Magnetism and anomalous Hall effect in Co-(La,Sr)TiO_3

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A systematic study of the magnetic properties and the Hall effect was performed on pulsed laser deposited 5% cobalt doped (La,Sr)TiO_3 thin films, especially grown at high substrate temperature. The system is found to be superparamagnetic in nature as evidenced by several protocols of magnetic measurements. Nevertheless, the anomalous Hall effect (AHE) is observed in the system, the profile of the measured Hall resistivity vs magnetic field being found to be identical to the magnetic hysteresis loops. This highlights the limitations of AHE as a tool to test the intrinsic nature of ferromagnetism in a diluted magnetic system, supporting our previous report for the Co:TiO_2 case [S. R. Shinde *et al.*, Phys. Rev. Lett. **92**, 166601 (2004)]. It is believed that the magnetic clusters polarize nearby electrons and the nonzero polarization leads to a net transverse current because of the spin dependent scattering, which gives rise to the observed AHE. We found that the magnitude of the AHE signal observed in the current extrinsic diluted magnetic semiconductor (DMS) is much lower (by a few orders of magnitude) than that found in the intrinsic long range ferromagnetic ordered DMS, which raises the possibility for using this magnitude, rather than the occurrence of AHE, as a criterion for intrinsic or extrinsic diluted magnetic system.

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I. INTRODUCTION

Recently, significant effort has been expended to realize spin-polarized current in semiconductor matrices so as to develop novel spintronic effects and applications. One of the major research directions being explored toward this objective is by dilute doping of a magnetic element into a semiconductor(s) to achieve room temperature ferromagnetism (FM) without changing the other application-worthy properties of the host.[1,2] Amongst the various materials being pursued for such so-called intrinsic diluted magnetic semiconductors (DMSs), oxide-based ones have perhaps been the most controversial because of the emergence of secondary phases and magnetic dopant clusters under different growth conditions.[3–5]

For a very long time, the anomalous Hall effect has played an essential role in establishing or negating the occurrence of intrinsic FM in DMS systems.[2,6] However, only few cases of AHE for oxide-DMS systems have been observed, mostly with reduced $TiO_{2-\delta}$ as the parent compound.[7–9] Very recently, our group reported the observation of the AHE in a highly reduced cobalt doped rutile $TiO_{2-\delta}$ wherein cobalt was found to form nanoclusters.[9] This questioned the AHE as a tool to test the intrinsic nature of DMS. Nevertheless, a systematic study of the AHE in relation to this extrinsic DMS has been lacking because of the insulating nature of TiO_2. Moreover, it is also important to establish in the interest of the DMS community that the cooccurrence of AHE and superparamagnetism is not unique in reduced Co-TiO_2 because the AHE is still being considered as an evidence of intrinsic FM of DMS systems.[10] In the work reported here, we have performed a systematic study of the Hall effect and magnetic properties in nonferromagnetic (La,Sr)TiO_3 with dilutely embedded cobalt clusters. This material is chosen for the following reasons. (1) Most recently, considerable interest has grown in novel oxide-based DMS systems of carrier mediated RKKY type,[11–14] including cobalt and lanthanum dual doped $SrTiO_{3-\delta}$ (Refs. 11 and 12) (Co-LSTO) which shows significant spin polarization when grown at certain growth conditions.[12] (2) Unlike TiO_2, (La,Sr)TiO_3 is a conductor;[15] hence, it is possible to do Hall measurements from room temperature down to very low temperatures.

In this work, we studied the pulsed laser deposited 5% cobalt doped (La,Sr)TiO_3 thin films, where evidences for ferromagnetic clusters are clearly noted. The magnetic moments of these nanoclusters do not couple with each other, as indicated by several superparamagnetism-type slow dynamic behaviors. Over the measured temperature range of 5–300 K, the anomalous Hall effect is seen, especially with loops below the blocking temperature. We found that this AHE contribution in an extrinsic DMS system is much smaller than that found in intrinsic III-V DMS systems such as Mn-GaAs.[2,16] Possible reasons of the AHE in our system are discussed. Importantly, we found that this material system appears to have an embedded cluster character under most experimental growth conditions examined in our work. This questions whether the system can, in fact, be stabilized into an intrinsic DMS state with only dilutely dispersed dopants without any degree of clustering. In our first paper,[11] based on the limited set of characterization protocols (which only evolved in later years with maturing of DMS research), we had suggested that this system appears to have an intrinsic DMS character. This was substantiated by later works of Herranz *et al.*[12] However, our detailed magnetic studies presented here seem to suggest an embedded cluster character for this system. The clusters are extremely fine which are

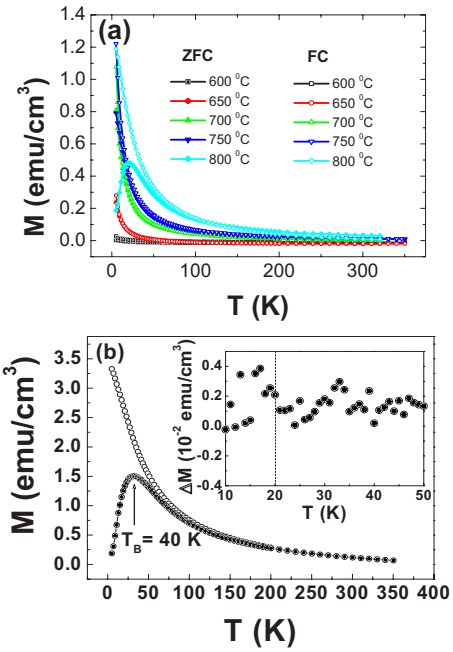

FIG. 1. (Color online) (a) Zero field cooled and field cooled magnetizations of Co-LSTO films (grown at 600–800 °C) in a magnetic field of 50 Oe as a function of temperature. (b) ZFCM (solid dots) and FCM (hollow dots) of Co-LSTO film grown at 850 °C as a function of temperature. The inset shows the difference of the magnetization between normal ZFC and aging ZFC.

therefore likely to evade detection unless specifically probed for. We also emphasize that, theoretically, the recently reported FM in Co-$LaSrTiO_3$ (Refs. 11 and 12) falls into the regime where the carrier concentration is larger than the moment concentration,[14] and this will lead to the frustrated state instead of the long range FM state if the RKKY interaction is applied.[17]

II. EXPERIMENT

The 5% Co:$La_{0.3}Sr_{0.7}Ti_{0.95}O_{3-\delta}$ thin films were grown on $LaAlO_3$(001) substrates by pulsed laser deposition at the oxygen partial pressure of 10^{-6} Torr. Such a low pressure is believed to be crucial in stabilizing the $LaSrTiO_3$ phase.[18] The growth temperature was varied from 600 to 850 °C. X-ray diffraction and high resolution transmission electron microscopy (TEM) were performed for structural characterization. The samples for Hall measurements were patterned by ion milling technique using a standard hall mask. The magnetization measurements were taken at the temperatures from 5 to 350 K using a superconducting quantum interference device magnetometer and the transport measurements were made by a Quantum Design physical property measurement system.

III. RESULTS AND DISCUSSION

A. Magnetic properties

Figure 1(a) shows the temperature dependence of zero field cooled magnetization (ZFCM) and field cooled magnetization (FCM) of the thin films grown at various temperatures (H=50 Oe, and H is perpendicular to film plane). Apparently, none of these samples shows ferromagnetic behavior; rather, they show a superparamagnetic type of behavior. We now focus on the systematic studies on the sample grown at 850 °C, which is the main part of this paper. As seen in Fig. 1(b), the FCM and ZFCM (under a magnetic field of 150 Oe) follow a Curie-like paramagnetic behavior at high temperatures and depart from each other below ~40 K, where a maximum appears for ZFCM, while the FCM continues to increase with decreasing temperature. This thermomagnetic irreversibility between ZFCM and FCM is a typical characteristic of superparamagnetism (SPM) or a (super) spin glass (SSG) where magnetic clusters couple. In our system, with only 5% cobalt in the (La,Sr)TiO_3 host, it is believed that the nanoclusters do not interact with each other, rendering a superparamagnetic system with the evidence of a nonflat FCM below the blocking temperature.[19] To confirm this argument, we performed the studies of ZFC memory effect on our system. In this protocol, the system is zero field cooled with and without an intermittent stop at 20 K for 10 000 s and then further cooled down to 5 K, after which the magnetization data were taken upon warming at a field of 150 Oe. The temperature dependence of the difference between ZFCM with and without an intermittent stop at 20 K does not show any cusp at 20 K [inset of Fig. 1(b)], indicating the absence of a memory effect, which establishes that the system is not a SSG.[19] Generally, in a superparamagnetic state, the Langevin function leads the nonhysteresis M vs H/T curves to superimpose into one curve[20] [M-H curves above 40 K in our case, Fig. 2(a)], while below ~40 K, M vs H data show hysteresis loops and the coercivity increases with the decrease in temperature [Fig. 2(b)]. Here, 40 K is the so-called blocking temperature (T_B), below which the moments of the single-domain FM particles start to be blocked due to the magnetic anisotropy. The blocking temperature relates to the average particle volume as $k_B T_B \sim KV/25$, where K is the anisotropy constant. If using K=4.5106 erg/cm^3 for cobalt,[20] we can get the average particle volume $V \sim 31$ nm^3 (diameter $D \sim 3.85$ nm). As shown in Fig. 2(c), the cobalt clusters with 3–5 nm size were indeed found to distribute randomly in thin films. On the other hand, as noted in Fig. 1(a), the blocking temperature decreases to below 5 K as the growth temperature decreases to 700 °C, indicating that the clusters are extremely tiny and may have been overlooked in our previous paper.[11]

B. Hall effect

The temperature dependence of longitudinal resistivity ρ_{xx} for the 5% Co-LSTO sample grown at 850 °C [shown in Fig. 3(a)] is identical to the undoped (La,Sr)TiO_3 case, with a strong electron-electron scattering dominated metallic behavior,[15] supporting the fact that cobalt does not incorporate into the LSTO matrix. The transverse resistivity ρ_{xy} against the magnetic field $\mu_0 H$ at various temperatures is plotted in Figs. 3(b) and 3(c). Although this system is not an intrinsic diluted magnetic system, as shown in the previous section, the AHE is clearly observed over the temperature

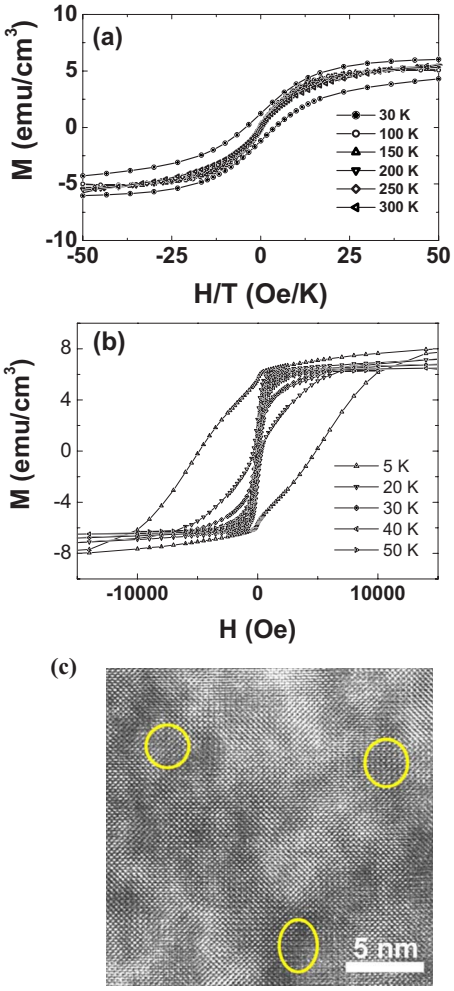

FIG. 2. (Color online) (a) M vs H/T data of the Co-LSTO film grown at 850 °C ($T \geqslant 50$ K). (b) M vs H data of the Co-LSTO film grown at 850 °C ($T \leqslant 50$ K). (c) The cross-section TEM image of the Co-LSTO film. Yellow circles indicate the nanosize clusters.

range of 5–300 K. The low temperature ρ_{xy} vs $\mu_0 H$ curves show hysteresis, while the high temperature ones do not. The Hall resistivity is usually expressed as $\rho_{xy}=R_0 B+\mu_0 R_s M$, where the first term is the ordinary Hall resistivity and the second one is the anomalous Hall resistivity. The R_0 and R_S are the ordinary and anomalous Hall coefficients, respectively; B and M are the applied magnetic field and spontaneous magnetization, respectively. The negative slope of our high field Hall data indicates the n-type carriers, which is expected for the LSTO system.[15] We subtracted the ordinary Hall resistivity (the carrier density n is $\sim 4\times 10^{21}$ cm^{-3}) and plotted the anomalous Hall resistivity vs magnetic field (ρ_{xy}^{AHE} vs $\mu_0 H$) in the insets of Figs. 3(b) and 3(c). For comparison, M vs $\mu_0 H$ together with ρ_{xy}^{AHE} vs $\mu_0 H$ for various temperatures is plotted in Figs. 4(a)–4(e). It can be seen that the ρ_{xy}^{AHE} vs $\mu_0 H$ and M vs $\mu_0 H$ curves almost coincide with each other, confirming that the AHE relates to those noninteracting magnetic clusters. The insets show the ρ_{xy}^{AHE} vs M which is almost linear. From $R_S=\rho_{xy}^{AHE}/\mu_0 M$, we can obtain the anomalous Hall coefficient $R_S \sim 0.03$ cm^3/C [as shown in Fig. 4(f)].

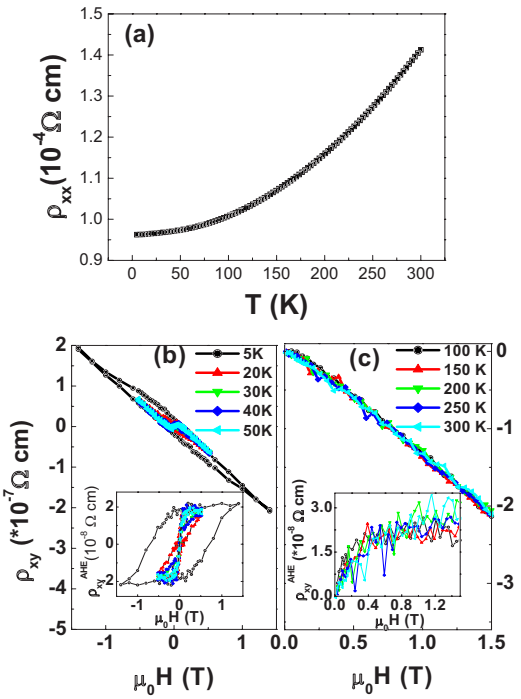

FIG. 3. (Color online) (a) The longitudinal resistivity ρ_{xx} as a function of temperature. (b) The Hall resistivity ρ_{xy} vs $\mu_0 H$ at low temperatures (around and below the blocking temperature). The inset shows ρ_{xy}^{AHE} vs $\mu_0 H$. (c) The Hall resistivity vs $\mu_0 H$ at high temperatures (above the blocking temperature). The inset shows ρ_{xy}^{AHE} vs $\mu_0 H$. The film was grown at 850 °C.

C. Mechanism of anomalous Hall effect in superparamagnetic cobalt and lanthanum dual doped $SrTiO_{3-\delta}$

In ferromagnetic materials, the origin of the AHE remains the subject of continuous debate and the current understanding is based on several possible mechanisms. (1) Bulk inversion asymmetry and structural inversion asymmetry of a material give rise to a band-structure-induced spin-orbit coupling effect called the Dresselhaus and the Rashba spin-orbit coupling, respectively. This kind of intrinsic spin-orbit coupling manifests itself as the Berry curvature in the momentum space and supposedly gives a dissipationless Hall current.[6,21] As this Hall effect originates from the intrinsic band structure of the material, it is commonly referred to the intrinsic anomalous Hall effect. The intrinsic anomalous Hall resistivity scales with the longitudinal resistivity as $\rho_{xy}^{AHE} \sim \rho_{xx}^2$. (2) In the presence of impurities, spin-orbit scattering in a solid gives rise to asymmetric scattering of spin-up electrons (preferentially in one direction) and spin-down electrons (preferentially in the opposite direction), which is called skew scattering.[22] In addition, an accompanying mechanism called side-jump scattering was proposed by Berger,[23] in which the electrons in the process of being scattered also undergo a lateral displacement due to the presence of an anomalous velocity induced by the spin-orbit coupling. The induced transverse Hall current due to both of these processes is directly proportional to the spin density $s=n\uparrow -n\downarrow$, where $n\uparrow$ and $n\downarrow$ are the spin-up and spin-down electron densities, respectively.[24,25] The AHE due to skew scat-

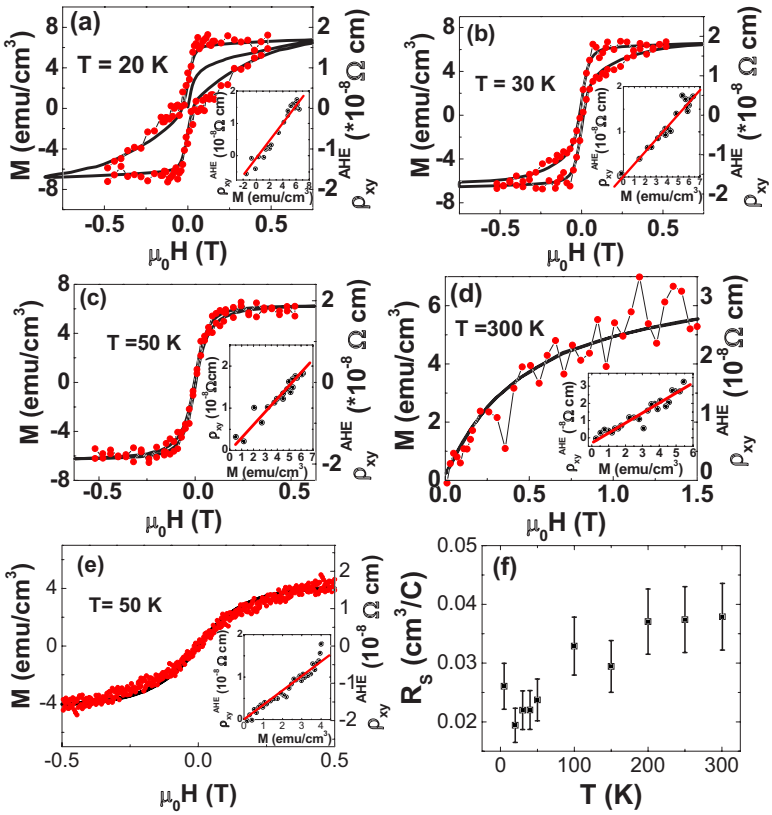

FIG. 4. (Color online) The comparison of the magnetic field dependence of anomalous Hall resistivity ρ_{xy}^{AHE} [red (dark gray) dots with right axis] and magnetization M (solid black line with left axis) at (a) 20 K, (b) 30 K, (c) 50 K, and (d) 300 K, for the film grown at 850 °C. (e) 50 K for the film grown at 750 °C. Insets show ρ_{xy}^{AHE} vs M for the respective temperatures [black dots are experimental data, and solid red (dark gray) lines are fitting data]. (f) R_s as a function of temperature for the sample grown at 850 °C.

tering and side-jump mechanisms is collectively known as the extrinsic anomalous Hall effect as it originates from extrinsic impurities. The scaling relationship for the extrinsic AHE is $\rho_{xy}^{AHE} \sim a\rho_{xx} + b\rho_{xx}^2$.

In our samples, both the ρ_{xx} and ρ_{xy} do not vary much with temperature. This makes it difficult to study the scaling relation between them. However, it would be fair to conclude that the AHE observed in our samples is not the intrinsic AHE since the embedded magnetic clusters do not change the band structure of the epitaxial $(La,Sr)TiO_3$ host, and $(La,Sr)TiO_3$ itself does not show any AHE.[15] Spin-orbit scattering due to impurities is therefore more likely to be the underlying mechanism for our observed AHE. The magnetic clusters polarize the nearby conduction electrons, as shown in Fig. 5(a). Before the clusters are magnetized by the external magnetic field, the net magnetization M is zero, and therefore the spin polarization is zero. Once the cluster magnetizations get oriented by the magnetic field, as shown in Fig. 5(b), a net electron spin polarization is induced ($s \sim M$), which gives rise to a nonzero transverse Hall current due to the extrinsic spin-orbit scattering. This picture is substantiated by the fact that the observed ρ_{xy}^{AHE} vs $\mu_0 H$ curve is almost identical to the M vs $\mu_0 H$ curve and therefore ρ_{xy}^{AHE} is proportional to the magnetization M (as seen in insets of Figs. 4(a)–4(e)), which is the case for the extrinsic AHE.[25] We note that for the case of intrinsic AHE, except for the known particular case of ferromagnetic Mn_5Ge_3 thin film,[26] the ρ_{xy}^{AHE} vs M profile is in general rather nonlinear.[6,27]

We note that the magnitude of the AHE (here defined by $\rho_{xy}^{AHE}/\rho_{xx}$) found in the extrinsic DMS is usually very small compared to that found in intrinsic DMS. For example, with similar doping level, $\rho_{xy}^{AHE}/\rho_{xx}$ in our Co-LSTO here and highly reduced Co-$TiO_{2-\delta}$ (Ref. 8) has values of the order of 10^{-4}, while in the intrinsic DMS with long range FM order,[2,16] i.e., (Ga, Mn)As, $\rho_{xy}^{AHE}/\rho_{xx}$ is $\sim 10^{-2}$. This is not

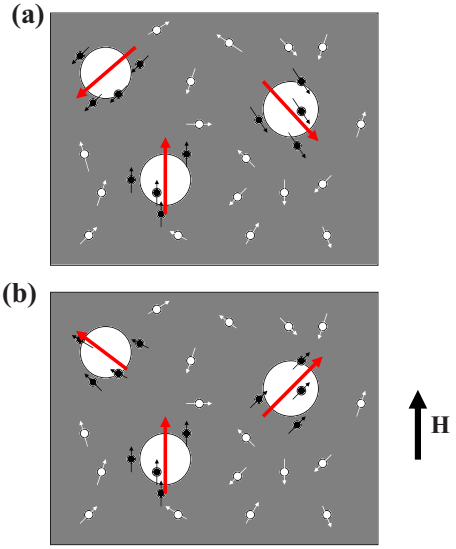

FIG. 5. (Color online) Schematic pictures of magnetic clusters and electrons inside a SPM sample as in our experiment (a) before and (b) during the cluster spins are oriented by the magnetic field H: the big white dots are clusters, the small black dots are electrons polarized by the clusters, and the small white dots are electrons not polarized by the clusters. Arrow crossing each dot indicates the spin orientation of each object.

surprising if one notes that $\rho_{xy}^{AHE}/\rho_{xx}$ is proportional to the spin polarization (s/n).[28] In our extrinsic DMS, the magnetic clusters do not couple to each other (superparamagnetism) but only polarize the carriers in the nearby region depending on the interaction strength. However, in the intrinsic DMS, especially for the carrier mediated type, the carriers strongly couple to the local magnetic ions which are more homogenously distributed. Therefore, the population of carriers contributing to AHE in the intrinsic DMS is much larger than that in the extrinsic case, which hence gives rise to a higher magnitude of AHE. Moreover, it has been found that the Berry-phase contribution of the AHE in the intrinsic DMS is more significant than the scattering contribution.[6,26,27] The low magnitude of the AHE in a system with dilutely dispersed magnetic clusters might be the reason that its existence has been neglected for a long time.

IV. SUMMARY

We report the observation of the anomalous Hall effect in superparamagnetic Co-$(La,Sr)TiO_3$ wherein noninteracting nanomagnetic clusters are dispersed in a crystalline LSTO matrix. The magnetic field dependence of the anomalous Hall resistivity is similar to that shown by the magnetization, indicating that the AHE here is related to the embedded superparamagnetic clusters. It is believed that the magnetic clusters polarize nearby electrons and the nonzero polarization leads to a net transverse current because of spin dependent scattering. We point out that the AHE signal here is much weaker than that observed in intrinsic DMS systems due to the less population of spin-polarized carriers, which might be considered as a criterion for the intrinsic nature of a DMS system.

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