Uncompensated magnetization and exchange-bias field in La$_{0.7}$Sr$_{0.3}$MnO$_3$/YMnO$_3$
bi layers: The influence of the ferromagnetic layer

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We studied the magnetic behavior of bilayers of multiferroic and nominally antiferromagnetic o-YMnO$_3$ (375 nm thick) and ferromagnetic La$_{0.7}$Sr$_{0.3}$MnO$_3$ and La$_{0.67}$Ca$_{0.33}$MnO$_3$ (8–225 nm), in particular the vertical magnetization shift $M_E$ and exchange bias field $H_E$ for different thickness and magnetic dilution of the ferromagnetic layer at different temperatures and cooling fields. We have found very large $M_E$ shifts equivalent to up to 100% of the saturation value of the o-YMO layer alone. The overall behavior indicates that the properties of the ferromagnetic layer contribute substantially to the $M_E$ shift and that this does not correlate straightforwardly with the measured exchange bias field $H_E$.

I. INTRODUCTION

In bilayers composed of antiferromagnetic (AFM) and a ferromagnetic (FM) phases a “horizontal” shift in the field axis of the hysteresis loops is generally observed after cooling them in a field applied at temperatures between the Néel $T_N$ and Curie $T_C$ temperatures. This “exchange-bias field” $H_E$ has been studied in different systems due to its fundamental importance as well as its technological relevance in spin-valve sensors, actuators and in high-density recording media and some details of the origin of $H_E$ are still a matter of discussion.

Less studied is the shift in the magnetization axis, i.e. the “vertical” $M_E$ shift in the hysteresis loop, probably because of its rather small relative values and its dependence on the cooling field $H_{FC}$. Recently, a maximum shift of 16% of the saturation magnetization was found in Fe$_3$Ni$_{1-x}$Fe$_2$/Co bilayers, which appeared to have an exchange bias field of its own. It was proposed that $M_E$ is related to uncompensated moments (UCM) at the AFM/FM interface and should have a direct correlation to $H_{FC}$. Element specific x-ray magnetic studies of FeF$_2$/Co$_{12}$ and CoO/Fe$_{12}$ layered structures confirmed the existence of this $M_E$ shift and revealed its relation to specific UCM in the AFM material.

Due to the limited number of studies on the $M_E$ effect it is of general interest to find systems with larger magnetization shifts, not only because of its fundamental interest but also because this shift provides a new degree of freedom in the hysteresis loop that may be well have some applicability in future devices. In this work we studied the exchange-bias shifts $H_E$ and $M_E$ of the hysteresis loops as a function of temperature $T$ and $H_{FC}$ for three AFM/FM bilayers having the same AFM layer but different thickness and dilution of the FM layer. We observed an unusually large uncompensated magnetization shift $M_E$ that is not simply correlated with $H_E$ and does not originate only from the AFM layer but from the FM one.

II. SAMPLE PREPARATION DETAILS AND X-RAY CHARACTERIZATION

We prepared bilayers composed of a FM La$_{0.7}$Sr$_{0.3}$MnO$_3$ (LSMO) layer (selected for its weak anisotropy and small coercivity) covering an AFM orthorhombic o-YMnO$_3$ (YMO) layer grown on (100) SrTiO$_3$ substrates of area $5 \times 5$ mm$^2$ for samples A and B and $6 \times 6$ mm$^2$ for sample C. For the deposition a KrF excimer laser (wavelength 248 nm, pulse duration 25 ns) was used and the optimal parameters found for o-YMO were 1.7 J/cm$^2$ with 5 Hz repetition rate, 800°C and 0.10 mbar for the substrate temperature and oxygen pressure during preparation. We have measured three bilayers, all of them with the same 375 nm thick o-YMO layer on STO substrates prepared always under the above mentioned conditions. To check the reproducibility of the found effects we have prepared a fourth bilayer with identical thickness as in sample A but instead of the LSMO FM layer we used La$_{0.67}$Ca$_{0.33}$MnO$_3$ (LCMO) deposited YMO and this last one on a (100)LSAT substrate.

For the FM LSMO layer, deposited immediately after the o-YMO one, the parameters were 10 Hz repetition rate and 0.35 (0.38) mbar oxygen pressure, 8 (30) nm thickness and at the same laser fluency and substrate temperature, for sample A (B). In order to corroborate the contribution of the FM layer in the $M_E$-shift we have decreased further the oxygen concentration to deposit the LSMO film in sample C (oxygen pressure 0.10 mbar) with a larger thickness of 225 nm decreasing in this way its coercivity. For the fourth LCMO/YMO bilayer the YMO
layer was grown under similar conditions as before but
the LCMO layer under an oxygen pressure of 0.55 mbar;
all other conditions as for the LSMO layers.

The epitaxial growth in the 00l direction for the o-
YMO and 100 for LSMO phases was confirmed by x-ray
diffraction using Cu-K$_\alpha$ line. As an example we show in
Fig. 1 the the x-ray spectrum of the single o-YMO layer
on STO. The preferential growth of the (00l) planes of
the orthorhombic phase YMO is clearly seen. Within the
experimental resolution no maxima due to the hexagonal
phase are observed. Figure 2 shows the x-ray spectrum
obtained for sample B. The main diffraction peaks from
the LSMO layer are observed as a weak shoulder near the
STO main maxima. Magnetization measurements were
performed with a superconducting quantum interference
device (SQUID) from Quantum Design in the tempera-
ture range between 5 K and 350 K.

In addition, we performed soft x-ray absorption and
circular dichroism measurements using the bending mag-
et beamline 6.3.1 at the Advanced Light Source in
Berkeley, CA (USA) and the elliptical undulator beam-
line 13.1 at the Stanford Synchrotron Radiation Light-
source, Stanford, CA (USA). For these measurements the
sample was mounted between the poles of an electromag-
net so that the x-rays are incident on the sample under
a grazing angle of 30° parallel to the direction of the
applied magnetic field. The x-ray absorption intensity was
monitored using the electron yield method. Hysteresis
loops were acquired by sweeping the external field while
monitoring the electron yield at the Mn L$_3$ and L$_2$
absorption resonance ($\approx$ 640 eV). This approach is surface
sensitive and in general it yields information only on the
first $\sim$ 5 nm of the sample. Assuming an exponential
escape depth of 2.5 nm, then 95% of the signal comes
from the top 6 nm of the sample. This is essentially our
probing depth. For a more detailed description of the
 technique see Refs. 10 and 11.

III. RESULTS

A. Single YMnO$_3$ layers

According to literature the o-YMO phase is AFM
with Néel temperature $T_N = 42 \pm 2$ K and with a ferro-
electric transition at $\sim 31$ K. In spite of its low $T_N$ this
material has several advantages for exchange bias stud-
ies. It belongs to the family of the perovskite manganite
RMnO$_3$ and the magnetic and electrical properties can
be changed by cation substitution keeping similar lat-
tice constants and therefore without drastic changes in
its structural properties. On the other hand, o-YMO is
a phase that was not thoroughly studied yet and the in-
fluence of its ferroelectric behavior, in spite of the low
temperature, might be used as a paradigm for potential
applications in magnetoelectric devices.

Figure 3(a) shows the magnetization loop of single o-
YMO layer. The hysteresis loop indicates a magnetiza-
tion at saturation of 1.8 emu/cm$^3$ at 5 K and at applied
fields $\mu_0 H > 0.5$ T in agreement with reported values. Figure 3(b) shows the magnetic moment of a single o-
YMO layer ($6 \times 6 \times 0.375$ $10^{-3}$ mm$^3$) on STO measured
as a function of temperature in ZFC and FC states at
an applied field of 0.05 T. A clear increase in $m(T)$ de-
creasing temperature is observed at $T \approx 42$ K. An hys-
teresis between ZFC and FC is observed already below
$T \sim 60$ K. As was shown in earlier studies on YMO we may expect to have persistent spin waves at temperatures
above $T_N$.

From the hysteresis loop shown in Fig. 3(a) one may

![FIG. 1. X-ray spectrum of the single YMO AFM layer on STO substrate.](image1)

![FIG. 2. X-ray spectrum of the bilayer sample B. The labels indicate the corresponding the main diffraction peaks.](image2)
speculate that the YMO film behaves as a ferro- or ferrimagnet and not as an antiferromagnet. In fact, a recent study suggests a change of the usual bulk antiferromagnetic state to a strain-dependent non-collinear magnetic one in thinner (≤ 120 nm) o-YMO films. Taking into account that our YMO layers are much thicker and show a different m(T) behavior (at ZFC and low applied fields the measured m(T) of our YMO films alone resembles practically the usual T—dependence found for antiferromagnets) as those reported in Ref. 18 we remark that the magnetic behavior of the o-YMO layers may correspond to the one observed in diluted antiferromagnets in external magnetic field (DAFF). It is well known that DAFF develop a domain state when cooled below T_N (sometimes with a spin-glass-like behavior) and this leads to a net magnetization, which couples to the external field, see e.g. Refs. 4, 7, 19–21.

From the measured temperature dependence of the magnetic moment and the observed scaling of the exchange bias field H_E with the inverse of the thickness of the LSMO layer for samples A and B, see section II, and the quantitative agreement of the obtained H_E and M_E shifts for the fourth sample (similar to sample A but with LCMO instead of LSMO) we may conclude that YMO behaves as an AFM or DAFF layer for the exchange bias effects. Whatever the real magnetic equilibrium state of our o-YMO films is, we may expect to see exchange bias effects when these films are coupled to a ferromagnet. Further examples for exchange bias effects in heterostructures with different ferro- or ferrimagnets can be seen in Refs. 22 and 23 and H_E effects, positive as well as negative, has been also observed in ferrimagnetic based bilayers.

B. La_{0.7}Sr_{0.3}MnO_3/YMnO_3 bilayers

Figure 4 shows the remanent moment for samples A and B measured increasing temperature at zero field, after cooling them to 5 K in a field of 0.1 T applied in-plane, i.e. a or b direction. Changes in slope of the remanence moment are observed near the Neél temperature onset T_N ∼ 50 K of the o-YMO layer. This increase of ∼ 8 K in T_N might be related to an exchange-bias or strain effect. An anomaly is also observed at T ∼ 20 K, as shown in Fig. 3(b), and already reported in the literature. The temperature dependence of the remanence measured in sample B shows a clear change of slope near the Curie temperature of the LSMO layer. In contrast, due to the lower LSMO thickness the remanent moment of sample A does not show a clear anomaly at T_C; similarly for sample C (not shown). For sample C we show in Fig. 4 the field cooled (FC) curve at 0.1 T: the absence of a marked anomaly at T_C and the smooth decrease of the magnetic moment with T demonstrates the expected strong magnetic dilution of the LSMO film. The existence of the FM state in this layer was confirmed through hysteresis loop measurements up to its ferromagnetic onset at T_C ∼ 300 K. The FC results presented below were obtained always after cooling the samples from T > T_C at zero field and after applying an in-plane field H_{FC} at 100 K > T_N.

Figures 3(a) and (b) show the hysteresis loops for ZFC and FC measurements at 5 K for samples A and B. A remarkable M_E shift of the same order of the saturation magnetic moment m_s is observed for sample A after FC from 100 K at μ_0H_{FC} = 0.5 T. For sample B the M_E shift is also clearer measured but it is smaller relative to m_s. The sign of the M_E-shift changes when the direction of H_{FC} changes, i.e. it has the same sign as that of H_{FC}. This indicates that the effective UCM layer is pinned in the direction of the cooling field, which means a ferromagnetic coupling.

In the determination of the M_E and H_E shifts we took special care to rule out effects due to minor hysteresis loops. Studying the behavior of the loops at different H_{FC} we conclude that no minor loops and a clear sat-
ulation behavior of the magnetic moment are obtained for $\mu_0H_{\text{FC}} \geq 0.2$ T at $T \geq 5$ K for samples A and B, see Fig. 4. For sample C, which has a more diluted and inhomogeneous FM layer, the hysteresis loops reveal no complete saturation at $\mu_0H_{\text{FC}} < 0.4$ T. However minor loop effects can be neglected also for this sample at $\mu_0H_{\text{FC}} \geq 0.2$ T, as the behavior of the coercive field $H_c$ vs. $H_{\text{FC}}$ indicates (see Fig. below).

We note that the value of $m_s$ obtained from the hysteresis loops depends on the applied $H_{\text{FC}}$. As example we show this effect for sample B where the hysteresis loop was measured after cooling the sample at $\mu_0H_{\text{FC}} = 2$ T, see Fig. 4b). This effect is due to the LSMO layer and indicates that the number of aligned domains can be changed with $H_{\text{FC}}$. In this case we expect that the $M_E$ effect will be strongly influenced by the FM layer since, as in the case of a diluted AFM layer, the formation and number of its domains that take part in the exchange bias coupling with the AFM layer can be enhanced leading to an increase of $M_E$. Note however that the $M_E$ effect is expected to decrease with $H_{\text{FC}}$, i.e. $M_E \to 0$ for $H_{\text{FC}} \to \infty$.

Note the opening of $\sim 1$ $\mu$emu of the hysteresis at the end of the loop at 0.5 T for sample A, see Fig. 4a). A similar opening is measured for all samples in agreement with the numerical results obtained with the domain state model for exchange bias proposed by Nowak et al.\textsuperscript{9,10}. The fact that the loops do not close indicates that uncompensated spins - pinned earlier during the field cooling - rotate and remain pinned in the opposite direction during the field sweep loop, reducing the final saturation moment. We note that in all three bilayers this opening remains of the order of 1…2 $\mu$emu, i.e. several times smaller than the $M_E$ shift, as we show below.

To characterize quantitatively the exchange bias $M_E$ effect and for a direct comparison with the saturation magnetic moments of each of the layers we define it as $m_{\text{shift}} = (m_{E}^+ + m_{E}^-)/2$, where $m_{E}^+$ and $m_{E}^-$ are the saturation moments at positive and negative fields. The shift in the field axis is defined as $H_E = (H_1^+ + H_1^-)/2$, where $H_1^+$ and $H_1^-$ are the coercive fields in upward and descending loop branches, respectively. We note that the $H_E$ values were obtained only after centering the hysteresis loop, subtracting the upward $M_E$ shift.

Figure 5 shows the coercivity $H_c$ (a), the exchange-bias $H_E$ (b) and the vertical shift in magnetic moment $m_{\text{shift}}$ (c) as a function of $T \leq 80$ K for sample B, measured after $\mu_0H_{\text{FC}} = 0.3$ T, as an example. A similar behavior is observed for samples A and C. Both, $H_C$ and $H_E$ show an anomaly at $T \lesssim 20$ K, in agreement with the behavior found in the remanence curve, see Fig. 4, suggesting that the transition at that temperature influences the exchange interaction. At $T \gtrsim 35$ K $H_E$ crosses zero and changes to positive. This sign change of $H_E$ from negative to positive increasing temperature was observed also in CoO/Co bilayers\textsuperscript{20} and suggests a change from direct ($J_{\text{interface}} > 0$) to indirect ($J_{\text{interface}} < 0$) interface inter-
action. As expected, $H_E(T)$ as well as $m_{\text{shift}}$ vanish at $T \gtrsim T_N$. In contrast to $H_E(T)$ no anomalous behavior is observed in $m_{\text{shift}}(T)$ at $T < T_N$, with exception of the slope change at $T \sim 20$ K, see Fig. 6(c).

Figure 7 shows the $H_{FC}$-dependence of $H_c$, $m_{\text{shift}}$ and $H_E$ for the three samples measured at 5 K. The decrease of $H_E$ from samples A to B agrees with the expected inverse proportionality of $H_E$ with the thickness of the FM layer. According to this thickness dependence sample C should show nearly one order of magnitude smaller $H_E$ than for sample B, in clear disagreement with the obtained result, see Fig. 7(c), suggesting that the magnetic dilution of this sample is responsible for the large observed $H_E$ field.

Regarding the $M_E$ effect and in agreement with the results in Co/CoO bilayers we observe a vanishing effect at zero and at large enough values of $H_{FC}$, see Fig. 7(b). Under the assumptions done in Refs. 8 and 19 the $M_E$ shift is mainly due to the AFM layer. According to this model, the largest $m_{\text{shift}}$ expected from our o-YMO layer, assuming complete saturation in the whole 375 nm thick layer, would be $m_{\text{YMO}} = 17 \mu$emu and 24.5 $\mu$emu from samples A or B and C, respectively. To estimate those numbers we have taken into account the measured magnetization at saturation of the single layers. The normalized $m_{\text{shift}}$ by the corresponding $m_{\text{YMO}}$, see Fig. 7(b), would indicate that it is necessary that 50% to 70% of the YMO layer should be responsible for the measured $m_{\text{shift}}$ at $H_{FC} \sim 0.5$ T. This percentage increases further for the diluter sample C at 0.2 $T \leq H_{FC} \leq 0.4$ T. Taking into account the 375 nm thickness of the YMO layer this assumption appears unlikely.

We remark that unexpected phenomena can occur at oxide interfaces. A recent study, for example, found an excess magnetization produced at the interface between...
STO and an AFM La$_{1/3}$Ca$_{2/3}$MnO$_3$ layer\cite{30} which origin remains unclear. In our case the large $m_{\text{shift}}$ values – actually a giant $M_E$ effect – indicate that a large contribution should come from the FM layer. Taking into account the saturation moments of the LSMO layers alone, we estimate for example that a thickness of the LSMO layer of less than 1.3 nm for sample B and < 10 nm for sample C should be enough to produce the observed $m_{\text{shift}}$ at $H_{\text{FC}} = 0.5$ T.

C. La$_{0.67}$Ca$_{0.33}$MnO$_3$/YMnO$_3$ bilayer

Further evidence for the reproducibility and robustness of the effects observed in the three LSMO/YMO bilayers reported in the last section are provided by the results of a LCMO/YMO bilayer with similar geometry and preparation conditions as sample A. Figure 8(a) shows the remanent magnetic moment of this bilayer after cooling the sample at 1 T applied field. The transition at the Néel temperature of the YMO layer is clearly seen as well as the change of slope at $\sim 20$ K. In Fig. 8(b) the hysteresis loops for three field cooled states at fields $H_{\text{FC}} = \pm 1$ T and 2 T are shown. At low $H_{\text{FC}}$ fields the exchange bias $M_E$- and $H_E$-effects are clearly observed whereas at high enough fields the $M_E$ effect vanishes, see Fig. 8(b). Figure 9 shows the $H_{\text{FC}}$-dependence for the three characteristics parameters. The observed $m_{\text{shift}}$ at $H_{\text{FC}} \leq 1$ T, see Fig. 9(b), is as large as the magnetic moment at saturation of the 375 nm thick YMO layer alone, indicating clearly that the FM layer should contribute to this effect near the interface.

Although in the LSMO/YMO bilayers we did not find any correspondence between the coercive field $H_c(H_{\text{FC}})$ and $m_{\text{shift}}(H_{\text{FC}})$, see Fig. 7, one may expect some correlation between them in case of a bilayer with a very thin (and diluted) FM layer. This may be so if we take into account the amount of the FM layer that remains pinned at the interface. In this case the smaller the effective thickness of the remained unpinned ferromagnetic layer the smaller might be $H_c$. Apparently this is observed in the (thin)LCMO/(thick)YMO bilayer. Indeed, the results shown in Fig. 7(d) indicate that when $m_{\text{shift}}$ decreases at $H_{\text{FC}} > 0.25$T, i.e. when the amount of UCM decreases, $H_c$ increases.

IV. DISCUSSION AND CONCLUSION

To further corroborate our conclusion that the observed vertical shift is mainly due to the FM and its interface region with the AFM layer we show the hysteresis loops acquired using x-ray magnetic circular dichroism in Fig. 11. For sample A we find a shift of about 5% using the surface sensitive approach measuring the response of the Mn ions within the LSMO FM layer only. The observed vertical shift is a clear indication that the FM layer is contributing to the $M_E$ effect and that the shift is not confined to the bulk of the AFM. Assuming that 95% of the secondary electrons detected in our experiment originate from the top 6 nm\cite{10,11}, we can conclude that the interfacial region of the FM/AFM layer contributes significantly more to the $m_{\text{shift}}$ compared to the surface layers of the FM. This result agrees with the estimate from the bulk SQUID measurements that one needs about 1 nm thick FM layer (for samples A as well as B) to account for the observed $m_{\text{shift}}$. Taking into account the previous statement that it is highly unlikely that the entire AFM bulk contributes to the shift we can conclude that the excess magnetization is produced predominantly at the FM interface during the field cooling process due to interfacial exchange coupling between the AFM and the FM as shown previously for the case of Co/FeF$_2$\cite{11}.

Using similar arguments on the importance of the magnetic dilution of the AFM layer\cite{9,19}, we argue that in our system the dilution of the FM layer may play a mayor role in the $M_E$ shift. In other words, the robust AFM
layer influences the magnetic behavior of the FM one, within a certain thickness from the interface. We note that some kind of $M_E$-shift were recently reported for ferromagnetic very thin hard/soft (3nm/12nm) DyFe$_2$/YFe$_2$ heterostructures. However, in that work the $M_E$ effect is in opposite direction to that of the applied $H_{FC}$, in contrast to our observations.

Furthermore, a comparison between the overall behavior obtained for $m_{\text{shift}}(H_{FC})$ and $H_E(H_{FC})$ indicates that there is no simple correlation between the two exchange bias effects. Note that $H_E$ decreases strongly from sample A to B, whereas $m_{\text{shift}}$ increases. Although element selective x-ray magnetic measurements would help to determine the penetration depth of the UCM in each of the layers, it is clear from our SQUID measurements that the o-YMO layer alone cannot be the reason for the observed giant $M_E$ effect, this is not the main message of our work.

In conclusion, our studies on LSMO/o-YMO bilayers and on a single LCMO/o-YMO bilayer found large uncompensated $M_E$ shifts, whose sign correlates with the direction of the cooling field $H_{FC}$. Both, the exchange-bias $H_E$ and $M_E$ effects, vanish near $T_N$ of the YMO layer. The large $m_{\text{shift}}$ values indicate that the AFM layer cannot be the only responsible but a certain thickness of the FM layer near the interface. This behavior can be actually understood taking similar arguments as those used for the AFM layer in the domain state exchange-bias model of Refs. 9 and 19. Tuning the thickness and magnetic dilution of the FM layer one should be able to obtain large $M_E$ shifts making it an effect worth to study in systems with $T_N > 300$ K. The different behaviors of $H_E$ and $M_E$ with temperature, cooling field and FM layer thickness indicate that these two phenomena are not correlated in a simple way.

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