Exchange bias associated with phase separation in the perovskite cobaltite $La_{1-x}Sr_xCoO_3$

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We report the observation of exchange bias phenomena in the hole-doped perovskite cobaltites $La_{1-x}Sr_xCoO_3$ (x=0.12, 0.15, 0.18, and 0.30) in which a spontaneous phase separation occurs. When the $La_{1-x}Sr_xCoO_3$ samples are cooled in a static magnetic field through a freezing temperature, the magnetization hysteresis loops exhibit both horizontal and vertical shifts. We also observed training effect of the exchange bias, which can be interpreted by a spin configurational relaxation model. Moreover, exchange bias in $La_{1-x}Sr_xCoO_3$ is strongly dependent on the measuring field and the cooling field due to the influence of magnetic field on the relative proportion of the coexisting phases. These results suggest that the intrinsic phase inhomogeneity in a spontaneously phase-separated system may induce an interfacial exchange anisotropy.

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I. INTRODUCTION

Exchange bias has often been observed in heterogeneous systems consisting of ferromagnetic (FM) and antiferromagnetic (AFM) spin structures. It manifests itself as a shift H_E of the magnetization hysteresis loop along the field axis when the system is cooled down in an external magnetic field through the Néel temperature of the antiferromagnet. This shift is due to an unidirectional anisotropy which is caused by the coupling of the ferromagnet to the antiferromagnet at the interface.[1] This effect was first discovered in 1956 by Meiklejohn and Bean when studying Co particles coated with a layer of AFM CoO.[2] Since then exchange bias has been observed in many different systems containing FM/AFM interfaces, such as inhomogeneous materials,[3,4] thin films consisting of bilayers and double superlattices.[5–7] In addition to FM/AFM interfaces, exchange bias has also been observed in other types of interfaces involving a spin glass (SG) phase (e.g., FM/SG).[8–13]

The hole-doped perovskite oxides, such as manganites and cobaltites, have drawn a lot of research attention since the early 1990s, mainly due to the discovery of the colossal magnetoresistance (CMR) effect in them. Recent progress in CMR materials has reached the conclusion that intrinsic phase separation should play a crucial role in understanding their peculiar physical properties.[14,15] Especially, recent studies have shown that the hole-doped cobaltites such as $La_{1-x}Sr_xCoO_3$ (LSCO) exhibit a particularly clear form of phase separation for a broad range of doping level x, as evidenced by many experimental results obtained using various techniques including electron microscopy,[16,17] nuclear magnetic resonance (NMR),[18–20] and small-angle neutron scattering (SANS).[21] Substitution of Sr^{2+} for La^{3+} induces Sr-rich clusters and La-rich matrix. Caciuffo *et al.* have observed Sr-rich clusters with sizes ranging from 8 to 40 nm.[16] The Sr-rich clusters are ferromagnetic metal due to the double exchange interaction between Co^{3+} and Co^{4+}, while the La-rich matrix is non-magnetic as similar to $LaCoO_3$. It has been proposed that the phase separation in LSCO is in the form of FM clusters embedded in a nonmagnetic matrix, with SG regions between them as interfaces.[18,19,22] Thus, the intrinsic phase separation leads to the FM/SG interfaces in LSCO. As mentioned above, exchange bias has been observed in some systems containing FM/SG interfaces. Therefore, it would be interesting to explore if exchange bias could exist in the intrinsically phase-separated cobaltites.

In this work, we have performed such a study in a series of $La_{1-x}Sr_xCoO_3$ (x=0.12, 0.15, 0.18, and 0.3) cobaltites. Exchange bias associated with phase separation was observed in all samples. We also observed training effect of the exchange bias, which can be described by a spin configurational relaxation model. Moreover, unlike conventional exchange bias phenomena, exchange bias associated with phase separation is strongly dependent on the cooling magnetic field as well as the measuring field. These findings suggest that in a spontaneously phase-separated system the exchange coupling at the interfaces between the FM regions and the surrounding SG regions may create an unidirectional anisotropy (exchange anisotropy) when the sample is cooled in a static magnetic field.

II. EXPERIMENTS

Polycrystalline $La_{1-x}Sr_xCoO_3$ (x=0.12, 0.15, 0.18, and 0.3) samples were prepared with solid state reaction method. A stoichiometric mixture of $SrCO_3$, Co_3O_4, and La_2O_3 powder was well ground and calcined twice at 800 and 950 °C for 24 h. Then, the resulting powder was pressed into pellets and sintered at 1100 and 1150 °C for 24 h, respectively. The x-ray diffraction experiments were carried out on the Rint Rigaku 1400 x-ray diffractometer. X-ray diffraction patterns show that the samples are single phase with rhombohedral structure (Fig. 1). The magnetization measurements were performed using a commercial Quantum Design superconducting quantum interference device (SQUID) magnetometer with applied magnetic fields H up to 50 kOe in the temperature range 5 K$\leq T \leq$360 K. The temperature dependence of magnetization was measured on warming with a low magnetic field (H=10 Oe) in both zero-field cooled (ZFC) and field cooled (FC) processes. In order to obtain a low field, the superconducting magnet of SQUID magnetometer was demagnetized before measurements. After each measurement

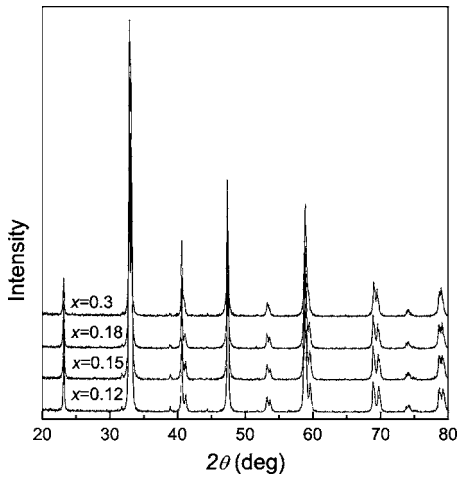

FIG. 1. Powder x-ray diffraction patterns of $La_{1-x}Sr_xCoO_3$ (x =0.12, 0.15, 0.18, and 0.3) samples at room temperature.

of the hysteresis loop at a given condition, the sample was warmed up to 360 K to demagnetize.

III. RESULTS AND DISCUSSION

Figure 2 shows the temperature dependence of magnetization with the ZFC and FC processes for $La_{1-x}Sr_xCoO_3$ (x =0.12, 0.15, 0.18, and 0.3) samples. All samples display a bifurcation of the ZFC and FC magnetizations at about 240 K which is the irreversibility temperature T_{irr}. The FC curve of x = 0.3 shows a "Brillouin-like" temperature dependence of the magnetization. With decreasing x the FC magnetization is considerably reduced and no longer shows a simple "Brillouin-like" FM behavior. In contrast, the ZFC magnetization is rather small for all samples. A peak is observed at 50, 65, 120, and 225 K for x=0.12, 0.15, 0.18, and 0.3, respectively. These magnetic behaviors were observed before and were regarded as cluster-glass behaviors. It has been known that there are Sr-rich FM regions and La-rich non-FM regions in LSCO within a broad doping level ($0 < x < 0.5$).[23,24] In our measurements, the irreversibility temperature T_{irr} implies the onset of FM ordering within the clusters. The peak temperature in ZFC magnetization is usually defined as T_f below which magnetic moments begin to freeze collectively. Recently, ^{59}Co NMR study has revealed that SG regions coexist with FM and non-FM regions in LSCO.[18,19] The magnetic relaxation experiments also demonstrate that a classical spin glass phase as well as intercluster interactions contributes to the glassy behaviors in LSCO.[22] It has been well known that a spin-disordered interface/surface layer is usually formed when a FM particle is embedded in a non-FM matrix[10] or the magnetic particle size is small enough (the finite size effect).[9] Taking into account the size of FM clusters ranging from 8 to 40 nm in LSCO,[16] it is more likely that the SG regions could exist at the interfaces between the FM clusters and the non-FM matrix. Therefore, exchange bias could be expected considering the coupling between the FM clusters and SG regions in the intrinsically phase-separated cobaltites.

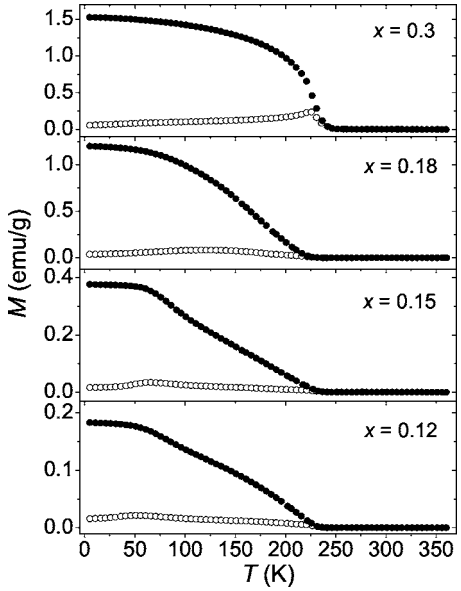

FIG. 2. Temperature dependence of the dc magnetizations with ZFC (open circles) and FC (solid circles) processes for $La_{1-x}Sr_xCoO_3$ (x=0.12, 0.15, 0.18, and 0.3) samples in 10 Oe.

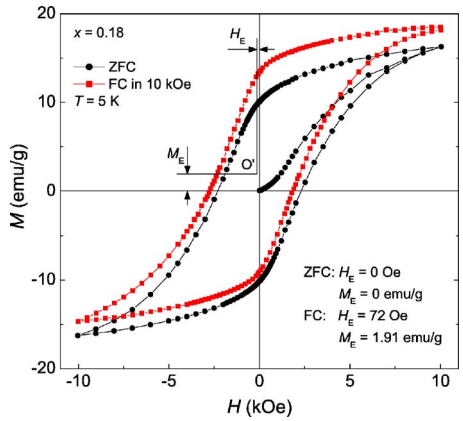

FIG. 3. (Color online) Hysteresis loops of $La_{0.82}Sr_{0.18}CoO_3$ at 5 K measured after zero-field cooling and field cooling in 10 kOe field.

To test this argument, we have measured the hysteresis loops of $La_{0.82}Sr_{0.18}CoO_3$ at 5 K with both the ZFC and the FC processes. For the ZFC process, the sample was cooled in zero magnetic field from 360 to 5 K. For the FC process, the sample was cooled in 10 kOe magnetic field from 360 to 5 K. Then, the hysteresis loops were measured between ±10 kOe. As shown in Fig. 3, while the ZFC magnetization has a normal hysteresis loop centered at zero field, it is clear that the FC hysteresis loop shifts both to the negative field and to the positive magnetization. Therefore, the gravity center of the FC hysteresis loop shifts to O' (72 Oe, 1.91 emu/g). As shown in Fig. 3, we define the shift of the gravity center along the field axis as the exchange-bias field H_E. The vertical shift of the gravity center is defined as the magnetization shift M_E.

One of the interesting characteristics in exchange-biased systems is the training effect, which describes the decrease of

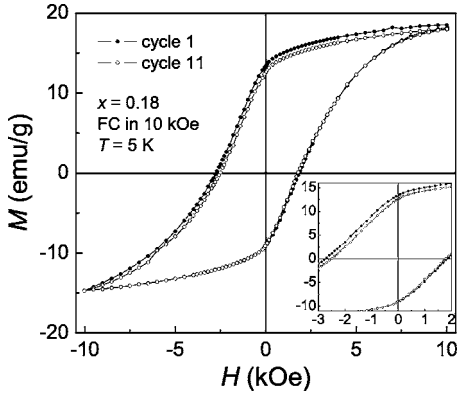

FIG. 4. Training effect of exchange bias in $La_{0.82}Sr_{0.18}CoO_3$. Plotted are the first and the eleventh loops at 5 K after field cooling in 10 kOe. Inset: enlarged view of the central region of the loops.

the exchange-bias field H_E when cycling the systems through several consecutive hysteresis loops.[1] For the $x=0.18$ sample, the consecutive hysteresis loops were measured at $T=5$ K, after field cooling in 10 kOe. The first and the eleventh loops are shown in Fig. 4. The inset of Fig. 4 shows the expansion of the low-field region. It is obvious that the training effect is present in our sample. Both the exchange-bias field and the magnetization shift decrease with magnetic field cycling. The number of field cycles n dependence of the exchange-bias field H_E and the magnetization shift M_E is shown in Fig. 5 (open circles and squares, respectively).

It is often found experimentally that the relationship between H_E (M_E) and n is given by a simple power-law

$$H_E - H_{E\infty} \propto 1/\sqrt{n}, \tag{1}$$

where $H_{E\infty}$ is the exchange-bias field in the limit of infinite loops.[1,25] The solid lines in Fig. 5 show the best fits with this empirical relation to both H_E and M_E data with field-cycle number $n>1$. The fitting curves show satisfactory agreement with the experimental data with $n>1$. We obtained the fitting parameters $H_{E\infty}=37$ Oe and $M_{E\infty}=1.534$ emu/g. It is also found that the data point at $n=1$ significantly exceeds the value when extrapolating the fit to $n=1$. Previous investigations have revealed the breakdown of the power-law behavior at $n=1$.[1,25] Although the power-law decay of exchange bias has been widely observed, its origin remained unexplained. Recently, Binek[26] considered the training effect in FM/AFM heterostructures in the framework of nonequilibrium thermodynamics. It was proposed that consecutively cycled hysteresis loops of the FM top layer trigger the spin configurational relaxation of the AFM interface magnetization toward equilibrium and a recursive formula is obtained to describe the n dependence of $H_E(M_E)$.[26] The relation is

$$H_E(n+1) - H_E(n) = -\gamma[H_E(n) - H_{E\infty}]^3, \tag{2}$$

where γ is a sample-dependent constant. The analytical approach is confirmed by experimental results obtained recently on a NiO(001)/Fe(110) heterostructure and a Co/CoO exchange-biased bilayer.[26,27] Using the initial value of $H_E(1)=72$ Oe obtained from experiments, $\gamma=2.738\times10^{-4}$ Oe^{-2} and $H_{E\infty}=34$ Oe, the theoretical data of H_E (solid circles in Fig. 5) are calculated from the implicit sequence (2). Similarly, the theoretical data of M_E (solid squares in Fig. 5) are obtained with $M_E(1)=1.91$ emu/g, $\gamma=2.701$ (emu/g)$^{-2}$, and $M_{E\infty}=1.495$ emu/g. It is found that both theoretical data are well coincident with experimental results not only for $n>1$ but also for $n=1$. Thus, the spin configurational relaxation model can describe our experimental results well. It is likely that the consecutive reversion of the FM cluster magnetization triggers the configurational relaxation of the interfacial SG spins toward equilibrium and causes the training effect.

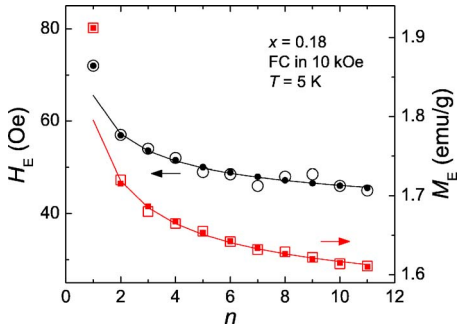

FIG. 5. (Color online) The number of field cycles n dependence of H_E and M_E (open symbols) for $La_{0.82}Sr_{0.18}CoO_3$ at 5 K after field cooling in 10 kOe. Solid lines show the best fits with Eq. (1) to the data for $n>1$. Solid symbols show the data generated from the recursive sequence (2) as described in the text.

All these behaviors described above, including the hysteresis loop shift along the field axis and the magnetization axis as well as the training effect, are the characteristics of exchange bias phenomenon. In some compounds, such as Gd_2CuO_4,[28] a shift of hysteresis loops is also observed, which is interpreted as the presence of undirectional anisotropies due to Dzyaloshinskii-Moriya interactions. However, for our sample, it seems that this mechanism is not appropriate due to the large magnetization of our sample and the shape of the initial magnetization curve as shown in Fig. 3. To reveal the origin of exchange bias in LSCO, we studied the temperature dependence of exchange bias. In these measurements, the sample was cooled down from 360 K to the measuring temperature with an applied field $H_{cool}=3$ kOe. Once the measuring temperature was reached, the magnetization loop was measured between −3 and 3 kOe. This process was repeated for every measuring temperature. As shown in Fig. 6, with increasing temperature H_E decreases and finally vanishes above $T\sim120$ K, in correspondence with the freezing temperature T_f. The temperature evolution of H_E is typical for exchange-biased systems with FM/SG interfaces.[11–13] As the sample is cooled through $T_C=240$ K with an applied magnetic field, the moments of FM clusters line up with the field, while the SG spins remain random. When cooling to $T<T_f$, in the presence of a magnetic field, the SG spins next to the FM clusters arrange along specific direction due to the exchange interaction at the FM/SG interface. In turn, below T_f, the SG spins at the interface exert a microscopic torque on the FM spins to keep them in their original direction. Thus, the magnetization loop is shifted

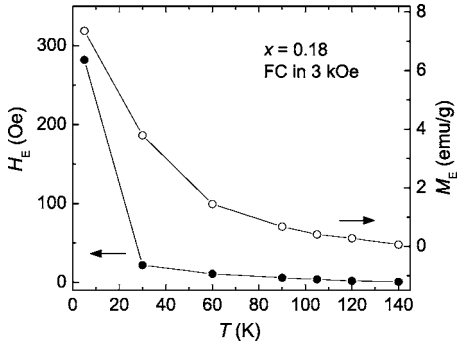

FIG. 6. Temperature dependence of H_E and M_E for $La_{0.82}Sr_{0.18}CoO_3$ after field cooling in 3 kOe.

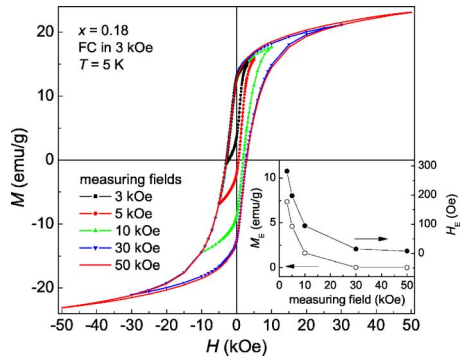

FIG. 7. (Color online) The FC (in 3 kOe) hysteresis loops for $La_{0.82}Sr_{0.18}CoO_3$ at 5 K with different measuring magnetic fields. Inset: measuring field dependence of H_E and M_E at 5 K.

along the field axis, i.e., exchange bias appears. In addition, the magnetization shift M_E is also observed at different temperatures below T_f, as shown in Fig. 6. This behavior can be ascribed to the competition of different energies at the FM/SG interface. The Zeeman energy of the FM clusters (E_Z), the anisotropy energy of the FM clusters (E_F) and of the SG (E_S), and the exchange energy at the FM/SG interface (E_{int}) are the four energy terms contributing to exchange bias in LSCO in an external magnetic field. An effective Zeeman energy $E_{Z_{\text{eff}}}=|E_Z|-|E_F|$ is introduced for convenience, and we will consider only the maximal absolute values of these energies. When $E_{Z_{\text{eff}}}<E_{\text{int}}$ and $E_{Z_{\text{eff}}}<E_S$, the effective Zeeman energy is too weak to overcome the interfacial energy barrier or to rotate the SG spins. Parts of FM spins will stay "frozen," and this will show up as a vertical magnetization shift. In our measurements, the maximal measuring field is 10 kOe, and the effective Zeeman energy is not strong enough so that it is reasonable to observe a shift along the magnetization axis. Recently, a vertical shift of the hysteresis loop is also observed in particle systems.[29,30] Furthermore, for spin glass, it is well known that there are many configurations of the ground state. In our sample, the exchange bias effect results from the pinning to FM moments by frozen SG spins along cooling field direction. It seems that there exists the training of the frozen spins. When the applied field consecutively cycles, some of frozen SG spins along cooling field direction may change their directions and fall into other metastable configurations, which would decrease the strength of exchange coupling at the interfaces. Therefore, the training effect in cobaltites can be well interpreted with the spin configurational relaxation model. In fact, the training effect of exchange bias in a FM/SG system (the γ-Fe_2O_3 coated Fe nanoparticles) has been well described using a modified Stoner-Wohlfarth model in which the spin configurational relaxation has also been considered.[31]

It has been reported that exchange bias is dependent on the cooling field in some systems.[11] In the phase-separated perovskite cobaltites, the proportion of coexisting phases can be affected by an external field. Therefore, it is necessary to explore the effect of magnetic field on exchange bias in LSCO. First, we studied the influence of measuring field on the exchange bias in $La_{0.82}Sr_{0.18}CoO_3$. For each measurement, the sample was cooled in a field of 3 kOe from 360 to 5 K, then the hysteresis loops were measured between ±3, ±5, ±10, ±30, and ±50 kOe, respectively. The results are shown in Fig. 7. When the measuring field is low ($H \leq 10$ kOe), the FC hysteresis loops always shift to the negative field and the positive magnetization, suggesting that an unidirectional anisotropy exists after the field cooling. However, when the measuring field is high enough, $H \geq 30$ kOe, the FC hysteresis loops do not show any shift, i.e., exchange bias disappears in high magnetic fields. The inset of Fig. 7 shows the measuring field dependence of the exchange-bias field H_E and the magnetization shift M_E. These shifts decrease rapidly with the growth of the measuring field and approach to zero around 30 kOe.

There should be two factors which contribute to this peculiar feature of exchange bias in $La_{0.82}Sr_{0.18}CoO_3$. On the one hand, as the measuring field increases, the effective Zeeman energy also increases. Accordingly, M_E decreases due to the reduction of the proportion of the FM spins staying "frozen." On the other hand, in phase-separated cobaltites, the FM clusters grow up with increasing applied magnetic field,[16] and the SG regions would somehow be destroyed by applied magnetic field. With the reduction of the SG regions and the increment of the size of the FM clusters, the relative proportion of the SG layers to the FM clusters significantly decreases. Once the field is high enough, the small portion of the SG spins cannot pin the huge moments of the FM region. Consequently, no exchange anisotropy exists. This effect is similar to that in the systems with FM/AFM interfaces, in which H_E decreases for thin enough AFM layers and is roughly inversely proportional to the thickness of the FM layers.[1]

Then we studied the influence of cooling field (H_{cool}) on the exchange bias in $La_{0.82}Sr_{0.18}CoO_3$. The sample was cooled down from 360 to 5 K under different applied field $0 \leq H_{\text{cool}} \leq 10$ kOe. After the temperature became stable, the field was set to $H=10$ kOe and the hysteresis loops were measured. Figure 8 shows the cooling-field dependence of the exchange-bias field H_E and the magnetization shift M_E. We notice that there are two regions for the variation of H_E (M_E) with H_{cool}. At low cooling field, H_E and M_E increase with the increase of cooling field. At high cooling field, H_E decreases with the increase of cooling field and M_E seems to be saturated. When the cooling field is low, it is not strong enough to make all of the FM clusters magnetized saturat-

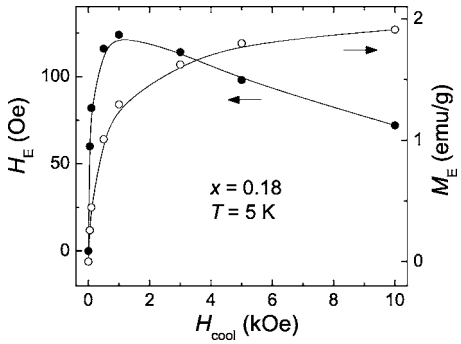

FIG. 8. Cooling field dependence of H_E and M_E for $La_{0.82}Sr_{0.18}CoO_3$ at 5 K. The lines are guides to the eyes.

edly. With the increase of the cooling field, the alignment degree of the FM clusters moments along a preferential direction is enhanced, which reduces the effect of averaging of the anisotropy due to randomness. Accordingly, more of SG spins at interface also align along the direction due to the exchange coupling. Therefore, H_E and M_E increase with the increase of the cooling field. When the cooling field is high enough, the FM moment is saturated and the "frozen" FM spins along the field vary little with the increase of the cooling field. Thus M_E also tends to be saturated. The reduction of H_E with increasing the cooling field is similar to those results in $La_{0.88}Sr_{0.12}CoO_3$, in which the effect of higher cooling field was explored.[32]

Since spontaneous phase separation occurs in LSCO for a broad doping level ($0<x<0.5$), it would be expected that exchange bias might appear in other LSCO samples. Especially, at a low x level the proportion of the FM phase is low, and consequently the FM clusters are small and well isolated. In this situation, the interfacial exchange coupling is expected to have a more significant effect. Exchange bias in $x<0.18$ should remain until a higher magnetic field than that in $x=0.18$.

To examine this view, we have also studied the hysteresis loops of $x=0.12$, $x=0.15$, and $x=0.3$. In Fig. 9, we show the FC hysteresis loops at 5 K for all samples with a cooling field of 3 kOe from 360 to 5 K and the maximum measuring field of 10 kOe. All samples exhibit exchange bias. The values of H_E and M_E are shown in the inset of Fig. 9. For $x=0.3$, the volume of the FM clusters exceeds the percolation threshold $x=0.18$. Many FM clusters are coupled ferromagnetically to one another, and even coalesce into bigger FM clusters. The high proportion of FM phase deduces the decrease of H_E and M_E as observed. For $x<0.18$, the exchange bias is stronger than that of $x=0.18$ as expected. However, the M_E of $x=0.12$ decreases abruptly. This could be mainly due to the small FM magnetization of $x=0.12$, which cannot provide enough magnetization to be frozen. In addition, we also measured the FC hysteresis loops of all samples with a high measuring field of 50 kOe. As shown in Fig. 10, exchange bias disappears in $x=0.18$ and $x=0.3$ samples. In contrast, as we have expected, for $x=0.15$ and $x=0.12$ samples, exchange bias remains even with a measuring field of 50 kOe, with $H_E=217$ and 257 Oe, respectively. Because the high measuring field leads to large effective Zeeman energy $E_{Z_{\text{eff}}}$ which can overcome exchange energy E_{int} and can make all of FM magnetizations reverse, no magnetization shift is observed for the two samples. Thus, the results of other LSCO samples further confirm the picture of exchange bias associated with phase separation in cobaltites.

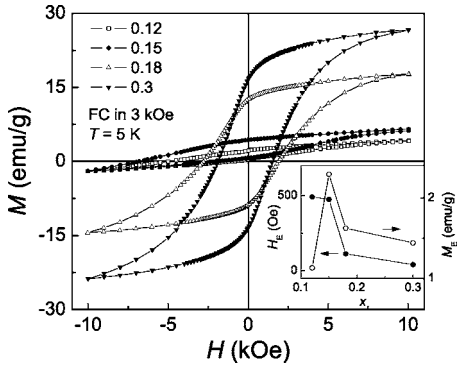

FIG. 9. Hysteresis loops of $La_{1-x}Sr_xCoO_3$ ($x=0.12$, 0.15, 0.18, and 0.3) samples at 5 K measured after field cooling in 3 kOe with measuring field between ±10 kOe. Inset: x dependence of H_E and M_E at 5 K.

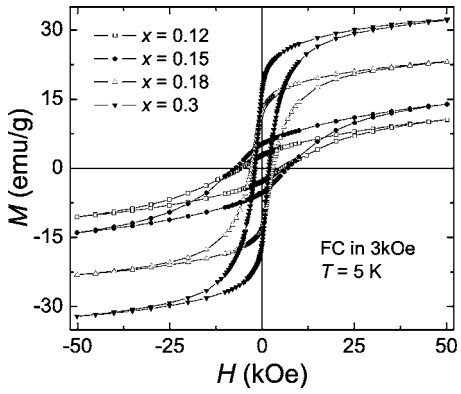

FIG. 10. Hysteresis loops of $La_{1-x}Sr_xCoO_3$ ($x=0.12$, 0.15, 0.18, and 0.3) samples at 5 K measured after field cooling in 3 kOe with measuring field between ±50 kOe.

IV. CONCLUSION

In summary, we have observed exchange bias phenomena associated with phase separation in cobaltites. When the LSCO samples are cooled in a magnetic field through a freezing temperature, the magnetization hysteresis loops shift to both the negative field and the positive magnetization. The exchange bias exhibits a training effect, which can be explained by a spin configurational relaxation model. Moreover, exchange bias in LSCO depends not only on the cooling field but also on the measuring field. These results suggest that in a spontaneously phase-separated system, the exchange coupling at the interfaces between the FM clusters and the SG regions may create an exchange anisotropy when the sample is cooled in a static magnetic field. Peculiarly, unlike conventional exchange bias phenomenon, exchange bias associated with phase separation can be completely removed in a high magnetic field due to the variation of coexisting phases with increasing magnetic field.

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