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Relaxation of low-temperature magnetoresistance and magnetization of polycrystalline $(La_{0.5}Eu_{0.5})_{0.7}Pb_{0.3}MnO_3$

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Abstract

Hysteresis and relaxation of magnetoresistance and magnetization of substituted $(La_{0.5}Eu_{0.5})_{0.7}Pb_{0.3}MnO_3$ lanthanum manganite in a low-temperature region (<40 K) are investigated. It is shown that at these temperature features of the magnetoresistive effect are determined mainly by spin-dependent tunnelling of carriers via insulating grain boundaries. As was demonstrated previously, the grain boundaries may be antiferromagnetically ordered. Therefore, relaxation of magnetization and resistance is determined by the processes of relative orientation of the magnetic moments of ferromagnetic domains neighbouring the antiferromagnetic boundary of ferromagnetic domains under the action of temperature fluctuations. It is shown that relaxation follows the logarithmic law within the time interval $t \sim 10^2$–3×10^3 s. A comparison between time evolutions of the magnetic moment and resistance shows that magnetoresistance and magnetization are related as $\delta R = \delta M^n$, where $n = 2.5$. The obtained value n is close to the characteristic value $n = 2$ for tunnel magnetoresistance of granular *ferromagnetic metal/insulator* systems.

1. Introduction

Until now, substituted manganese oxides, or manganites, $R_{1-x}A_xMnO_3$ (R is a trivalent rare-earth ion: La^{3+}, Nd^{3+}, Pr^{3+}, Sm^{3+}, etc and À is a divalent ion: Ca^{2+}, Sr^{2+}, Ba^{2+} or Pb^{2+}) have been a subject of intensive study because of their rich phase diagrams and physical properties sensitive to external factors [1–6], which make these materials promising for practical applications in spintronic devices. The variety of physical properties is caused by impurity phase stratification in these systems on a submicrometre scale [5, 6], which usually manifests itself in the coexistence of a ferromagnetic phase and an insulating phase with localized carriers. The effect of colossal negative magnetoresistance inherent in manganites originates from the increasing fraction of the conducting ferromagnetic phase in a magnetic field [1, 5, 6]. The ground state of the substituted manganites is determined by cation radii of atoms in the R positions and atomic disordering [1]. This state can be ferromagnetic metal or antiferromagnetic with charge ordering [7, 8]. The variation in the ground state of manganites upon doping is related to the competition between different interactions with similar energies, which allows the system to be in the phase stratification state, when two phases with different magnetic and electronic properties may coexist. This makes it possible to vary the properties of the system by the effect of different external factors.

The dynamic characteristics of the phases in manganites can be investigated by studying the relaxation of magnetization and resistance in external magnetic fields [9–11]. In such studies, energies of the interfaces between the conducting and insulating phases can be established by characteristic relaxation times. A value of relaxation is strongly temperature-dependent and can be positive or negative [10]. In addition, relaxation of magnetization being the manifestation of the metastable state in manganites is accompanied by hysteresis features in the field dependences of magnetoresistance. The aim of this study was to investigate the hysteresis features of magnetoresistance and relaxation of resistance and magnetization.

As an object for the study, we chose a polycrystalline $(La_{0.5}Eu_{0.5})_{0.7}Pb_{0.3}MnO_3$ sample, whose transport and

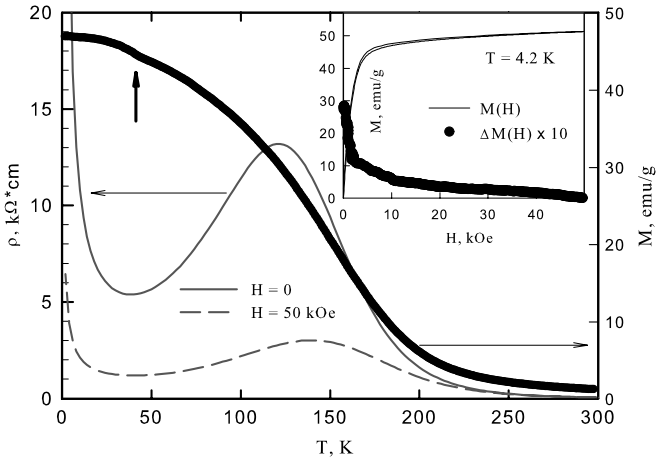

Figure 1. The $\rho(T)$ dependences of polycrystalline $(La_{0.5}Eu_{0.5})_{0.7}Pb_{0.3}MnO_3$ at $H = 0$ and 50 kOe (left axis) and $M(T)$ dependence at $H = 10$ kOe for the same sample (right axis). The inset shows the $M(H)$ dependence at $T = 4.2$ K and the value of the width of hysteresis loop $-\Delta M(H)$.

magnetic properties were investigated previously [12, 13]. The sample is characterized by considerable magnetoresistance and a hysteresis feature in the low-temperature (<40 K) region far below the temperature of the metal–insulator transition when the sample is already in the ground state of a conducting ferromagnet [1, 13]. In our opinion, the magnetoresistive effect at low temperatures is determined by spin-dependent tunnelling of carriers via insulating boundaries of the grains with antiferromagnetic ordering [12, 13]. Thus, unlike the relaxation processes in manganites being in the state of phase stratification, which manifests itself mainly near the metal–insulator transition, in our case, the main contribution to relaxation will by made by the interaction between ferromagnetic domains of the grains with the antiferromagnetic boundaries, whose relative orientation determines the low-temperature magnetoresistance in $(La_{0.5}Eu_{0.5})_{0.7}Pb_{0.3}MnO_3$.

2. Experimental

The polycrystalline $(La_{0.5}Eu_{0.5})_{0.7}Pb_{0.3}MnO_3$ sample for measurements was prepared from single crystals of this composition. The single-crystal samples were ground in an agate mortar, pressed and annealed at $T = 600\,^{\circ}$C for 6 h. Polycrystalline $(La_{0.5}Eu_{0.5})_{0.7}Pb_{0.3}MnO_3$ was characterized in detail in [12, 13].

The transport and magnetic properties were measured on a PPMS-6000 facility (Quantum Design) and a vibrating magnetometer adapted to the transport measurements. The latter were performed by a standard four-probe method. The sample's dimensions were $3 \times 3 \times 0.1$ mm^3. The sample was cooled in zero field.

3. Results and discussion

Figure 1 demonstrates the $\rho(T)$ dependences for the sample under study in zero external field and in the field $H = 50$ kOe. The metal–insulator (M–I) transition occurs near $T \approx 120$ K; in the low-temperature (below 40 K) region, the $\rho(T)$ dependence substantially grows. The value of the relative magnetoresistance $\Delta\rho/\rho(H = 0)$ at low temperatures is comparable to the value $\Delta\rho/\rho(H = 0)$ near the M–I transition (see the $R(T)$ dependence at $H = 50$ kOe in figure 1).

For a single crystal of the same composition, the $R(T)$ dependence below the M–I transition point is typical of a metal up to $T = 2$ K [12]. The value $\Delta\rho/\rho(H = 0)$ of the single-crystal sample has a maximum near the M–I transition and becomes insignificant at low temperatures.

Hence, the minimum of the $R(T)$ dependence at $T \approx 40$ K, the resistance growth in the low-temperature region and magnetoresistance of the polycrystalline sample at low temperatures result from the effect of the insulating grain boundaries.

The dependence $M(H)$ of the magnetic moment on the external field for the polycrystalline sample is shown in figure 1 (inset). The sharp growth of the dependence in the fields $H < 10$ kOe is followed by a weak linear increase in the range $H > 10$ kOe. Note the narrow yet visible hysteresis of the $M(H)$ dependence. Hereinafter, we denote the increasing external field by $H_\uparrow$ and the decreasing external field by $H_\downarrow$. The parameter $\Delta M(H) = M(H_\downarrow) - M(H_\uparrow)$ of the hysteresis dependence is also shown in the inset of figure 1 (the data for the $\Delta M(H)$ dependence are multiplied by 10). The value $\Delta M(H = 0) \approx 2.7$ emu g^{-1} corresponds to residual magnetization of the sample. For the single crystal, the $M(H)$ dependence at low temperatures saturates in the field $H \approx 10^4$ Oe; the $M(H)$ hysteresis is lower by at least an order of magnitude than that of the polycrystalline sample [12].

The temperature dependence of the magnetic moment $M(T)$ of the polycrystalline sample shown in figure 1 has a feature near $T \approx 40$ K. Investigations of specific heat of this polycrystalline sample also revealed anomaly at $T \approx 40$ K [12, 13]. According to the data of high-resolution transmission electron spectroscopy of the polycrystalline samples [13], a 3–5 nm-thick outer shell of a manganite grain is amorphous (the grain size is ~1–2 μm). This shell may be antiferromagnetically ordered [14, 15]. The magnetic (figure 1) and calorimetric data also indicate possible antiferromagnetic ordering of the grain shell.

The isotherms of magnetoresistance $R(H)$ of the polycrystalline sample at temperatures up to $T \approx 40$ K show a wide hysteresis. The isotherms of magnetoresistance of the single-crystal sample have no hysteresis [12] and the magnetoresistive effect decreases with temperature, which is typical of the substituted lanthanum manganites [5]. Consequently, the pronounced $R(H)$ hysteresis of the polycrystalline sample is related to the processes occurring at the grain boundaries. In this case, we should consider magnetoresistance in the F(M)–AF(I)–F(M) contact network, where F(M) is the ferromagnetic metal and AF(I) is the antiferromagnetic insulator.

The typical $R(H)$ hysteresis dependence for the polycrystalline sample under study at $T = 4.2$ K is presented in figure 2. Point $\boldsymbol{A}$ in the figure corresponds to resistance after zero-field cooling at $I = 1\,\mu$A. The $R(H)$ hysteresis dependence at the maximum applied field $H_{max} = 50$ kOe for

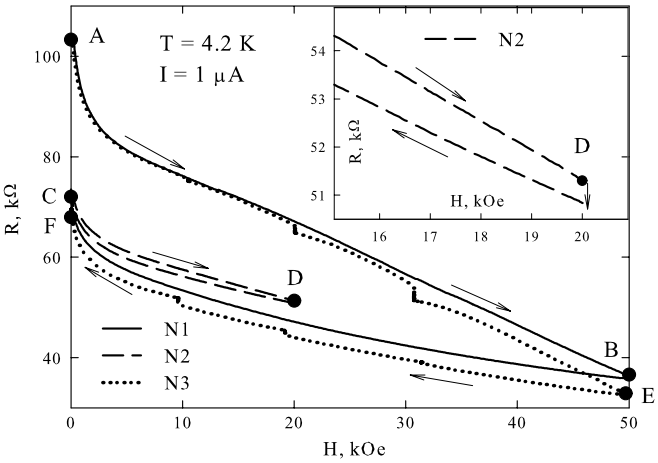

Figure 2. The typical $R(H)$ hysteresis dependences for the sample under study at $T = 4.2$ K.

the measuring time (∼1000 s) follows the path $\boldsymbol{A} \rightarrow \boldsymbol{B} \rightarrow \boldsymbol{C}$ (points $\boldsymbol{B}$ and $\boldsymbol{C}$ correspond to $R(H = 50\,\text{kOe})$ and the residual resistance $R(H_{\downarrow} = 0)$ after the external field is switched on/off). If the field is increased again from point $\boldsymbol{C}$, then the $R(H)$ dependence lies between the branches of the hysteresis dependence N1. Portion $\boldsymbol{CD}$ up to $H = 20$ kOe of the hysteresis dependence N2 in figure 2 illustrates this behaviour. If the external field is increased further up to 50 kOe, then the $R(H)$ dependence arrives at point $\boldsymbol{B}$. Upon further field cycling $0 \rightarrow 50\,\text{kOe} \rightarrow -50\,\text{kOe} \rightarrow 0$ with a constant sweep rate, the $R(H)$ dependence follows approximately portions $\boldsymbol{CDBC}$ and is symmetric relative to the ordinate axis.

As mentioned above, the magnetization hysteresis of the polycrystalline sample is rather narrow (see the inset of figure 1) as compared with the magnetoresistance hystersis. Therefore, the important role in the processes of spin-dependent tunnelling via the antiferromagnetic spacer that determine the magnetoresistance of the polycrystalline sample is played by orientation of the magnetic moments of the regions neighbouring the antiferromagnetic spacer. Indeed, the manganite grain size (∼1–2 μm) is sufficient to form a domain structure. Then, the magnetic moment of the polycrystalline sample is determined by the sum of three contributions:

$$M_{\text{CORE}} + M_{\text{ND}} + M_{\text{AF}},$$

where M_{CORE} is the magnetic moment of the ferromagnetic cores of the grains, M_{ND} is the magnetic moment of the domains near the antiferromagnetic spacer and M_{AF} is the magnetic moment of the antiferromagnetic spacer. Since the hysteresis of magnetization of the single crystal with this composition is insignificant (see above and [12]), the main contribution to the observed $M(H)$ hysteresis of the polycrystalline sample (the inset of figure 1) is probably made by the regions near the antiferromagnetic spacer, i.e. the ND regions. This means that there is a factor that prevents the orientation of the magnetic moments of the ND regions ($\boldsymbol{m}_{\text{ND}}$) along the field. It is reasonable to suggest that there exists an exchange interaction between the ND regions and the antiferromagnetic spacer. Then, the competition between the Zeeman energy $m_{\text{ND}} \cdot H \cos(\boldsymbol{m}_{\text{ND}}, \boldsymbol{H})$ and the energy of the exchange interaction with the antiferromagnetic spacer may provoke a situation where, in a certain field, the orientation of the $\boldsymbol{m}_{\text{ND}}$ vector is in the metastable state corresponding to the local energy minimum, which determines the hysteresis behaviour of $M_{\text{ND}}(H)$ and the small $M(H)$ hysteresis of the entire sample.

Magnetoresistance upon tunnelling via the insulating spacers is determined, in its turn, by the relative orientation of the magnetic moments $\boldsymbol{m}_{\text{ND}}$ to the left and to the right of the spacer. Therefore, magnetoresistance is more sensitive to the orientation of the magnetic moments of the domains neighbouring the spacers as compared with the magnetic moment of the sample. This fact determines the considerable $R(H)$ hysteresis. According to our data, above the antiferromagnetic transition temperature $T \approx 40$ K, $R(H)$ hysteresis vanishes, $M(H)$ exhibits a vary narrow hysteresis behaviour (at the level of experimental error).

Since any hysteresis is a manifestation of the metastable state, the measuring time of the hysteresis dependence, i.e. the relaxation processes, is of importance. The $R(H)$ hysteresis dependence N1 in figure 2 was measured for ∼1000 s. The dependence N3 in figure 2 was measured as follows. During the measurements of the $R(H)$ hysteresis dependence, the external field was increased up to a certain value and stabilized and relaxation of magnetoresistance $R(t)$ was fixed for ∼3000 s. Then, the external field was increased up to the next constant value and the $R(t)$ dependence was measured again. Such measurements were performed at 10, 20 and 30 kOe with increasing and decreasing fields ($H_{\text{max}} = 50$ kOe). The steps in the dependence N3 (figure 2) correspond to the $R(t)$ measurements. The total measuring time of the dependence N3 was ∼20 000 s. With the increasing and stabilized external field, the magnetoresistance value decreases, while on the back branch, i.e. with the decreasing and stabilized H, the $R(H_{\downarrow})$ value grows in time. This includes the case $H_{\downarrow} = 0$, where the observed growth of magnetoresistance is the maximum.

A noticeable shift of the dependence N1 relative to the dependence N2 (1000 s) down along the ordinate axis implies that the long stopping of the field scan is equivalent, to a certain extent, to the field variation. This concerns both $R(H_{\uparrow})$ and $R(H_{\downarrow})$ branches.

When the external field is constant, the direction of vector $\boldsymbol{m}_{\text{ND}}$ for the region neighbouring the antiferromagnetic spacer is determined by the competition between the exchange interaction with the antiferromagnetic spacer and the Zeeman energy $m_{\text{ND}} \cdot H \cos(\boldsymbol{m}_{\text{ND}}, \boldsymbol{H})$. This means that, at $H = H_{\uparrow} = \text{const}$, relaxation of the $R(t)$ dependence corresponds to a partial turn of vectors $\boldsymbol{m}_{\text{ND}}$ along the field, and at $H = H_{\downarrow} = \text{const}$ (after applying H_{max}), relaxation of $R(t)$ is determined by the disorientation of these vectors relative to the direction of the external field due to the effect of thermal fluctuations.

If after the $H = 0 \rightarrow H_{\text{max}} \rightarrow 0$ cycle a field lower than H_{max} is applied and fixed, the $R(t)$ dependence will decrease again. This behaviour is illustrated in the inset of figure 2 for magnetic prehistory of $\boldsymbol{ABCD}$. The break at point $\boldsymbol{D}$ ($H_{\uparrow} = 20$ kOe) corresponds to time relaxation during 3000 s.

Thus, the relaxation path (an increase or a decrease in $R(t)$) is determined by the direction of the field variation.

Therefore, the height of the potential barriers overcome by vector $\boldsymbol{m}_{\mathrm{ND}}$ is determined by the value of the maximum applied field. This behaviour can be schematically shown by a dependence of the energy of magnetic moment $\boldsymbol{m}_{\mathrm{ND}}$ in the external field on the angle $\varphi = \angle \boldsymbol{m}_{\mathrm{ND}}, \boldsymbol{H}$. A decrease in angle φ upon hopping via local potential barriers leads to a decrease in magnetoresistance, i.e. to the growth of the magnetic moment of the ND regions; an increase in φ leads to an increase in magnetoresistance. In this situation, stopping and holding the sample in the field $H_{\uparrow} = \mathrm{const}$ or $H_{\downarrow} = \mathrm{const}$ are equivalent to an increase or a decrease in the external field, respectively. In the case $H_{\downarrow} = 0$, vectors $\boldsymbol{m}_{\mathrm{ND}}$ disorient with time and the equilibrium value is apparently a quantity close to resistance of the sample upon zero-field cooling, $R(H_{\uparrow} = 0)$. In the decreasing field branch ($H = H_{\downarrow}$), on stopping the field ($H_{\downarrow} = \mathrm{const}$), the $R(t)$ dependence tends to a certain equilibrium value lower than $R(H_{\uparrow}, t = 0)$ at $H_{\uparrow} = H_{\downarrow}$, which implies the existence of an energy minimum[1].

Figure 3(*a*) shows the $R(t)$ dependences at $H_{\uparrow} = H_{\downarrow} = 30\,\mathrm{kOe}$ ($H_{\max} = 50\,\mathrm{kOe}$) in coordinates R and $\ln(t)$. For time $t \sim 10^2$–3×10^3 s, within experimental accuracy, the $R(t)$ dependences are approximated well by the logarithmic dependence

$$R(t)/R(t_0) \sim 1 \pm A \times \ln(t/t_0), \tag{1}$$

where A is a constant. The situation is analogous for other H values. Figures 3(*b*) and (*c*) demonstrate the dependences of residual resistance and residual magnetic moment after the external field $H_{\max} = 50\,\mathrm{kOe}$ is switched on/off. It can be seen that the logarithmic dependence

$$M(t)/M(t_0) = 1 - A' \times \ln(t/t_0) \tag{2}$$

exists also within the range $t \sim 10^2$–3×10^3 s. Regarding relaxation of magnetization in dc fields, due to smallness of the $M(H)$ hysteresis in the fields 10–50 kOe, the time variation in magnetization was kept within experimental accuracy.

Logarithmic relaxation of magnetization is known to be characteristic of spin glasses [16, 17] and was observed in ferromagnetic nanoparticles [18–20] and lanthanum manganites [10].

In the classical case of a small ferromagnetic particle considered by Néel [21], we have $M \sim M_0 \cdot \exp(-t/\tau)$, where τ is the characteristic relaxation time depending on the potential barrier overcome by the magnetic moment of a particle under the action of thermal fluctuations. Here, there are two allowed directions of the particle's magnetic moment: parallel and antiparallel to the field. However, the exponential dependence of relaxation is modified if one takes into consideration the distribution of the characteristic relaxation times, which may lead to logarithmic relaxation similar to (2) [16, 17, 22]. This concerns both spin glasses [16]

[1] To determine the equilibrium value of resistance at a certain field $H_{\uparrow} = H_{\downarrow}$, $H_{\max}$ should be higher than the field at which the $R(H)$ hysteresis vanishes. According to our data, the $R(H)$ hysteresis does not vanish up to $H = 90\,\mathrm{kOe}$. The $R(H)$ dependence does not saturate up to this value as well.

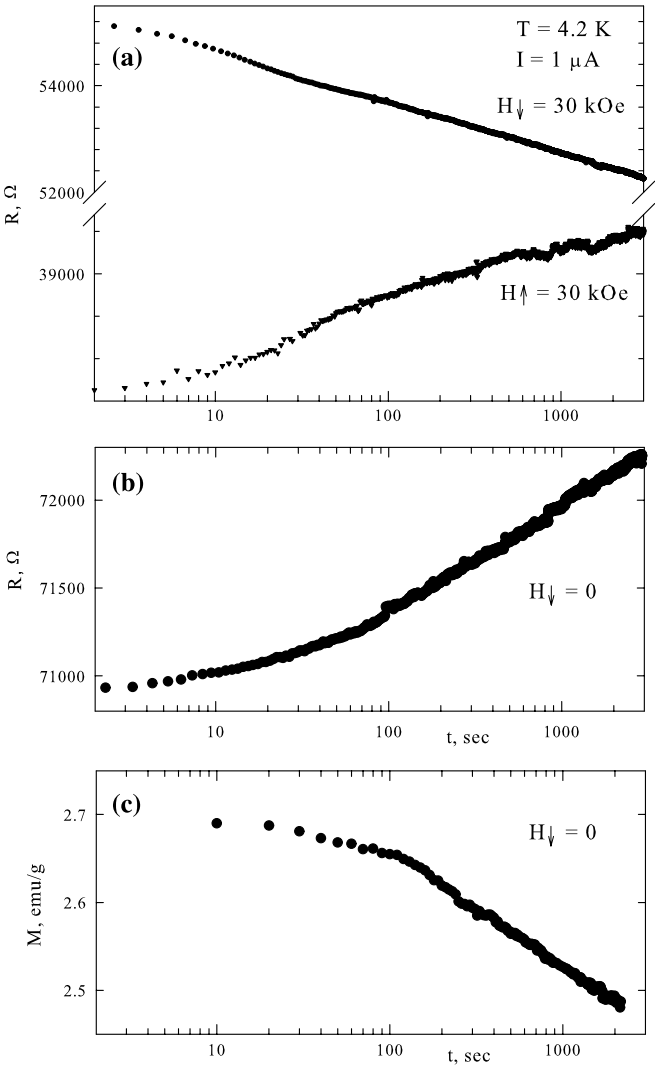

Figure 3. The $R(t)$ dependences at $H_{\uparrow} = H_{\downarrow} = 30\,\mathrm{kOe}$ (*a*), $H_{\downarrow} = 0\,\mathrm{kOe}$ (*b*) and $M(t)$ at $H_{\uparrow} = 0\,\mathrm{kOe}$ (*c*) in coordinates R and $\ln(t)$.

and small ferromagnetic particles [23]. Computer simulation of systems of ferromagnetic nanoparticles also shows that the $M(t)$ dependences may include portions with $M \sim \ln(t)$ [22, 24]. In [19, 20], the authors developed an alternative approach to the relaxation processes in an ensemble of interacting ferromagnetic nanoparticles that is analogous to the consideration of relaxation of magnetization in type-II superconductors [25]. If the barrier height for the magnetic moment of an individual particle depends on the resulting magnetic moment $U = U_0(1 - M/M_0)$ (M_0 was determined in [20]), then magnetization also changes in time following the law similar to (2).

In our case of magnetization relaxation, we may say that the processes occur in the regions neighbouring the antiferromagnetic spacer (ND regions); in other words, the data in figure 3(*c*) can be considered M_{ND} relaxation. This is probably the case that for magnetic moments $\boldsymbol{m}_{\mathrm{ND}}$ the exchange coupling with the antiferromagnetic spacer causes the vector $\boldsymbol{m}_{\mathrm{ND}}$ to overcome the external-field-dependent potential barrier.

The behaviours of the $M(t)$ and $R(t)$ dependences are interrelated and, in view of the aforesaid, it would be reasonable to suggest that R is a function of M_{ND} (the magnetic moment of the ND regions). At relaxation of the residual values (at $H_{\downarrow} = 0$) for the normalized quantities $\delta R(t) = \{R(t) - R(t^*)\}/R(H_{\uparrow} = 0)$ and $\delta M(t) = \{M(t^*) - M(t)\}/M(t^*)$, where t^* is the time of the beginning of the relaxation measurement, one may suggest the functional dependence

$$\delta R(t) = \delta M^n(t). \tag{3}$$

Substituting (3) into (2), we obtain

$$\delta R(t) = \{1 - A' \times \ln(t/t_0)\}^n. \tag{4}$$

In our case, the relaxation processes can be considered long-term: constant values in expressions (1) and (2) are much less than one: A, $A' \ll 1$. Accounting for the first term of the expansion of expression (4) in the Taylor series by small parameter A' yields the functional dependence $\delta R(t) = \{1 - A' \times n \times \ln(t/t_0)\}$, which is observed experimentally (figure 3(*b*)). Comparing the obtained expression with formula (1), we see that $A = A' \times n$.

The experimental data on the relaxation of residual values R and M presented in figures 3(*b*) and (*c*) are consistent with each other within the range $t \sim 10^2$–3×10^3 s at $n = 2.5$. In the calculation of $\delta R(t)$, the value $R(H_{\uparrow} = 0)$ was used that is equal to the resistance at point ***A*** in figure 2 and $t^* = 100$ s, i.e. after decreasing the external field to zero.

For the granular *ferromagnetic metal/insulator* films, it was predicted that $\delta R(H) = \delta M^2(H)$[Note 2] [26, 27], i.e. $n = 2$. Such behaviour was confirmed experimentally for the case when ferromagnetic particles do not interact with one another [27–31]. However, there might be deviations from this quadratic dependence when the particles interact [28, 32, 33] or the grains are considerably spread [34]. These factors may affect the obtained value of n in our case. Nevertheless, this value ($n = 2.5$) is close to the tunnel magnetoresistance predicted for a *ferromagnetic metal/insulator* granular system.

4. Conclusions

Being insulators, intergrain spacers in polycrystalline manganite determine the growth in resistance in the low-temperature region. The magnetoresistive effect at these temperatures is determined mainly by tunnel magnetoresistance on grain boundaries. The experimental data on magnetization and specific heat imply antiferromagnetic ordering of the spacers. It is reasonable to suggest that there is an exchange interaction between the antiferromagnetic spacer and ferromagnetic ND regions neighbouring the spacer. This apparently leads to the fact that for the directions of the magnetic moments of these regions there are many competing states with similar energies. These ND regions make relatively small contribution to the resulting magnetic moment of a sample as compared with the contribution of the grain core; therefore, the hysteresis is rather narrow. Orientation of the magnetic moments of the ND regions neighbouring the intergrain spacer is important for the tunnelling processes. This explains the large magnetoresistance hysteresis observed in polycrystalline manganite samples. The proposed mechanism is confirmed by the measurements of resistance relaxation in a dc field and after the field is switched off. Relaxation of magnetoresistance and the magnetic moment follows a logarithmic dependence within the range $t \sim 10^2$–3×10^3 s. Relaxation of the sample's magnetic moment observed in the sample may be thought of as originating from the disorientation of the magnetic moments of the ND regions, which also leads to resistance relaxation. A comparison of relaxations of the magnetic moment and resistance after the effect of the external field shows that the dependence of magnetoresistance on magnetization obeys the law $\delta R = \delta M^n$ at $n = 2.5$. The obtained value n is close to the characteristic value $n = 2$ for tunnel magnetoresistance of *ferromagnetic metal/insulator* granular systems.

[2] Here, normalized values R and M are calculated from the condition of saturation of the $R(H)$ and $M(H)$ dependences.

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