

Intrinsic metastability of low doped manganites: $\text{La}_{0.8}\text{Ca}_{0.2}\text{MnO}_3$ case

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Abstract. Transport properties of phase separated $\text{La}_{0.8}\text{Ca}_{0.2}\text{MnO}_3$ crystals in the aged highly resistive metastable state were studied. It was found that the coexistence of different ferromagnetic phases at low temperatures is sensitive to electric current/field. In a contrast with the previously studied low resistivity metastable states the high resistivity state exhibits positive magnetoresistance and significant current dependence of the resistivity even at temperatures much higher than the Curie temperature. Application of current pulses results in appearance of zero bias anomaly in the current dependent conductivity. Similarly to the low resistivity metastable states the memory of the resistivity can be erased only after heating of the sample to $T_e \approx 360$ K. After one year storage at room temperature the $\text{La}_{0.8}\text{Ca}_{0.2}\text{MnO}_3$ samples show clear signatures of aging. The aged samples spontaneously evolve towards high resistivity states. The results are discussed in the context of a coexistence of two ferromagnetic phases with different orbital order and different conductivity. The metallic ferromagnetic phase seems to be less stable giving rise to the experimentally observed electric field effects and aging.

PACS. 75.47.Gk Colossal magnetoresistance – 75.47.Lx Manganites – 71.30.+h Metal-insulator transitions and other electronic transitions

1 Introduction

Magnetic and transport properties of doped colossal magnetoresistive (CMR) manganites of the form $R_{1-x}A_x\text{MnO}_3$, where R is a rare-earth ion and A is a divalent ion such as Ca, Sr, Ba, etc. depend not only on the doping level x and the average radius of the A -site cation [1,2] but also on the magnetic [3], electric [4–6] and thermal [7] history of the sample. These properties of CMR manganites are closely related to pronounced electronic and magnetic phase separation (PS).

Metastability seems to be a generic feature of phase separated systems with two or more competing ordering mechanisms. Double exchange (DE) interactions establish ferromagnetic ordering in CMR manganites through a strong Hund's coupling between e_g electrons of neighboring Mn^{3+} and Mn^{4+} sites. However, for manganites doped below the percolation threshold x_c a ferromagnetic insulating phase incompatible with the DE mechanism has been observed [8–10]. According to the recent theoretical [11,12] and experimental [9,10,13–15] results the superexchange (SE) interactions and orbital ordering (OO) govern transport and magnetic properties of low-doped manganites hand in hand with the DE. The percolation threshold in $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ (LCMO) mangan-

ites is close to $x = 0.225$ [13]. The presence of two ferromagnetic phases with different orbital order and electronic properties in low doped LCMO ($x < x_c$) leads to pronounced metastable states with markedly different resistivities [4–6]. Metastable states are characterized by history dependent conductivity, magnetization and resistivity relaxation, memory effects, and strongly non-Gaussian conductivity fluctuations, see [1,3–5] and references therein.

The best way to reveal the metastable properties of low doped manganites is to subject a sample to abrupt changes of an experimental parameter such as e.g., current bias, magnetic field, or temperature. Using continuous current sweeps and short current pulses we have created various metastable states in $\text{La}_{0.82}\text{Ca}_{0.18}\text{MnO}_3$ and $\text{La}_{0.8}\text{Ca}_{0.2}\text{MnO}_3$ single crystals [4,5]. We have investigated in detail properties of low resistivity metastable states and found that they are strongly influenced by the thermal, magnetic, and electric history of a sample. Specific current pulse procedures allowed us to induce also high and very high metastable resistivity states in low doped LCMO crystals [5]. In the initial experiments current induced high and low resistivity states could have been relatively easily rejuvenated to the initial pristine state, the state appearing in the first measurements of the freshly fabricated samples, by means of a thermal

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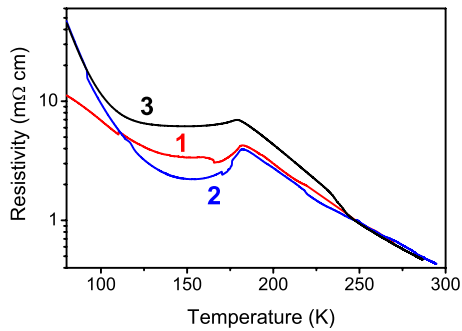


Fig. 1. Temperature dependence of the resistance of $\text{La}_{0.8}\text{Ca}_{0.2}\text{MnO}_3$ single crystal: curve (1) - the pristine resistive state, curve (2) - high resistivity metastable state (HRMS) created by an application of voltage pulses to the sample cooled down to 10 K in the pristine state, curve (3) - HRMS seen after 1 year storage of the sample at room temperature.

treatment at slightly elevated temperatures. However, with the elapsing time our samples show a tendency to evolve spontaneously towards higher resistivity states and it becomes progressively more and more difficult to bring them back to the “pristine” state. Such behavior poses a question about the ultimate stable state of low doped manganites. In this paper we address this issue by experimentally studying the properties and evolution of the high resistivity metastable state in the low doped $\text{La}_{0.8}\text{Ca}_{0.2}\text{MnO}_3$ single crystals.

2 Experimental and results

Single crystal of $\text{La}_{0.8}\text{Ca}_{0.2}\text{MnO}_3$ was grown by a floating zone method using radiative heating as described previously [5]. For electrical transport measurements $8 \times 3 \times 1.6 \text{ mm}^3$ sample with the longest dimension aligned with $\langle 110 \rangle$ axis was cut from the as-grown crystal. The detailed characterization of the same, performed almost immediately after its fabrication, was presented in reference [5]. In the present work we have used the same sample and the measuring setup as the one employed previously [5].

Figure 1 shows the temperature dependence of the resistance of the $\text{La}_{0.8}\text{Ca}_{0.2}\text{MnO}_3$ crystal recorded various resistivity states. Curve 1 shows the characteristic of a freshly made sample in its pristine state. After measurements of the pristine state properties a High Resistivity Metastable State (HRMS) has been created by means of applying at 10 K a series of 10 ms long voltage pulses with 10 V amplitude to the sample in a pristine state. The current within each pulse was limited to a maximum of 40 mA. This treatment caused an increase of the low temperature resistance by three orders of magnitude; from the initial pristine 10 Ω range to 2.5 k Ω range in a fully developed HRMS [5].

After creation of the HRMS the sample was stored for almost 1 year at room temperatures. The resistivity characteristic of the one year old sample is shown with the line 3. The R vs. T dependence has changed markedly with

respect to that of the freshly induced HRMS, shown with the curve 2 in Figure 1. Beside the general shape change $R(T)$ of an aged sample shows step-like resistivity change around 230–240 K. In the same time the magnetization vs T characteristic turned out to be exactly the same as the one recorded a year ago. In particular, Curie temperature T_C did not change and reminded exactly the same $T_C = 184$ [16]. This is reflected also in the R vs. T characteristics where the maximum marking the metal-insulator transition temperature became much less pronounced but it still coincides with the Curie temperature evaluated from magnetization measurements. Unchanged Curie temperature indirectly proves that significant changes in the resistivity characteristics are not caused by any possible oxygen loss during long term sample storage at room temperatures.

Freshly made low resistive metastable state is characterized by a number of sharp up and down resistance jumps occurring during recordings of the temperature dependencies of the resistance [5]. In the current induced HRMS the probability of occurrence of such jumps decreases with time and the number of current and thermal cycles. In general, the HRMS state appears to be more stable than the low resistivity metastable states and exhibits less jumps [5]. Observe that resistivity jumps have practically disappeared from the characteristics of the aged sample, see Figure 1.

Magnetoresistance $\text{MR} = [R(0) - R(H)]/R(H)$ of the $\text{La}_{0.8}\text{Ca}_{0.2}\text{MnO}_3$ crystal also depends on the resistivity state. Nevertheless, at temperatures well above T_C , when the sample is in a paramagnetic state, MR is practically absent independently of the actual resistivity state. The most pronounced negative MR is observed, as expected, in the vicinity of the metal-insulator transition. In a marked contrast to the pristine state exhibiting only negative MR, the low temperature MR of HRMS is positive at low fields and turns to be negative for fields exceeding 10 kOe, as shown in Figure 2. A similar behavior of MR has been previously observed in $\text{La}_{0.82}\text{Ca}_{0.18}\text{MnO}_3$ crystals [16]. It has been suggested that the positive MR may occur only in a predominantly insulating ferromagnetic ground state [17]. For a ferromagnetic phase with the dominant DE mechanism only the negative MR is expected. This predictions have been verified by MR measurements under the hydrostatic pressure. We have found that at temperatures below T_C already a modest pressure of 6.5 kbar considerably reduces the resistivity of a $\text{La}_{0.82}\text{Ca}_{0.18}\text{MnO}_3$ crystal and converts the positive MR of 3.5% in $H = 10$ kOe at $T = 77$ K to a fully negative one of 15% [16]. Changes in the sign of the MR were attributed to increasing of the volume of the ferromagnetic metallic phase with dominant DE interactions with increasing pressure.

One of the major problems in experimental studies of the electric current/field influence upon HRMS conductance is the Joule heating which may cause artifacts similar to those due to metastability and nonlinearity of transport properties [18,19]. To some extent it is possible to discern experimentally heating artifacts from the genuine metastability effects. Some specific effects in low doped

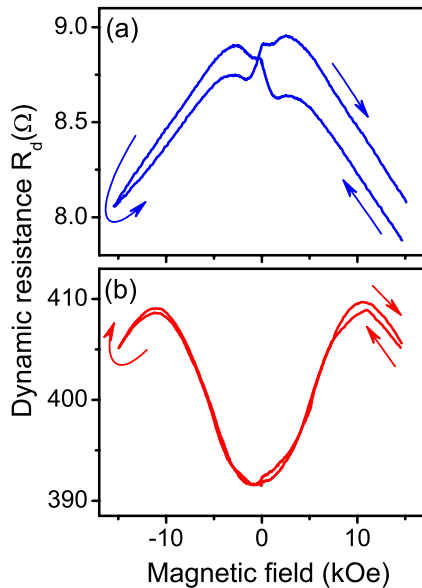


Fig. 2. Dynamic resistance of $\text{La}_{0.8}\text{Ca}_{0.2}\text{MnO}_3$ crystal vs. magnetic field at $T = 77$ K in the pristine (a) and HRMS state (b).

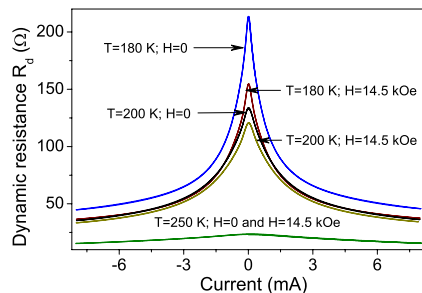


Fig. 3. Current dependence of the dynamic resistance of HRMS at various temperatures as measured in zero magnetic field and in $H = 14.5$ kOe.

manganites, such as e.g., the memory of the resistive state, persist for a long time after switching off the current [4, 5]. Moreover, experimentally observed resistivity changes under the influence of the current flow are frequently oppose to those expected from the heating mechanism. For example, the resistivity of $\text{La}_{0.82}\text{Ca}_{0.18}\text{MnO}_3$ at low temperatures has a semiconducting-like character ($dR/dT < 0$) and simple heating should result in resistivity decrease, contrary to the effects observed in the experiments [5, 6].

Figure 3 shows experimental recording of the dynamic resistance R_d as a function of bias current obtained at temperatures around and above T_C . Bias current significantly decreases the resistance even at temperatures much higher than the T_C . At room temperature current flow of 8 mA results in 15% decrease of the resistance. Even if the resistivity at high temperatures is very low we cannot exclude that the observed effect may be, at least partially, due to the local inhomogeneous overheating.

With temperature decreasing towards the Curie temperature the shape of $R_d(I)$ dependence changes. A bell-like form at high temperatures evolves towards a narrow

maximum centered at zero current, see Figure 3. As expected, the effect of the applied magnetic field are practically absent at high temperatures $T \geq 250$ K. The highest MR is observed in the vicinity of T_C , compare curves recorded at zero applied field with those obtained at field = 14.5 kOe in Figure 3. Such behavior of is typical for most of the CMR systems.

As already discussed in our previous paper, application of current pulses to $\text{La}_{0.8}\text{Ca}_{0.2}\text{MnO}_3$ crystal induces metastable resistivity states characterized by a relatively long lifetime, significantly exceeding the time scale of an experiment [5]. The pulse treatment procedure is most efficient when current pulses are applied at low temperatures $T \ll T_C$.

Figure 4a shows the effect of an application of a single strong pulse with the magnitude 40 V and 500 ms length to a sample in a HRMS which has been aged for a year at room temperatures. During the pulse treatment the sample was immersed directly in the liquid nitrogen bath and the maximum current was limited to 200 mA. The $R_d(I)$ characteristic of the aged HRMS is shown with curve (1) while the pulse modified one with curve (2). In our previous investigations in which a recently fabricated samples were employed, freshly enforced metastable states could have been erased by a heat treatment at temperatures $T > 360$ K. After such thermal treatment the sample has always rejuvenated to the resistivity state very close to the pristine one [5]. The situation in the aged sample turned out to be markedly different.

In the first test we have heated the pulse-treated sample to the room temperature and maintained it at $T = 300$ K for 48 hours. After two days the sample was cooled back to 77 K and $R_d(I)$ dependence was again recorded. The $R_d(I)$ curves recorded before and after the room temperature treatment were found to be identical. Any significant changes in the sample resistivity were observed only after rising the temperature of the treatment to $T = 360$ K. In a difference to the situation observed in a freshly made sample the resistance of the aged one depends on the time of permanence at 360 K.

Figure 4b illustrates changes in the $R_d(I)$ dependence during a multi-stage heat treatment at $T = 360$ K. The zero bias asymmetry could have been eliminated only after a prolonged, almost 24 hours treatment, see Figure 4b. However, even an extended high temperature treatment was unable to restore the original pristine state of the one year old sample. On the contrary, the final zero current dynamic resistance after the treatment is now significantly higher than the one seen before the application of a pulse to the original HRMS. It seems that the sample has now a tendency to evolve towards more and more insulating state. The HRMS in the aged sample turns out to be more stable than in a fresh one. Spontaneous resistivity jumps do not appear on $R(T)$ characteristics and the memory of the aged HRMS state is much more robust. It cannot be rejuvenated to anything close to a pristine state with low resistivity. All these factors point out to an apparent strong aging of the LCMO crystals even when maintained at room temperature.

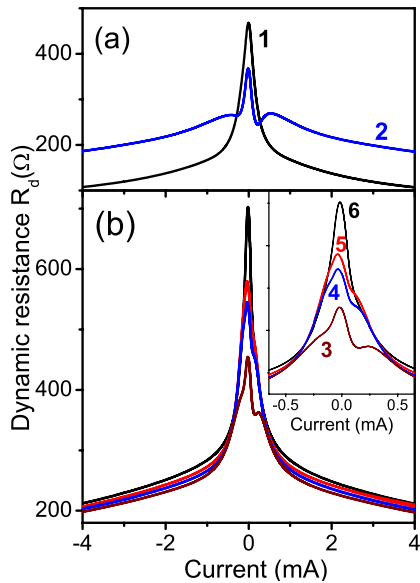


Fig. 4. (a) Dynamic resistance vs. current of $\text{La}_{0.8}\text{Ca}_{0.2}\text{MnO}_3$ crystal at $T = 77$ K before (curve 1) and after (curve 2) application of a strong current pulse; (b) dV/dI vs. I dependencies recorded subsequently after various stages of the high temperature treatment after application of a strong pulse: after 24 h at 320 K - curve (3), after 2 h at 360 K - curve (4), after another 2 h at 360 K - curve (5), after 24 h at 360 K - curve (6). Heat treatment at 360 K for times longer than 24 h was found not to influence in a significant way the resistive state. Inset: the dependencies from Figure 4b at low current range in the expanded current scale.

3 Discussion

A key element for understanding our experimental results is a pronounced phase separation of manganites which in low doped LCMO leads to a coexistence of two ferromagnetic phases with different orbital order [1]. It should be underlined that the resistivity of a PS system does not depend solely on the ratio between volumes occupied by the coexisting phases, but also in a crucial way depends on the distribution of the conductive and insulating domains, their size, and shape. External stimuli such as temperature gradients, applied magnetic field, or electric current/field, may affect the topology of the coexisting phases and even cause a change in their volume ratio [20]. Under the influence of an external stimulus the percolating path of conductive domains can be therefore “more insulating” or “more metallic”, leading to differences between transport properties of metastable resistive states in the same sample. The subtle balance between metastable resistive states with distinct electronic properties is an indicative of the closeness of energies of the coexisting phases.

A number of features observed in the electric transport of the aged HRMS in $\text{La}_{0.8}\text{Ca}_{0.2}\text{MnO}_3$ crystals have been previously observed in the pristine state of a less doped $\text{La}_{0.82}\text{Ca}_{0.18}\text{MnO}_3$. In both cases one sees a pronounced change in the slope of the resistivity at $T \approx 230$ K. A step-like change in $R(T)$ slope in $\text{La}_{0.82}\text{Ca}_{0.18}\text{MnO}_3$ has been ascribed by us to the Jahn-Teller (JT) structural phase

transition [4]. Recently, high resolution X-ray diffraction and neutron powder diffraction of low doped LCMO samples have confirmed that for temperatures below doping dependent T_{JT} the crystal symmetry indeed changes from the orthorhombic $Pnma$ to monoclinic $P2_1/C$ [21]. Both $x = 0.18$ and $x = 0.2$ crystals demonstrate a difference in the sign of the low temperature magnetoresistance between high and low fields. Most importantly however, the dynamic resistance of the both systems depends strongly on an applied electric current/field. This influence is seen in a wide temperature range up to the room temperature.

The pronounced current dependence of the low temperature resistivity in $\text{La}_{0.82}\text{Ca}_{0.18}\text{MnO}_3$ has been interpreted as a signature of spin polarized tunnelling through spontaneously formed intrinsic tunnel barriers [4]. The nonlinear conductivity at high temperatures is a hallmark of persistence of the ferromagnetic domains in a paramagnetic matrix at temperatures much higher than the Curie temperature T_C . The existence of small magnetic clusters of the size of 12 \AA well above T_C in $\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ has been directly verified by small-angle neutron scattering combined with magnetic susceptibility and volume thermal expansion measurements [22].

Strong and weak external stimuli will create different metastable states in low doped manganites [5]. The dominant effect of the weak currents consists likely in a reversible injection of spin-polarized carriers into specific ferromagnetic domains. Charges accumulated at phase boundaries of the ferromagnetic metallic regions can be driven by electric field/current and literally pull the boundaries into insulating regions thus rising the volume of the metallic phase [23]. On the other hand, strong currents, as those appearing in the pulse treatment, most likely act through the associated strong electric fields. Such fields are capable of inducing local changes to the orbital order in a less conducting phase and/or changing the overall topology of an insulating phase and the domain-wall configuration.

Our experiments show that metastable resistivity states induced by the pulse treatment can be erased by thermal processing at $T > T_e \approx 360$ K. The experimentally determined memory erasing temperature T_e coincides closely with the upper temperature limit for the Q_2 structural distortions established from the structure measurements [24]. T_e coincides therefore with the upper temperature limit of the coexistence of the orbitally ordered and disordered phases. The memory erasing temperature T_e is also very close to the critical temperature T_{sp} of the vibronic ferromagnetic state marking to the upper limit of the spinodal phase segregation into hole-rich ferromagnetic vibronic clusters within paramagnetic orbitally ordered or disordered matrix [2].

Recently, Ahn et al. [25] suggested that the micrometer-scale PS in manganites is self-organized by intrinsic energy landscapes containing hierarchical energy barriers for relieving of the strain. Application of a sufficiently strong electric current/field may shift domain boundaries in a metastable pinning landscape and cause pronounced conductance changes. Domain walls pinned at

boundaries between ferromagnetic phases with different orbital order can be directly coupled to the strain fields in the sample. According to the above model the domain formation is self-sustained. External stimuli may influence the topology of the coexisting metallic and insulating regions and cause significant changes in the transport properties. Consequently, memory erasing temperature T_e can be identified with the temperature at which the intrinsic energy landscape with hierarchical energy barriers coupled to the strain fields is formed, or as a temperature at which thermal fluctuations start to exceed the pinning energy, such that at $T > T_e$ the domain walls are not effectively pinned by the hierarchical pinning landscape.

The memory erasing temperature T_e can be alternatively identified with the Griffiths temperature T_G [26]. In his original work Griffith considered a percolation-like problem for a random Ising ferromagnet in which some of the lattice sites are occupied by magnetic ions, whereas the remaining ones are either empty or occupied by non-magnetic atoms. The existence of the high temperature scale with $T_G > T_C$ and appearance of the Griffith phase in the temperature range $T_C < T < T_G$ was latter related to the very mechanism of CMR [1]. In the context of the quenched disorder scenario T_C corresponds to the magnetic transition temperature in a system with disorder while T_G corresponds to the Curie temperature of the undiluted system. In the context of a general scenario of clustered states T_G can be associated with the temperature at which clusters start to form.

Reports on the systematic experimental investigations of the Griffith phenomenon are very rare and started to appear only very recently. Generally T_G is determined as a temperature of a sharp downturn in the temperature dependence of the inverse of the magnetic susceptibility $\chi^{-1}(T)$ above T_C [27–29]. T_G has been also determined from the heat capacity data [27] and electron spin resonance spectra [29]. The experiments show that the Griffith temperature of the $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$ system is practically independent on the doping level, at least for $x < x_C$ [29]. This result is consistent with our experimental observations that the memory erasing temperature T_e is practically identical for both $x = 0.18$ and $x = 0.2$ LCMO. Moreover, the Griffith temperature for $\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ was found to be $T_G = 376$ K, a value very close to our $T_e \approx 360$ K [27].

The fact that our sample is slowly aging at room temperatures and evolves towards higher resistivity is a direct consequence of the fact that all the threshold temperatures discussed above, T_{sp} , T_G , and the upper limit of the order-disorder phase coexistence are higher than the room temperature. Therefore a sample maintained at room temperatures can be already in a metastable state prone for aging. On the basis of the currently available data we cannot discriminate which from the above discussed mechanism is responsible for aging processes. This issue requires more experimental work with a new theoretical guidance.

The data clearly indicate that at room temperatures the conducting ferromagnetic phase is much less stable

than the insulating one. Applied electric pulses and aging at room temperature appear to enhance a decay of the more conductive orbitally disordered metastable state toward the thermodynamically more stable orbitally ordered insulating FM state. Double exchange interactions in the ferromagnetic metallic state arise from the itinerancy of the charge carriers. Therefore, any reduction in one-electron bandwidth W may destabilize the ferromagnetic metallic state. It is worth to point out to a close similarity of our results with those of Tokura et al. obtained for $(\text{NdSm})_{0.5}\text{Sr}_{0.5}\text{MnO}_3$ system. The ferromagnetic metallic state at $T < T_C$ was found to be mostly metastable in nature, and existing at low temperatures only as an under-cooled state [30].

In summary, the existence of a variety of metastable resistivity states may be attributed to a coexistence of distinct phases having different magnetic and orbital order at different temperature ranges. High resistive metastable state exhibits nonlinear conductivity in a wide temperature range from low temperature up to the room temperature and a positive magneto-resistance at low temperatures. The properties of HRMS of $\text{La}_{0.8}\text{Ca}_{0.2}\text{MnO}_3$ crystal resemble closely those of $\text{La}_{0.82}\text{Ca}_{0.18}\text{MnO}_3$ crystals. High resistivity metastable state can be created by a suitable relatively strong current pulse treatment. Once the system is driven into such a state its resistance becomes more stable and possesses a memory. These effects can be ascribed to different stability of the coexisting ferromagnetic phases. The metallic phase seems to be less stable what results in slow aging of the samples stored at room temperatures towards more stable higher resistivity state.

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