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2014

Tian, Y. F., Lebedev, O. I., Roddatis, V. V., Lin, W. N., Ding, J. F., & Hu, S. J., et al. (2014).
Interfacial magnetic coupling in ultrathin all-manganite $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3\text{-TbMnO}_3$
superlattices. *Applied Physics Letters*, 104(15), 152404-.

<https://hdl.handle.net/10356/104121>

<https://doi.org/10.1063/1.4871701>

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Citation: *Applied Physics Letters* **104**, 152404 (2014); doi: 10.1063/1.4871701

View online: <http://dx.doi.org/10.1063/1.4871701>

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Interfacial magnetic coupling in ultrathin all-manganite $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3\text{-TbMnO}_3$ superlattices

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(Received 5 November 2013; accepted 6 April 2014; published online 16 April 2014)

We report the growth and magnetic properties of all-manganite superlattices composed of ultrathin double-exchange ferromagnetic $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ and noncollinear multiferroic TbMnO_3 layers. Spontaneous magnetization and hysteresis loops are observed in such superlattices with individual $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ layers as thin as two unit cells, which are accompanied by pronounced exchange bias and enhanced coercivity. Our results indicate substantial interfacial magnetic coupling between spin sublattices in such superlattices, providing a powerful approach towards tailoring the properties of artificial magnetic heterostructures. © 2014 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4871701>]

The rapid advancements of modern thin film growth techniques have enabled the exploration of a wide range of epitaxial ultrathin oxide films and heterostructures with emergent functionalities. The long-range collective behaviors of spins in magnetic materials are well known to evolve with the film thickness, and the ground states in ultrathin films are results of energetic competition between surface, interface and dipolar anisotropies.^{1–3} Transition metal oxides offer rich phase diagrams with fascinating magnetic orders as a result of the coupling between multiple degrees of freedom.⁴ Particularly, mixed-valence manganites have attracted lots of interest after the discovery of colossal magnetoresistance (CMR) effect.^{5,6} However, it has been extensively reported that Curie temperature and magnetic moment of the ferromagnetic (FM) manganite films decreases as the films become thinner, which has been ascribed to both extrinsic and intrinsic effects such as structural and compositional inhomogeneities, interfacial charge transfer, and strain-induced orbital reconstruction.^{7–11} In a flurry of recent studies, ultrathin manganite films were incorporated in epitaxial heterostructures as a result of expectation of emergent physics such as tailored interfacial magnetic coupling and geometrically confined doping in such artificial low-dimensional systems.^{12–15} Epitaxial superlattices (SLs) of complex oxides give access to fascinating physical phenomena as a result of magnetic frustration, charge transfer, and orbital reconstruction across interfaces.^{16–22} In particular, exchange bias, as a prototypical interfacial magnetic interaction between different spin orders, was reported in some manganite-based heterostructures and SLs.^{23–29}

Unlike CMR manganites such as $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ (LSMO) where double exchange interactions lead to collinear ferromagnetism and metallic states, rare-earth manganite TbMnO_3 (TMO) offers multiferroic properties, coupling ferroelectric and magnetic orders.^{30–32} As a result of the competing exchange interactions, TMO displays sinusoidal antiferromagnetic Mn^{3+} spin ordering at the Néel temperature $T_N \sim 41$ K, spiral Mn^{3+} spin ordering at the ferroelectric transition temperature $T_{\text{lock}} \sim 28$ K, and long range ferromagnetic Tb^{3+} spin ordering below $T_{\text{Tb}} \sim 7$ K. Orthorhombic-structured TMO is compatible with other perovskites, and epitaxial thin films have been fabricated.^{33,34} Recently, TMO-based heterostructures including rectifying junctions and manganite bilayers were investigated.^{35–37} The correlation effects involving multiple degrees of freedom are expected to be enhanced at interfaces in ultrathin heterostructures. However, so far there has been no report on the fabrication and characterization of TMO-based SLs.

In this work, we fabricated a series of epitaxial manganite SLs composed of ultrathin LSMO and TMO layers, and studied their magnetic properties. Remarkably, sizable low-temperature magnetism with hysteresis was stabilized in the SLs down to a LSMO layer thickness of two unit cells (u.c.), which is accompanied by drastically enhanced coercive field of more than 3000 Oe. Furthermore, exchange bias was observed in the SLs, which can be attributed to the strong magnetic coupling across the LSMO-TMO interface.

Epitaxial $[\text{m/n}]_x$ SLs (as schematically shown in Fig. 1(a)), consisting of m u.c. of LSMO and n u.c. of TMO, repeated x times, were grown on TiO_2 -terminated (001) SrTiO_3 (STO) single-crystal substrates using pulsed laser deposition (PLD) technique assisted by reflection high-energy electron diffraction (RHEED). The growth took place at 750 °C under an oxygen pressure of 0.05 millibars. A laser fluence of $\sim 1.5 \text{ J/cm}^2$ was

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FIG. 4. (a) and (b) Magnetic hysteresis loops of various LSMO-TMO SLs measured at 10 K. Here, the magnetic moments are normalized to the whole volumes of the samples (a) or to the LSMO unit cell (b). (c) The ZFC and FC M-T curves for various SLs measured at 200 Oe. (d) Coercivity (H_C , dashed bars) and exchange bias field (H_E , solid bars) of various SLs.

we did not fabricate any SL with layers of single u.c. because of the cation mixing at the interfaces cannot be completely excluded. Assuming the magnetic signal solely comes from the LSMO layers, we re-plot the magnetization data in Figure 4(b) with a unit of $\mu_B/u.c.$ and the saturation magnetization values of all the SLs are roughly at the same level. In general, the saturation magnetization of the SLs is lower than the value of $3.7 \mu_B/u.c.$ expected for bulk LSMO, which indicates suppression of collinear spin ordering in the LSMO-TMO SLs. Nevertheless, the stabilization of magnetic order with hysteresis in such ultrathin LSMO-TMO SLs is quite remarkable, considering that the critical LSMO layer thickness to exhibit ferromagnetism is 3 u.c. for LSMO single layers.⁷ Symmetry breaking, charge transfer, and spin frustration at the LSMO-substrate and LSMO-air interfaces are often cited to account for the suppressed magnetism in ultrathin LSMO films.^{7,11,38} At the LSMO-TMO interface, the Mn ions interact not only with each other like in the double-exchange LSMO but also with the spin sublattices in TMO. Particularly, in the $[2/n]_x$ SLs, all the magnetic Mn ions in LSMO interface with the TMO layers. The proximity of TMO apparently plays the decisive role in stabilizing the spin alignment in the LSMO layer. Figure 4(c) shows the ZFC-FC M-T curves for the SLs, where similar behavior of bifurcation between ZFC and FC curves was observed. Such bifurcation was previously reported for both manganite heterostructures^{35,39} and phase-separated manganite films.^{40,41} In our SLs, it likely comes from the magnetic frustration at the LSMO-TMO interfaces.

Careful examinations of the hysteresis loops reveal the emergence of exchange bias H_E together with a giant enhancement of coercivity H_C in the LSMO-TMO SLs. Here, H_C is defined as $H_C = |H_+ - H_-|/2$, where H_+ and H_- denote the right and left coercivity field, respectively. As shown in Figure 4(d), among the SLs we measured the $[4/8]_{16}$ sample shows the highest exchange bias of ~ 170 Oe at 10 K. Its value of H_C at 10 K is ~ 3200 Oe; in comparison, H_C is only ~ 90 Oe for 8 u.c. LSMO thin films.³⁵ A similar

effect of coercivity enhancement was previously reported in other manganite-based heterostructures such as LSMO/La₂CuO₄ bilayer (705 Oe at 5 K)³⁹ and LSMO/BiFeO₃ bilayer (850 Oe at 7 K).²⁴ This observation of the enhanced magnetic anisotropy in LSMO-TMO SLs unambiguously underscores the significant exchange interaction and spin pinning across the LSMO-TMO interfaces.

Considering that the exchange bias is highest in the $[4/8]_{16}$ LSMO-TMO SL, we measured its temperature dependent magnetic properties. The data in Figure 5(a) reveal that the irreversible temperature decreases with increasing magnetic field, which are consistent with the existence of spin frustration.^{26,39} Figure 5(b) shows the magnetization vs. magnetic field data measured from 5 to 300 K. Both coercivity and exchange bias quickly decreases on increasing temperature, indicating weakened exchange coupling in the SLs due to thermal excitation.

More insights on the magnetic interactions in LSMO-TMO SLs can be obtained by examining the temperature dependence of H_C and H_E as shown in Figure 6. Remarkably, the exchange bias emerges near the T_N of bulk TMO. In macroscopic TMO single crystals, sinusoidal antiferromagnetic Mn³⁺ spin ordering occurs at $T_N \sim 41$ K, which however may not fully develop in ultrathin SLs with TMO layers of a few u.c. due to the symmetry breaking and nanoscale confinement. Nevertheless, the temperature dependence shown in Figure 6 suggests that the strong magnetic interaction and spin pinning only appear at low temperatures, and the onset temperature is intriguingly close to the T_N of bulk TMO. In addition, as shown in Figure 6(a), H_E shows the exponential temperature dependent decay following a phenomenological expression: $H_E = H_E^0 \exp(-T/T_0)$. This exponential temperature dependence has previously been observed in diverse systems such as manganite nanoparticles, bilayer, and superlattices,^{26,29,42,43} which can be ascribed to the competing magnetic interactions and spin frustration at the interfaces. In contrast, H_C deviates from this exponential law at low temperatures (Figure 6(b)),

FIG. 5. (a) The ZFC and FC M - T curves for the [4/8]₁₆ SL measured under different magnetic fields after correcting the diamagnetic substrate contribution. (b) Hysteresis loops for the [4/8]₁₆ SL measured at different temperatures.

FIG. 6. Temperature dependence of the exchange bias field H_E (a) and the coercivity H_C (b) measured on the [4/8]₁₆ SL. The red solid line in (a) is exponential theoretical fitting to H_E .

suggesting that H_E and H_C in the low-temperature regime do not share exactly the same origin.

It is insightful to compare the magnetic properties of LSMO-TMO SLs with those of bilayers. Since SLs contain multiple interfaces, much more than the single interface in bilayers, we expect stronger interfacial magnetic coupling in SLs. Indeed, in the LSMO (8 u.c.)-TMO (40 nm) bilayer, H_E is 42 Oe and H_C is 1200 Oe at 5 K.³⁵ On the other hand, in the [4/8]₁₆ SL sample, both H_E and H_C are greatly enhanced, i.e., H_E is found to be 320 Oe and H_C is 3320 Oe at 5 K. The remarkable enhancement illustrates a stronger interfacial exchange interaction in the SLs compared to the bilayer counterparts. This clearly underscores ultrathin SLs as a powerful approach towards achieving effective exchange coupling in manganite heterostructures. The fact that the largest exchange bias and coercivity enhancement was observed in the [4/8]₁₆ SL suggests that a certain layer thickness of TMO is needed to achieve effective exchange coupling at the TMO-LSMO interfaces. On the other hand, thicker TMO layers are harder to grow in the layer-by-layer mode, and the rougher interfaces may result into the lower saturation magnetization. For example, after the saturation magnetization is normalized according to the total LSMO thickness, the value of the [4/4]₅ SL ($2.93 \mu_B/\text{u.c.}$) is higher than that of [4/8]₁₆ SL ($2.25 \mu_B/\text{u.c.}$). Furthermore, nanoscale domains were observed in TMO thin films,⁴⁴ and their interactions with the magnetic domains in LSMO will be investigated in the future.^{45,46}

In summary, the emergence of significantly enhanced coercivity and exchange bias in all-manganite LSMO-TMO SLs underscores such unit-cell-controlled heterostructures as a viable route towards tailored artificial magnetic materials. It is quite remarkable that sizable magnetization with hysteresis can be stabilized in LSMO-TMO SLs with the layers being as thin as two u.c. The competing exchange coupling mechanisms within the spin sublattices are responsible for the observed rich magnetic behaviors. We believe that insights on the magnetic phenomena in such artificial heterostructures and advancements in designing interfacial coupling will lead to technological breakthroughs in exploring future magnetic materials.

Y.F.T. and S.S.Y. acknowledge the support from NSF No.51125004, 111 Project No. B13029, the National Basic Research Program of China No. 2013CB922303 and IIFSDU. Authors are grateful to Professor A. L. Chuvilin for using a Helios 600 DualBeam instrument in CIC NanoGUNE and FIB specimen preparation.

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