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Thermal hysteresis in low-frequency noise of $La_{0.7}Sr_{0.3}Mn_{0.92}Fe_{0.08}O_3$ thin films at low magnetic field

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Abstract

The low-frequency $1/f$ voltage noise of epitaxial $La_{0.7}Sr_{0.3}Mn_{0.92}Fe_{0.08}O_3$ thin films has been studied as a function of temperature (80–300 K) and magnetic field (up to 1500 G). It is found that the noise power spectral density (PSD) shows a peak at $T \approx 238$ K which coincides with the peak temperature of the resistivity, and is below the Curie temperature. When a small magnetic field of 1500 G is applied, the noise PSD shows a large thermal hysteresis and changes with the magnetic history. This thermal hysteresis, lacking at $B = 0$, is distinctively different from other reported results on manganites without Mn-site doping. The origin of the noise peak at $B = 0$ is analysed on the basis of the magnetic fluctuation, and the scaling analysis of the normalized noise PSD is consistent with a three-dimensional random-void percolation model. The thermal hysteresis of the noise PSD further supports the assertions of a contribution from magnetic fluctuations and of a spin-dependent conduction process in Fe-doped colossal-magnetoresistance materials.

1. Introduction

A great deal of attention has been paid to mixed-valence perovskite manganites with the general formula $RE_xA_{1-x}MnO_3$ (RE = rare earth and A = divalent metal). Besides the so-called colossal magnetoresistance (CMR) effect, a strong interplay of charge, spin, and lattice system has also been found for these systems [1–3]. Due to the competition between interactions of comparable strength, a rich electronic phase diagram has been observed. The doped manganites exhibit ferromagnetic metallic (FMM), ferromagnetic insulating (FMI), paramagnetic metallic (PMM), paramagnetic insulating (PMI), charge-ordering (CO), and orbital-ordering ground states in different cases. Recent theoretical works indicate that, as the competition between different states is so delicate, a phase separation generally exists [4]. Experimentally, the coexistence of metallic and insulating phases in $La_{1-x}Ca_xMnO_3$ has been confirmed by scanning tunnelling microscopy [5]. Evidence of an inhomogeneous FMM state

and small polarons in $La_{1-x}Sr_xMnO_3$ has been found using pair-density functional techniques and is reported in [6, 7]. Further experimental results support the existence of both lattice and magnetic polaronic states near the ferromagnetic ordering temperature, T_c. These features lead to a picture where the metallic delocalized carriers and insulating thermally activated polarons coexist near T_c [8]. The spin-dependent process of transportation of these carriers plays a key role in the understanding of the CMR effects.

Noise measurement has been proven to be a powerful tool for probing dynamic behaviours of the charge carriers in manganites, providing more insight into the underlying mechanism of the CMR effects [9–15]. In the noise measurement, the charge carriers behave as fluctuators which cause resistivity fluctuations. Alers *et al* [15] and Wang *et al* [16] measured the $1/f$ noise of, respectively, $La_{0.6}Y_{0.07}Ca_{0.33}MnO_3$ and $Nd_{0.7}Sr_{0.3}MnO_3$ epitaxial thin films. The observed giant noise was attributed to the resistivity fluctuation caused by magnetic fluctuations through a spin-dependent transportation process. Raquet *et al* [10] measured the random telegraph noise (RTN) in $La_{2/3}Ca_{1/3}MnO_3$ films as a function of temperature and magnetic field, and reported on observation of giant RTN in the resistance fluctuation. They considered the origin of the RTN to be a dynamic mixed-phase percolative conduction process, where manganese clusters switch back and forth between two phases that differ in their conductivity and magnetization. Podzorov *et al* [9] discovered an unprecedented magnitude of the $1/f$ noise near the Curie temperature (T_c) in low-T_c manganite polycrystal and single-crystal bulk samples of $La_{5/8-x}Pr_xCa_{3/8}MnO_3$ with $x = 0.35$. They suggested that the so-called Curie temperature for the low-T_c materials is a percolation transition temperature rather than the temperature at which the long-range ferromagnetic phase transition occurs. The scaling analysis of the $1/f$ noise is consistent with the percolation model of conducting domains randomly distributed in an insulating matrix.

In this paper, in order to clarify the effects of Fe doping on the CMR mechanism, we measured the resistivity and low-frequency noise for Fe-doped epitaxial thin films of $La_{0.7}Sr_{0.3}Mn_{0.92}Fe_{0.08}O_3$ (LSMFO), with temperature ranging from 80 K to 300 K and for small magnetic fields up to 1500 G. This sample is chosen because

(1) despite the fact that $La_{0.7}Sr_{0.3}MnO_3$ is a canonical high-T_c CMR material, noise measurements on this system have been few [17],
(2) LSMFO shows a separation between the electronic and magnetic phase transition temperatures and an enhancement in magnetoresistance (MR) at a 4 kOe field.

2. Experiments

LSMFO thin films of size 2×10 mm^2 and typical thickness 2500 Å were deposited on (001)-oriented $SrTiO_3$ substrates using a 248 nm KrF excimer laser (Lambda Physik). The as-deposited films were post-annealed at 680 °C for 30 minutes under an O_2 pressure of 400 mbar prior to cooling to room temperature at a rate of 15 °C min^{-1}. The crystal structure of these films was checked by x-ray diffraction and strong (001) reflections were found. We found that the structure does not change with the Fe doping. From the XRD patterns we also found that all the samples were single phase without detectable secondary or impurity phases; the detailed results will be published elsewhere [18].

The electrotransport properties were measured using the standard four-probe technique. The gold pads were prepared by electron-beam evaporation and electrical wires were attached by indium soldering on the LSMFO thin films. The thickness of each gold pad was 100 nm and the separation distance for each contact was 2 mm. The contact resistance was much less than 1 Ω and the contact noise, which was checked by varying the resistance of the

ballast resistor, was shown to be negligible in our experiment. In the noise measurement, we used a battery as a current source and the biasing current was 0.1 mA; the corresponding current density was 25 A cm^{-2}. The spectra of the $1/f$ noise in the voltage fluctuation were measured between 1 and 100 Hz. The battery-generated dc current was applied to the sample through an in-series ballast resistor. The resistance of the ballast resistor was chosen to be at least 1000 times greater than that of the sample. The ac signal (because the dc signal was filtered by a large capacitor) between the voltage probes was fed to an EG&G 1900 low-noise transformer, then to an EG&G 5113 pre-amplifier, and the amplified output was measured by an HP 35670A spectrum analyser. The background noise (thermal noise and preamplifier noise) was measured when the sample current was zero. All the noise signals reported in this paper are shown with the background subtracted. In all measurements, the temperature was controlled by a LakeShore 340 Temperature Controller and the sample was located inside a vacuum chamber, which was immersed in liquid nitrogen. The temperature stability during each measurement was better than 0.01 K and, in order to remove thermal fluctuations and drift, all noise data were measured when the system was in thermal equilibrium. The direction of the applied magnetic field was perpendicular to the sample surface.

3. Results and discussion

Figure 1 shows the temperature dependence of the normalized noise power spectral density (PSD), $S_n(f) = S_V(f)/V^2$, at $f = 9$ Hz for two different cases, i.e., $B = 0$ G (a) and $B = 1500$ G (b). Here the error bar of $S_n(f)$ at each temperature is below 10%. The inset shows the temperature dependence of the resistivity data for two different applied magnetic fields, $B = 0$ G (solid line) and $B = 1500$ G (dotted line). When $B = 0$ G, a noise peak appears near 238 K and this temperature is not changed with thermal warming and cooling, i.e., there is no thermal hysteresis. Furthermore, at this temperature, the resistivity also has a maximum value as shown in the inset of figure 1 and this reveals the metal–insulator (MI) transition temperature (T_p), which is obtained as 238 K for $B = 0$ G. Although the position of T_p is not changed with cooling or warming, it shifts to lower temperature with applied magnetic field; e.g., $T_p(B = 1500\ \text{G})$ is 4 K below $T_p(B = 0\ \text{G})$ as shown in the inset of figure 1. In our previous work [18] we found that with Fe doping, there appears a discrepancy between T_p and T_c, and in the LSMFO thin films with $x = 0.08$, T_p (=238 K) is about 20 K below T_c (=258 K) which is marked in the inset of figure 1. Generally, the coincidence of the noise peak with the resistivity peak supports the PMI-to-FMM phase transition temperature. Thus below T_p, the sharp decrease in noise PSD occurs due to transition of the material from a randomly aligned spin state to an aligned spin state.

In figure 1(b), we present results of the noise PSD measurement when a magnetic field of 1500 G is applied. First the sample is cooled down from room temperature to 80 K (field cooling; FC) and then it is ramped up from 80 K to room temperature (field warming after field cooling; FW-AFC). We repeat the experiment by first cooling the sample down to 80 K without applying a magnetic field, only applying the magnetic field when the temperature is ramped up (field warming after zero-field cooling; FW-AZFC). During the FC process, the noise PSD is found to decrease slightly, but below $T = 270$ K it increases and a broad peak is observed (full triangles in figure 1(b)). At temperatures below T_p, the noise PSD decreases sharply. However, different noise PSD behaviours are found for FW-AFC (full circles) and FW-AZFC (open squares) processes. No noise peak was found for the FW-AFC process and the noise PSD is generally 2–3 orders of magnitude lower than that of the FC process. However, at temperatures well below T_p, the magnitudes are the same. This low level of noise could be explained well by the alignment of the core spins of the Mn ions.

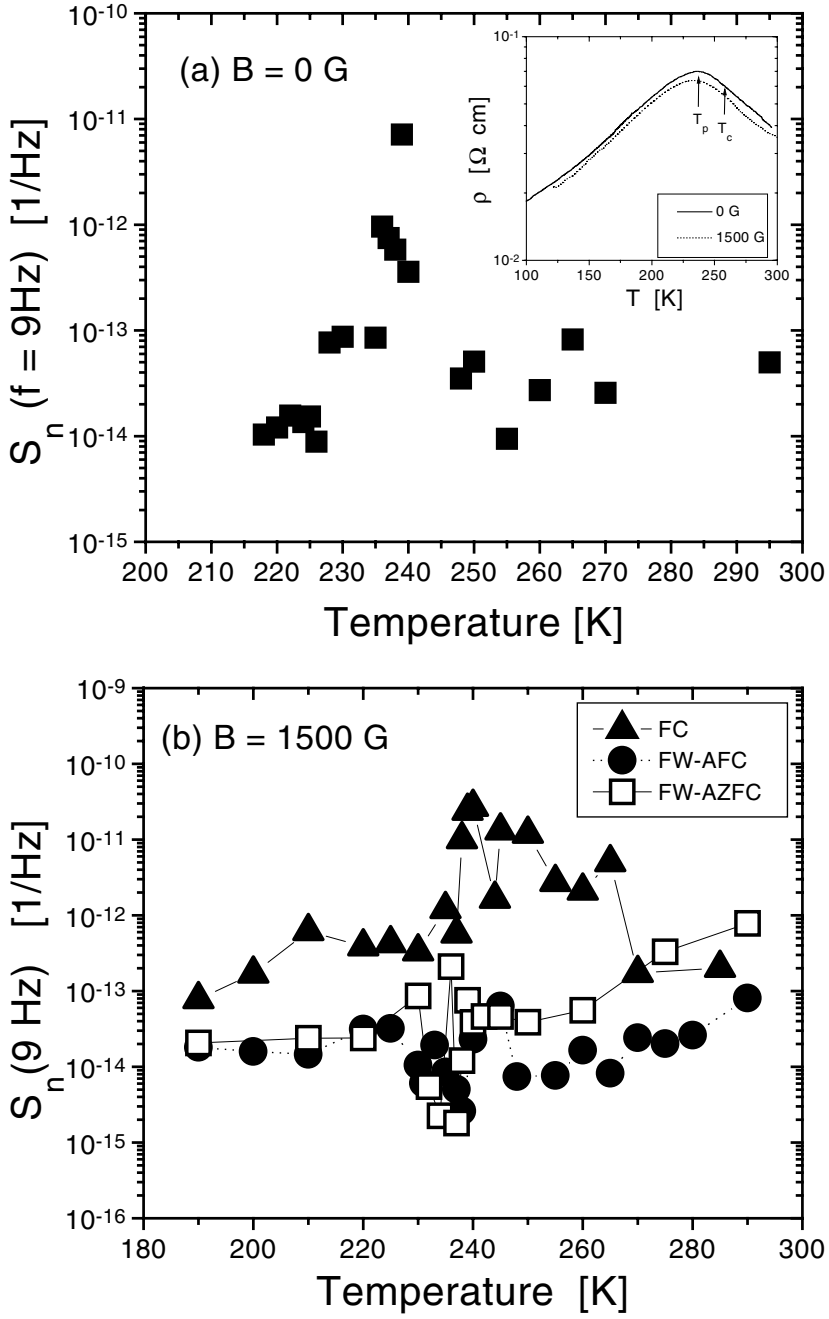

Figure 1. Temperature dependences of the normalized noise PSD, $S_n(9\ \text{Hz})$, at $B = 0$ G (a) and $B = 1500$ G (b). In (b), field cooling (full circles) and zero-field cooling (open squares) are also shown. The inset shows the temperature dependence of the resistivity for two different applied magnetic fields, $B = 0$ G (solid line) and $B = 1500$ G (dotted line).

Another interesting observation can be made if figure 1(b) is compared with figure 1(a). At temperatures near and above T_p, the noise PSD at $B = 1500$ G is larger than (for FC)

or similar to (for FW-AFC) that at $B = 0$ G. This is contrary to what is observed in the resistivity data. This suggests that above T_p, the increase in the noise PSD cannot be due to the alignment of the core spin of the Mn ions. This temperature dependence behaviour suggests that the increase of the noise PSD could be due to thermal fluctuations of the magnetic spins or domains. Moreover, below T_p the magnitude of the noise for FW-AZFC is similar to that for FW-AFC, but near T_p there appears a noise peak, and above T_p the magnitude of the FW-AZFC noise is one order of magnitude larger than that for FW-AFC. This also suggests that the thermal fluctuation of the magnetic spin or domain is dominant above T_p and depends on the sample's magnetic history.

In order to compare the magnitude of the $1/f$ noise PSD in CMR materials for different material systems, in practice one determines the Hooge parameter, γ (which is a dimensionless quantity) [19]:

$$\gamma = f^{\alpha} n \Omega \frac{S_V(f)}{V^2}. \tag{1}$$

Here V is the dc voltage across the sample, n is the charge-carrier density, and Ω is the sample volume. Typically γ is between 10^{-5} and 10^{-1} for metals and about 10^{-3} for semiconductors [20]. For CMR samples the published γ-values are more than 4–7 orders of magnitude larger than those for metals [20–22]. Figure 2 shows the temperature dependence of the Hooge parameter, $\gamma(T)$. In the calculation of $\gamma(T)$, we took the charge-carrier density, n, as 0.5×10^{22} cm^{-3} for $La_{2/3}Sr_{1/3}MnO_3$ thin film [23] and assumed that n does not change with temperature. At room temperature, the γ-value ($\gamma = 4.4 \times 10^3$) is significantly larger than those of conventional metals [19]. Moreover, the γ-values of these CMR oxides are comparable to the γ-values of metallic multilayers exhibiting giant magnetoresistance (GMR) [22]. This reveals that the intrinsic $1/f$ noise level in CMR materials may be abnormally high, and the spin-polarized conduction process seems to be related to the intrinsic origin of the noise [24].

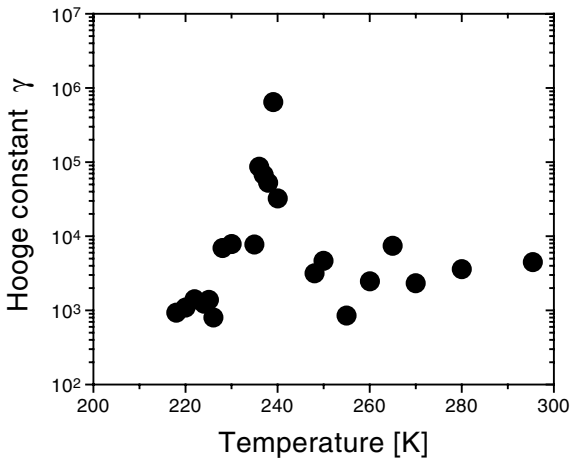

Figure 2. The temperature dependence of the Hooge parameter, $\gamma(T)$, at $B = 0$ G. At room temperature, $\gamma(T) = 4.4 \times 10^3$.

There are many things that can affect the voltage noise in manganites, and in this paper we consider the effects of charge carriers, spins, and mixed-phase percolation processes.

First, we consider the status of the charge carriers (the fluctuators in the voltage noise measurement) for the LSMFO thin films. For CMR materials, it is possible that there are several ground states in certain temperature ranges, such as FMM, FMI, PMM, PMI, CO,

and orbital-ordering ground states. In LSMFO systems, the disorder caused by Fe doping can lead to fluctuation of the on-site energy experienced by the carriers. The on-site energy fluctuation plays a key role in determining the electronic state of the manganites for the disorder model [25,26]. Within the Hamiltonian model, it is composed of two terms; one is the double-exchange energy and the other is the random on-site energy. In the $La_{1-y}Sr_yMnO_3$-type manganites, the random on-site energy arises mainly from the doping disorder of the divalent metal ions [26]. By doping with Fe, we can change the degree of disorder. A separation of the electronic and magnetic phase transition temperatures ($T_c \neq T_p$) was observed in LSMFO; i.e., with decreasing temperature, LSMFO thin film changes from a PMI state to a FMI state, and finally to a FMM state [18]. In this inhomogeneous material, domains, clusters, or islands exist in each phase and their free energies will be more or less the same. As this leads to the system consisting of many energy minima, and charge carriers find it very easy to fluctuate in position among these energy minima, this may result in giant fluctuation noise; i.e., giant fluctuation noise may arise from the competition between different ground states. In fact, recently, in noise measurements on $La_{1-x}Ca_xMnO_3$ single crystals and thin films with $x \approx 0.33$, the noise was found to originate from the local electronic states of the CMR material fluctuating between more and less conductive states [11].

Second, considering the spin-dependent scattering of the polarized conduction electrons, another mechanism has been suggested to correlate the enhanced noise with various magnetic fluctuation phenomena in CMR oxides [27]. In this picture, the enhanced noise ratio originates from the fluctuation of the canting angles of the Mn spins or the magnetic domain fluctuations. The fluctuations of the canting angles of the Mn spins and the domain orientations will cause fluctuations in the intra-domain resistance and inter-domain resistance, respectively. The overall noise ratio is affected by both factors and determined by whichever is the dominant one. According to this model, a giant noise peak is predicted near the critical magnetic phase transition temperature and this is supported by our experiments. For LSMFO thin films, we found that the noise peak is located at 238 K, which coincides with the resistivity peak temperature T_p. This temperature is also near the magnetic critical temperature (=258 K) for the film [18]. Furthermore, with decreasing temperature the magnetic alignment becomes stable and the noise level will become lower as shown in figure 1(a) for $T < T_p$. This reveals that the magnetic fluctuation is one of the main mechanisms.

It is suggested that the noise peak at T_p and its surprising amplitude are direct consequences of the mixed-phase percolation process, e.g. metallic and thermally activated (polaronic) transport [10,28,29]. In the percolation model, the normalized noise PSD and the resistivity, ρ, increase as the fraction (p) of metallic particles approaches the percolation threshold (p_c) [9,30]:

$$S_n \propto (p - p_c)^{-\kappa} \tag{2}$$

$$\rho \propto (p - p_c)^{-t}. \tag{3}$$

Here, κ and t are the critical exponents of the normalized noise PSD and the resistivity. It is convenient to represent S_n as a function of ρ:

$$S_n \propto \rho^{\kappa/t} \tag{4}$$

and it can be fitted by a power law with $\kappa/t = 4.41 \pm 0.18$. These values are somewhat larger than the values obtained theoretically from the continuum percolation model. In order to compare in detail with the percolation model, we have to obtain values of κ and t, and it is convenient to use some quantity that is proportional to p, e.g., the magnetization, M. It is known [31] that $M \propto (T_p - T)^{\beta}$ with $\beta \approx 0.3$ and $\rho \propto M^{-2} \propto (T_p - T)^{-t}$ with $t = 2\beta$. From our experimental data for M [18], we found that $\beta = 0.403 \pm 0.016$—and these values

are close to 0.3—and from the resistivity data we obtained $\beta \approx 0.47$; thus $t = 0.95 \pm 0.01$. The dependence of S_n is close to the power law $S_n \propto (T_p - T)^{-\kappa}$ with $\kappa = 1.569 \pm 0.216$ on the metallic side of the transition as shown in figure 3.

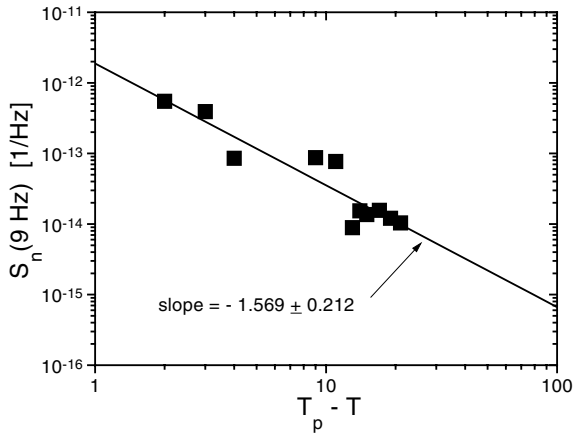

Figure 3. A log–log plot of the normalized noise PSD, $S_n(9\ \text{Hz})$, versus $T_p - T$ for $B = 0$ G. Here the slope is -1.569 ± 0.216.

When we compare our experimentally obtained values of κ and t with others, we find that κ is in very good agreement with the 3D continuum percolation model of random-void classes, where $\kappa = 1.56 \pm 0.1$ for 3D [32]. However, t is not in good agreement with this 3D random-void model. In the random-void model, insulating regions are randomly placed in a conducting matrix, while in our LSMFO thin films, FMI regions are randomly located in a FMM conducting matrix. Moreover, the effect of Fe doping produces an insulating site, and Fe ions act as trapping centres and block the conduction path of e_g electrons [18]. Since there is no CO insulator among our LSMFO samples, the resistivities of the insulating phase and metallic phase should be similar as shown in the inset of figure 1, and thus t is small and in this case the resistivity will not follow the original percolation model. However, as the noise PSD is basically caused by the switching between the clusters, it still follows the original percolation model.

Moreover, during this mixed-phase percolation process, instead of the dynamic switching between different electronic states, it is the magnetic fluctuation that plays the key role in causing the giant noise in LSMFO thin films. This is magnified by the large thermal hysteresis of the noise PSD, as shown in figure 1(b). Raquet *et al* [10] have argued that the fluctuation/switching between different electronic states can be represented by a statistical probability. They emphasized that the final states for RTN noise are never exactly the same following thermal cycling. Since the thermal hysteresis that we observed here is stable, we conclude that the dynamic phase separation mechanism, if it is operative at all, cannot be the main origin of the giant $1/f$ noise. Instead, the origin of the thermal hysteresis of the noise PSD can be related to the magnetization history of the sample. In the LSMFO epitaxial thin films studied here, some local magnetic frustration is generally expected due to the oxygen deficiency or the Fe-doping-induced disorder. Therefore, corresponding to the different magnetization histories (FC or ZFC), the film is in different magnetic states showing different magnetic stabilities. Through the spin-dependent conduction process, this will lead to the different noise levels apparent in figure 1(b). This explanation further leads us to the conclusion that the magnetic fluctuations are the main source of noise.

4. Conclusions

The electronic and magnetic transport properties of high-quality $La_{0.7}Sr_{0.3}Mn_{0.92}Fe_{0.08}O_3$ epitaxial thin films are studied by measuring the resistivity and low-frequency noise. At $B = 0$ G, the noise PSD shows a peak near T_p, which is below the Curie temperature, and there is no thermal hysteresis. The coincidence of the noise peak with the resistivity peak at $T = T_p$ reveals the phase transition from FMI to FMM states. The noise peak at T_p can be explained by a mixed-phase percolation process, in which the magnetic fluctuations (the fluctuations of the canting angles between Mn core spins or the magnetic domain fluctuations) play a key role. The noise generated by switching of FMI and FMM states due to the magnetic fluctuation will follow the three-dimensional random-void percolation model. At $B = 1500$ G, there is a thermal hysteresis in the noise PSD, and this suggested that the dynamic phase separation mechanism, if it is operative at all, cannot be the main origin of the giant $1/f$ noise observed in the $La_{0.7}Sr_{0.3}Mn_{0.92}Fe_{0.08}O_3$ thin films. The thermal hysteresis in the noise could be related to the magnetization history of the sample, as local magnetic frustration is expected from the Fe-doping-induced disorder and oxygen deficiency in the films. Moreover, the change of the noise PSD with both the thermal and the magnetic field history at $B = 1500$ G further supports the assertion of contributions from magnetic fluctuations and a spin-dependent conduction process in Fe-doped CMR material. At room temperature, the γ-value ($\gamma = 4.4 \times 10^3$) is significantly larger than those of conventional metals. Moreover, the γ-values of these CMR oxides are comparable to the γ-values of metallic multilayers exhibiting giant magnetoresistance (GMR), and this reveals that the intrinsic $1/f$ noise level in CMR materials may be abnormally high.

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