Materials Research Express

**PAPER**

Study of transport, magnetic and magnetocaloric properties in Sr\(^{2+}\) substituted praseodymium manganite

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**Abstract**

The magnetotransport, magnetic and magnetocaloric properties in the polycrystalline Pr\(_{1-x}\)Sr\(_x\)MnO\(_3\) (0.20 \(\leq x \leq 0.40\)) have been studied. The X-ray diffraction peaks for the composition \(x = 0.20, 0.33\) and \(0.40\) have been indexed with orthorhombic structure having Pnma space group, whereas \(x = 0.25\) composition has been indexed to R-3c space group having rhombohedral symmetry. The substitution of Sr\(^{2+}\) at Pr\(^{3+}\) site shows an increase in metal-insulator transition temperature (\(T_{MI}\)) and ferromagnetic ordering temperature (\(T_C\)). The composition, \(x = 0.25\) shows the higher isothermal magnetic entropy change, \(|\Delta S_m| = 2.84 \text{ J kg}^{-1} \text{K}^{-1}\) with RCP = 116.33 J kg\(^{-1}\) at \(\Delta H = 3\) Tesla. The high RCP value (\(=151.16\) J kg\(^{-1}\)) with \(|\Delta S_m| = 2.41\) J kg\(^{-1}\) \(\text{K}^{-1}\) at \(\Delta H = 3\) Tesla for the composition, \(x = 0.40\), implies that \(\delta T_{fhhn}\) plays a significant role.

1. Introduction

Perovskite manganites having common formula R\(_{1-x}\)A\(_x\)MnO\(_3\) where ‘R’ is a rare-earth cation (La\(^{3+}\), Pr\(^{3+}\), Y\(^{3+}\), Nd\(^{3+}\) etc.) and ‘A’ an alkali or alkaline earth cation (Ca\(^{2+}\), Sr\(^{2+}\), Ba\(^{2+}\), Na\(^{+}\), K\(^+\), etc.) show a lot of interesting properties, arising from the strong interplay between spin, charge, orbital and lattice degrees of freedom [1–9]. Colossal magnetoresistance (CMR) phenomena observed in these manganites has been mainly explained through double exchange mechanism [10], phase separation [11] and spin-polarized tunnelling effect [12]. Besides the CMR properties, in the vicinity of ferromagnetic (FM) to paramagnetic transition temperature the manganites also show large magnetocaloric effect (MCE). These correlations of magneto-transport and magnetic properties in manganites make it suitable for a wide range of applications; such as in magnetic sensors, bolometric devices and magnetic refrigeration, which add to their multifunctionality and are studied continuously in recent years [12–18]. However, the prime challenge in these materials remains due to their complex transport and magnetic behaviour as well as study of the fundamental physics involved [1–4]. In this paper, we have discussed in detail the magnetotransport and magnetocaloric properties of Pr\(_{1-x}\)Sr\(_x\)MnO\(_3\) (0.20 \(\leq x \leq 0.40\)) perovskite manganite.

2. Experimental

The polycrystalline Pr\(_{1-x}\)Sr\(_x\)MnO\(_3\) (0.20 \(\leq x \leq 0.40\)) have been synthesized using the nitrate route. Powder of Pr\(_6\)O\(_{11}\), SrCO\(_3\) and MnO\(_2\) were taken in stoichiometric ratio. The powders were ground and calcined several times between 800 °C and 1200 °C for 24 h with intermediate grindings. The powders thus, obtained were pressed into a pellet form at 10 MPa pressure and finally sintered at 1400 °C for 30 h with a cooling down to room temperature in air. The phase formation and structural characterization was carried out using powder x-ray diffraction (Bruker AXD-8 advance, CuK\(_{\alpha}\) radiation) at room temperature. DC electrical resistivity as a function of temperature and magnetic field down to 5 K was measured using the standard four-probe technique by means of resistivity/magnetoresistance set-up along with 8 T Oxford-Superconducting magnet at CSR.
Indore Centre. The magnetization (M) measurements as a function of temperature (T) and Magnetic field (H) were performed using Superconducting Vibrating Sample Magnetometer (VSM) (Versa Lab).

3. Result and discussion

3.1. Structural study

The x-ray diffraction (XRD) patterns of Pr$_{1-x}$Sr$_x$MnO$_3$ (0.20 $\leq x \leq$ 0.40) along with Rietveld analysis of all the compositions carried out using Fullprof program are shown in the figure 1. We do not observe any impurity in this XRD pattern except for a very small and broad peak around 2$\theta$ = 32°, which could be due to a very small quantity Mn$_2$O$_4$ & Mn$_3$O$_4$ (111) phase. The intensity of this impurity peak in x = 0.20 composition is less than 1% of that of the (020) peak of highest intensity and weakens in later compositions. Similar results of the presence of impurity phase are reported in JCPDS-862337 and 22. The XRD pattern of x = 0.20, 0.33 and 0.40 compositions have been indexed to Pnma space group having orthorhombic symmetry, whereas, of x = 0.25 composition has been indexed to R-3c space group having rhombohedral symmetry. Inset of figure 1 shows highest intense peak in the XRD pattern for each composition.

The compositions with x = 0.20, 0.33, 0.40 having single intense peak and x = 0.25 having bifurcation in intense peak suggests the crystallization of their structure in orthorhombic and rhombohedral symmetry respectively in accordance with the Rietveld analysis. The relevant structural parameters obtained are tabulated in table 1. It has been observed that the unit cell parameter and cell volume decreases with increase in Sr$^{2+}$ concentrations for x = 0.20, 0.33 and 0.40. It may be understood here that as Sr$^{2+}$ (1.31 Å) has higher ionic radii compared to Pr$^{3+}$ (1.18 Å), hence generally the substitution may lead to increase in lattice parameter. The increase in the unit cell parameters and cell volume for x = 0.25 is understood due to its rhombohedral structure. Markovich et al [9], for Pr$_{1-x}$Sr$_x$MnO$_3$ single crystal (where, x = 0.22, 0.24, 0.26) also observed decrease in lattice parameter with increasing Sr$^{2+}$ concentration and attributed it to a progressive decrease of Jahn–Teller distortions. Additionally, authors also suggested that for compositions with Sr$^{2+}$ (x > 0.3), a structural transition to R-3c space group having rhombohedral symmetry must take place citing reference Boujelben et al [19].

Further, Knizek et al [20] in the composition range 0 $< x <$ 0.5 suggested the crystallization of compositions in orthorhombic symmetry having Pbnm space group and reported that the lattice volume decreases with increasing Sr$^{2+}$ concentration. Chand et al [21], also reported the decrease in lattice parameter
with Sr$^{2+}$ concentration $x = 0.2, 0.3$ and $0.5$ in Pr$_{1-x}$Sr$_x$MnO$_3$. Nasari et al [22, 23] indexed the XRD pattern for Pr$_{0.6}$Sr$_{0.4}$MnO$_3$ having orthorhombic structure. Hence, the variation in lattice structures in our study are in accordance with the above discussed reported literature. The variation in structural parameters may be due to different method of preparation [7].

3.2. Magnetotransport study

The temperature dependent resistivity $\rho(T)$ plot in the absence (0 T) and presence of magnetic field 5 T and 8 T are shown in figures 2(a)–(d). All the compositions show distinctive metal–insulator transition (TMI), which increases for compositions with Sr$^{2+}$ concentration $x = 0.20–0.33$ and decreases for $x = 0.40$. The decrease in resistivity and increase in TMI indicates that with Sr$^{2+}$ substitution, the ferromagnetic metallic state becomes more dominant. Additionally, the decrease in resistivity along with the increase in TMI with the application of magnetic field also suggests the dominance of the magnetic field in enhancing the FM nature of the compositions. For lower composition $x = 0.20$, a large change in resistivity is observed around TMI, the metal to insulator transition becomes broader and shifted to a higher temperature side with the application of magnetic field as shown in figure 2(e). The temperature dependence of magnetoresistance, defined as (MR)(%) = [(\rho(H) – \rho(0))/\rho(0)] x 100] is also shown along with the resistivity curve in figures 1(a)–(d). Negative MR has been observed in the entire temperature range of measurement for all the compositions, however for compositions, $x = 0.20$ and 0.25, MR is nearly 80% around respective TMI makes its suitable for the device application.

3.3. Magnetic properties study

The temperature dependent magnetization (T), of the compositions at 100 Oe down to 50 K is shown in the inset of figure 3. All the compositions show paramagnetic (PM) to ferromagnetic (FM) transition. Transition temperature $T_C$ is determined from the minimum of dM/dT versus T curve. It can be observed that with the increase in Sr$^{2+}$ concentration $T_C$ increases significantly, from 150 K to 286 K, which clearly indicates the strengthening of ferromagnetism in accordance with the transport properties. As we increase Sr$^{2+}$ (x), a lower saturation magnetization ($M_S$) is expected, as Sr is practically non-magnetic element. However, there is a crossover for $M_S$ values between $x = 0.20$ and 0.25, breaks this systematic. As mentioned earlier in XRD section, $x = 0.20$ and 0.25 have orthorhombic and rhombohedral structural respectively. This structural transition may have lead to the increase in $M_S$ value of $x = 0.25$ composition [19]. In order to understand the magnetic behavior of the compositions in the PM region above $T_C$, we studied the inverse of DC magnetic susceptibility (M/H) as a function of temperature (T) shown in figure 3. In high temperature region, much above $T_C$, the DC magnetization data in paramagnetic region follows the Curie–Weiss (CW), expressed as $\chi = C/(T - \theta_{CW})$ as shown in figure 3.

Here $C$ is a constant and can be defined as $C = b\chi_0^2 N$, where $\mu_{eff}$ is the effective magnetic moment, ‘b’ is universal constant, ‘N’ is concentration of magnetic moments and $\theta_{CW}$ is the CW temperature. $\mu_{eff}$ calculated from the linear fitting to the $\chi^{'-1}(T)$ curves are tabulated in table 1 along with that expected from the theoretical model. The theoretical effective moment for each case can be written as $g\sqrt{(S + 1)} \mu_B$ for (Mn$^{3+}$ and Mn$^{4+}$) and $g\sqrt{(I + 1)} \mu_B$ for Pr$^{3+}$ where $g$ is the gyromagnetic factor, $S$ is the spin angular momentum, $J(L \pm S)$ is total angular momentum, $L$ is orbital angular momentum and $\mu_B$ is the Bohr magneton. The theoretical values

### Table 1. Lattice parameters obtained from Rietveld fittings to XRD pattern, Best fit values obtained from M(T) in PM region using CW law. MCE $\Delta \delta$ and RCP value.

| Pr$_{1-x}$Sr$_x$MnO$_3$ | x = 0.20 | x = 0.25 | x = 0.33 | x = 0.40 |
|-------------------------|----------|----------|----------|----------|
| a(Å)                    | 5.459(9) | 5.479(7) | 5.448(0) | 5.442(3) |
| b(Å)                    | 7.719(5) | 5.479(7) | 7.701(4) | 7.678(9) |
| c(Å)                    | 5.493(3) | 13.396(9)| 5.482(1) | 5.484(1) |
| V(Å$^3$)                | 231.533(1)| 348.346(6)| 230.013(4)| 229.186(0)|
| $T_{MI}$(K)             | 100      | 200      | 253      | 230      |
| (+)MR% at $T_{MI}$(8 T) | 86       | 88       | 66       | 45       |
| $T_C$(K)                | 150      | 186      | 261      | 286      |
| $\theta_{CW}$           | 0.0200   | 0.0199   | 0.0180   | 0.0171   |
| $\mu_{eff}$             | 160      | 190      | 236      | 268      |
| $\mu^{'eff}$            | 6.10     | 6.04     | 5.71     | 5.51     |
| $\mu^{''eff}$           | 4.90     | 4.64     | 4.25     | 3.95     |
| $-\Delta S_{m}^{\text{MCE}}$ (J/kgK)(3 T) | ... | 2.84 | 2.36 | 2.41 |
| $\delta T_{\text{ thermo}}$(K) | ... | 40.96 | 50.60 | 62.72 |
| RCP (J/kg)(3 T)          | ... | 116.33 | 119.42 | 151.16 |
of $\mu^{3+}(\text{Mn}^{3+})$, $\mu^{4+}(\text{Mn}^{4+})$ and $\mu^{3+}(\text{Pr}^{3+})$ are $\sim 4.88\mu_B$, $\sim 3.87\mu_B$ and $3.58\mu_B$ respectively. The calculated $\mu_{\text{eff}}^{\text{cal}}$/formula unit for the composition e.g. for $x = 0.33$ can be written as,

$$\mu_{\text{eff}}^{\text{cal}} = \sqrt{[(0.67)\mu_{\text{eff}}^{\text{Pr}^{3+}} + 0.33\mu_{\text{eff}}^{\text{Mn}^{4+}}]}. $$

The $C$, $\theta_{\text{CW}}$, $\mu_{\text{eff}}^{\text{cal}}$ and $\mu_{\text{eff}}^{\text{exp}}$ values so obtained from the best fit to the experimental data are listed in Table 1. The obtained value of $\mu_{\text{eff}}^{\text{exp}}$ are found to be greater than that of $\mu_{\text{eff}}^{\text{cal}}$. The difference between the experimental effective paramagnetic moment and the calculated can be explained by the existence of FM clusters within the PM phase, evidenced by the downturn in $\chi^{-1}(T)$ curve near $T_C$ with the decreasing temperature [24, 25]. This downturn in $\chi^{-1}(T)$ suggests the deviation from CW law with the decreasing temperature and is an indication of non-analytical behaviour of magnetization arising from magnetic inhomogeneities [26–29].
3.4. Magnetocaloric effect (MCE)

In order to study the change in magnetic entropy \( \Delta S_M \) with respect to the temperature \( T \), the magnetic field \( H \) dependent magnetizations \( M \) measured up to 3 T (within the instrumental limit) at the interval of 2 K and are shown in figures 4(a)–(d). As shown in the inset of figures 4(a)–(d), the observed positive slope for all studied temperatures specifies that the magnetic transition between the FM and PM phase is of the second order. The pragmatic second order transition without any thermal and magnetic hysteresis suggests that the compositions are suitable for refrigeration application.

The magnetic isotherms measurements have been carried out with the difference of 2 K \( (\Delta T) \), we have adopted a suitable fitting approach to obtain apparent maximum entropy change \( -\Delta S_M^{\text{max}} \) and full width at half maximum \( \delta T_{\text{fwhm}} \) and to further calculate relative cooling power (RCP). We used the Gaussian Asym equation, \( y = y_0 + A \times \exp(-0.5((x - xc)/w)^2) \), where \( y_0 \) is the offset, \( A \) denotes the amplitude, \( w \) is a
parameter specifying the width called Gaussian width of the and $x_c$ represents the abscissa of the peak. Fitting $-\Delta S_M \text{ versus } T$ plot with the said equation, the obtained best fit values $(y_0 + A)$ represents $-\Delta S_M^{\text{max}}$ and $\delta T_{\text{fwhm}} = 2w \times \sqrt{\ln 4}$. The $-\Delta S_M^{\text{max}}$, $\delta T_{\text{fwhm}}$ and RCP values are tabulated in table 2. It can be observed that $-\Delta S_M^{\text{max}}$ is maximum for $x = 0.25$ composition having value $2.84 \text{ J kg}^{-1} \text{ K}$ at 3 T. The relative cooling power (RCP), defined as $\text{RCP} = -\Delta S_M^{\text{max}} \times \delta T_{\text{fwhm}}$, which provides a measure of the amount of heat transfer between hot and cold sinks during one ideal refrigeration cycle. The obtained RCP values of all the compositions are presented in figure 5(d). The higher RCP value $151.16 \text{ J kg}^{-1}$ for $x = 0.40$ composition suggests that $\delta T_{\text{fwhm}}$ plays a significant role.

4. Conclusions

Perovskite manganite $\text{Pr}_{1-x}\text{Sr}_x\text{MnO}_3$ with $x = 0.20, 0.25, 0.33$ and 0.40 are grown in single phase. The compositions $x = 0.20, 0.33$ and 0.40 crystallize in orthorhombic structure with Pnma space group, whereas $x = 0.25$ crystallize in Rhombohedral structure with $R 3c$ space group. Both metal to insulator transition and Curie temperature increases with increase in Sr$^{2+}$ concentration. Magnetic measurements revealed that all the compositions undergo a second order magnetic transition with the PM–FM transition near room temperature. Through thermodynamic Maxwell relations, the isothermal entropy change ($-\Delta S_M$) has been determined. The entropy behaviour also suggests typical second order transition in all the studied compositions. The
compositions with Sr$^2^+$ concentration $x = 0.25$ and $x = 0.40$ shows a good magnetocaloric effect, indicating its potential application for refrigerant applications.

Acknowledgments

This work is supported by UGC DAE CSR, Indore (CSR-IC/CRS-89/2014-2018) and SERB-DST, New Delhi (EMR/2016/005424) grants. AKS (Project fellow II) gratefully acknowledges UGC DAE CSR and GC (IRF) SERB-DST for fellowship. Authors thank Dr Mukul Gupta and Layanta Behera for XRD measurements. We are grateful to Dr Rajeev Rawat and Sachin Kumar for resistivity/ MR measurements at UGC-DAE Consortium for Scientific Research, Indore. Authors are thankful to Nanomaterials Core Characterization Facility (NCU) Virginia Commonwealth University, Richmond, USA for VSM facility.

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