Magnetoresistive dynamics and noise in low-strain manganite films

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ac magnetoresistance measurements of low-strain epitaxial films of $La_{0.7}Ca_{0.3}MnO_3$ on NdGaO₃ substrate show an out-of-phase response which switches sign near the metal-insulator transition, indicating at least two types of slow contributors to the magnetoresistance and suggesting the role of a third phase. These films show noise properties very distinct from films under tensile strain. A narrow peak in the noise found at the transition is non-Gaussian but lacks distinct persistent few-state fluctuators. Such fluctuators only appear some 10 K below the transition, indicating that conduction in the nominally metallic phase remains highly inhomogeneous. Comparison of discrete fluctuator sizes in a range of materials indicates that strain constraints limit the sizes in films.

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I. INTRODUCTION

The $R_{1-x}A_x$ MnO₃ series of perovskite manganties (with R a rare earth and A a divalent cation) has become a topic of increasing interest in recent years due to its wide variety of phases and the proclivity of some of these phases to coexist. The reasons for the stability of the phase coexistence are not yet fully understood, but evidence suggests that chemical disorder, structural disorder, and strain interactions can all play important roles.¹⁻³ The manganite La_{1-x}Ca_xMnO₃ (LCMO) is known to have a phase transition between ferromagnetic metal (FM) and paramagnetic insulator (PI) with an extended range of coexistence-i.e., FM clusters within a PM matrix near a transition temperature T_c . The growth of the FM clusters, either through decreasing temperature (T) or increasing magnetic field (H), eventually leads to a sharp increase in conductivity, potentially giving rise to the colossal magnetoresistance (CMR) found near T_c . Evidence that the inhomogeneities include another type of correlations, likely to be of the antiferromagnetic insulator (AFI) type, in bulk, nominally unstrained LCMO has been provided by extra periodicity found in transmission electron microscopy⁴ (TEM) and neutron scattering⁵ and by other analysis of neutron scattering.⁶

Such studies provide detailed spatial information, but do not provide much information on the dynamics. Internal friction measurements⁷ provide some evidence on dynamics, but without sensitivity to small regions. In this study, we use noise, including non-Gaussian effects, and ac magnetoresistance measurements to elucidate the dynamics in the phase coexistence regime. We find complicated results which do not follow expectations for a simple two-phase picture. We find distinct indications of AFI effects near T_c (as suggested by neutron scattering^{5,6}) and unmistakable evidence of strongly inhomogeneous current flow well below T_c .

We will conclude with a comparison of the noise results in these and other samples. A surprising approximate universality of fluctuating domain sizes across ranges of temperatures, materials, and types of domains in films, but not in bulk samples, strongly indicates that strain constraints are crucial in setting domain sizes.

II. EXPERIMENTAL METHODS

The sample is an "optimally doped" La_{0.7}Ca_{0.3}MnO₃ film grown on an untwinned 1-mm-thick NGO (001) substrate using laser ablation. Atomic force microscopy demonstrated atomic step-flow growth, indicating a coherently strained film.⁸ The growth details are outlined in Ref. 9. The low degree of lattice mismatch beween the film and substrate (0.1%) gives a T_c of approximately 260 K, which is also typical of bulk samples. The NGO substrate produces an nearly orthorhombic strain,¹⁰ unlike the tetragonal strain found in films on SrTiO₃. The film is grown to a 50 nm thickness and subsequently patterned into a 3.7-mm-long bridge (see Fig. 1) and a 2.1-mm wire. The wires in both cases are 50 μ m wide. Gold contact pads were then deposited using lift-off. Imperfections in the lithography leave the bridge unbalanced to approximately 1 part in 1000 at very low frequency.



FIG. 1. Shows the sample bridge geometry and schematically indicates the measurement circuit. The top amplifier provides a signal which can be linearly combined with the main cross-bridge voltage (middle amplifier) to nearly eliminate any contact noise. The bottom amplifier is used to measure net sample R.

Resistance fluctuations in the arms also randomly throw the bridge out of balance, allowing a measurement of the resistance fluctuation spectrum through

$$S_{AB}(f) = V_S^2 \frac{S_R(f)}{R^2},$$
 (1)

where R is the resistance of the bridge, $S_R(f)$ is the spectrum of its fluctuations, V_S is the voltage applied to the bridge, and $S_{AB}(f)$ is the spectrum of voltage fluctuations across the bridge. Equation (1) assumes that the fluctuations in the different arms of the bridge are independent. The role of the bridge is to cancel out current fluctuations, which can be significant due to the difficulty of making good electrical contact with the sample. Any current fluctuations that are not subtracted out by the bridge can, in large part, be removed in software by simultaneously monitoring the current fluctuations via the wire-wound series resistor shown in Fig. 1. This series measurement serves an additional purpose, allowing us to determine the sign of the ΔR of discrete fluctuations, which cannot be determined from pure bridge measurements, via the cross spectrum with the bridge imbalance voltage. The net voltage across the bridge was also monitored in a four-terminal arrangement for net R measurements.

Since, with the exception of some discrete individual fluctuators, the resistance noise found had the standard nearly 1/f spectrum, we can characterize its magnitude by a single number:

$$v_N \equiv v_S \frac{fS_R(f)}{R^2},\tag{2}$$

where v_S is the volume of the sample in the bridge. This normalized form facilitates comparison of noise magnitudes between samples with different sizes and geometries.

Current was provided via lead-acid batteries and a series resistor. The voltage fluctuations were amplified via SR550 preamplifiers before being sent to a SR640 low-pass filter for antialiasing and further amplification. The resulting voltage timetraces were then read via an analog-to-digital converter (ADC), with all subsequent analysis performed using homebuilt software. T was measured with an Si diode, with reproducibility of about 10 mK. Although the absolute accuracy is not that good, we give T's to high precision purely for internal comparisons.

The magnetoresistance measurements were taken on the single 2.1-mm wire using a standard four-probe geometry. The sample was driven at 10 μ A of constant current. To probe the frequency response, a 650-Oe dc magnetic field with a small added ac component was applied along the direction of current flow, using a standard room-temperature electromagnet. The resulting ac sample voltage was amplified and sent to a lock-in amplifier. Using a Hall effect sensor, the magnetic field was monitored real time and used as the reference input to the lock-in amplifier. Identical filter chains were used for the signal and reference, to avoid phase shifts, and it was checked that the amplifiers and filters gave no significant relative phase shift. This dc field biases the sample well above its main coercive field of ~50 Oe.¹⁰ We define a complex ac magnetoresistive coefficient by



FIG. 2. (Color online) R(T) and $v_N(T)$ are shown at H=0 in part (a) and at H=3700 Oe in part (b). High-*T* points were taken on averaging at fixed *T*, rather than on continuous *T* sweeps, because of low sample noise-to-background ratios.

$$R(t) - R_0 = \chi'_R H_{\rm ac} \cos(\omega t) + \chi''_R H_{\rm ac} \sin(\omega t), \qquad (3)$$

where the applied field is of the form $H_0+H_{ac} \cos(\omega t)$. In the particular measurements presented, we used a 2.3-Oe (rms) ac field at 5 Hz. This low frequency slows data taking but avoids phase shifts due to cable capacitance.

III. RESULTS AND INITIAL ANALYSIS

We first outline some qualitative features of the results, before examining aspects of them in more detail. The metalinsulator transition is sharp and little shifted from the bulk transition temperature T_c of approximately 260 K as found previously for similar films on NGO (Refs. 10 and 11) and as expected from the good lattice match (<0.1%) to the substrate. Figure 2 illustrates the R(T) and the noise magnitude versus T. Unlike in a variety of different film samples on subtrates which produce more tensile strain, a narrow peak appears in the low-frequency conductance noise at T_c . Previous noise results11 on LCMO on NGO found a peak of similar width and normalized magnitude. However, we find excess noise, which we shall show is associated with CMR effects, for $T < T_c$, in contrast to the previous findings¹¹ of no detected CMR noise below the peak but substantial fielddependent noise above the peak. Above T_c the 1/f noise in our samples drops to very low levels, not distinguishable



FIG. 3. The in-phase and out-of-phase responses of the sample voltage to ac magnetic field at 5 Hz, with an rms amplitude of 2.3 Oe, in a dc field of 650 Oe parallel to the current. The current bias on the sample is 10 μ A. Just near T_c , a small out-of-phase response of anomalous sign appears. The sample was cooled at H = 0 and data taken on warming in field.

from a variety of metals or highly doped semiconductors with no special domain effects.¹² Much of the noise below T_c appears in many narrow peaks in $v_N(T)$, which we shall see arise from discrete switchers.

The effect of moderate magnetic fields on the noise is somewhat surprising. At 3700 Oe, the main noise peak in $v_N(T)$ broadens, reduces in height, and shifts to lower *T*, even though the effects on R(T) are small, as illustrated in Fig. 2. That is very difficult to explain in a simple twocomponent picture of the transition, since the noise peak occurs at different points in the R(T) curve for different *H*. Although the maximum temperature at which the switching noise is found decreases in the particular data set shown, that result was not consistently reproducible.

When *R* is a unique function of the net magnetization at some fixed *T*, the noise associated with magnetic fluctuations can be related to χ_R'' by a fluctuation-dissipation relation.¹³ Figure 3 displays both components of $\chi_R(T)$, measured at 5 Hz. χ_R' peaks near the temperature of maximum dR/dT(nominally T_c) and tails off smoothly in both directions. Its high-temperature tail, though small, extends well above T_c . Prior evidence indicates that small FM clusters form in this range, as seen in ultrasonic measurements¹⁴ and neutron scattering.¹⁵ χ_R'' tracks χ_R' over much of the temperature range, but shows a dramatic deviation near T_c , with χ_R'' changing sign. Just above T_c , χ_R'' leads rather than lags the in-phase response.

We checked carefully to make sure that this anomalous out-of-phase magnetoresistance was not an experimental artifact. The out-of-phase result is not affected by nulling the lock-in input with the reference signal, ruling out phase error in the lock-in. The only way the measurement circuit could give rise to a *T*-dependent phase shift would be via RC effects in the sample-cable-amplifier portion. In these 5 Hz measurements, we calculate that such effects should be negligible. At any rate, since the anomalous leading response occurs in the region of high *R*, changing to a more typical lagging response in the low-*R* range, the effect has the wrong sign to be due to such capacitative phase shifts.

Thus the data give evidence of a slow positive MR response in the same regime where the MR is mainly fast and negative. Since the second law of thermodynamics requires that the out-of-phase magnetic susceptibility $\chi''(\omega) > 0$ for all ω , *R* cannot be solely a function of the magnetization. In this regime, the magnetization is expected to closely follow the FM volume fraction, at least for $H > H_c$, so it seems that *R* is not simply a function of the FM volume fraction. One might fit the positive χ''_R and the negative χ'_R in a two-phase FM-PI model, by postulating that the response to an increase in *H* is an initial increase in FM volume followed by a slow decrease in connectivity of FM domains, but such postulates would seem contrived.

A more plausible scenerio is provided by the inclusion of the AFI regions suggested by previous work.^{4–6} In this threecomponent picture, the equilibrium phase-conversion kinetics is characterized by two different relaxation times. If the slowest process is the growth and shrinkage of the AFI component, we believe that the anomalous χ_R'' would be produced by strain constraints. If we ignore anisotropy, the partial strain constraint created by the substrate means that an increased amount of the densest phase, FM,¹⁶ shifts the equilibrium between the other two components to favor the least dense, AFI,⁶ which also is the most insulating. It would be



FIG. 4. The noise power in a fixed bandwidth is shown as a function of *H* history at T=264.5 K. All noise data were taken at H=0. A well-defined field scale of about 100 Oe is required for switching the noise properties, which are not symmetrical under field reversal within the field range used.



FIG. 5. (Color online) Noise magnitudes in the 6-11-Hz band taken at several *T* show large slow fluctuations. Magnitude fluctuations for Gaussian noise under these averaging conditions would be only about 1%. The fluctuations were uncorrelated with *T* drifts of the cryostat, which at any rate were more than an order of magnitude too small to be relevant. In a narrow *T* window—e.g., 265.77 K—the noise magnitude shows continual random drift.

expected that this anomalous response would be masked by ordinary slow components of the main FM-PI CMR response at lower T, since an abrupt decrease in suspected AFI volume fraction below T_c has been reported.⁶

Noise data taken in the same regime where the anomalous $\chi_{R}^{"}$ is found provide further evidence of a very slow domain dynamics, probably also due to AFI regions. Figure 4 shows the noise magnitude (in a fixed bandwidth) measured at H=0 after the sample is cycled to some field (H cycle). The data show clear dependence of the resistance noise on field history, with at least two different noise signatures. Fields of under 100 Oe have little effect, but fields of over about 200 Oe substantially change the noise. Increasing the field to thousands of Oe has little further effect. The sign of the field matters, so the overall sample state breaks time reversal symmetry in a way that cannot be undone even by fields well above the coercive field. Thus there is a magnetic order parameter (the only plausible sort that would break timereversal symmetry) which is associated with much larger coercive fields than those found for the ferromagnetism. That amounts to a standard signature of antiferromagnetism.

The noise results provide further information on the magnetic structure, although at present only some of the interpre-



FIG. 6. (Color online) Time traces for an individual fluctuator at T=195 K for three different H are shown.



FIG. 7. Approximate values for the dimensionless entropy difference between the states are shown for a collection of two-state fluctuators. These values are estimated to an accuracy of typically a factor of 1.25 from the T dependence of the noise magnitude.

tation is clear. The fluctuations in noise power are much larger and much slower than found for Gaussian noise,¹² as shown in Fig. 5. Just above the temperature of the anomalous out-of-phase ac magnetoresistive feature, the noise magnitude shows particularly large and persistent slow fluctuations. Unlike noise in similarly doped LCMO bulk samples or other films,^{17–20} no discrete persistent identifiable fluctuators were found in or near this peak.

Unlike in previous reports on LCMO on NGO,¹¹ we find a collection of two-state switchers starting at about 235 K and extending to lower *T*, seen as spikes in the noise in Fig. 2, one of which is illustrated in Fig. 6. These do not merely represent a change in statistical properties of the noise but rather a new noise source above the level present in the range from 235 K to 250 K. We performed thermodynamic measurements^{18,20,21} using the dependence of the Boltzmann factor giving the ratio (*r*) of occupancy between the two states to obtain the differences between them in magnetic moment, $\Delta\mu$, and dimensionless entropy, $\Delta\sigma$:

$$\Delta \mu = -kT \frac{d \ln(r)}{dH}, \quad \Delta \sigma = T \frac{d \ln(r)}{dT} + \ln(r). \tag{4}$$

Each switcher shows up as a peak in $v_N(T)$, with a width of approximately $T/\Delta\sigma$. Figure 7 shows a collection of these



FIG. 8. (Color online) A comparison of the ratios of changes in entropy and magnetic moment ratio for a collection of fluctuators from this and other works (Refs. 18–21) on similar compositions of LCMO and approximate thermodynamic parameters from bulk material (Ref. 22). "STO" films are on $SrTiO_3$ substrates.

 $\Delta\sigma$ values. In some cases we also measured the field dependence of the noise magnitude, from which one obtains $\Delta\mu$, the magnetic moment difference between the two states, via Eqs. (4).

The ratio $\mu_{\rm B}\Delta\sigma/\Delta\mu$ (where μ_B is a Bohr magneton), which can be used to distinguish between magnetic rotations and fluctuations of the FM volume, is shown in Fig. 8, along with results from some other related samples and bulk thermodynamic properties²² for comparison. In all cases checked, this ratio was fairly typical for fluctuations of the FM volume.

In previous work on film samples under tensile strain, we found occasional cases in which the high resistance state had lower entropy and/or higher magnetic moment than the lower-resistance state, opposite to expectations for simple FM-PI fluctuations.¹⁸ We argued that the substrate strain constraints favor anticooperative changes in which some region goes FM while some adjacent region goes PI.¹⁸ The thermodynamic parameters depend on the *net* volumes of these regions. However, because of the strong inhomogeneity of the current flow, the resistance change can sometimes be more sensitive to the minority phase change-—a sort of "electoral college" effect. Here we found one fluctuator with a typical $\mu_B \Delta \sigma / \Delta \mu$, but with the anomalous sign of ΔR , confirming that such effects are widespread in film samples.

The kinetic pathway for the fluctuators is also of some interest. Although the ratio of the lifetimes of the two states is uniquely determined by the thermodynamic differences between them, the separate lifetimes depend also on the transition state or states. By separately measuring the dependence of the lifetimes of the two states on *T* and *H*, we can see whether these transition states have intermediate values of μ and σ , as would be expected if the mechanism is motion of a domain wall.²¹ The results on several fluctuators analyzed here were similar to those found in bulk, with domain wall motion.

The most striking feature of the two-state fluctuators is that they can be detected individually. As in a variety of other samples, the typical $\Delta R/R$ is orders of magnitude larger than the volume fraction of the sample inferred from $\Delta \mu$. It seems impossible to explain such effects without a very inhomogeneous current density^{18,20}—not a surprise very close to T_c but not otherwise expected in the midst of the FM regime. This current inhomogeneity suggests that there should be some domain structure well into the FM phase that remains important for the transport properties. Generic domain structures are expected to show aging effects,²³ and these would be expected to show up more dramatically in noise properties than in net resistance. Figure 9 shows the aging of the noise at 86 K over a period of hours. The aging is very substantial, including both a systematic component and random components, expected for non-Gaussian noise with large contributions from individual sites.

The symmetry under field changes of the noise also provides some evidence concerning the domain structure. As shown in Fig. 10, it is common for the same fluctuator to appear at both +H and -H on sweeps up to 2.5 kOe. Thus in this regime any breaking of time-reversal symmetry can be undone by these relatively small fields, in contrast to the results near T_c .



FIG. 9. (Color online) The aging of the noise power in two octaves after dropping T to 86.5 K is shown. R dropped by about 1% over this period.

IV. DISCUSSION

The ac magnetoresistance, noise, and aging experiments all point to more complicated dynamics than have usually been considered. We can offer at least partial explanations for the observations.

Near T_c both the anomalous out-of-phase ac magnetoresistance and field history dependence of the noise indicate that AFI components play a significant role. In particular, the detailed noise properties show broken time-reversal symmetry not reversible by fields of a few kOe, well above the FM coercive field.

The existence of a sharp noise peak at T_c seems to be a consistent difference between samples on NGO and films under more tensile strain, regardless of deposition method. At the peak in $v_N(T)$, there is a stable quenched background which breaks time-reversal symmetry. However, the fluctuations in this peak do not break up into persistent two-state components, unlike other noise results near T_c .^{18,19,21} This distinction suggests that the role of quenched disorder is smaller in the NGO samples than in others, even single-



FIG. 10. (Color online) The noise volume is shown on successive field sweeps at T=198 K. Each sweep took 1000 s. Although there are distinct changes over time, in many instances the same fluctuators appear at +*H* and -*H*. *H_c* appears as a spike near 50 Oe. The scale refers to the top trace, the others being shifted for viewing.

crystal samples made by atomic layer-by-layer epitaxy¹⁸ and bulk single crystals.²¹ It is possible that the slow fluctuations in noise magnitude just near T_c are closely connected to the anomalous slow magnetoresistance. We suspect that the lownoise window below T_c found in our NGO samples may be lost in samples under tensile strain because in them the AFI correlations should be more prominent over a broader range of T.

Below T_c , noise results demonstrate that the current distribution remains highly inhomogeneous in the nominally FM phase, since discrete fluctuators can show many orders of magnitude larger effects on R than they would for homogeneous transport. Unlike some of the fluctuators found near 20 K in other films,²⁰ the discrete fluctuators found near 200 K here have significant entropy changes, indicating that they are largely switches between different phases, not magnetic rotations. Concerted fluctuations exist in which FM to PI conversion of one region is accompanied by PI to FM conversion of another, as expected on the basis of substrate strain constraints. The inhomogeneous current distribution is likely associated with the same sort of domain organization which gives rise to aging effects in the noise. The partial symmetry of detailed noise versus H results indicates that well below T_c there are no substantial AFI regions giving broken time-reversal symmetry impervious to applied fields in the kOe range, in contrast to data taken near T_c .

The low-noise window just below T_c is a bit surprising on the basis of previous noise measurements, 11,17-19,21,24-29 but strongly reminiscent of the pattern of internal friction versus T found in bulk polycrystalline material with similar composition.⁷ For that material, the frequency dependence of the internal friction showed that it arose from thermally activated processes, for which motion of boundaries between FM and PI domains was the proposed mechanism.⁷ That idea is consistent with our results, but leaves open the explanation for the slow dynamics at the main noise peak at T_c . We suggest that the slow dynamics at T_c is promoted by the AFI regions, which give nearly static breaking of time-reversal symmetry. If, as previously claimed, these regions largely disappear just below T_c , low-frequency noise and friction would not reemerge until T were low enough to again give a more nearly stable domain structure.

V. FLUCTUATOR SIZES: COMPARATIVE DISCUSSION

It is becoming increasingly difficult to dismiss as a coincidence the similarity of the sizes of the fluctuating domains in a variety of regimes of different manganite films. The change in magnetic moment associated with the discrete fluctuators found here is about $6 \times 10^4 \mu_B$, indicating a fluctuation volume of about 3×10^4 unit cells. This turns out to be remarkably similar to many other film results and quite different from similar discrete fluctuations in bulk material, which can have much larger sizes—e.g., 6×10^5 unit cells.²¹ There are five or six distinct cases in which about the same size scale arises in films. These include

(i) fluctuations between FM and PI states near T_c in atomic layer-by-layer single-crystal LCMO films on SrTiO₃ (STO),¹⁸

(ii) similar effects to (i) in laser-ablated films on $LaAlO_3$,¹⁹

(iii) fluctuations of magnetic orientation near 20 K (far below T_c) in atomic layer-by-layer (ALL) single-crystal LCMO films on STO,²⁰

(iv) fluctuators near 20 K in laser-ablated films on ${\rm LaAlO}_3,^{19}$

(v) the fluctuators found here in ablated films on NGO in a regime somewhat below T_c but far above the 20 K regime, and

(vi) fluctuators both at and below T_c in new data (to be described in the thesis of A.P.) on films like those described in this paper but subject to damage from an electron beam.

In addition, the field scale for changing the noise at T_c in the films described here was comparable to that for the discrete fluctuators, although since no discrete fluctuators were identified it is hard to be sure that corresponds to a domain size.

Thus there should be some reasonably general explanation for the size scale in films. This explanation should be thermodynamic, since if the scale were simply determined by post-selection of some range of kinetic barriers via the choice of experimental frequency range, one would expect similar results for the bulk. Anticooperative effects due to clamping of strain changes by the substrate^{18,30} provide an obvious way by which the film thickness can limit coherence lengths for a variety of different fluctuation types, without any fine-tuning of disorder. To reiterate the argument previously made for FM-PI fluctuations,^{18,30} if two different states have different in-plane strain, then the growth of one state favors the growth of the opposite state in its vicinity. In other words, regardless of the linear term in the free energy due to average stress (which shifts the temperature of phase equilibria) there will be a term which is quadratic in the effective stress on the substrate. When the free energies of the two pure states, including the linear strain term, are tuned via T and H to be about equal, the quadratic strain term favors an intermediate strain state, with textures on scales smaller than the film thickness. In mixed-domain regimes, that term will be minimized by forming a mosaic on distance scales shorter than the distance to the substrate, at most the thickness of the film. Roughly similar mosaic distance scales should emerge for a variety of different fluctuation modes, as long as their strain change is large enough.

We believe that this collection of data thus provides strong evidence for the importance of strain effects in limiting coherence sizes in films. The larger coherence sizes found in bulk cannot have precisely the same origin. However, the manifest importance of strain effects on mesoscopic texture in films supports pictures of more complicated martensitelike strain effects in bulk.^{31,32}

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