Pressure-induced magnetization reversal in the lightly electron-doped manganite compound (Ca,Sr)Mn$_{0.95}$Sb$_{0.05}$O$_3$

Takahiro Fujiwara, Michiaki Matsukawa, Satoru Kobayashi, Shigeki Nimori, and Ramanathan Suryanarayanan

1) Department of Materials Science and Engineering, Iwate University, Morioka 020-8551, Japan
2) National Institute for Materials Science, Tsukuba 305-0047, Japan
3) Laboratoire de Physico-Chimie de L’état Solide, CNRS, UMR8182, Université Paris-Sud, 91405 Orsay, France

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We report here on the dc magnetization as a function of temperature and pressure up to 1 GPa in the lightly electron-doped manganite compound (Ca$_{1-x}$Sr$_x$)Mn$_{0.95}$Sb$_{0.05}$O$_3$ with its fixed carrier content. When the magnetic field is small (<500 Oe), diamagnetic magnetization is observed for $x \leq 15\%$, which changes to positive values for $x > 15\%$. However, on an application of pressure on the samples with $x = 16\%$ and 17%, magnetization reverses sign and diamagnetism reappears. We present a magnetic phase diagram as a function of temperature and pressure. To understand better the dynamical properties of this system, we have carried out the ac magnetic susceptibility as a function of frequency in the presence of an external dc field of 0 and 100 Oe. Our results indicate the existence of magnetic frustration between diamagnetic clusters and canted antiferromagnetic matrix.

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I. INTRODUCTION

Manganese oxides with pervoskite structure have been extensively investigated since the discovery of a colossal magnetoresistance (CMR) effect. The spontaneous insulator to metal transition and its associated CMR effect are well explained on the basis of the double-exchange (DE) model between Mn$^{3+}$ and Mn$^{4+}$ ions. Furthermore, the phase-separation model, where the ferromagnetic (FM) metallic and antiferromagnetic (AFM) insulating clusters of competing electronic phases coexist, strongly supports experimental studies of manganites.

CaMnO$_3$, the end member of the Ca$_{1-x}$La$_x$MnO$_3$ system, undergoes a G-type antiferromagnetic transition around $T_N \sim 120$ K, accompanied by a weak ferromagnetic component where each spin of the Mn ions is antiparallel to the nearest neighbors of Mn. In recent years, the electron-doped manganite system ($x < 0.5$) has attracted much attention because of the possibility of observing a negative magnetoresistance effect similar to that observed in its counterparts, in the so-called hole-doped manganites, for $x > 0.5$.

In addition to a large amount of researches on the A-site substituted electron-doped manganites, it has been shown that substitution at the Mn site of CaMnO$_3$ with higher valence ions than 4+ such as CaMn$_{1-2x}$M$_{2x}$M$_3$O$_{16}$+O$_3$, with M = Nb, Ta, V, Ru, and CaMn$_{1-2x}$M$_{2x}$M$_{2x}$O$_{16}$+O$_3$, with M = Mo, W creates Mn$^{3+}$ ions, i.e., electrons leading to the CMR effect. For CaMn$_{0.96}$Mo$_{0.04}$O$_3$ system, the low-temperature magnetic ground state is better described by a canted AFM magnetic structure than by a phase-separated state.

Thus, examining the physical properties of the Mn-site substituted compositions would be interesting for our understanding of the electronic phase diagram of electron-doped manganites.

The negative magnetization phenomena in manganites were originally reported in compounds with two sublattices of Mn ions and rare-earth ions (Nd, Gd, Dy), such as NdMnO$_3$10,11, (La,Gd)MnO$_3$12, (Nd,Ca)MnO$_3$13, (Gd,Ca)MnO$_3$14, and (Dy,Ca)MnO$_3$. Some of these studies were discussed on the basis of a ferrimagnetic scenario leading to a negative magnetization at temperatures below a compensation temperature, where Mn and some rare-earths sublattices are antiferromagnetically coupled. Moreover, a phase separation model between ferromagnetic clusters and the canted AFM matrix was proposed, in order to account for a possible origin of the negative magnetization in manganites.$^{10,13}$

Earlier, a negative magnetization in CaMnO$_3$ with B-site substitution had been reported. Recently, we demonstrated the effect of the hydrostatic pressure on magnetic, transport, and thermal transport properties in the lightly electron-doped manganite CaMn$_{1-x}$Sb$_x$O$_3$. Anomalous magnetization reversals were clearly observed for $x = 0.05$ and 0.08 in the field-cooled magnetization while the application of external pressure induced a suppression of the negative magnetization.

In this paper, we report the influence of the chemical pressure on the temperature dependence of the dc magnetization in CaMn$_{0.95}$Sb$_{0.05}$O$_3$ by using the substitution of Sr with its larger ion radius at a Ca site, to examine the physical role of local lattice distortion in the magnetization reversal phenomenon. In the next section, the experimental outline is described. In Sec. III, we display the temperature dependence of the electrical resistivity and the dc magnetic susceptibility for (Ca$_{1-x}$Sr$_x$)Mn$_{0.95}$Sb$_{0.05}$O$_3$ ($x = 0.0, 0.10, 0.15, 0.16, 0.17,$ and 0.2). The effect of the hydrostatic pressure on the magnetic phase diagram is presented, to examine anomalous diamagnetic properties. Furthermore, to understand better the dynamical properties of the present system, we
measured the temperature dependence of the ac magnetic susceptibility of \((\text{Ca}_{1-x}\text{Sr}_x)\text{Mn}_{0.95}\text{Sb}_{0.05}\text{O}_3\) under an application of the dc magnetic field. In the final section, the summary is given.

II. EXPERIMENT

Polycrystalline samples of \((\text{Ca}_{1-x}\text{Sr}_x)\text{Mn}_{0.95}\text{Sb}_{0.05}\text{O}_3\) \((x=0.0, 0.05, 0.10, 0.15, 0.17, \text{and } 0.2)\) were prepared by using a solid-state reaction method. The stoichiometric mixtures of high purity \text{CaCO}_3, \text{Mn}_3\text{O}_4, \text{Sb}_2\text{O}_5\text{ and SrCO}_3\text{ powders were calcined in air at } 1000^\circ\text{C for } 24\text{ h}}. The products were then ground and pressed into cylindrical pellets. The pellets were finally sintered at 1400 ~ 1450°C for 12 h. X-ray diffraction data revealed that all samples had almost a single phase with an orthorhombic structure \((Pnma)\text{)}\). The lattice parameters and unit cell volume increase with increasing the Sb content because the ion radius of Sb\(^{5+}\) (0.61Å) is greater than the value of Mn\(^{4+}\) (0.54Å). In addition, the Mn\(^{4+}\) ions are replaced by Mn\(^{3+}\) ions (0.645 Å) with one extra electron, which contributes to the increased cell volume.\(^{22}\) The X-ray photoemission spectroscopy (XPS) on the \text{CaMn}_{0.8}\text{Sb}_{0.2}\text{O}_3 (Sb20%) sample was performed at room temperature by using the Perkin-Elmer PHI-5600 system. The XPS data strongly support that the valence of the Sb ion is 5+ because the XPS profile of the Sb20% sample behaves like that of Sb2O5 in comparison to the data for Sb2O3 (Fig. II(b)).

The dc magnetization measurement was carried out at 1400 ~ 1450°C for 12 h. X-ray diffraction data revealed that all samples had almost a single phase with an orthorhombic structure \((Pnma)\text{)}\). The lattice parameters and unit cell volume increase with increasing the Sb content because the ion radius of Sb\(^{5+}\) (0.61Å) is greater than the value of Mn\(^{4+}\) (0.54Å). In addition, the Mn\(^{4+}\) ions are replaced by Mn\(^{3+}\) ions (0.645Å) with one extra electron, which contributes to the increased cell volume.\(^{22}\) The X-ray photoemission spectroscopy (XPS) on the \text{CaMn}_{0.8}\text{Sb}_{0.2}\text{O}_3 (Sb20%) sample was performed at room temperature by using the Perkin-Elmer PHI-5600 system. The XPS data strongly support that the valence of the Sb ion is 5+ because the XPS profile of the Sb20% sample behaves like that of Sb2O5 in comparison to the data for Sb2O3 (Fig. II(b)).

The dc magnetization measurement was carried out
using the commercial superconducting quantum interference device (SQUID) magnetometers at Iwate University and at the National Institute for Materials Science (NIMS). The ac magnetic susceptibility measurement was measured as a function of temperature for frequencies ranging from 1 Hz to 1000 Hz under the ac magnetic field of 5 Oe at NIMS. In particular, to remove the influence of a remanent magnetic field, the SQUID magnetometer with the option of a magnet reset mode was used in zero-field-cooled measurements.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Effect of Sr-substitution on electrical transport

First of all, the temperature dependence of the electrical resistivity $\rho$ for the Sr-substituted $\text{(Ca}_{1-x}\text{Sr}_x)\text{Mn}_{0.95}\text{Sb}_{0.05}\text{O}_3$ compound ($x=0.0$, 0.10, 0.15, 0.17, and 0.2) is presented in Fig.2. The inset shows the resistivity as a function of Sr content from 0% up to 20% at low and high temperatures. The magnitude of $\rho$ at room temperature is not largely affected by Sr-substitution, indicating the carrier content almost fixed (chemical pressure effect). At low temperatures, the resistivity follows a monotonous increase with increasing...
Sr content, which originates from the randomness effect due to Sr-substitution. As previously reported, it is clear evidence for the electron doping that the behavior of resistivity at high temperatures for the lightly Sb-substituted parent samples shows a metallic conduction accompanied by the negative Seebeck coefficient.

**B. Effect of chemical and physical pressures on dc magnetization**

Now, let us show in Fig. 4 the temperature dependence of the field-cooled magnetic susceptibility for the Sr-substituted (Ca$_{1-x}$Sr$_x$)Mn$_{0.95}$Sb$_{0.05}$O$_3$ recorded under H=100, 200, and 500 Oe. For the parent sample, we observed a typical negative magnetization curve at lower temperatures below 46 K under 50, 100, and 200 Oe with lowering $T$. As pointed out in a previous work, the substitution of Sb$^{5+}$ with its larger ionic radius at the Mn site introduces a local lattice distortion, resulting in a variation of the local easy axis of magnetization through the Jahn-Teller active Mn$^{3+}$O$_6$ octahedra. This finding is closely related to the stabilization of the diamagnetic spin configuration. It is well known that a larger Sr$^{2+}$ substitution at the Ca site in the Ca$_{1-x}$Sr$_x$MnO$_3$ system increases both the lattice volume and the bond angle of Mn-O-Mn, and a rise in the Néel temperature $T_N$. For the parent CaMnO$_3$ with $T_N=123$ K, the bond angle of Mn-O-Mn is estimated to be 157-158.8° whereas the Sr20% substitution at the Ca site enhances its bond angle up to 161-162°, resulting in $T_N=141$ K. In the present sam-
Fig. 5: (Color online) Temperature dependence of field-cooled magnetic susceptibility for the Sr-substituted (Ca$_{1-x}$Sr$_x$)Mn$_{0.95}$Sb$_{0.05}$O$_3$ recorded at $H = 100$, 200, and 500 Oe under a hydrostatic pressure of 1.0 GPa. (a) $x = 0.10$, (b) $x = 0.15$, (c) $x = 0.16$, and (d) $x = 0.17$. The arrow of (c) points to the pressure-induced magnetization reversal from the ambient curve. Magnetic phase diagram of the Sr-substituted (Ca$_{1-x}$Sr$_x$)Mn$_{0.95}$Sb$_{0.05}$O$_3$ collected at 100 Oe under the ambient and hydrostatic pressures. (e) 0 GPa, and (f) 0.8 GPa and 1.0 GPa. PM, c-AFM, and DM denote paramagnetic, canted antiferromagnetic, and diamagnetic phases, respectively. For comparison, we present in (e) the diamagnetic phase diagram from the data taken under $H = 200$ Oe and 500 Oe.

Spinels, the Sr doping up to 20% causes a slight increase of $T_N$ from 104 K at $x = 0$ to 107 K at $x = 0.2$. In addition to its local lattice distortion due to Sb, we need to take into account for the lightly electron doping effect on the Ca$_{1-x}$Sr$_x$MnO$_3$ system. The temperature dependence of the magnetization of the present system is quite different from that of the Sb-free (Ca,Sr) based manganite compound. Upon increasing Sr content up to 15%, we continue to obtain the diamagnetic behavior at lower temperatures although it is accompanied by a gradual drop of its negative magnetization. At further Sr contents beyond 15%, such a diamagnetic signal vanishes and instead of it a weak ferromagnetic behavior appears. The Sr-substitution at the Ca site suppresses a canted spin state in a direction opposite to that of the applied field. The effect of chemical pressure due to the higher Sr-substitution relaxes the local lattice deformation around the Sb ion existing at B-site, preventing a diamagnetic response observed for the lower samples below Sr15%. We note that the low temperature magnetization reversal is associated with the existence of a prominent peak located near 85 K, independent of Sr content up to 15%.

Next, to further examine the effect of the lattice on the behavior of negative magnetization, we tried to measure the temperature dependence of the magnetic susceptibility for the Sr-substituted (Ca$_{1-x}$Sr$_x$)Mn$_{0.95}$Sb$_{0.05}$O$_3$ under a hydrostatic pressure. Figure 5 displays the field-cooled magnetization curves collected at several magnetic
Fig. 6: (Color online) The real and imaginary parts of ac magnetic susceptibility of (Ca$_{1-x}$Sr$_x$)Mn$_{0.95}$Sb$_{0.05}$O$_3$ as a function of temperature collected at zero dc magnetic field for frequencies ranging from 1 Hz to 1000 Hz. The amplitude of the ac magnetic field $H_{ac}$ was set to be 5 Oe. The arrows point to the direction of increasing frequencies. For $x=0.0$, (a) $\chi'$ and (b) $\chi''$. For $x=0.05$, (c) $\chi'$ and (d) $\chi''$. For $x=0.10$, (e) $\chi'$ and (f) $\chi''$. The ac magnetization data are recorded under ZFC condition. The arrows point to the direction of increasing frequencies.

fields under 0.8 GPa. The $M/H$ data for both the parent and Sr5% doped samples shows no diamagnetic behavior. We define as $T_{DM}$ a characteristic temperature where a magnetization curve starts to reach a diamagnetic signal. At Sr=10%, the magnitude of $T_{DM}$ at 100 Oe is substantially reduced from 60 K at 0 GPa down to 20 K at 0.8 GPa. For the higher Sr15% substituted sample, the diamagnetic behavior is not largely affected. However, on an application of pressure on the two samples with Sr16% and Sr17% just above the critical content of Sr=15%, the magnetization reverses its sign and diamagnetism reappears as shown in Figs. 4(d) and 4(e). It is true that the application of the external pressure suppresses the diamagnetic behavior of the lower Sr doped samples, but in the case of Sr contents near the critical value of 15% it assists the appearance of the diamagnetic state. Furthermore, the $M/H$ curve of the samples with Sr15%, Sr16%, and Sr17% under the application of external pressure of 1.0 GPa is presented in Fig. 5. For the Sr16% sample, the diamagnetic behavior survives even in 1.0 GPa, while the magnetization of the Sr17% sample exhibits no such a negative tendency. Our findings are summarized in the phase diagram given in Fig. 5. The diamagnetic area existing in the phase diagram is reduced at the presence of the external pressure except for in the neighborhood of the critical content around Sr15%. For the Sr17% sample, the chemical pressure effect also completely destroys a diamagnetic behavior, as commonly observed in the higher Sr-substitution at the Ca site beyond 15%. On the other hand, it is true that an application of the external pressure of 0.8 GPa recovers a magnetization reversal, as well as the Sr16%
Fig. 7: (Color online) The real and imaginary parts of ac magnetic susceptibility of (Ca_{1−x}Sr_x)Mn_{0.95}Sb_{0.05}O_3 as a function of temperature collected at zero dc magnetic field for frequencies ranging from 1 Hz to 1000 Hz. The amplitude of the ac magnetic field $H_{ac}$ was set to be 5 Oe. The arrows point to the direction of increasing frequencies. For $x = 0.15$, (a) $\chi'$ and (b) $\chi''$. For $x = 0.17$, (c) $\chi'$ and (d) $\chi''$. For $x = 0.20$, (e) $\chi'$ and (f) $\chi''$. The ac magnetization data are recorded under ZFC condition. The arrows point to the direction of increasing frequencies.

sample does so. However, increasing further pressure up to 1 GPa again suppresses its diamagnetic signal. These complicated properties are quite intriguing phenomena, which are in their origin close to the interplay between the local lattice distortion and various magnetic interactions. To our knowledge, the magnetization reversal induced by external pressure has not been reported so far and is an additional novel phenomenon of the physics of manganites.

A theoretical work on lightly electron-doped manganite CaMnO$_3$ predicted that spin canting in the G-type antiferromagnetic structure is realized by electron doping through the double exchange mechanism. The weak FM component observed in the Ce-substituted CaMnO$_3$ is well explained by the spin-canting G-type AFM state with the double-exchange hopping of electrons. For the lightly electron-doped samples with the higher substitution of Sr16%−Sr20% , we believe that a weak ferromagnetic trend in their $MT$ curves is compatible with the present canted AFM scenario. Moreover, as previously referred, the larger Sb substitution at the Mn site introduces the local lattice distortion of Mn$^{3+}$O$_6$ associated with $e_g$-electron doping and then changes the orbital state of $e_g$-electron through the local John-Teller effect. As a result, it gives rise to a considerable variation in local spin configuration existing at the nearest neighbor of its Sb ion, leading to the formation of diamagnetic clusters within its matrix. The canted AFM matrix stabilized by light electron doping contributes to a weak ferromagnetic component. On the other hand, the diamagnetic clusters are simultaneously formed through the local lattice deformation due to partial the substitution of Sb with its
larger ionic radius. One supposes that both the former and latter contributions coexist in the phase diagram of the present system, which is dependent on the Sr content and/or the applied pressure.

C. Frequency and dc magnetic field dependences of ac magnetic susceptibility

Now, we tried to carry out the ac magnetic susceptibility measurements for \((\text{Ca}_{1-x}\text{Sr}_x)\text{Mn}_{0.95}\text{Sb}_{0.05}\text{O}_3\) \((x=0.0, 0.05, 0.1, 0.15, 0.17, \text{ and } 0.2)\), in order to clarify the dynamical effect on the diamagnetic state. The temperature dependence of the real and imaginary parts, \(\chi'\) and \(\chi''\), are registered at zero \(dc\) magnetic field with increasing frequency \(f\) ranging from 1 Hz to 1000 Hz, as shown in Figs. 8 and 9. For the parent Sr0% sample, a sharp peak in both \(\chi'\) and \(\chi''\) is observed around 100 K, which is good agreement with the \(dc\) magnetic measurement. Upon increasing \(f\), a second peak of \(\chi'\) and \(\chi''\) located at 90 K slightly shifts towards lower temperatures. For lower Sr doping up to 10%, the amplitude of \(\chi''\) shows its strong decay with increasing frequency. When the Sr content exceeds 15%, the frequency dependence of ac magnetic susceptibility almost disappears. The frequency dependence of the ac magnetization indicates the signature of a spin-glass like character, but a substantial decrease of the peak in \(\chi''\) with increasing \(f\) is in contrast to the behavior of conventional spin-glass systems\(^{23}\), as previously pointed out in the phase-separated Pr\(_{0.7}\)Ca\(_{0.3}\)MnO\(_3\).\(^{24}\) The clear second peak in \(\chi''\) observed at the parent sample is rapidly suppressed.
due to the lower Sr-substitution. From the present data, we point out that the higher Sr-substitution above 15% gives no frequency dependences of the ac magnetic susceptibility, indicating the stability of the magnetic state against small disturbances. In general, the imaginary part $\chi''$ of the ac magnetic susceptibility represents magnetic energy dissipation. The magnitude of $\chi''$ around 100 K is decreased from $\sim 10^{-4}$ emu/g at Sr0% through $1.5 \times 10^{-5}$ emu/g at Sr10% down to $10^{-6}$ emu/g at Sr17%, which also supports frustrated magnetic properties of the lower Sr-substituted samples.

To further determine the differences in the spin dynamics between zero-field-cooled and field-cooled conditions, we measure the dc magnetic field dependence of the ac magnetic susceptibility for $(Ca_{1-x}Sr_x)Mn_{0.95}Sb_{0.05}O_3$ ($x=0.0, 0.05, 0.1, 0.15, 0.17,$ and $0.2$). Figures. 9 and 10 show the temperature variation of the real and imaginary components, $\chi'$ and $\chi''$ with a superimposed dc field ($H_{dc}=100$ Oe). For the parent and lower Sr doped samples, the temperature dependence of $\chi'$ and $\chi''$ strongly depends on the cooling processes. In particular, the magnitude of the imaginary part of the parent sample near 80 K varies from $\sim 10^{-4}$ emu/g at zero dc field through $\sim 2 \times 10^{-5}$ emu/g at ZFC scan down to $\sim 2 \times 10^{-6}$ emu/g at FC scan. These tendencies in the ac magnetic susceptibility are common properties for the lower Sr doped samples. On the other hand, the data for the Sr17% and Sr20% samples exhibit no superimposed dc field dependence although there is a large hysteresis between the ZFC and FC dc curves of both the samples as depicted in the inset of Figs. 8(e) and (f). From the ac magnetization data, the temperature variation of the imaginary com-

![Fig. 9: (Color online) The real and imaginary parts of ac magnetic susceptibility of Sr substituted $(Ca_{1-x}Sr_x)Mn_{0.95}Sb_{0.05}O_3$ measured at 10 Hz under a superimposed dc field ($H_{dc}=100$ Oe). For $x=0.15$, (a) $\chi'$ and (b) $\chi''$. For $x=0.17$, (c) $\chi'$ and (d) $\chi''$. For $x=0.20$, (e) $\chi'$ and (f) $\chi''$. For comparison, the ac data in the absence of dc magnetic field are given (•10Hz). The ac magnetization data are recorded as a function of temperature under ZFC and FC conditions.](image)
ponent $\chi''$ depends on cooled processes of the samples under dc field, which is closely related to the appearance of the field-cooled magnetization reversal in the present system. In particular, we expect that there exist various magnetic frustrations between diamagnetic clusters and canted AFM matrix, contributing to the dynamical behavior of the ac magnetic susceptibility. When the external pressure or the larger ion substitution relaxes the local lattice distortion associated with antiparallel spin component in a direction opposite to that of the applied field, the magnetic frustration related to the diamagnetic clusters is then removed from the canted AFM matrix, resulting in the dc field independence of $\chi''$. However, the origin of the magnetization reversal under a given pressure is not understood well at the present moment.

IV. SUMMARY

We have reported on the temperature dependence of the dc magnetization under several pressures in the lightly electron-doped manganese compound $(\text{Ca}_{1-x}\text{Sr}_x)\text{MnO}_3$ with its fixed carrier content. For $x \leq 15\%$, diamagnetic behaviors were detected in the weakly field-cooled magnetization ($H < 5000\text{Oe}$). For the Sr content $> 15\%$, the behavior of the negative magnetization vanishes. On an application of the external pressures on the Sr16% and 17% substituted samples, magnetization reverses its sign, and diamagnetism reappears. We present a magnetic phase diagram including the temperature dependent magnetization reversal both under ambient and hydrostatic pressures. The ac magnetic susceptibility measurement was performed to examine the dynamical properties of the corresponding system. For the lower Sr-substituted samples, both the real and imaginary parts of ac magnetic susceptibility exhibit their strong frequency dependence and are sensitive to field-cooled processes. Our findings strongly indicate the existence of magnetic frustration between diamagnetic clusters and canted antiferromagnetic matrix. To our knowledge, the magnetization reversal induced by external pressure is an additional novel phenomenon of the physics of manganites.

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* Electronic address: matsukawa@iwate-u.ac.jp

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