Exchange-bias properties in permalloy deposited onto a Pt/Co multilayer

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A shift in the hysteresis loop along the magnetic field axis, H_E , is induced in a permalloy layer (with in-plane anisotropy) deposited onto a Pt/Co multilayer ML (with perpendicular anisotropy) by applying a strong in-plane magnetic field, H_0 , to the sample prior to measure the low field hysteresis curve. No field cooling is required to induce H_E . This shift is accompanied by a vertical displacement of the loop and an enhancement of coercivity, H_C . These effects originate from the coupling between the permalloy layer and an uncompensated in-plane magnetic moment in the Pt/Co ML induced after saturating the ML with the initial large in-plane field (H_0). The training effects and the temperature dependencies of H_E and H_C are investigated. If H_0 is applied perpendicular to the film plane, the multilayer preserves a single domain state with perpendicular magnetization in the field range where permalloy reverses and, as a consequence, no loop shift or coercivity enhancement are observed.

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I. INTRODUCTION

Exchange bias refers to the shift of the hysteresis loop, along the magnetic field axis, typically observed in exchange interacting antiferromagnetic (AFM)-ferromagnetic (FM) materials.¹⁻⁴ During the last two decades this effect has been extensively investigated, mainly due to its technological applications in magnetic sensors based on spin-valves or tunnel junctions structures, where exchange-biased bilayers constitute an essential part.⁵ The hysteresis loop shift is often accompanied by an increase of coercivity, which can be used to enhance performance of hard magnetic materials.⁶ It is noteworthy that exchange bias effects have also been observed in ferrimagnetic (Ferri)-AFM,7 Ferri-Ferri,8 and Ferri-FM (Refs. 9 and 10) exchange interacting materials. Some particular exchange coupled soft-hard ferromagnetic or ferrimagnetic materials (i.e., spring magnets) can also exhibit hysteresis loop shifts. This occurs when the soft layer reverses its magnetization, while maintaining the magnetization of the hard phase tightly stuck along the easy directions.^{11,12} In spring magnets, the interphase dipolar interactions have been shown to play an important role in inducing exchange bias.¹³

Usually, to induce exchange bias, a field cooling procedure through the Néel or Curie temperature of the AFM or Ferri material is necessary. This field cooling often requires the material to be heated to above room temperature since, for technological applications, exchange bias effects need to be present at and above room temperature. In some cases this can result in structural deterioration of the layers. Nevertheless, it has been recently demonstrated that the magnitude of exchange bias in FM-AFM bilayers, particularly when the anisotropy of the AFM is low, can be modified by applying sufficiently large fields, without need of varying temperature. This might be due to field-induced changes in the domain structure of the AFM.^{14,15}

In this article, we demonstrate that when a soft permalloy (Py) layer (with in-plane anisotropy) is deposited onto a [Pt/Co] ML (with perpendicular anisotropy) a shift in the hysteresis loop of this soft magnetic layer can be induced at

room temperature by applying a large in-plane magnetic field to the system. This large in-plane field induces an in-plane magnetic moment in the ML, which is able to pin the Py layer during its magnetization reversal (analogously to an AFM layer in FM/AFM exchange coupled systems). An enhancement of coercivity is also observed. Interestingly, the loop shift induced at room temperature is preserved at high temperatures, up to T=550 K. Moreover, this loop shift can be erased by simply applying a sufficiently large field along the perpendicular-to-film direction. Hence, this kind of multilayered structure (i.e., ML with perpendicular anisotropy + soft magnetic layer with in-plane anisotropy) may be used as a novel system for exchange bias applications.

II. EXPERIMENTAL PROCEDURE

A magnetic multilayer (ML) with the composition $Pt(20 \text{ nm}) / [Co(0.6 \text{ nm}) / Pt(1.8 \text{ nm})]_5 / Co(0.2 \text{ nm}) / Py(3)$ nm)/Cu(2 nm), where Py denotes permalloy (i.e., $Ni_{81}Fe_{19}$) was deposited at room temperature onto a thermally oxidized Si substrate by dc magnetron sputtering. The thin Co(0.2 nm) layer was introduced in order to avoid interdiffusion between the Pt and the Py and to enhance the perpendicular anisotropy of the ML. For comparison, a similar ML without Py was also deposited. The base pressure was 5.3 $\times 10^{-6}$ Pa, while the Ar pressure during deposition was 0.25 Pa. To induce exchange bias, a large magnetic field $(H_0=15 \text{ kOe})$ was applied at room temperature along the thin film plane. This field is enough to fully saturate both the Py and the Pt/Co ML. After removing H_0 , hysteresis loops were recorded in-plane in a vibrating sample magnetometer (VSM) using a maximum field, $H_{hvst,Max}$ of variable intensity, ranging from 0.7 to 15 kOe. Note that, contrary to most studies on exchange bias, no field cooling was required to induce exchange bias effects in this system. Hysteresis loops were measured at temperatures varying from 150 to 600 K. Training effects (i.e., variations in the magnitudes of the coercive field, H_C , and the exchange bias, H_E , when successive hysteresis loops are recorded¹) were investigated at room



FIG. 1. In continuous line is the hysteresis loop, measured at room temperature, of the Pt(20 nm)/[Co(0.6 nm)/Pt(1.8 nm)]₅/Co(0.2 nm)/Py(3 nm)/Cu(2 nm) system after applying a large positive field, $H_0=15$ kOe, along the thin film plane. In discontinuous line is the same hysteresis loop after correcting for the vertical shift (i.e., after centering along the magnetization axis). The inset shows the angular dependence of H_E , where θ designates the angle between the direction of H_0 and the direction along which the hysteresis loop has been measured (always in the thin film plane).

temperature. The angular dependence of H_E was also studied. Hysteresis loops with the magnetic field applied in-plane were also recorded after pre-magnetizing the system using the H_0 =15 kOe field applied perpendicular to the thin film plane. Moreover, hysteresis loops with the magnetic field applied perpendicular to plane were also recorded by an extraordinary Hall effect, a technique which is particularly sensitive to the perpendicular component of the magnetization.¹⁶ The magnetic domain structures after pre-magnetizing the system both in-plane and perpendicular-to-plane were investigated by magnetic force microscopy (MFM).

III. RESULTS AND DISCUSSION

Shown in Fig. 1 (continuous line) is the hysteresis loop, measured at room temperature, of the Pt(20 nm)/ $[Co(0.6 \text{ nm})/Pt(1.8 \text{ nm})]_5/Co(0.2 \text{ nm})/Py(3 \text{ nm})/Cu(2 \text{ nm})$ system after applying a large field, $H_0 = 15$ kOe, along the thin film plane. The hysteresis loop exhibits both a vertical shift, $\Delta M/M \sim 20\%$ (where ΔM is the difference of saturation magnetization between the measured loop and the loop centered in the M axis, see discontinuous line) and a shift along the magnetic field axis, H_E . The value of H_E , after correcting for the vertical shift, is 68 Oe. It should be noted that a vertical shift of the hysteresis loop has also been occasionally reported in FM-AFM exchange biased bilayers, although, in that case, its magnitude is very small, i.e., typically less than 1%.17,18 The vertical shift in FM-AFM bilayers is generally attributed to the existence of uncompensated AFM spins at the FM-AFM interface. Taking into account the physical dimensions of the sample and the amplitude of the magnetization it can be deduced that the measured hysteresis loop corresponds basically to the Py layer signal, although a minor contribution from the Pt/Co ML (particularly from the last Co layer in the ML), which would rotate together with Py, cannot be completely excluded. However, the presence of a vertical shift indicates that, after application and removal of the large H_0 magnetic field, a net uncompensated magnetic moment from the Pt/Co ML is retained inplane in the field range where the hysteresis loop is measured, i.e. (-1500, 1500 Oe). From the magnitude of the vertical shift it can be estimated that, in spite of the perpendicular effective anisotropy of the Pt/Co ML, around 15% of its overall magnetization remains, in fact, oriented towards the H_0 direction after removal of this field. This net in-plane magnetization component of the Pt/Co ML, which couples to the Py, is likely to be responsible for H_{E} . The angular dependence of H_E is shown in the inset of Fig. 1. θ designates the angle between the direction of H_0 and the direction along which the hysteresis loop has been measured (always in the thin film plane). The curve evidences the unidirectional character of the loop shift, typical of FM-AFM exchange coupled systems. Note that, to overcome the training effects of the system (which will be discussed later in the text), several loops were successively recorded along the H_0 direction before starting to measure the angular dependence of H_E . This is why the value of H_E along 0° in Fig. 1 (inset) is somewhat smaller than the previously mentioned value, H_E =68 Oe, of the first hysteresis loop. It is also worth mentioning that no significant differences in the shape of the loops and the angular dependencies of H_E and H_C are observed if one changes the in-plane direction along which H_0 is applied. This indicates that the sample is magnetically isotropic in the plane, since it is polycrystalline in nature and therefore there is no magnetic preferred orientation originating from the film microstructure.

It is noteworthy that in this system, after application of the H_0 in-plane field, the magnitudes of H_E , H_C and vertical shift $(\Delta M/M)$, measured along the H_0 direction, are found to depend on the maximum field used to perform the hysteresis loop, $H_{\text{hvst,Max}}$. As can be seen in Fig. 2, both H_E and $\Delta M/M$ are roughly constant in a relatively broad $H_{hvst,Max}$ range, i.e., 700–2000 Oe. For larger $H_{\rm hyst,Max}$ values, H_E and $\Delta M/M$ progressively decrease and, finally, they tend to vanish for $H_{\rm hvst,Max}$ > 5000 Oe, although values for $\Delta M/M$ as large as 2% are still observed for $H_{\text{hvst,Max}} = 10$ kOe, indicating that small nonreversed in-plane magnetization component is preserved even for rather large values of $H_{\rm hyst,Max}$. A good correlation between H_E and the vertical shift has also been reported in FM-AFM exchange coupled bilayers, proving that both effects are closely related to each other.¹⁷ On the contrary, the value of the coercive field, H_C , progressively increases with $H_{hyst,Max}$ and tends to level off for $H_{\rm hyst,Max} > 5000$ Oe. The decrease of $\Delta M/M$ and H_E with $H_{hyst,Max}$ can be attributed to the progressive dragging of the net in-plane magnetic moment of the Pt/Co ML as H_{hyst.Max} becomes larger. As the in-plane magnetic moment of the Pt/Co ML starts to reverse during the in-plane hysteresis loop, the vertical shift and H_E tend to decrease but, conversely, H_C progressively increases, as a result of the extraenergy required to switch the in-plane magnetic moment arising from the Pt/Co ML.

Figure 3(a) shows the hysteresis loop measured by the extraordinary Hall effect along the perpendicular to film direction of the $Pt(20 \text{ nm})/[Co(0.6 \text{ nm})/Pt(1.8 \text{ nm})]_5/$



FIG. 2. Dependence of the loop shift, H_E (in (a)), coercivity, H_C (in (b)), and vertical shift, $\Delta M/M$ (in (c)), measured at room temperature along the H_0 direction, on the maximum field used to perform the hysteresis loop, $H_{hyst,Max}$. The lines are guides to the eye.

Co(0.2 nm)/Py(3 nm)/Cu(2 nm) system after saturating it in a 12 kOe field applied perpendicular to the film direction. It can be seen that the loop is, in fact, the overlapping of two hysteresis loops, one hard axis loop corresponding to Py, which has an in-plane anisotropy, and a square loop corresponding to the Pt/Co ML. The good squareness of the loop is an indication that the Pt/Co ML remains in a single domain state after H_0 being applied along the perpendicular to the film direction. Interestingly, a completely square hysteresis loop (with a remanence to saturation ratio, $M_R/M_S=1$) is obtained in $Pt(20 \text{ nm})/[Co(0.6 \text{ nm})/Pt(1.8 \text{ nm})]_5$ (i.e., the ML without Py). Figure 3(b) shows the hysteresis loop, measured in-plane by VSM, of the Pt/Co/Py system, after saturating it using $H_0=15$ kOe applied perpendicular to the film direction. It can be seen that, contrary to the case where H_0 is applied in-plane (Fig. 1), no loop shift or coercivity enhancement are observed in this case. The loop is fully symmetric along the vertical axis. This is because the Pt/Co is in a single domain state with an out-of-plane magnetic moment. Therefore, no in-plane magnetic moments are induced in the Pt/Co ML and, hence, the Py is free to rotate along the in-plane direction. This means that exchange bias can be induced or deleted at a fixed temperature just by applying sufficiently large fields along the in-plane or perpendicular to plane directions, respectively. In fact, we found that a field of 6 kOe, applied perpendicular to the thin film plane, is required to completely erase an exchange bias induced by applying a strong (e.g., $H_0 = 15$ kOe) in-plane field.

In order to get deeper insight on the origin of the hysteresis loop shift of this system, MFM images were



FIG. 3. (a) Hysteresis loop of the Pt(20 nm)/[Co(0.6 nm)/ Pt(1.8 nm)]₅/Co(0.2 nm)/Py(3 nm)/Cu(2 nm) multilayer measured along the perpendicular-to-film direction by the extraordinary Hall effect after saturating the system perpendicular to the film direction; (b) Hysteresis loop of the same multilayer, measured inplane by VSM, after saturating it using H_0 =15 kOe applied perpendicular to the film direction.

performed in both the $Pt(20 \text{ nm})/[Co(0.6 \text{ nm})/Pt(1.8 \text{ nm})]_5$ $Pt(20 \text{ nm}) / [Co(0.6 \text{ nm}) / Pt(1.8 \text{ nm})]_{5} / Co(0.2 \text{ nm}) /$ and Py(3 nm)/Cu(2 nm) systems after applying H_0 along the inplane and perpendicular to plane directions. Shown in Fig. 4(a) is the MFM image of the Pt(20 nm)/[Co(0.6 nm)/Pt(1.8 nm)]₅ multilayer obtained at zero field after applying the $H_0=15$ kOe in-plane field. The image reveals that the in-plane field induces the formation of segmented stripe domains in the ML, which are of about 0.2 μ m in width. Similar domain structures have been reported in the literature in magnetic multilayers with out-of-plane anisotropy after saturating them in-plane.^{19,20} Due to the perpendicular anisotropy of the Pt/Co ML, the magnetization direction in each of these domains is essentially perpendicular to the thin film plane. However, although it cannot be directly observed by MFM, it is known that, in order to avoid divergences in the magnetic flux density, it is energetically favorable that the magnetization inside each domain progressively tilts towards the in-plane direction as it approaches the surface of the ML, i.e., forming closure domains.^{21,22} In these closure domains the magnetic moments are oriented in-plane. Conversely, when the H_0 field is applied along the perpendicular to film direction, a featureless magnetic contrast is observed in the $Pt(20 \text{ nm})/[Co(0.6 \text{ nm})/Pt(1.8 \text{ nm})]_5 \text{ ML} [see Fig. 4(b)],$ hence confirming that the system is in a single domain state. MFM observations have also been carried out in the $Pt(20 \text{ nm}) / [Co(0.6 \text{ nm}) / Pt(1.8 \text{ nm})]_5 / Co(0.2 \text{ nm}) /$ Py(3 nm)/Cu(2 nm) ML. Figure 4(c) shows the MFM image of this system after applying H_0 along the in-plane direction.



FIG. 4. (Color online) (a) Magnetic force microscopy (MFM) image of the Pt(20 nm)/[Co(0.6 nm)/Pt(1.8 nm)]₅ multilayer obtained at zero field after applying H_0 =15 kOe in-plane field; (b) MFM image of the same multilayer after applying H_0 =15 kOe along the perpendicular to film direction; (c) MFM image of the Pt(20 nm)/[Co(0.6 nm)/Pt(1.8 nm)]₅/Co(0.2 nm)/Py(3 nm)/Cu(2 nm) system after applying H_0 =15 kOe along an in-plane direction; (d) MFM image of the same multilayer after applying H_0 =15 kOe along an in-plane direction and subsequently applying a small in-plane field (H=130 Oe) during the imaging.

In this case, the interpretation of the domain pattern is more complex due to the overlapping between the domain structure of the Pt/Co ML and that of the Py layer and the shielding effect exerted by the Py layer. However, if MFM images are subsequently recorded under the presence of a small inplane magnetic field of variable intensity, significant changes in the domain pattern are observed. For instance, in Fig. 4(d), where a field H=130 Oe has been applied in-plane during the imaging, it can be seen that some magnetic domains (indicated by the circles) remain unchanged whereas some others (indicated by the squares) disappear. Since small variations in the applied field are able to significantly alter the domain pattern, this suggests that the observed magnetic domains could mainly correspond to Py. However, the fact that some domains remain rather stable in size while others change abruptly may be an indication of the role of the Pt/Co in-plane magnetic moments in partially pinning the Py layer. In view of the MFM results, it can be argued that, since the Pt/Co ML exhibits a strong perpendicular anisotropy (which arises from the hybridation between the electronic d states of Pt and Co at the Pt/Co interfaces²³), when the ML is premagnetized using a large in-plane field, the ML is left in a multidomain state, where configurations similar to closure domains are likely to form at the ML/Py interface. A schematic diagram of the domain configuration that is likely to form in the [Pt/Co]+Py system after application of the H_0 in-plane field is shown in Fig. 5(a). For comparison, Fig. 5(b) shows a domain pattern with a zero net in-plane magnetic moment, which could be obtained, for instance, after demagnetizing the sample using an AC in-plane magnetic field, as has been reported in the literature.^{21,22} The net in-



FIG. 5. (a) Schematic diagram of the domain configuration that is presumably formed in the [Pt/Co]/Py system after application of a strong H_0 in-plane field. For comparison, (b) shows a domain configuration with compensated in-plane magnetic moment, which could be obtained after demagnetizing the sample.

plane magnetic moment that is induced by the large H_0 initial in-plane field could stem from an enlargement of the closure domains oriented parallel to H_0 at the expenses of those oriented antiparallel to H_0 [as shown in Fig. 5(a)]. The coupling between Py and the net in-plane magnetization of the ML would then account for the observed shift of the hysteresis loop. It is noteworthy that similar arguments have been used to explain the exchange bias effects in the TbCo (Ferri)/Py system.^{9,10} Interestingly, an in-plane hysteresis loop on the [Pt/Co]/Py system was also performed after demagnetizing it using an in-plane AC field and no loop shift was observed, although the coercivity remained rather large, i.e., H_C =110 Oe. It should be noted that the observed exchange bias, apart from being due to direct exchange interactions between Py and the net in-plane magnetic moment of the Pt/Co ML,



FIG. 6. First and twentieth hysteresis loops of the Pt (20 nm) / $[Co (0.6 nm) / Pt (1.8 nm)]_5 / Co (0.2 nm) / Py (3 nm) / Cu (2 nm) system obtained after applying <math>H_0=15$ kOe and subsequently measuring using maximum fields $H_{hyst,Max}=1500$ [in (a)] and 3500 Oe [in (b)], respectively. Note that the loops are shown in the as-measured state, i.e., no corrections for the vertical shift have been performed in any of them.



FIG. 7. Dependence of H_E [in (a)] and $\Delta M/M$ (b) on the loop number, *n*, for the Pt(20 nm)/[Co(0.6 nm)/Pt(1.8 nm)]₅/Co(0.2 nm)/Py(3 nm)/Cu(2 nm) system after applying H_0 = 15 kOe along an in-plane direction, for both $H_{hyst,Max}$ =1500 and 3500 Oe. In (c) the loop shifts have been normalized to $H_{E,Max}$, i.e., the loop shift corresponding to first hysteresis loop after application of the in-plane H_0 =15 kOe field. Note that the lines are guides to the eye.

could also originate from magnetostatic coupling between the two materials. Namely, the Pt/Co ML has a significant remanence resulting in a varying stray field in the Py film, which could also pin domain walls. Finally, it should be noted that the domain pattern of the Pt/Co ML alone [as shown in Fig. 4(a)] should not be extrapolated to be the same as in [Pt/Co]+Py, since the presence of the Py layer is likely to also influence the domain configuration of the Pt/Co ML, particularly the domain size because of the direct exchange coupling between the top Co layer and the Py and the influence of the Py layer on the overall magnetostatic energy of the system.

As already commented, this system also exhibits training effects, i.e., variations in the magnitudes of H_E , H_C , and $\Delta M/M$ when repeated loops are performed after application of the H_0 in-plane field. Training effects are sometimes also observed in FM-AFM systems and they usually follow a $1/(n)^{1/2}$ law, which means that the magnitude of the loop shift decreases with the inverse $\frac{1}{2}$ power of *n*, where *n* is the hysteresis loop number.^{1,24,25} Figures 6(a) and 6(b) show the first and twentieth loop of the Pt(20 nm)/[Co(0.6 nm)/Pt(1.8 nm)]₅/Co(0.2 nm)/Py(3 nm)/Cu(2 nm) ML mea-



FIG. 8. Dependence of H_E and H_C on temperature. The loops have been measured upon heating after first applying the in-plane H_0 field at room temperature and then cooling the system to T=150 K. To overcome training effects, several loops were first recorded at T=150 K before starting the overall temperature dependence measurements. The lines are guides to the eye.

sured, respectively, using maximum fields $H_{\text{hyst,Max}} = 1500$ and 3500 Oe. The dependence of H_E and $\Delta M/M$ on the loop number is shown in Figs. 7(a) and 7(b). It can be seen that in both cases H_E and $\Delta M/M$ decrease when repeated loops are successively recorded. However, contrary to many exchange bias systems, ^{1,24,25} the dependence of H_E on the loop number can neither be fitted to a $n^{1/2}$ nor a log or exponential type of decay. The reduction is particularly steep during the first 3 loops. Additionally, for $H_{hyst,Max}$ =3500 Oe, the loop shift practically vanishes after a few hysteresis loops, whereas for $H_{\text{hyst,Max}}$ =1500 Oe the observed decrease in H_E is, at most, of about 35%. This can be more clearly seen in Fig. 7(c), where the dependence of $H_E/H_{E,Max}$ (where $H_{E,Max}$ is the loop shift corresponding to the first loop) on the loop number is presented. This effect can be understood taking into account that larger $H_{hvst,Max}$ values favor more dragging of the Pt/Co in-plane moment, as also suggested by the results in Fig. 2. As a consequence, during successive loops, if $H_{\rm hvst,Max}$ is large enough, the domain configuration probably changes progressively from that shown in Fig. 5(a) into the one in Fig. 5(b), where the net in-plane magnetic moment stemming from unequal closure domains has vanished. It is worth mentioning that if one plots the ratio $\Delta M/(H_E M)$ as a function of n (not shown), one obtains a roughly constant value, of around 0.22, for both $H_{hyst,Max}$ =1500 Oe and $H_{hyst,Max}$ =3500 Oe, again pointing out the existent correlation between H_E and $\Delta M/M$.

Finally, the temperature dependencies of H_E and H_C have been also investigated. This has been carried out by first applying the in-plane H_0 field at room temperature, then cooling the system to T=150 K and, finally, measuring hysteresis loops at different temperatures ranging from 150 to 600 K. As for the angular dependence, in order to overcome the training effects, several loops (ten) were recorded at T=150 K before measuring the overall temperature dependence. Shown in Fig. 8 are the temperature dependencies of H_C and H_E . Similar to FM-AFM exchange coupled bilayers,¹⁻⁴ both H_E and H_C decrease when increasing temperature probably due to the progressive loss of anisotropy in the Pt/Co ML and the concomitant thermally induced uppinning of magnetic domains. It is noteworthy that for sufficiently low temperatures (T < 200 K) the loops are even slightly open, a clear indication of partial dragging of the Pt/Co in-plane magnetic moment. Interestingly, the loop shift induced at room temperature is maintained up to T = 550 K. This makes this system appealing for technological applications.

In conclusion, a shift in the hysteresis loop has been obtained in a Py layer, with in-plane anisotropy, coupled to a Pt/Co ML with perpendicular anisotropy. The loop shift is induced, without need of any field cooling process, by applying a strong in-plane magnetic field to the system. After removal of this field, an in-plane magnetic moment in the Pt/Co ML is retained, which presumably couples to the Py,

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inducing also a vertical shift of the hysteresis loop. As in FM-AFM bilayers, an enhancement of coercivity is also observed. The loop shift induced at room temperature is preserved, upon heating, up to T=550 K. To erase the exchange bias and the coercivity enhancement it is necessary to apply a strong magnetic field along the perpendicular to film direction. The exchange bias can also be erased by cycling in large in-plane fields.

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