

## Driving the magnetization reversal below the blocking temperature in exchange biased NiFe/NiO

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The temperature dependence of the exchange bias field and coercive field was studied in a polycrystalline NiFe layer coupled with a diluted NiO layer. The temperature behavior of both fields is modified by cooling the bilayer below the Curie, Neel, and/or blocking temperatures. Below these temperatures, the presence of double hysteresis loops demonstrates the key role of the NiFe multidomain state during the cooling procedure. © 2013 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4820249>]

Uniaxial anisotropic properties in magnetism offer a broad diversity of hysteretic behavior. Over recent decades, a new kind of anisotropy, named unidirectional (or exchange) anisotropy, has been discovered<sup>1,2</sup> and thoroughly studied to build fundamental understanding of magnetization reversal and provides a basis for its applied uses in magnetic data reading devices, among others.<sup>3,4</sup> This exchange anisotropy is observed in ferromagnetic(F)/antiferromagnetic(AF) bilayers and revealed by a hysteresis loop shift along the field axis ( $H_e$ ) and coercive field ( $H_c$ ) enhancement. Experimental observations and theoretical predictions have shown that domain wall (DW) formation in the F and/or in the AF should be considered in the reversal mechanism in order to obtain a reasonable value for  $H_e$ .<sup>5</sup> Mauri *et al.* suggested the presence of parallel DW at the interface.<sup>6</sup>  $H_e$  would then depend on the energy stored in this wall. This model does not show an enhancement of the coercivity for a perfect system (in a case with no disorder and zero temperature). Defects within this DW are predicted to enhance the anisotropy locally and result in DW pinning, leading to the  $H_c$  enhancement.<sup>7</sup> Instead of a parallel DW proposed in these models, Malozemoff,<sup>8</sup> followed by Nowak *et al.*,<sup>9</sup> predicted that non-uniform magnetization reversal, via the creation of a multidomain structure in the AF, was a driving mechanism of exchange anisotropy. In their models, interfacial or AF bulk defects lead to a random field that breaks the AF layer into domains over its area.  $H_e$  originates from the interfacial pinned spins, i.e., the non-reversible part of interfacial spins. The  $H_c$  enhancement originates from the reversible process driven by the AF uniaxial anisotropy that creates additional critical fields that hinder the DW motion in the F layer.<sup>10,11</sup>

The temperature behavior of pinned and unpinned spins should depend on the particular thermal conditions. Previous experiments have shown that different behaviors could be obtained by cooling down an exchange coupled system through one of the following ordering temperatures: the F Curie temperature, the AF Neel temperature, or the blocking

temperature  $T_b$  (defined here as the temperature below which  $H_e \neq 0$ ).<sup>12–18</sup> Double hysteresis loops arising from two types of domains have already been reported in F/AF bilayers when samples were cooled through an ordering temperature.<sup>12–18</sup>

Here, we show that the temperature behavior of  $H_e$  and  $H_c$  may be modified by cooling the F/diluted AF system well below one of the previously mentioned ordering temperatures. We will discuss our results considering the multidomain state of the ferromagnet and show that a direct interaction of the external field with pinned or unpinned spins during cooling is not the driving mechanism.

A bilayer of  $\text{Ni}_{81}\text{Fe}_{19}$ (20 nm)/NiO(200 nm) was deposited on a Si substrate by RF sputtering under a 300 Oe magnetic field, with a base pressure of  $10^{-7}$  mbar. In this paper, the direction of the applied field during growth is considered as the positive direction. No annealing treatment was performed. The NiO layers were deposited at an argon pressure of  $4 \times 10^{-3}$  mbar by sputtering from a  $\text{Ni}_2\text{O}_3$  target. The NiO prepared at this pressure is a diluted AF and its stoichiometry corresponds to that of  $\text{Ni}_{0.38}\text{O}_{0.62}$ . Therefore, in the rest of this paper, NiO corresponds to  $\text{Ni}_{0.38}\text{O}_{0.62}$ . Compositional and structural characterization were performed using field emission scanning electron microscopy, transmission electron microscopy, energy dispersive x-ray spectroscopy, x-ray reflectometry, and x-ray diffraction techniques. It showed the uniform and polycrystalline nature of NiO layers grown at the argon pressure of  $4 \times 10^{-3}$  mbar.<sup>19,20</sup> The NiFe layer was also found to be polycrystalline using XRD analysis with a NiFe (111) rocking curve of about  $20^\circ$ . Temperature dependent magnetic measurements were performed between 10 K and 300 K using a superconducting quantum interference device (SQUID) magnetometer.

The temperature dependence of  $H_e$  and  $H_c$  was first probed using the following protocol: a positive (negative) external saturation field of 2200 Oe ( $-2200$  Oe) was applied during field cooling. The procedure was stopped every 25 K to measure M-H loops. The M-H loops were measured between  $\pm 2200$  Oe. In the case of the negative field cooling,

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the field was set at  $-2200$  Oe after recording each M-H loop and before the decrease of  $T$ . The 300 K hysteresis curve, i.e., the starting point, is the same prior to positive and negative field cooling, as shown in Figure 1(a). It should be noted that there was no training effect observed in the sample studied here. Figures 1(b) and 1(c) show that a positive field cooling procedure results in an increase of  $H_e$  and  $H_c$  with a decrease in  $T$ . However, negative field cooling results in the opposite phenomenon for  $H_e$ : a large decrease of  $H_e$  with a decrease in  $T$ . Such an effect could either arise from a straightforward interaction between the diluted AF and the external field, or from the interfacial interaction with the F negatively magnetized.

In the case of experimental protocol which involved a cooling through  $T_b$ , previous experiments have already shown that the straightforward interaction with the external field is not a driving mechanism: the key role of a weak external field was only to orient the ferromagnetic layer during the cooling.<sup>12,15,21</sup> In the same case of experimental protocol which involved a cooling through  $T_b$ , previous studies have already shown that the pinned spin population was influenced by the F magnetic state well below  $T_b$ .<sup>22,23</sup> In the study presented here, experimental protocols occur well below  $T_b$ . Indeed, it should be noted that  $H_e \simeq 9$  Oe at 380 K

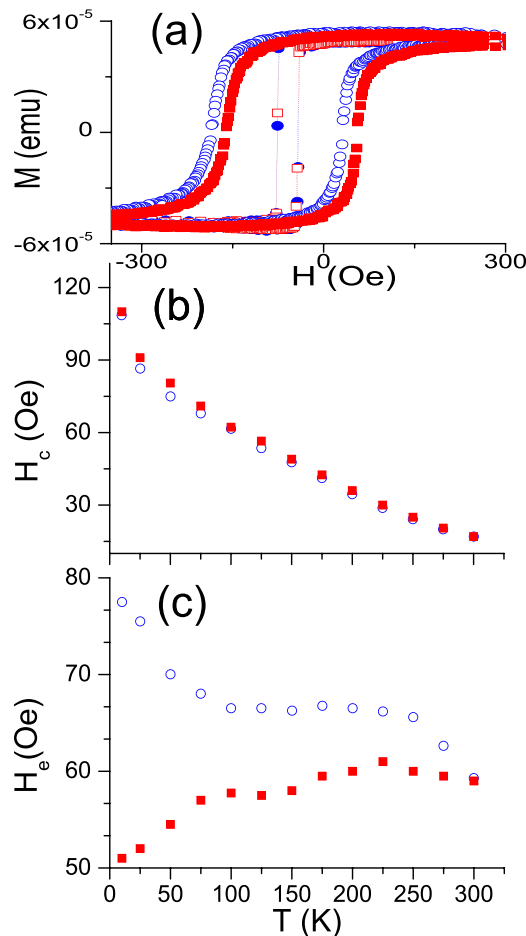


FIG. 1. (a) Magnetization loops obtained at 300 K before positive (●) and negative (□) field cooling; magnetization loops obtained at 10 K after positive (○) and negative (■) field cooling. (b)  $H_c$  evolution with temperature for positive (○) and negative (■) field cooling. (c)  $H_e$  evolution with temperature for positive (○) and negative (■) field cooling.

(not shown in Figure 1(c)), indicating that  $T_b$  is greater than 380 K for this sample. Thus, if the straightforward interaction with an external field is ruled out, then the present study would show that the pinned spin population may be modified even if a sample is not driven through any of its ordering temperatures.

The coercivity, driven by the uncompensated and unpinned spin population of the diluted AF,<sup>19,20</sup> is hardly affected by the direction (sign) of the field cooling, as can be seen in Figure 1(b). Furthermore, the shape of the hysteresis loop does not depend on the sign of the external saturation field during the cooling procedure, as shown in Figure 1(a) for results obtained at 10 K.

In order to determine which mechanism drives the  $H_e$  dependence with  $T$  below the ordering temperature, i.e., either the F magnetization or the interaction with the external field, we field cooled our system in a  $-25$  Oe field. This field corresponds to a positively magnetized F for  $T > 150$  K, and to a negatively magnetized F for  $T < 150$  K, as shown in Figure 2. If the F magnetization drives this mechanism, then we should observe a modification of the magnetization reversal around 150 K. Figure 2 shows just such a phenomenon.

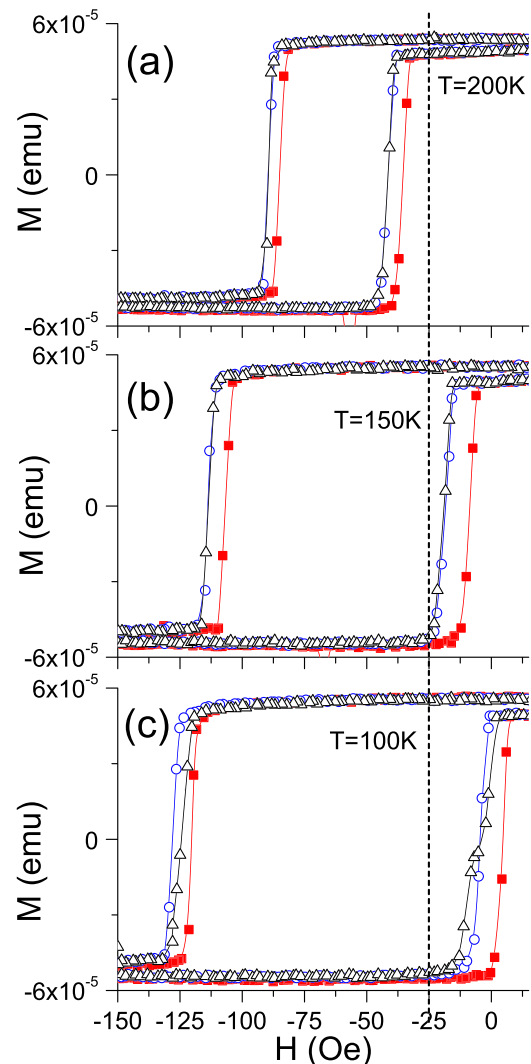


FIG. 2. Magnetization loops obtained after positive (○), negative (■), and  $-25$  Oe (△) field cooling: (a) at 200 K, (b) at 150 K, and (c) at 100 K. The vertical line is a guideline to identify  $H = -25$  Oe.

Indeed, for  $T > 150$  K, the hysteresis loops are identical to the ones observed for the positive field cooling procedure, i.e., when the system at  $+2200$  Oe was cooled. However, for  $T < 150$  K, the magnetization reversal is modified, and the hysteresis loop exhibits an inflection point around  $-10$  Oe. Thus, below  $T_b$ , the  $-25$  Oe cooling experiment shows that the driving mechanism of the reduced  $H_e$  relies on the F magnetization during the field cooled (FC) procedure.

In order to further understand the modification of the magnetization reversal, the following experimental protocol was applied to maintain a multi-domain F state during field cooling. In this protocol, the sample is field cooled from  $300$  K to  $10$  K, in steps of  $25$  K, in a non-constant field. In fact, the temperature was stabilized every  $25$  K, where  $M(H)$  loops were acquired to determine  $H_c$  and  $H_e$ . The sample was then field cooled to the next temperature at this  $H_c$  value for the next  $25$  K. In this paper, we will refer to this procedure as the “coercive cooling procedure.” Figure 3 shows that the non-homogeneous F domain state during cooling results in a transition from one broad loop above  $275$  K to two non-separated subloops from  $275$  K to  $10$  K. The shift and width of the top (bottom) subloop are obtained using  $H_1^{\text{top}}$  and  $H_2^{\text{top}}$  ( $H_1^{\text{bottom}}$  and  $H_2^{\text{bottom}}$ ), as shown in Figure 3(a).  $H_1^{\text{top}}$  and  $H_2^{\text{top}}$  correspond to the half of the positive saturated magnetic moment.  $H_1^{\text{bottom}}$  and  $H_2^{\text{bottom}}$  correspond to the half of the negative saturated magnetic moment. The widths are similar for both subloops, as shown in Figure 3(b). This is expected since the results shown in Figure 1 revealed that the coercivity is hardly affected by whether the field cooling is positive or negative. However, Figure 3(c) shows that the  $T$  dependence shift of the top subloops exhibits a similar behavior than the one obtained through a negative field cooling (i.e., it decreases as the temperature decreases), whereas the  $T$  dependence shift of the bottom subloop exhibits a similar behavior to the hysteresis loops obtained through a positive FC procedure.

This phenomenon, observed in the present study, may be explained by the presence of magnetic moments that got pinned in the sample during the growth of the sample in the applied field, and those moments do not change throughout the entire experimental procedure described in this paper. These provide a  $H_e$  of about  $60$  Oe at  $300$  K. On top of that, there are some other moments that get pinned at lower temperatures and possibly with smaller strength and anisotropy. The direction (sign) of the latter moments depends on the cooling field or the local magnetization of the F layer during cooling. The combination of the former and the latter pinned moments provides an average exchange bias field. For this reason, the two curves for  $H_e$  in Figures 1(c) and 3(c) are almost symmetric with respect to the horizontal line of  $H_e = 60$  Oe. It should be noted that repeating similar measurements while warming up the sample would be of interest as it could increase the symmetry of the results at higher temperatures. A previous study has shown that the pinned spin population may be altered in the sense that angular changes in the direction of the exchange field can be obtained if the angle between the applied field and the sample was modified below  $T_b$ .<sup>24</sup> The study presented here indicates that changes of magnetization reversals may occur without any modification of such an angle.

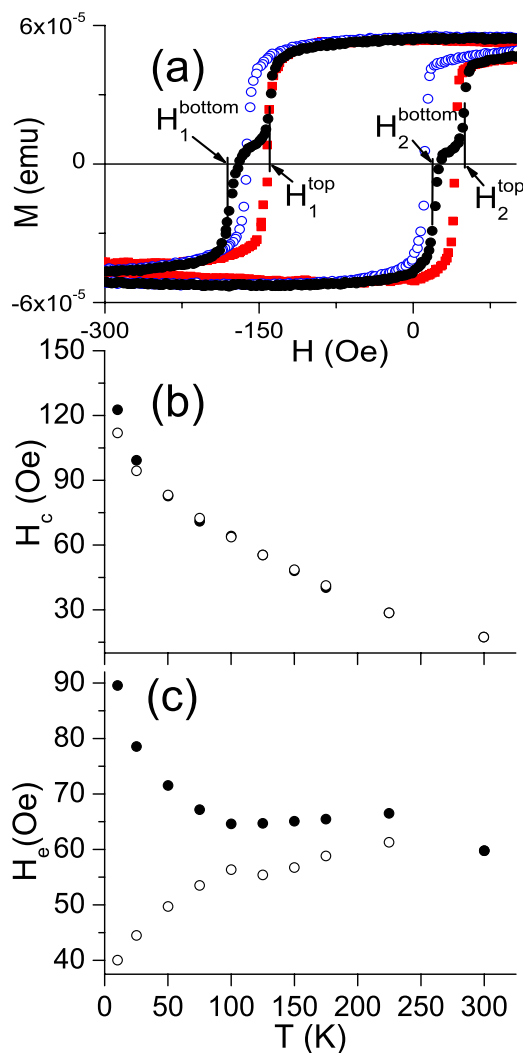


FIG. 3. (a) Magnetization reversal loops obtained at  $25$  K after positive ( $\circ$ ), negative ( $\blacksquare$ ), and coercive field ( $\bullet$ ) cooling procedures. (b)  $H_c$  evolution with temperature for the coercive cooling procedure, for top ( $\bullet$ ) and bottom ( $\circ$ ) subloops. (c)  $H_e$  evolution with temperature for the coercive cooling procedure, for top ( $\bullet$ ) and bottom ( $\circ$ ) loops.

Furthermore, the presence of two subloops in the coercive cooling procedure suggests that the coercive cooling procedure results in a non-uniform exchange over the sample area. There are two spatially separated subsystems in the AF, each with a net but a different frozen AF pinned moment. Hence, each AF subsystem gives rise to local exchange bias on the ferromagnet and to a non-uniform exchange over the sample area. This non-uniformity of the exchange arises from the non-uniform magnetic state of the F during cooling. This mechanism has previously been observed after field cooling through an ordering temperature.<sup>12–18</sup> In the present study, it is demonstrated that it can be activated at any temperature below  $T_b$ . This constitutes another degree of freedom for modifying the F magnetization reversal using exchange anisotropy. This mechanism differs from the previous results<sup>12–18</sup> showing a reduced  $H_e$  below  $T_b$ , as a non-uniform  $H_e$  over the sample area is demonstrated. Finally, the presence of a non-uniform exchange bias over the sample area suggests that the multidomain structure in the diluted AF plays a key role.

In conclusion, our results show that the multidomain state within the ferromagnet provides a way to modify the exchange anisotropy and magnetization reversal. This multidomain state is obtained by following a field cooling procedure around the temperature dependent coercive fields. The demonstration of a modification of the magnetization reversal well below  $T_b$ , over a small temperature range, has relevance for various applications. First, it shows that just a small  $T$  change below  $T_b$  may result in a large modification of the reversal. Such a change could be induced by laser heating or a current. Second, it demonstrates that a given exchange bias within a device could be compromised by temperature fluctuations in a magnetic field, even below  $T_b$ . Finally, it also suggests that the performance of high frequency devices based on the permeability of exchange biased systems could also be compromised if subjected to temperature changes.

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