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Interface exchange coupling in a CoPt/NiO bilayer

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ABSTRACT

The effects originating from the proximity between the ferromagnetic and the antiferromagnetic phase of a CoPt/NiO bilayer, grown at 670 K by Pulsed Laser Deposition, have been investigated from the point of view of the chemical properties (through Hard X-ray Photoelectron Spectroscopy, HAXPES) and of the magnetic behavior (by measuring hysteresis loops in the temperature range 5–300 K both after cooling in zero external field and after cooling from $T = 380$ K in a field of 1 T). At $T = 5$ K, the coercivity, measured after zero-field-cooling, is ~168 mT, to be compared to that of a reference CoPt layer of ~87 mT. Such magnetic hardening of the ferromagnetic CoPt phase is ascribed to the magnetic exchange interaction at the interface with the antiferromagnetic NiO phase, which is also responsible for the horizontal shift of the loop, observed only after field-cooling (exchange bias effect). Actually, the latter effect persists up to room temperature (exchange fields $\mu_0 H_{ex}$ ~60 mT and ~8 mT were observed at $T = 5$ and 300 K, respectively). Hence, it can be deduced that the CoPt and NiO phases are efficiently coupled by the exchange interaction, despite the chemical inhomogeneity observed at the interface region. In fact, the HAXPES analysis reveals that a chemical reduction of the NiO phase takes place in the interface region, resulting in the formation of metallic Ni. On the other hand, this inhomogeneity of the interface is proposed to be at the origin of the peculiar shape of the field-cooled loop at $T = 5$ K, featuring a double reversal of the magnetization.

1. Introduction

One of the main issues of nanomagnetism is the study of the effects originating from the proximity between different magnetic phases. In the recent past, the research in this field has led to resounding discoveries of interesting physical phenomena, paving the way for spintronics and for the appearance of a next generation of magnetoelectronic devices [1,2]. The study of proximity effects in such composite systems necessarily implies a deep knowledge of compositional and microstructural features of materials, strongly affecting the magnetic behavior.

One of the most investigated magnetic phenomena is the exchange bias (EB) effect [3], which plays a crucial role in spin-valve based structures and devices [4]. It consists in the horizontal shift of the hysteresis loop because of the magnetic exchange interaction at the interface between a soft ferromagnetic (FM) layer and an anisotropic antiferromagnetic (AFM) one, with nanometric thicknesses. The effect usually occurs when the FM/AFM system is cooled in a static magnetic field (H_{cool}) through the Néel temperature T_N of the AFM, being the Curie temperature of the FM larger than T_N. However, a number of experimental investigations [5–7] have established that, if the AFM layer has a nanocrystalline or disordered structure, it may be sufficient to field-cool across a characteristic temperature above which the anisotropy energy barriers of the AFM phase are so low that they can be easily overcome by the AFM spins. This is related to an actual distribution of T_N in an inhomogeneous AFM system, consisting of regions of different anisotropy. Hence, below the characteristic temperature, the final arrangement of the AFM spins is determined by the exchange coupling with those of the adjacent FM phase, so as to minimize the interface exchange interaction energy. During a hysteresis loop, the AFM spins exert a torque action on the FM spins, resulting in the appearance of an unidirectional anisotropy (usually indicated as exchange anisotropy), revealed by the horizontal shift of the loop.

The role of H_{cool} is to magnetically saturate the FM layer in a single domain state, which maximizes the EB effect. Indeed, if the sample is cooled in zero field from a demagnetized FM state, no loop shift is observed, due to the existence of magnetic domains with different magnetization orientations. Moreover, the shift is often accompanied by a coercivity enhancement, whose origin is only one of the numerous controversial items in the EB phenomenology. A proposed explanation assumes that, due to local variations of anisotropy in an inhomogeneous AFM, a fraction of the AFM spins, subjected to strong local anisotropy, exerts the torque action on the FM spins resulting in the loop shift, whereas another fraction, with low local anisotropy, is

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dragged by the FM spins during the magnetization reversal, resulting in an additional anisotropy contribution for the FM component and producing the coercivity enhancement [8]. In our opinion, if this description is realistic, a coercivity enhancement should be observed also in the zero-field-cooled state, i.e. when no loop shift is experienced, unless the direction of this additional anisotropy changes on a length scale shorter than the ferromagnetic exchange length of the FM phase, so that its contribution is averaged out. An increase of the coercivity, even when zero-field-cooled, has been reported for core–shell FM/AFM nanoparticles, with respect to the same particles without the AFM shell [9,10]. However, this item hasn't been systematically addressed in layered systems and unambiguous information is missing.

In this context, we have studied the proximity effects in the CoPt(FM)/NiO(AFM) system produced by Pulsed Laser Deposition (PLD). In particular, we report on a typical sample showing a drastic increase in the coercivity measured at low temperature after zero-field-cooling, with respect to a reference CoPt layer, and showing EB effect only after field-cooling. The role of the interface exchange interaction in determining these behaviors is discussed, also considering the chemical and structural features of the interface as derived from XPS analysis.

2. Experimental details

The CoPt/NiO system was prepared by a sequential deposition at T = 670 K of NiO and CoPt on a Si (100) substrate in a PLD high vacuum chamber. Depositions were performed by focusing a pulsed KrF Lambda Physik excimer laser (l = 248 nm, pulse duration = 17 ns) on a rotating Ni target. The spot energy fluence was 3 J/cm^2, and the pulse repetition rate was 2 Hz for a total of 7200 pulses. Substrate and target were assembled in a frontal geometry at 50 mm reciprocal distance. The PLD chamber was evacuated up to a base pressure of 1×10^{-8} Pa prior to the film deposition. The NiO layer (nominal thickness = 20 nm) was grown at 5×10^{-5} Pa in a reactive atmosphere produced by an O_2 dynamic gas flow through a needle valve [11]. Then, after recovering the vacuum at the background pressure, a thin CoPt film of about 5 nm was deposited on top by ablating a composite Co and Pt target, at the same growth temperature. A CoPt/Si reference sample was prepared in the same deposition conditions.

Hard X-ray Photoelectron Spectroscopy (HAXPES) measurements in the energy range up to 6.5 keV were performed at the ID32 beamline of the European Synchrotron Radiation Facility in Grenoble, France. The X-ray beam was monochromatized by a Si(111) double crystal monochromator and focused on the sample with Be compound refractive lenses. SPECS Phoibos 225 electron analyzer was mounted at 90° with respect to the incident photon beam such that a geometry of incidence angle of 45° corresponding to 45° take-off angle for photoelectrons could be used. Analyzer entrance slit size and pass energy were chosen such that the energy resolution of XPS spectra is limited by the X-ray beam energy bandwidth which is about 470 meV at 3.5 keV and 870 meV at 6.5 keV. XPS data were recorded at 3.5 keV and 6.5 keV to vary the probing depth of this experimental method. With increasing kinetic energy, the mean free path of the ejected photoelectrons increases significantly and allows one to retrieve depth-sensitive chemical state composition at interfaces buried at depths of tens of nanometers (for a review see e.g. Ref. [12]). Survey spectra and high resolution spectra of Ni 2*p*, Co 2*p*, and Pt 4*f* were recorded and analyzed; Si 2*s* substrate signal can be easily identified in the survey spectra confirming that the whole bilayer has been probed. Fermi edge signal of a clean Au foil was measured for calibration of the XPS data in energy.

Zero-field-cooled (ZFC) and field-cooled (FC) magnetic hysteresis loops were measured at different temperatures in the 5–300 K range on the CoPt/NiO bilayer and on the CoPt reference sample, using a Superconducting Quantum Interference Device magnetometer (maximum field 5.5 T). In the field-cooling procedure, the CoPt/NiO film was cooled from T = 380 K down to the measuring temperature under an applied field of $\mu_0 H_{cool} = 1$ T, parallel to the film plane; then, the field was increased up to $\mu_0 H = 2$ T and the hysteresis loop was measured. Finally, the loops were corrected for the diamagnetic contribution of the Si substrate.

3. Results and discussion

3.1. HAXPES analysis

It has been shown [13] that Co and NiO are prone to chemical reduction at their interface. The redox reaction creates metal cations of the oxide and oxidized metal atoms in the vicinity of the interface. The level of oxygen diffusion depends on several factors such as sample preparation conditions, initial roughness of the interface and its structural order. Similar effects are to be expected at the CoPt/NiO interface. Fig. 1a shows Ni $2p_{3/2}$ spectra of the bilayer at 6.5 keV and 3.5 keV of incident photon energy corresponding to HAXPES effective attenuation lengths in the range of 10 and 5 nm, respectively, considering the nominal chemical profile of the sample. The spectra can be decomposed into three main components: metallic Ni contribution at binding energy $E_b = 852.9(1)$ eV, main NiO line at 854.5(2) eV and the oxide shake-up satellite at 855.9(3) eV. These values are in agreement with various XPS studies of clean and oxidized nickel [13]. We observe an increase in the relative intensity of the metallic component as the photon energy is lowered. Since this corresponds to a lower photoelectron kinetic energies, the recorded signal is more surface sensitive. If we assume the same energy dependence of the various Ni 2*p* component cross-sections (valid likely with the exception of the exact form of the shake-up satellite) we can conclude that the reduced metallic Ni is at the top of the NiO layer, i.e. at the NiO/CoPt interface.

The corresponding Co $2p_{3/2}$ spectra and their deconvolution are shown in Fig. 1b. The relaxed energy resolution does not allow for unambiguous identification of possible oxide states such as Co_3O_4 and

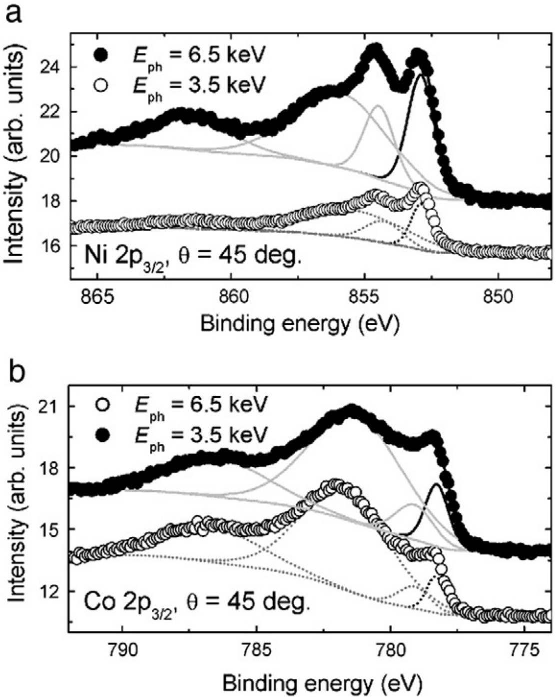

Fig. 1. (a) Ni $2p_{3/2}$ and (b) Co $2p_{3/2}$ XPS spectra recorded at two different photon energies E_{ph}. Shirley profile has been used for background subtraction.

CoO so these are overall represented by a Voigt profile centered at $E_b = 781.3(2)$ eV. Broad feature at 786.4(3) eV corresponds to the oxide component shake-up satellite [14]. Metallic-like component (corresponding to CoPt layer) is at the lowest binding energy, 778.2(1) eV, slightly above 778.84 eV for clean Co reported in Ref. [15] but in agreement with Ref. [14]. Quantitative analysis of these XPS data is complicated by the exposure of the sample to air prior to HAXPES measurement which resulted in the oxidation of the top layer. The observed cobalt oxide components can thus be present at the NiO/CoPt interface or/and at the sample surface. But the metallic-like component of the Co $2p_{3/2}$ spectra is clearly more intense in the 6.5 keV data than at 3.5 keV, which means that the metallic-like signal comes from the bulk of the sample. This conclusion is corroborated by comparison with XPS data recorded at very shallow take-off angle (10°) with $E_{ph} = 6.5$ keV where, despite the high kinetic energy of photoelectrons, the metallic-like component is hardly discernible.

3.2. Magnetic analysis

The ZFC hysteresis loop measured on the CoPt/NiO bilayer at T = 5 K is shown in Fig. 2a; for comparison, the loop relative to the CoPt reference sample is also displayed. The latter has a coercivity $\mu_0 H_{C\_ZFC}$ ~87 mT. This value is larger than that expected for a CoPt film of the soft fcc phase, whose magnetocrystalline anisotropy coefficient is of the order of 10^4 J/m^3 [16]. In fact, it approximately corresponds to just one half of the anisotropy field 2 K/M$_s$, where M$_s$ is the saturation magnetization, assumed of 700 kA/m [17]. Coercivity values ranging from some hundreds of Oe till a few Tesla are measured in CoPt layers after proper annealing treatments, inducing, depending on the annealing temperature, a partial or complete structural transition to the chemically ordered tetragonal CoPt L1$_0$ phase with high-anisotropy [18].

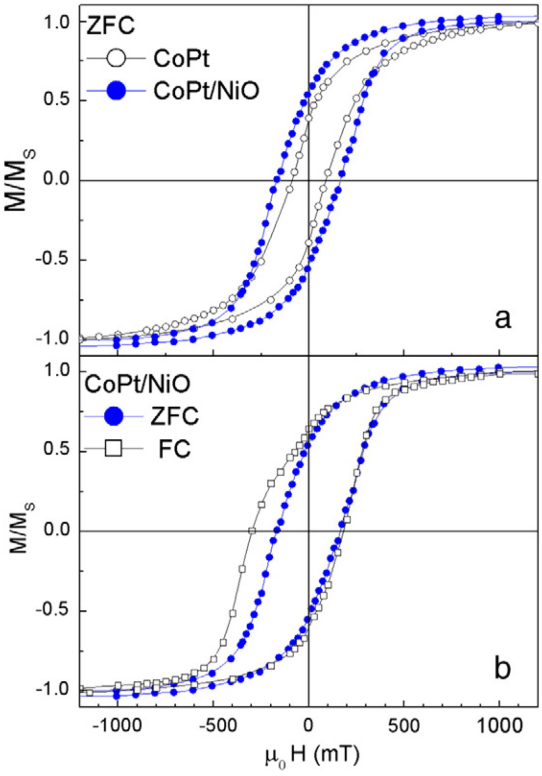

Fig. 2. (a) Zero-field-cooled (ZFC) hysteresis loops measured at the temperature T = 5 K on the reference CoPt sample (–○–) and on the CoPt/NiO bilayer (–●–); (b) hysteresis loops for the CoPt/NiO bilayer, measured at T = 5 K in the ZFC state (–●–) and in the field-cooled (FC) state (–□–). The curves are normalized to the magnetization value at saturation (M$_S$).

Accordingly, we infer that a partial chemical ordering may be present in the investigated sample, which makes the FM layer inherently magnetically harder, compared to the materials traditionally employed in EB systems.

In the CoPt/NiO film, $\mu_0 H_{C\_ZFC}$ ~168 mT and also the normalized remanent magnetization is enhanced with respect to the CoPt sample (~0.6 against ~0.4, Fig. 2a). Hence, the proximity with the NiO component determines a magnetic hardening of the CoPt layer. In Fig. 2b, the hysteresis loop measured at T = 5 K on CoPt/NiO after field-cooling is shown together with the ZFC loop already displayed in Fig. 2a. The FC loop is clearly shifted along the horizontal axis, revealing the presence of EB effect; the exchange field, expressed as $H_{ex} = -(H_{right} + H_{left})/2$ being H_{left} and H_{right} the points where the loop intersects the field axis, is ~60 mT. The EB effect unambiguously discloses the existence of the magnetic exchange coupling at the FM/AFM interface.

On this basis, we propose that the cause of the intrinsic magnetic hardening of the CoPt/NiO sample, as results from the ZFC loop, is the exchange interaction between the FM spins and the AFM spins, of lower anisotropy, which are dragged during the magnetization reversal, resulting in an enhanced effective anisotropy with respect to the CoPt layer alone.

Thus, the FM/AFM interface exchange interaction brings about the onset of an exchange anisotropy term with unidirectional character, which manifests itself only after field-cooling, giving rise to EB, since it is averaged out in the ZFC state; in addition, the interface exchange interaction causes a net increase of the effective anisotropy energy barriers of the FM layer. In other words, we can say that the FM/AFM coupling provides an additional anisotropic contribution, which is not completely averaged out in the ZFC state and determines the coercivity increase with respect to the FM layer alone. Actually, the effect of this contribution can be further enhanced by field-cooling. In fact, in the FC loop the coercivity $H_{C\_FC} = (H_{right} - H_{left})/2$ is ~241 mT, considerably higher than in the ZFC case (Fig. 2b). This can be explained considering that, since the field-cooling process selects a preferential magnetization orientation of the FM layer, it acts to reduce the effect of macroscopic averaging of the anisotropy usually associated to the presence of a number of space-distributed easy axes [19], namely, in this case, of magnetic domains with different magnetization orientations.

The curves of H_{C_ZFC}, H_{C_FC} and H_{ex} as functions of temperature are shown in Fig. 3. H_{ex} decreases with increasing T, indicating a progressive reduction of the strength of the FM/AFM coupling. H_{C_FC} and H_{C_ZFC} decrease as well and their difference reduces with rising T and it is less than 20 mT at T = 300 K. However, the analysis of the same curves normalized to their values at T = 5 K (inset of Fig. 3) clearly reveals that the thermal evolution of H_{C_ZFC} and H_{C_FC} is very similar. This suggests that the prevailing source of anisotropy in CoPt/NiO must be the same both in the ZFC and in the FC state, so as to result in a similar decreasing rate of H_{C_ZFC} and H_{C_FC} with rising T. In particular, the extent of the decrease of both H_{C_ZFC} and H_{C_FC} passing from 5 to 300 K corresponds to that undergone by H_{ex} (inset of Fig. 3), which supports our picture that the exchange coupling with the NiO component is responsible for the magnetic hardening of the CoPt layer.

This is remarkable considering that the CoPt phase we have produced is inherently quite hard. On the other hand, this also implies that its exchange length is shorter than the traditional soft FM component of EB bilayers. Actually, this may be the characteristic which allows the magnetic hardening to be experienced in our system. In fact, the favorable condition may be realized that the ferromagnetic exchange length of the FM layer is shorter than the length over which the additional anisotropy contribution, arising from the exchange interaction, changes orientation. Therefore, the latter is not averaged out.

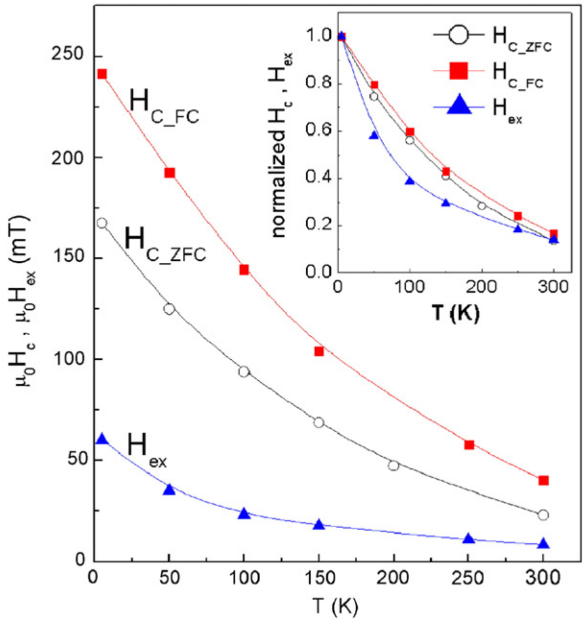

Fig. 3. ZFC coercivity (H_{C_ZFC}), FC coercivity (H_{C_FC}) and exchange field (H_{ex}) for the CoPt/NiO bilayer shown as functions of T. In the inset, the same curves are normalized to their values at T = 5 K.

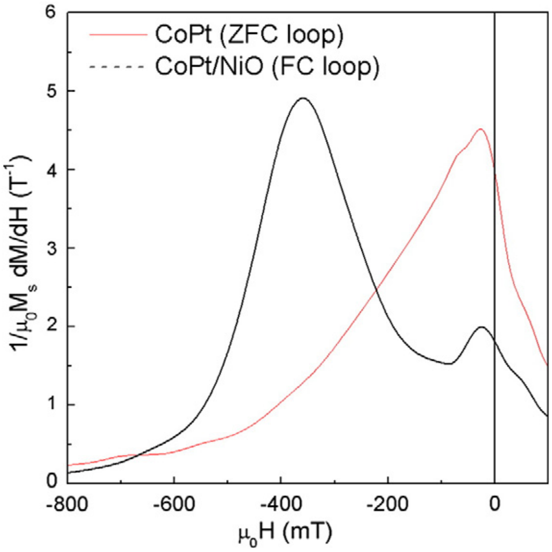

Fig. 4. Derivative of the descending branch of the ZFC loop of the CoPt reference sample (solid line) and of the FC loop of the CoPt/NiO bilayer (dashed line), both measured at T = 5 K.

Therefore, despite the chemical and structural disorder characterizing the interface region and revealed by the HAXPES analysis, the exchange interaction between CoPt and NiO definitely determines the overall magnetic behavior of the sample. In this respect, it should be noted that at T = 300 K a considerable value of μ_0H_{ex} ~8 mT is measured. Usually, in EB systems exploiting the NiO phase, the blocking temperature T_B, above which the EB effect disappears, lies below 300 K [20]. The main reason for this behavior is generally assumed to be the weak magnetic anisotropy of the NiO phase that is unable to exert a torque action on the magnetic moments of the FM component at high temperature, in spite of the fact that the Néel temperature of bulk NiO, above which it becomes paramagnetic, is 524 K. Moreover, it is known that other factors, such as the grain size of the AFM phase [6], the crystallinity degree [21] or the combination of structural and magnetic disorder [22,23] may affect the strength of the FM/AFM exchange coupling. To compare the magnitude of the EB effect in different systems, the parameter $\Delta E = H_{ex}\, t_{FM}\, M_{S,}$ (t_{FM} is the thickness of the FM layer) is often adopted, which corresponds to the interface energy per unit area, according to the simple model for EB by Meiklejohn and Bean [3]. In our case, at T = 300 K, ΔE is ~0.03 mJ/m^2. A ΔE value of the same order (~0.05 mJ/m^2) can be calculated for the NiFe/NiO system described in Ref. [24], which is one of the few literature cases of T_B higher than room temperature.

Hence, the reduction of NiO at the interface with CoPt, leading to the appearance of a fraction of metallic Ni, does not seem to have any particular detrimental effect on the extent of the EB effect. On the other hand, it should be also considered that the chemical reduction of NiO at the interface with the metallic FM layer may occur also in other EB systems (as some reports seem to confirm [25]), namely may be common to coupled systems exploiting NiO.

Actually, a certain influence of the inhomogeneity of the interface region on the magnetic behavior of the sample is to be expected. In this respect, a feature deserving some attention is the shape of the FC hysteresis loop at T = 5 K (Fig. 2b). In particular, the trend of the descending branch of the loop (from $\mu_0H = 2$ T to $\mu_0H = -2$ T) is consistent with a double reversal process of the magnetization, indicating the coexistence of FM regions with a different switching field. In Fig. 4, the derivative of the descending branch of the FC loop of CoPt/NiO and of the ZFC loop of CoPt at T = 5 K (shown in Fig. 2) are compared. The curve for the CoPt reference sample features a peak at ~25 mT, which can be assumed as a rough measure of the mean switching field. In the curve relative to CoPt/NiO, two broad peaks are visible, one in correspondence to that in the CoPt sample and one at much higher field, around 350 mT. A possible explanation for this behavior, which we have observed in a number of CoPt/NiO samples produced in the same way, is that the strength of the exchange coupling with the NiO phase is not homogeneous along the whole interface, but it varies in space so that in some regions of the CoPt layer the effects caused by the proximity with the NiO layer tend to vanish. In our opinion, this can be tentatively ascribed to the chemical inhomogeneity of the interface in our sample, which can be finally depicted as a transition region of irregular thickness, very far from the ideal model of a well-defined FM/AFM interface.

4. Conclusions

The magnetic exchange interaction between the CoPt and the NiO layers strongly determines the magnetic behavior of the whole composite sample, in spite of the fact that the interface region, across which the coupling takes place, features an inhomogeneous chemical profile, as revealed by HAXPES.

In particular, we have proposed that, besides of giving rise to an unidirectional exchange anisotropy for the spins of the FM layer, the exchange interaction produces a net increase of the effective anisotropy energy of the FM phase. The presence of the unidirectional anisotropy is revealed only by field-cooling the sample, which produces a loop shift (EB). On the other hand, the net increase of the FM effective anisotropy is revealed by the strong enhancement of the coercivity and of the normalized remanence measured at low temperature in the CoPt/NiO bilayer even after zero-field-cooling, with respect to a reference CoPt sample. The EB effect is also accompanied by an increase in H_{C_FC}, compared to H_{C_ZFC}. In this respect, we have proposed that the field-cooling process, selecting a preferential magnetization orientation in the FM layer, just enhances the effect of magnetic hardening experienced in the ZFC state.

The thermal evolution of H_{ex}, H_{C_ZFC} and H_{C_FC} supports this description. In fact, with rising T, the three parameters show a similar decreasing trend, indicating that the main source of anisotropy for the CoPt phase, both in the ZFC and in the FC state, must be the exchange interaction with the NiO layer whose strength reduces progressively with temperature.

The effect of magnetic hardening produced in the ZFC state has been seldom described in literature, but, actually, the possibility of exploiting the FM/AFM exchange interaction to modify the coercivity of a FM material, without inducing a concomitant EB effect, may open important technological perspectives. In the case of the investigated system, the CoPt phase is inherently harder than the FM materials typically used in EB systems and the H_{C_ZFC} enhancement is definitely remarkable since, at T = 5 K, $\mu_0H_{C\_ZFC}$ is ~87 mT in the single-phase CoPt sample and ~168 mT in the CoPt/NiO bilayer.

The chemical inhomogeneity of the interface region, which obviously implies also a structural and magnetic inhomogeneity, does not seem to alter the extent of the EB effect which, actually, persists up to T = 300 K. On the other hand, we have proposed that this inherent disorder of the interface region may cause a sort of spatial modulation of the exchange coupling strength. This conclusion is supported by the observation that also the magnetization reversal process is not at all homogeneous since different values of the switching field have been distinguished by analyzing the FC loop at T = 5 K, including that corresponding to the reversal in the reference CoPt sample. Further studies are in progress to better elucidate this last phenomenon.

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