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Magnetic relaxation in $La_{0.250}Pr_{0.375}Ca_{0.375}MnO_3$ with varying phase separation

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We have studied the magnetic relaxation properties of the phase-separated manganite compound $La_{0.250}Pr_{0.375}Ca_{0.375}MnO_3$. A series of polycrystalline samples was prepared with different sintering temperatures, resulting in a continuous variation of phase fraction between metallic (ferromagnetic) and charge-ordered phases at low temperatures. Measurements of the magnetic viscosity show a temperature and field dependence which can be correlated to the static properties. Common to all the samples, there appears to be two types of relaxation processes: at low fields associated with the reorientation of ferromagnetic domains and at higher fields associated with the transformation between ferromagnetic and nonferromagnetic phases.

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I. INTRODUCTION

The perovskite manganites with composition $Ln_{1-x}R_xMnO_3$, where "Ln" is a lanthanide and "R" is an alkaline earth, are a particularly important class of materials because of the close coupling between their structural, magnetic, and transport properties.[1] Recent work has suggested that the magnetoelectronic ground state of many of these materials can be inhomogeneous due to the coexistence of a ferromagnetic (FM) metallic phase and an antiferromagnetic (AFM) charge-ordered (CO) phase and that this phase separation governs many of the observed physical properties of these materials.[2–6]

The coexistence of ferromagnetic and antiferromagnetic tendencies in the manganites suggests that there should be glassy behavior associated with the frustration of the different interactions. Indeed, logarithmic time dependence has been observed in the relaxation of both resistivity and magnetization after sudden changes in magnetic field,[7–17] and there have been numerous reports of spin-glass-like behavior in these materials[18–22] (although the qualitative details of the behavior are not necessarily consistent with the conventional behavior of spin glasses[23]). While this sort of glassy behavior and the slow relaxation can be attributed to domains in ferromagnets, to superparamagnetism, or to conventional spin-glass phases associated with local disorder, another possibility in the phase-separated materials is that the relative fractions of the different phases slowly adjust to changes in the external environment. This explanation is consistent with the observation that the relaxation effects seem to be especially pronounced in materials near $x=0.50$,[8,11,15,16] where there is often a crossover between predominantly ferromagnetic conducting and antiferromagnetic charge ordered states[24] and phase separation appears to dominate the physical properties.[2,25]

In this work we investigate a series of polycrystalline samples with nominal composition $(La_{0.250}Pr_{0.375})Ca_{0.375}MnO_3$ in which the relative fraction of the coexisting phases is varied, but the charge-ordering and Curie temperatures remain virtually constant. In particular, we focus on the temperature and magnetic field dependence of the magnetic relaxation after a sudden decrease in the applied magnetic field. We find that the magnetic viscosity has a complex temperature dependence which correlates well with the static properties of the material. Furthermore, we can identify two different relaxation mechanisms, at large and small applied magnetic fields, which appear to be associated with fluctuations between ferromagnetic and charge-ordered or paramagnetic phases and reorientation of ferromagnetic domains, respectively.

II. EXPERIMENTAL DETAILS

Our polycrystalline samples of $La_{0.250}Pr_{0.375}Ca_{0.375}MnO_3$ were prepared from La_2O_3, Pr_6O_{11}, $CaCO_3$, and Mn metal by a citrate gel technique. Stoichiometric amounts of the starting materials were dissolved in diluted nitric acid (HNO_3) to which was added an excess of citric acid and ethylene glycol to make a metal complex. After all the reactants had completely dissolved, the solution was mixed and heated on a hot plate resulting in the formation of a gel. The gel was dried at 300 °C, then heated to 600 °C to remove the organic and to decompose the nitrates. The resulting powder was pressed into pellets, each of which was sintered at a different temperature (900, 950, 1000, 1100, 1200, and 1300 °C). The samples we studied are thus labeled by their sintering temperature. All the samples showed single-phase x-ray patterns, and the average grain sizes [estimated by scanning electron microscopy (SEM)] increased with increasing annealing temperature as discussed below:

The resistivity and magnetization of the samples were measured in Quantum Design PPMS cryostats and MPMS superconducting quantum interference device (SQUID) magnetometers respectively. The latter systems were also used to study the time dependence of the magnetization with the following procedure: (1) the superconducting magnet was de-gaussed by oscillating the magnetic field, (2) the sample was zero-field cooled from 300 K to a base temperature T_0, (3) the sample was held at $T=T_0$ for a time $t_{w1}=1000$ s, (4) the magnetic field was ramped up to a value H_a for another

$t_{w2} = 1000$ s, and (5) the magnetic field was reduced to near zero and the magnetization was recorded for a time interval longer than 3500 s. The end field was chosen to be 0.002 T (which is slightly higher than the typical magnitude of the residual field in these cryostats), in order to avoid a reversed residual field. The applied magnetic field H_a was set to 0.05, 0.1, 0.2, 0.3, 0.5, 1, 2, and 4 T (it takes 200 s to ramp the field to 1 T) and the time-dependent measurements were performed at 2, 5, 10, 25, 50, 75, 100, 130, and 160 K. We fit the relaxation of the magnetization with the form $M(t) = M(t=0)[1 - S(T,H_a)\log_{10}(t/\tau_0)]$, where $S(T,H)$ is the magnetic viscosity which characterizes the relaxation process.[26,27]

We found that the magnetometers were subject to a residual time-dependent magnetic field of the form $H'(t) = H'_0 + a \log_{10}[t]$ (presumably attributable to relaxation in the magnet or in nearby magnetic materials in the building). The residual field parameters, i.e., H'_0 and a, depended on the applied field H_a and were temperature independent (typical values were $a = 0.014$ Oe and $H'_0 = 24.7$ Oe for $H_a = 0.05$ T, $a = 0.142$ Oe and $H'_0 = 3.92$ Oe for $H_a = 4$ T.) The magnetic moment induced in the $La_{0.250}Pr_{0.375}Ca_{0.375}MnO_3$ samples by this field was obtained from the measured low-field susceptibility and then subtracted from the measured magnetic moment in order to obtain the actual magnetic viscosity. This correction was small, but significant when S approached zero.

III. RESULTS

A. Time-independent measurements

We chose to examine a composition from the series $(La_{5/8-y}Pr_y)Ca_{3/8}MnO_3$ in which the varying Pr fraction (y) controls the fraction of the ferromagnetic metallic phase in the ground state. Previous studies have demonstrated two-phase coexistence, involving a predominantly FM metallic state at small y, a predominantly charge-ordered state at large y, and the relative fractions changing with y.[28–31] Furthermore, there is strong evidence that all of the magnetotransport properties are dominated by the coexistence of the two phases, since the application of a magnetic field simply converts the sample from the charge-ordered state to the ferromagnetic conducting state. Kiryukhin *et al.*[32] examined the phase diagram for $y = 0.35$, and they found that with decreasing temperature the compound first undergoes a transition to a predominantly charge-ordered phase at $T_{CO} \approx 200$ K and then an insulator-metal transition at $T \sim 70$ K to a mixed phase consisting of CO insulating, paramagnetic, and FM metallic regions. They also concluded that a non-charge-ordered paramagnetic phase is present below T_{CO} which coexists with the charge-ordered phase and that it (rather than the charge-ordered phase) evolves into the ferromagnetic metallic phase below T_C in zero magnetic field.

Due to the strong coupling between the lattice and other physical properties, the strain associated with the grain boundaries and other types of structural defects can dramatically affect the electrical and magnetic behavior of polycrystalline manganites.[33,34] In our series of polycrystalline samples with $y = 0.375$, we find that the grain size increases monotonically with annealing temperature, as shown in Fig. 1. Since this composition is near the edge of the compositional transition between the metallic and insulating ground states, one might expect a dramatic effect on the resistivity from this change in grain size. Indeed, we do observe a striking difference between the samples in the temperature-dependent resistivity which is plotted in Fig. 2 in zero applied magnetic field and in a field of 10 T. For samples 900 and 950, the resistivity increases monotonically with decreasing temperature in zero magnetic field to the lowest temperature we could measure. For the other samples, the zero-field resistivity begins to decrease at a temperature near the onset of ferromagnetism seen in the magnetization data (discussed below). All samples have a sharply suppressed resistivity in the high magnetic field, and this large magne-

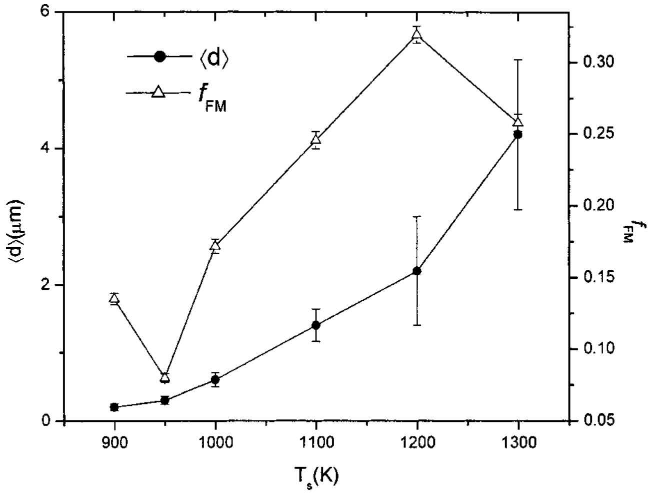

FIG. 1. Average grain size of the polycrystalline samples (from scanning electron microscopy) and the volume fraction of ferromagnetic phase at 5 K [from $M(H)$ data as described in the text].

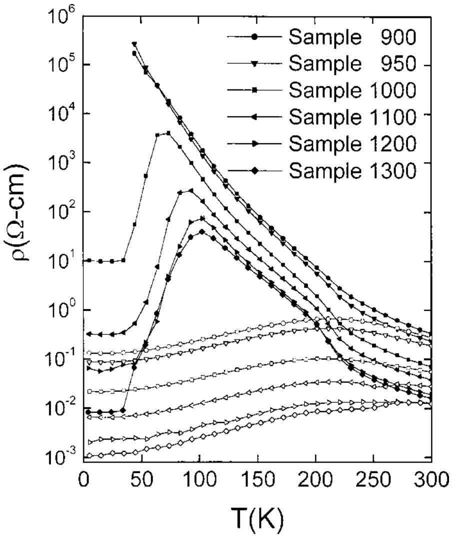

FIG. 2. Temperature dependence of the resistivity on cooling in zero magnetic field (solid symbols) and in $H = 10$ T (open symbols) of the entire series of samples.

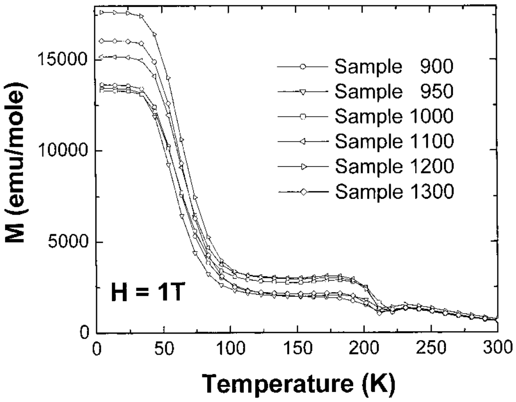

FIG. 3. Magnetization as a function of temperature measured on cooling of the entire series of samples.

toresistance is presumably associated with a field-induced ferromagnetic metallic state indicated by the saturated moment observed in magnetization measurements as a function of field (see Fig. 4 below). The onset of charge ordering is discernable as a steep change in the resistance around $T_{\mathrm{CO}} \sim 220\,\mathrm{K}$ for all samples, and T_{CO} can be determined from a maximum in the slope of the resistivity. The electrical behavior of the samples is similar to that reported previously[28,29,35] for $(\mathrm{La,Pr,Ca})\mathrm{MnO}_3$ in which the magnetotransport data could be described in terms of percolative transport through the ferromagnetic metallic regions which increased in volume with La content. The resistivity data indicate that the fraction of the metallic phase analogously increases monotonically with annealing temperature (and grain size) in the series of samples studied here and also increases with applied magnetic field at all temperatures below T_{CO}.

In Fig. 3, we show the temperature dependence of the magnetization of the samples in a 1-T magnetic field, showing a feature near $T_{\mathrm{CO}} \sim 200\,\mathrm{K}$ and a sharp rise at lower temperatures where part of the sample becomes ferromagnetic. The Curie temperature $T_C \sim 85\,\mathrm{K}$ can be determined from these data as the maximum in the temperature derivative. In Fig. 4, we show the field dependence of the magnetization, $M(H)$, after zero-field cooling at various temperatures for sample 1100. This plot demonstrates paramagnetic behavior at high temperatures $[M(H)$ is linear$]$ and evidence for ferromagnetism at the lowest temperatures [in that there is downward curvature of $M(H)$]. Between T_{CO} and T_C, there is a small ferromagnetic rise in $M(H)$ and then upward curvature—presumably indicating the presence of a small ferromagnetic fraction which grows to encompass the entire sample in a sufficiently strong field. At the lowest temperatures $M(H)$ exhibits a plateau at around 1 T which presumably corresponds to fields which are sufficient to align the moments of those regions which are ferromagnetic at zero field, but which are not sufficient to transform charge-ordered regions into ferromagnetism. We can estimate the volume fraction which is ferromagnetic after zero-field cooling (f_{FM}) by taking the ratio of the plateau magnetization to the theoretically expected magnetization ($3.625\mu_{\mathrm{B}}$/f.u.) for complete alignment of all the Mn moments. The values of f_{FM} at 5 K for all the samples are plotted in Fig. 1, and it is notable that the ferromagnetic fraction at low temperature increases with annealing temperature as the samples become more conducting. Furthermore, when f_{FM} exceeds the three-dimensional percolation threshold of 14.5% (Ref. 36) for samples 1000–1300, the low-temperature zero-field state of the samples changes from insulating to conducting. Thus we can conclude that the relative fractions of the two phases are changing across our sample series, but that the individual phases (as characterized by T_{CO} and T_C) are unchanged. It is interesting to note that our observed dependence of f_{FM} on grain size and annealing time is opposite that seen by Levy *et al.* in $\mathrm{La}_{0.5}\mathrm{Ca}_{0.5}\mathrm{MnO}_3$,[37] perhaps due to the inverted order of T_C and T_{CO} in that material compared to $(\mathrm{La}_{5/8-y}\mathrm{Pr}_y)\mathrm{Ca}_{3/8}\mathrm{MnO}_3$. Due to the smooth variation of f_{FM} in our series, we can thus examine the relaxation process as a function of the fractions of the coexisting phases, as described in the following section.

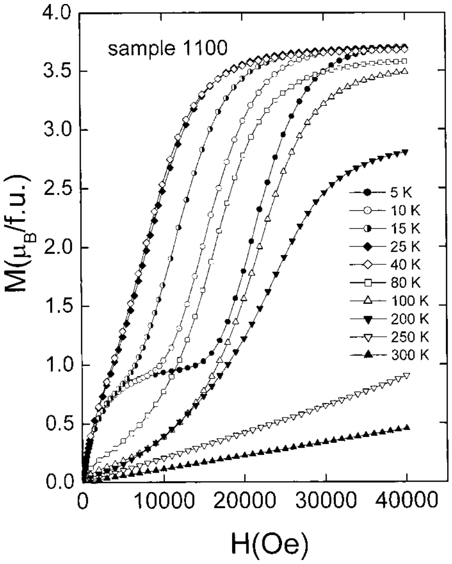

FIG. 4. Magnetization vs field (after zero-field cooling), measured at various temperatures for sample 1100.

B. Relaxation measurements

We studied the magnetic relaxation of each of the samples in the series using the procedure described above to obtain the magnetic viscosity. We focused our studies on low temperatures ($T \leqslant 160\,\mathrm{K}$) in order to examine the regions in which two-phase coexistence clearly dominates the behavior. Figure 5 shows representative profiles of the magnetic relaxation for sample 1000 with an applied field of $H_a = 0.05\,\mathrm{T}$ and different temperatures [Fig. 5(a)] and with different applied magnetic fields for $T = 130\,\mathrm{K}$ [Fig. 5(b)]. The magnetization in each case was normalized to the corresponding value at $t=0$ (immediately after the field was stabilized near zero) and $M(t)/M(0)$ was plotted versus $\log_{10}(t)$. The asymptotic slopes of these curves, i.e., the long-time logarithmic relaxation rates, were used to estimate the magnetic viscosity,

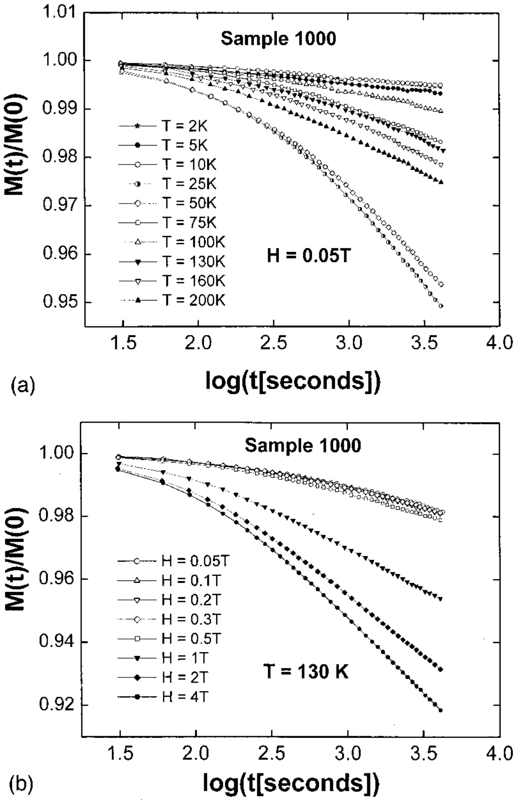

FIG. 5. (a) Profiles of the magnetization relaxation for sample 1000 for $H_a = 0.05$ T and a range of temperatures. (b) Profiles of the magnetization relaxation for sample 1000 at 130 K for different applied magnetic fields (H_a).

$$S = \frac{1}{M_0} \frac{dM}{d(\log_{10}(t))},$$

which was then corrected for the residual field as described above.

The temperature dependence of S for sample 1100 is shown in Fig. 6 for a range of applied magnetic fields (H_a). As can be seen in this figure, the temperature dependence of S has a characteristic behavior in low applied fields (open symbols, $H_a \leqslant 0.3$ T) and a different characteristic behavior in larger fields (solid symbols, $H_a \geqslant 0.5$ T). The difference between the low- and high-field regimes can also be seen by plotting the same data (Fig. 7) as a function of the applied field at different temperatures. Although S is small and nearly independent of the applied field at the lowest temperatures, for all higher temperatures there is a clear difference between the two field regimes. At $T = 25$ and 50 K, S has a large magnitude for small fields, but approaches zero rapidly up to $H_a \sim 0.5$ T where it saturates. For temperatures above 50 K, S has weak field dependence up to $H_a \sim 0.5$ T: then, it dramatically increases in magnitude for larger fields.

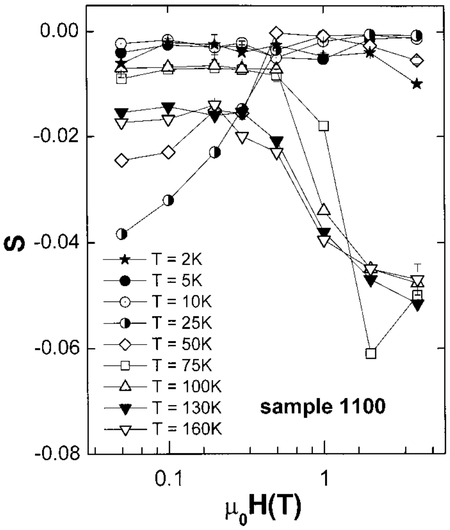

FIG. 7. Field dependence of the magnetic viscosity at different temperatures for the sample 1100.

Since the magnetic viscosity appears to have different behavior for low and high values of the applied magnetic field H_a, we must conclude that there are two different relaxation processes responsible for the magnetic viscosity in this series of materials. We first discuss the small H_a processes shown in Fig. 8, where S has an almost constant small magnitude at the lowest temperatures ($T \lesssim 10$ K), has the largest magnitude just below T_C and then has a small and almost

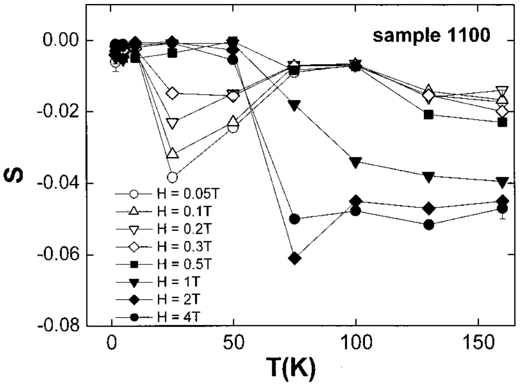

FIG. 6. Temperature dependence of the magnetic viscosity for sample 1100 in different applied magnetic fields (H_a). Note the two different characteristic behaviors at low and high fields as designated by the open and solid symbols, respectively.

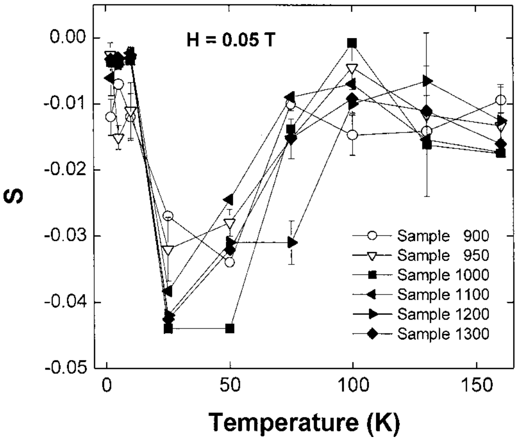

FIG. 8. Temperature dependence of the magnetic viscosity S for all the investigated samples for a 0.05-T applied magnetic field.

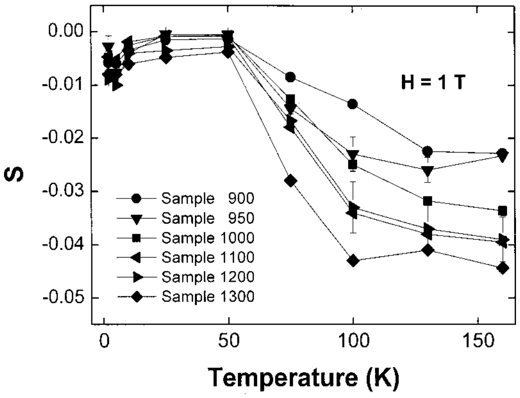

FIG. 9. Temperature dependence of the magnetic viscosity for all the investigated samples for a 1-T applied magnetic field.

temperature-independent value above T_C. We do not attribute the observed relaxation processes in this low-field range to changes in the relative fractions of ferromagnetic and nonferromagnetic regions because there is little magnetoresistance at such low fields (which indicates that there is little conversion from charge order to metallic conduction). There is also no consistent variation among the obtained values of S between the samples in the series, even though the resistivity and equilibrium magnetization data discussed above indicate a changing ferromagnetic fraction among the samples. We attribute the relaxation in this low-field regime instead to relaxation of the moment orientation of the ferromagnetic regions in the material which exist even after zero-field cooling. This explanation is consistent with the largest viscosity being observed just around T_C where the moments are more easily reoriented. We also observe that S at the lowest temperatures has a larger magnitude for samples 900 and 950 than for all of the other samples. Those samples are below the percolation threshold in their ferromagnetic fraction, which presumably would have an effect on the magnetic relaxation processes due to anisotropy factors.

The temperature dependence of S is quite different at the higher magnetic fields as shown in Figs. 9 and 10. In this field regime, S is almost constant for $T<T_C$, but then increases abruptly in magnitude near T_C and for higher temperatures, i.e., in the predominantly charge-ordered state. This temperature dependence is qualitatively similar to that observed by Lopez *et al.* in $La_{0.5}Ca_{0.5}MnO_3$ after reducing the field from 5 T (the only magnetic field from which they studied relaxation). We attribute relaxation in this regime to the fraction of ferromagnetic volume decreasing after the removal of the magnetic field, i.e., to the relaxation of the proportions of the coexisting phases. This view is supported by the data in Fig. 11 where we plot S for $H_a=1$ T as a function of sample number (where the higher numbered samples were sintered at higher temperature and have a larger ferromagnetic phase fraction as discussed above). While the data are somewhat noisy at the lowest temperatures (due to the smaller magnitude of S), we see that S increases in magnitude significantly (by a factor of 2 or more) with increasing ferromagnetic phase fraction in this high-field regime. Since both T_C and T_{CO} are approximately constant for all of the samples, the change in S among the samples can be attributed to the changing ferromagnetic fraction and thus the relaxation itself to relaxation of the proportions of the coexisting phases. The increase in S we observe just above T_C in this field regime is also consistent with this explanation. While for $T<T_C$ the ferromagnetic fraction remains roughly constant after removing the magnetic field, the application of a strong magnetic field to the CO phase converts some fraction of it to a ferromagnetic conducting phase (see Fig. 4). After reducing the applied magnetic field, the system reverts to the equilibrium predominantly charge-ordered state,[28] and we are apparently observing the relaxation of that process. An interesting point in the data is the trend of increasing S with the increasing sample number which correlates with an increased fraction of the ferromagnetic state within the predominantly charge-ordered matrix. In other words, the system relaxes faster when the end point has a larger fraction of the minority ferromagnetic phase. This result should give insight into the dynamics of the conversion process between charge order and ferromagnetism with the application and removal of magnetic field.

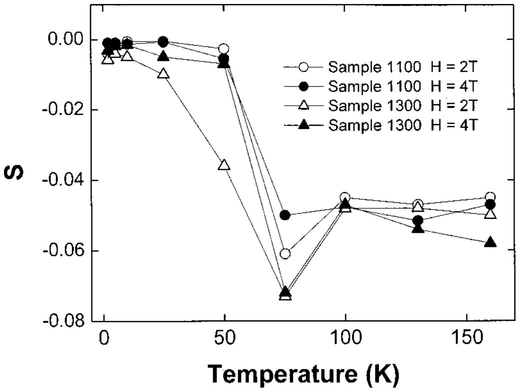

FIG. 10. Temperature dependence of the magnetic viscosity S for the samples 1100 and 1300 for $H_a=2$ and 4 T.

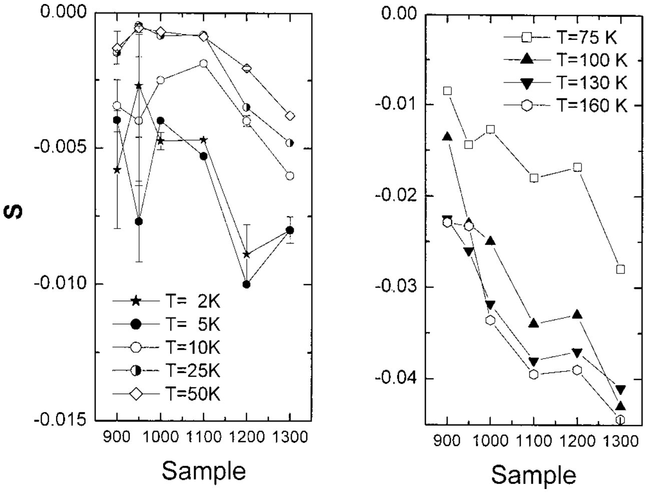

FIG. 11. Magnetic viscosity as a function of the sample sintering temperatures, measured at different temperatures and $H_a=1$ T.

IV. CONCLUSIONS

In summary, we have examined the relaxation properties of a series of phase separated manganite materials in which the relative phase fraction was varied through sintering of the samples. We find that two different relaxational processes are important in these systems corresponding to either the relaxation of moment orientation or the relaxation of phase fraction. This latter process is unique to the phase-separated nature of the manganites and may constitute a new sort of magnetic glassiness—based on the macroscopic yet dynamic coexistence of two dissimilar phases. This would add an additional aspect to the many unique properties of the perovskite manganites, but further studies of the detailed dynamics of phase separation, through microscopic probes or theoretical simulations, will be needed to fully characterize the nature of these processes.

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