Magnetic-field-induced structural changes in the electron doped manganites $\text{Ca}_x\text{Sm}_{1-x}\text{MnO}_3$ ($x = 0.8, 0.85$)

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Abstract

We studied the correlation between magnetic, electrical, structural, and magnetostriction properties of the electron doped manganites $\text{Ca}_x\text{Sm}_{1-x}\text{MnO}_3$ ($x = 0.85, 0.8$). The paramagnetic to antiferromagnetic transition in both the compounds while cooling is accompanied by an abrupt increase of the spontaneous volume thermal expansion ($\Delta V/V = 0.07 \%$ for $x = 0.85$ and $0.25 \%$ for $x = 0.2$). The $x = 0.15$ exhibits multiple phase separation at 5 K: G-type, and C-type antiferromagnetic phases in orthorhombic ($Pnma$) and monoclinic ($P2_1/m$) structures respectively. Magnetic study on $x = 0.85$ also suggest ferromagnetic regions possibly in $Pnma$ structure coexist with the antiferromagnetic phases. The magnetization ($M = 1.2 \mu_B$) of $x = 0.85$ does not reach the value expected for the complete alignment of Mn spins even at 12 T and at 12 K. Metamagnetic transitions (C-type to Ferromagnetic)
in both compounds are accompanied by contraction of volumes under high magnetic fields. We suggest that a high magnetic field induces $P2_1/m$ (high volume) to $Pnma$ (low volume) structural transition. This is also supported by the neutron diffraction study.
I. INTRODUCTION

In spite of extensive investigations on the hole doped, Mn$^{3+}$: $t_{2g}^3e_g^1$ rich manganites, there are scarcely few reports on the electron doped, Mn$^{4+}$: $t_{2g}^3e_g^0$ rich compounds. Recently, electron-doped CaMnO$_3$ compounds regained experimental and theoretical interests motivated by the following facts: 1. The compounds $x > 0.5$ in the series RE$_{1-x}$Ca$_x$MnO$_3$ (RE = La, Nd, Pr, etc.,) are antiferromagnetic insulators and the antiferromagnetic state is insensitive to the external magnetic field of $\mu_0 H = 5-7$ T normally employed in many laboratories. However, magnetoresistance of about 82% was first reported by Mahendiran et al. in La$_{0.1}$Ca$_{0.9}$MnO$_3$ (Mn$^{4+}$ = 85%) around the Neel temperature ($T_N$). 2. Investigations by Chiba et al. on Bi$_{1-x}$Ca$_x$MnO$_3$ and by Maignan et al. on RE$_{1-x}$Ca$_x$MnO$_3$ (RE = Nd, Sm, Gd, etc..) revealed that magnetoresistance exists over a wide range ($0.96 \leq x \leq 0.8$) and the maximum magnetoresistance was again obtained for Mn$^{4+}$ $\approx$ 85%. In contrast to the hole-doped (rich in Mn$^{3+}$: $t_{2g}^3e_g^1$) compounds whose resistivity ($\rho$) becomes insensitive to a fixed external magnetic field as size of the rare earth ion decreases, a large magnetoresistance independent of the rare earth ion is observed in the electron doped manganites. 3. Magnetization of the electron doped compounds for $0.85 < x < 1$ surprisingly increases below $T_N$ whose origin is attributed to the formation of ferromagnetic clusters and their growth with $x$ in the G-type antiferromagnetic background although canted antiferromagnetic structure for certain $x$ value cannot be completely ruled out. Interestingly $\rho$ also exhibits a maximum around $T_N$ in compounds ($x = 0.97-0.88$) which have higher magnetization. 4. Electron-doped manganites serve as a laboratory to study the dynamics of excess electrons ($e_g$ electrons of Mn$^{3+}$: $t_{2g}^3e_g^1$) in an antiferromagnetic matrix with compositionally tunable Jahn-Teller interactions and model to verify electronic phase separation proposed theoretically. When intersite Jahn-Teller interactions becomes dominant the G-type simple two sublattice antiferromagnetic order in Sm$_{1-x}$Ca$_x$MnO$_3$ ($x = 1$) changes to the C-type antiferromagnet (ferromagnetic linear chains with inter chain antiferromagnetic coupling) for $x = 0.87-0.8$ without charge ordering and with charge ordering for $0.8 < x$.
The composition \( x = 0.5 \) is generally a CE-type antiferromagnet (zig-zag ferromagnetic chains with inter chain antiferromagnetic coupling) with charge and cooperative Jahn-Teller ordering occurring at \( T > T_N \). Most of the existing studies have attributed the large magnetoresistance in manganites to the magnetic field induced ferromagnetic alignment of the \( t_{2g}^3 \) core spins either from the antiferromagnetic or the paramagnetic state although few investigations\(^3\)\(^4\) suggest that magnetically tunable lattice distortion could play an important role. However, the sign of lattice deformations either in zero or non zero magnetic field is not predicted theoretically so far and the existing experimental results are mostly on the hole doped \((x < 0.5)\) or \( x = 0.5 \) compounds\(^3\). The purpose of this paper is to investigate the nature of the lattice distortion in absence and in presence of a external magnetic field in the electron doped \( \text{Sm}_{1-x}\text{Ca}_x\text{MnO}_3 \) series \((x = 0.85, 0.8)\) which undergo simultaneous magnetic (paramagnetic to a C-type antiferromagnetic) and structural transitions (orthorhombic to monoclinic) with lowering temperature\(^3\).

II. EXPERIMENT

We measured magnetization of polycrystalline samples of \( \text{Ca}_x\text{Sm}_{1-x}\text{MnO}_3 \) \((x = 0.85, 0.8)\) using a commercial vibrating sample magnetometer up to 12 T and zero field four probe resistivity was also measured in the temperature range \( 5K < T < 300 K \). The structural characterization of our samples was already reported\(^3\)\(^4\). Linear thermal expansion \((\Delta L/L)\) in zero magnetic field was measured for \( 20 K < T < 300 K \) using the strain gauge technique. The volume thermal expansion was calculated using \( \Delta V/V = 3\Delta L/L \). Magnetostriction by the strain gauge method was measured in a pulsed magnetic field up to 14 T with the magnetic field parallel and perpendicular to the measuring direction. The volume magnetostriction of randomly oriented polycrystallites was calculated through \( \Delta V/V = \lambda_{par} + 2\lambda_{per} \) where \( \lambda_{par} \) and \( \lambda_{per} \) are parallel and perpendicular magnetostric tions. The anisotropic magnetostriction was found to be negligible and not presented in this paper. Neutron powder diffraction spectra at \( T = 100 K \) in 0 T and 6 T on \( \text{Ca}_{0.85}\text{Sm}_{0.15}\text{MnO}_3 \) with \( \text{Sm}^{154} \) isotope were car-
ried out in the high resolution spectrometer (D2B instrument) at Institute Laue-Langvin (ILL), Grenoble using the wavelength of 1.594 Angstrom.

III. RESULTS

Fig. 1(a) show temperature dependence of the inverse magnetic susceptibility (H/M.) for $x = 0.85$ and 0.8 while cooling from 300 K. The arrows mark the onset of C-type antiferromagnetic ordering ($T_{NC} = 112$ K for $x = 0.85$ and 150 K for $x = 0.8$). The paramagnetic to a C-type antiferromagnetic transitions are accompanied by orthorhombic ($Pnma$ space group) to monoclinic ($P2_1/m$) structural transitions. The magnetic transition in these compounds are hysteretic as shown already. Earlier neutron diffraction study on $x = 0.85$ showed that in addition to the C-type ordering in the monoclinic phase, another antiferromagnetic ordering of G-type also sets in orthorhombic phase around $T_G = 118$ K and its phase fraction increases from 5% at 100 K to 9 % at 10 K. Due to its smaller phase fraction and closeness of $T_G$ to $T_C$, its signature is not very clear in the magnetic measurements.

The Curie-Weiss fit (dark lines) for $T > T_{NC}$ gives Curie-Weiss temperatures $\theta_p = 119$ K ($x = 0.85$) and 135 K ($x = 0.8$). The experimentally determined effective moments $P_{eff} = 4.386 \mu_B$ ($x = 0.85$), $4.238 \mu_B$ ($x = 0.8$) are slightly higher than the theoretical values $P_{eff} = 4.162 \mu_B$ for $x = 0.85$ and $4.215 \mu_B$ for $x = 0.8$. The $x = 0.8$ composition is expected to undergo a paramagnetic to a C-type antiferromagnetic transition below $T_{NC} = 150$ K without coexistence of phases at low temperature based on the measurements on similar compounds.

Fig. 1(b) shows the zero field resistivity $\rho(T)$ of $x = 0.85$ and $x =0.8$ while cooling from 300 K to 5 K and warming back to 300 K. We clearly see hysteresis in resistivity around $T_{NC}$. The crystallographic transition accompanying magnetic transition are marked. At 300 K, $\rho(x = 0.8) > \rho(x = 0.85)$. In contrast to the insulating behavior of $\rho(T)$ in the paramagnetic phase of hole-doped compounds, $\rho(T)$ of these two electron-doped compounds decreases as the temperature decreases from 300 K and shows a broad minimum at $T_{min} = 155$ K (
x = 0.85), 210 K (x = 0.8) as shown in the inset and then jumps at \( T_{NC} \) (\(< T_{min}\)). In between \( T_N \) and 5 K, \( \rho(T) \) increases by four orders of magnitude in \( x = 0.85 \) and eight orders of magnitude in \( x = 0.8 \). The resistivity minimum above \( T_{NC} \) can be recaptured in non adiabatic polaronic picture when the thermal and polaron activation energies are equal. We were able to fit the data using the relation \( \rho(T) = \rho_0 T^{1.5} \exp(E_a/k_B T) \) with \( E_a = 0.017 \) eV and \( E_a = 0.023 \) eV. With increasing content of the the rare earth ion, \( T_{min} \) shifts to much higher temperatures.

Fig. 1(c) shows the volume thermal expansion (\( \Delta V/V \)) with respect to \( V \) value at 300 K. As T decreases from 300 K, the lattice shrinks in the paramagnetic region and then shows a rapid increase at \( T_{NC} \). In the entire temperature range (300 K- 20 K), the overall change in \( \Delta V/V \) is larger for \( x = 0.85 \) than \( x = 0.8 \). However, around \( T_{NC} \) the volume of \( x = 0.8 \) increases by 0.35 % which is five times larger than in \( x = 0.85 \) (\( \Delta V/V \approx 0.07 \) % around \( T_{NC} \)). These differences are intimately tied to the degree of localization of charges (for example, resistivity of \( x = 0.8 \) at 5 K is 5 orders of magnitude higher than \( x = 0.8 \)), the strength of antiferromagnetic exchange interactions and structural distortions. Here, it should be mentioned that the magnitude of \( \Delta V/V \) in \( x = 0.85 \) is very sensitive to the stoichiometry. All previous studies on manganites undergoing transition either from a paramagnetic insulator to a ferromagnetic metal or from a ferromagnetic metal to a charge-ordered antiferromagnetic insulator showed that volume contracts at the transition temperature in contrast to the volume expansion behavior seen here.

Figs. 2(a)-(d) illustrate the influence of external magnetic fields on the spin and lattice degrees of freedom. The field dependence of the magnetization and the magnetostriction data were taken after zero field cooling to a predetermined temperature. At 12 K (\( T << T_{NC} \)) \( M(H) \) of \( x = 0.85 \) [Fig. 2(a)] shows a rapid increase at low fields (\( \mu_0 H < 0.5 \) T), gradually increases up to 2.5 T and then shows a rapid, but a smooth increase until the maximum field of \( \mu_0 H = 12 \) T. The trend of the high field data suggest that \( M(H) \) can increase even beyond \( \mu_0 H = 12 \) T. Upon decreasing H, \( M(H) \) exhibits a huge hysteresis. The low field
behavior is caused by the presence of ferromagnetic regions in antiferromagnetic phase as suggested previously. The rapid increase of M(H) above 2.5 T is due to the field induced transition from the C-type AF to ferromagnetic state. It should be noted that the maximum magnetic moment at 12 K even at a field as high as 12 T is 1.45 $\mu_B$ which is much less than 3.1 $\mu_B$ expected for a fully ferromagnetic alignment of Mn spins. This means only a part of the sample transforms into ferromagnetic state and the rest remaining in antiferromagnetic state. We will touch upon this aspect in the later part of the paper. As T increases the width of hysteresis decreases and finally at 175 K and 225 K, M(H) is linear in H as expected for a paramagnet. Fig. 2(b) shows M(H) of x = 0.8 very close but below $T_N$. First we note that the ferromagnetic like low field behavior seen in x = 0.85 is absent in x = 0.8. Second, the metamagnetic transition is rather sharp and the width of the hysteresis loop is nearly same (= 1.5 T) for T = 140 K and 145 K. The magnetic moment even at a field as high as 12 T is $\approx 1.2 \mu_B$ (see 145 K data) which is far below the value of 3.2 $\mu_B$ expected for a fully ferromagnetic alignment of Mn$^{3+}$ and Mn$^{4+}$ spins. The critical field $H_C$ for the metamagnetic transition, as determined from the inflection point of the rapidly increasing part of M(H) in the field-up mode increases rapidly from 8 T to 11 T as the temperature decreases by 5 K from 145 K.

The field-induced metamagnetic transition is also accompanied by a dramatic change in volumes. The volume magnetostriction ($\omega$) isotherms in Fig. 2(b) and 2(d), for x = 0.8 and x = 0.85 respectively, show that the lattice volume contracts upon transition from the antiferromagnetic to the metamagnetic state. The reverse transition is hysteretic. The maximum volume change at 13.7 T is -0.135 % at 25 K for x = 0.85 and decreases to -0.085 % at 100 K. As like the magnetization, the hysteresis in $\omega$ also decreases with increasing temperature. At 120 K, $\omega$ is positive and is 0.0185 %. The volume change is negligible for x = 0.8 at 12 K. An appreciable magnetostriction in x = 0.8 appears only in between 120 K and 150 K. The maximum value of magnetostriction $(\Delta V/V)_{max}$ increases with H, reaches a maximum at 135 K and then decreases again. Fig. 3 is complementary to Figs.
2(c) and (d) and it compares the values of the volume magnetostriction at the maximum field $\mu_0 H = 13.7 \, \text{T}$ for $x = 0.85$ and 0.8 as a function of temperature. The $(\Delta V/V)_{\text{max}}$ is zero far away from $T_{NC}$ and at very close but above $T_{NC}$ it has smaller positive value and, a rapid increase occurs just below $T_{NC}$. The peak in $(\Delta V/V)_{\text{max}}$ for $x = 0.8$ is due to the fact that the maximum available field is insufficient to induce a complete antiferromagnetic to ferromagnetic transition.

IV. DISCUSSION

Now we turn attention to the origin of the volume contraction under a magnetic field. The paramagnetic to C-type AF transition in the electron doped compounds under investigation is accompanied by orthorhombic ($Pnma$) - monoclinic ($P2_1/m$) structural transition. The C-type antiferromagnetism is coupled to polarization of the $d_{z^2-r^2}$ orbitals of Mn$^{3+}$ sites along the c-axis. The $x = 0.85$ composition exhibits multiple phase separation. It was found that about 9 % of the G-AF phase in $Pnma$ structure also coexists with the C-phase ($P2_1/m$ structure) at 10 K in zero field in $x = 0.85$. In addition, a third phase which is ferromagnetic also appears to be present in $x = 0.85$. But the volume fraction of the ferromagnetic phase is extremely sensitive to sample preparation. The ferromagnetic phase is in $Pnma$ structure, if one extrapolates the trend seen in $x = 0.9$ compound. When $H$ is increased from 0 T, the magnetic contribution due to domain wall displacement in ferromagnetic phase dominates at low fields. As $H$ increases further above 5 T at 12 K, a metamagnetic transition takes place and a portion of the C-AF phase is converted into a ferromagnetic phase. Since, the magnetic moment does not saturate even at the highest field of 12 T, ferromagnetic phase and antiferromagnetic phase coexists in between 4 T and 12 T at 12 K. Because of the strong coupling between the C-type AF and $d_{z^2-r^2}$ orbitals, the destruction of the C-type AF is accompanied by disordering of $d_{z^2-r^2}$ which drives $P2_1/m$ to $Pnma$ structural transition. Since the $P2_1/m$ phase higher volume than the $Pnma$ phase, the field induced monoclinic to orthorhombic transition leads to a volume
contraction as seen in the magnetostriction data (Fig. 2(c)). If this is to be true, we can expect to see an increase in the \textit{Pnma} phase fraction above at high fields. This is what indeed found in the neutron diffraction study\cite{19}. While small angle neutron scattering study and elaborate powder neutron diffraction study in Ca_{0.85}Sm_{0.15}MnO_3 will be published by some of us elsewhere\cite{19}, we show the result most relevant to this work. Fig. 4 shows the schematic diagram of the nuclear phases of x = 0.85 at 100 K in absence of a magnetic field and in presence of H = 6 T. The \textit{P2_1/m} phase (C-AF) which is 91 % at 0 T reduces to 44 % at 6 T and correspondingly the \textit{Pnma} phase (G-AF) increases from 9 % at 0 T to 56 % at 6 T. A large ferromagnetic component of = 1.7 \mu_B is also found in the \textit{Pnma} phase\cite{19}. We believe that similar field induced structural transition also takes place in x = 0.8 compound.

V. SUMMARY

In summary, we have shown that the paramagnetic to the C-type antiferromagnetic transition in the electron doped manganites Ca_xSm_{1-x}MnO_3 (x = 0.80, 0.85) is accompanied by an abrupt volume expansion (\Delta V/V = 0.07 % in x = 0.85, 0.35 % in x = 0.8) and orthorhombic (Pnma) to monoclinic (P21/m) structural transitions. The x = 0.85 compound is magnetically and structurally phase separated with two types of antiferromagnets ,G-(Pnma) and C- (P21/m) and a minority ferromagnetic phase (Pnma) coexisting together. Magnetization study shows that metamagnetic transition (C-AF to ferromagnetic) field increases from 5 T in x = 0.85 to more than 12 T in x = 0.8 at 12 K. Isothermal magnetostriction measurements shows high volume to low volume transition under a magnetic field which has been suggested to structural transition (\textit{P2_1/m} to \textit{Pnma}) under a field. The driving factor for this structural transition is the strong coupling between the C-type antiferromagnetic order and the d_{3z^2-r^2} orbital order. The field induced structural transition in x = 0.85 compound is supported by neutron diffraction study.
VI. ACKNOWLEDGMENTS

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**Figure captions**

**Fig. 1** Temperature dependence of the inverse susceptibility $(H/M)$ measured under $\mu_0H = 0.01$ T in $\text{Ca}_x\text{Sm}_{1-x}\text{MnO}_3$ ($x = 0.8, 0.85$). G- and C-type antiferromagnetic phases coexist down to the lowest temperature in $x = 0.15$. PM -paramagnetic phase. (b). Temperature dependence of the zero field resistivity of $x = 0.85$ and $x = 0.8$ while cooling and warming. The inset shows the resistivity minimum in the paramagnetic state (c). Temperature dependence of the spontaneous volume thermal expansion of $x = 0.8$ and $x = 0.85$. The structural transitions are also marked.

**Fig. 2** Magnetization isotherms of (a) $x = 0.85$ and (b) $x = 0.8$. Volume magnetostriction isotherms of (c) $x = 0.15$ and (d) $x = 0.8$.

**Fig. 3** Temperature dependence of the volume magnetostriction $(\Delta V/V)$ of $x = 0.85$ and 0.8 at the the maximum field $\mu_0H = 13.7$ T.

**Fig. 4** A schematic diagram of the volume phase fractions of nuclear phases in 0 T and 6 T at 100 K in $\text{Ca}_{0.85}\text{Sm}_{0.15}\text{MnO}_3$. 
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M. R. Ibarra and J. M. de Teresa in ref. 2; page 83

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FIG. 1
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FIG. 2
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FIG. 3
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FIG. 4
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Volume Phase Fraction (%)

0  20  40  60  80  100

Nuclear Phases

Pnma (0T) (G-AF)

Pnma (6T) (G-AF)

P2_1/m (0T)-(C-AF)

P2_1/m (6T)-(C-AF)