Size-dependent electronic-transport mechanism and sign reversal of magnetoresistance in Nd$_{0.5}$Sr$_{0.5}$CoO$_3$

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Abstract

A detailed investigation of electronic-transport properties of Nd$_{0.5}$Sr$_{0.5}$CoO$_3$ has been carried out as a function of grain size ranging from micrometre order down to an average size of 28 nm. Interestingly, we observe a size induced metal–insulator transition in the lowest grain-size sample while the bulk-like sample is metallic in the whole measured temperature regime. An analysis of the temperature dependent resistivity in the metallic regime reveals that the electron–electron interaction is the dominating mechanism while other processes like electron–magnon and electron–phonon scatterings are also likely to be present. The fascinating observation of enhanced low temperature upturn and minimum in resistivity on reduction of grain size is found due to electron–electron interaction (quantum interference effect). This effect is attributed to enhanced disorder on reduction of grain size. Interestingly, we observe a cross over from positive to negative magnetoresistance in the low temperature regime as the grain size is reduced. This observed sign reversal is attributed to enhanced phase separation on decreasing the grain size of the cobaltite.

(Some figures may appear in colour only in the online journal)

1. Introduction

Cobaltites are one of the most interesting systems amongst the strongly correlated electron materials having various fascinating electronic and magnetic properties like manganites. Similar to manganites, cobaltites possess close interplay between charge, orbital, spin and lattice degrees of freedom [1–5]. However, they have one extra degree of freedom, the spin state of Co ion. The multiple spin state found in Co is a consequence of the comparable values of the crystal field splitting energy and Hund’s coupling energy [1–5]. In a material like La$_{1-x}$Sr$_x$CoO$_3$ the Co$^{3+}$ and Co$^{4+}$ ions may have three different spin states each, namely the low spin (LS: $t_{2g}^6e_g^0$ for Co$^{3+}$ and $t_{2g}^5e_g^0$ for Co$^{4+}$), the intermediate spin (IS: $t_{2g}^3e_g^1$ for Co$^{3+}$ and $t_{2g}^2e_g^1$ for Co$^{4+}$) and the high spin (HS: $t_{2g}^1e_g^2$ for Co$^{3+}$ and $t_{2g}^0e_g^2$ for Co$^{4+}$) states [1–5]. Spin state transition may occur under influences like change of temperature [2, 5]. The observed ferromagnetism in cobaltites originates most likely from double exchange interaction between Co$^{3+}$ and Co$^{4+}$ species similar to the case of doped manganites, but dependent on Co spin state. Another most interesting feature of cobaltites is the phase separation effect [4, 6–8] like that of manganites. The low temperature phase of cobaltite materials like La$_{1-x}$Sr$_x$CoO$_3$ has been found to be inhomogeneous where ferromagnetic (FM) regions coexist with various non-FM regions or spin glass regions [7, 8]. Although La$_{1-x}$Sr$_x$CoO$_3$ is one of the most studied cobaltite systems, other classes of rare earth cobaltites (i.e. Pr, Nd, Eu, etc based systems) [9–13] have also been investigated to some extent. Like the La-based cobaltites, the system Nd$_{1-x}$Sr$_x$CoO$_3$ with a lower bandwidth is also known to have a variety of magnetic and electronic properties depending on the hole doping level.

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For instance, in the low doped regime this material displays a phase separated state and glassy magnetic behaviour [9, 10]. This system also exhibits ferrimagnetic ordering below a certain temperature in a particular doping regime [14]. As far as the electronic-transport properties are concerned, Nd$_{1-x}$Sr$_2$CoO$_3$ displays insulating behaviour in the low doped regime and metallic behaviour in the heavily doped regime [9]. However, in any case, the physics of cobaltites has remained less understood compared to that of manganites to date. In particular, regarding the recent trend of investigation on materials with reduced dimension (nanomaterials), cobaltites are comparatively less explored. We here present our results of investigations on different magnetic and electronic-transport properties of Nd$_{0.5}$Sr$_{0.5}$CoO$_3$ in a wide range of grain sizes of this material. According to earlier reports this system in bulk form is metallic up to room temperature and undergoes a transition into a ferromagnetic state below around 225 K [9, 15]. On reduction of grain size down to 28 nm, a complete reversal of electronic- and magneto-transport properties is observed, while a moderate influence on magnetic properties is also found.

2. Experimental details

We synthesized our polycrystalline Nd$_{0.5}$Sr$_{0.5}$CoO$_3$ samples with varying grain size through a chemical route known as pyrophoric reaction [16]. We employed high purity Nd$_2$O$_3$, Sr(NO$_3$)$_2$ and Co(NO$_3$)$_2$·6H$_2$O in stoichiometric proportion and dissolved in distilled water with the proper amount of HNO$_3$. Triethanolamine (TEA) was then added to the solution with a molar ratio to the metal ions of 4:1:1 (Nd/Sr:Co:TEA=1:1:4). This solution was stirred and heated at 180°C until combustion took place. The obtained fluffy powder was divided into four parts, ground thoroughly and calcinated in air (5 h) at different temperatures to obtain polycrystalline Nd$_{0.5}$Sr$_{0.5}$CoO$_3$ samples with different grain sizes. The powder samples were pressed into pellets and again sintered in air at the same temperature for 1 h. The samples are designated as S1150, S950, S850 and S750 after their corresponding sintering temperature in degrees Celsius. The phases of the samples were checked by a high resolution x-ray diffraction technique. The average grain size was estimated by employing a transmission electron microscope (TEM) and a field emission scanning electron microscope (FESEM). The electronic-transport measurements were carried out employing a closed cycle helium refrigeration cryostat fitted inside a superconducting magnet (8 T) in the temperature range of 2–300 K (Cryogenics Ltd, UK). The magnetic properties of the samples were investigated employing a homemade vibrating sample magnetometer (77–300 K) [17] and homemade ac susceptometer (77–300 K). Both these instruments employ a high precision lock-in amplifier (SR830) for signal detection and a PID temperature controller (Lakeshore 331S) to control and monitor the sample temperature.

3. Results and discussion

The room temperature x-ray diffraction patterns of the S1150 and S750 samples are shown in figure 1. No impurity phase is observed in the x-ray diffraction patterns of these samples or in the other intermediate grain-size samples. The x-ray data of all the samples were refined using the Rietveld method assuming the Imma space group of orthorhombic structure. The fitted curves are also shown in figure 1. The obtained lattice parameters and unit cell volumes are listed in table 1. We observe that the change in lattice parameters and unit cell volumes is very small with the change of grain size. This change is also non-monotonic with grain size. This indicates that the grain size has only a negligible influence on structural distortions. The TEM image of the S750 sample shown in figure 2(a) displays a nanometric size of the grains. The distribution of grain size plotted in the inset of figure 2(a) displays a log normal type distribution. The average grain size of S750 is found to be about 28 nm. The high resolution lattice image of S750 in figure 2(b) exhibits that the crystallographic structure is not prominent at the boundary region of the grains (marked by arrows) indicating the disorder in the grain surfaces. The FESEM images of the S850, S950 and S1150 samples are shown in figures 2(c)–(e), respectively. The average grain sizes estimated from the
plots of grain-size distribution of these samples are given in Table 1. The S1150 sample has an average grain size in the micrometre regime. This indicates that the sample should display bulk-like behaviour. Further, we experimentally checked the stoichiometry of our samples through EDS (energy-dispersive x-ray spectroscopy) measurements. There is a possibility of change in stoichiometry in the samples due to the change in calcination temperature. The EDS curves of the S1150 and S750 samples are shown in Figure 3. From these spectra the calculated chemical formulae of the samples are found to be $\text{Nd}_{0.51}\text{Sr}_{0.47}\text{Co}_{1.02}\text{O}_{2.99}$ for the S1150 and $\text{Nd}_{0.51}\text{Sr}_{0.48}\text{Co}_{1.01}\text{O}_{2.99}$ for the S750 sample, respectively. We find that there is negligibly small change of stoichiometry in the samples when the grain size is varied by changing the calcination temperature.

The dc magnetic studies of the samples were carried out in terms of measuring the temperature dependent magnetization. The data were taken at 100 Oe field during heating after cooling the sample at zero field (ZFC) or at 100 Oe field (FC) as shown in Figure 4. All the samples display an FM–paramagnetic (PM) transition at around 225 K ($T_C$). This transition temperature is in agreement with the previously reported value for this system [9]. Although $T_C$ is not found to change much with variation of grain size, the S750 sample displays a much broader FM–PM transition compared to the other samples. This may be attributed to the presence of grain-size distribution. One interesting observed feature is the huge bifurcation between ZFC and FC magnetization of all the samples. This indicates that the low-temperature ferromagnetic phase is highly anisotropic in nature. This anisotropic magnetic behaviour in cobaltites is very common and has been discussed earlier [18, 19]. This is mainly magnetocrystalline anisotropy added to the surface anisotropy originating from surface disorder. One possible
The origin of the magnetic anisotropy is the presence of Co ions as pointed out by different groups earlier[19]. We find that the ZFC magnetization falls sharply just below $T_C$ with decrease of temperature. This is due to the fact that the spins become randomly oriented along their respective anisotropy directions with the lowering of temperature or thermal energy. When the sample is cooled under field, the spins try to align to the field direction instead of falling into the anisotropy barrier. This gives rise to the bifurcation between the ZFC and FC curves. We notice that the bifurcation between the ZFC and FC magnetizations is non-monotonic with the grain size. The maximum bifurcation is found in the S850 and S950 samples. This behaviour is clearly observed from the plot of $M_{FC} - M_{ZFC}$ as a function of temperature shown in the inset of figure 4. We also notice that the magnitude of ZFC magnetization of the samples also displays non-monotonic change with grain size. This is possibly due to the complicated nature of the phase separation of the samples. This phase separated state is affected non-monotonically by the surface disorder. This also indicates that the intrinsic anisotropy of the samples has a non-monotonic dependence on the grain size in the samples. Obviously, when the disorder effect is very high (for S750) the overall magnetization is found to be the lowest as the surface becomes nearly a non-magnetic shell in most of such cases. Further, we study the linear and non-linear ac magnetic susceptibilities of these samples as shown in figure 5. The real parts of the first order or linear susceptibilities ($\chi'_R$) of all the samples show similar behaviour to that found in the dc magnetization study. In this case also we see a transition at around $T_C$ for all the samples. In the inset of figure 5, we have shown the variation of second order ac susceptibility ($\chi''_R$) as a function of temperature. For the entire series of samples we observe a peak at around $T_C$. We know that $\chi''$ appears due to the presence of symmetry breaking spontaneous magnetization in a magnetic system[20]. It ($\chi''$) displays a peak around the FM–PM transition because of the onset of spontaneous magnetization. Thus, our results undoubtedly show that the transition at 225 K is really an FM–PM transition. Next we investigate the electronic-transport properties of our samples and their dependence on grain size. Figure 6 displays the resistivities of all the samples as a function of temperature measured with and without the application of magnetic field. We find that the zero field temperature dependent resistivities of the samples exhibit interesting variation with grain size. Firstly, the magnitude of resistivity increases with decrease of grain size. The overall resistivity increases by roughly one order of magnitude in S750 compared to that of the S1150 sample. The S1150 sample displays metallic behaviour ($d\rho/dT > 0$) in the measured temperature range. The nature of the resistivity and its magnitude for the S1150 sample are consistent with those reported previously for bulk polycrystalline Nd$_{0.5}$Sr$_{0.5}$CoO$_3$[15]. Moreover, we observe that all the samples display an anomaly in the resistivity around their corresponding FM–PM transition temperatures. Interestingly, this anomaly takes the form of a metal–insulator transition for S750 showing a peak (inset of figure 6(d)) at around 230 K (change of sign of $d\rho/dT$). In an earlier report on the La$_{0.5}$Sr$_{0.5}$CoO$_3$ system[21], it was found that on reduction of grain size the metallic behaviour is suppressed into an insulating behaviour. However, in our case we observe the metal–insulator transition in a particular sample of nanometric grain size. Furthermore, a significant change in the nature of resistivity with change of grain size is also observed around the low temperature regime. We find an upturn and shallow minimum in resistivity (at $T_{\text{min}}$) for the S950 ($T_{\text{min}} = 45$ K, table 3) sample in the low temperature regime. This upturn of resistivity is enhanced in the S850 sample and becomes extremely high exhibiting a resistivity minimum at a very high temperature, $\sim$180 K, for the S750 sample. The resistivities of the samples show negligible dependence on magnetic field as is evident from the resistivity measured at 8 T magnetic field.
Figure 6. The measured resistivities of the (a) S1150, (b) S950, (c) S850 and (d) S750 samples as a function of temperature at zero field (symbols). The lines are the same measured at 8 T magnetic field. The inset of (d) shows the magnified image around the metal–insulator transition of the S750 sample.

field. It is found that only around \( T_C \) is the magnitude of resistivity slightly suppressed for all the samples. Such a weak field dependence of the resistivity is consistent with previous reports on cobaltite systems [4, 11]. This also indicates that unlike manganites processes like spin polarized tunnelling are not significant in our systems.

We now try to analyse and find out the possible mechanism of the temperature dependence of resistivity in our samples. In manganites, the temperature dependent resistivity in the metallic regime has been well described through electron–electron (e–e), electron–magnon or electron–phonon scattering as reported earlier by a large number of groups [22, 23]. However, for cobaltites the electronic-transport mechanism is still not explored extensively. Thus, without any pre-assumption we try to fit the \( \rho \) versus \( T \) data of the S1150 sample in the metallic regime with a general power law,

\[
\rho = \rho_0 + \rho_n T^n.
\]  

The parameter \( \rho_0 \) is the residual resistivity. We find a very good fitting as shown in inset (a) of figure 7. Interestingly, the obtained value of \( n \) is 2.1. We know that in the metallic regime the inelastic e–e interaction has a \( T^2 \) dependence [22, 24]. So, we infer from the result of fitting that the dominant interaction in the metallic regime is e–e type. Since \( n \) is not exactly equal to 2, there is an indication that other mechanisms might be present. So, next we try an equation of the form

\[
\rho = \rho_0 + \rho_e T^2 + \rho_p T^p
\]  

to fit the experimental data assuming that e–e interaction is already present. The last term is supposed to represent any other process like electron–magnon or electron–phonon scattering. In this case also a very good fitting is obtained as shown in figure 7 (main panel). The obtained value of \( p \) is 4.4 (±0.5). We know that the second order electron–magnon scattering goes as \( T^4 \). So looking at the value of \( p \) we can primarily attribute the third term on the right hand side of equation (2) to electron–magnon scattering. However, the error in the value of \( p \) indicates that electron–phonon scattering which has a \( T^5 \) dependence in the temperature regime below the Debye temperature cannot be completely ruled out. We applied equations (1) and (2) in the metallic regimes of the S950 and S850 samples also. The fits employing equation (2) for the S950 and S850 samples are shown in insets (b) and (c) of figure 7, respectively. The obtained fitting parameters of all the samples are summarized in table 2. Clearly, we find a gradual deviation (increase) of
Table 2. The obtained parameters employing equations (1) and (2) in the metallic regimes of the S1150, S950 and S850 samples. $R^2$ values of the fittings are also given.

| Sample | $\rho_0$ (10^{-4} \, \Omega \text{ cm}) | $\rho_e$ (10^{-9} \, \Omega \text{ cm K}^{-2}) | $\rho_p$ (10^{-15} \, \Omega \text{ cm K}^{-n}) | $p$ | $R^2$ |
|--------|-----------------|-----------------|-----------------|-----|-----|
| S1150  | 0.68            | 1.07            | 0.17            | 4.4 | 0.9998 |
| S950   | 4.93            | 3.36            | 47.13           | 4.0 | 0.9990 |
| S850   | 9.69            | 4.33            | 25.85           | 4.2 | 0.9888 |

Table 3. The positions of resistivity minima ($T_{min}$) and the obtained parameters on employing the formula $\rho = \rho_0 - \rho_{ee} T^{1/2} + \rho_p T^n$ in the low temperature regime of the samples. $R^2$ values of fittings are also given.

| Sample | $T_{min}$ (K) | $\rho_0$ (10^{-3} \, \Omega \text{ cm}) | $\rho_{ee}$ (10^{-5} \, \Omega \text{ cm K}^{-1/2}) | $\rho_p$ (\Omega \text{ cm K}^{-n}) | $p$ | $R^2$ |
|--------|---------|-----------------|-----------------|-----------------|-----|-----|
| S950   | 45      | 0.53            | 0.76            | 8.52×10^{-7}    | 0.7 | 0.9990 |
| S850   | 75      | 1.13            | 1.53            | 2.40×10^{-15}   | 5.0 | 0.9714 |
| S750   | 180     | 6.83            | 24.88           | 4.06×10^{-11}   | 3.2 | 0.9983 |

The most fascinating observation in the transport properties is the enhancement of the low temperature minima due to size reduction as mentioned earlier. We attempt to find out the possible origin of this resistivity upturn. Previously, the upturn of resistivity in various materials including manganites was attributed to phenomena like the Kondo effect [25], e–e interaction [26], the Coulomb blockade (CB) effect [27], etc. The Kondo effect is observed in low resistive diluted magnetic alloys due to the coupling of conduction electron spins with the magnetic impurity moment in the low temperature regime. This gives rise to a low temperature upturn of resistivity which is drastically suppressed on application of magnetic field. As the temperature is increased, various temperature dependent inelastic scattering processes come to play and as a result a minimum in the resistivity is formed. Assuming the Kondo effect the resistivity in the low temperature regime can be written as

$$\rho = \rho_0 - \rho_{ee} \ln T + \rho_p T^n.$$  \hspace{1cm} (3)

Here we have included the inelastic scattering term in the form of a general power law as $\rho_p T^n$ which increases with increase of temperature. Another origin of minima in the resistivities found in different disordered (structural and compositional) materials has been attributed to the elastic e–e interaction. In this case the upturn of resistivity takes place due to the quantum interference effect. In such disordered systems, at low temperatures, the mean free path of the electrons becomes small and the electrons are best described as diffusing from site to site by multiple elastic scattering. Previously, the low temperature resistivity minima found in some polycrystalline manganites have been attributed to this e–e interaction effect [28, 29]. Including the correction term due to e–e interaction and the inelastic scattering term the resistivity in the low temperature regime can be described as

$$\rho = \rho_0 - \rho_{ee} T^{1/2} + \rho_p T^n.$$  \hspace{1cm} (4)
On the other hand, the resistivity due to the Coulomb blockade effect is empirically given as

$$\rho = \rho' \exp \left( \sqrt{\frac{\Delta}{T}} \right),$$

(5)

Here $\Delta$ is equivalent to the charging energy of an electron. This energy is required as the grains become charged when an electron leaves a grain.

We apply equations (3)–(5) individually and also different combinations of these equations to fit the low temperature resistivity data of the S750 sample. To determine the best fit we plot the difference between experimental and fitted data with temperature as shown in inset (a) of figure 8. We find that the Coulomb blockade effect (equation (5)) gives an extremely bad fit to the experimental data. This indicates that the Coulomb blockade effect which is generally expected to be present in granular materials is not responsible for the low temperature upturn of resistivity of the S750 sample. We notice that the best fit is obtained on application of equation (4) (figure 8 and inset (a)) and the combination of equations (3) and (4). However, when we combine equations (3) and (4), a good fit is achieved with a negative value of the coefficient $\rho_n$ which is meaningless and unphysical. Furthermore, the observed very small effect of magnetic field on the resistivity minima also indicates that the Kondo effect is very unlikely in our samples. So we conclude that the elastic e–e interaction is the dominant mechanism which gives rise to the low temperature upturn and resistivity minimum in the S750 sample. The resistivity of this sample is of the order of Mott’s maximum metallic resistivity ($\sim 10 \text{ m}\Omega \text{cm}$) indicating the plausibility of e–e interaction in it. We also employed equation (4) on the other samples and observed reasonably good fits to the experimental data (inset (b) of figure 8). The obtained parameters are listed in table 3. From table 3 it is clear that the e–e interaction coefficient ($\rho_{ee}$) monotonically increases with decrease of grain size. The coefficient $\rho_{ee}$ is explicitly given by $\rho_{ee} = 0.0309\sqrt{\frac{\kappa_B}{\hbar^2 D^2_0} e^2}$ [30], where $D$ is the diffusion constant.

While measuring the magnetoresistance (MR($H$, $T$) = $\frac{\Delta (H,T) - \rho (0,T)}{\rho (0,T)}$) of these samples we observed a peculiar sign reversal of the MR in the low temperature environment.
regime. From the MR versus $H$ curves shown in figure 10 we observed that the S1150 sample displays a very small but positive MR ($<$1%) at 3 K. The MR of S950 at the same temperature is also positive and small ($<$0.5%). However, a small negative MR was observed for sample S850. This negative value of MR is enhanced upto 4.5% in the S750 sample at that temperature. This clearly demonstrates that on reduction of grain size negative MR is enhanced in our samples in the low temperature regime. The positive MR, which is a common property of materials, observed in S1150 may be due to the Lorentz force on the charge carriers. However, the observed negative MR can be attributed to the phase separation effect. It is an extensively discussed issue that in manganites the large negative MR originates partially due to the existence of phase separation and field induced percolation of phases. With decrease of grain size, the phase separation effect is possibly enhanced in our samples. The negative MR due to this phase separation competes with the positive MR and displays the highest value in S750. The enhancement of phase separation is, we think, due to the structural disorder which can disturb the spatial extent or long range correlation of individual phases. Furthermore, we have observed a prominent hysteresis in the MR of the S750 sample at 3 K (very weak for S850 at 3 K). Such hysteresis was previously attributed to the presence of magnetic hysteresis in the corresponding sample [23]. On the other hand, around the FM–PM (225 K) transition all the samples exhibit negative MR as shown in figure 10. This is also visible in figure 6. Such negative MR is generally attributed to the suppression of spin disorder on application of magnetic field in this temperature regime. This can also be partially due to the intrinsic enhancement of the phase separation around the transition temperature of such materials.

4. Conclusions

In summary, we have carried out a detailed investigation of the electronic-transport properties with additional magnetic characterization of polycrystalline $\text{Nd}_{0.5}\text{Sr}_{0.5}\text{CoO}_3$ with variation of grain size down to 28 nm. All the samples display a ferromagnetic–paramagnetic transition at around 225 K. We observe a metal–insulator transition in the lowest grain-size sample while the highest grain-size bulk-like sample is metallic up to room temperature. The temperature dependence in the metallic regimes of the samples is best described through e–e interaction and electron–magnon or electron–phonon interaction. We have also observed a huge enhancement of the low temperature resistivity upturn as the grain size is reduced. This upturn is attributed to the elastic e–e interaction generally found in disordered materials. The enhancement of this interaction with reduction of grain size is attributed to enhanced disorder in the sample (surface effect). At very low temperature the observed change of sign of the magnetoresistance from positive to negative with decrease of grain size has been attributed to the enhanced phase separation effect.

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