Coexistence of ferromagnetic and glassy behavior in the $La_{0.5}Sr_{0.5}CoO_3$ perovskite compound

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Dc magnetization, ac susceptibility, and zero-field-cooled relaxation measurements are carried out for a cluster glass compound of $La_{0.5}Sr_{0.5}CoO_3$. The temperature dependence of the magnetic properties could be distinguished into two regimes: a high-temperature regime with the time-independent parameters originating probably from the intracluster ferromagnetism, and a lower-temperature regime where the freezing of clusters takes place with a considerabe frequency dependence of a shoulder in $\chi'(T)$ and a hump in $\chi''(T)$. The cusp in $M_{\mathrm{ZFC}}(T)$ at T_a, as well as the high-temperature maximum in $\chi'(T)$ are discussed in terms of an existence of the local anisotropy inside the clusters. The high-temperature maximum in $\chi''(T)$ is interpreted in connection to the reversibility temperature, T_r. Empirically, we found $T_r \propto -H$ while $T_a \propto -H^{0.58}$. We report also the long-time relaxation and the ageing phenomenon in this compound. The ageing effect is much more pronounced in the ferromagnetic state than in the cluster glass state and the system does not reach equilibrium for time scales up to 10^4 s. The relaxation and ageing effects are attributed to the cluster growth slowed down by the presence of the frustration. [S0163-1829(99)00105-8]

I. INTRODUCTION

The substitution of Sr^{2+} for La^{3+} in $La_{1-x}Sr_xCoO_3$ converts an adapted number of the trivalent Co to tetravalent Co introducing a predominantly ferromagnetic (FM) order due to the double-exchange interactions between Co^{4+} and Co^{III} ions.[1–3] On the other hand, the $Co^{3+}-Co^{3+}$ and $Co^{4+}-Co^{4+}$ couplings are antiferromagnetic (AF) due to the superexchange interactions. Frustration may appear in consequence of the coexistence and the competition of AF and FM interactions. Randomness may be achieved by the random occupation of the different valence Co ions in the crystal. These conditions may be sufficient for the system to possess spin-glass-like behavior at low temperature if AF and FM interactions are comparable.

Recently, the magnetic phase diagram of $La_{1-x}Sr_xCoO_3$ ($x \leqslant 0.5$) has been reported,[4,5] in which the system with $x \leqslant 0.18$ was reported as a spin-glass (SG) phase. At $x=0.18$ there is a very sharp transition of the magnetic characterization and the system was identified as ''cluster-glass'' (CG) with $0.18 \leqslant x \leqslant 0.5$. A remarkable feature of the system is that its magnetic behavior changes drastically with x in the SG region while it changes only slightly as x increases from 0.2 to 0.5. From ac susceptibility results, the behavior of $La_{1-x}Sr_xCoO_3$ has been explained by rather complicated interaction processes caused by the transitions of spin states of Co^{3+} ions.[6]

The $La_{0.5}Sr_{0.5}CoO_3$ compound has recently been chosen as a typical CG member of the $La_{1-x}Sr_xCoO_3$ system in several studies.[4,5,7] Itoh *et al.* observed a large difference between zero-field-cooled (ZFC) and field-cooled (FC) magnetization, $M_{\mathrm{ZFC}}(T)$ and $M_{\mathrm{FC}}(T)$ respectively, and a cusp in $M_{\mathrm{ZFC}}(T)$, but could not observe the ageing effect, a signature of a variety of frustrated ferromagnets. Moreover, a comparison of the static and dynamic response of this CG to those of SG, reentrant spin glass (RSG), and canted spin systems by magnetic measurements has been presented.[7] In this paper, we represent the magnetic properties of the $La_{0.5}Sr_{0.5}CoO_3$ compound verified from the magnetic measurements. The physical meanings of the specific temperatures of the system are analyzed and phenomenologically discussed in terms of intracluster ferromagnetism and magnetic glassy behavior of the cluster system. The time-dependent magnetization $m(t)$ and the relaxation rate $S(t)$ at 10 and 180 K, with wait times of $10^2, 10^3, 10^4$ s, are also presented. The results are discussed in the light of the ''droplets'' model[18,19] adopted for the ferromagnetic clusters.

II. EXPERIMENTS

The $La_{0.5}Sr_{0.5}CoO_3$ compound was prepared following the conventional solid-state reaction method. X-ray diffraction shows that the sample is of almost single phase with cubic structure. There is an indistinct peak in the x-ray-diffraction pattern indicating a possible signature of CoO, which is known as an antiferromagnet with $T_N=291$ K.

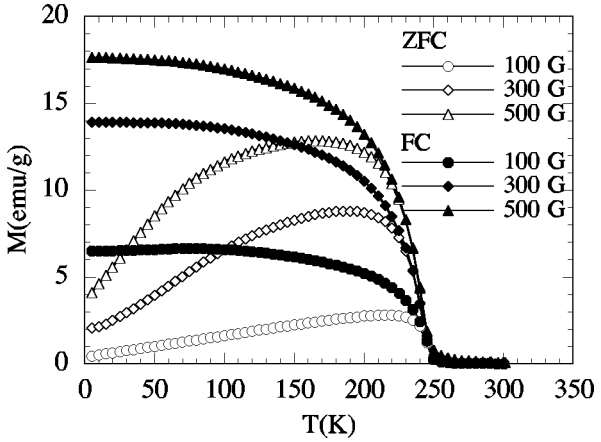

FIG. 1. Typical $M_{\mathrm{ZFC}}(T)$ and $M_{\mathrm{FC}}(T)$ measured at 100 (circles), 300 (diamonds), and 500 G (triangles). The empty symbols are for $M_{\mathrm{ZFC}}(T)$ and the solid symbols for $M_{\mathrm{FC}}(T)$. $M_{\mathrm{ZFC}}(T)$ attains a maximum at temperature denoted by T_a and T_r the temperature where $M_{\mathrm{FC}}(T)$ and $M_{\mathrm{ZFC}}(T)$ start deviating. See text for detail.

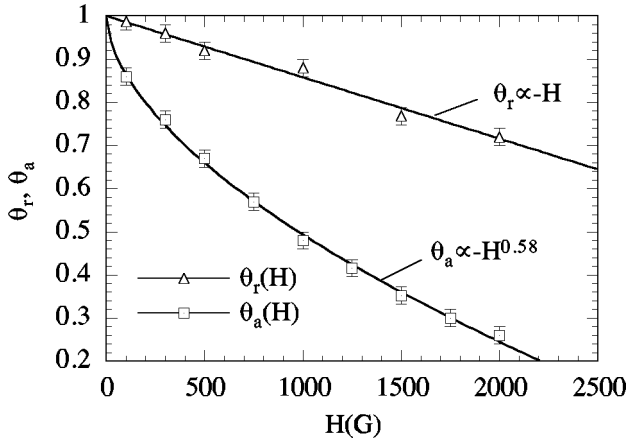

FIG. 2. The field dependences of the reduced temperatures $\theta_r = T_r/T_c$ (triangles) and $\theta_a = T_a/T_c$ (dotted squares). The solid lines are the fitting results showing that $\theta_r \propto -H$ and $\theta_a \propto -H^{0.58}$. Some data points are extracted from Fig. 1

However, our magnetic measurements show no anomaly around 291 K implying an as small as negligible amount of the CoO remnants. The magnetization measurements were performed by a Quantum Design MPMS superconducting quantum interference device (SQUID) magnetometer. In the ZFC measurements the sample was cooled down from room temperature to 5 K in zero field before a measured field was applied. In the FC measurements the sample was cooled in an applied field from room temperature to 5 K. All of the $M_{\mathrm{ZFC}}(T)$ and $M_{\mathrm{FC}}(T)$ data was collected during warming the sample up to 300 K. The temperature-dependent ZFC-susceptibility measurements were carried out in a commercial Lakeshore 7225 susceptometer using the same ac applied field $H_{\mathrm{ac}}^{\mathrm{rms}} = 10$ G for frequencies $\omega/2\pi = 15$, 125, and 1000 Hz. A noncommercial SQUID magnetometer was used for the relaxation measurements. The sample was rapidly cooled in zero field from a reference temperature of 270 K to the measured temperatures T_m, and was kept for different wait times t_w. The relaxation of magnetization $m(t)$ was recorded as a function of the time elapsed after an application of a probing field $h = 0.5$ G.

III. RESULTS AND DISCUSSION

A. dc magnetization

In Fig. 1 some typical $M_{\mathrm{ZFC}}(T)$ and $M_{\mathrm{FC}}(T)$ curves with different applied fields H_{ex} are presented. The paramagnetic (PM)-ferromagnetic phase transition occurs at $T_c \approx 250$ K in accordance with that of $La_{0.5}Sr_{0.5}CoO_3$ reported in several previous studies[4,5,7] and $M_{\mathrm{ZFC}}(T)$ shows a cusp, namely at T_a, where it attains a maximum. $M_{\mathrm{ZFC}}(T)$ and $M_{\mathrm{FC}}(T)$ are substantially identical in a region $T_r \leqslant T \leqslant T_c$ implying a reversibility of magnetization, T_r denotes the temperature where $M_{\mathrm{ZFC}}(T)$ and $M_{\mathrm{FC}}(T)$, with a fixed field, start merging. With higher fields T_a and T_r both shift to lower temperatures and the $M_{\mathrm{ZFC}}(T)$ cusp is broadened. In an attempt to compare to ordinary SG in validity of the De Almeida-Thouless (AT) line,[8] we have plotted the reduced temperatures $\theta_a = T_a/T_c$ and $\theta_r = T_r/T_c$ vs H_{ex} in Fig. 2 and found that these reduced temperatures could scale with H_{ex} as

$$\theta_{a,r} \propto 1 - \frac{C_{\mathrm{AT}}}{T_c} H_{\mathrm{ex}}^{n}. \tag{1}$$

The best fits are obtained with $C_{\mathrm{AT}} \approx 0.36$ and $n \approx 1.0$ for the case of θ_r and $C_{\mathrm{AT}} \approx 2.37$ and $n \approx 0.58$ for the case of θ_a. For SG, where T_c is replaced by the spin-glass transition temperature, mean-field theory predicts $n = \frac{2}{3}$ and this value is firmly endorsed by very many experimental results. However, this is only a necessary (but not a sufficient) feature of a SG transition. In ordinary SG systems, the cusp in $M_{\mathrm{ZFC}}(T)$ is governed by SG dynamic transition and usually observed in the temperature region where the SG transition takes place. We assume that, in $La_{0.5}Sr_{0.5}CoO_3$, the $M_{\mathrm{ZFC}}(T)$ cusp may be governed by a local anisotropy field acting on the spins inside each cluster. Magnetic moments of the spins may be frozen in directions energetically favored by their local anisotropy or by the external field if the system is cooled down from high temperature in a zero or nonzero field, respectively, leading to a difference between $M_{\mathrm{ZFC}}(T)$ and $M_{\mathrm{FC}}(T)$. In this case, probably, there would be a competition between the local anisotropy and the influence of the external field acting on the spin moments. The former is predominant at $T < T_a$ and so is the later at $T > T_a$. We thus suppose that the broad cusp at T_a marks a crossover region where the average anisotropy energy, $E_{\mathrm{a}}(T) = \mathbf{H_a}(T) \wedge \mathbf{M}(T)$, here $\mathbf{H_a}(T)$ and $\mathbf{M}(T)$ are the temperature dependences of the average anisotropy field and magnetization vectors, respectively, and the energy $E_{\mathrm{ex}}(T) = \mathbf{H_{ex}} \wedge \mathbf{M}(T)$ caused by the external field, are comparable. In the reversibility region, $T \geqslant T_r$, the sample would be substantially ''saturated'' in the applied field.

In our measurements, there is still a slight field dependence of magnetization in the reversibility region and the lack of a complete saturation in high fields (up to 5.5 T) indicating an existence of some magnetic moments being misaligned with the applied field. The misaligned moments might either be associated to some related impurities or, as supposed in Ref. 9, be attributed to the moments not belonging to the ferromagnetic clusters and retain their random ori-

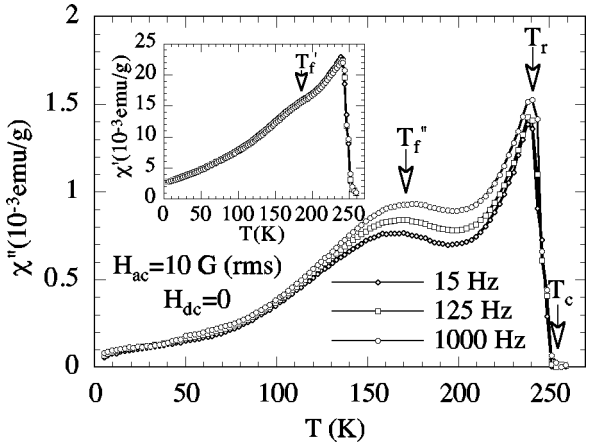

FIG. 3. $\chi''(T)$ measured with ac field $H_{\rm ac}^{\rm rms}=10$ G for $\omega/2\pi=15$ (diamonds), 125 (squares), and 1000 Hz (circles). The inset shows $\chi'(T)$ correspondingly. The arrows mark the specific temperatures. See text for detail.

entations. However, we suggest that this or maybe all the behavior mentioned above are very similar to those of a canted spin system and we should consider seriously the picture of local spin distortions introduced by De Gennes for $La_{1-x}Ca_xMnO_3$,[10] taking into account the possibility of bound states and the self-trapping of Zener electrons.[3] In that picture, there is a nonhomogeneously canted arrangement of spins surrounding the hole impurities (for instance, Sr^{2+} ions in the present case) forming the clusters as a result of the double-exchange couplings.

B. ac susceptibility

ac susceptibility measurements are a compatible technique usually used to search for the magnetic glassy behavior in a material. In Fig. 3, we present the temperature dependence of the ZFC out-of-phase susceptibility $\chi''(T)$, the inset shows the corresponding in-phase $\chi'(T)$. At about 250 K, both the susceptibilities abruptly and rapidly decrease to zero, exhibiting the FM-PM phase transition. Despite the time-dependent dynamics due to the blocking process of the clusters, the temperature dependence of $\chi'(T)$ including the maximum at 240 K may be qualitatively explained similarly as we have dealt with $M_{\rm ZFC}(T)$ implying a resemblance in physical meaning of this maximum to the cusp observed in the dc $M_{\rm ZFC}(T)$ curves. This maximum is frequency independent and being positioned by the ac field amplitude. On the other hand, as can be seen in Fig. 3, there also exists a maximum in $\chi''(T)$ at 240 K independent of frequency. Thus the two maxima in $\chi'(T)$ and $\chi''(T)$ seemingly appear at the same temperature. However, we suppose that they may be not associated and might be differently originated. It is well known that $\chi''(T)$ reflects the magnetic energy dissipation in the sample and, naturally in this case, $\chi''(T)$ is proportional to the area of the hysteresis loop within one period of the ac field at an equilibrium temperature. The high-temperature maximum in $\chi''(T)$ is therefore assumed to appear at the temperature where the sample is substantially saturated when the field has just reached to the peak value of the ac amplitude and the hysteresis loop area has therefore an extreme value. Further increasing temperature will reduce not only the coercive force but also the saturated magnetization leading to an abrupt decrease of $\chi''(T)$ towards zero. So, it seems that the temperature position of this maximum is corresponding to T_r observed in the dc magnetization measurements. Because there is no hysteresis of magnetization in the PM phase, T_c should be determined as the position where $\chi''(T)$ is essentially equal to zero as indicated in Fig. 3.

We want to emphasize that because of the small differences between T_a, T_r, and T_c, especially in low fields which are usually applied for ac susceptibility measurements, one might therefore easily be confused and determine mistakenly the physical meanings of these temperature positions. Our arguments for T_a and T_r are also in agreement with the results reported by Mukherjee *et al.*,[7] who found that the high-temperature maxima in the susceptibilities shift to lower temperature with an increase of ac field amplitude whereas no frequency dependence of these maxima is observed. Furthermore, also reported in Ref. 7, there is no difference between ZFC and FC susceptibilities above these maxima. The frequency independence of T_a and T_r convincingly indicate that the high-temperature maxima in $\chi'(T)$ and $\chi''(T)$, together with the cusp in $M_{\rm ZFC}(T)$ are entirely associated with the FM state originated from the intracluster ferromagnetism and, unlike canonical SG systems, not associated to the freezing of the magnetic moments. This behavior of $La_{0.5}Sr_{0.5}CoO_3$ is somehow similar to that of a reentrant spin glass, $(Fe_{0.2}Ni_{0.8})_{75}P_{16}B_6Al_3$, where in the high-temperature FM phase, there exists in susceptibilities a field-dependent cusp governed by coercive force H_c.[11]

With lowering temperature, the cluster glassy behavior is exposed by a shoulder in $\chi'(T)$ at T_f' and a hump in $\chi''(T)$ at T_f'' as marked by the arrows in Fig. 3 (see Fig. 3 and the inset therein). T_f' and T_f'' are frequency dependent and considerably shift to lower temperatures with lowering frequency. The exact position of the shoulder in $\chi'(T)$, T_f', as well as its frequency dependence is rather difficult to determine. The hump in $\chi''(T)$ is much clearer and is determined as $T_f''\approx 164.5$, 167.5, and 175 K for $\omega/2\pi=15$, 125, and 1000 Hz, respectively. An attempt at fitting $T_f''(\omega/2\pi)$ data to conventional slowing down,

$$\frac{\tau}{\tau_o}\propto\left(\frac{T_f''-T_f}{T_f}\right)^{-z\nu} \tag{2}$$

gives $T_f=162.6$ K, while $T_f=157.7$ K is obtained by fitting to the Volgel-Fulcher scaling law,

$$\frac{\tau}{\tau_o}\propto e^{E_a/k(T_f''-T_f)}. \tag{3}$$

These values are all relatively higher than those reported in Ref. 7 leading to suggestion that the average size as well as the distribution of the clusters may vary in samples prepared by different methods. In fact, a drastic change of the magnetic and electrical properties of the samples prepared by different synthesis techniques has recently been reported.[12] One may therefore connect the glassy behavior of $La_{0.5}Sr_{0.5}CoO_3$ to the blocking of superparamagnetic single-domain particles, where the freezing process is resulted from a distribution of particle sizes and therefore relaxation times.[13] If we consider the system as an assembly of clusters,

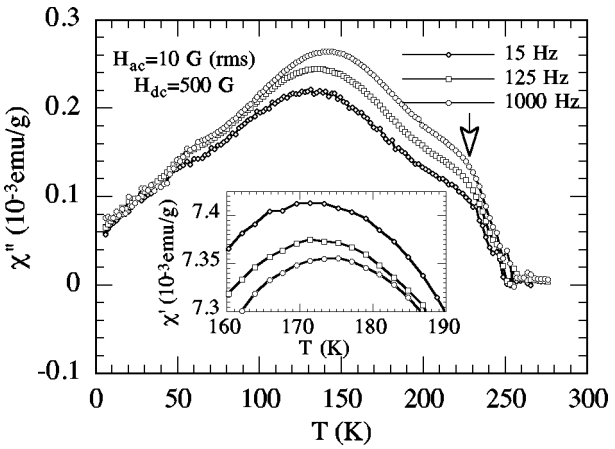

FIG. 4. $\chi''(T)$ measured with $H_{ac}^{rms}=10$ G for $\omega/2\pi=15$ (diamonds), 125 (squares), and 1000 Hz (circles) in a superimposed dc field of 500 G. The inset shows an enlargement of the corresponding $\chi'(T)$ around the freezing temperature. The arrow marks the singular point around 230 K in consistence to T_r with $H_{ex}=500$ G shown in Fig. 1.

for a certain field and frequency, with decreasing temperature, the larger size clusters will be frozen at higher temperature when their relaxation time τ becomes longer than the characteristic time of the measurement, $\tau_m=2\pi/\omega$. We suppose that, being strikingly different (and additional) to the conventional superparamagnetic single-domain particles, the clusters in $La_{0.5}Sr_{0.5}CoO_3$ would also change their sizes with temperature, and together with the presence of frustration, leading to a long-time relaxation and an age-dependent effect, which we discuss in Sec. III C. Another characteristic feature is that the intercluster couplings in this system are not negligible and hence their effects may be inevitable.

Figure 4 presents $\chi''(T)$ measured in a superposed dc field of 500 G. The susceptibility is drastically suppressed by the biasing field. There is a notable abrupt decrease in $\chi''(T)$ towards zero at around 230 K (marked by an arrow). This anomaly seems frequency independent in position and probably is according to the high-temperature maximum in $\chi''(T)$ measured with the zero-biasing field plotted in Fig. 3. Interestingly, the temperature value of this anomaly is quite consistent with the value of T_r for the case of 500 G dc applied field (Fig. 1). Moreover, the application of the superposed dc field would tend to order the spins in the field direction and therefore hinder the randomly freezing process of the cluster moments causing a drastic shift of T_f'' to lower temperature. Another remarkable feature is that $\chi''(T)$ seemingly tends to be frequency independent at low temperature for both cases: with and without a superimposed field (Figs. 4 and 3) indicating a rather slow response of the system to the probing field with the measured frequencies.

C. Relaxation and ageing effect

In contrast with the results previously reported in Ref. 4, for $La_{0.5}Sr_{0.5}CoO_3$, we have observed the long-time relaxation of magnetization and the ageing effect with time scale as long as 10^4 s. The ageing effect is clearly seen not only at temperature below but also well above the freezing temperature of the cluster moments. Figures 5(a) and 5(b) present the

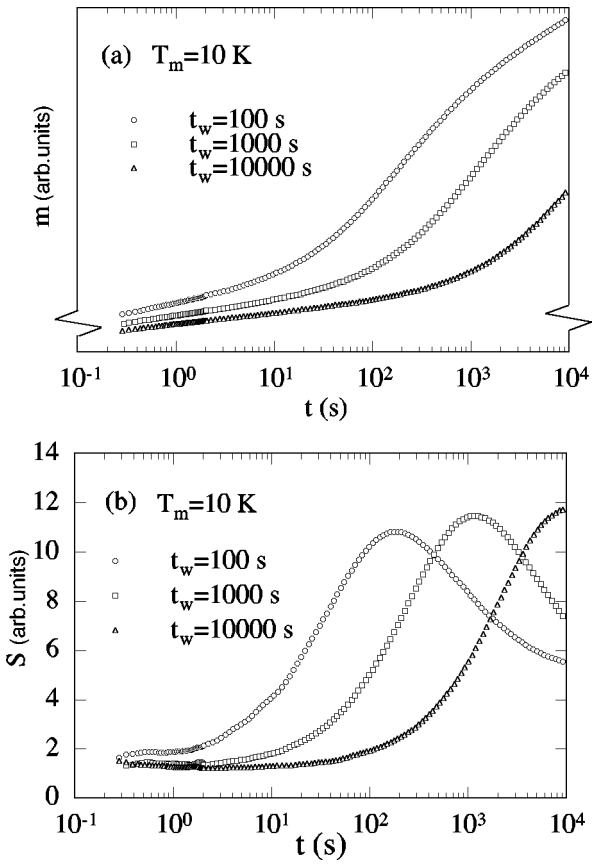

FIG. 5. ZFC relaxation magnetization $m(t)$ (a) and corresponding relaxation rate $S(t)$ (b) measured at $T_m=10$ K with wait time $t_w=100$ (circles), 1000 (squares), and 10 000 s (triangles) using probing field $h=0.5$ G.

time-dependent relaxation of the ZFC magnetization $m(t)$ measured at $T_m=10$ K with $t_w=10^2,10^3,10^4$ s and the corresponding relaxation rates

$$S(t)=\frac{1}{h}\frac{\partial m(t)}{\partial \ln t}. \tag{4}$$

The similarly measured procedure is also applied for the relaxation measurements at $T_m=180$ K and the results are presented in Figs. 6(a) and 6(b). It is clear that the relaxation rate, at both measured temperatures and with all the wait times, attains a maximum at the elapsed time very close to the wait time indicating convincingly an age-dependent effect, which is often observed in SG, and recently, even in the ferromagnetic phase of a reentrant spin glass[11,14,15] and the two-dimensional random-exchange Ising ferromagnet $Rb_2Cu_{0.89}Co_{0.11}F_4$.[16,17] Furthermore, those reports show that the relaxation rate in the FM phase is much more pronounced than that in the SG phase. In comparison, a similarity of the ageing effect is found for $La_{0.5}Sr_{0.5}CoO_3$, as one can compare in Figs. 5(b) and 6(b), that the relaxation at 180 K is much faster and even stronger than that at 10 K. Using the phenomenological domain growth theory for interpreting the ageing effect in $Rb_2Cu_{0.89}Co_{0.11}F_4$, the authors in Ref. 17 have attributed the faster relaxation in the FM phase to the more steady domain growth.

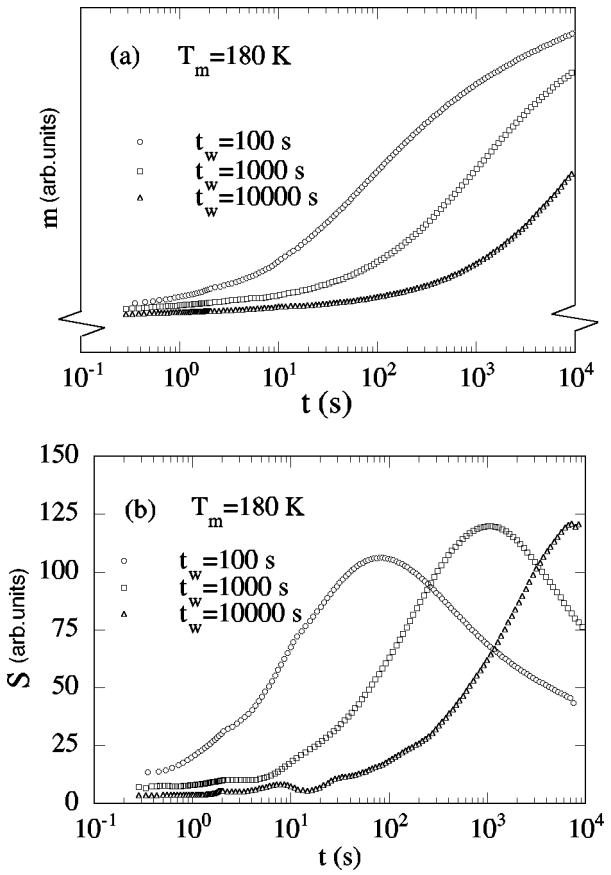

FIG. 6. ZFC $m(t)$ measured at $T_m = 180$ K (a) and corresponding relaxation rate $S(t)$ (b) with $t_w = 100$ (circles), 1000 (squares), and 10 000 s (triangles) using the probing field of 0.5 G.

The long-time relaxation observed for $La_{0.5}Sr_{0.5}CoO_3$ evidences an important role of the frustration, especially at 180 K, it is well above T_f where the system is in FM state. The cluster formation and the competing coexistence of the $Co^{4+}-Co^{III}$ double-exchange interaction and the superexchange interaction of $Co^{3+}-Co^{3+}$ and $Co^{4+}-Co^{4+}$ probably set in simultaneously. Thus the frustration may exist in the FM region even at temperatures close to T_c so that the whole system is not in a true ferromagnetic state. The long-time relaxation and ageing effect at both temperatures lower and higher than the freezing temperature of clusters convince us that this is an inherent property of the studied system. We suppose that double-exchange couplings are entirely dominant inside each cluster and the frustration only appears in a ''transition region'' between the cluster and the intermedia. At high temperature, since the clusters relax on very short-time scales then the long-time relaxation of the system is expected to be mostly contributed from a layer near the surface of each cluster where frustration is supposed to be strong. Thus the ageing effect can be regarded as the evolution of the clusters and frustration plays the part of pinning centers slowing down the evolvement. The slower and weaker relaxation at 10 K should be attributed to the stronger frustration. We also suggest that the cluster growth at high temperature could be probably responsible for the blocking at T_f.

It is also important to note that there certainly exists a limiting field for the visibility of the ageing effect.[14] Itoh *et al.* have used a probing field of 100 G and the ageing signal could not be detected.[4] We suppose that such a high field might be enough to suppress entirely the effect.

IV. CONCLUSION

Magnetic properties of $La_{0.5}Sr_{0.5}CoO_3$ have been represented. In the temperature region close to T_c the magnetic behavior probably reflect the predominant intracluster ferromagnetism, however, because of the presence of the frustration, this is not a true FM state. The cusp in $M_{ZFC}(T)$ at T_a as well as the high-temperature maximum in $\chi'(T)$ are probably governed by a local anisotropy (or intracluster anisotropy) field acting on spins inside each cluster changing their arrangement with temperature. The specific temperatures T_a and T_r observed in dc measurements could be retrieved in $\chi'(T)$ and $\chi''(T)$, respectively. T_r is found to be proportional to $-H$, while $T_a(H)$ follows closely the AT line with $n = 0.58$. The glassy behavior is signaled with lowering temperature by a frequency-dependent shoulder in $\chi'(T)$ and a hump in $\chi''(T)$, which shifts to higher temperatures with higher measuring frequency, indicating a process of the clusters freezing. The long-time relaxation and ageing effect observed at both temperatures lower and higher than the freezing temperature indicate an important role of the frustration and could be interpreted by adopting the ideas of the growing domains model.[18,19] The more pronounced aging effect at high temperature is explained by the dimer frustration on the surface of the clusters. The cluster growth with lowering temperature may be responsible for the blocking of the clusters at T_f. The magnetic properties of $La_{0.5}Sr_{0.5}CoO_3$ are quite similar to those of RSG and the obtained results also indicate clearly a coexistence of ferromagnetic and magnetic glassy behavior.

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