Exchange bias in antiferromagnetic-ferromagnetic-antiferromagnetic structures with out-of-plane magnetization

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Exchange bias effects are investigated in antiferromagnetic- (AF₁-) ferromagnetic- (F-) antiferromagnetic (AF₂) structures, where the F consists of a [Pt/Co] multilayer with perpendicular anisotropy and the two AF layers are composed of IrMn. The AF₁ and AF₂ thicknesses are varied so that the two IrMn layers exhibit different blocking temperatures. After field cooling, enhancements of the coercivity H_C and exchange bias field H_E are observed in the AF₁-F-AF₂ structures with respect to the two subsystems with a single AF layer (i.e., AF₁-F and F-AF₂). For all systems, the magnitude and sign of H_E can be subsequently tailored by field cooling processes under fields of different sign while H_C remains constant. The net effect of having two AF-F interfaces is roughly the sum of each individual interface contribution.

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INTRODUCTION

Exchange bias refers to the shift of the hysteresis loop, along the magnetic field axis, which is typically observed in exchange interacting ferromagnetic- (F-) antiferromagnetic (AF) materials.¹⁻⁴ The loop shift, denoted as H_E , is often accompanied by an enhancement of the coercivity H_C and is commonly induced by field cooling the F-AF system from above the Néel temperature of the AF. During the last decade, this phenomenon has been extensively investigated both from fundamental and technological points of view, mainly due to its applications in the development of magnetic random access memories (MRAM) and read heads based on spin valves or magnetic tunnel junction devices, where F-AF bilayers constitute an essential part.^{5,6} The majority of F-AF bilayers where exchange bias has been studied exhibit an in-plane magnetic anisotropy. However, recently, exchange bias effects have also been induced along the perpendicular-to-film direction, both in continuous multilayers⁷⁻¹⁵ or in lithographed nanostructures.¹⁶ In some cases, perpendicular exchange bias is observed at room temperature, hence making these structures appealing for the implementation of spin valves or tunnel junctions with perpendicular anisotropy.^{9,10}

In thin film form, exchange bias has been primarily investigated in F-AF bilayer structures.¹⁻⁴ Exchange bias effects in more complex systems such as F-AF-F trilayers or in multilayers containing several F-AF or ferrimagnetic-AF interfaces have also been reported, both in systems with in-plane^{17–27} or with out-of-plane anisotropy.^{12–15} In particular, it has been shown that when two or more F layers are separated by AF spacers, they can become coupled through the spacer (ferromagnetically, antiferromagnetically or at 90°), resulting in a range of interesting phenomena. In the case of F-AF multilayers with ferromagnetic coupling between the F layers, an enhancement of coercivity has been observed, which may be appealing for thin film magnets.^{22,23} Such H_C enhancement is attributed to either an effective magnetic surface anisotropy introduced on top of each F by the subsequent AF layer²⁴ or to the interlayer coupling between the successive F layers through the AF spacers.²² Although exchange bias effects in F-AF-F and [AF-F] multilayers have been explored to some extent, the effects of the coupling in AF-F-AF trilayers (i.e., a single F surrounded by two adjacent AF layers) have been far less investigated. The few existing studies of this type concentrate mainly on the chemical or structural changes occurring at the F-AF interfaces and their effects on the strength of the coupling.^{28–31} Comparative studies between AF-F-AF trilayers and F-AF bilayers with analogous composition have not been performed so far. Hence, the way exchange bias properties will be modified when a F layer is simultaneously coupled with two adjacent AF layers, instead of one, has not been systematically examined.

In this article, we investigate exchange bias effects in AF_1 -F- AF_2 structures, where the F is a [Pt/Co] multilayer with a perpendicular to plane effective magnetic anisotropy and the two AF layers are IrMn with different thickness, displaying different blocking temperatures (i.e., temperature at which exchange bias vanishes upon heating). For simplicity, we will refer to the AF₁-F-AF₂ structures as the "trilayer" systems and the AF₁-F and F-AF₂ structures as "bilayers." Our results show that, after field cooling a given trilayer from above the blocking temperature of the two AF, both H_C and H_E are enhanced with respect to the two constituent bilayers. Actually, the H_C and H_E values in the trilayers are roughly the sum of those from each individual AF-F interface. Moreover, due to their different blocking temperatures, the relative orientation of the two AF layers composing the trilayer systems can be tuned by combining successive field cooling processes under fields of different sign. The resulting H_E is determined by the magnitude and sign of the exchange bias field induced at each interface. Conversely, the coercivity is found to remain constant, independent of the coupling direction, both in the AF₁-F-AF₂ or in the AF₁-F or F-AF₂ systems.

EXPERIMENTAL

Three series of multilayers with the compositions (i) buffer(5 nm)/IrMn(7 nm)/Pt(0.4 nm)/[Co(0.4 nm)/ AF_1 -F, Pt(2 nm)₄, denoted as (ii) Pt(0.4 $nm)/[Co(0.4 nm)/Pt(2 nm)]_3/Co(0.4 nm)/Pt(0.4 nm)/$ denoted $IrMn(t_{IrMn,top})/Pt(2 nm),$ as F-AF₂, and Cu(5 nm)/IrMn(7 nm)/Pt(0.4 nm)/[Co(0.4 nm)/ (iii) $Pt(2 nm)]_3/Co(0.4 nm)/Pt(0.4 nm)/IrMn(t_{IrMn,top})/Pt(2 nm)$ denoted as AF₁-F-AF₂ (where Cu is a buffer layer) were deposited onto thermally oxidized Si wafers by dc magnetron sputtering. For the AF₁-F stack several buffer layers Cu, Ta, and Pt were preliminary tested. Although the thickness of the bottom IrMn layer AF_1 is kept constant to 7 nm, the AF_2 thickness values t_{AF_2} range from 2 to 15 nm for both F-AF₂ and AF₁-F-AF₂ structures. A thin Pt spacer was introduced between the AF and the first and/or last Co layer in the F multilayer to enhance the strength of the coupling.¹⁰ The base pressure was 5.3×10^{-6} Pa, whereas the Ar pressure during deposition was 0.25 Pa. All depositions were performed at room temperature. The samples were then annealed at 450 K (above the blocking temperature T_B of all systems) for 0.5 h and subsequently cooled under a field of $H_{\rm FC}$ =-2.5 kOe, applied perpendicular to the film plane, to set the unidirectional exchange anisotropy in this direction. Hysteresis loops, applying the magnetic field perpendicular to the thin film direction, were subsequently measured at room temperature using an extraordinary Hall effect setup, a technique which is particularly sensitive to the perpendicular component of the magnetization.³² Thermal activation effects on exchange bias properties were evaluated by subsequently field cooling the samples under a positive field (i.e., $H_{\rm FC}$ = +2.5 kOe) from temperatures ranging from 300 to 450 K, after the standard ($H_{\rm FC}$ =-2.5 kOe) cooling procedure.³³ Note that the top IrMn layer thickness was purposely varied in order to be able to tune the blocking temperature distribution of AF_2 with respect to the one of AF_1 .

RESULTS AND DISCUSSION

Representative hysteresis loops for the AF₁-F (with a Cu buffer layer) and F-AF₂ stacks, measured along the perpendicular to film direction, after perpendicular field cooling from 450 K under $H_{\rm FC}$ =-2.5 kOe, are shown in Fig. 1. It can be seen that the loops display a rather square shape, with a remanence to saturation magnetization ratio close to 1. This indicates that the samples exhibit an effective magnetic anisotropy perpendicular to the film, which is known to arise from the hybridization between the electronic d states of Pt and Co at the Pt/Co interfaces.³⁴ Furthermore, the loops exhibit a shift along the magnetic field axis, i.e., a perpendicular exchange bias. It should be noted that, for the same IrMn thickness (e.g., $t_{AF_1} = t_{AF_2} = 7$ nm) the magnitude of H_E is larger when the IrMn is deposited on top of the [Pt/Co] multilayer, i.e., F-AF₂, than at the bottom, i.e., AF₁-F. This difference in the exchange bias properties between top and bottom configurations is in qualitative agreement with results reported in F-AF bilayers with in-plane anisotropy. Structural differences (e.g., grain size, texture, or interface roughness)



FIG. 1. Hysteresis loops corresponding to the AF₁-F (with $t_{AF_1}=7 \text{ nm}$) and F-AF₂ (with $t_{AF_2}=2$, 5, 7, and 15 nm) bilayers measured by extraordinary Hall effect along the perpendicular to film direction, after field cooling ($H_{FC}=-2.5 \text{ kOe}$) along the same direction from T=450 K.

between the two configurations have been proposed as the most reasonable explanations for this effect.^{35,36} Changes in the microstructure could also explain the slight decrease in the squareness ratio (i.e., remanence to saturation ratio) in the F-AF₂ system with respect to AF₁-F. Other effects of the microstructure can be seen in the role of the buffer layer, where Cu maximizes the exchange bias properties for AF₁-F, compared to Ta or Pt buffers. Actually, Cu is often used as buffer for the growth of IrMn,^{35,37} although perpendicular exchange bias in IrMn—[Pt/Co]_n multilayers where the IrMn is deposited onto other buffer layers, such as Pt, has also been reported.³⁸ In the following, only results on structures with Cu buffer layer will be presented.

Shown in Fig. 2 are the dependences of H_E and H_C on t_{AF_2} for the F-AF₂ bilayers. It is observed that H_C and H_E follow the same behavior as observed for many in-plane systems using IrMn as AF.³⁹⁻⁴¹ Namely, both $H_E(t_{AF})$ and $H_C(t_{AF})$ exhibit a peak for relatively thin AF layers. In the F-AF₂ sample with $t_{AF_2}=2$ nm virtually no exchange bias is observed because the AF thickness is so small that the IrMn spin lattice is not sufficiently pinned, so that it is dragged by the magnetization of the adjacent [Pt/Co] multilayer when it switches. The inverse relationship between H_E and t_{IrMn} ob-



FIG. 2. Dependence of (a) the exchange bias field H_E and (b) the coercivity H_C on the AF₂ thickness t_{AF_2} for the F-AF₂ systems. The lines are guides to the eye.

served for $t_{IrMn} > 5$ nm can be understood within the framework of Malozemoff's model, where it is predicted that H_E is inversely proportional to the AF domain size (closely related to the AF thickness).⁴² The domain state model can also account for this relationship between H_E and t_{IrMn} since it predicts a reduced number of AF domain walls for thick layers, which should result in a H_E decrease.³⁹

Shown in Fig. 3 are some hysteresis loops for the AF₁-F-AF₂ structures for $t_{AF_1}=7$ nm and $t_{AF_2}=2$, 5, 7, and 15 nm. When comparing these loops with those of the AF₁-F and F-AF₂ bilayers, it can be seen that the loop shape is more square in the trilayers than in the F-AF₂ systems. This indicates that the presence of the bottom IrMn layer in the full AF₁-F-AF₂ structures improves the out-of-plane anisotropy. This effect might have a magnetic, rather than structural, origin. Specifically, the presence of two, instead of one, AF layers induces an extra out-of-plane anisotropy when the trilayer is field cooled along the perpendicular to film direction, consequently improving the squareness of AF₁-F-AF₂ structure. A similar effect has been observed in some F-AF multilayers, where it is possible to induce exchange bias effects along the perpendicular to film direction even when the F layers exhibit, intrinsically (i.e., without the presence of the AF), an in-plane magnetic anisotropy.^{13,15} In these systems, the hysteresis loops, measured along the perpendicular to film direction, also become more square and the appearance of perpendicular exchange bias is accompanied with an enhancement of coercivity. Remarkably, the improvement of the out-of-plane anisotropy induced by an AF has been proposed as a way to extend the temperature range of out-of-plane behavior in multilayers.⁴³

Apart from the squareness improvement, in Fig. 3 it can be also seen that both H_E and H_C for the AF₁-F-AF₂ structure are significantly larger than in the corresponding AF₁-F and F-AF₂ constituent bilayers. Closer inspection of the loops reveals that the H_E for the AF₁-F-AF₂ structure is



FIG. 3. Hysteresis loops corresponding to the AF₁-F-AF₂ system (with t_{AF_1} =7 nm and t_{AF_2} =2, 5, 7, and 15 nm) systems measured by extraordinary Hall effect along the perpendicular to film direction, after field cooling (H_{FC} =-2.5 kOe) along the same direction from *T*=450 K.

actually the sum of the loop shifts of the two constituent AF_1 -F and F-AF₂ stacks after field cooling. This is indicated in Fig. 4, where the dependences of H_E and H_C in the AF_1 -F-AF₂ trilayers are plotted as a function of t_{AF_2} . The discontinuous line in Fig. 4(a) is the arithmetic sum of exchange bias fields of the AF₁-F and F-AF₂ bilayers, which virtually overlaps with the experimental values obtained for the AF₁-F-AF₂ trilayers. Conversely, the sum of the coercivities of AF₁-F and F-AF₂ [discontinuous line in Fig. 4(b)] is slightly larger than H_C (AF₁-F-AF₂). This is because part of the measured H_C does not originate from the coupling with



FIG. 4. Dependence of (a) the exchange bias field H_E and (b) the coercivity H_C on the AF₂ thickness t_{AF_2} for the AF₁-F-AF₂ systems. Note that the continuous lines are guides to the eye. The crosses and discontinuous lines indicate the calculated H_E and H_C values that would be obtained for the AF₁-F-AF₂ systems if they were the arithmetic sum of the H_E and H_C values of the two AF₁-F and F-AF₂ counterparts.

the AF layers but it is the intrinsic H_C of the [Pt/Co] multilayer, which is of the order of a 12 Oe, similar to the H_C of the F-AF₂ system with $t_{IrMn2}=2$ nm, where exchange bias effects are almost inexistent. In fact, if one takes into account that $H_C([Pt/Co]) \sim 12$ Oe then, again, the coercivity enhancement (i.e., due to the coupling with the AF layers) of the AF₁-F-AF₂ stack is essentially the sum of coercivities of the corresponding AF₁-F and F-AF₂ structures. This additive character of the exchange bias properties of the two F-AF interfaces is maintained for all the investigated t_{AF_2} . In particular, the maximum in H_E and H_C for $t_{AF_2}=5$ nm, observed for F-AF₂, is mimicked in the AF₁-F-AF₂ structure. This additive behavior arises because, during magnetization reversal, the magnetization of the [Pt/Co] multilayer is simultaneously pinned, along the same direction, by the two adjacent AF layers and, consequently, the F-AF contact area increases, thus enhancing the total energy necessary to overcome by the F layer during reversal. This is of particular interest for the case of H_C , since some authors reporting H_C enhancements in F-AF multilayers with more than one F layer attribute such effect to the interlayer coupling between the several F layers,^{22,23} hence neglecting that H_C can also be enhanced simply because of the simultaneous coupling of each F with the two adjacent AF layers. Furthermore, it should be noted that the results of our work might shed light to the origin of the H_C enhancement, typically observed in F-AF powder mixtures, where the F particles are surrounded by several AF grains, thus resulting in multiple F-AF interfaces.44

The effect of varying the relative orientation of the coupling between the F layer and each of the AF layers has also been investigated. For this purpose, all the samples (AF₁-F, F-AF₂ and AF₁-F-AF₂ multilayers), which had al-



FIG. 5. Dependence of the exchange bias field H_E (———) and the coercivity H_C (———) of the AF₁-F (with $t_{AF_2}=7$ nm) and the F-AF₂ (with $t_{AF_2}=2$, 5, 7, and 15 nm) bilayers on the annealing temperature T_{ann} from which the samples are field cooled in positive field ($H_{FC}=2.5$ kOe) after the standard field cooling process from T=450 K, using a negative field ($H_{FC}=-2.5$ kOe). The lines are guides to the eye.

ready been field cooled from T=450 K in a negative field, $H_{\rm FC}$ =-2.5 kOe, were again field cooled under a *positive* field, $H_{\rm FC}$ = +2.5 kOe, from several annealing temperatures T_{ann}, located between room temperature and 450 K. Shown in fig. 5 are the dependencies of H_E and H_C on T_{ann} for different t_{AF_2} for the AF₁-F and F-AF₂ systems. It can be seen that H_E tends to decrease, changing sign, when increasing T_{ann}. Actually, the combination of two consecutive field cooling processes under fields of different sign, allows probing thermal activation effects in exchange bias systems.33 Namely, if thermal activation effects are present at $T=T_{ann}$, the initially induced (i.e., with cooling in negative field) exchange bias will be reduced to some extent due to some loss of the AF pinning strength, i.e., some grains in the AF layer can become superparamagnetic if their local blocking temperature is below T_{ann} . Since the samples are subsequently cooled again using a positive field, the AF grains affected by the thermal activation at T_{ann} will be then realigned but in the opposite direction, leading to an overall reduction of H_E when measured at room temperature. It can be seen in Fig. 5 that for the F-AF₂ systems, the average blocking temperature



FIG. 6. Dependence of the exchange bias field H_E (—O—) and the coercivity H_C (—**A**—) of the AF₁-F-AF₂ trilayers on the annealing temperature T_{ann} from which the samples are field cooled in positive field (H_{FC} =2.5 kOe) after the standard field cooling process, from T=450 K, using a negative field (H_{FC} =-2.5 kOe). The continuous lines are guides to the eye, whereas the dashed and dotted lines are, respectively, the calculated H_C and H_E values that are obtained from the arithmetic sum of the H_E and H_C measured in the two constituent bilayers AF₁-F and F-AF₂ after field cooling from T_{ann} .

(temperature at which H_E crosses zero) $T_{B,ave}$ increases for larger t_{AF_2} values, from $T_{B,ave} \sim 340$ K for $t_{AF_2}=5$ nm to $T_{B,ave} \sim 410$ K for $t_{AF_2}=15$ nm (for $t_{AF_2}=2$ nm $T_{B,ave}$ is below room temperature). This reduction of $T_{B,ave}$ with decreasing t_{AF} is in agreement with many exchange bias systems.¹ Additionally, it should be noted that for the same thickness of the AF₁ and AF₂ layers, $t_{AF}=7$ nm, $T_{B,ave}$ of AF₁-F systems is substantially lower, $T_{B,ave}(AF_1-F)$ ~ 340 K, than for the F-AF₂ system [$T_{B,ave}(F-AF_2)$ ~ 380 K]. This has probably a microstructural origin. For example, differences in crystallite size, roughness or defects between AF₁ and AF₂ can account for this different blocking temperature. It should be noted that, for all bilayer systems, although H_E decreases with T_{ann} , H_C remains insensitive to the coupling direction.

Plotted in Fig. 6 are the dependencies of H_E and H_C on T_{ann} for different t_{AF_2} for the AF₁-F-AF₂ systems. For

comparison, $H_F(AF_1-F) + H_F(F-AF_2)$, and $H_C(AF_1-F)$ $+H_C(\text{F-AF}_2)$ (obtained from the data in Fig. 5) are also given in Fig. 6 as dotted and dashed lines, respectively. Similar to the results obtained after the standard field cooled process, H_C and H_E of the AF₁-F-AF₂ systems after the second field cooling process are also roughly the sum of the two H_E and H_C values of the AF₁-F and F-AF₂ counterparts. The slight difference in measured and calculated H_C is due, as before, to the intrinsic H_C of the [Pt/Co] multilayer. Moreover, Figs. 5 and 6 reveal some effects which are worth mentioning. For example, for T_{ann} = 360 K, the AF₁ layer in the AF₁-F system has essentially reversed its orientation due to the field cooling in negative field (see left pannel in Fig. 5). However, for the same T_{ann} =360 K and for t_{AF_2} =15 nm, the AF₂ layer in F-AF₂ system is still mainly oriented in the upwards direction (bottom pannel in Fig. 5). As a result, one can infer that in the corresponding AF₁-F-AF₂ multilayer, the two AF layers will be roughly antiparallel to each other. As a result, one would expect this sample to exhibit virtually no exchange bias at this temperature. Interestingly, this is indeed what is observed in Fig. 6. In addition, the $T_{B,ave}$ of the AF₁-F-AF trilayers is lower than for the F-AF₂ bialyers for the same t_{AF_2} . This is best seen for large t_{AF_2} , e.g., comparing the lower panels of Figs. 5 and 6. For example, $T_{B,ave}(F-AF_2)$ ~400 K for $t_{AF_2}=15$ nm for F-AF₂, while $T_{B,ave}(AF_1-F_2)$ AF_2 ~ 370 K for the trilayer with the same thickness. This arises because the trilayer has thermal activation effects at both F-AF interfaces. Hence, $T_{B,ave}$ in the trilayers is somewhat an average of the $T_{B,ave}$ of the two interfaces. Finally, it is worth mentioning that, as it occurs for the bilayers, H_C in the trilayers is also roughly constant for whatever the orientation of the coupling in the two F-AF interfaces. This is because H_C depends on the irreversible dragging of AF spins, which enhances the Zeeman energy required for the reversal of the F layer and this does not depend on the coupling direction but only on its strength. These results, apart from their fundamental importance, are of particular technological interest both for the processing of thin film permanent magnets and for advancement in the implementation of novel types of magnetoelectronic devices.

CONCLUSIONS

In summary, the exchange bias effects in AF₁-F-AF₂ structures have been investigated and compared to those in AF₁-F and F-AF₂ constituent substructures. It was observed that the exchange bias energy in the AF₁-F-AF₂ sandwich is approximately given by the algebric sum of the two exchange bias coupling energies separately measured in AF₁-F and F-AF₂ bilayers. The sign in this algebraic sum can be modified by different annealing conditions (parallel or antiparallel annealing). In contrast, the coercivity of the F layer in AF₁-F and F-AF₂ is roughly given by the sum of the coercivity of the AF₁-F and F-AF₂ bilayers minus the coercivity of the AF₁-F and F-AF₂ bilayers minus the coercivity of the F layer alone. In some particular cases it is thus possible to obtain $H_E \sim 0$ in the AF₁-F-AF₂, while having nonzero H_E values of opposite sign in the two AF₁-F and F-AF₂ counterparts. Our results demonstrate that the coerciv-

ity enhancement typically observed in F-AF multilayers containing more than one F film does not necessarily originate uniquely from interlayer coupling between the several F layers but also from the additive exchange coupling effects of each F with its neighboring AF layers.

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