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Gate control of ferromagnetic insulating phase in lightly-doped $La_{0.875}Sr_{0.125}MnO_{3-\delta}$ film

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The electric field effect on the lightly doped $La_{0.875}Sr_{0.125}MnO_{3-\delta}$ (LSMO) thin film in electric double-layer transistors was investigated by measuring transport properties of the film under various gate voltages. It was found that the positive gate bias leads to an increase of the charge-orbital ordering (COO) transition temperature and a decrease of the Curie temperature T_C, indicating the suppression of ferromagnetic metal (FMM) phases and preference of COO/ferromagnetic insulator (FMI) with the hole depletion by gate bias. Such different electric field effects can be ascribed to the weakening of the ferromagnetic interaction and enhancement of Jahn-Teller (JT) distortion caused by the transformation of JT inactive Mn^{4+}-ions to JT active Mn^{3+}-ions. Moreover, a step-like increase in the high temperature region of the ρ-T curve, which is related to the transition of cooperative JT distortion, was found to develop with increasing the positive bias, indicating that the cooperative JT distorted phase is stabilized by the depletion of holes in LSMO film. These results demonstrate that the modulation of holes via electric field strongly affects the balance between energy gains of different interactions and thus produce different effects on the competing FMI, FMM, and cooperative JT distorted phases in LSMO film. © *2016 AIP Publishing LLC*.
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Hole doped-$La_{1-x}Sr_xMnO_3$ exhibits various phase transitions due to the strong coupling among charge, spin, orbital, and lattice. In the lightly doped zone, two successive structural phase transitions occur from a high temperature pseudocubic phase to an intermediate Jahn-Teller (JT) distorted orthorhombic phase and then to a low temperature pseudocubic phase. The most striking feature in this type of materials is the observation of the charge orbital ordering and insulating property in a ferromagnetic background. Specifically, when $x = 0.125$, a phase transition from paramagnetic to ferromagnetic accompanying an insulator to metal transition occurs at around 183 K (Refs. 1–3) which is well explained by the double-exchange (DE) mechanism. At a lower temperature of about 150 K, another metal to insulator transition occurs in the ferromagnetic region which results in a ferromagnetic insulating (FMI) phase in the low temperature region.[1–3] This FMI phase cannot be explained solely within the framework of the DE mechanism. Many experimental and theoretical studies have revealed that charge and orbital ordering (COO) takes place in this FMI phase, accompanying with the localization of charge carriers and strong distortion of MnO_6 octahedra.[2,4–6] Meanwhile, experiments showed that the properties of $La_{0.875}Sr_{0.125}MnO_3$ thin film vary greatly from that of the bulk materials due to the epitaxial strain from the mismatch between the substrate and the thin film. Particularly, the COO-induced metal-insulator (MI) transition is suppressed in the (001)-oriented films due to the clamp of the in-plane lattice by the substrate[7–9] but is achieved in (011)-oriented film by introducing anisotropic strains which allows a lattice deformation within the orbital ordering plane required by the concurrent JT effect with the formation of COO.[9–11] More recently, Wang *et al*. achieved a COO transition in the (001)-oriented $La_{0.875}Sr_{0.125}MnO_3$/PMN-PT thin film by introducing a large in-plane tensile strain and breaking out of the straitjacket from substrate.[12] These experiments demonstrated that the epitaxial strain and resulted lattice distortion is a key factor that influence the FMI phase and COO transition in $La_{0.875}Sr_{0.125}MnO_{3-\delta}$ (LSMO) thin films. However, introducing extra lattice strain from various substrates might bring about additional parameters such as defects, microstructures, and so on, which restricts the further investigation on the mechanism underlying various interesting properties in lightly doped $La_{1-x}Sr_xMnO_3$ film.

On the other hand, the carrier density is a key parameter that controls the physical properties of manganites. Experiments showed that the modulation of carrier concentration by carrier doping in manganites could result in various interesting phenomena including the charge orbital ordering.[13,14] Recently, lots of interest has been focused on electrostatically modulating physical properties of manganite thin film by using the configuration of electric field transistors.[15–17] In particular, many attempts have been made in the research of electric field effect using all oxide-based field effect transistors (FETs), composed of manganites and dielectrics/ferroelectrics, either for control of the different phases or for developing new electronic devices.[18–22] Different from the conventional chemical doping which is most widely used to modulate the charge carrier concentration, electric field approach does not introduce impurity and can continuously change the charge carrier concentration by varying the gate voltage. However, the low available gate bias and the inherent Thomas-Fermi screening effect[23] prevent its further application on the manganite thin film. More

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recently, quite a number of experimental investigations demonstrated that the electric double-layer transistor (EDLT) is a good alternative to the FETs for controlling the properties of the manganites via carrier density modulation.[24–32] To form an EDLT device, the ionic liquid/electrolyte is used as the gate dielectric materials instead of the conventional solid dielectric. Compared with the traditional solid dielectric, ionic liquids or electrolytes can provide very high electric fields and thus a large modulation of the carrier density (up to 10^{14}–10^{15} cm^{-2}) before breakdown occurs,[27,28] which considerably enlarge the research range of carrier concentration. Some investigations have been performed on FETs configurations composed of ionic liquid and manganite films.[26–28,30–32] However, most of previous researches focused on the gate control of insulator-metal transition (accompanied with the establishment of ferromagnetic ordering) and related percolative transport in specific systems. There has been no report so far on using EDLT to explore the effect of carrier modulation (field effect) on the competing various phases and corresponding transport properties in the low-doped LSMO with strong coupling among charge, spin, orbital, and lattice. Moreover, it has been previously demonstrated that the electrostatically doped carriers are more localized than those induced by chemical doping.[30] Thus, to investigate the evolution of the transport behavior of the lightly hole-doped LSMO film under the EDLT configuration would be unique and quite helpful for understanding the complex competition and related mechanism among the specific COO(FMI), ferromagnetic metal (FMM), and JT-distorted phases.

In this letter, we present a report of the electric-field-control of the competing FMI, FMM, and JT distorted phase in the lightly doped LSMO thin film by modulating the carrier concentration. A EDLT device with LSMO as channel was used to investigate the transport properties under various gate voltages. The N, N-diethyl-N-methyl-N-(2-methoxyethyl)ammonium bis(trifluoromethylsulphonyl)imide (DEME-TFSI), which is a binary salt with high polarity and composed of nitrogen-containing cations and imide anions, was chosen as the ionic liquid due to its higher dielectric constant and ionic conductivity than other electrolyte dielectrics. Moreover, it is solvent-free and can be used on lots of materials, especially suitable for manganite oxide films, on which it can be exposed to moderate voltages avoiding redox reactions. Our results revealed that the hole depletion induced by positive gate bias favors the FMI state and suppresses the FMM phase. Moreover, we found that the highly insulating state induced by positive gate voltage cannot return back to the original state after the positive voltage was removed and a negative one was even applied. Such interesting phenomena can be ascribed to the different accumulation and migration effect of oxygen vacancies driven by different polarities of the bias, which results in an irreversible oxygen vacancy distribution and thus an irreversible resistivity behavior in the channel film of LSMO. Furthermore, an unusual step-like change in the ρ-T curve, which is supposed to be related to the transition of cooperative JT distortion, was found to develop with the positive gate bias applied. Further applying negative bias resulted in the disappearance of such cooperative JT distortion transition. The induced transition between JT active Mn^{3+} and inactive Mn^{4+} ions by carrier injection was regarded as the main reason for such preference. These unexpected results indicate that the modulation of carriers (holes) strongly affect the stability of the cooperative JT distorted phase in LSMO film.

The thin LSMO film was deposited on a (001)-oriented single-crystal substrates of $SrTiO_3$ by the pulsed laser deposition (PLD) technique. A KrF excimer laser ($\lambda = 248$ nm) with a pulse energy of 180 mJ and a frequency of 2 Hz was used to ablate the stoichiometric target. The LSMO target was prepared by conventional solid reaction method. During the deposition, the temperature of the substrate was kept at 700 °C and the oxygen pressure at 0.01 Pa. After deposition, the film was cooled down to room temperature in vacuum. Considering the tradeoff between a large electric field effect and a small resistivity of the film, we used the film with 20 nm thickness to fabricate the device and performed transport investigations. The crystalline structure of the film was checked by means of X-ray diffraction (XRD) using Cu-K_α radiation on Bruker AXS D8-Discover. Meanwhile, the surface morphology of the film was investigated by atomic force microscopy (Seiko-SPA400). Magnetization was measured with a superconducting quantum interference device (SQUID-VSM). The sample was patterned using photolithography into a standard Hall-bar geometry with the side gate set next to the channel. The schematic of the sample is illustrated in Fig. 1(a). A droplet of ionic liquid, DEME-TFSI, was used to cover both the channel and the gate enabling the ions to move freely as a gate voltage was put on (see Fig. 1(b)). When a positive gate voltage is applied, the ion migration in DEME-TFSI depletes holes in the LSMO channel. In contrast, a negative voltage results in accumulation of the holes in the channel. The gate voltage dependent

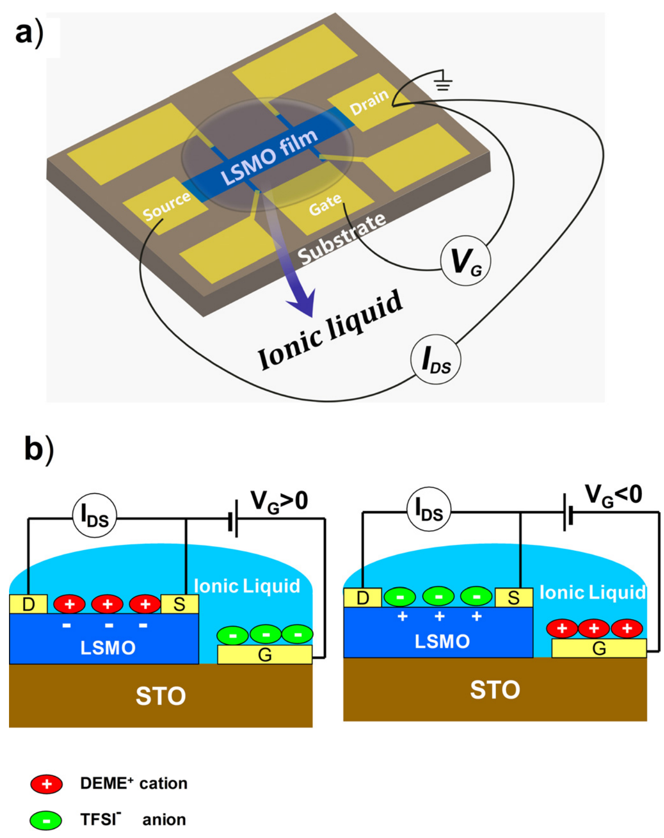

FIG. 1. The schematic illustration of the (a) patterned sample and (b) EDLT.

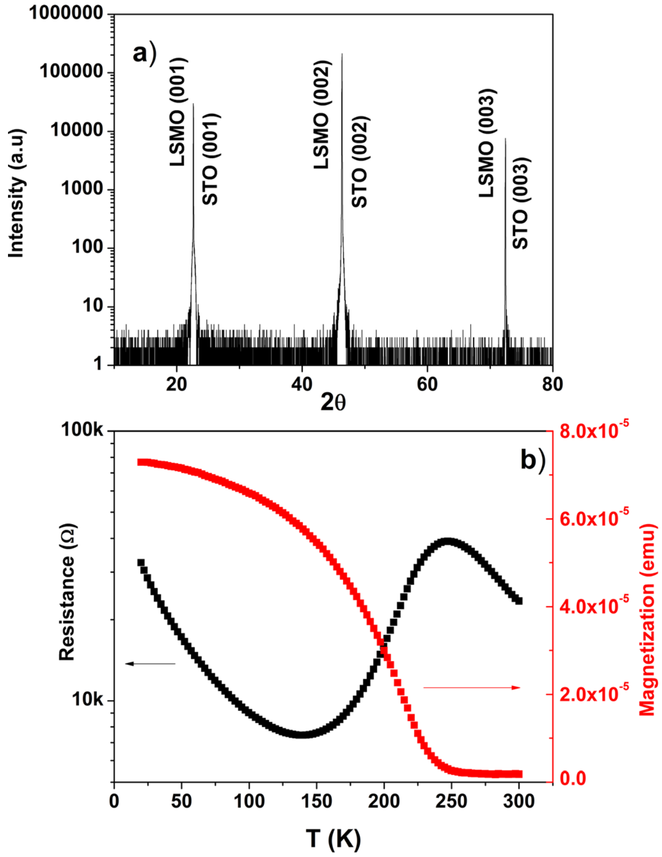

FIG. 2. (a) X-ray diffraction pattern of the LSMO/STO structures. (b) The original temperature-dependent resistance and magnetization curves of the LSMO film.

transport properties were measured in the SQUID-VSM with *in situ* electric fields applied on the gate. The measurements were carried out in the sequence of positive gate voltages, zero, and negative ones. All measurements were performed after the gate voltage was kept for 30 min at 300 K to ensure a sufficient migration of the polarized ions in DEME-TFSI. During the measurements, the gate voltage was kept as the sample was cooled down.

To suppress the influence from the lattice mismatch, we chose the single crystalline $SrTiO_3$ (STO) which has a very small lattice mismatch with LSMO as the substrate for the growth of LSMO film. Figure 2(a) shows an X-ray θ-2θ scan profile of the 20 nm-thick LSMO film, which indicates that the film was epitaxially grown on the substrate and no other phases or textures are observed. The reflections from the film were found to overlap with those from STO substrate completely, which indicates a nearly pseudomorphic growth and small strain induced by substrate taking into account that lattice parameters of LSMO (a = 3.902, b = 3.907, c = 3.896) are very close to that of the STO (a = 3.905). The surface morphology is investigated by atomic force microscopy (AFM). The surface roughness RMS was found to be ~0.9 nm, indicating the good quality of the film. The resistance and magnetization versus temperature curves for the film are shown in Fig. 2(b). Two successive MI transitions were identified from the R-T curve. The one in the higher temperature happens in the vicinity of the Curie temperature, and another in the lower temperature suggests a COO transition.[2–4] The low oxygen pressure during the deposition and post annealing leads to oxygen deficiencies in the film, which increases the distortion of MnO_6 octahedra and reduces the influence from the substrate, promoting the formation of charge-orbital ordering at low temperature. Previous researches have demonstrated that the COO transition at low temperature could be induced by either epitaxial strain or deficiencies.[12,33]

Figure 3 presents the temperature-dependent resistance (ρ-T) for different applied gate voltages measured on a 20 nm thick LSMO channel. Firstly, one can find that the resistance in the original state shows a bulk-like behavior

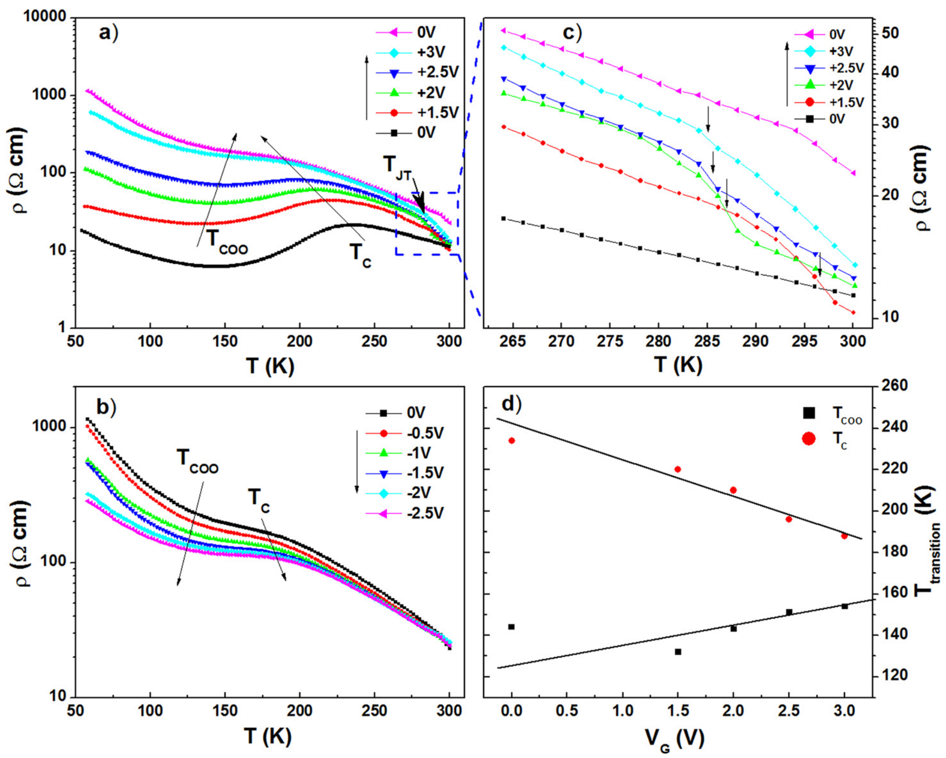

FIG. 3. Temperature dependent resistivity for (a) the positive gate bias and (b) the negative gate bias. (c) The details of (a), showing the detail of the abnormal change in resistivity related to cooperative JT distortion. (d) The gate voltage dependent T_C and T_{COO} upon positive bias.

similar to the one measured on the as-prepared film. Various positive gate voltages were applied to the channel through the gate electrode which is connected with the channel by the ionic liquid. As shown in Fig. 3(a), the resistance of the channel shows an increase in the whole measurement range with the application of positive gate voltage, which should be ascribed to the reduction in the carrier concentration due to the gate voltage induced depletion of holes in the LSMO channel. Meanwhile, with the increase of the gate voltage, the channel film gradually converts into a complete insulating state. Specially, when the gate voltage reaches +3 V, a highly insulating state appears in the entire temperature range and an obvious plateau is observed in the ρ-T curve. Since LSMO is hole-doped, when a positive gate voltage is applied, holes in the channel are depleted (see Fig. 1(b)), resulting in the reduction of carrier concentration. Measurements of Hall effect were performed in the LSMO Hall bar in order to estimate the sheet carrier concentration n_s. The n_s values under zero gate voltage were calculated to be in the range of 1.8×10^{14} to $7.2 \times 10^{14}\,cm^{-2}$ for different temperatures, whose magnitudes are consistent with previous reports for mixed-valence manganites.[24,28] Furthermore, it was found that the calculated carrier density decreases by an order of magnitude, while a positive gate voltage of +2 V was applied on EDLT. Specially, the modulation of sheet carrier density upon +2 V is as large as $6.4 \times 10^{14}\,cm^{-2}$ at 300 K. Such value of the modulated carrier density is at least ten times more than that can be achieved from conventional gate dielectrics and surely will cause the increase of channel resistance. Opposite effect is expected for the negative gate voltage. As shown in Fig. 3(b), the resistance of the channel decreases monotonously in the whole measuring temperature range with the increase of the magnitude of the negative gate voltage, which should be ascribed to the negative gate voltage induced injection of holes from the interface between ionic liquid and the channel film. However, the film remains the highly insulating state and cannot return back to the original state even at a voltage of −2.5 V. Such different situation for positive and negative gate voltage might be related to the migration of the oxygen vacancies in the channel film. Positive (negative) gate bias polarity will drive the oxygen vacancies to migrate away from (towards) the interface, hence resulting in different distribution of oxygen vacancy and lattice distortion. Considering the oxygen vacancies could affect the doping intensity,[34,35] such different distribution would lead to different carrier mobility and thus different conductivity in the channel. Moreover, previous research has demonstrated that the negative gate voltage can accumulate oxygen vacancies at the interface,[35] which will make the distribution discrepancy caused by different bias polarity irreversible to some extent. Thus, it might be reasonable that the resistance of the channel upon negative gate bias cannot completely return back to the original one. Moreover, the compared atomic force microscopy investigations of the surface quality of the channel film before and after the gating sequence show that no remarkable change occurs in the surface morphology and the surface RMS roughness, which suggests that the observed different transport behaviors under positive and negative gate voltages should not be owing to the possible damage of the film induced by the electrochemical reaction between the ionic liquid and the channel film.

Moreover, the successive metal-insulator transitions, related to the formation of ferromagnetic ordering and charge-orbital-ordering, respectively, show different responses to the gate bias. It was found that the peak temperature for the transition at higher temperature region (Curie temperature T_C, defined as the maximum point where $d\rho/dT = 0$) shifts towards lower temperature (from 234 K to 188 K) when the gate voltage increases from 0 V to +3.0 V. On the contrary, the transition point of the MI transition at low temperature (T_{COO}, defined as the minimum point where $d\rho/dT = 0$) first decreases to a lower temperature of 132 K at the voltage of +1.5 V and then shifts progressively to higher temperatures (from 132 K to 154 K, see Fig. 3(d)) with further increasing the gate voltage. Such opposite variations in T_C and T_{COO} indicate that the positive gate voltage can suppress the FMM state and favor the COO state via reducing the concentration of charge carriers. Actually, the reduction of carrier (hole) concentration leads to a weaker ferromagnetic interaction according to DE mechanism and thus the suppression of ferromagnetic ordering.[13] As a result, the FMM transition temperature decreases with the increase of gate voltage. On the other hand, the depletion of holes turns JT inactive Mn^{4+}-ions to JT active Mn^{3+}-ions,[3] which introduces JT distortion in the MnO_6 octahedra around the Jahn-Teller active site. Thus, with increasing the gate voltage, the lattice distortion is largely enhanced, which causes the channel film to favor the COO state at low temperature and thus an increase of T_{COO}.[12] The observed opposite shift of T_C and T_{COO} with the increase of the negative gate voltage (see Fig. 3(b)) further supports such explanations.

Interestingly, upon cooling, a step-like increase of the resistance appears around 298 K when a voltage of +1.5 V is applied on the gate electrode, indicating a discontinuous transition. With further increase of the gate voltage, such discontinuous transition keeps and its start-point moves slightly towards low temperature (see Fig. 3(c)). However, when a negative voltage was applied on the gate electrode, such discontinuous transition disappears. Previous researches by resonance X-ray scattering on the bulk LSMO[2,3,36] have confirmed that this transition is related to the appearance of cooperative JT distorted phase. However, such a transition was scarcely observed in LSMO thin films probably due to the limitation of cooperative JT distortion by the substrate. In this study, applying positive gate voltages introduces much more distorted MnO_6 octahedra in the channel film via turning JT inactive Mn^{4+}-ions to active Mn^{3+}-ions by depleting holes as discussed above, which will largely increase the energy gain connected to electron-phonon interaction. As a result, the stability of the cooperative Jahn-Teller distorted phase is enhanced and the discontinuous phase transition appears around room temperature. On the contrary, applying negative gate voltage induces hole doping in the LSMO thin film, turning Mn^{3+}-ions back to Mn^{4+}-ions. As a result, the stability of the cooperative JT distorted phase is reduced and the transition disappears in the measuring temperature range.

To conclude, we studied the electric field effect on the competing phases in $La_{0.875}Sr_{0.125}MnO_{3-\delta}$ thin film by measuring transport properties of the EDLT device under various gate voltages. The results revealed that the depletion (injection) of holes in the LSMO channel by positive (negative) gate voltage results in an increase (decrease) in the channel resistance. It was believed that the change of the carrier concentration should be responsible for these observations. Besides, two more important effects have also been demonstrated, which supports the appearance and evolution of the cooperative JT distorted phase upon bias. Firstly, we found that the positive gate voltage causes an opposite variation in T_C and T_{COO}, indicating the suppression of FMM and preference of COO(FMI) with the reduction of charge carrier concentration. Moreover, an unusual step-like increase in the ρ-T curve, which is related to the transition of cooperative JT distortion, was observed when the positive gate bias was applied. Further applying negative bias results in the disappearance of such cooperative JT distortion transition, indicating that the stability of the cooperative JT distorted phase is enhanced by the depletion of holes in LSMO film. These results illustrate that the carrier modulation via electric field approach can change the balance between energy gains of different interactions and thus produce different effects on the competing FMI, FMM, and cooperative JT distorted phases in LSMO film. In addition, it suggests that the electric field approach using EDLT device can serve as a powerful tool to unravel the underlying mechanism for unusual transport phenomena relating to FMI, FMM, and cooperative JT distorted phases.

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