Exchange bias effect involved with tunneling magnetoresistance in polycrystalline $\text{La}_{0.88}\text{Sr}_{0.12}\text{CoO}_3$

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Abstract – We report the exchange bias (EB) effect along with tunneling magnetoresistance (MR) in polycrystalline $\text{La}_{0.88}\text{Sr}_{0.12}\text{CoO}_3$. Analogous to the shift in the magnetic hysteresis loop along the field ($H$)-axis a shift is clearly observed in the MR-$H$ curve when the sample is cooled in a static magnetic field. Training effect (TE) is a significant manifestation of EB effect which describes the decrease of EB effect when sample is successively field-cycled at a particular temperature. We observe TE in the shift of the MR-$H$ curve which could be interpreted by the spin configurational relaxation model. A strong field-cooled (FC) effect in the temperature as well as time dependence of resistivity is observed below spin freezing temperature. The unusual MR results measured in FC mode are interpreted in terms of intragranular interface effect between short-range ferromagnetic clusters and spin-glass matrix giving rise to the EB effect. EB effect in MR has been observed in bilayer or multilayer films which has not yet seen in a polycrystalline compound. EB effect involved with tunneling MR and semiconducting transport property attributed to the intragranular intrinsic nanostructure is promising for the spintronic applications.

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Introduction. – The exchange bias (EB) effect is a manifestation of the unidirectional anisotropy which occurs in a system having heterostructure composed of ferromagnetic (FM) and antiferromagnetic substances when it is cooled through the Néel temperature in a static magnetic field [1–3]. A semiconducting system exhibiting EB effect is fascinating because EB coupling provides an additional degree of freedom for controlling the conduction process which has a significant impact in spintronic applications [4]. Recently, investigations on the spin-polarized transport, tunneling magnetoresistance (TMR), and exchange coupling in the semiconducting FM bilayer or multilayer films have played a central role in spintronics [5,6].

The doped cobaltites $\text{La}_{1-x}\text{Sr}_x\text{CoO}_3$ with perovskite structure are the model systems for the investigation on complex magnetoelectronic phase separation [7]. At a low doping the semiconducting transport properties were reported where FM metallic clusters embedded in a non-FM insulating matrix have been confirmed by neutron and NMR results [8–10]. With increasing $x$ the fraction of the FM metallic cluster increases and it coalesces together for $x > 0.18$ leading to the long-range FM ordering and metallic conductivity. Wu et al. recently reported an interesting scenario of semiconducting transport properties exhibiting glassy magnetic behavior in the single crystals of $\text{La}_{1-x}\text{Sr}_x\text{CoO}_3$ for $x \leq 0.18$ [11]. They suggested that the unusual glassy transport phenomena were attributed to the magnetoelectronic phase separation into nanoscale FM clusters embedded in a non-FM matrix. Recently, significant EB effect through the shift in magnetic hysteresis (MH) loop has been reported in polycrystalline as well as single crystalline compounds, $\text{La}_{1-x}\text{Sr}_x\text{CoO}_3$ suggesting the spontaneous phase separation between FM and spin-glass (SG) like states [12–14].

Recent reports demonstrate that the EB effect can also be evidenced through the shift in magnetoresistance-field (MR-$H$) curve for bilayer or multilayer films [15–18]. To the best of our knowledge, the signature of the EB effect in the MR-$H$ curve is not reported in a compound. In this letter, we report a considerable EB effect through the systematic shift, training effect, strong field-cooled (FC)
effect in the temperature and time dependences of resistivity ($\rho$) below the spin freezing temperature in polycrystalline $La_{0.88}Sr_{0.12}CoO_3$. Large MR at low temperature is interpreted in terms of an intragranular tunneling mechanism across the magnetic tunnel barrier. Current research has been focused in developing the advanced materials for spintronic applications in the artificial heterostructure derived from FM semiconductors [19]. Here, we demonstrate that the intrinsic heterostructure attributed to the spontaneous phase separation in the grain interior may provide a useful platform in searching new materials for spintronic applications.

**Experimental.**– A polycrystalline compound with composition $La_{0.88}Sr_{0.12}CoO_3$ was prepared by the chemical route which was described in our previous report [20]. As-synthesized sample was heated at 873 and 1273K where the average grain sizes are found to be $\sim 35$ and $\sim 240$ nm, respectively which were confirmed by Transmission Electron Microscopy (TEM) using a microscope (ZEOL JEM-2010). For simplicity we address the sample with larger grain size as bulk while the sample with smaller grain size is defined as nanoparticle. TEM images are shown in figs. 1(a) and (b) for the bulk and nanoparticle, respectively. A high-resolution TEM (HRTEM) image of the nanoparticle is further shown in fig. 1(c) where a single lattice plane is noticed with spacing $\sim 0.367$ nm which matches with the spacing of the (110) plane with rhombohedral structure ($R\overline{3}c$) having lattice parameters, $a = 0.538$ nm and $\alpha = 60.4^\circ$ obtained from the X-ray powder diffraction. Experimental results for the bulk is mainly presented here while the results for the nanoparticle is compared with the bulk in fig. 3. The single phase of the rhombohedral structure was confirmed by a powder X-ray diffractometer (Seifert XRD 3000P) using CuK$_\alpha$ radiation. Powder X-ray diffraction patterns of the bulk and nanoparticle are shown in fig. 1(d) where the diffraction peaks could be indexed in the rhombohedral structure ($R\overline{3}c$) with lattice parameters, $a = 0.539$ nm and $\alpha = 60.7^\circ$ for the bulk which are in accordance with the previous reports [7,21]. Measurements of the negative MR defined as $\frac{\rho_H - \rho_0}{\rho_0}$ were carried out using a superconducting magnet system (Cryogenic Ltd., UK), where $\rho_H$ and $\rho_0$ are the resistivities in static and zero magnetic field. dc magnetization was measured using a commercial superconducting quantum interference device, SQUID magnetometer (MPMS, XL).

**Results and discussions.**– Zero-field cooled (ZFC) and field-cooled (FC) magnetizations as a function of temperature ($T$) measured at 200 Oe are shown in fig. 2 for the bulk. A peak in the ZFC magnetization is observed around $\sim 55$ K ($T_f$) with a large FC effect below $\sim 197$ K ($T_c$) which is highlighted in the inset of the figure. The magnetization results are in excellent agreement with the phase diagram proposed by Wu et al. where $T_c$ is the onset of Curie temperature involved with the short-range FM clusters and $T_f$ is the spin freezing temperature behaving like a SG transition temperature [7].

A symmetric MH loop (solid line) measured at 5 K is shown in fig. 3 for the bulk where magnetization does not show any saturating tendency at 50 kOe which is consistent with the cluster-glass state proposed for $La_{0.88}Sr_{0.12}CoO_3$ [13]. MR-$H$ curve (solid line) at 5 K having large MR ($\sim 57\%$) at 50 kOe is also displayed in fig. 3 for the bulk. MR (broken line) was also measured for the nanoparticle where magnitude of MR is decreased drastically than that of the bulk counterpart. Since grain boundary region is considerably enhanced by decreasing the grain size than its bulk counterpart, the large decrease in the magnitude of MR for the nanoparticle.
reveals that the grain boundary effect does not contribute any significant role where grain interior mechanism leads to the large MR. Intragranular giant magnetoresistance (GMR) attributed to the spontaneous phase separation has been proposed by Wu et al. where tunneling between FM metallic clusters across the non-ferromagnetic semiconducting matrix was pointed out for the interpretation of GMR effect in La$_{0.85}$Sr$_{0.15}$CoO$_3$ [8]. The results are analogous to the negative intergranular MR previously observed in artificial structures composed of nanoscale FM particles embedded in an insulating or metallic nonmagnetic matrix where orientation of the magnetization axes, the density, and the size of the FM entities are crucial to interpret GMR effect [22–24]. In the present observation the smaller magnitude of MR at 5 K and 50 kOe for the nanoparticle than its bulk counterpart is suggested due to the intragranular mechanism involved with the size and the density of FM metallic clusters in the non-ferromagnetic semiconducting matrix for the nanoparticle which are different from the bulk analogue. MR–H curve attributed to the tunneling mechanism exhibits a peak in MR at zero magnetization [19]. For example, the Co$_{16}$Cu$_{84}$ alloy exhibiting SG-like behaviour at low temperature shows a peak in the MR–H curve at zero magnetization [22]. Here, a peak is observed at $H'_C$ (8.8 kOe) which is much larger than the coercivity ($H_C = 5.8$ kOe) in the MH loop. The results are similar to that observed in the double perovskite, Sr$_2$FeMoO$_6$ [25]. The MH loop and the MR–H curve for the nanoparticle are illustrated in fig. 3 by the broken lines. $H_C$ is increased to 6.8 kOe while $H'_C$ does not change significantly. Since the anisotropy of SG spins is much stronger than FM spins, the coercivity of the SG compound is typically much higher than that of the FM compound. Thus the system composed of FM and SG components should have smaller average coercivity than the individual SG component where $H_C$ obtained from the MH loop provides the average coercivity. In the present observation TMR is attributed to the intragranular effect where tunneling between FM clusters takes place through the SG matrix. In such a case the tunneling barrier is set by the anisotropy of the SG spins where $H'_C$ represents the coercivity of the SG component. We note that the value of $H'_C$ does not change with the average particle size. The results further indicate that the anisotropy of the SG component remains unaltered, despite the grain interior nanostructure is modified due to the change in particle size which is reflected in the substantial change in the MR–H curve.

When the sample was cooled down to 5 K from 250 K with a cooling field, $H_{cool} = 10$ kOe, the MH loop is shifted along the negative $H$-axis. The shift is absent while cooling the sample in the ZFC mode which is shown in fig. 4(a). The central part of the loop is highlighted in fig. 4(b). If $H_{c1}$ and $H_{c2}$ are the negative and positive coercivities of the shifted loop, the magnitude of the EB field ($H_E$) is defined as $H_E = |H_{c1} - H_{c2}|/2 \approx 650$ Oe which is consistent with the previous report [13]. Interestingly, a large shift in the MR–H curve is also observed at 5 K for cooling the sample in the FC mode which is absent in the ZFC mode. We further notice that the shift is negative for $H_{cool} = 10$ kOe while it is positive for $H_{cool} = -10$ kOe. The shifted MR–H curves in between ±50 kOe are illustrated in fig. 4(c) for $H_{cool} = \pm 10$ kOe which are further highlighted in fig. 4(d) around the origin. The peak in the MR–H curve measured from 50 kOe to −50 kOe is shifted along negative $H$-axis while the curve from −50 kOe to 50 kOe is remained almost unchanged (see the broken curve in fig. 4(d)) for $H_{cool} = 10$ kOe. Exchange bias field ($H_{MR}^E$) obtained from the shifted MR–H curve is estimated.
The training effect (TE) is one of the significant results as $H_{\text{MR}}^{\text{TE}} = |H_p - H_q|/2 \approx 940$ Oe, where $H_p$ and $H_q$ correspond to the field of the peak positions at $p$ and $q$, respectively. $H_{\text{MR}}^{\text{TE}}$ is noted much larger than the value from the MH loop, despite the values of $H_{\text{E}}/H_C$ ($\approx 11.2\%$) and $H_{\text{MR}}^{\text{TE}}/H_C$ ($\approx 10.7\%$) are nearly the same. The qualitative interpretation of a value of $H_{\text{MR}}^{\text{TE}}$ larger than that of $H_E$ is discussed at the end of the text. We further note that the peak height in the MR-$H$ curve at $p$ decreases considerably compared to the height at $q$ where position and height at $q$ are almost the same compared to the symmetric MR-$H$ curve obtained in the ZFC mode. On the other hand, the peak height at $r$ decreases considerably with respect to the height at $s$ for $H_{\text{cool}} = -10$ kOe. The results clearly exhibit a spin-valve-like character in the MR-$H$ curve analogous to that observed in films exhibiting the EB effect [15–18].

The training effect (TE) is one of the significant results of the EB effect which describes the decrease of the EB effect when the sample is successively field-cycled at a particular temperature. TE is typically observed in the MH loop [2,3] which is recently reported in the MR-$H$ curve only in a very few spin valve systems exhibiting the EB effect [26,27]. In fig. 5(a) typical signature of TE is illustrated in dMR/d$H$ with $H$ plots at $5$ K up to $5$ successively cycles ($\lambda$). Curves passing through dMR/d$H = 0$ in the positive and negative $H$-axes are highlighted in the top and bottom inset, respectively which correspond to the field at the peak position in the shifted MR-$H$ curves. A large decrease of $H_{\text{MR}}^{\text{TE}} \sim 22\%$ is observed in between first and second cycles. The decrease of $H_{\text{MR}}^{\text{TE}}$ is fitted satisfactorily with the empirical relation, $H_{\text{MR}}^{\text{TE}}(\lambda) - H_{\text{MR}}^{\text{TE}}(\lambda = \infty) \propto 1/\lambda$. The solid straight line in fig. 5(b) exhibits the best fit of $H_{\text{MR}}^{\text{TE}}$ against $\lambda^{-1/2}$ ($\lambda \geq 2$) with $H_{\text{MR}}^{\text{TE}}(\lambda = \infty) \approx 566.5$ Oe. The above empirical relation does not fit the sharp decrease between first and second cycles in accordance with the reported results in MH [3,12] as well as in MR-$H$ [26]. Binek proposed a recursive formula in the framework of spin configurational relaxation to understand the training effect which describes the ($\lambda + 1$)-th loop shift with the $\lambda$-th one as [28]

$$H_{E}^{\text{MR}}(\lambda + 1) - H_{E}^{\text{MR}}(\lambda) = -\gamma[H_{E}^{\text{MR}}(\lambda) - H_{E}^{\text{MR}}(\lambda = \infty)]^3,$$

where $\gamma$ is a sample-dependent constant. Using $\gamma = 2.5 \times 10^{-6}$ Oe$^{-2}$ and $H_{E}^{\text{MRI}} = 503.5$ Oe the whole set of data (filled circles) could be generated which matches satisfactorily with the experimental data (fig. 5(b)). In case of spin configuration relaxation model [28] the expression was developed in the frame work of nonequilibrium thermodynamics where TE for the FM/AFM heterostructure was correlated with the relaxation of the FM spins exchange coupled with the AFM spins toward equilibrium. A similar effect might also be proposed here where the relaxation of the FM spins exchanged coupled with the SG spins at the interface toward equilibrium leads to the TE in the MR-$H$ curve.

The semiconducting temperature dependence of $\rho$ measured during the warming mode in zero field ($\rho_0$) and in field (50 kOe) under ZFC ($\rho_{\text{ZFC}}$) and FC ($\rho_{\text{FC}}$) modes are shown in fig. 6(a). $\rho_{\text{ZFC}}$ deviates from $\rho_0$ below $\sim 40$ K which is much below $T_{\text{f}}$ at 55 K. Furthermore, a considerable deviation between $\rho_{\text{ZFC}}$ and $\rho_{\text{FC}}$ is observed where the magnitude of $\rho_{\text{FC}}$ is significantly higher than $\rho_{\text{ZFC}}$ below $\sim 40$ K. A strong FC effect in the time dependence of $\rho$ is observed under the following experimental protocol. The sample was cooled down to 5 K from 250 K with $H_{\text{cool}} = 50$ kOe and then $\rho$ was measured with time ($t$) after the removal of the magnetic field. The plot of $\rho(t) - \rho(0)/\rho(0)$ vs. $t$ is illustrated in fig. 6(b) by the open circles where $\rho(0)$ defines the resistivity at $t = 10$ s. The relaxation of magnetization is involved with the activation against distribution of anisotropy barriers for the glassy magnetic compound where time dependence of magnetization typically follows the stretched exponential with a critical exponent ($\beta$) for $0 < \beta < 1$. We fit the time dependence using a stretched exponential function, $\rho(t) - \rho(0)/\rho(0) = A + B \exp(t/\tau)^\beta$, where $\tau$ is the relaxation time. The satisfactory fit is shown by the continuous curve in fig. 6(b) with $\tau = 2435$ s and $\beta = 0.94$, suggesting the glassy magnetic behavior in the transport property. We observe a considerable increase
FM and spin-glass regions.

 Modes. FM, FFM, and SG represent the ferromagnetic, frozen H in ZFC (bulk). Cartoon of the phases separation scenario within a grain. H \rho (b) Time dependence of \[ \rho (t) - \rho (0) \] at 5 K measured with \( H = 0 \) and 50 kOe in FC and ZFC modes, respectively, for the bulk. Cartoon of the phase separation scenario within a grain in ZFC (\( H_{\text{cool}} = 0 \)) (left inset) and FC (\( H_{\text{cool}} \neq 0 \)) (right inset) modes. FM, FFM, and SG represent the ferromagnetic, frozen FM and spin-glass regions.

 of \[ \rho (t) - \rho (0) / \rho (0) \] up to \( \sim 11\% \) at \( t = 5.6 \times 10^3 \) s. On the other hand, a very small decrease of \[ \rho (t) - \rho (0) / \rho (0) \] around \( \sim 1\% \) is observed below 100 s when the sample is cooled down to 5 K from 250 K in the ZFC mode and then \( \rho (t) \) is recorded with time at 50 kOe field. The results clearly demonstrate that strong time dependence of \( \rho \) at 5 K in the FC mode is involved with the EB effect.

 A cartoon of the phase separation within a grain is proposed in the left inset of fig. 6(b) where short-range FM regions or clusters are embedded in a SG matrix. When the sample is cooled in the FC mode, a new layer consisting of frozen FM (FFM) spins arises at the FM/SG interface due to the pinning of the FM spins by the rigid SG spins. The layers of FFM spins by replacing the surface region of the FM clusters at the vicinity of FM/SG interface are depicted in the right inset of fig. 6(b). The appearance of new layer causes the marked deviation between \( \rho_{\text{FC}} \) and \( \rho_{\text{ZFC}} \) where \( \rho_{\text{FC}} \) is larger in magnitude than \( \rho_{\text{ZFC}} \) below 40 K. Moreover, the appearance of new layer leads to the strong time dependence in \( \rho \) satisfying the stretched exponential which is typically observed in the time evolution of magnetization characterizing the glassy magnetic behavior. The results indicate that frozen FM spins pinned by the SG spins at the FM/SG interface reveal the glassy magnetic behavior in the transport property, although it was originally FM spins before field cooling.

 Since the size and the density of FM clusters within the grain is close to the percolation threshold, tunneling among the FM metallic clusters takes place through the barrier involved with the anisotropy of SG spins. The tunneling barrier is further modified by the appearance of new layers comprising of FFM spins having unidirectional anisotropy which gives rise to the unidirectional shift in the MR-\( H \) curve. The polarization direction of FFM spins is strongly influenced by the direction of the cooling field leading to the spin valve mechanism in MR. We noticed that the decreasing MR-\( H \) curve (from 50 kOe to \(-50 \) kOe) is modified considerably retaining nearly unchanged the increasing MR-\( H \) curve (from \(-50 \) kOe to 50 kOe) where the peak in the decreasing curve is shifted along the negative \( H \)-axis (see figs. 4(c) and (d)) for positive cooling field. On the other hand, the increasing MR-\( H \) curve is modified for the negative cooling field giving rise to the spin valve mechanism. We further note the marked difference between exchange bias field estimated from the different measurement techniques where anisotropies manifested by the coercivities are considerably different for the magnetization and resistivity measurements with \( H_C = 5.8 \) kOe and \( H_C' = 8.8 \) kOe, respectively for the bulk. The simple intuitive model [29] as well as sophisticated theories [30,31] seem to agree that the exchange bias effect should be larger for larger anisotropy of the AFM substance in a FM/AFM heterostructure which has been elaborately described by Nogués et al. [3]. Although the experimental investigations [32–34] dealing with the role of the anisotropy seem to agree with the theories, any quantitative conclusions from them could not be established because of the difficulties for extracting the anisotropy of the AFM component from the heterostructure. In the present observation the larger EB field is found to be associated with the larger coercivity obtained from the MR-\( H \) curve which is in accordance with the proposed theories [29–31] as well as the experimental results [32–34]. Until now, different aspects of the EB effect has been extensively investigated through the magnetization studies which is rather less focused in the MR measurement. Thus a clear understanding of the EB effect correlated with the coercivity of the highly anisotropic substance is still lacking at the microscopic level especially, for the MR measurements.

 Conclusions. – In conclusion, the compound exhibits large magnetoresistance which is involved with the intragranular spin-polarized tunneling mechanism where the tunneling barrier is further modified by the appearance of frozen ferromagnetic spins due to the field-cooling. Frozen ferromagnetic spins give rise to the glassy transport behavior and more importantly the exchange bias effect. Exchange bias field obtained from the magnetoresistance...
is much larger than the value of exchange bias field measured from magnetic hysteresis loop. Semiconducting transport properties associated with the spin-polarized tunneling mechanism and the considerable exchange bias exhibiting spin-valve-like feature in bulk La_{0.88}Sr_{0.12}CoO_3 is attributed to the intrinsic nanostructure in the grain interior which creates a tremendous impact in searching new materials for spintronics applications.

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