PHYSICAL REVIEW B, VOLUME 64, 144404

Magnetic relaxation phenomena and cluster glass properties of $La_{0.7-x}Y_xCa_{0.3}MnO_3$ manganites

R. S. Freitas* and L. Ghivelder
Instituto de Física, Universidade Federal do Rio de Janeiro, C.P. 68528, Rio de Janeiro, RJ 21945-970, Brazil

F. Damay
Materials Department, Imperial College, Prince Consort Road, London SW7 2BP, United Kingdom

F. Dias
Instituto de Física, Universidade Federal do Rio Grande do Sul, C.P. 15051, Porto Alegre, RS 91501-970, Brazil

L. F. Cohen
Blackett Laboratory, Imperial College, Prince Consort Road, London SW7 2AZ, United Kingdom

(Received 5 October 2000; revised manuscript received 27 April 2001; published 11 September 2001)

The dynamic magnetic properties of the distorted perovskite system $La_{0.7-x}Y_xCa_{0.3}MnO_3$ $(0 \leqslant x \leqslant 0.15)$ have been investigated by ac susceptibility and dc magnetization measurements, including relaxation and aging studies. All investigated samples display a metal-insulator transition. As yttrium is added in the compounds the overall results show evidence for the gradual appearance of a cluster glass behavior. For the $x=0.15$ sample, magnetization measurements as a function of time at various temperatures show that the magnetic relaxation is maximum at a given temperature, well below the ferromagnetic transition. This maximum coincides in temperature with a frequency-dependent feature in the imaginary part of the ac susceptibility, associated with a freezing process. This is interpreted as due to ferromagnetic clusters, which grow with decreasing temperature down to a temperature at which they freeze due to severe intercluster frustration.

DOI: 10.1103/PhysRevB.64.144404 PACS number(s): 75.50.Lk, 75.30.Vn, 72.80.Ga

I. INTRODUCTION

Extensive study of the doped perovskite manganites $La_{1-y}Ca_yMnO_3$ revealed a very rich and complex phase diagram, which includes colossal magnetoresistance (CMR) behavior near the transition temperature of the metallic ferromagnetic compositions, $0.15 < y < 0.50$.[1] The ferromagnetic (FM) interaction appears as a consequence of the manganese mixed-valence state. Substitution of La^{3+} by Ca^{2+} in antiferromagnetic $LaMnO_3$ changes the Mn^{3+}/Mn^{4+} ratio and produces holes in the e_g orbitals. The simultaneous para-ferromagnetic (PM) and metal-insulator transitions are basically understood within the framework of the double-exchange theory,[2] which considers the transfer of one e_g electron between neighboring Mn^{3+} and Mn^{4+} ions through the path Mn-O-Mn.

For a given e_g concentration the tolerance factor of the perovskite structure, proportional to the average ionic size of the A site, was shown to have a drastic influence on the physical properties of manganites. Compounds of the type $(La_{0.7-x}R_x)Ca_{0.3}MnO_3$ ($R=$Pr, Y, Dy, or Tb), where the Ca concentration is close to an optimum value in relation to the FM interaction, are particularly suited to investigate this effect: replacing La^{3+} with smaller rare-earth ions decreases the Mn-O-Mn bond angle and thus affects the hopping of the e_g electrons and weakens the double-exchange interaction, while the antiferromagnetic superexchange term is only marginally affected and therefore increase in importance.[3] Consequently, both double-exchange and superexchange interactions will compete more strongly depending on the structural distortion, giving rise to a magnetically disordered state. A magnetic and electronic phase diagram of several manganite systems plotted against the tolerance factor has been published on several investigations.[4,5] Lowering the value of the average ionic size of the A site initially reduces the transition temperature T_C, and below a critical value the low-temperature phase changes to a spin-glass insulating state. Preliminary studies[6] have additionally shown that the FM metallic phase of $La_{0.7-x}Y_xCa_{0.3}MnO_3$ $(x=0.15)$ displays signatures of a magnetic cluster glass state.

The purpose of the present work is to investigate the dynamic properties of this disordered phase by means of magnetic relaxation measurements. One characteristic feature of glassy systems is that the magnetization of a sample cooled in zero field to a certain temperature depends on how long it is held in zero field before the magnetic field is applied. This is the so called aging effect. Furthermore, if $M(t)$ is differentiated with respect to ln t, the derivative $S=\partial M/\partial(ln t)$ displays a maximum which shifts to higher t for longer values of the wait time, t_w. These effects have been observed on a well-characterized cluster glass compound, the cobaltite $La_{0.5}Sr_{0.5}CoO_3$, where glassy behavior and intracluster ferromagnetism were shown to coexist.[7,8] More recently, on $Nd_{0.7}Sr_{0.3}MnO_3$, it was shown that disorder and frustration may occur in the ferromagnetic phase.[9]

Within this context, this paper presents low-temperature magnetic relaxation measurements on $La_{0.7-x}Y_xCa_{0.3}MnO_3$, with $0 \leqslant x \leqslant 0.15$. As yttrium is added in the compound, the results evidence the gradual appearance of glassy signatures within the ferromagnetic metallic phase, prior to the existence of the insulating spin-glass phase which occurs with higher rare-earth doping at the La site.[4,5] Relaxation measurements probe the out-of-equilibrium state of the system,

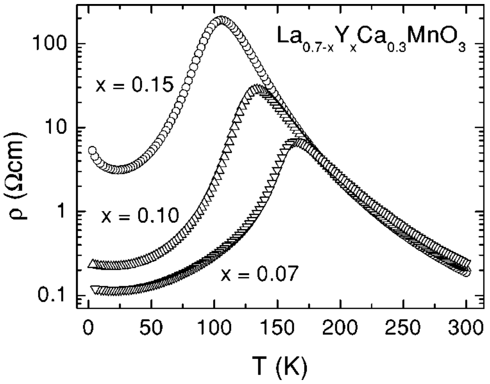

FIG. 1. Temperature dependence of the resistivity of the studied compounds.

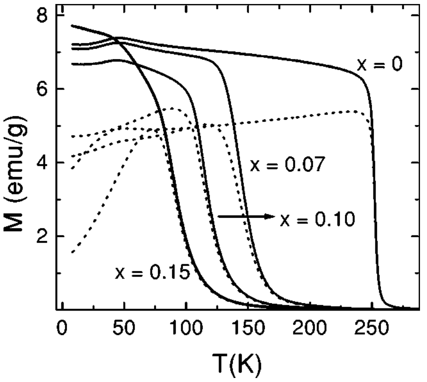

FIG. 2. Field-cooled (solid lines) and zero-field-cooled (dotted lines) dc magnetization of $La_{0.7-x}Y_xCa_{0.3}MnO_3$, measured with 5 mT.

where the clear observation of a peak in the magnetic relaxation as a function of temperature and the observation of aging effects further characterize the cluster glass properties of this system.

II. EXPERIMENT

The investigated compounds are polycrystalline samples of $La_{0.7-x}Y_xCa_{0.3}MnO_3$, with $x=0$, 0.07, 0.10, and 0.15, prepared by standard solid-state reaction. X-ray analysis confirmed a single-phase orthorhombic perovskite structure. Magnetic measurements were made with an extraction magnetometer (Quantum Design PPMS) when the applied field was greater then 5 mT. Low-field data, $H=0.3$ mT, were taken with a superconducting quantum interference device (SQUID) magnetometer (Quantum Design MPMS). Zero-field-cooled relaxation measurements were performed on all samples at different temperatures between 10 and 250 K, and for applied fields varying from 5 mT to 0.5 T. The samples were cooled from a reference temperature in the paramagnetic state with a constant cooling rate and kept at a target temperature for a certain wait time t_w. Thereafter, a dc field was applied and the magnetization was recorded versus the elapsed time *t*. The samples were additionally characterized by transport measurements, made by a conventional ac four-probe method. The results are shown in Fig. 1. All samples display a metal-insulator transition, below which a metallic-like electrical conductivity is observed. A similar behavior was previously reported for these compounds.[10]

III. RESULTS AND DISCUSSION

To recall the characteristic magnetic behavior of $La_{0.7-x}Y_xCa_{0.3}MnO_3$ compounds, Fig. 2 displays the zero-field-cooled (ZFC) and field-cooled (FC) temperature dependences of the dc magnetization of the samples $x=0$, 0.07, 0.10, and 0.15, measured at $H=5$ mT. The small hump visible at low temperatures in most of the FC data is due to a problem in the experimental apparatus. The results of Fig. 2 clearly show a standard FM transition for the $x=0$ sample at $T_C=252$ K, as observed in numerous investigations.[1] For the doped compounds ($x>0$) the ZFC curves evolve continuously, with higher yttrium concentration, to a cusplike anomaly at a temperature T_a just below the Curie-like temperature T_C, defined as the maximum inflection of the FC data. The T_C values obtained are 143, 114, and 89 K, for $x=0.07$, 0.10, and 0.15, respectively. An important point to be noted here is that the FC magnetization continues to increase strongly below the irreversibility temperature T_r at which ZFC and FC curves merge, a typical feature of various cluster glass systems.[11,12] This effect is more pronounced on the $x=0.15$ sample. On the other hand, in canonical spin-glass systems the FC magnetization shows a nearly constant value below T_r. It worth noting that due to the fact that cluster glasses exhibit finite-range ferromagnetic ordering below T_C, this system may show some features similar to those found in reentrant spin-glass (RSG) systems, which undergo a PM-FM transition at T_C and have a lower freezing temperature. Nevertheless, it may be noted that in several RSG systems, such as NiMn,[1314] AuFe,[15], and FeZr,[16] the irreversibility temperature T_r occurs at temperatures well below T_C (when the measuring field is higher then the coercive field), whereas in a cluster glass the irreversibility arises just below T_C,[7] as observed in Fig. 2 for the yttrium-doped samples.

On the other hand, magnetization curves measured as a function of field, $M(H)$, do show signatures of a RSG behavior. The low-field portion of a typical hysteresis loop measured after the ZFC condition at 2.0 K, a temperature well below T_r, is shown in the inset of Fig. 3. The curve displays an S shape in the virgin branch, with a positive curvature at low fields, a typical characteristic of both canonical SG and RSG systems.[7,17,18] In the same way, the variation of the coercive field H_C with temperature, plotted in Fig. 3, is quite similar to those usually observed in RSG systems, where H_C increases rapidly as the temperature is lowered below T_C.[18,19] Such conflicting results evidence a more complex glassy behavior in these compounds.

Susceptibility measurements are a very efficient way to evidence glassy behavior. The temperature dependence of the ac susceptibility of $La_{0.55}Y_{0.15}Ca_{0.3}MnO_3$ is presented in Fig. 4. The variation with temperature of the in-phase susceptibility χ', shown in the inset of the figure, is comparable to that of the dc M_{ZFC}, with an onset around 120 K related to the paramagnetic to ferromagnetic transition, followed by a maximum at $T=79$ K, close to the M_{ZFC} cusp temperature

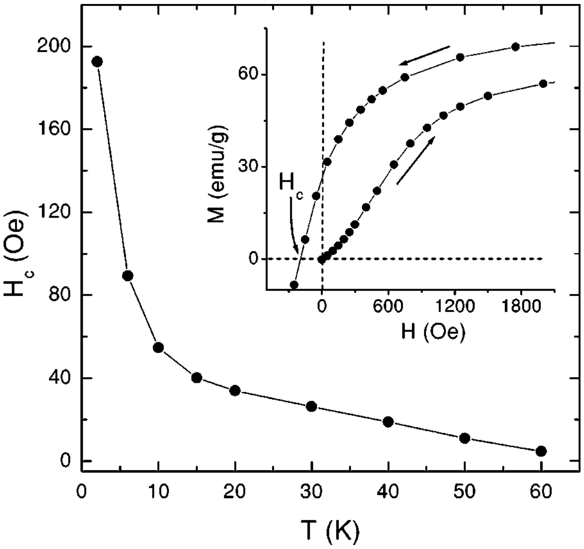

FIG. 3. Temperature dependence of the coercive field H_C obtained from $M(H)$ measurements after the application of $H=9$ T. The inset shows the low-field portion of the $M(H)$ measurements taken at 2.0 K. The lines are only a guide to the eyes.

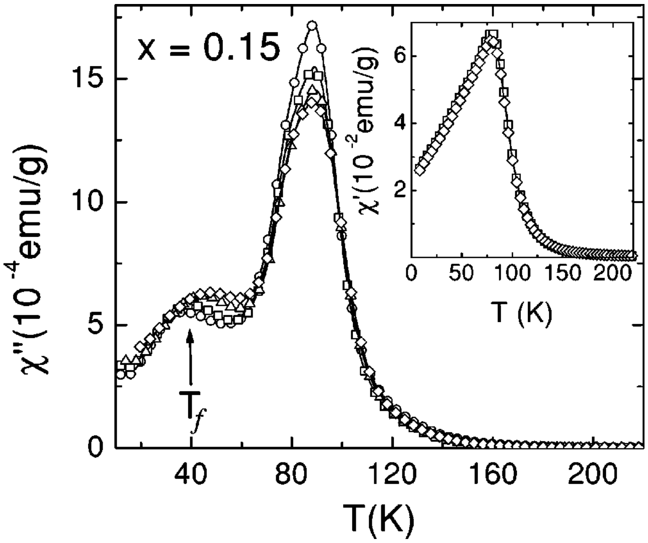

FIG. 4. Out-of-phase susceptibility (χ'') of $La_{0.55}Y_{0.15}Ca_{0.3}MnO_3$, measured with an ac field $h_{ac}=0.5$ mT and frequencies $f=90$ (circles), 250 (squares), 700 (triangles), and 2000 Hz (diamonds). The position of a frequency-dependent feature in the data is indicated by T_f. The inset shows the in-phase susceptibility (χ') of the same sample.

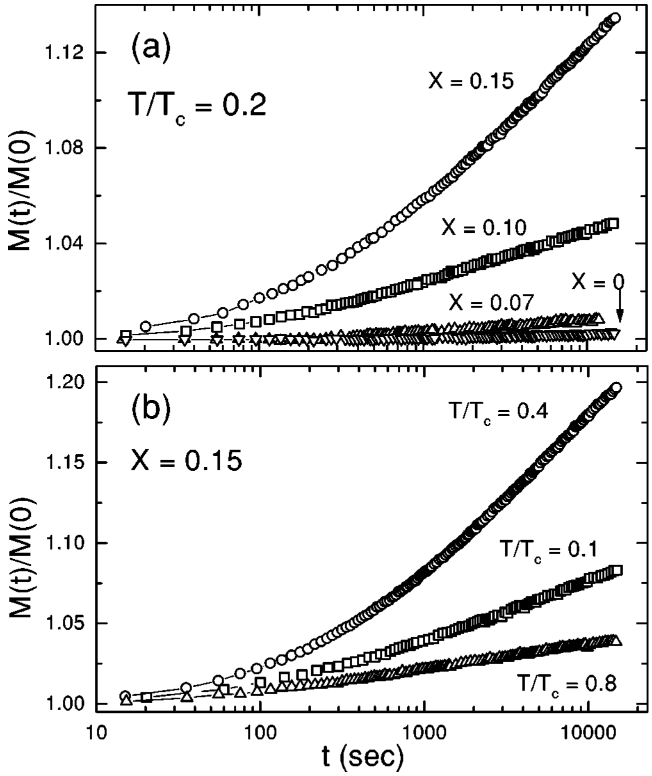

FIG. 5. (a) Normalized zero-field-cooled magnetization of $La_{0.7-x}Y_xCa_{0.3}MnO_3$ measured as a function of time, with $H=5$ mT at a reduced temperature $T/T_C=0.2$; (b) The same measurement for $La_{0.55}Y_{0.15}Ca_{0.3}MnO_3$, at $T/T_C=0.1$,0.4, and 0.8.

$T_a=72$ K. This maximum is not frequency dependent, which suggests that the FM state originates from intracluster ferromagnetism. Moreover, if the χ' maximum and T_a are related, the cusp on the M_{ZFC} curve is the dc signature of these FM interactions, rather than a consequence of cluster freezing. The out-of-phase susceptibility χ'' also shows a maximum at $T=87$ K, which is frequency independent. It has been suggested[8] that the temperature at which this maximum occurs is related to the irreversibility temperature $T_r=88$ K obtained on the dc magnetization curves. Therefore the maximum in χ'' at higher temperatures is also related to the intracluster FM interactions. However, an additional feature clearly visible in χ'' is the presence of a hump, at $T_f \sim 40$ K. This broad peak is frequency dependent and shifts towards higher temperatures with increasing frequency, a characteristic feature of the dynamics of spin-glass systems. A similar behavior has previously been reported in other manganite samples.[20] This double-peak structure in χ'' is progressively less visible for lower Y doping, and it is not present in the Y=0 sample.

In order to gain further information on the underlying nature of this cluster glass system, we have measured the long-time relaxation of the magnetization, with a time scale greater than 10^4 s. Figure 5(a) displays the normalized $M(t)/M(0)$ data for all samples measured at a reduced temperature $T/T_C=0.2$, with $H=5$ mT (this probing field is within the linear response regime). As Y is added in the compounds the data show a gradual increase of $M(t)/M(0)$, at any given time. Quantitatively, after 10^4 s, the fractional change of the magnetization is 0.18%, 0.93%, 4.5%, and 12% in the samples with $x=0$, 0.07, 0.10, and 0.15, respectively. Relaxation data at different temperatures for $La_{0.55}Y_{0.15}Ca_{0.3}MnO_3$ are shown in Fig. 5(b). The slope of $M(t)/M(0)$ increases below T_C, reaches a maximum at $T/T_C \simeq 0.4$, and decreases for lower temperatures. This evolution of the magnetization relaxation behavior with temperature is indicative of the development of magnetic clusters as the temperature is lowered. It is noteworthy that

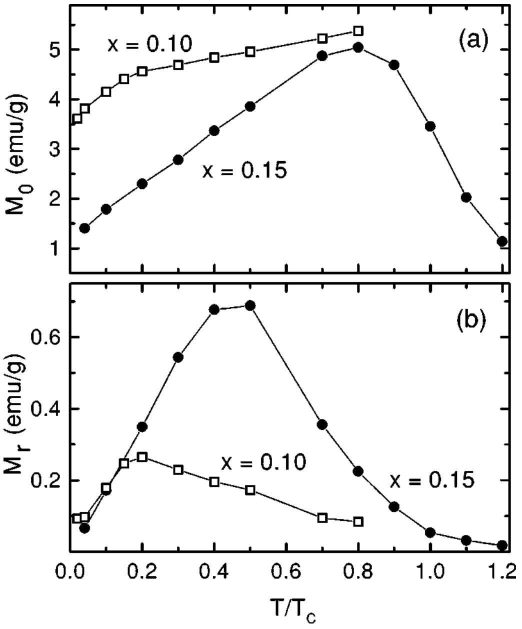

FIG. 6. Evolution of the fitting parameters M_0 and M_r (see text for details) as a function of a reduced temperature T/T_C, for $x=0.10$ and 0.15 samples.

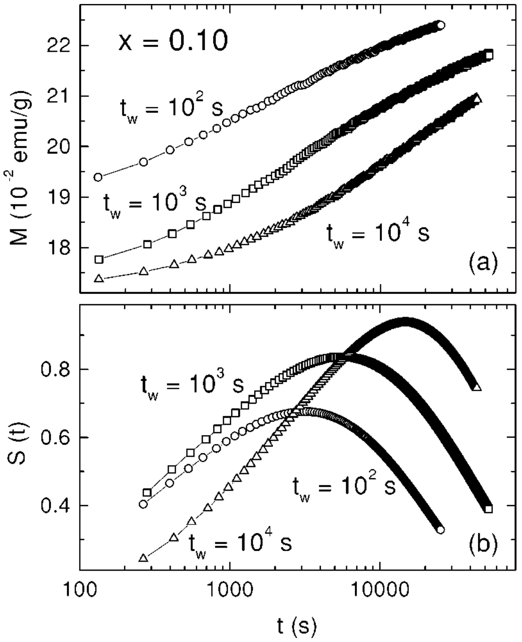

FIG. 7. (a) Zero-field-cooled relaxation magnetization and (b) the corresponding relaxation rate $S(t)=\partial M/\partial \ln t$, of $La_{0.60}Y_{0.10}Ca_{0.3}MnO_3$, measured at $T/T_C=0.2$ with $H=0.3$ mT after different wait times $t_w=10^2$, 10^3, and 10^4 s.

relaxation is observed not only below the freezing temperature T_f, but also up to the FM transition temperature T_C, which indicates that the system is not in a true ferromagnetic state. This is reinforced with measurements of $M(H)$ above and below T_C (not shown), where Arrot plots of M^2 vs H/M do not yield straight lines. Previous observations in RSG systems[21] also show that relaxation is present above the freezing temperature of the spin-glass state.

Among the various functional forms that have been proposed to describe magnetization as a function of time, one of the most popular is a stretched exponential of the form

$$M(t)=M_0-M_r\exp\left[-\left(\frac{t}{\tau_r}\right)^{1-n}\right], \tag{1}$$

where M_0 relates to an intrinsic FM component and M_r to a glassy component mainly contributing to the relaxation effects observed. The time constant τ_r and the parameter n are related to the relaxation rate of the spin-glass-like phase. The values of n are scattered between 0.48 and 0.6, in agreement with previous results.[20] The variations of M_0 and M_r with reduced temperature are shown in Fig. 6 for the samples with $x=0.10$ and 0.15. As expected, M_0 and M_r depend strongly upon temperature. The dependence of M_0 with T/T_C evidences the FM transition at $T/T_C\simeq 1$. As observed in the temperature dependence of M_{ZFC}, the FM component first reaches a maximum at $T/T_C\simeq 0.8$, which corresponds to T_a (M_{ZFC} cusp temperature) and decreases further with decreasing temperature. The FM component M_0 is larger for $x=0.10$ compared to $x=0.15$, in agreement with its stronger FM behavior. The variation of M_r with T/T_C shows that for $x=0.15$ the relaxing component first increases from $T/T_C\simeq 1$ to $T/T_C\simeq 0.45$, as a consequence either of an increase of the cluster numbers or of the growth of the cluster sizes, and decreases for $T/T_C<0.45$. The resulting maximum in M_r coincides with the temperature of the frequency-dependent lower maximum in the out-of-phase susceptibility, which supports the idea that it is related to the beginning of cluster freezing. A similar behavior is observed for $x=0.10$, but at a lower temperature, with the maximum in M_r occurring around $T/T_C\simeq 0.20$. The relaxing component M_r in the $x=0.10$ sample is also much lower than in $x=0.15$, due to a less pronounced glassy behavior. The relaxation time τ_r is extremely sensitive to any noise in the data. However, our fitting (not shown) indicates that τ_r increases with decreasing temperature for both $x=0.15$ and $x=0.10$, suggesting a regular stiffening of the spin relaxation, which is compatible with an increase of the cluster sizes and enhanced intercluster frustration.

To further investigate the dynamic magnetic behavior of these metallic glass compounds the wait time t_w dependence of the long-time-relaxed magnetization, or aging effect, was studied. The results were obtained with a small dc field $H=0.3$ mT. Additional measurements (not shown) as a function of the applied field revealed that the magnitude of the aging effect is reduced when the field increased and totally disappears at $H=10$ mT. Magnetization versus time was measured with wait times $t_w=100$, 1000, and 10 000 s, before application of the magnetic field. Results for the $x=0.10$ sample, measured at $T/T_C=0.2$, are plotted in Fig. 7(a). This temperature was chosen because it corresponds to the maximum in the relaxing component M_r. It is clear from the figure that the measured magnetization strongly depends on t_w, which confirms the existence of aging processes. Macroscopically, aging means that the system becomes "stiffer" for larger wait time; i.e., the measured magnetiza-

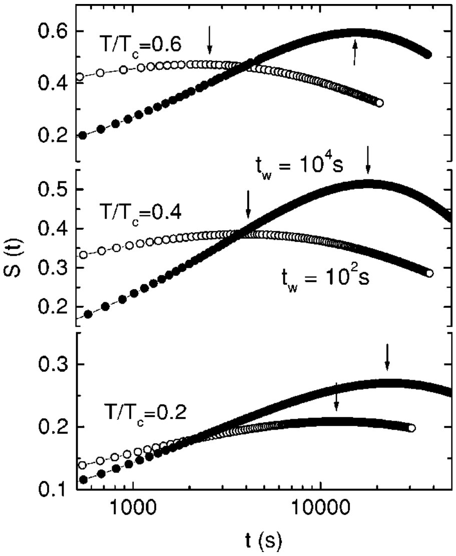

FIG. 8. Relaxation rate $S(t)=\partial M/\partial \ln t$ of $La_{0.55}Y_{0.15}Ca_{0.3}MnO_3$ measured after different wait times $t_w=10^2$ (open circles) and 10^4 (solid circles) at $T/T_C=0.2,0.4,0.6$. The arrows indicate the peak position of each data curve.

tion is lower for higher t_w, as observed in the data. The corresponding time-dependent relaxation rate $S(t) =\partial M/\partial(\ln t)$ is plotted in Fig. 7(b). The $S(t)$ curves were obtained by taking the derivative of a polynomial fit of the magnetization data. It is readily observed that $S(t)$ reaches a maximum, corresponding to an inflection point on the $M(t)/M(0)$ curves, which shifts to longer observation times for longer values of t_w. These results are similar to those reported for various glassy systems.[8,9,22,23] It may be pointed out that the maximum in $S(t)$ is not centered on t_w as expected. However, it is believed that this is related to the additional time required for stabilizing the temperature and the magnetic field applied.

For $La_{0.55}Y_{0.15}Ca_{0.3}MnO_3$, $S(t)$ has been calculated at various temperatures, and the results are shown in Fig. 8. Aging effects are observed at $T/T_C=0.2$, 0.4, and 0.6. The wait time dependence of the peak in $S(t)$ clearly increases with temperature. Aging effects are more pronounced at higher temperatures, i.e., when the FM state is stronger, as reported previously for $La_{0.5}Sr_{0.5}CoO_3$ (Ref. 8) or for the two-dimensional ferromagnet $Rb_2Cu_{0.89}Co_{0.11}F_4$.[24] This may be explained by a more steady cluster growth in the FM phase. Larger clusters give rise to a slower response due to larger free-energy barriers and, correspondingly, a larger number of spins to be simultaneously flipped; the smaller the cluster, the more rapidly it will relax. At $T/T_C=0.2$, on the contrary, the relaxation is slow [Fig. 6(b)] and the aging effect small, because the regular cluster growth with decreasing temperature leads eventually to a blocking of the clusters at T_f and below. It may also be noted in the data that the maximum in $S(t)$ occurs at higher observation times for lower temperatures. This effect is mostly experimental in origin at low t_w (10^2 s), because t_w is of the same order of the time it takes to reach the measuring temperature, which effectively increases the wait time at low temperatures. However, at high t_w (10^4 s), the wait time is much higher than the time for temperature stabilization, and the shift in the maximum of $S(t)$ with temperature may be associated with the slower response time of the clusters at low temperatures. It is worth mentioning that the measuring field of 0.3 mT may not be in the linear regime, which could affect these results. However, larger fields, outside the linear regime, mostly change the intensity but not the position of the maximum in $S(t)$.[9]

IV. CONCLUSIONS

The peak observed in the temperature dependence of the relaxing component of the magnetization, M_r, or equivalently a maximum in the slope of $M(t)/M(0)$ at a given temperature [Fig. 5(b)], combined with the aging effects observed for $La_{0.55}Y_{0.15}Ca_{0.3}MnO_3$, and to a lesser extend for $La_{0.6}Y_{0.10}Ca_{0.3}MnO_3$, evidence the importance of magnetic frustration in these samples and establish the existence of cluster glass properties over a broad temperature range. The enhanced lattice distortion resulting from the presence of cationic size mismatch on the perovskite A site weakens the Mn^{3+}-O-Mn^{4+} FM double-exchange interactions and favors frustration between the ferromagnetic and antiferromagnetic superexchange interactions. This competition is sufficient to suppress long-range FM order, giving rise to the appearance of spatially confined ferromagnetic clusters, which in turn are responsible for the observed glassy behavior of the system due to frustrated interaction among their magnetic moments. It is important to stress that the observed cluster glass behavior occurs within the metallic phase, as opposed to the standard spin-glass phase of the manganites, which occurs in the insulating phase. From the behavior of M_r, it is inferred that clusters start to form just below the FM transition T_C. As the temperature is lowered, the size and/or number of these magnetic clusters increase, leading to a maximum in the relaxing component of the magnetization. The slower (weaker) relaxation at lower temperatures can be attributed to the freezing of the clusters, similar to the blocking of the magnetic moments in a superparamagnet, due to an enhanced intercluster frustration resulting from oversized domains.

The overall relaxation results presented here demonstrate the existence of a magnetic glassy behavior within the metallic phase, which plays an important role in the physics of these compounds. An important issue which remains to be verified is whether the observed cluster glass properties in $La_{0.7-x}Y_xCa_{0.3}MnO_3$ develop uniformly distributed due to disorder or enhanced canting within the FM phase or possibly due to phase-separated spin-glass regions in a FM background. It is also worth noting that M vs H measurements are similar to that obtained in RSG systems, indicating the existence of more complex frustration effects and disorder in this system. Within the phase segregation scenario, it is possible that the FM clusters and the FM background give rise to different glassy behavior.

ACKNOWLEDGMENTS

We thank F. Wolff and P. Pureur for assistance with the SQUID measurements and M.A. Gusmão for helpful discussions. This work was partially financed under PRONEX/FINEP/CNPq Contract No. 41.96.0907.00. Additional support was given by FUJB. The sample preparation was funded by the UK-EPSRC.

[*] Corresponding author. Electronic address: freitas@if.ufrj.br

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