

# Nonlinear electrical characteristics of the low-bandwidth manganites $R_{1-x}Ca_xMnO_3$ ( $R=Pr,Nd,Ho,Er;x=0.3-0.5$ )

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We have investigated nonlinear electrical characteristics in low bandwidth manganites  $R_{1-x}Ca_xMnO_3$  with  $R=Pr,Nd,Ho,Er$  and  $x=0.3-0.5$ . In all these materials we observe strong nonlinear  $I-V$  characteristic that is manifested in negative differential resistance (NDR) and breakdown voltages  $V_{br}$  as low as few volts. These effects are accompanied by intense Joule heating that seems to be inseparable from the effect itself. We present different types of measurements with the aim to resolve the origin of the phenomena. Melting of insulating state is observed regardless of its origin—antiferromagnetic, charge ordered, or paramagnetic. This nonlinearity is found to have microscopic origin, indicating that it is some sort of percolation effect. Hysteretic effects also indicate that the heating is not the only cause of NDR. Enhancement of conductivity is proposed to arise from double exchange induced by local magnetic field.

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## INTRODUCTION

Manganese oxides, widely known as manganites, are studied for more than 50 years<sup>1</sup> but keep sparking scientific and technological interest even today. They became particularly popular after the discovery of giant/colossal magnetoresistance in 1990s. Recently, the scientific interest in manganites shifted to “electroresistance,” i.e., current/voltage induced nonlinear phenomena. First reports date 1997 when colossal decrease of resistance was found in  $Pr_{0.7}Ca_{0.3}MnO_3$  upon the application of large voltage.<sup>2</sup> Since then, a significant amount of publications report nonlinear current/voltage effects in manganite. Initially, investigations were limited to materials with charge order (CO) ground state<sup>3-8</sup> but later nonlinear effects are found in materials without CO.<sup>9-11</sup> Driven by potential applications, recent research has shifted to thin films and heterostructures.<sup>12-16</sup>

Manganites are known to exhibit complex interplay between spin, charge, and orbital degree of freedom resulting in rich phase diagram.<sup>1</sup> The general formula is  $R_{1-x}A_xMnO_3$  where  $R$  stands for rare earth (La, Pr) and  $A$  for any divalent atom (Ca, Sr). They crystalize in a perovskite structure with manganese ion in the center of (cubic) cell and  $R$  ions on the corners. The possibility of different valence of Mn ion ( $Mn^{3+}$  or  $Mn^{4+}$ ) enables continuous tuning with  $0 < x < 1$ . Electronic properties are largely influenced by ionic radii  $R$  and  $A$ . If these radii are larger, the manganese  $3e_g$  electron hopping bandwidth  $W$  is large, resulting in metallic behavior. This is the case for  $La_{1-x}Sr_xMnO_3$  (LSMO) and  $x > 0.3$ . Reducing the radii of  $R$  and  $A$  yields to the decrease of  $W$  and thus results in insulating behavior. Typical example is  $Pr_{1-x}Ca_xMnO_3$  (PCMO) and compounds with the rare earth ion following Pr in periodic table of elements (Nd, Ho, Er, ...).

Accordingly to historical discoveries of nonlinear characteristics in manganites, first interpretations involved melting of CO states. Together with the development of phase separation theory,<sup>1</sup> current was assumed to grow and coalesce metallic ferromagnetic domains in insulating CO background. Reports of electroresistance in systems without CO

undermined these interpretations. Recently, it was found that electrical current even destroys ferromagnetic metallic state in  $La_{0.8}Ca_{0.2}MnO_3$  (Ref. 16) leading to the insulating state.

One of the most important problems this research is facing are the effects of Joule heating. The currents necessary for nonlinear effects are always on the limit of Joule heating. This is especially true for thin films where even relatively small currents result in huge current densities and thus in significant local heating. Therefore, we have concentrated our research on bulk materials, either single crystals or polycrystal, hoping that we can eliminate the heating problem. This is the aim of this work: To distinguish intrinsic (if any)  $I-V$  nonlinearity in manganites to those originating in contact or thermal effects.

## EXPERIMENT

We have studied the wide range of low-bandwidth (insulating) manganites. We have concentrated on  $R_{1-x}Ca_xMnO_3$  compounds with increasing atomic number of rare earth ion ( $R=Pr,Nd,Ho,Er$ ). The bandwidth  $W$  in the above list is continuously decreasing which is reflected in the increase of corresponding room temperature resistivities from  $10^{-1}$   $\Omega$  cm for Pr to 300  $\Omega$  cm for Er sample. The samples examined were single crystals of  $Pr_{0.6}Ca_{0.4}MnO_3$  (PCMO) and polycrystalline  $Pr_{0.6}Ca_{0.4}MnO_3$  and  $Nd_{0.5}Ca_{0.5}MnO_3$  (NCMO) and  $(Ho,Er)_{5/8}Ca_{3/8}MnO_3$  (HCMO,ECMO). Details about the sample preparation can be found elsewhere.<sup>17</sup> PCMO and NCMO exhibit charge ordering at temperatures around 250 K that in resistivity curves can be seen as small anomalies.<sup>18</sup> HCMO and ECMO lack any ordering for  $T < 350$  K. In addition, we have also measured single crystals of  $La_{0.9}Sr_{0.1}MnO_3$  (LSMO) that is also insulating in the whole temperature region. Ferromagnetic and charge ordering transitions in the latter are below  $T=150$  K and only data above this temperature are presented here.

Polycrystalline samples are sintered in pellets of different sizes, the largest having dimensions of  $10 \times 3 \times 2$  mm<sup>3</sup>. Single crystals had comparable size of up to 15 mm<sup>3</sup>. By

comparison of single crystal and polycrystal data we are able to eliminate possible influence of grain boundaries as origin of the effect. All samples show negative magnetoresistance (MR), typical for mixed valence manganites. MR effect in the HCMO and ECMO sample is in the order of a few percent, while PCMO and NCMO exhibit colossal MR at low temperatures.<sup>18</sup> The contacts have been made on either pre-evaporated gold or directly with silver paste. They have been covering/embedding the whole cross section of the sample, aiming to achieve uniformity of current passing through the sample. Thus, the nominal current densities are many orders of magnitude lower than is the case in thin films. All of the above materials are semiconducting with the activation energy of  $\sim 0.1$  eV.

All the measurements reported here have been performed in four contact configuration. Two contact measurements are always hindered by low (electric) field nonlinearity due to the Schottky contacts.<sup>19</sup> In all the manganite samples measured in two-contact capacitance configuration, we have found large capacitance. This capacitance would always give unphysically high dielectric constant of the order of 1000. Additionally, relatively low bias voltages of up to 1 V would induce both the decrease in resistance and capacitance. Non-linear current-voltage characteristics and decrease of capacitance with bias are signatures of Schottky diode. Based on this finding, one should take extreme caution when interpreting two-contact measurements on thin films or heterostructures.

Few different experimental setups are used for current sources and voltage measurements. In one typical setup, current source is Keithley sourcemeter K2410 that also enables us to track 2-contact voltage (resistance). Separate voltmeter (K182 or K195) was used to measure 4-contact resistance. All the data in this report are presented in the “raw” format of current “ $I$ ” and voltage “ $V$ ” since the nonlinearity is non-uniform effect and effective cross section concept can be misleading. Different sweeps of current, voltage, or time are performed in order to distinguish intrinsic nonlinear effects from those coming from Joule heating. Additionally, measurements in Fig. 2 were done with the standard platinum thermometer sitting on the top of the large polycrystalline pellet.

**RESULTS**

As an example of nonlinearity typical for all manganites studied we start with the standard 4-contact measurements on HCMO material. HCMO is the paramagnetic insulator and lacks any sort of structural or magnetic ordering in the examined temperature window. Current voltage characteristic of HCMO is presented in Fig. 1. The data are taken for a set of currents while measuring the voltage. The temperatures are indicated in the figure. For small currents we obtain nearly Ohm’s law, but at higher currents the voltage saturates and even decreases. This gives negative differential resistance (NDR), similarly to results reported by other groups in PCMO.<sup>3,4,10</sup> Similar to Ref. 10, where NDR is attributed to enhancement of double exchange<sup>20</sup> by electrical current, our system does not exhibit charge ordering. Our finding indicate

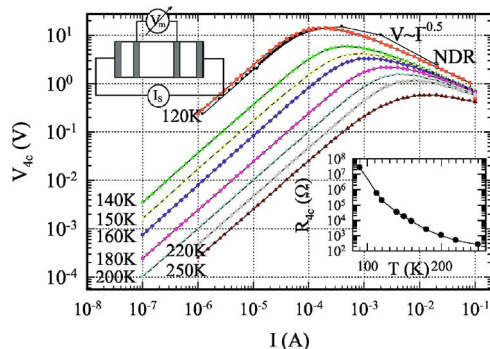


FIG. 1. (Color online)  $I$ - $V$  characteristics of  $\text{Ho}_{5/8}\text{Ca}_{3/8}\text{MnO}_3$  polycrystal measured at temperatures as indicated in the figure. The inset shows the temperature behavior of ohmic resistance of HCMO material. Contact configuration is shown in the upper left corner.

that NDR is a common effect for all manganites that are semiconducting/insulating. For HCMO, NDR is found as high as at room temperature (RT). This make it suitable for thorough tests of nonlinearity at RT.

During measurements presented in Fig. 1 we observed the time dependence of the voltage for each step of current. The voltage would need a few seconds to decrease to its equilibrium after each step of current. This induced us to study the time dependence of resistance in several different setups. Figure 2 presents one type of time dependence measurement that is particularly illustrative. Resistance is measured in 4-contact configuration with fixed voltage  $V_5$  set on current contacts (see inset of Fig. 2). Simultaneously we have mea-

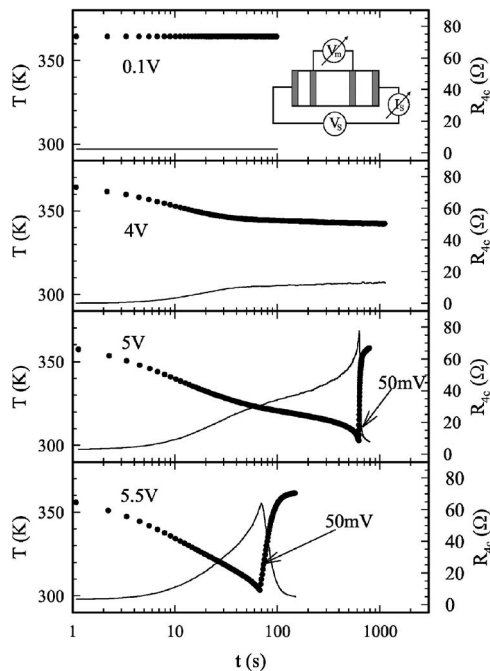


FIG. 2. Time dependence of resistance  $R$  (circles, right axis) for fixed voltages that are set on current contacts: 0.1, 4, 5, 5.5 V.  $R$  is calculated from both measured values: Current passing through the sample and voltage taken at voltage contacts. Lines are temperatures (left axis) of thermometer attached on the top of the sample. Polycrystal HCMO.  $T=295$  K.

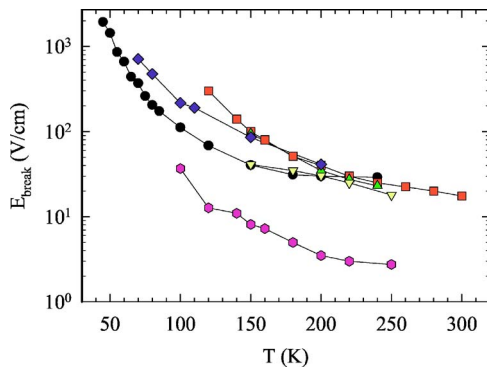


FIG. 3. (Color online) Breakdown electric field  $E_{br}$ , as a function of temperature  $T$  for different material: Polycrystal (black circles) and single crystal (blue diamonds) of PCMO, polycrystal of NCMO (pink hexagons), single crystal of LSMO (yellow triangles down), polycrystals of HCMO (red squares), and ECMO (green triangle up).

sured the current passing through the sample and the voltage on separate voltage contacts. The resistance obtained from these measured values is plotted versus time. Four voltages are shown in Fig. 2: 0.1 V (black circles) that gives perfectly Ohmic resistance, 4 V (red squares) that shows a finite decrease of resistance in time, 5 V (green triangles up), and 5.5 V (blue triangles down) that show divergence in current (vertical drop in resistance). At the point when the current reaches the compliance limit of the instrument (set to 0.3 A), the voltage is switched to 50 mV, i.e., to the Ohmic regime. This allows us to follow the relaxation after the application of high voltages. Simultaneously, the temperature (lines, left axis) of the platinum thermometer shows a drastic increase of the temperature of the sample. The heating of the sample can be assumed even more drastic since the sudden increase of heat at the point of current divergence cannot be adequately transmitted to a thermometer barely lying on the top of it.

From the results presented in Fig. 2 it is obvious that the current/voltage nonlinearity is associated with strong Joule heating. A larger current induces larger Joule heating, which in turns lowers the resistance and increases the current. This leads to an avalanche and finally divergence of current at fixed voltage. This is analogous to NDR in Fig. 1—an increased value of current heats up the sample and voltage starts to decrease. Analogously, one can sweep the voltage  $V_S$  set on the current contacts and at certain breakdown voltage  $V_{br}$  ( $\sim 5$  V in the case of HCMO in Fig. 2) the current diverges. Such measurements have been performed for all the materials mentioned above. The breakdown electric fields from these measurements  $E_{br} = V_{br}/d$  ( $d$  is the length of the sample), that coincide with measurements shown in Fig. 2, are shown in Fig. 3 as a function of temperature. In all samples except NCMO  $E_{br}$  is falling almost on the same line while increasing with decreasing temperature. This increase follows rather closely the behavior of low-field resistance in all samples. Note that breakdown voltage is found in single crystals as well, which excludes the grain boundaries as the possible origin of the effect. Are these breakdown voltages just points where a heating avalanche starts or they corre-

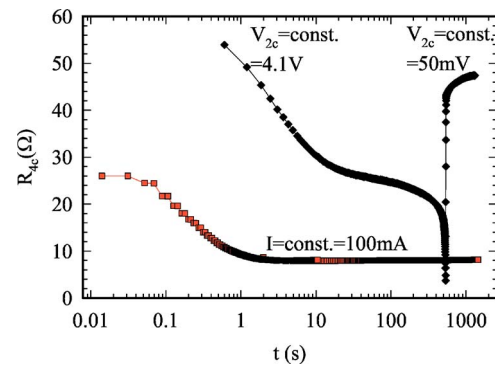


FIG. 4. (Color online) Constant voltage (black diamonds) and constant current (red squares) measurements on HCMO polycrystal. Contact configuration as in Figs. 2 and 1, respectively.  $T = 295$  K.

spond to some inherent microscopic process in manganites?

In Fig. 2 we saw that resistance decreases with time upon the application of constant voltage  $V_S$ . This current increase is accompanied by intense heating. In Fig. 4, we show complimentary time measurements on HCMO for fixed large current ( $I_{const} = 100$  mA, contact configuration like in Fig. 1). This is a different HCMO sample, therefore, the resistance and  $V_{br}$  are slightly different. The constant current and constant voltage methods yield to rather different response, but one that is easily explainable. When the current is fixed, the heating is constant and resistance does not decrease with time (at  $t > 2$  s). What is important in Fig. 4 is the fact that even at fastest d.c. measurements one cannot obtain low field (ohmic) resistance ( $68 \Omega$  in the case of this sample).  $R \approx 26 \Omega$  for fastest measurement at  $t = 10$  ms. This means that if the origin of nonlinearity is heating, it has to be local heating on the nodes of the current path. The whole sample (pellet  $10 \times 3 \times 1.5$  mm<sup>3</sup>) cannot heat up quickly to equilibrate the temperature in less than 10 ms. The decrease of  $R$  shown in Fig. 4 at  $t < 2$  s from 26 to  $8 \Omega$  is then supposedly the heating effect. However, this decrease will be seen in different circumstances, as shown below.

Measurements of the above nonlinear effects are inevitably influenced by thermal contact of the sample with its environment. However, we did not notice too much influence of thermal contact on breakdown voltages/electric fields presented in Fig. 3. The nonlinear data were reproducible for a few different thermal configurations. It looks like the breakdown voltage is not predominantly influenced by thermal contacts: The increase of temperature seems to be a consequence of large currents and not vice versa. To elucidate this, we intended to improve the thermal sink of the sample. One way is to dip the sample directly into the liquid nitrogen. Unfortunately, this limits our choice of materials: HCMO and ECMO had too large  $V_{br}$  at  $T = 77$  K to be measurable. Therefore, we turned back to CO materials PCMO and NCMO that have lower resistivity. Figure 5 shows the current voltage characteristic of PCMO single crystal dipped directly into liquid nitrogen.

Here, the usual configuration from Fig. 1 is used: The sweep of current ( $I = 0 \rightarrow 10$  mA  $\rightarrow -10$  mA  $\rightarrow 0$ ) is passed through the current contacts while measuring voltage on voltage contacts. NDR is observed for  $I > 3$  mA. The hyster-

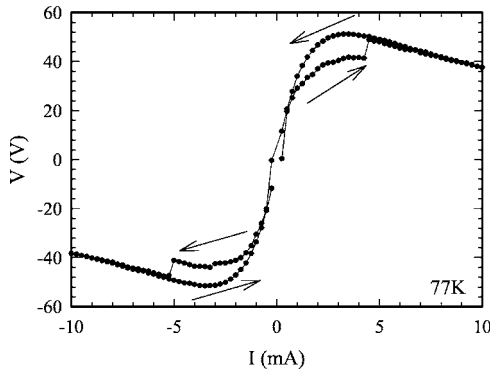


FIG. 5.  $I$ - $V$  characteristics for PCMO single crystal dipped in liquid nitrogen. Contact configuration as in Fig. 1. Current is swept  $I=0 \rightarrow 10 \text{ mA} \rightarrow -10 \text{ mA} \rightarrow 0$ .  $T=77 \text{ K}$ .

esis is found starting at currents as low as  $I=1 \text{ mA}$  ( $j=25 \text{ mA/cm}^2$ ). This hysteresis indicates that the effect is not related to heating problems. It is hard to imagine that the sample, once in NDR regime presumably from heating, would become more resistive (presumably cool down) as it happens at  $I=5 \text{ mA}$ . At  $I=1 \text{ mA}$  the applied power is  $70 \text{ mW}$ . This amount when dispersed over the mass of sample is several orders of magnitude smaller than in thin films.<sup>8,12,13</sup> It is important to note that we observe a very similar effect on PCMO polycrystal dipped in liquid nitrogen.<sup>21</sup> Interestingly enough, in polycrystal the hysteresis effect starts also at  $I=1 \text{ mA}$  ( $j=8 \text{ mA/cm}^2$ ), although applied power at that current was  $30 \text{ mW}$ .

In Fig. 6 we show fast time response of the same PCMO single crystal dipped into liquid nitrogen. Here we present voltage as measured on current contacts versus time upon the application of current. Values of currents in milliamperes are given in the figure. Data presented are 2-contact measurements, but are identical to 4 contact measurements in the time region  $t > 0.1 \text{ s}$  (lack of fast measurements for  $t < 0.1 \text{ s}$  is the reason why we present 2-contact data). In the case of “low” current ( $0.1 \text{ mA}$ ) one can see time independent response. With increasing current, voltage increases but becomes also time dependent. For the case of  $2 \text{ mA}$ , 2-contact resistance at  $\Delta t=10 \text{ ms}$  is  $V=175 \text{ V}$ . After approximately  $1 \text{ s}$  it lowers to  $V \approx 80 \text{ V}$ . Note the similarity with constant cur-

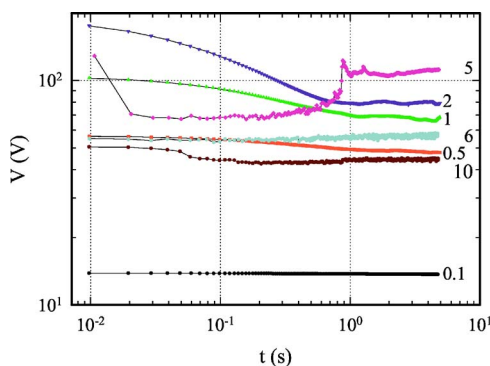


FIG. 6. (Color online) Time dependence of 2-contact voltage for a set of currents. Values of currents in milliamperes are given in the figure. Single crystal of PCMO.  $T=77 \text{ K}$ .

rent method trace of HCMO in Fig. 4, despite very different thermal environment in these two cases. With the current increasing further, the voltage starts to decrease even at  $\Delta t=10 \text{ ms}$ . This leads to  $\text{NDR}—V(10 \text{ mA}) < V(1 \text{ mA})$ . Note that these measurements are equivalent to those in Fig. 5. Even more, the instability/hysteresis region around  $I=5 \text{ mA}$  in Fig. 5 is reappearing as instabilities around  $5 \text{ mA}$  in Fig. 6. Similar instabilities as that for  $I=5 \text{ mA}$  in Fig. 6 are recorded at all currents between  $4$  and  $6 \text{ mA}$ . These instabilities, invoking discontinuous jumps toward higher voltages/resistances, are clearly opposite to what is expected from heating.

Let us estimate the incoming heating power in Fig. 6, assuming that all the incoming power goes into the heat. Consider traces of  $I=0.1 \text{ mA}$  and  $I=10 \text{ mA}$ . In  $\Delta t=10 \text{ ms}$  and with  $I=10 \text{ mA}$  the total incoming energy is  $\Delta E = V \cdot I \cdot \Delta t = 50 \text{ V} \cdot 10 \text{ mA} \cdot 10 \text{ ms} = 5 \text{ mJ}$ . Heat capacity of the PCMO system at  $T=77 \text{ K}$  is approximately  $30 \text{ J/mol/K}$ .<sup>22</sup> The sample in Fig. 6 has a mass of  $m=51 \text{ mg}$ . From our  $\Delta E=5 \text{ mJ}$ , the temperature increase  $\Delta T$  might be just up to  $\Delta T = \Delta E / (C \cdot n_{\text{mol}}) = 0.67 \text{ K}$ . Using  $R \sim \exp(\Delta/T)$  with activation energy  $\Delta \approx 1000 \text{ K}$ ,<sup>19</sup> one gets a ratio of high and low resistances  $R_1/R_2=1.12$ . This invokes that the pure heating of  $\Delta E=5 \text{ mJ}$  can lower the resistance from  $R_1(0.1 \text{ mA})=138 \text{ k}\Omega$  just to  $R_2=123 \text{ k}\Omega$ . On the contrary, measured  $R(10 \text{ mA})=50 \text{ V}/10 \text{ mA}=5 \text{ k}\Omega$  gives the ratio  $R(0.1 \text{ mA})/R(10 \text{ mA})=27.6$ . It is evident that heating is not responsible for NDR as observed in Fig. 6.

## DISCUSSION AND CONCLUSION

We have studied nonlinear current-voltage characteristics in low bandwidth manganites, whose temperature dependence of resistance is semiconducting. Our measurements show that all manganites in temperature range of their semiconducting behavior exhibit nonlinearity at high voltages, resulting in NDR. NDR is associated (if not influenced) by heating, as shown in the HCMO case (Fig. 2). Such a heating appears to be local, as indicated by Fig. 4. It is difficult to distinguish if nonlinearity in Fig. 2 is caused exclusively by local, microscopic heating on the preferable current paths or is triggered with some other inherent process. If one extrapolates the constant current curve in Fig. 4 toward shorter times, the low field (ohmic) resistance ( $R_0=68 \Omega$ ) of that HCMO sample would be met at times  $t < 100 \mu\text{s}$ . In accordance with this estimate, pulsed measurements on HCMO at RT have shown no nonlinearity for pulses shorter than  $100 \mu\text{s}$ .

However, measurements on PCMO at  $T=77 \text{ K}$  indicate that the origin of  $I$ - $V$  nonlinearities might be unrelated to Joule heating. In the tight thermal contact with the cryostat, PCMO material exhibits two intriguing effects, seemingly separated. First is negative differential resistance that in other cases/samples seems to be related to heating. The second is hysteresis in the current cycling.

As for the former, NDR in Fig. 5 occurs for the applied power of less than  $1 \text{ W}$ . No excessive boiloff of liquid nitrogen is observed during these measurements. Even more, upon the sudden application of a large current ( $\sim 10 \text{ mA}$ ,

Fig. 6) resistance of the sample, it switches to a low resistive state in less than 10 ms, when calculated incoming heat is by far insufficient. Further, fast measurements on PCMO single crystal show heating-like effects for intermediate currents (1 mA) but no such effects [constant  $V(t)$ ] for currents above critical 5 mA. The above arguments indicate that low resistive state at  $I > 5$  mA in Fig. 6 or NDR in Fig. 5 are not related to heating. Even more, if currents  $I > 5$  mA are not heating the sample, the curving at  $I = 1-5$  mA (at  $t < 1$  s) might come from inherent nonlinearity and not heating. This in turn questions the curving of constant current trace of HCMO presented in Fig. 4.

As for the latter effect, jumps from low resistive (LR) to high resistive (HR) states also indicate that the  $I-V$  curve in PCMO is not related to heating. Note that current induced LR  $\rightarrow$  HR transition is reported also in  $\text{La}_{0.8}\text{Ca}_{0.2}\text{MnO}_3$ .<sup>16,23</sup> Authors attribute it to the electroelastic effect,<sup>24</sup> i.e., lattice distortion that is coupled to the charge transport. Presently, we can only speculate if hysteresis in Fig. 5 is the same type of electroelastic effect like in the above cases. We might just add that the existence of the same hysteretic effect in both polycrystalline and single crystal PCMO indicates that effect is not related to defects (crystal twinning or grain boundaries).

Finally we should note that high-field nonlinear effects presented in this article are accompanied also with nonlinearity at much lower voltages/currents. PCMO, NCMO, and LSMO samples show significant  $I-V$  nonlinearity even when the applied power is much smaller than  $1 \mu\text{W}$  (or  $I < 1 \mu\text{A}$ ). This low field nonlinearity becomes more pronounced with the increase of the current and eventually leads to NDR and/or hysteretic effects like in Fig. 5. It is interesting to note, however, that HCMO and ECMO show almost a perfect Ohmic low at low currents. This invokes the most sig-

nificant differences between these two groups of materials: The former are either ferromagnetic (LSMO) or very susceptible to magnetic field (PCMO and NCMO) at low temperatures. The latter are paramagnets relatively insensitive to the magnetic field. However, HCMO shows even at  $T = 300$  K negative magnetoresistance of 4.3% at  $B = 9$  T. This indicates that it is indeed a manganite with double exchange. This relative insensitivity of HCMO to magnetic field (when compared to PCMO or NCMO) might be related to less pronounced electroresistance at low currents. In a naive picture, one can speculate that parts of the sample on the local path of the electrical current exhibit large local magnetic fields. This in turn enhances double exchange and consequently electrical conductivity. For PCMO and NCMO even small currents are sufficient for this effect. In HCMO currents necessary for electroresistance have to be much larger and the whole effect is screened by Joule heating.

To conclude, we have shown that strong current-voltage nonlinearities like NDR are often screened by Joule heating. This heating is shown to be local (microscopic). Existence of Joule heating at relatively low current densities in bulk samples (for HCMO sample in Fig. 2  $I = 100$  mA yields  $j = 1.7 \text{ A/cm}^2$ ) indicate that Joule heating is a much more severe problem for measurements on thin films. However, fast measurements and hysteretic effects in strict thermal environments indicate that heating is a consequence of inherent nonlinearities and not vice versa. This suggests that electroresistance is the consequence of double exchange enhancement common to all manganites.

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