Charge transfer and interfacial magnetism in (LaNiO$_3$)$_n$/LaMnO$_3$$_2$ superlattices

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(Dated: January 31, 2013)

(LaNiO$_3$)$_n$/LaMnO$_3$$_2$ superlattices were grown using ozone-assisted molecular beam epitaxy, where LaNiO$_3$ is a paramagnetic metal and LaMnO$_3$ is an antiferromagnetic insulator. The superlattices exhibit excellent crystallinity and interfacial roughness of less than 1 unit cell. X-ray spectroscopy and dichroism measurements indicate that electrons are transferred from the LaMnO$_3$ to the LaNiO$_3$, inducing magnetism in LaNiO$_3$. Magnetotransport measurements reveal a transition from metallic to insulating behavior as the LaNiO$_3$ layer thickness is reduced from 5 unit cells to 2 unit cells and suggest a modulated magnetic structure within LaNiO$_3$.

PACS numbers: 68.65.Cd, 73.21.Cd, 75.47.Lx, 81.15.Hi

I. INTRODUCTION

In recent years, there has been a great amount of interest in the novel electronic and magnetic states that emerge at interfaces between dissimilar complex oxide materials. In the most widely studied systems, these new behaviors arise as a result of interfacial charge redistribution and a resulting reconstruction of the orbital and spin degrees of freedom. The interfacial charge redistribution can arise in several ways. For example, at the widely studied LaAlO$_3$ (polar)/SrTiO$_3$ (non-polar) interface, this is said to result from a polar discontinuity. Charge redistribution may also result from differences in chemical potential across an interface, as in LaTiO$_3$/SrTiO$_3$. In magnetic systems, such as short-period LaMnO$_3$/SrMnO$_3$ superlattices, “charge leakage” between neighboring Mn$^{3+}$/Mn$^{4+}$ sites can give rise to intermediate valency and a ferromagnetic, metallic ground state at the interface. Interfacial magnetism is linked to the charge state and bonding between the nearest neighbor B-site transition metal cations, and is very sensitive to the details of the structure. In stoichiometric oxide heterostructures requires precise control and understanding of the interface charge states.

In this work, we examine the role of charge transfer in a series of digital superlattices that combine metallic LaNiO$_3$ with insulating antiferromagnetic LaMnO$_3$. In this model system, each BO$_2$ atomic plane ($B$ = Mn, Ni) is sandwiched between two identical LaO layers. Through x-ray spectroscopy measurements we show the Mn to be in a 4+ oxidation state, while that of Ni is intermediate between 2+ and 3+, showing conclusively the presence of a charge transfer at the [001] LaNiO$_3$/LaMnO$_3$ interface. The superlattices are found to have a net magnetic moment, with the magnetism residing on both the Mn and Ni sites. We also observe a transition from metallic transport to insulating behavior as the thickness of the LNO layer is reduced below 4 unit cells. These results, combined with magnetotransport measurements, point to the existence of a metallic state with inhomogeneous magnetism in LaNiO$_3$ with a mixture of Ni valence states.

II. MATERIALS AND EXPERIMENTAL

LaMnO$_3$ (LMO) has been extensively investigated as the parent compound of the prototypical perovskite colossal magnetoresistive (CMR) oxides. In stoichio-
metric bulk LMO with \( t_{2g}^3e_g^1 \) occupancy, a cooperative Jahn-Teller distortion lifts the degeneracy of the half-filled \( e_g \) band, producing an orbitally-ordered \( A \)-type antiferromagnetic insulating ground state. In thin films, however, LMO often exhibits ferromagnetism with a Curie temperature of \( \sim 150 \) K and a saturation moment close to \( 4 \mu_B/\text{Mn} \) due to cation deficiency and strain. The LMO investigated here shows insulating behavior with a gap of \( \sim 125 \) meV \( \mu_B \) a low saturation moment, and high coercivity, consistent with an antiferromagnetic spin arrangement. \( \text{LaNiO}_3 \) (LNO), on the other hand, is a paramagnetic metal where the \( \text{Ni}^{3+} \) ion adopts a low spin, orbitally degenerate \( t_{2g}^5e_g^1 \) electronic configuration. Strong mixing between the \( d^7 \) and \( d^8L \) \( (L \) denotes a ligand hole on the oxygen) configurations is expected, as LNO is significantly more covalent than divalent nickel compounds. The introduction of \( \text{Ni}^{2+} \) through oxygen deficiencies or Ce-doping has previously been shown to increase the resistivity of LNO and induce a transition to an insulating state. Under our growth conditions, the resistivity of an 80 unit cell thick LNO film showed metallic behavior, with \( \rho(T = 5 \text{ K}) = 35 \) \( \mu\Omega \cdot \text{cm} \), comparable to the lowest value for stoichiometric bulk samples.

Epitaxial \([\text{LNO}_n/\text{LMO}_m]_n\) superlattices with \( 2 \leq n \leq 5 \) unit cells were grown on \((001)\) \( \text{TiO}_2 \)-terminated \( \text{SrTiO}_3 \) single-crystal substrates using ozone assisted molecular beam epitaxy (MBE). The total heterostructure thickness was kept constant at \( \sim 80 \) unit cells (\( ~30 \) nm) by adjusting the stacking periodicity, \( m \). Elemental materials were evaporated sequentially from effusion cells using a block-by-block technique described previously under an \( \text{O}_2 \) partial pressure of \( 2 \times 10^{-6} \) Torr, with the substrate maintained at 690°C. After growth, x-ray reflectivity (XRR) was used to confirm the thickness and determine the interfacial and surface roughnesses; the crystal structure and epitaxy were investigated with x-ray diffraction (XRD). The in-plane resistivity was measured as a function of temperature in a four-point geometry for \( 2 \text{ K} \leq T \leq 300 \) K, while the Hall coefficient was measured using the Van der Pauw method in fields of up to 9 T. Measurements of the net sample magnetization were carried out as a function of temperature and magnetic field using a superconducting quantum interference device (SQUID) magnetometer, while cation specific magnetic properties were investigated using x-ray magnetic circular dichroism (XMCD). To determine the electronic properties, the superlattices were examined with x-ray absorption spectroscopy (XAS) in the soft x-ray regime at beamline 4-ID-C of the Advanced Photon Source. The spectra were measured in both the surface-sensitive total electron yield and bulk-sensitive fluorescence yield modes, and were aligned to a NiO (Ni\(^{2+}\)) standard measured simultaneously with the superlattice samples.

FIG. 1. (Color online) X-ray reflectivity (a) and high-resolution x-ray diffraction (b) scans for a series of \([\text{LNO}_n/\text{LMO}_m]_n\) superlattices on \( \text{SrTiO}_3 \).

### III. RESULTS

In order to obtain the intrinsic properties of the LMO/LNO interfaces, we require these to be atomically sharp with precise control of the stacking periodicity. The thickness and lattice parameters of our samples were determined by x-ray reflectivity and high-resolution x-ray diffraction, as shown in Figs. 1(a) and 1(b), respectively. Pronounced Bragg reflections indicate the interfaces are abrupt, as confirmed by fitting of the reflectivity curves using the Parratt formalism, which also showed the superlattice periods to typically be within 1% of the nominal thicknesses. We observe a “double-peak” structure in the XRR scan for the \( n = 4 \) sample, where the superlattice density modulation is not an exact integer number of unit cells, and for this sample estimate the error in stacking periodicity to be around 3%. The average c-axis lattice constant is obtained from the \( 002 \) scans performed around the (002) reflection as shown in Fig. 1(b) and is found to decrease monotonically with increasing LNO thickness from 0.385 nm for \( n = 2 \) to 0.382 nm when \( n = 4 \) in contrast with \( (\text{LaNiO}_3)_n/(\text{SrMnO}_3)_2 \) superlattices, where the largest out-of-plane lattice parameter was found for \( n = 4 \).

Figure 2(a) shows the temperature dependence of the magnetization of the four superlattices in addition to LMO and LNO films. The magnetization was measured while warming the samples in a field of 500 Oe applied in the plane of the film after field cooling in 500 Oe (solid symbols) and zero field cooling (open symbols). On lowering the temperature, all of the samples except the LNO film, exhibit a rise in magnetization below a nominal transition temperature \( T_c \sim 150 \) K, corresponding to the onset of magnetic order. Hysteresis loops (Fig. 2(b)) measured along the [100] direction at 5 K displayed a systematic reduction in coercive field with increasing \( n \), as shown in the inset to Fig. 2(b). The pure LNO film is antiferromagnetically ordered, as evidenced by the large coercivity and small saturation moment, \( M_s < 0.2 \).
\( \mu_B/\text{Mn} \).

To probe the microscopic origins of the observed metal-insulator transition and magnetic ordering, we carried out both x-ray absorption spectroscopy (XAS) and x-ray magnetic circular dichroism (XMCD) measurements near the Mn and Ni \( L_{2,3} \) absorption edges. Measurements were performed in fields of up to 5 T applied in the plane of the sample. Both bulk-sensitive fluorescence yield and surface-sensitive total electron yield configurations were found to give similar results. XAS spectra measured at \( T = 15 \) K are shown in Figs. 3(a) and (b) for the superlattices with \( n = 2 \) and \( n = 4 \), along with spectra for Mn\(^{3+} \) and Mn\(^{4+} \) from Ref. [24] and measured reference spectra for Ni\(^{2+} \) and Ni\(^{3+} \). We find that for both the \( n = 2 \) and \( n = 4 \) superlattices the valence of Mn is nearly Mn\(^{4+} \), unlike in bulk LMO, where Mn is in the 3+ oxidation state. The Mn\(^{4+} \) ion that results has an empty \( e_g \) manifold as in CaMnO\(_3\), which is expected to quench the Jahn-Teller distortion and may give rise to \( G \)-type antiferromagnetic ordering. The Ni valence is close to Ni\(^{2+} \) in the \( n = 2 \) structure, while signatures of both Ni\(^{2+} \) and Ni\(^{3+} \) are found in the superlattice with 4 layers of LNO. The Ni \( L_3 \)-edge spectra is similar to that reported for bulk \( R \)NiO\(_3\) materials below the metal-insulator transition temperature,[23] and for short-period LaNiO\(_3\)/LaAlO\(_3\) superlattices[29,30] where the two-peak feature was attributed to the formation of charge-ordered states. In our samples, we are unable to distinguish this scenario from a simple mixed valence picture without charge-ordering. The results suggest that each Mn donates one electron to a nearby Ni cation at the interface, as in the double perovskite La\(_2\)NiMnO\(_6\).[28] The Ni\(^{2+} \) is expected to have a doubly-degenerate, high-spin (\( S = 1 \)) \( e_g \) manifold, with antiferromagnetic coupling to neighboring Ni\(^{2+} \) cations, as in La\(_2\)NiO\(_4\). The competing exchange interactions at the interface may result in a frustrated magnetic state, as recently proposed to explain the exchange-bias effects observed in La\(_{0.75}\)Sr\(_{0.25}\)MnO\(_3\)/LaNiO\(_3\) superlattices.[29]

From the amplitude of the XMCD signal, we are able to estimate the net magnetization of the Mn and Ni cations in the superlattices with \( n = 2 \) and \( n = 4 \), as the magnetic field is varied from 0.1 T to 5 T, with the temperature held constant at 15 K (Figs. 3(c) and (d)). We find that the saturation Mn magnetization in both samples is enhanced compared to the pure LMO film (Fig. 2(b)), and the measured value of \( \sim 2 \mu_B/\text{Mn} \) is inconsistent with purely antiferromagnetic ordering in the LMO, though the Mn is in a \( 4+ \) state. This may occur by several mechanisms. We note that recent experimental work...
has shown that weak ferromagnetism can arise through phase separation in lightly electron-doped CaMnO$_2$. Also, tensile strain has been shown theoretically to favor an A-type antiferromagnetic arrangement that may be susceptible to canting, leading to a net magnetization. We also find a net moment on the Ni cations, in agreement with Ref. 29 which we estimate to be 0.35 $\mu_B$/Ni at 5 T for both $n = 2$ and $n = 4$. From the shapes of the Mn and Ni $L_{2,3}$-edge spectra, we determine that the net moment of LMO and LNO layers are coupled ferromagnetically. To check the internal consistency of our magnetization estimates, we have computed the net magnetization of the superlattices by weighting the magnetization of the individual cations, as shown by the solid squares in Figs. 3(c) and (d). We find good agreement between the net magnetizations obtained from XMCD measurements and from SQUID magnetometry measurements (open circles in Figs. 3(c) and (d)).

Figure 4(a) shows the variation of the resistivity of the LNO/LMO superlattices and LNO and LMO thin films as a function of temperature. In previous studies of LaNiO$_3$/LaAlO$_3$, LaNiO$_3$/SrTiO$_3$, and LaNiO$_3$/SrMnO$_3$ superlattices, a transition from metallic to insulating behavior was observed as the thickness of the LNO layer was reduced below 3 unit cells; the same thickness we find here. The fact that this transition occurs at the same thickness here is surprising, as the average valence of the nickelate layer is lower due to charge transfer observed at the LMO/LNO interface. The resistivity of the $n = 2$ superlattice is well described by a two-dimensional variable-range hopping (VRH) model (Fig. 4(b)) given by,

$$\rho = \rho_\infty \exp \left[ \frac{(T_0/T)^{1/3}}{\xi} \right],$$

where $T_0$ is a characteristic temperature related to the density of states at the Fermi energy, $N(\xi_F)$, and the localization length, $\xi$, by

$$T_0 \approx \frac{13.8}{k_B N(\xi_F) \xi^2}.$$  

Two-dimensional VRH has previously been observed in ultrathin (5 unit cell) LNO films and (LaNiO$_3$)$_2$/SrMnO$_3$ superlattices. In the $n = 2$ sample, $T_0 \approx 1.31 \times 10^6$ K and the mean hopping energy $\Delta E \approx 0.28 k_BT_0^{1/3} T^{2/3}$, exceeds $k_BT$ over the entire temperature range studied (60 K $\leq T \leq 300$ K). The density of states in bulk LNO is $\sim 1.1 \times 10^{19}$ eV$^{-1}$ m$^{-3}$ from which we estimate $\xi \sim 0.07$ nm. This localization length is incompatible with traditional theories of variable-range hopping where the localization length must be greater than the Ni-Ni distance and this may be due to the fact that the density of states we use is largely overestimated, consistent with our observation of Ni$^{2+}$ in this sample (Fig. 2(b)). This is similar to previous reports on hole-doped manganites, where a reduced $N(\xi_F)$ was proposed to explain the insulating behavior. This may occur due to the formation of a pseudogap.

To probe the origin of the thickness dependent metal to insulator transition, we performed magnetotransport measurements on the superlattices. Figure 4(c) shows the magnetic field dependence of the magnetoresistance ratio (MRR) at $T = 5$ K for the superlattices with $n = 3$ to 5. (MRR is defined as $[R(H) - R(0)/R(0)]$.) All of the superlattices show negative magnetoresistance, which is largest in the $n = 3$ sample, and decreases in the metallic samples. The orientation dependence of the resistance was investigated in the $n = 4$ superlattice by rotating the sample about the [100]-axis in a fixed field of 9 T (J is along the [100] crystallographic direction and is maintained perpendicular to the magnetic field). The resistance follows a $\cos^2 \theta$ dependence, where $\theta$ is the angle between the magnetic field and the normal to the film plane. This is further evidence that the carriers in LNO are scattering off magnetic moments in the superlattice. Empirically, the transverse resistivity of a ferromagnetic conductor may be written,

$$\rho_{xy} = R_H B + R_S \mu_0 M,$$

where $R_H$ is the ordinary Hall coefficient and $R_S$ is the anomalous Hall coefficient. We determine $R_H$ from the high-field ($H > 4$ T) slope of the anti-symmetric transverse resistivity [i.e., ($\rho_{xy}(+H) - \rho_{xy}(-H))/2$], while the anomalous Hall resistivity, $\rho_{xy}^{AHE} = R_S \mu_0 M$, is found by extrapolating the high-field slope to $H = 0$ T. Figure 4(c) shows $R_H$ as a function of temperature for the metallic superlattices, where conduction is assumed take place through the LNO layers only. All of the samples show a positive Hall coefficient, consistent with holotype conduction and in quantitative agreement with ultrathin LNO films and mixed-valence La$_{2-x}$Sr$_x$NiO$_4$ samples. $R_H$ is found to decrease non-linearly with increasing temperature, which may point to the presence of two carrier types with different mobilities, as suggested by recent band structure calculations of LNO. The strong temperature dependence of $R_H$ has also been attributed to antiferromagnetic correlations in the metallic phase of systems on the verge of an insulator to metal transition, such as V$_{2-x}$O$_3$ and cuprate superconductors. Assuming the current is confined to the LNO layers, we estimate the carrier concentration at $T = 5$ K for the metallic superlattices within a single-band model and find $p \approx 6.5 \times 10^{21}$ cm$^{-3}$ (0.38 holes per unit cell) and $p \approx 8.2 \times 10^{22}$ cm$^{-3}$ (0.48 holes per unit cell) for samples with $n = 4$ and $n = 5$, respectively. These values agree well with the expected number of holes per LNO unit cell, assuming each Ni cation at an interface donates a single hole to the neighboring Mn atom.

As shown in Fig. 4(e), we observe a negative anomalous Hall resistivity in the $n = 4$ sample below the magnetic transition temperature $T_C \sim 150$ K. The magnitude of the measured anomalous Hall resistivity is comparable to that previously observed in other perovskite oxide systems. In the $n = 5$ sample, however, $\rho_{xy}^{AHE}$ van-
The anomalous Hall effect arises from the spin-orbit interaction in the presence of broken time-reversal symmetry. In homogeneous magnetic systems, $R_S$ is predicted to scale with the longitudinal resistivity: $R_S \propto \rho_{xx}^\gamma$, with $\gamma = 1$ for skew-scattering and $\gamma = 2$ for the quantum mechanical side-jump mechanism. In magnetically inhomogeneous systems, such as Co/Pt superlattices and granular Co-Ag, values of $\gamma$ greater than 2 have been reported. We analyze the anomalous Hall effect in the $n = 4$ sample qualitatively by plotting $\rho_{AHE}$ as function of magnetization measured in the plane of the superlattice (Fig. 3 (inset)). The linear dependence suggests that $R_S$ is independent of temperature. However, $\rho_{xx}$ has a minimum at $T = 90$ K, ruling out a simple scaling relation between $R_S$ and $\rho_{xx}$. A lack of scaling between $R_S$ and $\rho_{xx}$ is also observed in colossal magnetoresistive manganites, where $R_S$ is sharply peaked above the Curie temperature. This behavior is attributed to Berry phase effects arising from fluctuating non-collinear spin textures. This is, however, in stark contrast to our samples, where $R_S$ is temperature independent. This may be due to a frozen interfacial spin texture, though a quantitative model is lacking.

IV. CONCLUSIONS

In conclusion, detailed magnetotransport, magnetic, and spectroscopic measurements were carried out on atomically sharp [001] (LaNiO$_3$)$_n$/([LaMnO$_3$])$_2$ superlattices. We have unambiguously evidence of the transfer of electrons from Mn to Ni, and ferromagnetic coupling of the net magnetization on the Mn and Ni sites. As the LNO layer thickness increases, the average Ni valence changes from Ni$^{2+}$ ($n = 2$) to Ni$^{2.5+}$ ($n = 4$), and is accompanied by a transition from insulating behavior to...
metallic transport. Detailed magnetotransport measurements suggest the carriers in the LaNiO$_2$ layer scatter off the magnetic Ni sites at the interface, and that the magnetization on the Ni is inhomogeneous on the nanometer scale. This work opens new avenues for the creation of artificial oxide heterostructures with tailored electronic and magnetic properties.

V. ACKNOWLEDGMENTS

Work at Argonne National Laboratory, including the use of the Center for Nanoscale Materials, was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Science, under Contract No. DE-AC01-06CH11357. J.H., B.N.-C. and A.B. acknowledge the support of the Division of Materials Science, DOE BES.

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