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Non-linear susceptibility studies of $La_{0.85}Ca_{0.15}Mn_{0.95}Fe_{0.05}O_3$

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Abstract

Linear and non-linear dynamic properties of a re-entrant ferromagnetic polycrystalline bulk $La_{0.85}Ca_{0.15}Mn_{0.95}Fe_{0.05}O_3$ sample are studied using AC susceptibility. This sample composition shows a transition from paramagnetic (PM) to ferromagnetic (FM) at around 174 K and to spin glass (SG) at ∼100 K. The dynamic behaviour is investigated in the intermediate temperature range ($T_g < T < T_c$). A prominent non-linear susceptibility is observed in this range, signifying the decomposition of the FM network into clusters by a random magnetic field which is expected to be generated by PM spins in the FM region. The results are supported by a study of the AC frequency and DC field effect on the non-linear susceptibility.

1. Introduction

The colossal magnetoresistance compounds (CMR) are transition metal oxides. These compounds have perovskite structure and are represented as $R_{1-X}A_XMnO_3$, where R is a trivalent rare earth metal and A is a divalent alkaline earth metal. These are highly correlated systems with a subtle balance of spin, charge, orbital and lattice degrees of freedom, which leads to a complex phase diagram and to the coexistence of various forms of ordering [1, 2]. A profound knowledge of these ordering processes and their mutual interactions is essential for a better understanding of CMR. For example, the simultaneous observation of charge and orbital ordering in $Pr_{1-X}Ca_XMnO_3$ has revealed that the charge ordering couples to the magnetic degree of freedom and drives the orbital ordering [3].

Enormous interest in these compounds stems from the variety of phases with changing doping level X. These phases include paramagnetic (PM) to antiferromagnetic (AFM) for $0.5 < X < 1$, paramagnetic (PM) to ferromagnetic (FM) for $0.2 < X < 0.5$ and in some compounds it is reported that at low temperature, the re-entrant spin glass (RSG)

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transition followed by spin canting state occurs. The magnetic behaviour in these compounds is understood on the basis of the double-exchange theory proposed by Zener [4].

Many studies have been done through doping of La sites, which results in lattice defects and ultimately influences the double exchange. However, far fewer studies have been conducted on doping of Mn sites, which are at the heart of double exchange. Dho *et al* [5] have studied manganites with Cr doping at Mn sites, namely $La_{0.46}Sr_{0.54}Mn_{1-y}Cr_yO_3$, and observed multiple magnetic transitions, PM $\rightarrow$ FM $\rightarrow$ AFM and to a re-entrant spin glass for $y = 0.02$. Yang *et al* [6] and Ahn *et al* [7] have shown that amongst various doping elements (Ni, Cr, Fe, etc), Fe has the most devastating effect on T_c; hence it is worthwhile to investigate the influence of doping by other elements at Mn sites. Doping at Mn sites, in general, may cause (i) strong lattice distortions, depending upon the size mismatch between Mn and the doping ions, (ii) magnetic dilution caused by doping with non-magnetic ions like Al^{3+}, In^{3+}, etc and (iii) additional magnetic coupling by doping with magnetic ions like Cr, Co, Ni, etc. These effects may be interrelated and all of them can affect the magnetic behaviour.

In this work we have studied Mn site substitution with $y = 0.05$ in $La_{0.85}Ca_{0.15}Mn_{1-y}Fe_yO_3$. In this composition, competing FM and AFM interactions are present. Recently, a spin glass like transition at around $T \sim 100$ K has been reported in $La_{0.85}Ca_{0.15}Mn_{0.95}Fe_{0.05}O_3$ with a FM transition at $T \sim 175$ K [8]. In general the RSG transition is a topic of much controversy. It has been argued that the spin glass features start just after the FM transitions [9], whereas other reports [10] stress that the FM transition is followed by a spin-canted state that later shows a spin glass type transition. Furthermore, there is an ongoing debate on whether the spin glass transition temperature (T_g) represents a true phase transition in the thermodynamic sense or is just a dynamical freezing temperature [5, 11].

In the canonical spin glass phase, disordering takes place and any long range spin order is completely destroyed, i.e., there is no residual magnetization. However RSG, in contrast undergoes successive transitions; one is between the PM phase and long range ordered phase (either FM or AFM phase) at high temperature, and the other is between the ordered phase and the RSG phase at low temperature. Takayama [12] has calculated explicitly the linear and non-linear magnetic susceptibility of mean field Ising models for the RSG system using Parisi's replica symmetry breaking (RSB) and predicted that both the FM and AFM systems undergo transitions between the replica symmetry (RS) phase and the RSB phase at the transition temperature (T_g), while maintaining finite spontaneous magnetization. The intermediate temperature region between the two phase transitions is not well understood and there are very few experimental studies exploring the dynamics of the system in this region. The intermediate temperature region between the FM and SG transitions is thus worth investigating to clarify these conjectures. It is the main objective of this work to investigate the low temperature phase transitions in $La_{0.85}Ca_{0.15}Mn_{0.95}Fe_{0.05}O_3$ compounds.

2. Experiment

The samples under investigation were prepared by a standard solid state method, and the details are as previously described [13]. The x-ray diffraction pattern indicated that both the parent and the Fe doped bulk polycrystalline samples have cubic perovskite structure without any other secondary or impurity phases, with almost the same lattice parameter. The resistivity of the sample was measured with the conventional four-probe method in the temperature range of 4–300 K. AC susceptibility measurements were performed by using a home made susceptometer in the temperature range of 77–300 K. Second- and third-harmonic non-linear susceptibilities were measured using the digital DSP Lock-in Amplifier of Stanford Research Systems, model SR830. In our previous studies, we have demonstrated that the parent compound

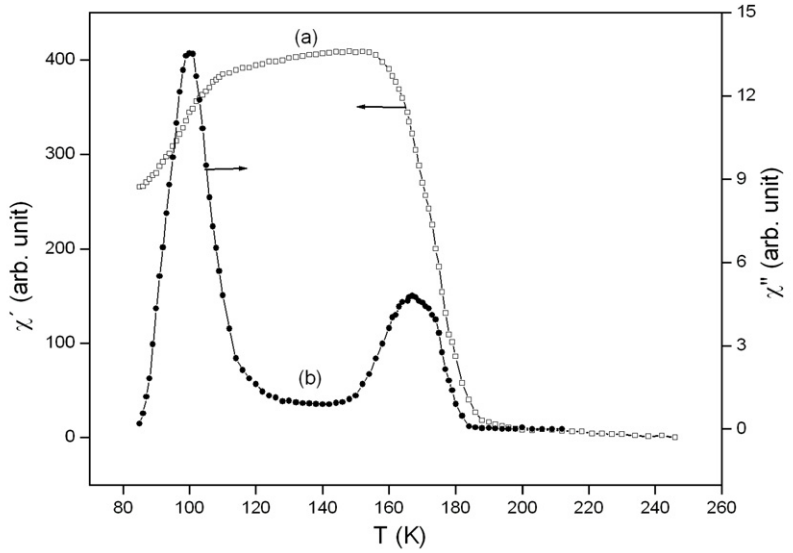

Figure 1. The temperature dependences of the AC susceptibility for $La_{0.85}Ca_{0.15}Mn_{0.95}Fe_{0.05}O_3$ at a frequency of 573.3 Hz and $h_{ac} = 2$ Oe: (a) real part of the susceptibility (χ'); (b) imaginary part of the susceptibility (χ'').

$La_{0.85}Ca_{0.15}MnO_3$ shows a PM to FM transition at a critical temperature of $T_c = 205$ K and its resistivity data in the temperature range 77–300 K show an insulator to metallic transition at ∼208 K which almost coincides with the PM–FM transition temperature [8].

3. Results and discussion

Typical data for the Fe doped composition of the in phase and out of phase parts of the AC susceptibility χ' and χ'' are shown in figure 1. The probing field h_{ac} in this case was 2 Oe at frequency $f = 573.3$ Hz. From this plot it is evident that the system undergoes a PM to FM transition in the temperature range 190–160 K. The approximate PM to FM Curie transition temperature (T_c) thus obtained is 174 K. It is evident in figure 1 that the maximal plateau of χ' occurs at 142 K and then there is a slight decrease in χ' as we further decrease the temperature. Finally there is a sharp decline initiating at temperature ∼100 K.

The out of phase part χ'' in figure 1, shows a peak at 171 K that indicates a PM to FM transition. On further decreasing the temperature a second peak is observed at ∼100 K, in harmony with the sharp decline of χ' around the same temperature region. The second peak is much stronger than the PM to FM transition peak. It is also noticeable that the out of phase part is non-zero in a FM region, between 160 and 125 K.

The DC resistive behaviour of this sample is shown in figures 2(a), (b); unlike the parent compound, this sample exhibits insulating behaviour down to the lowest temperature ∼20 K and there is no metal–insulator transition observed for this composition over the entire range of temperature. However, there is a very sharp increase in resistivity below $T \sim 150$ K, indicative of some very substantial scattering phenomena. The logarithmic behaviour of this resistivity is shown in figure 2(b); the slope above 150 K is quite constant, while a clear change in slope is evident below 150 K. It has been suggested that Fe^{3+} ions replace Mn^{3+} because of the ionic sizes being the same, i.e., 0.70 Å [14]. Doping with Fe^{3+} causes depletion of the Mn^{3+}/Mn^{4+}

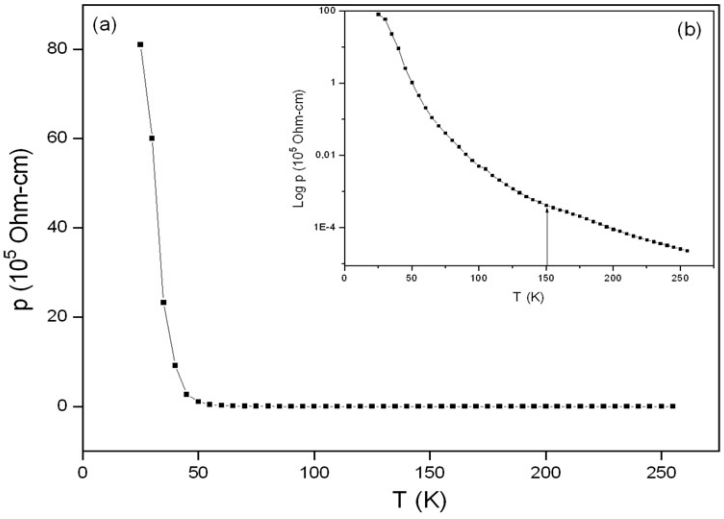

Figure 2. Main figure: temperature dependence of the resistivity for the $La_{0.85}Ca_{0.15}Mn_{0.95}Fe_{0.05}O_3$ composition. Inset: resistivity as in the main figure shown on a log scale. The arrow indicates the temperature ($T \sim 150$ K) where the temperature dependence of the resistivity appears to change significantly.

ratio, the population of the hopping electrons, and the number of available hopping sites. Thus, double exchange is suppressed, resulting in the reduction of the ferromagnetism and metallic conduction [7]. Along with this change, the χ' and χ'' also show different behaviour (vide infra) in the low temperature region (figure 1). We may argue that this change originates from the spin blocking or freezing effects, which shows typical signatures of RSG (it should be mentioned here that these signatures are frequency and field dependent (v, H_{dc}, h_{ac})). In our previous work [8] we have conducted series of experiments to confirm these signatures.

The FM and SG transitions have been studied extensively in the past [15, 16]; however, less attention has been given to the intermediate temperature region. It has been argued that the long range order of the ferromagnetism is perturbed long before the re-entrant spin glass transition occurs. However, no evidence of any phase transition is found in the AC susceptibility of Fe doped compounds.

To investigate this further, we have performed non-linear susceptibility studies on our system. The study of the non-linear susceptibility is an important experimental tool in identifying the SG and RSG behaviour. The χ_2 and χ_3 measurements can reveal that the system shows FM–SG behaviour. In the presence of a magnetic field the magnetization of a FM material can be expressed as

$$m = m_0 + \chi_1 h + \chi_2 h^2 + \chi_3 h^3 + \cdots \tag{1}$$

where m_0 is the spontaneous magnetization, χ_1 is the linear susceptibility, χ_2 and χ_3 are the non-linear susceptibilities. But for a direct PM–SG transition the magnetization m is expressed as an odd power series [17] as

$$m = \chi_1 h + \chi_3 h^3 + \chi_5 h^5 + \cdots. \tag{2}$$

It has been shown both theoretically and experimentally that at least in some systems the non-linear term χ_3 diverges at transition temperature characterizing the SG transition [18, 19]. Dub *et al* [20] have shown the non-linear (χ_2, χ_3) data and found that in the RSG or mixed

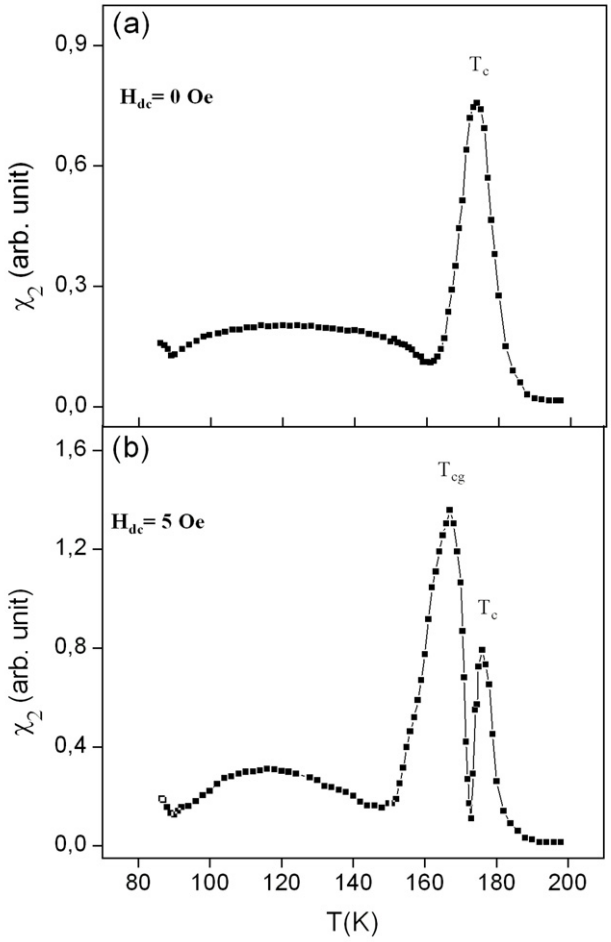

Figure 3. Temperature dependence of the non-linear harmonic susceptibility χ_2 for the $La_{0.85}Ca_{0.15}Mn_{0.95}Fe_{0.05}O_3$ composition at a frequency of 573 Hz and $h_{ac} = 9$ Oe showing the DC field effect: (a) $H_{dc} = 0$ Oe; (b) $H_{dc} = 5$ Oe.

state, where FM and SG state coexist, the χ_2 term would appear at and around SG transition temperature. In this case χ_3 is expected to show non-diverging behaviour, which is also an indication of the RSG or cluster glass (CG) state [21]. Omerzu *et al* [22] also argued that in the case of strictly zero external field, the even power in (2) should be zero. But if not precisely compensated, a static field of typically 0.5 Oe is present, which breaks the symmetry and makes the even power appear.

The measurements of the harmonics were done in the same manner as for the fundamental susceptibility except that the lock-in amplifier was set to either the second-or third-harmonic mode as required. The sample was cooled in zero magnetic field and the non-linear component (χ_2) of the AC susceptibility was logged; the probing field h_{ac} in this case was 9 Oe having frequency $f = 573.3$ Hz, as shown in figure 3(a). A peak at around 174 K is observed signifying a PM to FM transition; after the initial peak, a small but broad maxima or plateau is observed. Note that, at low temperature where the SG like transition was found to be completed, χ_2 is decreased.

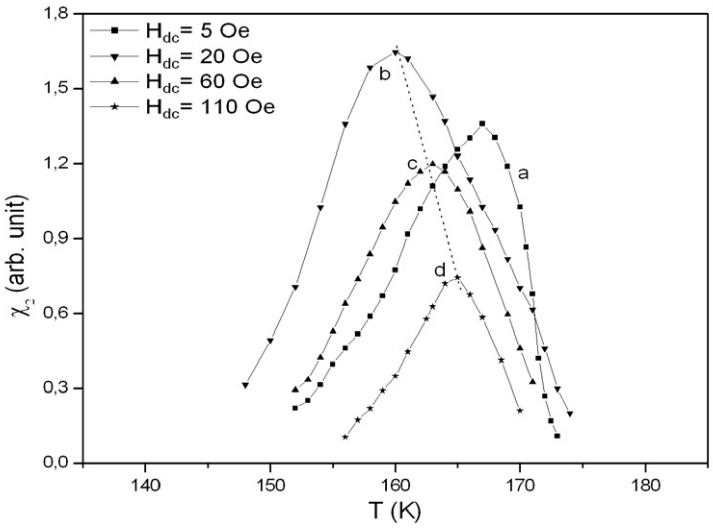

Figure 4. Temperature dependence of the non-linear harmonic susceptibility χ_2 for the $La_{0.85}Ca_{0.15}Mn_{0.95}Fe_{0.05}O_3$ composition at a frequency of 573 Hz and $h_{ac} = 9$ Oe showing the shifts in peak position (T_{cg}) caused by applying different DC fields: (a) $H_{dc} = 5$ Oe; (b) $H_{dc} = 20$ Oe; (c) $H_{dc} = 60$ Oe; (d) $H_{dc} = 110$ Oe.

We performed a similar experiment applying a DC field. In this case the sample was zero-field cooled and then a DC field with range from $0 < H_{dc} < 110$ Oe perpendicular to the AC field was applied, and the χ_2 was observed while warming the sample. Typical data for χ_2 are plotted in figure 3(b), for $H_{dc} = 5$ Oe, with the same conditions as above (figure 3(a)). It is evident from the figure that the zero-field peak (PM $\rightarrow$ FM) is split into two separate peaks by applying a DC field; one appeared at the FM transition ($T_c = 176$ K) while a second peak occurred at slightly lower temperature ($T = 167$ K).

More detailed plots of the DC field effects on the low temperature peak are shown in figure 4, covering the range $5 < H_{dc} < 110$ Oe. We note that this peak ($T = 167$ K) shows a non-monotonic temperature dependence. We represent this peak in χ_2 as T_{cg} (for reasons to be illustrated later), and observed that for small DC fields, T_{cg} remains constant. For $H_{dc} > 20$ Oe this peak shifts to lower temperature (for $H_{dc} = 5$ Oe, $T_{cg} = 166.5$ K, while at $H_{dc} = 110$ Oe, T_{cg} shifts to 164.5 K).

We further determined the frequency dependence of χ_2 (133 Hz $< f <$ 3.533 kHz). After zero-field cooling, the data for representative frequencies at $h_{ac} = 9$ Oe and $H_{dc} = 40$ Oe were plotted; see figure 5. There is no frequency dependence at the FM transition temperature (T_c). However the peak position T_{cg} was found to be frequency dependent and shifted to higher temperature with increase of the frequency, i.e., it went from 161 K at $f = 133.33$ Hz to 164 K at $f = 3533$ Hz.

To decipher the nature of the intermediate phase and to establish whether the SG phase or RSG phase exists in our system, we studied the third harmonic of the AC susceptibility (χ_3). During these investigations the sample was zero-field cooled, and the data were taken while warming the sample, as shown in figure 6. It is worth mentioning here that χ_3 is expected to be non-zero whenever the AC response is non-linear. It is evident from the figure that χ_3 shows two peaks, one at the PM to FM transition and a second peak at the RSG transition; however the important point is that χ_3 remains non-zero even at intermediate temperature,

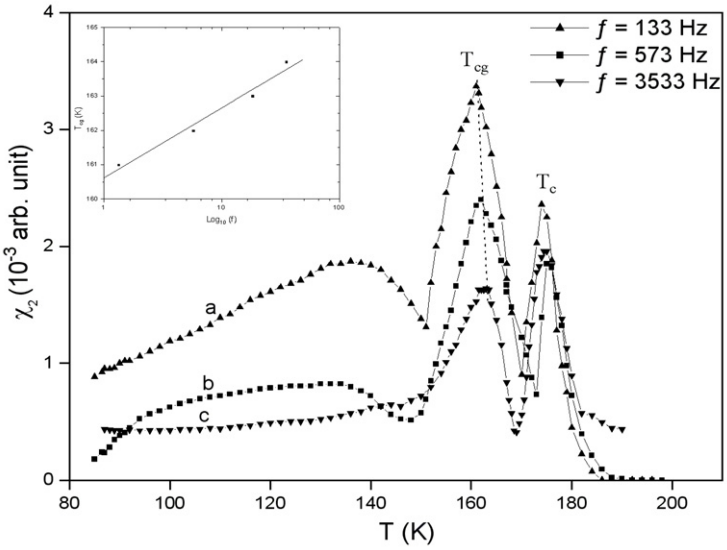

Figure 5. Temperature dependence of the non-linear harmonic susceptibility χ_2 for the $La_{0.85}Ca_{0.15}Mn_{0.95}Fe_{0.05}O_3$ composition at different frequencies with $h_{ac} = 9$ Oe and $H_{dc} = 40$ Oe representing the shifts in peak position and peak intensity obtained by increasing the frequency: (a) $f = 133$ Hz; (b) $f = 573$ Hz; (c) $f = 3533$ Hz. The inset shows the variation of the T_{cg} peak temperature with different frequencies in the range 133–3533 Hz.

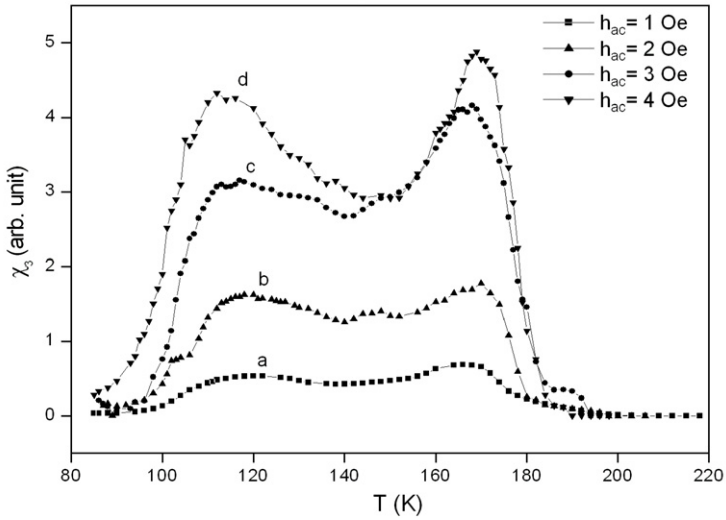

Figure 6. Temperature dependence of the non-linear harmonic susceptibility χ_3 for the $La_{0.85}Ca_{0.15}Mn_{0.95}Fe_{0.05}O_3$ composition at a frequency of 573 Hz indicating the effect of different AC fields on both the peak temperatures (T_c and T_{rsg}): (a) $h_{ac} = 1$ Oe; (b) $h_{ac} = 2$ Oe; (c) $h_{ac} = 3$ Oe; (d) $h_{ac} = 4$ Oe.

indicating the presence of a FM region in this range. We also observed the effect of different AC fields and noted that the peak positioned at ∼171 K is found to be independent of the AC field but the second peak positioned at ∼110 K is sensitive to the AC field. This second

peak shifts to low temperature on increasing the AC field. Similar results have been reported previously [17, 20, 23].

In order to interpret our results, we start by summarizing the basic understanding of long range order, specifically the FM long range order, and frustration effects leading to the SG state in CMR and related compounds. To account for our results we first illustrate the role of Fe substitution in these compounds. It is well known that by Fe doping one changes the Mn^{3+}/Mn^{4+} ratio, thus producing superexchange interaction and hence promoting AFM interaction in addition to FM double-exchange interaction. In a system with FM double-exchange interaction dominating, the AFM (superexchange) interaction may cause magnetic disorder and frustration. Thus the system in this case is expected to show a low temperature RSG phase [5, 24–28].

The nature of RSG and FM phases in re-entrant ferromagnets is basically understood in terms of a mean field picture. However, in the mean field picture, a true re-entrance from the FM phase to the normal SG phase is not predicted. To substantiate our results we make use of a phenomenological model proposed by Aeppli *et al* [29] and Suzuki *et al* [30] where a phenomenological random field picture is used to explain neutron scattering experiments. The model is based on decomposition via the frustration mechanism of spin system with exchange interactions of random sign into SG like and FM networks. In this picture the system in the FM phase consists of regions which would order ferromagnetically, and other regions forming PM clusters. Actually the phase separation effect is responsible for the creation of small hole-rich FM particles in the PM region at T well above T_c [31, 32]. The frustrated spins in the PM clusters can generate random molecular fields, which act on the unfrustrated spins in the infinite FM network. In the FM phase well above T_{rsg}, the fluctuations of the spins in PM clusters are so rapid that the FM network is less influenced by them and their effect is only to reduce the net FM moment. On decreasing the temperature toward T_{rsg}, the thermal fluctuations of the spin in the PM clusters become slower [33]. The coupling between the PM clusters and the FM network becomes important and the molecular field from the slow PM spins acts as a random magnetic field. This causes break-up of the FM network into finite sized clusters.

We assume that the perturbation of the long range FM order due to frustrated spins starts as soon as the PM to FM transition has taken place. Thus, decomposing the FM network into clusters, these clusters get smaller and smaller as the temperature is decreased, as is clear in figure 1, where a continuous decline in χ' with temperature occurs. Moreover the rounded maximum in χ' (figure 1) indicates the formation of finite clusters, compared to the much sharper one in the case of long range FM ordering [34]. We also stress that the intermediate phase is not a result of a true phase transition in a thermodynamic sense, and is therefore difficult to observe in a usual out of phase part of the AC susceptibility.

As we discussed earlier (figures 3(a), (b)), the χ_2 peak splits into two peaks, separated by $\Delta T \sim 10$ K. The second peak that we referred to as T_{cg} substantiates the fact that FM clusters respond to the magnetic field in unison to provide a net magnetization [35]. Although the effect of the magnetic field on the T_{cg} position may be non-monotonic, which is still not established (figure 4), one can argue for the formation of clusters at higher temperature with increasing DC field, which is understandable as the DC field helps in stabilizing the long range order [36]. The frequency dependence of the peak (T_{cg}) in χ_2 is also consistent with either a cluster glass or a FM cluster system, but it can be quantified through the frequency dependence of the peak temperature as given by the peak position (T_{cg}). As shown in figure 5 (inset), we find that T_{cg} is linear in the logarithm of the frequency with a normalized slope $p = \Delta T_{cg}/(T_{cg}\Delta \log f)$. We find $p = 0.013$, which is not only lower than typical values for canonical spin glass systems but also different from those reported for FM clusters with FM insulating states [37]. The shift in peak temperature and peak magnitude obtained by increasing the frequency is qualitatively

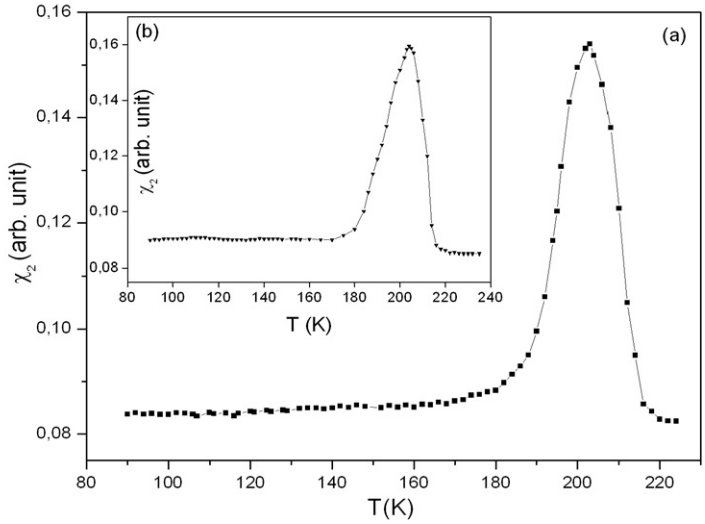

Figure 7. Temperature dependence of the non-linear harmonic susceptibility χ_2 for the $La_{0.85}Ca_{0.15}MnO_3$ composition at a frequency of 573 Hz and $h_{ac} = 9$ Oe showing that there is no splitting of T_c obtained by applying a DC field: (a) $H_{dc} = 0$ Oe; (b) $H_{dc} = 20$ Oe.

different from the behaviour of most spin glasses, in which we expect an increase of the peak magnitude as frequency is increased [38]; our data thus appear to support the existence of ferromagnetic clusters. A non-zero signal for χ_3 and χ'' in the temperature range 160–125 K may also support the above results. The average cluster size cannot be estimated accurately as the size varies with the magnitude of the applied magnetic field. However, a very rough estimate of 50 Å was obtained just below T_c [31, 39]. χ_3 exhibits a non-diverging broad peak at ∼110 K, while χ'' also shows a peak at around the same temperature; thus our non-linear data appear to exclude the existence of a true SG phase and instead indicate that a RSG phase occurs in $La_{0.85}Ca_{0.15}Mn_{0.95}Fe_{0.05}O_3$ [21, 34]. The effect of an AC field (figure 6) on the low temperature peak in χ_3 is also in agreement with this result.

In order to authenticate the above conjecture as not due to some spurious effects, we have conducted similar experiments on the parent compound (without Fe doping). We do not find any anomaly in the non-linear AC susceptibility χ_2 of these compounds, in either zero DC field or in the presence of a DC field ∼20 Oe, as shown in figure 7.

In summary we have extensively studied the non-linear components of the AC susceptibility of Fe doped CMR compounds. Both the second and third harmonics of the AC susceptibility have been studied, as a function of temperature, for various DC fields and AC frequencies. We have substantiated our results using the frustration induced ferromagnetic cluster model. We presume that the frustration effects proliferate gradually, right after the FM transition. However complete spin glass like transitions occur at a much lower temperature, $T_g \sim 100$.

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