Internal friction evidence of the intrinsic inhomogeneity in La$_{0.67}$Ca$_{0.33}$MnO$_3$ at low temperatures

Y. Q. Ma, W. H. Song, R. L. Zhang, J. M. Dai, J. Yang, and J. J. Du

Key Laboratory of Internal Friction and Defects in Solids, Institute of Solid State Physics, Chinese Academy of Sciences, Hefei 230031,
People's Republic of China

Y. P. Sun*

Key Laboratory of Internal Friction and Defects in Solids, Institute of Solid State Physics, Chinese Academy of Sciences,
Hefei 230031, People's Republic of China
and National Laboratory of Solid State Microstructures, Nanjing University, Nanjing 210093, People's Republic of China

C. Z. Bi, Y. J. Ge, and X. G. Qiu

National Laboratory for Superconductivity, Institute of Physics and Center for Condensed Matter Physics, Chinese Academy of Sciences,
P.O. Box 603, Beijing 100080, People's Republic of China

The ferromagnetic manganite La$_{0.67}$Ca$_{0.33}$MnO$_3$ is systematically investigated below Curie temperature T_C by measurements of internal friction $Q^{-1}(T)$, Young's modulus $E(T)$, resistivity $\rho(T)$, and magnetization $M(T)$. Accompanying single metal-insulator transition at T_p and single paramagnetic-ferromagnetic (PM-FM) transition, three peaks at temperatures 260, 222, and 105 K are observed in the spectra of internal friction $Q^{-1}(T)$, respectively. The two peaks at 260 and 105 K are of the characteristic of the phase transition, which are related to the magnetic transition from a PM disorder state to a FM order state and to the intrinsic inhomogeneity due to the electronic phase separation, respectively. The broad peak around 222 K is actually composed of two subpeaks located at 222 and 234 K, respectively. This broad peak is of the characteristic of relaxation originating from the thermally activated motion of insulating and conduction domains. The activation energy and the relaxation time at infinite temperature are determined as 0.32 eV, 2.0×10^{-12} s for the 222-K subpeak and 0.38 eV, 2.5×10^{-13} s for the 234-K subpeak, respectively.

I. INTRODUCTION

Since the colossal magnetoresistance (CMR) effect in perovskite manganese oxides $R_{1-x}A_x$MnO$_3$ (R is a trivalent rare earth such as La, Nd, Pr, etc., A is a divalent dopant such as Ca, Sr, Ba, Pb, etc.) is discovered, it has been extensively studied in past years because of its value in fundamental physics and its potential applications.[1–3] For CMR materials with doping level around $x=0.33$, the measurement of the temperature dependence of resistivity of this material shows a metal-insulator transition (MIT) from a low-temperature metallic phase to a high-temperature insulating phase characterized by the peak at T_p. Accompanying the MIT, a magnetic transition from a high-temperature paramagnetic (PM) phase to a low-temperature ferromagnetic (FM) phase characterized by the Curie temperature T_C at which spontaneous magnetization appears. It has long been thought that the MIT and FM-PM transition are qualitatively correlated via the double exchange (DE) interaction mechanism, i.e., the hopping of e_g electrons from the e_g orbital of Mn^{3+} to the neighboring empty e_g orbital of Mn^{4+} ions through $2p$ orbital of O^{2-} ions in between.[4] The hopping of the e_g electrons is enhanced in the FM state in which the core spins (t_{2g} electron spin) of Mn^{3+} and Mn^{4+} are parallel. In turn, the hopping e_g electrons promote FM order because they energetically prefer to polarize the spins in their vicinity. The hopping e_g electron does not pay strong Hund coupling energy (J_H) if both e_g and t_{2g} electron spins are parallel. The electron-spin scattering is minimized in this process and the

kinetic energy of e_g is optimized. The applied magnetic field can suppress the spin disorder around T_C and shift both T_p and T_C to higher temperatures, which results in the CMR effect. However, Millis *et al.*[5] argued that the DE mechanism alone was not sufficient to describe some features of the CMR effect, such as the magnitude of the resistivity at the high temperature and CMR. In order to explain the CMR effect quantitatively, a strong electron–Jahn-Teller–phonon coupling arising from the distortion of the MnO$_6$ octahedra due to the Jahn-Teller effect and the orbital-ordering effects due to two e_g orbitals per Mn ion should be considered in the framework of the DE model. In addition, recently, a phase separation (PS) theory has been proposed to account for the CMR effect.[6–9] Anyway, the exact origin of the CMR effect observed in manganites has not fully been elucidated so far.

Recently, the question as to whether the metallic FM phase in manganites is homogeneity has attracted attention because it intimately links to the intrinsic properties of the doped manganites. Several papers have reported that the FM phase in La$_{1-x}$Ca$_x$MnO$_3$, which is a typical DE system, has a complicated character.[10–14] Fäth *et al.* investigated La$_{1-x}$Ca$_x$MnO$_3$ single crystals and thin film with x of about 0.3 using scanning tunneling spectroscopy (STS) and found a phase separation where inhomogeneous structures of metallic and insulating phases coexisted.[10] Heffner *et al.* studied La$_{0.67}$Ca$_{0.33}$MnO$_3$ polycrystalline sample using the muon spin relaxation (μSR) technique and found that at least two spatially separated FM regions existed, which possessed temperature-dependent volumes and very different Mn-ion

spin dynamics.[11] The Mössbauer spectroscopy in $La_{0.7}Ca_{0.3}Mn(^{57}Co)O_3$ compound showed two FM phases, $M1$ and $M2$.[12] The coexistence of two FM phases were also observed using ^{55}Mn NMR.[13] Except for the inhomogeneity observed in $La_{1-x}Ca_xMnO_3$ compound with x of about 0.3, this phenomenon is also observed in $La_{1-x}Sr_xMnO_3$ poly-crystalline and film samples with $x=0.3$ by means of ^{55}Mn NMR (Ref. 13) and STS,[14] respectively.

The internal friction measuring technique is a nondestruc-tive but very sensitive tool in studying defects and micro-scopic processes in solids[15–18] including electron strongly correlated materials, such as cuprate high-temperature super-conductors and manganites.[15,16,18] Very recently, the glassy dynamics of the inhomogeneous metallic phase in $La_{1-x}Ca_xMnO_3$ with $0.33<x<0.5$ was investigated in the wide temperature range over 100 K up to the high tempera-ture near 900 K by the measurement of the anelastic spectra.[18] In this paper, we report the study of the FM phase below MIT in the $La_{0.67}Ca_{0.33}MnO_3$ polycrystalline sample in the temperature range of 80–300 K by measurements of internal friction $Q^{-1}(T)$ and Young's modulus $E(T)$ at a zero magnetic field.

II. EXPERIMENTAL TECHNIQUES

Polycrystalline sample $La_{0.67}Ca_{0.33}MnO_3$ was prepared by the standard solid-state reaction. Stoichiometric and La_2O_3, $CaCO_3$, MnO_2 powders were mixed and ground, and then calcined at 1000 and 1200 °C for 24 h for several times with intermediate grinding. After the final grinding, the material was sintered at 1400 °C for 24 h and slow cooled to room temperature. The dimension of samples used in Q^{-1} mea-surement is $80\times4\times3$ mm^3. Powder x-ray-diffraction mea-surement was performed using a Philips PW 1700 x-ray dif-fractometer with Cu K_α radiation at room temperature. The resistivity ρ of $La_{0.67}Ca_{0.33}MnO_3$ was measured by the con-ventional four-probe technique. The temperature dependence of magnetization was measured using a Quantum Design commercial superconducting quantum interference device (SQUID) magnetometer. $Q^{-1}(T)$ and $E(T)$ were measured by the free decay method of a resonant bar in acoustic fre-quency range with magnitude of kHz and in the temperature range of 80–300 K using warming mode in a helium gas environment at the rate of 0.3 K per minute under zero ap-plied magnetic field. The sample was excited electromagneti-cally in the fundamental and the first free flexural modes, whose resonant frequencies f are in the ratios 1:2.76. The internal friction Q^{-1} is defined as follows:[15]

$$Q^{-1}=\frac{1}{n}\ln\left(\frac{A_0}{A_n}\right),\tag{1}$$

where n is the number of the vibration cycles, while the amplitude attenuates from A_0 to A_n. The Young's modulus E is given by
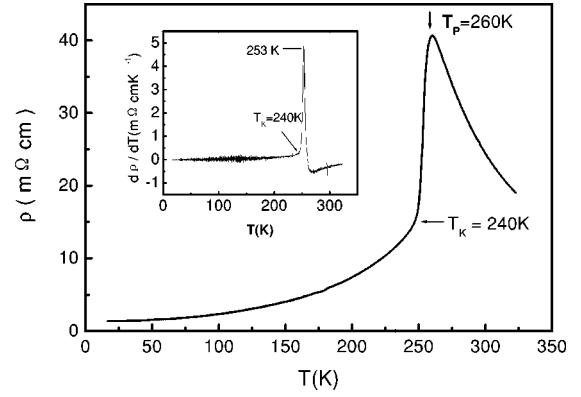
$$E=\frac{4\pi^2 sdl^4}{m^4 I}f^2,\tag{2}$$

FIG. 1. Temperature dependence of the resistivity for $La_{0.67}Ca_{0.33}MnO_3$ at zero magnetic field. The inset shows $d\rho/dT$ versus temperature, and T_K is defined as the temperature below T_P at which $d\rho/dT$ begins to rise rapidly.

where f is the resonant frequency, s is the cross-sectional area, d the density, l the length, I the moment of inertia of the sample. In addition, m is 4.730 for the vibrating mode in the fundamental mode, in which the sample is suspended at two points whose span is $0.224l$ from two free ends of the sample. We define the resonant frequency f in the fundamen-tal mode at room temperature (300 K) as measuring fre-quency $f_0=2.56$ kHz, thus the measuring frequency in the first free flexural mode as $2.76f_0$, i.e., 7.06 kHz. Based on Eq. (2), E is proportional to the square of the resonant fre-quency, i.e., $E\propto f^2$. Therefore we substitute f^2 for the Young's modulus E.

III. RESULTS AND DISCUSSION

Powder x-ray diffraction measurement at room tempera-ture indicates that the investigated sample $La_{0.67}Ca_{0.33}MnO_3$ is a single phase. All diffraction peaks can be indexed by orthorhombic lattice with space group *Pbnm* and lattice pa-rameters of $a=5.4788$ Å, $b=5.4709$ Å, and $c=7.7459$ Å, which reveals the sample is chemically homogeneous.

The temperature dependence of resistivity $\rho(T)$ in $La_{0.67}Ca_{0.33}MnO_3$ is shown in Fig. 1. A distinct MIT occurs at the temperature $T_p=260$ K. T_K, defined as the tempera-ture below T_P at which $d\rho/dT$ begins to rise rapidly with increasing temperature, is about 240 K; T_s^{max}, defined as the temperature corresponding to the maximum of $d\rho/dT$, is 253 K (shown in the inset of Fig. 1). Below T_K, the resis-tivity ρ of the sample decreases monotonously with the de-crease of the temperature and without any detectable anomaly in $\rho(T)$, which seems to imply that the sample lies in a homogeneous metallic state in the low-temperature range below T_K.

The temperature dependence of magnetization $M(T)$ of the sample under an applied field of $H=0.01$ T and field-cooled mode is shown in the main panel of Fig. 2. It shows that the sample undergoes a PM-FM transition at Curie tem-perature $T_C=260$ K defined as the inflection point on the $M(T)$ curve with decreasing temperature. Furthermore, M is almost temperature independent in the whole temperature re-
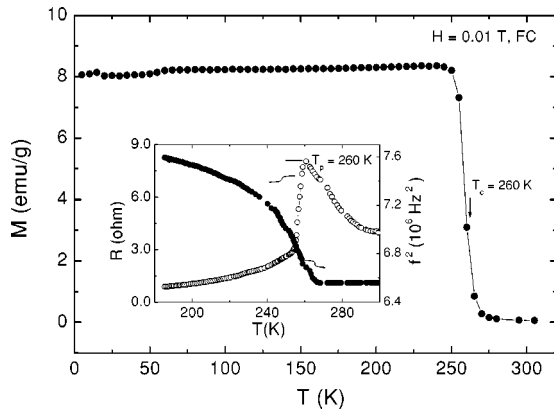
FIG. 2. Temperature dependence of magnetization of $La_{0.67}Ca_{0.33}MnO_3$ under the field-cooled mode at an applied magnetic field of 0.01 T. The inset shows the temperature dependence of the resistance R and Young's modulus E measured *in situ* in the neighborhood of MIT.

FIG. 4. Temperature dependence of the internal friction Q^{-1} under the different measuring frequencies of 2.56 and 7.06 kHz around 260 and 105 K as the background internal friction is subtracted.

gion below T_C, which implies that the magnetic moment of the sample does not vary evidently with the decrease of the temperature. In other words, the sample seems to lie in a homogeneous FM state below T_C.

Based on electric transport and magnetic measuring results, one usually seems to think that the sample should lie in a homogeneous FM metallic state below T_C. To test whether this viewpoint is real, both Q^{-1} and E are measured.

The temperature dependence of Q^{-1} and E (denoted using measuring frequency square f^2) is shown in Fig. 3. $Q^{-1}(T)$ exhibits three peaks located at 260, 222, and 105 K, respectively. The similar peaks at about T_C and just below T_C are also detected by Cordero *et al.*[18] However, for our measurement, another peak at lower temperature of 105 K is observed, which may be ascribed to our lower temperature measurement range. Combined with the result of magnetization $M(T)$ as shown in Fig. 2, the Q^{-1} peak at 260 K is suggested to originate from the change of magnetic entropy in the process of magnetic transition.[16,18] That is to say, the magnetic entropy of the sample will take place a large loss when the sample undergoes a transition from magnetically
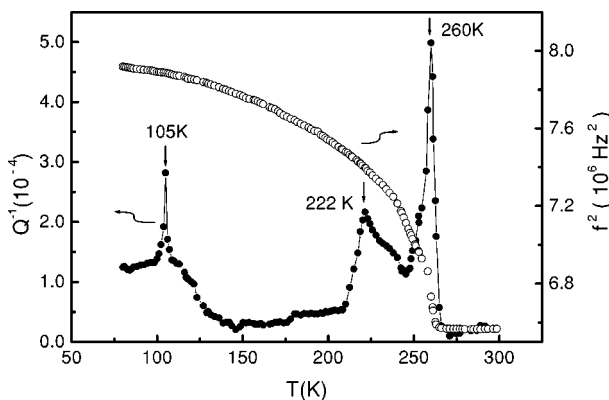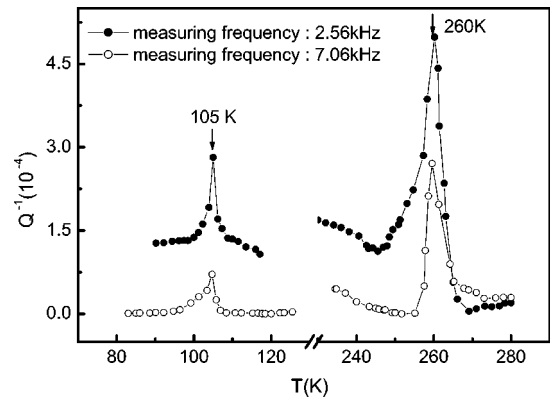

FIG. 3. Temperature dependence of the internal friction Q^{-1} and Young's modulus at measuring frequency of 2.56 kHz for $La_{0.67}Ca_{0.33}MnO_3$ at zero magnetic field.

disordered paramagnetic state to ordered ferromagnetic one. To further identify the nature of this peak, we measure $Q^{-1}(T)$ around 260 K under different measuring frequencies of 2.56 and 7.06 kHz. The result is shown as in Fig. 4. It shows that the position of the peak does not shift with raising frequency. It reveals that the peak is of the feature of phase transition-induced Q^{-1} peak. It elucidates further that this peak is caused by the PM-FM transition. In addition, a prominent softening of E can be observed in the temperature range of $T_k < T < T_p$ from Fig. 3 with increasing temperature. This kind of large softening of E around T_C was also observed by Cordero *et al.*[18] As is well known, E is close related to cohesive force among atoms and is proportional to the inverse of the static strain if the external force is kept in a constant. Therefore the variation of E also actually reflects the information of lattice variation. The large softening of E in the vicinity of 260 K actually means that there exists an obvious lattice distortion accompanied by FM-PM transition, which is driven by Jahn-Teller effect due to Mn^{3+} ions. This seems to be consistent with recent neutron powder-diffraction and x-ray-absorption fine-structure measurement results.[19-21]

To probe the correlation between E and electric transport behavior around MIT T_p, the temperature dependence of resistance R and E of the sample is measured *in situ* simultaneously in the temperature range of 180–300 K as shown in the inset of Fig. 2. It reveals that E occurs an evident softening in the neighborhood of T_p, which is similar to the result shown in Fig. 3. As $T > T_p$, E almost does not change with temperature. The result demonstrates the E occurs a remarkable change in the process of MIT, which implies that there exists a close correlation between charge and lattice. In addition, this result also implies that MIT occurring at 260 K is of the character of the first order because the large variation of E reveals the variation of the volume of lattice as suggested in Ref. 22 recently. In addition, a very small resistivity $\rho(T)$ hysteresis (not shown here) around 260 K during the process of cooling and warming measurement also reveals the character of the first-order transition. Combined the results of Q^{-1}, E with ρ in the vicinity of 260 K, it unam-

biguously indicates the sample exists a strong charge-spin-lattice coupling.[23]

Compared with electric transport and magnetization measurements, the measurement of Q^{-1} seems to supply more fine information for the sample because there exists extra two Q^{-1} peaks located at 105 and 222 K except for the prominent Q^{-1} peak of 260 K discussed above as shown in Fig. 3. We suggest these two peaks may be related to the microscopic behavior of the sample. First, the nature of the 105-K peak is investigated by changing the measuring frequency. The result indicates that the position of 105-K peak does not shift with raising measuring frequency from 2.56 to 7.06 kHz as shown in Fig. 4, which reveals the characteristic of the phase-transition Q^{-1} peak. The similar Q^{-1} peaks appear in $La_{0.8}Ca_{0.2}MnO_3$, $La_{0.7}Sr_{0.3}MnO_3$, and $Nd_{0.7}Sr_{0.3}MnO_3$ at the temperatures of 98, 118, and 110 K, respectively.[24] In general, the first-order transition is accompanied by hysteresis and a discontinuous change in volume. No hysteresis phenomenon is observed in the curves of $\rho(T)$ around 105 K (not shown here) by measuring cooling and warming $\rho(T)$ curves and no evident anomalous variation is detected in the $E(T)$ curve around 105 K. Therefore we suggest that the transition at the 105-K peak is of the characteristic of the second-order phase transition. The optical density of infrared measurement also shows anomalous behavior in the vicinity of 100 K, which suggests that this anomalous behavior is related to the phase separation.[24] Compared with the very sharp 105- and 260-K peaks, the 222-K peak is much broader in the curve of $Q^{-1}(T)$, indicating it perhaps has a fully different origin from the other two peaks. By changing measuring frequency, it is found that the peak shifts to higher temperature with the increase of frequency (2.56 and 7.06 kHz), which demonstrates that this peak is associated with a thermally activated relaxation process. The relaxation character of about 220 K is also obtained by Cordero *et al.*[18] In Ref. 18, the authors fit the curve and suggest that it is a single peak which is attributed to the glassy dynamics of some inhomogeneous phases. For our observed 222-K peak, we find that this peak can be fit by two Debye peaks with distribution in relaxation time by nonlinear fitting method,[25] which indicates that the 222-K broad peak is actually composed of two subpeaks located at 222 and 234 K, respectively when the background internal friction is subtracted. The fitting result is plotted in Fig. 5, which shows that fitting data is almost in superposition with experimental one. The shift of peak positions of two subpeaks with changing frequency is shown in the inset of Fig. 5. For a thermally activated relaxation process, the relaxation time τ generally follows the Arrhenius relation[26]

$$\tau = \tau_0 \exp(E/k_B T), \qquad (3)$$

where τ_0 is the preexponential factor (or the relaxation time at infinite temperature), E is the activation energy of the relaxation process, and k_B is the Boltzmann constant. At the Q^{-1}-peak position, the condition $\omega\tau = 1$ is satisfied, where $\omega = 2\pi f$ is the angular frequency of measurement. By fitting, the relaxation parameters E and τ_0, which are 0.32 eV, 2.0 $\times 10^{-12}$ s for the 222-K peak and 0.38 eV, 2.5×10^{-13} s for
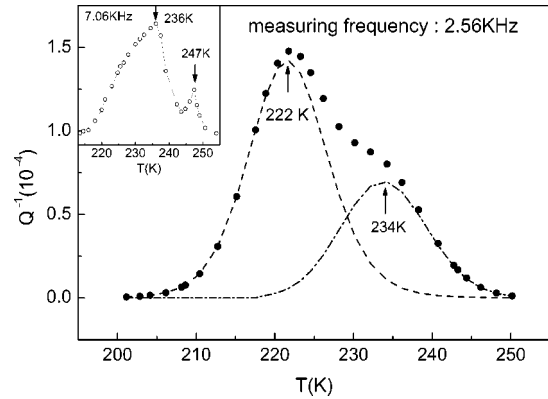

FIG. 5. Temperature dependence of the internal friction peak around 222 K at measuring frequency of 2.56 kHz. The circles are experimental data points after subtracting background internal friction. The dashed and dashed dot lines are the fitting lines of the 222- and 234-K peaks, respectively. The inset is the temperature dependence of the internal friction peak around 222 K at a measuring frequency of 7.06 kHz (the background internal friction is also subtracted).

the 234-K peak, are obtained. Two different activation energies may correspond to two different relaxation units, which will be discussed below. By the way, from Fig. 2(b) in Ref. 18, the peak just below T_C also seems to be composed of two subpeaks. That is to say, both the peak just below T_C observed by Cordero *et al.* and by us may be of the same origin.

As to the origin of Q^{-1} peaks of 105 and 222 K, it is reasonable to suggest that they are closely related to the intrinsic inhomogeneity of the sample. In fact, as Becker *et al.* argued, the broad MIT implied the sample is inhomogeneous.[14] For our studied sample, Fig. 1 shows that there exists a 20-K difference between T_P and T_K. Generally, for a completely homogeneous sample, one would expect that $\rho(T)$ curve has a sharp transition and T_K should be very close to T_P when the sample undergoes a transition from the low-temperature FM metallic phase into the high-temperature PM insulating phase at the Curie temperature T_C. Therefore we could suggest that the sample may exist an intrinsic inhomogeneity consisting of insulating and conducting domains in the FM background below T_C as suggested by STS, μSR, Mössbauer spectroscopy, and NMR techniques.[11–13] As to the 105-K Q^{-1} peak, we suggest that it originates from the electronic phase separation. That is to say, as $T < 105$ K, the sample may behave as a homogeneous single FM metallic phase. As the temperature is increased to 105 K, an electrically insulating phase appears. In other words, the sample exhibits an electronic phase separation at 105 K. Based on the character of the 105-K Q^{-1} peak described above, this electronic phase separation is of the character of the second-order phase transition. Hence, as $T > 105$ K, the sample comprises both metallic and insulating domains. The fraction of insulating domains increases and the fraction of metallic domains decreases with increasing temperature. Furthermore, we suggest that the increase of the fraction of insulating domains is achieved by the increase of the number of insulating domains and not achieved by the

growth of the size of insulating domains. That is to say, the increase of the fraction of insulating domains is achieved by the nucleation growth of the insulating domain and not achieved by the mobility growth of the insulating domain boundary. With increasing temperature, more and more e_g electrons are bound in the insulating domains, which results in the motion of the insulating and metallic domain boundary. Some noise in the slope of the resistivity occurred just below 105 K (see inset in Fig. 1) is perhaps related to the appearance of insulating domains. In terms of above the calculation of the 222-K Q^{-1} peak, activated energies of domain boundary motion are 0.32 and 0.38 eV for insulating and metallic domains, respectively. Considering the fact the activated energies are very small, it is reasonable to suggest that the relaxation mechanism of the 222-K peak is related to the change of e_g electrons' state—from itineration to localization. In the temperature range of 105–222 K, though the relative volumes of insulating and metallic domains are changed, the percolative path constituted by metallic domains does not become fully destroyed and the sample keeps up percolative conduction causing the sample to exhibit metallic behavior. The microscopic intertwining of the insulating and metallic domains due to their thermally activated relaxation motion leads to the remarkable increase of resistivity and to the melting of the percolative path giving rise to the occurrence of MIT. The broad MIT is ascribed to the development of the insulating area and the thermally activated relaxation motion of the insulating and metallic domains with temperature.

Why does the sample occur the electronic phase separation at low temperature below MIT? We suggest that the electronic phase separation is closely related to the local distortion disorder of the MnO_6 octahedron which belongs to the electronic phase separation induced by disorder as argued by Moreo et al.[7] Our results can be well explained by recent results of high real-space-resolution atomic pair distribution functions obtained by Billinge et al. using high-energy x-ray powder-diffraction technique.[27] For the $La_{0.67}Ca_{0.33}MnO_3$ polycrystalline sample, at the low temperature of 20 K, they find that the sample exhibits single Mn-O bond length of 1.96 Å and there is no evidence of any long bond caused by Jahn-Teller distortion, which means there does not exist the distortion of the MnO_6 octahedron and the sample lies in an entirely delocalized homogeneous metallic phase. However, when the temperature is increased to 100 K, the Mn^{3+}-O-long bonds of 2.18 Å begin to appear and persist further to room temperature of 300 K, which means the appearance of Jahn-Teller distortion of the MnO_6 octahedron. This distortion results in the localization of the e_g electron and gives rise to the appearance of the insulating phase. Based on the schematic phase diagram obtained by Billinge et al.,[27] it seems to be reasonable that the 105-K Q^{-1} peak is suggested to originate from the electronic phase separation induced by the local distortion disorder of the MnO_6 octahedron. Based on the above discussion, the relation between the $Q^{-1}(T)$ spectrum and the Mn-O bond length can be plotted schematically in Fig. 6. In fact, the gradual decrease of E with the increasing temperature shown in Fig. 3 also seems to consist with the variation of Mn-O bond lengths
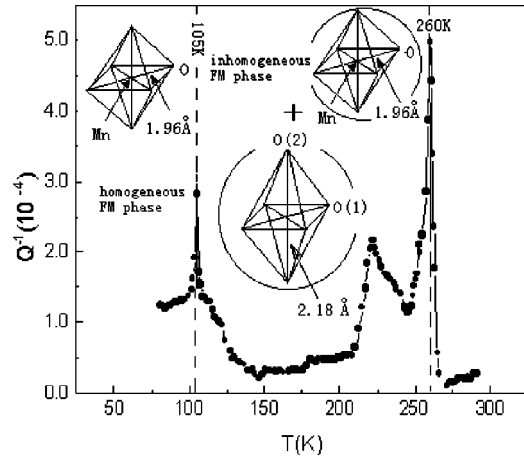

FIG. 6. Schematic MnO_6 octahedron in homogeneous and inhomogeneous FM phases.

with temperature caused by the local distortion of the MnO_6 octahedron. Because the appearance of local Jahn-Teller distortion leads to the increase of globally average $\langle Mn\text{-}O \rangle$ bond length. The increase of the $\langle Mn\text{-}O \rangle$ bond length with increasing temperature was also corroborated by neutron diffraction.[28] It is understandable that the increase of Mn-O bond length results in the decrease of E.

IV. CONCLUSION

In summary, the behavior of the optimally doped $La_{0.67}Ca_{0.33}MnO_3$ sample below MIT is investigated by internal friction Q^{-1} and Young's modulus E measurements. In addition to the 260-K Q^{-1} peak correlated with PM-FM transition being observed, the other two Q^{-1} peaks located at 105 and 222 K are also detected. We suggest that the 105-K peak originates from the electronic phase transition induced by the local distortion disorder of the MnO_6 octahedron, which gives rise to the coexistence of metallic and insulating domains in the temperature region of 105 K$<T$ $<T_p$. The results reveal the nature of the intrinsic inhomogeneity of the sample in the FM phase. The broad peak around 222 K is of the dynamic characteristic of insulating and conduction domains and reflects the thermally activated relaxation motion of two kinds of domains. Our results indicate that the internal friction method seems to be a useful technique in studying the coupling between spin, charge and lattice of CMR materials. The further work of Q^{-1} in other manganites is in progress.

*Corresponding author. Email address: ypsun@issp.ac.cn

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