



Research articles

Study of devitrification of kinetically arrested magnetic phase and magnetocaloric effect for nanocrystalline $\text{Pr}_{0.65}(\text{Ca}_{0.7}\text{Sr}_{0.3})_{0.35}\text{MnO}_3$ compound

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ABSTRACT

Magnetic and magnetocaloric effect of nanocrystalline $\text{Pr}_{0.65}(\text{Ca}_{0.7}\text{Sr}_{0.3})_{0.35}\text{MnO}_3$ compound have been investigated. In addition to the charge ordering and antiferromagnetic ground state, several fascinating physical properties were appear depending upon the controllable phase separation scenario. The different proportions of the magnetic phases appeared at the low temperature is directly related with the history of applied magnetic field and temperature. Moreover, the nucleation (arrested magnetic phase) and devitrification nature of magnetic phase were addressed considering different widths of the supercooling and kinetic arrest bands. Additionally, nanocrystalline compound exhibits the large inverse magnetocaloric effect at low temperature. The inverse magnetocaloric effect arises possibly due to the arrested antiferromagnetic phase of this compound.

1. Introduction

Several intriguing phenomena governed by the coexisting magnetic phases have been gained an utmost attention to the current research direction from the fundamental as well as application point of view [1–10]. As a magnetic phase separated system, doped perovskite manganite stands in the forefront position [11,12]. Generally undoped perovskite manganites having general formula LnMnO_3 exhibits antiferromagnetic ground state. However depending upon the substitution of trivalent ‘Ln’ site with suitable divalent ions (Sr^{2+} , Ca^{2+} etc.) introduced different magnetic phases within the doped compounds [13–15]. Additionally it is also reported that the phase separation may appear due to the size reduction of a bulk compound having single magnetic phase [16,17]. One of the most well known and versatile source of the phase separation in doped manganites system is mismatching of the A-site cationic radius [18]. In strongly correlated electronic system magnetic and electron transport properties are strongly influenced by each other [19,20]. Due to doping, in addition to the phase separation, several other existing phenomena appears in doped manganites which are charge ordering, magnetocaloric effect, meta magnetic transition, colossal magneto resistance etc.

Charge ordering is the real space ordering of the Mn^{3+} and Mn^{4+} ions within the crystals. It is almost a general formula for nearly half doped manganites. It is also well documented that due to the application of external magnetic field or particle size reduction charge

order antiferromagnetic phase become fragile [21–29]. Magnetocaloric effect (MCE) is quantified by the isothermal magnetic entropy change or adiabatic temperature change of a magnetic material when it is exposed in a magnetic field. Due to the application of the external magnetic field, the randomness of the magnetic moments within the material were influenced greatly. Regarding this context, study of the magnetocaloric effect in such phase separated system is important.

Previously it is well established that in addition to the application perspectives, MCE can be used as a powerful tool to elucidate the nature of the ground state of a magnetic material [10,30]. Among the manganites family, $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$, $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$, $\text{Pr}_{1-x}\text{Ca}_x\text{MnO}_3$, $\text{Pr}_{1-x}\text{Sr}_x\text{MnO}_3$ are extensively studied during several decades [29,31–33]. Depending upon the doping element and doping fraction, compound exhibits different magnetic ground state. Regarding this context one point should mentioned here that as ionic radius of ‘Sr’ is comparatively large from ‘Ca’, generally double exchange mechanism should be more favorable in ‘Sr’ doped compound. By tuning the proportionate fraction of ‘Ca’ and ‘Sr’, co-existing magnetic phases were appear in La-Ca-Sr-MnO or in Pr-Ca-Sr-MnO compounds. For single magnetic phase antiferromagnetic compound, field induced meta-magnetic transition generally take place at very high magnetic field [34]. In contrast to that for a phase separated materials such transition may occur at comparatively lower magnetic field [35]. Previously, Biswas et al. have reported that not only in bulk compounds, phase coexistence

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phenomena may be persists even in nanocrystalline sample also [10]. Additionally, in nanocrystalline compound due to enhance surface effect many fascinating physical properties were also present. Considering such contexts, in our present study we have selected nanocrystalline $\text{Pr}_{0.65}(\text{Ca}_{0.7}\text{Sr}_{0.3})_{0.35}\text{MnO}_3$ compound. According to the reported studies, this nanocrystalline compound possesses a paramagnetic to charge order antiferromagnetic transition at $T \sim 210$ K. With further lowering the temperature some fraction of the compound undergoes a ferromagnetic transition ($T \leq 100$ K) and a mixed ground state (FM and AFM) is appears at low temperature region [10,36]. The response of magnetic phases with temperature is strongly depends upon the exchange coupling between the magnetic moments. In case of ferromagnetic compound generally magnetization decreases with temperature below T_C for a multi domain compounds. In a phase separated (ferromagnetic and antiferromagnetic) compound, due to the presence of the interfacial distorted magnetic unit cells, it is quite expected that the magnetization dynamics should be different.

The aim of the present study is to understand the phase separation in the presence of different magnetic field and explore the dynamics of the different phases (FM and AFM) at low temperature. Regarding this context, it is important to mention that phase transformation (due to applied field) can be easily take place in case of the spontaneously phase separated sample compared to the robust charge ordered compounds. Hence, it is quite expected to get the pronounced effect of temperature and applied magnetic field on the dynamical phase separation scenario of nanocrystalline $\text{Pr}_{0.65}(\text{Ca}_{0.7}\text{Sr}_{0.3})_{0.35}\text{MnO}_3$ compound.

Our study indicates that some fraction of high temperature magnetic phase is kinetically arrested at low temperature and it is very much sensitive with the application of the cooling magnetic field. Depending upon the phase fractions at the low temperature, the devitrification of the arrested phase starts at different temperature values. Moreover, the competing nature of the different magnetic interactions at different temperature regime have also been addressed.

2. Sample preparation, characterizations and measurements

Nanocrystalline $\text{Pr}_{0.65}(\text{Ca}_{0.7}\text{Sr}_{0.3})_{0.35}\text{MnO}_3$ was prepared by using well known sol-gel method. Here 99.99% pure Pr_2O_3 , CaCO_3 , SrCO_3 , MnO_2 were taken as starting components. At first Pr_2O_3 was preheated at 500°C for 5 h. Then appropriate amount of HNO_3 was mixed with all the components for the conversion of carbonate to their nitrate form. But for the clear solution of MnO_2 at first oxalic acid was mixed to make it oxalate and then add some HNO_3 for its nitrate form. Some millipore water was added with all the nitrate form of the components. When each and every components make clear solution, they are mixed into a large container and stirred up with a magnetic stirrer at room temperature. After adding adequate amount of citric acid, the solution was heated at $80\text{--}90^\circ\text{C}$ until the gel was formed. Then black porous powder was produced by heating the gel at $200\text{--}230^\circ\text{C}$. The powder was then palletized and make the nanocrystalline compound of $\text{Pr}_{0.65}(\text{Ca}_{0.7}\text{Sr}_{0.3})_{0.35}\text{MnO}_3$ at 900°C for 3 h. To characterize the nanocrystalline compound, room temperature x-ray diffraction measurement was carried out using Rigaku-TTRAX-III diffractometer. To determine the average particle size of the nanoparticles a scanning electron microscope have been utilized. A super conducting quantum interference device (SQUID-VSM) was used for magnetization measurements within the temperature range of $2\text{--}300$ K and magnetic field variation up to 70 kOe.

3. Results and discussion

Room temperature x-ray diffraction measurement indicates the single phase nature of the sample. Average particles size calculated from x-ray line width broadening and scanning electron microscopy image

and it was found to be ~ 40 nm. The details about the characterization of the nanocrystalline compound is reported earlier in ref [36].

Magnetization as a function of temperature of the nanocrystalline compound have been measured in three different protocols.

Zero Field Cooled Warming (ZFCW): In this method, at first sample was cooled down at zero external magnetic field from room temperature (300 K) to a lower temperature (5 K). then an external magnetic field of magnitude 1 kOe was applied on the nanocrystalline compound and had taken magnetization vs. temperature data during warming from 5 K to 300 K.

Field Cooled Cooling(FCC): In this method, an external magnetic field of magnitude 1 kOe is applied on the nanocrystalline compound at room temperature and magnetization vs temperature data was taken during cooling from room temperature (300 K) to lower temperature (5 K).

Field cooled Warming (FCW): in this method, magnetization vs temperature data was taken during warming from lower temperature (5 K) to room temperature (300 K) in the same previous external magnetic field of magnitude 1 kOe.

From the temperature dependent magnetization study near $T = 200$ K temperature, charged ordered anti-ferromagnetic transition is clearly visible. Such feature was also reported earlier [37,38]. With further lowering the temperature compound under goes to a ferromagnetic transition. Interestingly the ferromagnetic phase fraction at lower temperature is depend on the magnetic field. The increasing nature of the magnetization with increasing magnetic field implies the transformation of antiferromagnetic state to the ferromagnetic state in this compound. Additionally the irreversible nature of the magnetization during FCC and FCW protocols again confirms the phase co-existing nature of the compound at the low temperature. It is worth mentioning that, oxygen non-stoichiometry plays a vital role in several compounds and transition temperature is markedly modified [39,40]. However, in case of present compound the magnetic transition temperatures are nicely matches with the previous reported studies [37,38]. Hence, it may be assumed that oxygen non-stoichiometry is not significant in this case. In doped manganites, spin clusters are often observed even in the paramagnetic region (inset of Fig. 1). The effective magnetic moment of the compound have been calculated from the linear fitting of temperature dependent inverse susceptibility data (in paramagnetic region) and it is found to be ~ 7.009 . This value is larger than the theoretical value (5.40), indicating the existences of the spin clusters in the paramagnetic region.

Fig. 2 shows isothermal magnetization for PCSMO nanoparticles. In the absence of magnetic field the sample was first cooled down at several constant temperatures then applying magnetic field magnetization was recorded. A field induced meta-magnetic transition was observed for this nanocrystalline compound. The critical magnetic field (field required for metamagnetic transition) decreases with increasing temperature up to 60 K. However it further increases with the temperature above 60 K. The reversible nature of magnetic isotherms, after the field induced meta-magnetic transition, shows the soft ferromagnetic nature of this compound up to 60 K temperature. In the low temperature region, such reversible nature due to magnetic field cycling (except first quadrant of M-H) states that the fraction of antiferromagnetic part was converted into ferromagnetic and only ferromagnetism nature persists for subsequent field cycling. But above this temperature an interesting phenomena was observed where reversible nature of magnetic field cycling was suppressed. The irreversible nature enunciates that the transformed fraction of antiferromagnetism was partially recovered and it never lost its memory (shown in Fig. 2(f) and (g)). The nature of isothermal magnetization at different temperature indicates the evolution of ferromagnetic interaction at different temperature region. At very low temperature region ($T < 80$ K) the ferromagnetic interaction is more dominated. However, above 80 K temperature the antiferromagnetic interaction competes with ferromagnetic interaction which resulting a phase separated magnetic state in the studied compound.

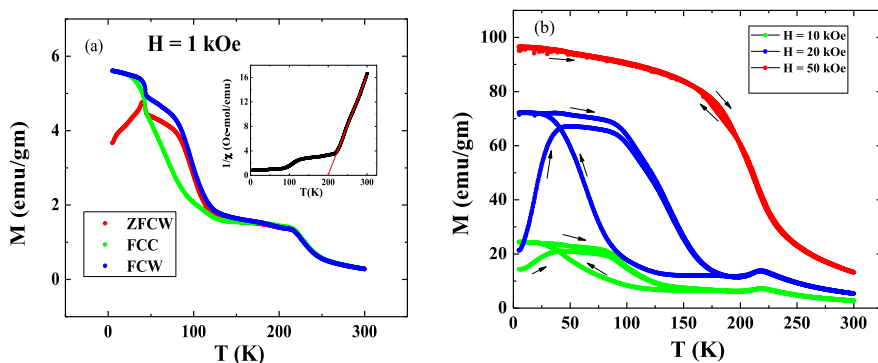


Fig. 1. (a) Shows the variation of magnetization with temperature at $H = 1$ kOe external magnetic field for nanocrystalline PCSMO compound at three different protocols ZFCW, FCC and FCW. Inset: temperature dependent inverse susceptibility (FCW data) (red line indicate the linear fitting). (b) Temperature dependent magnetization in the presence of different external magnetic field. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

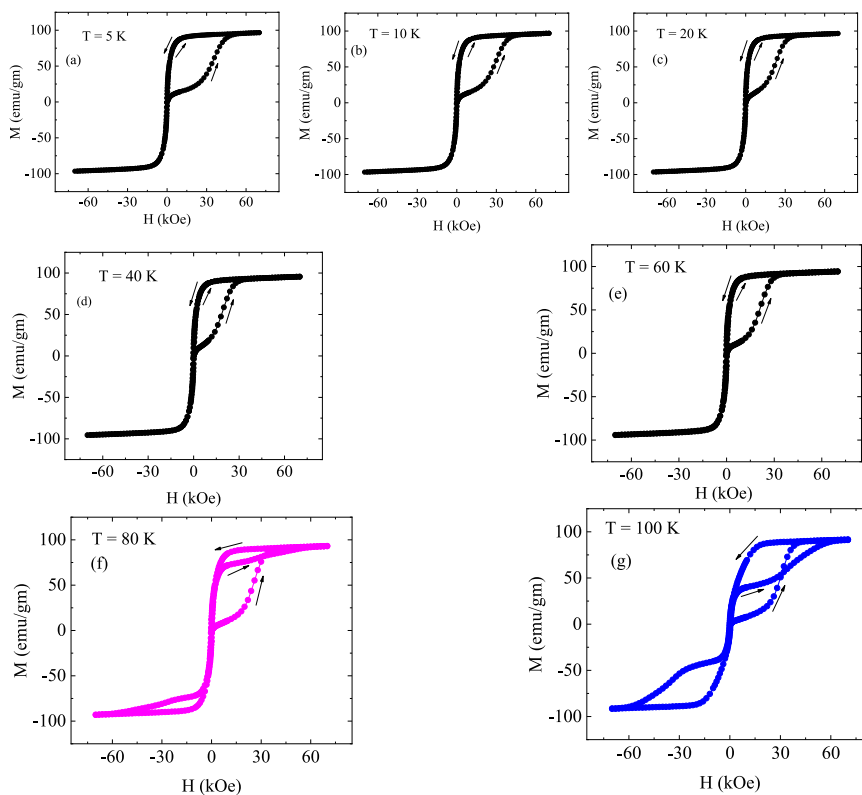


Fig. 2. (a)–(g) represents magnetization as a function of external magnetic field at several fixed temperature for nanocrystalline PCSMO compound after cooled down the compound to the fixed temperature in the absence of magnetic field.

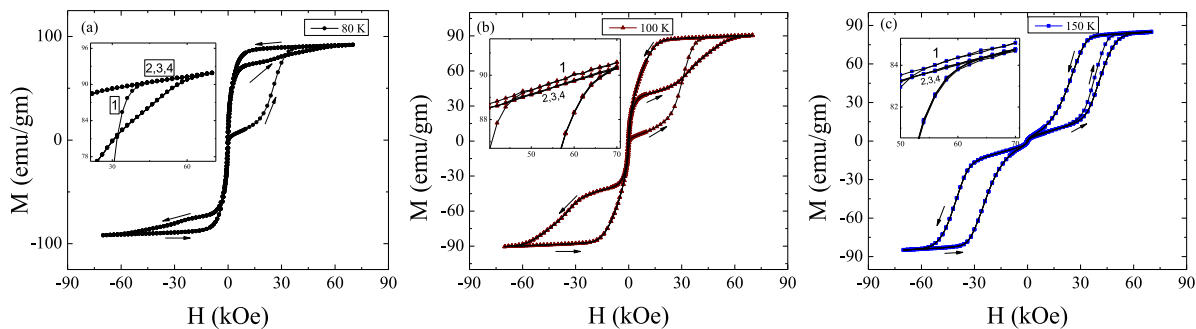


Fig. 3. Isothermal magnetization as a function of external magnetic field. Four M-H loops (assigned as '1','2','3' and '4') were measured subsequently at each temperature.

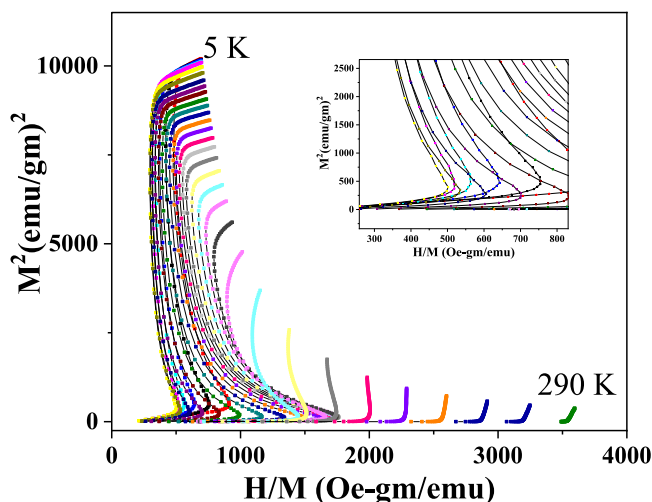


Fig. 4. Variation of the square of magnetization (M^2) with H/M for different temperatures. The negative slope indicates the first order nature of the transition. Inset indicate the enlarge view of the plot.

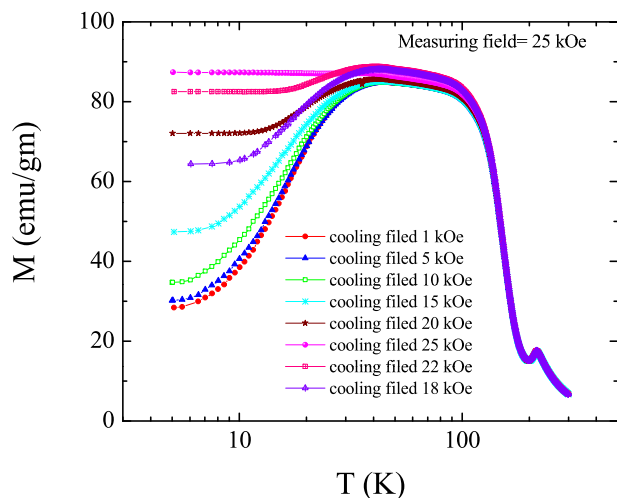


Fig. 5. Magnetization as a function of temperature for nanocrystalline PCSMO compound in the presence of 25 kOe magnetic field. Sample was cooled down in the presence of different cooling fields (as mention in figure). Magnetization data was recorded during warming cycles.

Generally, in such phase separated compound, magnetic training effect is almost a generic phenomenon [41]. It is well established that in a phase separated compound the training effect is dominated when the ferromagnetic and antiferromagnetic interaction are in comparable [41]. Considering those facts, we have studied the training effect of this phase separated compound at $T = 80$ K, 100 K, 150 K temperature (shown in Fig. 3). The well pronounced training effect in this compound again indicates the comparable nature of the different magnetic interactions especially at 100 K and 150 K.

According to Banerjee criterion, first order and second order magnetic phase transitions can be distinguished from Arrott plot (M^2 vs. H/M). The positive and negative slope (shown in inset of Fig. 4) of the Arrott plot corresponds to second order and first order magnetic phase transitions respectively [42,43]. The M^2 vs. H/M isotherms between 5 K to 290 K for nanocrystalline PCSMO are shown in Fig. 4. Here the presence of negative slope of the isotherms clearly indicates the first order phase transition for this nanocrystalline compound.

As discussed above, from the temperature and the field dependent magnetization studies of the nanocrystalline PCSMO compound it is

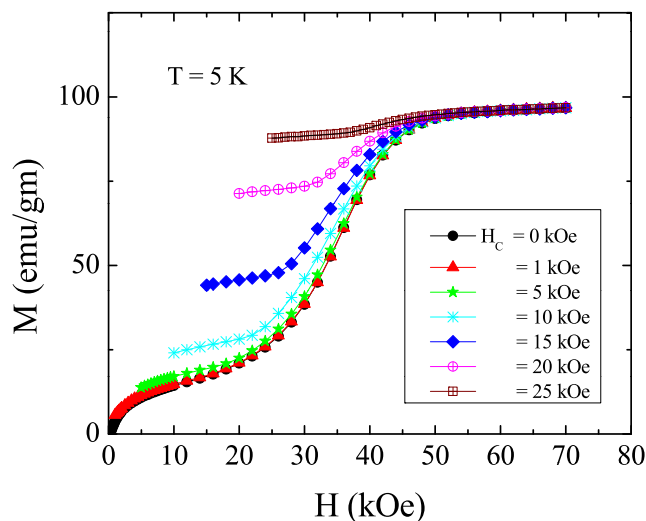


Fig. 6. The variation of magnetization with magnetic field for nanocrystalline PCSMO compound. Data was taken after cooled down the compound at 5 K temperature at different cooling magnetic field values (as mention in figure).

clear that a phase separated magnetic state is stabilized at the low temperature region. Additionally, at low temperature, sample transformed into a ferromagnetic state via a field induced metamagnetic transition. For such phase separated sample, kinetic arrest of different magnetic phases is a very common phenomena. Generally, when the sample is cooled down in the presence of lower magnetic field (smaller than the critical field) some portion of the high temperature magnetic phase (AFM, in this case) will get arrested. However, in the presence of higher cooling magnetic field value (greater than the critical field), phase separated nature is disappear at low temperature region. To explore the nature of the kinetically arrested magnetic phase at low temperature, we have cooled down the sample in the presence of different magnetic fields. Magnetization as a function of temperature data was recorded at $H = 25$ kOe magnetic field during warming which is shown in Fig. 5. It is reported earlier by Rawat et al. different nature of the devitrification of kinetically arrested magnetic phase may appear according to the different width of the phase transition bands [44]. From the above temperature dependent magnetization study, it is noticeable that the starting temperature of devitrification of kinetically arrested antiferromagnetically arrested phase is depends upon the cooling magnetic field. With increasing cooling field, the starting temperature of devitrification also increases.

To understand the increasing nature of temperature for the devitrification of antiferromagnetic phase with cooling magnetic field, we consider the different nature (magnetic interaction strength) of the antiferromagnetic domains as reported earlier by Pi et al. for similar phase separated compounds [41]. According to their studies, there are two types of antiferromagnetic domains namely “weak” and “strong”. Due to application of the external magnetic field (smaller than the charge ordered melting field) “weak” domains are easily flipped along field direction however the “strong” domains are remains in antiferromagnetic state [41]. In the present study, due to the increasing cooling field, the fraction of “weak” antiferromagnetic domains are decreases and hence the devitrification temperature of the remaining antiferromagnetic part increases.

The influences of such different kind of antiferromagnetic domains (having different interaction strength) is reflected in metamagnetic transition during isothermal magnetization measurements. Magnetic isotherms are plotted at temperature $T = 5$ K for the various external cooling magnetic fields (shown in Fig. 6). This graph indicates magnetization value increases with the increase of cooling fields which is

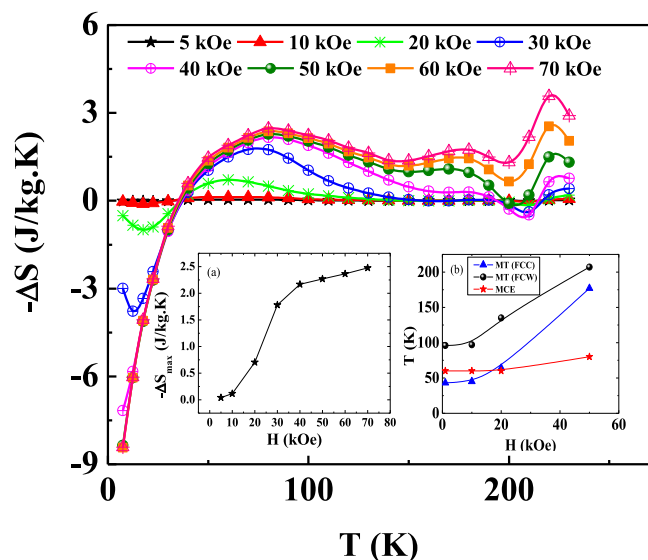


Fig. 7. Magnetocaloric entropy change vs. temperature at different external magnetic field for nanocrystalline PCSMO compound. Inset: (a) variation of the maximum normal magnetocaloric entropy change with field. (b) magnetic phase diagram constructed from FCC, FCW and MCE data.

because of increasing ferromagnetic portion with the increase of cooling magnetic field in the phase separated (coexisting of ferromagnetic and anti-ferromagnetic domains) PCSMO nanocrystalline compound. Another very interesting fact is observed which is critical magnetic field of metamagnetic transition (anti-ferromagnetic to ferromagnetic transition) increases with the increase of cooling magnetic fields. As discussed above, during the cooling of the sample under magnetic field, “weak” antiferromagnetic domains are converted into ferromagnetic part and it is increases with increasing cooling magnetic field. As a result, the requirement of the magnetic field for the metamagnetic transition is increases with the cooling magnetic field. Except this phenomena, interfacial strain between FM and AFM domain, predominant surface spin disorders and spin pinning play also important role [36, 45].

MCE is a powerful tool for identifying different magnetic ground states. It is defined as magnetic entropy change of a magnetic materials in the presence of external magnetic field. According to Maxwell’s thermodynamic relation, isothermal magnetic entropy change is quantified as

$$\Delta S = \int_0^H (\partial M / \partial T) dH \quad (1)$$

Especially for the phase separated compound, there are several reports where magnetic and magnetocaloric effect is extensively studied [46–53]. Phan et al. reported the systematic magnetocaloric measurements on $\text{La}_{5/8-y}\text{Pr}_y\text{Ca}_{3/8}\text{MnO}_3$ ($y = 0.275$) single crystals compound having complex multiple-phase transitions [46]. According to their study, the dynamic strain-liquid phase is strongly affected by an applied magnetic field. However, the frozen glassy phase field independent [46]. The modification of physical properties due to size reduction was reported by Bingham et al. [47]. The detailed magnetocaloric effect study indicate the smooth trend to stabilization of ferromagnetic state in nanoscale regime and significant phase competition [47]. The study regarding the modification of transition temperature and broadening with size reduction is reported earlier [48]. In contrast to the bulk counterpart the magnetic properties were markedly modified in the nanoscale regime and in thin film [48]. We have calculated magnetocaloric entropy change by using the one quadrant magnetic isotherms measured at different temperatures. The variation of magnetocaloric

entropy change with respect to temperature at several constant magnetic field for nanocrystalline PCSMO is shown in Fig. 7. Signature of charge ordered antiferromagnetic state is clearly visible near $T \sim 200$ K temperature. Moreover, a broad hump around $T \sim 100$ K, manifests the ferromagnetic ordering of the magnetic moments in nanocrystalline PCSMO compound. Below $T = 40$ K, magnetocaloric entropy change revert its sign for different constant magnetic field and inverse magnetocaloric effect (IMCE) is found at low temperature region. Regarding this context it should mention that in case of the polycrystalline sample such nature was absent [54]. In our present study, inverse magnetocaloric entropy change gives its maximum value 9 J/kg K at 50 kOe external magnetic field and with increasing field this value remains constant. Initially, the value of the IMCE was increased with increasing magnetic field up to 40 kOe . Above this field, the value of entropy change is saturated. To explore the origin of IMCE, it is worth mentioning that the magnetocaloric entropy change was calculated from the magnetic isotherms measured in ZFC protocols. As mentioned earlier, at low temperature region both ferromagnetic and antiferromagnetic components coexist (as the high temperature AFM part is arrested). Due to the application of magnetic field, antiferromagnetic counterpart enhanced the magnetic entropy when field is applied. This phenomena may be the possible origin for the IMCE of this compound. Such nature might be disappear when the magnetocaloric effect will be calculated from the FCC/FCW magnetization data. The variation of the normal magnetocaloric entropy change ($-\Delta S_{max}$) with external magnetic field is given in the inset (a) of Fig. 7. The rate of change of maximum entropy was initially small. However, after field induced metamagnetic transition it increases rapidly and almost saturates. A possible magnetic phase diagram constructed from FCC, FCW and MCE data is shown in inset (b) of Fig. 7.

4. Conclusions

In summary, the experimental findings indicate that the magnetic and magnetocaloric properties of phase separated compound are very much sensible on the temperature and magnetic field. Additionally, at low temperature, magnetic phase fractions are depend on the cooling magnetic field. At low temperature region, the devitrification field and temperature of kinetically arrested magnetic phase are depend on the ferromagnetic phase fraction present at the low temperature region. Moreover, at intermediate temperature range where the competing magnetic interactions are comparable, pronounced training effect was observed. At low temperature range, arrested magnetic phase influenced the magnetocaloric properties of this nanocrystalline compound.

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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