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Research articles

# Experimental observation of weak antiferromagnetic correlation between blocked spins in the nanocrystalline $La_{0.45}Ca_{0.55}MnO_3$ compound at low temperature

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ABSTRACT

In the present study, an attempt has been taken to probe the meta-stable nature of the magnetic ground state in the nanocrystalline $La_{0.45}Ca_{0.55}MnO_3$ compound at the low temperature through magnetic and magneto-transport properties and compared the results from the observation with the bulk polycrystalline form of the $La_{0.45}Ca_{0.55}MnO_3$ compound. The charge-ordered antiferromagnetic ground state in the bulk compound transformed into a ferromagnetic state in the nanocrystalline sample. Additionally, in the nanocrystalline form of the compound, a short-range antiferromagnetic correlation between the blocked spin is noticed at the low temperature region ($T \leqslant 50$ K). Such weak magnetic interaction between magnetic spins or sublattices is probed through the magneto-transport and magnetocaloric measurements study in the nanocrystalline $La_{0.45}Ca_{0.55}MnO_3$ compound.

## 1. Introduction

In the previous two decades, the doped perovskite manganite compounds were extensively studied owing to their charismatic physical properties. The inflexible ground state in undoped $RMnO_3$ compounds (R = trivalent ion) become flexible with the substitution of trivalent site by the bivalent elements like Ca, Sr, Ba etc. Competing nature of the different type of magnetic interactions induced by the doping of bivalent ions originate many stimulating phenomena in this class of materials [1–11]. Moreover, a strong correlation was observed in between the magnetic and magneto-transport properties of the doped perovskite manganites [12,13]. The ferromagnetic ground state in the bulk manganite sample favors metallic nature where as antiferromagnetic correlation prefers insulating ground state of the system [14,15]. However, for the nanocrystalline or disorder ferromagnetic compounds, a different nature was also observed [16–20]. Surface spin disorder in nanoparticles is a very decisive parameter in order to determine the magneto-transport properties. Due to the insufficient percolation paths, disorder ferromagnetic nanoparticles exhibit the strong insulating nature in temperature dependent resistivity study [21,22]. In such a system, the change in resistivity is very small due to any feeble change of magnetic configuration. In contrast to that, as magnetocaloric effect (MCE) is associated with the change of magnetic entropy directly, it may be used as a primary sensitive tool to detect such feeble transitions [23–25]. Physical properties of low dimensional manganites were extensively studied due to their charming responses over their surface spin randomness mediated complicated magnetic configuration.

After the discovery of colossal magneto-resistance (CMR) by Jin et al. in 1994 in low dimensional La-Ca-$MnO_3$ manganites, this compound was widely studied having different Ca-doping concentration and dimensions [26]. According to the phase diagram for the bulk $La_{1-x}Ca_xMnO_3$ compound, the whole Ca-doping range (0 to 1) may be divided into three broad regions [27–31]. When $x < 0.5$, ferromagnetic ground state appears and for $0.5 < x < 0.87$, charge ordered antiferromagnetic ground state was noticed. However, for $x > 0.87$, compound shows canted antiferromagnetic like ground state at the low temperature region. A drastic change in the physical properties were found in the compound having composition in different regions [20,32]. On the other hand, except composition, the physical properties are also very much sensitive on the dimension of the studied compound. Various striking physical properties are evolved with the modification of the already existed ground state with varying the dimension of the system. Due to

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the energetically favorable phase separation scenario, a core–shell type structure was formed in the nanocrystalline compound. Magnetically, the core part exhibits the same characteristic nature as the parent bulk compound, whereas, the magnetic ground state of the shell section behaves differently with respect to the core part [20,33–36]. Core shell structures are not only possible scenario occurring during the size -reduction in case of doped manganite compound. Sarkar et al. had earlier reported the effect of size reduction in the nanocrystalline $La_{0.5}Ca_{0.5}MnO_3$ compound. They have shown that the long-range charge-orbital ordering state present in the high temperature region was kinetically arrested and this will prevent the change of crystal structure upon cooling the system. As a result, low temperature ground state is highly destablized and the freezing of the crystal structure was explained by the increment of surface pressure acting on the nanoparticle sample [37,38].

It is reported that for the charge ordered antiferromagnetic nanoparticle, in the shell part generally a short-range type ferromagnetic correlation has been observed between the surface spins. An opposite nature was found for the nanoparticles of ferromagnetic compound [17,21,22]. Dong et al. reported theoretically about the quantitative ferromagnetic fraction with the dimension of the antiferromagnetic nanoparticles [17]. The nanoparticles with very small size possesses generally a strong ferromagnetic interaction between the magnetic sublattices, suppressing the charge-ordered antiferromagnetic nature either partially or fully [17].

A common phenomena called “spin-blocking” has been noticed in case of the nanocrystalline compound at the low temperature region. When the sample was cooled down in the absence of any external magnetic field, below a particular temperature, some tiny domains get randomly pinned in their respected positions. The temperature at which such blocking of magnetic spins start to appear is called the “blocking temperature” ($T_B$). However, the magnetic nature of such pinned spins are not still clear till date.

In our earlier report, we have established the coexistence of both antiferromagnetic (AFM) and ferromagnetic (FM) phases in the ground state by studying the magnetotransport properties (basically from the magnetoresistance (MR) plots) (predominant phases) [20]. In our present study, we have probed the presence of AFM interaction between the blocked spins (the quantitative amount is very small) through the magnetocaloric effect study. It was established that any feeble magnetic transition can be highlighted from the temperature dependence of magnetic entropy change curves. To serve our purpose we have choose a very well known charge ordered antiferromagnetic compound $La_{0.45}Ca_{0.55}MnO_3$. We have studied the physical properties of bulk as well as nanocrystalline compound having particle size ~70 nm [20]. Our study reveals that antiferromagnetic part markedly suppressed in nanocrystalline compound and predominant ferromagnetism appears. However, a very weak antiferromagnetic correlation is present in between the blocked spins (in the shell as well as interface between core and shell part) at the low temperature region. Additionally, an insulator–metal transition takes place in presence of the external magnetic field.

## 2. Experimental details/sample preparation, characterizations and measurements

We have used the well-known sol–gel chemistry technique to prepare the bulk as well as nanocrystalline $La_{0.45}Ca_{0.55}MnO_3$ compounds [20,39,40]. The precursor gel was prepared by using the pre-heated $La_2O_3$, $CaCO_3$ and $MnO_2$ having purity greater than 99.99 %. Suitable amount of the citric acid and oxalic acid were also taken. The details about the precursor gel preparation procedure is given in Refs. [12,20,39,40]. After the formation of the gel, it was decomposed at slightly higher temperature and as a result, black porous powder was obtained. The powder sample was pelletized by using hydrostatic pressure and final heat treatment was performed depending upon the requirements. To achieve the nanocrystalline form of the compound, final sintering was performed at 900 °C for 3 h and final heat treatment at 1400 °C for 36 h has been performed to get the bulk form of the compound.

X-ray diffraction (XRD) measurements were carried out in a Rigaku-TTRAX-III diffractometer for both the bulk and nanocrystalline compounds, suggesting the chemically single phase nature of the compounds and the compounds adopted an orthorhombic crystal structure with *Pmma* space group which was discussed in our earlier report [20]. Additionally, the Transmission Electron Microscopy (TEM) study was performed to determine the average particle size of the nanocrystalline compound and the calculated average particle size was found to be 70 nm [20].

Magneto-transport measurements were performed by utilizing four probe technique in longitudinal geometry using a variable temperature cryostat (Cryogenic, U.K.) equipped with a 9 Tesla super conducting magnet. A Super conducting Quantum Interference Device (SQUID) (Quantum Design) was employed to study the magnetic and magnetocaloric properties of the compounds.

## 3. Results and discussion

The magnetization of the bulk and nanocrystalline compound were measured in two different measurement protocols namely the zero-field-cooled (ZFC) warming and field-cooled (FC) warming. In case of the ZFC measurement, the sample was first cooled down to the lowest measuring temperature ($T = 5$ K) from the room temperature in the absence of any external magnetic field. At $T = 5$ K, a desired static magnetic field (in this present study, $H = 100$ Oe) was applied and the magnetization data were recorded during the warming cycle from $T = 5$ K to $T = 300$ K. Whereas, in the FC measurement protocol, sample was cooled down form room temperature to low temperature in the presence of desired external magnetic field (in the present study it was 100 Oe) and the magnetization data were collected during the warming cycle as like ZFC magnetization data. Similarly as reported in earlier studies, at the lower temperature region $T \leqslant 150$ K, the charge ordered antiferromagnetic response of the bulk sample manifests by very small magnetization [20]. The peak at $T \sim 250$ K in the temperature dependent magnetization data of the bulk compound indicates the dominating signature of charge order state as shown in Fig. 1(a). In addition, a small bifurcation in between the ZFC and FC magnetization has been observed due to the blocking of magnetic spins at the low temperature region where both thermal and magnetic energy is insufficient to align the magnetic spins along the applied magnetic field direction.

However, a different result has been noticed in the temperature dependent of magnetization data in case of nanocrystalline compound. In contrast to the bulk counterpart, the magnetic ground state is drastically modified in case of nanocrystalline sample. The temperature dependent of magnetization curves indicates the dominating ferromagnetic ground state of the sample with Curie temperature, $T_C \sim 235$ K as shown in Fig. 1(b). A strong bifurcation between ZFC and FC magnetization data has been noticed below the spin blocking temperature ($T_B \sim 200$ K). It is quite common trend for nanocrystalline compound of similar materials [41]. Such different nature of ZFC and FC magnetization signifies the existence of sufficient spin blocking and spin pinning center in the nanocrystalline compound. In the ZFC measurement, since the sample was cooled down in the absence of magnetic field, such pinned or blocked spins are get randomly arrested and resulting the lower value of the magnetization at the low temperature region. During the magnetization measurements in the warming cycle (ZFC), the temperature fluctuation helps to loose the already pinned magnetic spins, resulting the increment in the value of the magnetization with temperature below $T_B$. On the other hand, the sample was cooled down in the presence of magnetic field in case of FC magnetization measurement protocol (applied from the paramagnetic region), the magnetic spins are already aligned along the applied magnetic field

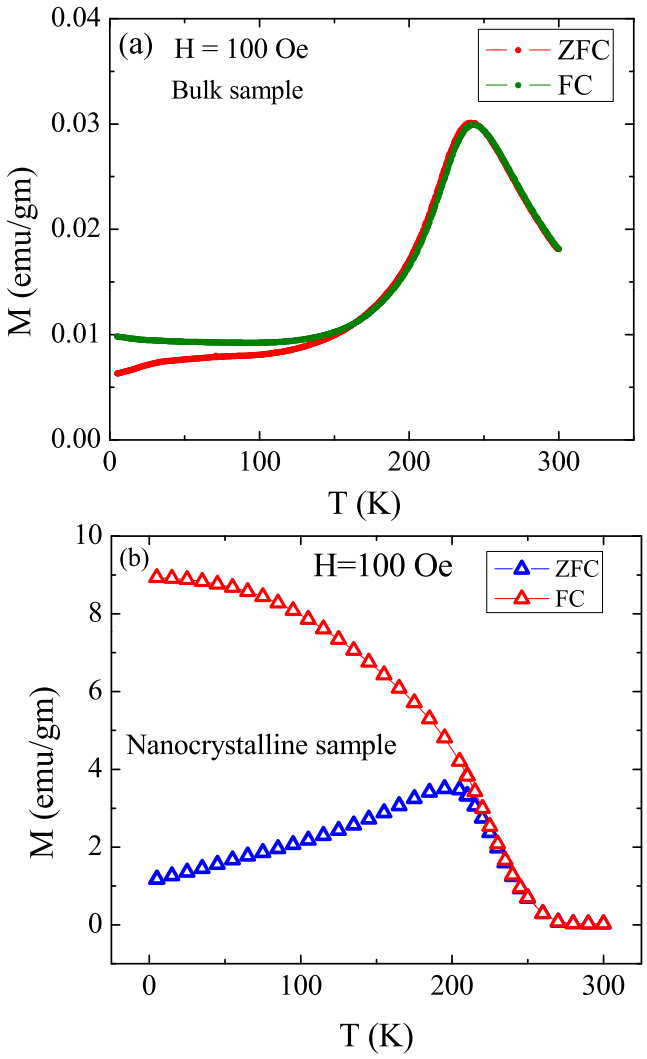


**Fig. 1.** Magnetization (M) as a function of temperature in the presence of H = 100 Oe external magnetic field for (a) bulk and (b) nanocrystalline $La_{0.45}Ca_{0.55}MnO_3$ compounds in two different protocols namely, zero-field-cooled warming (ZFC) and field-cooled-warming (FCW).

direction and as a outcome large magnetization value is achieved at the low temperature region.

A generic nature of the charge-ordered antiferromagnetic compound, called "insulating nature" has been observed in the temperature dependent resistivity data with the reduction of the temperature [5,12]. However, in some cases depending upon the availability of the sufficient percolation paths, the well-pronounced insulator–metal transition appears in the nanocrystalline compound [42–44]. In this present study, variation of the temperature dependent resistances of the bulk and nanocrystalline compound are presented in Fig. 2(a) and (b) respectively. For the bulk sample the resistance measurements were carried out down to 60 K (before reaching measurement limit). However in case of the nanocrystal, it was 5 K. In both the cases, dominant insulating nature reveals the insufficient percolation paths for conduction electrons even in the presence of 10 kOe external magnetic field. However, in case of the nanocrystalline compound, slight decreasing nature of the resistance in the presence of magnetic field indicates the fragile nature of the insulating state. This fragile insulating state basically consists of some randomly oriented non-interacting ferromagnetic domains, where 10 kOe magnetic field is not enough alone to create necessary percolation path for conduction of electrons in the system, and hence the conduction electrons are trapped within the domains, resulting insulating ground state of the system. Although, due to the activation of magnetic spins in presence of 100 Oe magnetic field, a FM like signature is observed in the M-T curve for the nanocrystalline compound. To clearly visualize the fragile nature in the nanocrystalline compound in presence of 10 kOe magnetic field, we have calculated the magnetoresistance (MR) of both the compounds (bulk nd nanocrystalline). MR is defined as the change of resistance of a compound in the presence of the external magnetic field. It is generally calculated by using the mathematical expression as suggested by Mohamed et al. [45],

$$MR\% = \frac{R(H) - R(0)}{R(0)} \times 100\% \quad (1)$$

Surprisingly, the pronounced enhancement of the magnetoresistance in the nanocrystalline compound is clearly identified and displayed in Fig. 2(c). Moreover, at very low temperature region (T < 50 K), magnetoresistance of the nanocrystalline compound get reduced. The origin of such anomalous nature is discussed in the later part of this paper.

Form the above discussion regarding the magnetic and magneto-transport properties of bulk and nanocrystalline compound, we can summarize two interesting points which are:

(i) In contrast to the bulk counterpart, nanocrystalline $La_{0.45}Ca_{0.55}MnO_3$ compound exhibits predominant ferromagnetic nature with sufficient spin blocking at the low temperature region which is evident from the huge bifurcation in between ZFC and FC temperature dependent magnetization curves [38,46].

(ii) The insulating state in nanocrystalline compound is more fragile with respect to the bulk sample and an anomalous nature in magnetoresistance has been noticed at T ⩽ 200 K.

To recognize the different magnetic phases induced in the ground state of the nanocrystalline $La_{0.45}Ca_{0.55}MnO_3$ compound, magnetocaloric effect study can be used as an influential tool. To calculate the change of magnetic entropy (ΔS) in presence of different external magnetic fields, a set of virgin field dependent magnetization curves are recorded at various temperatures ranging from 5 K to 310 K with an interval of 10 K as shown in Fig. 3. For ferromagnetic (antiferromagnetic) compound the magnetization generally decreases (increases) with increasing temperature. However, In the present study the signature of the both ferromagnetic (low field region) and antiferromagnetic (high field region) phases are present. A linear dependence of magnetization with magnetic field is observed at T = 300 K data, suggesting the paramagnetic state of the system. The isothermal magnetic entropy changes (ΔS) according to classical Maxwell's thermodynamic relation is given by [47]

$$\Delta S_M(T,H) = S_M(T,H) - S_M(T,0) \quad (2)$$

$$= \int_0^H (\partial M/\partial T)dH$$

Fig. 4 shows the variation of magnetic entropy changes (ΔS) with temperature for the low field values. The peak observed at around T ∼ 230 K due to the ferromagnetic ordering of magnetic ions present in the studied nanocrystalline compound. This peak position is almost same as the $T_C$ of the compound. Additionally, at the low temperature region (T ∼ 50 K), a cross-over from positive to negative value of −ΔS has been observed. This crossover of magnetic entropy change is coined as inverse magnetocaloric effect (IMCE) [48–50]. Basically, IMCE can be observed due to the dominant nature of the antiferromagnetic ground state or mixed magnetic phases (AFM and FM) at the low temperature region. In our system, we already mentioned the presence of weak antiferromagnetism along with ferromagnetic components in the ground state of the

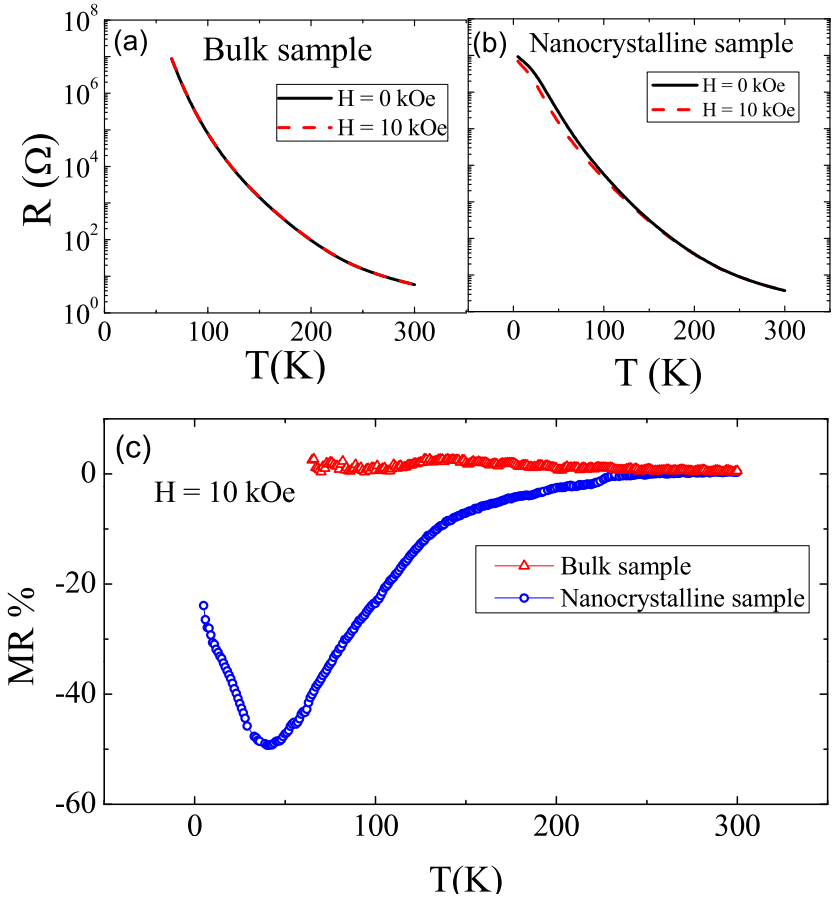


**Fig. 2.** Resistance (R) as a function of temperature in the absence and in the presence of 10 kOe external magnetic field for (a) bulk and (b) nanocrystalline $La_{0.45}Ca_{0.55}MnO_3$ compounds, (c) Temperature dependence of the magnetoresistance (MR) in the presence of 10 kOe external magnetic field for both the bulk and nanocrystalline $La_{0.45}Ca_{0.55}MnO_3$ compounds.

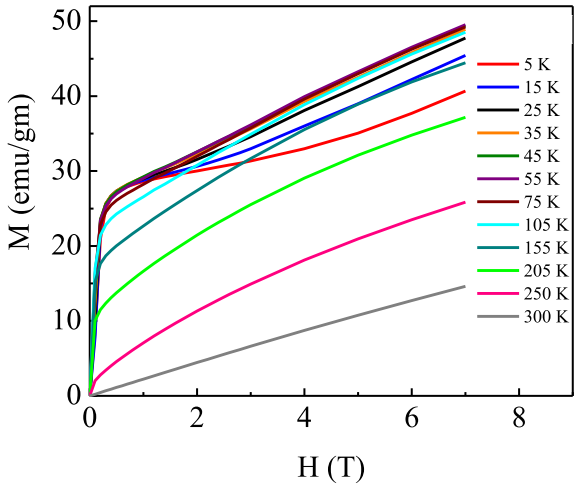


**Fig. 3.** Isothermal magnetization curves for nanocrystalline $La_{0.45}Ca_{0.55}MnO_3$ compound, recorded under ZFC condition for some selected temperatures.

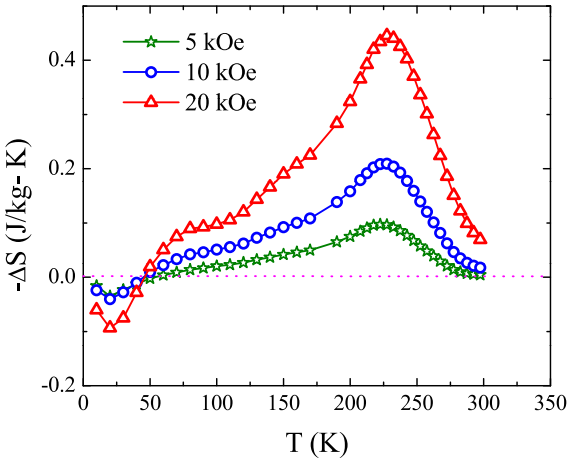


**Fig. 4.** Magnetocaloric entropy change (ΔS) as a function of temperature in the presence of different external magnetic fields for nanocrystalline $La_{0.45}Ca_{0.55}MnO_3$ compound.

studied systems. Even in the field change of 70 kOe, the dominating signature of antiferromagnetism is still persists and we are unable to detect the peak position up to our measuring temperature scale as shown in Fig. 5. This may be due to the randomly arranged ferromagnetic domains in the shell part of the nanocrystalline system where 70 kOe magnetic field is not enough to orient the magnetic spins of each domain along the field direction. The obtained maximum value of entropy change (ΔS) is ~1.72 J/kg-K for a magnetic field variation of 0 kOe → 70 kOe.

The confirmation of the presence of low temperature weak antiferromagnetism is also given by the isothermal magnetization data recorded under ZFC protocol as shown in the inset of Fig. 5. Although the value of magnetization sharply increases at low field region (signature of ferromagnetism), it still not saturates at higher field values and the

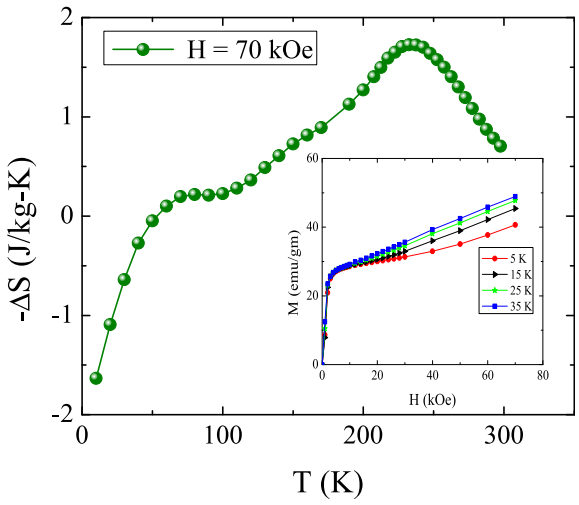


**Fig. 5.** Magnetocaloric entropy change (ΔS) as a function of temperature in the field variation of 70 kOe for nanocrystalline $La_{0.45}Ca_{0.55}MnO_3$ compound. The inset shows the virgin M-H curves recorded at low temperature regions.

magnetization increases with increasing the temperature at higher field values, a characteristic for antiferromagnetic system. This obtained experimental results further confirms the fact that the ground state of the studied nanocrystalline system consists of mixed magnetic phases (both ferromagnetic and weak antiferromagnetic) at low temperature region. Such development of weak antiferromagnetic correlation between the 'shell clusters' act as extra spin scattering center and resulting the reduced magnetoresistance at the low temperature region as pointed out in Fig. 2(c).

## 4. Conclusions

To summarize the present study, we have performed a comparative study on the magnetic and magnetotransport properties of both nanocrystalline and bulk polycrystalline $La_{0.45}Ca_{0.55}MnO_3$ compounds and mainly focused on the magnetocaloric effect of the nanocrystalline form of the compound. An antiferromagnetic phase has been found along with the ferromagnetic ground state in case of the nanocrystallie form of the compound. The presence of such antiferromagnetic correlation in the low temperature limit of nanocrystalline system is verified from the temperature dependent of magnetic entropy change ( $-\Delta S$) data which is further supported by the magneto-transport study. However, the temperature dependent magnetization measurements do not exhibits such antiferromagnetism in the low temperature region of the nanocrystalline system where well-pronounced ferromagnetism appears at $T_C \sim 235$ K. This phenomenon is well-described by considering the antiferromagnetic correlation between the blocked spins in nanocrystalline compound.

## CRediT authorship contribution statement

**Kalipada Das:** Conceptualization, Formal analysis, Supervision, Writing - original draft, Writing - review & editing. **Dipak Mazumdar:** Data curation, Formal analysis, Visualization, Writing - original draft, Writing - review & editing. **P. Dasgupta:** Data curation, Writing - review & editing. **Sanjay Kumar:** Data curation, Writing - review & editing. **I. Das:** Conceptualization, Supervision, Writing - review & editing.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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