Structural domain and spin-ordering–induced glassy magnetic phase in single-layered manganite $\text{Pr}_{0.22}\text{Sr}_{1.78}\text{MnO}_4$

S. Chattopadhyay, S. Giri and S. Majumdar(a)

Department of Solid State Physics, Indian Association for the Cultivation of Science, Jadavpur, Kolkata 700032, India

received 21 December 2011; accepted in final form 16 March 2012; published online 24 April 2012

PACS 75.25.Bk – Orbital, charge, and other orders, including coupling of these orders
PACS 75.50.Lk – Spin glasses and other random magnets
PACS 75.47.Lx – Magnetic oxides

Abstract – The single-layered manganite $\text{Pr}_{0.22}\text{Sr}_{1.78}\text{MnO}_4$ undergoes structural transition from high temperature tetragonal phase to low temperature orthorhombic phase below room temperature. The orthorhombic phase was reported to have two structural variants with slightly different lattice parameters and Mn-3$d$ levels show orbital ordering within both the variants, albeit having mutually perpendicular ordering axis. In addition to orbital ordering, the orthorhombic variants also order antiferromagnetically with different Neél temperatures. Our magnetic investigation on the polycrystalline sample of $\text{Pr}_{0.22}\text{Sr}_{1.78}\text{MnO}_4$ shows large thermal hysteresis indicating the first-order nature of the tetragonal to orthorhombic transition. We observe magnetic memory, large relaxation, frequency-dependent ac susceptibility and aging effects at low temperature, which indicate spin-glass–like magnetic ground state in the sample. The glassy magnetic state presumably arises from the interfacial frustration of orthorhombic domains with orbital and spin orderings playing crucial role toward the competing magnetic interactions.

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One of the intriguing aspects of $\text{AMnO}_3$-type perovskite manganites ($\text{A}=\text{alkaline earth or rare-earth element}$) is the frequent observation of a glassy magnetic state at low temperature, which is believed to arise from the competing interactions between finite-size ferromagnetic (FM) and antiferromagnetic (AFM) clusters [1]. $\text{AMnO}_3$ compounds actually belong to a wider class of materials known as Ruddlesden-Popper (RP) phase with general formula $\text{A}_n\text{Mn}_n\text{O}_{3n+1}$, and they may be thought of as the $n=\infty$ member in the RP phase. There also exist several other manganites in the RP phase with fascinating crystal structures. The lowest possible value of $n$ is 1 and this corresponds to $\text{A}_2\text{MnO}_4$-type single-layered manganites [2–8]. These compounds crystallize in a tetragonal $\text{K}_2\text{NiF}_4$-type structure where $\text{MnO}_2$ layers remain separated from one another through $\text{A}^{2+}$ ions and construct a quasi–two-dimensional (2D) lattice structure.

Similar to $\text{AMnO}_3$ compounds, doping with rare-earth ions at the $\text{A}$ site of $\text{A}_2\text{MnO}_4$ produces mixed valency where both $\text{Mn}^{3+}$ and $\text{Mn}^{4+}$ ions can exist. However, unlike the perovskite system, single-layered manganites (SLMs) do not show FM-metallic ground state and colossal magnetoresistance is absent. Rather, in many situations, they undergo charge ordering/orbital ordering (CO/OO) which ultimately favors an AFM ground state [7,9,10]. Due to the absence of a finite FM region or cluster, SLMs are unlikely to have a cluster glass-like state. However, it should be kept in mind that FM bonds between $\text{Mn}$ atoms are actually present in SLMs, which along with superexchange-mediated AFM bonds can give rise to long-range C- or CE-type AFM structures. Many SLMs show a complex interplay between orbital, magnetic, structural and charge degrees of freedom, and it is pertinent to investigate the magnetic ground state of such materials, particularly in the presence of coexisting FM and AFM bondings.

In this work, we chose a suitable SLM with nominal composition $\text{Pr}_{0.22}\text{Sr}_{1.78}\text{MnO}_4$ (PSMO) for such magnetic investigations. The series of compounds, $\text{Pr}_{1-x}\text{Sr}_{1+x}\text{MnO}_4$ ($0.75 \leq x \leq 0.9$) are reported to undergo a structural phase transition from tetragonal (TG) to orthorhombic (OR) phase with lowering of $T$. Interestingly, there are two orthorhombic variants OR1 and OR2 with unequal $b/a$ ratios ($a$ and $b$ are the lattice
parameters). Both the variants undergo orbital ordering, although with mutually orthogonal orientations. In the OR1 phase, OO takes place in the $d(3z^2-r^2)$ orbital of $\text{Mn}^{3+}$ ions, whereas, it is the $d(3y^2-r^2)$ orbital that stabilizes in the OR2 phase. The structural phase transitions, i.e. TG to OR1 and TG to OR2 also occur at two different temperatures. Transmission electron microscopy reveals the presence of alternating arrays of orbitally ordered OR1 and OR2 domains [11] at low temperature. Similar orthorhombic domain structure is also observed in few other compositions among SLs with general formula $\text{R}_{1-x}\text{Sr}_{1+x}\text{MnO}_4$ ($\text{R} = \text{La}, \text{Nd}$) [12–15].

With further lowering in temperature ($T$), OR1 phase in PSMO undergoes a C-AFM transition at $T_{N1}$ where the orbitally ordered OR2 phase still remains magnetically disordered. Finally at $T = T_N2$ ($< T_{N1}$), C-type AFM (C-AFM) transition of the OR2 phase occurs. The ground state of PSMO is constructed by arrays of two coexisting C-AFM phases associated with OR1 and OR2, respectively.

Our investigation based on the dc and ac susceptibilities, relaxation, aging, and memory measurements reveals the presence of an unusual glassy magnetic phase in the ground state of PSMO even in the absence of any obvious microscopic FM clusters.

A polycrystalline sample of PSMO was prepared following the conventional solid state reaction route as described elsewhere [16]. The sample was characterized using X-ray powder diffraction (Cu–K$\alpha$), and it was found to be single phase with tetragonal crystal structure (space group: $I4/mmm$; lattice parameters: $a = 3.78\,\text{Å}$, and $c = 12.21\,\text{Å}$). Magnetization ($M$) and ac susceptibility measurements were performed in a Quantum Design SQUID magnetometer.

Figure 1(a) depicts the $T$ variation of the dc magnetic susceptibility ($\chi = M/H$, $H$ being the applied magnetic field) between 3 K and 320 K measured in zero-field–cooled heating (ZFCH), field cooling (FC), and field-cooled heating (FCH) protocols. The inset shows the ZFCH curve for an applied field of 100 Oe. Panel (b) shows isothermal magnetization as a function of applied field ($H$) at three different temperatures. The inset shows the enlarged view of the curves in the low-field region.

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\caption{(Colour on-line) Panel (a) shows the dc magnetic susceptibility measured with an applied field of 500 Oe in the zero-field–cooled heating (ZFCH), field-cooled heating (FCH) and field-cooling (FC) protocols. The inset shows the ZFCH curve for an applied field of 100 Oe. Panel (b) shows isothermal magnetization as a function of applied field ($H$) at three different temperatures. The inset shows the enlarged view of the curves in the low-field region.}
\end{figure}

In order to probe the spin dynamics of the low temperature orbitally ordered phase, we measured $M$ as a function of time ($t$) at different constant $T$ well below the thermal hysteresis region. In fig. 2(a), $M$ has been depicted in a normalized form $M(t)/M(0)$, where $M(0)$ is the value
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Fig. 2: (Colour on-line) Panel (a) shows the isothermal time dependence of magnetization [$M(t)$] measured at three different temperatures. Here the data have been normalized by the initial value of the magnetization [$M(0)$]. The solid lines are fit to the data with stretched exponential form of relaxation. Panel (b) shows the magnetic viscosity ($S = \frac{1}{H} \partial M}{\partial (ln(t))}$) at 6 K in the ZFC state as a function of time. Here the three different curves correspond to measurement at 100 Oe after initial wait at zero field of $t_w = 1500$, 3500 and 5500 s, respectively.

of $M$ at the start of the relaxation measurement. All the data carry clear signature of magnetic relaxation and it is as large as 1.6% at 18 K for $t = 3600$ s. Such large relaxation of $M$ indicates that the magnetic state at low temperature is associated with some sort of metastability. Interestingly, the magnitude of relaxation decreases with decreasing temperature. This is presumably an indication of thermal effect, where thermal fluctuations associated with the frozen spins diminishes with decreasing $T$. In addition, the system consists of structural domains (OR1 and OR2), and the number of such domains (and hence the number of interfaces) is found to increase with increasing $T$ [12]. Therefore, the enhanced relaxation with $T$ can also be related to the increasing number of such domains. The relaxation data can be best fitted with a modified stretched exponential function given by [19–22],

$$M(t) = M_i - M_s \exp[-(t/\tau)^{\beta}].$$

This particular relation is widely used to describe the relaxation behaviour of glassy magnetic systems. Here, $M_i$ corresponds to the initial magnetization, $M_s$ is the contribution from a glassy part, $\tau$ is the time constant and the exponent $0 < \beta < 1$ is linked with the distribution of energy barriers among metastable states. For an ordered FM system, the numbers of local energy minima shrinks to a single global energy minimum and $\beta$ becomes unity. In PSMO, the fitted values of $\beta$ for all $T$’s remain between $\sim 0.55$ to $\sim 0.6$ which falls within the range of $\beta$ values reported earlier for different spin glass (SG)-like systems [23,24].

The analysis of relaxation data indicates that PSMO has a metastable ground state with a distribution of local energy minima. Such energy landscape can correspond to either a disordered magnetic state or a SG like phase. Simple relaxation measurement cannot conclusively distinguish between them. In order to elucidate the exact magnetic state of PSMO, aging effect was studied carefully. The sample was zero-field–cooled down to 6 K followed by an isothermal aging at $H = 0$ for a particular wait time $t_w$. After that $H$ was increased to 100 Oe and $M$ was measured as a function of $t$.

The experiment was repeated for different values of $t_w$, namely 1500 s, 3500 s and 5500 s. Using the resulting wait-time–dependent $M(t)$ data, it is possible to calculate the relaxation rate/magnetic viscosity $S(t) = \frac{1}{M} \partial M}{\partial (ln(t))}$ [20].

For an SG, $S(t)$ shows a peak at $t_p \approx t_w$. Figure 2(b) describes the $S(t)$ graph for three different values of $t_w$’s as mentioned before. Evidently, $S(t)$ curves corresponding to $t_w = 1500$, 3500 and 5500 s show their peaks at $t_p \sim 1100$ s, 2000 s and 2500 s, respectively. Such systematic shift in peak position with varying $t_w$ is a signature of aging and regarded as a strong evidence for the presence of glassy magnetic phase. The obtained values of $t_p$ do not match exactly with $t_w$ and they are always smaller than the actual wait time. A similar effect was previously observed in glassy magnetic systems where finite spin clusters rather than individual atomic spins are associated with the spin freezing phenomenon [25,26].

Figure 3(a) depicts the field stop experiment based on $M(T)$ measurement in the FC mode [27–29]. The sample was cooled down to 3 K in 100 Oe with intermediate stops ($H$ being reduced to 0 at the stops) of duration 1 h each at 18 K and 12 K (curve $M_{FC}$). Subsequently, the sample was heated back to 30 K and $M$ was measured as a function of $T$ (curve $M_{FC+H}$). Clearly, while heating $M$ shows distinct anomaly (marked at $T_{S1}$ and $T_{S2}$) exactly at the same temperatures where the sample was allowed to age for 1 h during cooling. Those anomalies are clearly absent in the reference field-cooled heating data ($M_{H}$), which was recorded without any stop while cooling. Such signature of magnetic history is a convincing evidence for the glassy magnetic state. However, noninteracting superparamagnetic nanoparticles can show similar field stop memory effect originating from the distribution of magnetic relaxation time among the particles [30,31]. To rule out such possibility in PSMO, we measured magnetic memory in the ZFC condition, which has the same protocol as that field stop memory experiment baring the
Fig. 3: (Colour on-line) Panel (a) depicts field stop memory measurement for PSMO with an applied magnetic field of 100 Oe. \( M_{\text{stop}} \) denotes the cooling curve at 100 Oe with stops at 18 K and 12 K for 3600 s each. During the stop, the magnetic field was reduced to zero. \( M_{\text{FC}}^{\text{mem}} \) corresponds to the curve during continuous heating maintaining 100 Oe field after cooling the sample down to the base temperature with stops at 18 and 12 K. \( M_{\text{FC}}^{\text{ref}} \) denotes the reference FCH curve after the field cooling without any stop. Panel (b) shows the difference curve \((M_{\text{FC}}^{\text{mem}} - M_{\text{FC}}^{\text{ref}}})\) as obtained from the ZFC memory measurement. In this measurement protocol, the sample was initially zero-field cooled to 3 K with a stop of 10800 s at 18 K and then continuously heated back to 60 K in presence of 100 Oe field (denoted as \( M_{\text{FC}}^{\text{mem}} \)). Whereas \( M_{\text{FC}}^{\text{ref}} \) is the magnetization data during heating just after the zero-field cooling of the sample without any stop. Panel (c) shows the magnetic relaxation data at \( H = 100 \) Oe in the zero-field–cooled state measured at 18 K along with an intermediate measurement at 12 K. Panel (d) shows the temperature variation of the real part of the ac susceptibility data measured at different frequencies.

The observed glassy phase is further supported by the \( t \)-dependent memory measurements (fig. 3(c)). The sample was allowed to relax in 100 Oe field at 18 K in the ZFC condition. After 3600 s, \( T \) was decreased to 12 K without altering \( H \) and the relaxation was recorded for another 1 h. In the last segment of this \( M(t) \) measurement, the sample was again heated back to 18 K without changing the magnetic field. Interestingly the system starts to relax from a point where it ended up in the first segment. It means that the system can remember its earlier state in spite of such negative \( T \) cycling, which is a clear evidence for the SG like ground state of PSMO. Such observations of memory is in line with the Hierarchical and Droplet Model description of spin glasses [27,29,30,32–35].

It is now pertinent to investigate the onset temperature for the SG freezing. We performed magnetic memory experiment (both temperature and time dependent) at various \( T \) range. The field stop experiments on the \( M(T) \) curve (similar to what described in fig. 3(a)) fail to show any signature of memory above about 50 K. The \( t \)-dependent memory measurements (similar to what described in fig. 3(c)) also rules out the possibility of SG-like phase above about 50 K. The \( t \)-dependent memory measurements (similar to what described in fig. 3(c)) also rules out the possibility of SG-like phase above about 50 K. This is also supported by the \( T \) variation of ac susceptibility (\( \chi_{\text{ac}} \)) measurement on the sample at different frequencies. The real part of \( \chi_{\text{ac}} \) (\( \chi'_{\text{ac}} \)) shows a clear peak around 50 K (see fig. 3(d)) and the peak shifts to higher \( T \) with increasing frequency. It is to be noted that no other prominent anomaly in \( \chi_{\text{ac}} \) was observed in the entire temperature range (5–300 K) of measurement (curve not shown here).

Among single-layered manganites, a glassy magnetic state was previously reported in the case of Eu_{0.5}Sr_{1.5}MnO_{4}, La_{1.1}Sr_{0.9}MnO_{4} and Pr_{1-x}Ca_{x+1}MnO_{4}.
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Fig. 4: (Colour on-line) (a) Different structural and magnetic phases of PSMO as a function of temperature. The figure also shows the zero-field-cooled magnetization data measured at 100 Oe. Panel (b) shows the orthorhombic structural variants OR1 and OR2 along with the spin and orbital arrangements in the ground state. Here x and y orbital axes correspond to the a and b crystallographic axes of the sample, respectively.

(0.35 ≤ x ≤ 0.5) [8,32–34]. Unlike A MnO$_2$-type perovskite manganites, the long-range FM state is absent in single-layered systems, and therefore a relatively easy picture of competing interactions between FM and AFM clusters leading to a glassy phase cannot be invoked. The glassy magnetic phase in the above-mentioned single-layered manganites was explained on the basis of the fragmentation of the charge-orbital ordered state down to the nanometer scale. This leads to the mixture of AFM and FM bonds in the case of the CE-AFM structure of those SLMs resulting in competing interaction. It was found that such competing magnetic interaction in the nanometer scale can only exist in underdoped and optimally doped (x ≤ 0.5) single-layered samples of general formula T$_{1-x}$D$_x$O$_2^-$Mn$_{1-x}$O$_2^-$ (T and D are, respectively, trivalent and divalent cations). For x > 0.5, the SG state disappears due to the presence of extra holes [34].

The presently studied sample is actually an overdoped composition (x = 0.78), and therefore the above model of SG arising from the nanoscale disorder is not applicable here. The most likely scenario for such glassy phase is the existence of different orthorhombic variants with dissimilar spin structures. In fig. 4(a), we have depicted different phases of PSMO as obtained from our magnetic measurements and previous reports. The transition from TG to OR phase takes place over a wide temperature range (approximately 250 K to 40 K), which is characterized by the thermal hysteresis in M as depicted in fig. 1. The most important observation is that both the OR variants have C-type antiferromagnetically ordered state at least below 125 K, and these magnetically ordered variants coexist with each other down to the lowest T.

In fig. 4(b), we have shown two such variants along with their spin and orbital arrangements in the MnO$_2$ layer. The e$_g$ orbitals are shown for Mn$^{3+}$ ions only where they are occupied by electrons. The e$_g$ orbitals are aligned either along x (for d(3x$^2$ − r$^2$)-type OO) or y (for d(3y$^2$ − r$^2$)-type OO) direction and their orientations are mutually orthogonal in two variants. The spin structure in PSMO strongly depends upon the nature of the orbital ordering. The ordered orbitals along the x or y direction favour the electron transfer leading to FM double-exchange interaction only along the respective directions. This along with the Mn-O-Mn--type superexchange give rise to C-type antiferromagnetism, where we get antiferromagnetically coupled ferromagnetic chains along the x or y direction within a single MnO$_2$ layer.

The FM exchange interaction, which originates from the electron transfer along the ordered orbitals, will have the same direction as the axis of orbital ordering (here either x or y). This will in turn favour the spins to lie along the x or y direction. As a result, similar to the OO state, two orthorhombic variants will also have mutually perpendicular spin anisotropy axis (see fig. 4(b)).

The FM and AFM bonds in a homogeneous C-type AFM phase do not introduce magnetic frustration. However, the situation can be quite different at the interface of the two orthorhombic variants with mutually perpendicular spin anisotropy axis. As shown in fig. 4(b), such spin arrangements can introduce frustration at the interface. If one looks carefully, the spins in a chain are ferromagnetically coupled along the x-axis in the left variant (OR1), however, in the right variant (OR2) spins are antiferromagnetically coupled along the same x-direction. Therefore, the variants can introduce competing magnetic interaction in the interfacial region. Such competition along with disorder or defects due to doping can give rise to spin freezing. It was already predicted theoretically that anisotropy can indeed play a key role for the development of SG state [35]. The average size of the variants in PSMO was reported to be about few hundred nanometers, and so at low temperature there will be enough interfacial frustrated spins for the SG like states to evolve. In the above-discussed model, we have considered the interface to be perfectly sharp. However, in reality it may be rough and it can introduce additional randomness in spin structure. The above scenario is quite similar to the cluster glass in perovskite manganites and cobaltites with coexisting FM and AFM domains [36].
The only difference is that here the coexisting structural domains are all AFM but with unequal spin anisotropy axis.

It is evident from fig. 4 that magnetic frustration in the system can actually exist below 125 K where both OR1 and OR2 become antiferromagnetically ordered. However, we failed to see characteristic features (such as memory in the $t$- and $T$-dependent $M$) of a spin glass above about 50 K. $\chi_{ac}$ also shows a clear frequency-dependent anomaly around 50 K signifying the onset of spin freezing. Therefore, despite the prevailing spin frustration, the system only shows the SG phase below about 50 K. This might be related to the thermal fluctuations of spin, which only become weak enough below 50 K for spin freezing to occur. However, it is to be noted that magnetic relaxation is present even temperatures as high as $\sim$ 200 K, and it completely vanishes only above the region of hysteresis. This observed relaxation at higher $T$ is presumably not connected to the glassy magnetic phase or frustration, rather it corresponds to the landscape of metastable states associated with the disorder driven FOPT [37]. The time-dependent nucleation of a particular phase within the region of FOPT can also give rise to slow dynamics of $M$ provided the coexisting phases have distinctive magnetic character.

In conclusion, we observed a glassy magnetic state in single-layered manganite PSMO. Due to the quasi-2D structure, SLMs do not sustain metallic FM phase, and therefore the coexistence of FM/AFM clusters cannot be the cause of the glassy state. The likely origin is the existence of structural domains with C-type antiferromagnetic ordering, where spin alignment is closely related to the nature of the orbital ordering in two adjacent structural domains. The interface between two domains can give rise to spin frustration leading to a SG-like state. Such scenario can be important in understanding the glassy magnetic state in several magnetic oxides.

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SC wishes to acknowledge the Council of Scientific and Industrial Research, India.

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