Strongly inhomogeneous conduction in cobaltite films: Non-Gaussian resistance noise

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Strongly non-Gaussian conductance fluctuations are found in relatively large (e.g., 1×10^{-10} cm^3) samples of (001)-oriented ~50-nm-thick epitaxial films of the nominally ferromagnetic and metallic perovskite cobaltite $La_{0.5}Sr_{0.5}CoO_3$. Field dependences of Boltzmann factors of individual fluctuators show them to be magnetic regions with of order 1×10^4 Bohr magnetons. Given the size of the fluctuating regions, the size of the discrete conductance fluctuations would be anomalously large unless the conductance were strongly inhomogeneous as expected for magnetoelectronic phase-separation effects known to occur in some bulk cobaltites at lower concentrations or with polycrystalline structure. Temperature dependences of Boltzmann factors indicate small energy and entropy changes, more like magnetic rotations than like fluctuations between phases. The films, like bulk samples of the same composition, show resistance increasing with temperature in Van der Pauw measurements; but resistance in strip-shaped regions weakly decreases with temperature, indicating particularly extreme effects of the inhomogeneous magnetic orientation dependent conductivity.

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The doped perovskite cobaltites, e.g., $La_{1-x}Sr_xCoO_3$ (LSCO), form complicated magnetoresistive materials with inhomogeneous magnetic and electronic phases.[1,2] Prior experimental work using techniques as varied as bulk probes[2,3] transmission electron microscopy (TEM),[4] neutron diffraction,[4] Co (Ref. 5) and La NMR,[6] small-angle neutron scattering,[7] and inelastic neutron spectroscopy[8] has conclusively proven that these materials can undergo magnetoelectronic phase separation. At low doping ($x \ll 0.5$) the system forms nanoscale clusters of ferromagnetic (FM) metal embedded in a nonferromagnetic matrix. As x increases these clusters increase slightly in size and greatly in density leading to coalescence into a long-range ordered ferromagnetic network and a coincident percolation metal-insulator transition.[2–8] In bulk, many details of the ferromagnetic clusters (e.g., their size[7,8] and their influence on transport properties,[7,9] and their formation above the Curie temperature[10]) are well established, but the nature of the nonferromagnetic matrix is still under debate.[11] Polycrystallinity can induce phase inhomogeneity even at $x=0.5$,[2] for which single crystals are nominally single phase.[12] Very little is known about the effect of dimensional confinement (e.g., growth in thin film form) on these magnetic phase-separation effects.[13]

At high x standard Van der Pauw (VdP) measurements of resistance (R) vs temperature (T) on bulk crystals indicate metallic conductivity, i.e., monotonically increasing $R(T)$ at low T,[2,12] suggesting that metallic regions percolate through the material as expected from the above discussion. In this paper, we present non-Gaussian resistance noise results on epitaxial films with $x=0.5$, finding that small magnetic regions have disproportionately large effects on R, as often found in materials with very inhomogeneous conductivity, e.g.,[14,15] similar to polycrystalline bulk samples and to lower-x bulk single crystals. More surprisingly, we find that $R(T)$ measured in patterned samples with narrow-strip conductance paths is a *decreasing* function of T, in contrast to $R(T)$ measured by the VdP method on the same films. We conclude that the unusual conductance properties of these films probably result from insulating domain walls in a three-dimensional domain structure driven by strain constraints.

45–55-nm-thick epitaxial (001)-oriented $x=0.5$ LSCO films were deposited on $SrTiO_3$ (001) substrates by high-pressure off-axis reactive dc magnetron sputtering. Details of fabrication, structural characterization (by various modes of x-ray diffraction, x-ray reflectivity, TEM, and atomic force microscopy), and magnetic measurements (magnetometry, magnetotransport, and polarized neutron reflectometry) have been described elsewhere.[13] Briefly summarizing the characterization results, these films are epitaxial and oxygen stoichiometric[13] with several properties (Curie temperature $T_C=240$ to 250 K, magnetization at high field, and VdP resistivity) close to their bulk values.[13] Unfortunately substrate backgrounds have prevented checking for saturation in the dependence of the film net magnetization on high fields and create a few percent uncertainty in the magnetization values.

As-deposited films contain a small amount of CoO impurity phase, which can then be removed by oxygen annealing.[13] They also contain low-angle grain boundaries extending through the film thickness with a density of roughly several per micrometer not affected by annealing.[13] Annealing has negligible effects on the resistivity and magnetization.[13] Annealing does slightly change the lattice parameter consistent with minor strain relaxation.[13]

For this study, the samples (both annealed and as-deposited) were patterned in standard eight-probe or six-probe patterns as illustrated in Fig. 1 using ion milling in some cases and simple scratching in others. The ion milling used mechanical masks to avoid any heating or chemical effects associated with lithography. In these patterns the current and the voltage probe paths can be constrained to run down a relatively narrow shared strip similar to two-probe measurements but without contact artifacts since the paths

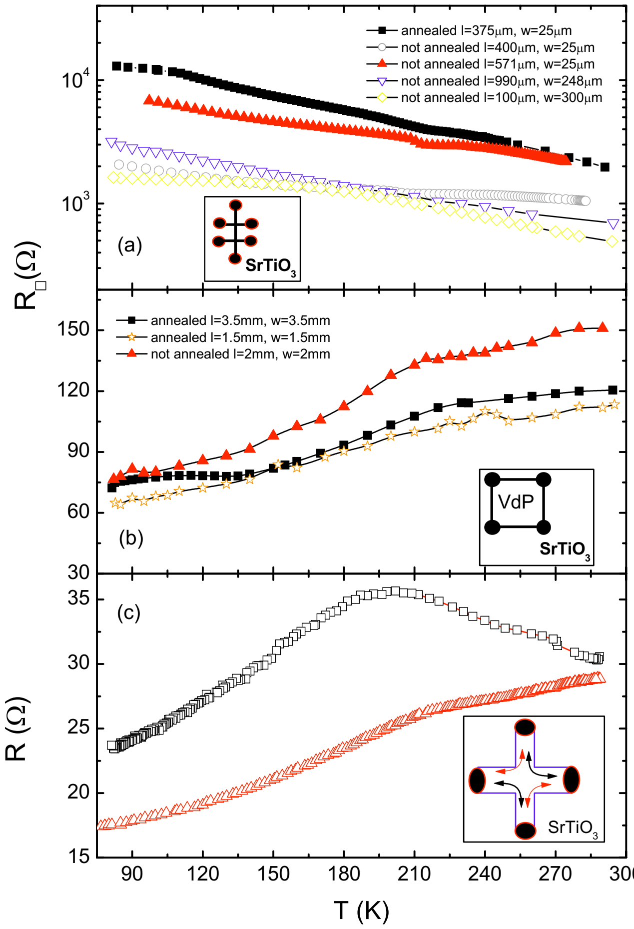

FIG. 1. (Color online) Resistance as a function of temperature measured in several $La_{0.5}Sr_{0.5}CoO_3$ samples employing the configurations shown in the insets: (a) a conventional four-probe technique (on six-probe or eight-probe strip patterned samples), (b) a Van der Pauw method, or (c) four-probe cross measurements, using the two different voltage-current pairings on part of the sample shown as open triangles in (a). The data represented by solid symbols in (a) and (b) were obtained by simultaneous measurements of *both* strip and VdP configurations on the same films.

diverge before reaching well-separated current and voltage contacts.

Measurements of $R(T)$ are shown (Fig. 1) both as taken using patterned strips [Fig. 1(a)] and via the VdP method [Fig. 1(b)]. In every case the VdP $R(T)$ was an increasing function, i.e., metallic looking, in the range from 80 to 290 K just as found in similar measurements on high-x bulk crystals.[7,12] The VdP results were qualitatively independent of which of the two possible configurations were used for current and voltage lead pairs, although differences of up to a factor of two appeared in the value of R. In every case, however, the patterned strip result gave a *decreasing* $R(T)$ regardless of whether the sample was patterned by scratching or by ion milling. The form of the strip $R(T)$ results was nearly independent of which inner voltage contacts were used when the outermost contacts were used for the current leads. Annealed samples as might be expected from the prior characterization behave like the as-deposited samples.[13]

Since we were concerned that the strip results might come from some sort of sample damage, we made sure to include tests of single films by both transport methods without any sort of processing steps in between measurements. In one case, one of the patterned samples (made by scratching) was used in a roughly Van der Pauw-type geometry with current and voltage paths overlapping in a crosslike intersection of lines with width at about 0.25 mm as shown in the inset to Fig. 1(c). Current flowed between two adjacent arms of the cross with voltage measured on the other two. The data in Fig. 1(c) show a crossover from $R(T)$ weakly increasing (triangles) or decreasing (squares) above about 200 K to more strongly increasing below 200 K for either choice of paired adjacent leads. A speculative account of why these different geometries produce such different results is given toward the end of this paper after some background facts are established by noise results.

In all cases the strip $R(T)$ (and the VdP measurements when available) showed the sharp onset of some thermal hysteresis at and below 240 K as shown in Fig. 2(a). This onset of thermal hysteresis corresponded approximately to the onset of ferromagnetic order, as found in other measurements,[13] and thus presumably indicates the phase transition. In the two samples measured down to 4.2 K, the increase in $R(T)$ saturated below about 10 K. Thus when we refer to “insulating” behavior, it is a shorthand for the decreasing $R(T)$ regime but not true insulating behavior with a diverging $R(0)$.

Magnetoresistance measurements [Fig. 2(b)] on a strip sample as it is cooled in an in-plane field show the onset of some complicated (nonmonotonic in H) effects near about 200 K (see lower inset) turning to simple weak negative magnetoresistance at low T with no sign of saturation at the largest fields (4.5 T) reached. The likely explanation for this low-T negative magnetoresistance not saturating within our field range is discussed in our conclusions.

Conductance noise measurements were performed on patterned strip samples in a liquid-nitrogen transfer-line cryostat mounted in an electromagnet. Although convenient at low fields, this setup did not allow noise measurements above $|H|=0.3$ T. The noise measurements employed dc currents from battery sources, ac-coupled low-noise amplifiers, high-frequency low-pass filters, and digitation rates in the range 200–400 Hz. (Unfortunately the low-pass filter was not a standard antialias filter, so the higher octaves in that frequency range do not give reliable noise powers, and small corrections are needed in the lower octaves.) As shown in Fig. 3, these measurements revealed ordinary 1/f-type noise (equal noise power per octave) above the magnetic ordering temperature. Below the magnetic ordering temperature there were large variations in the spectral slope as a function of T and between samples. The typical spectral slope was less than one (more noise power in higher octaves) in this regime for unknown reasons. Annealed and unannealed samples showed similar noise magnitudes and variations in spectral slope. The absolute magnitudes of the noise normalized by sample volume were comparable to some of the noisier manganite films reported, particularly, ones with poor substrate-sample lattice matches (e.g., Ref. 16).

The noise magnitude increases as T is lowered. Given that the spectral slopes are consistently less than 1.0, an effect of

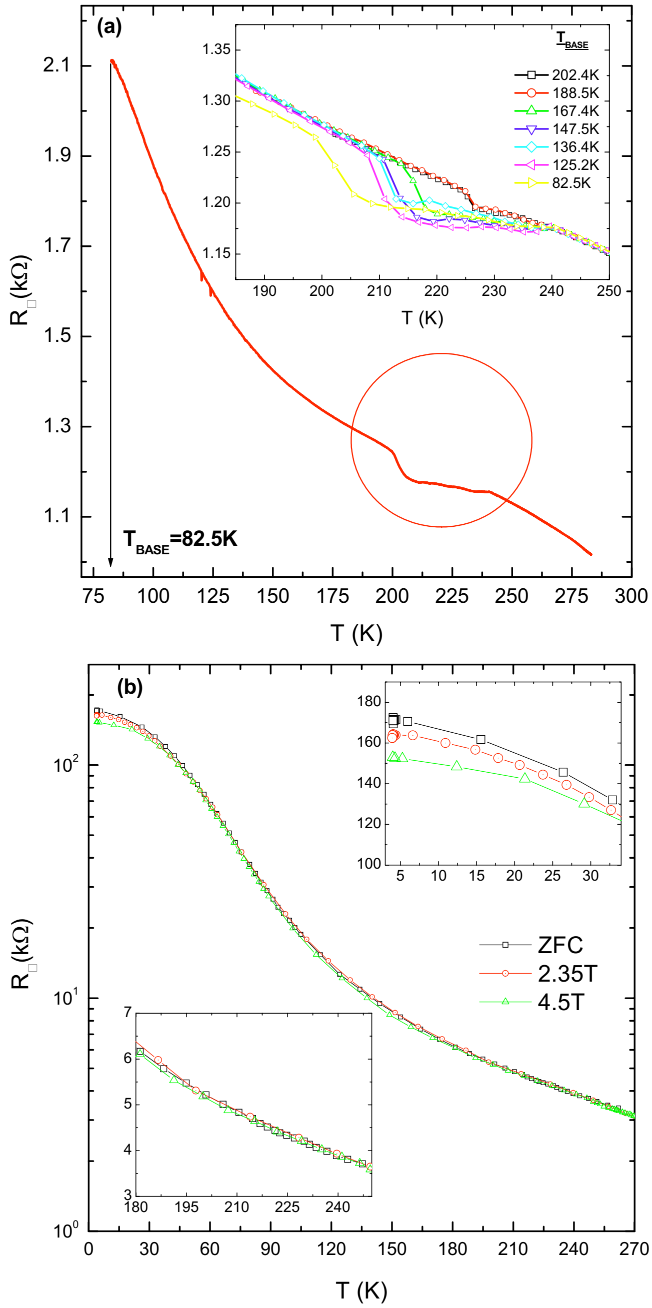

FIG. 2. (Color online) (a) The resistance (expressed per square) is shown under different thermal histories. A (circled) feature appears in $R(T)$ in the 200–240 K range taken on a strip-patterned sample shown as solid triangles in Fig. 1(a) as shown upon heating from a base T (T_{BASE}) of 82 K. Thermal hysteresis in R (T with a variety of different curves taken on warming from different base temperatures is shown in detail in the inset). All curves collapse at 240 K. Those with base T_{BASE} above 185 K overlap each other and show a sharp feature at 226 K. (b) $R(T)$ expressed as resistance per square taken in a strip sample, with length=1 mm, width =0.5 mm, and thickness=54 nm, cooling under fields of 0, 2.35, and 4.5 T. The saturation of R at low T was also found in the other sample measured down to 4.2 K. The insets show blowups of parts of the same plot in the same units.

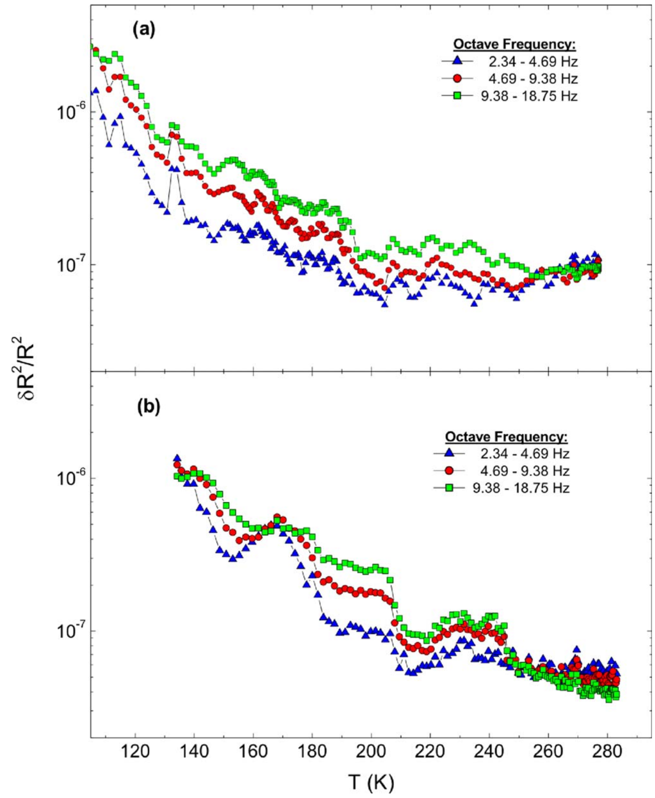

FIG. 3. (Color online) Normalized noise powers in several octaves are shown as a function of T in two samples. (a) Annealed sample with length=375 μm and width=25 μm [shown by solid squares in Fig. 1(a)]. (b) Not annealed sample with length =571 μm and width=25 μm [shown by solid triangles in Fig. 1(a)]. Both samples were patterned by ion milling. The temperature sweep rate was approximately 0.5 K/min.

this sign, which is somewhat stronger than the observed T dependence, is expected via the Dutta-Horn relation between kinetics and T dependence.[17] The overall T dependence of the noise magnitude is complicated by the thermodynamic freezing of fluctuators and by T-dependent changes in the overall conduction properties.[18]

Non-Gaussian noise effects were also evident throughout this low-T regime despite the relatively large dimensions of these samples. Switchers with two or more discrete R levels were found in this range. Only those with just two clear levels as illustrated in Fig. 4 were analyzed in detail. We analyzed all those found while sweeping T at $H=0$ in one sample with some data missing because of the usual experimental limitations (e.g., finding fluctuators before the magnet was set up). The fractional magnitude $\Delta R/R$ of the individual resistance steps is roughly 1×10^{-3} for this record typical of the larger two-state switchers found. In a sample with a net volume of $\sim1\times10^{-9}$ cm^3 in the shared current-voltage path that step size would indicate if the current flow were nearly homogeneous that the fluctuating regions would have volumes of at least $\sim1\times10^{-12}$ cm^3 or even larger if the fractional local resistivity fluctuations were less than of order unity.

The free-energy difference between the two states of a fluctuator $\Delta F=\Delta E-kT\Delta\sigma$ (where $\Delta\sigma$ is the dimensionless

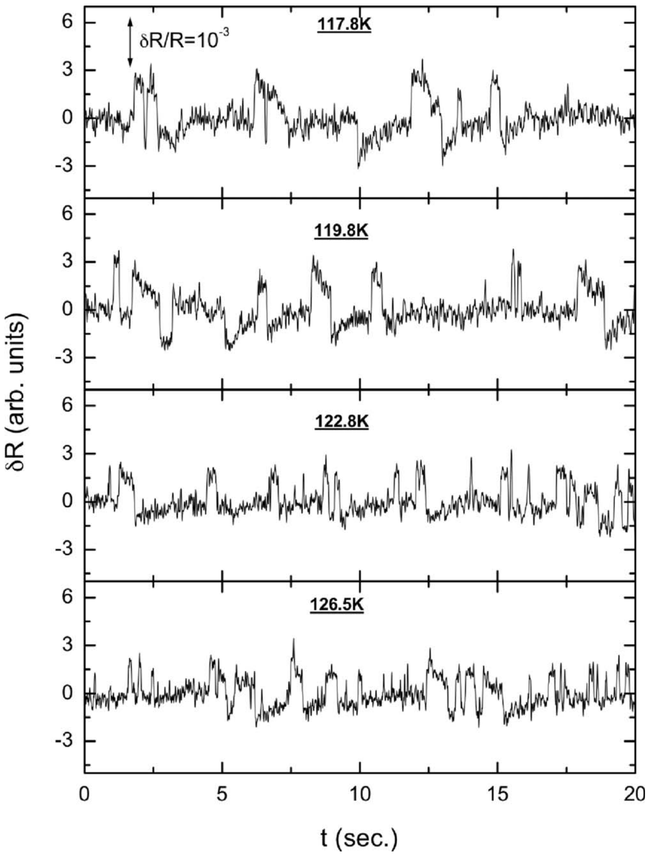

FIG. 4. Resistance as a function of time monitored at different temperatures for one of the switchers. Measurement performed on patterned sample employing a four-probe configuration. Sample: not annealed, patterned by ion milling, with length=571 μm, width=25 μm, and thickness=48 nm, shown as solid triangles in Fig. 1(a).

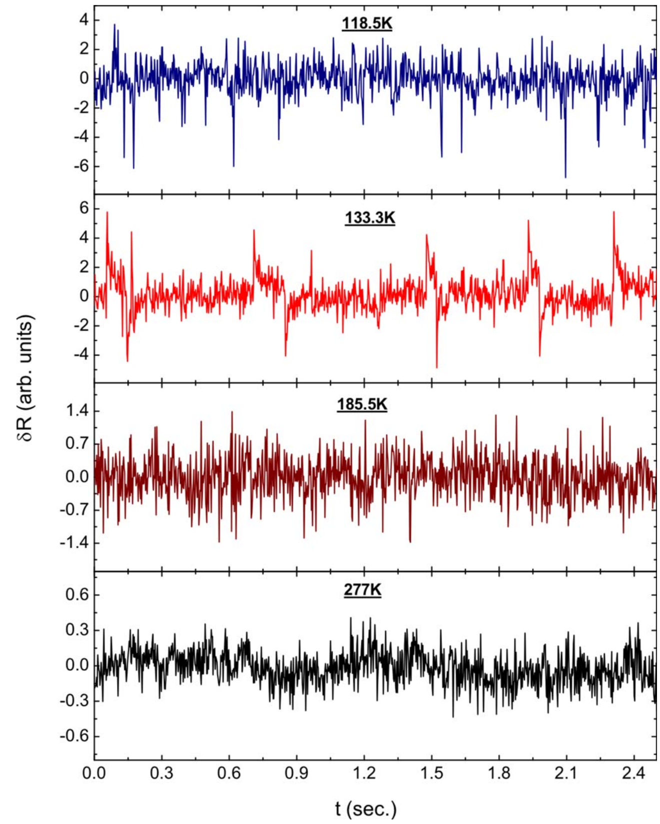

FIG. 5. (Color online) Resistance as a function of time monitored at different temperatures in an annealed sample, patterned by ion milling, with length=571 μm, width=25 μm, and thickness =48 nm, shown as solid squares in Fig. 1(a). Occasional large two-state switchers are apparent below about 250 K in some cases (133 K) with the appearance affected by the high-pass filtering of the preamplifier.

entropy difference, ΔE is the energy difference, and k_B is Boltzmann's constant) is given by $\Delta F = -kT\ln(r)$ where r is the ratio of the times spent in each of the two configurations from records such as those in Fig. 4. Therefore we can use the standard thermodynamic relations

$$\frac{\partial \Delta F}{\partial T} \equiv -k\Delta\sigma \quad \text{and} \quad \frac{\partial \Delta F}{\partial H} = \Delta\mu$$

to compute $\Delta\sigma$, ΔE, and the magnetic-moment difference $\Delta\mu$ by taking derivatives with respect to T and H.

These thermodynamic derivatives were studied in detail in two as-deposited samples. Although we did not perform thermodynamic derivative measurements on the annealed sample, Fig. 5 illustrates that it contained similar two-state systems. The Boltzmann factor vs H data [see Fig. 6(a) and Table I] consistently show $d\ln(r)/dH$ of roughly 1×10^4 μ_B/kT for $|H| < \sim 500$ Oe indicating that the fluctuations are between configurations differing in moment by roughly 1×10^4 μ_B along the direction of the applied in-plane field. Measurements with H normal to the sample plane showed much lower sensitivity indicating that the magnetization was in-plane consistent with direct magnetic measurements on similar films.[13] At larger fields it is difficult to track these individual fluctuators, and the pattern of changes in r vs H is irregular indicating that r is also affected by changes in the local environment from neighboring domains. Such noise results are all familiar from measurements on manganites.[19]

The T dependences of the r's of the different two-state fluctuators were very weak as illustrated in Fig. 6(a). The implication is that the energies of the two configurations differ by less than around 2500 K (in Boltzmann units) as summarized by the low ΔE's in Table I. Then the actual ratio r at any given T can be used to infer the dimensionless entropy difference $\Delta\sigma$ between the configurations with typical values being $|\Delta\sigma| < 10$, which are very small considering that the vector moment differs by of order 1×10^4 μ_B.

The $\Delta\mu$'s found are large enough to indicate that these are magnetic fluctuators presumably involving ferromagnetic clusters similar to those found in bulk samples.[2–9,11] $\Delta\mu/\mu_B$ then should give the approximate number of unit cells in an individual fluctuator. Values of about 1×10^4 are consistently found at least five orders of magnitude lower than would be guessed assuming near-homogeneous conductivity from $\Delta R/R$. As with colossal-magnetoresistive manganite films,[14,20,21] the disproportion between the small portions of the sample involved in the fluctuations and their relatively

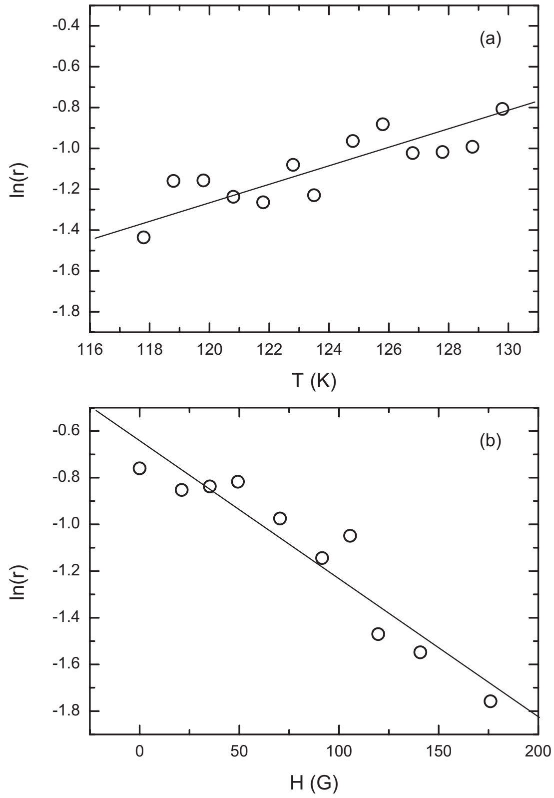

FIG. 6. (a) Logarithm of the ratio (r) between the time with high R to that with lower R as a function of temperature for the fluctuator shown in Fig. 4. (b) Magnetic-field dependence of r measured at T=129.8 K. The lines are linear fits.

large effects on R is compelling evidence that the conductance follows strongly inhomogeneous paths. Fluctuations do not show up much if they occur off the conducting paths or in parts shorted by nearby conducting regions, but an occasional fluctuator located at a node in a conduction path shows up disproportionately. In manganites, such noise effects have been found in at least two distinct regimes: near a metal-insulator transition and deep in a low-temperature metallic regime.[14,20–22] (Similar effects are found in Fe-SiO_2 nanocomposites.[15]) Fluctuators in the first regime show energy and entropy differences near that expected from bulk thermodynamic measurements of the transition between FM and paramagnetic (PM) phases; but in the low-T regime, these scalar parameters often show much smaller changes[20] indicating that the fluctuations are mainly magnetic rotations. In the regime where scalar fluctuations predominate, one expects something like a percolative conduction path since there is a mixture of metallic and insulating regions. The more surprising low-temperature conduction inhomogeneity has been tentatively attributed to a network of tunnel-barrier insulating domain walls between FM domains.[20]

In these cobaltite films the regime in which the conduction noise is dominated by magnetic rotations extends up to a high fraction of the Curie temperature. It is likely that the strong current inhomogeneity found in the noise, the anomalous geometry dependence of the $R(T)$ measurements, and the sensitivity to magnetic rotations all have the same origin: a network of insulating regions between magnetic domains. The saturation of the strip $R(T)$ at low T indicates that the insulating barriers would be thin enough to allow some tunneling conductance. A picture in which such regions give rise to interdomain resistance, which depends on relative orientation of domain magnetizations, has been suggested already for bulk cobaltites at low x with a mixture of metallic and insulating components.[12] Similar domain-wall noise effects have been found at grain boundaries in manganites[22] and have been proposed to account for low-T noise in ordered manganite films.[20]

The key issues remaining are why the films with x=0.5 (which would give a pure FM phase in bulk) should break up into domains and how those would give rise to the peculiar geometry dependence of the transport. We proceed to give an explanation of the domain origins and to speculate on how they might give rise to the effects we observed.

The formation of domains in films can be driven by strain constraints.[23] The anisotropic magnetostriction in this cobal-

TABLE I. Detailed thermodynamic and kinetic properties are shown for individual fluctuators. Error bars are shown for the most important quantities.

Switcher	T (K)[a]	E_A (K)[b]	f_A(Hz)[c]	ΔE (K)[d]	$\Delta\sigma$[e]	$\Delta\mu/\mu_B$[f] (±15%)
α1	133.6–147.5	2800	3×10^9	420±6	5±0.35	3.1×10^4 at 137 K
α2	153.5–165.5	3500	2×10^{10}	2560±500	16±4.1	-
α3	154.3–177.5	2650	1×10^8	1550±250	7±0.2	4.8×10^4 at 173.3 K
α4	117.8–129.8	2500	3×10^9	−375±20	−2±0.3	1.3×10^4 at 129 K
α5	195.7–203.8	5060	1×10^{12}	1200±300	6±1.5	-
α6	102.8					4.7×10^3 at 102.8 K

[a]T gives the temperature range over which data were taken on the switcher.
[b]E_A gives the Arrhenius activation energy of the net switching rate.
[c]f_A gives the attempt rate; r is the ratio between the time spent in the state with high R to that with the lower R.
[d]$\Delta E=-k_BT^2\partial \ln(r)/\partial T$ is their thermodynamic energy difference.
[e]$\Delta\sigma=-\ln(r)-T\partial \ln(r)/\partial T$ is the dimensionless entropy difference.
[f]$\Delta\mu=-k_BT\partial \ln(r)/\partial H$ is the magnetic-moment difference along the applied field with μ_B being the Bohr magneton.

tite (at least at $x=0.3$) is even larger than in manganites,[24] so strain clamping by the substrate favors formation of 90° domain-wall textures even more strongly in the cobaltites.[23] In both types of materials, the proximity of insulating nonferromagnetic phases can lead to insulating domain walls having lower free energy than any standard magnetic distortion, and domain walls in manganites are known to show anomalously high resistance.[25] The expected size of the domains[23] given substantial uncertainty about what the domain wall free energy would be is consistent with the fluctuator sizes found via noise. The resistance would then be expected to arise from somewhat disordered domain walls, which is very sensitive to domain fluctuations. There would be no particular mystery about how the strip $R(T)$ and the non-Gaussian noise effects arise from such a model. The very large fields required to obtain much negative magnetoresistance would also make sense since the characteristic energy scale per unit cell for strain-driven phase changes would have to be roughly of the order of kT_C corresponding to fields of around 100 T. Thus we would not expect at our largest fields either to saturate the magnetoresistance or to suppress the noise.

The VdP $R(T)$ data are stranger. It is important to establish that the VdP $R(T)$ does not result from macroheterogeneities (e.g., associated with grain boundaries), which happen to isolate current from voltage leads. Depending on the orientation of such defects, they could (if insulating) isolate current from voltage leads in one VdP orientation but could actually have the opposite effect in the 90°-rotated configuration. The grain boundaries in these films had approximately random orientations[13] so they should lead to no systematic geometry dependence of the form of $R(T)$ on a large scale. They are dense enough so that on the multimicrometer scale of our sample patterns, the statistical fluctuations in their numbers should not be important. The largest asymmetries we found for any sample were for the small cross illustrated in Fig. 1(c), and even in this case the low-T data were roughly symmetrical and completely unlike the strip data from the same sample. We see no way for the systematic qualitatively different behavior of all the VdP-type geometries and the strip geometries to arise from any blotchy pattern of conductivities in two dimensions (2D). It may also seem odd that the difference between strip and VdP data extends up well above T_C. Since there is strong evidence in bulk of nonstatic magnetic clusters in this regime,[10] however, the transport properties (other than thermal hysteresis and low-frequency noise) would not be expected to show any sharp break here.

For the VdP measurements, the overlap between the actual current pattern and the path probed by the voltage contacts is less close than in the strip samples. Given the domain sizes estimated from the switchers in the noise, our films would be several domains thick allowing the easy conduction networks to pass over each other in one patch of the sample. Although both paths overlap with positive average dot product, they enter the overlap region from different directions and may pass along different networks, which become less connected at lower T. We speculate that this difference may be sufficient to shift $R(T)$ from a decreasing function of T to a weakly increasing function of T. The data on the crosslike sample show a crossover between these two types of behavior as would be expected if the high-T regime were more nearly homogeneous on the scale of that sample with increasing separation into different current paths around the onset of magnetic order.

This interpretation of the VdP anomaly would rely on the domains being smaller than the film thickness since there are no bypasses in 2D. Since the strain-driven domain size should scale approximately as the square root of film thickness for large thicknesses,[23] thinner films should crossover to qualitatively different behavior. VdP results on films thinner than 5 nm indeed show $R(T)$ *decreasing* with T.[13] Although other systematic differences between such very thin films and the ones we studied could be present, that result is consistent with an explanation involving parallel conduction networks in three dimensions (3D). We do not know whether the peculiar geometrical effects would arise generically for highly variable conducting links or depend on special geometrical features of the domain pattern.

In summary, the noise data allow a firm conclusion that the conductance in these films is highly inhomogeneous and is very sensitive to fluctuations in orientations of magnetic domains. It is then likely that resistance from almost-insulating (with low-T tunneling conductance) domain walls accounts for the noise and the inhomogeneity. Known strain effects should produce networks of 90° domain walls. We speculate that there is a close relation between these noise effects and the strikingly anomalous geometry dependence of transport measurements. It would be very interesting to take similar data on films for which substrate-induced strain gave out-of-plane magnetization, since then strain effects would not produce the domain walls, which we believe are important for the anomalous effects.

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