*[7] showed that a PM with a modest induced moment could achieve the full strength of the exchange coupling without the necessity of a fully aligned FM state. It seems that the results presented here, see Fig. 5(b), are in accordance with their observation.

Light-ion bombardment affects the NiO/NiCu bilayer in a rather different manner than the annealing. That is, antiparallel anneal changes mainly H_{C2}, whereas the irradiation decreases H_{C1} only; also, the shape of the hysteresis loops is changed gradually when the ion fluence is increased for $\mathbf{H}_{\rm IB}$ in the antiparallel configuration, though no significant variation of the shape of the loops with the annealing temperature was observed. The evolution of the two subloops in Fig. 6 measured after irradiation with $\varphi_{\rm IB} = 180°$ evidences that the number of uncompensated spins at the interface, effectively parallel to $\mathbf{H}_{\rm IB}$ and oppose to the original EB direction, gradually increases with the ion fluence, indicating that ion bombardment with sufficiently high fluences might reverse the EB direction completely. In the unconventional EB system studied here, however, such a postdeposition treatment has not been able to enhance the $H_{\rm EB}$ as observed in bilayers with $T_C > T_N$.[13,24] The same holds for the here-performed magnetic annealing.

It is believed that the realization of EB in bilayers with $T_C \ll T_N$ might have important implications in technological applications[7,9] seeing that one can expand the search for FM/AF bilayers with optimized performance finding suitable values of $H_{\rm EB}$ and H_C near RT without looking upon the condition $T_C > T_N$. Our work indicates that magnetic annealing and ion bombardment seem very promising for such tuning. Certainly, more systematic research needs to be conducted on unconventional EB systems showing higher shift field and/or coercivity than that studied here to better clarify the role of each of the postdeposition treatments' parameters on the anisotropy and coupling variations, which will be the aim of a forthcoming study.

To conclude, this paper reports experimental results obtained on an unconventional EB system where $T_C \ll T_N$ with emphasis on establishing EB bias through two types of postdeposition treatments. Annealing with magnetic field of different strengths and applied at distinct in-plane directions and/or from different temperatures, as well as light-ion irradiation, also in a presence of an in-plane magnetic field, were employed. It was found that although the EB can be completely reversed by means of either of these postdeposition treatments when the treatment magnetic field is antiparallel to the EB direction induced during the film's growth, these treatments provide very little variation of the EB field value. These results seem to support the hypothesis that the interfacial moments adjacent to the AF layer are likely to be responsible for the exchange biasing in the paramagnetic state.

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