Fizika Nizkikh Temperatur, 2004, v. 30, No. 5, p. 551–557

Magnetoresistive study of the antiferromagnetic–weak ferromagnetic transition in single-crystal $La_2CuO_{4+\delta}$

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Received December 4, 2003

Resistive measurements were made to study the magnetic field-induced antiferromagnetic (AF) – weak ferromagnetic (WF) transition in the La_2CuO_4 single crystal. The magnetic field (dc or pulsed) was applied normally to the CuO_2 layers. The transition manifested itself in a drastic decrease of the resistance in critical fields of 5–7 T. The study is the first to display the effect of the AF–WF transition on the conductivity of the La_2CuO_4 single crystal in the direction parallel to the CuO_2 layers. The results provide support for the three-dimensional nature of the hopping conduction of this layered oxide.

PACS: 74.72.Dn, 75.30.Kz, 75.50.Ee

1. Introduction

Transport and magnetic properties of cuprate $La_2CuO_{4+\delta}$ have attracted considerable attention in the area of superconducting research. This is a parent compound for one of the family of high-temperature superconductors, and study of its properties is considered to be important for elucidation of still unclear nature of superconductivity in cuprates. Stoichiometric La_2CuO_4 ($\delta = 0$) is an antiferromagnetic (AF) insulator with a Néel temperature T_N of about 320 K, but doping it with bivalent metals (such as Sr) or with excess oxygen ($\delta \neq 0$) leads to destruction of the long-range AF order and a decrease in T_N [1–3]. A fairly high doping results in a transition to a metallic state.

The perovskite crystal lattice of La_2CuO_4 is orthorhombic (below about 530 K) consisting of CuO_2 layers separated by La_2O_2 layers (the latter consisting of two buckled La–O layers) [2,3]. In the *Bmab* space group the CuO_2 layers are perpendicular to the **c** axis and parallel to the *ab* plane [3]. The CuO_6 octahedra are tilted in a staggered way; the tilting is uniform in a given *cb* plane. The AF state is strongly connected with crystal lattice features [4]. The magnetic state is determined by d^9Cu^{2+} ions with spin $S = 0.5$. In the CuO_2 planes, the magnetic structure is characterized by a simple two-dimensional (2D) AF array with nearest neighbors having antiparallel moments [4]. Due to the above-mentioned tilting of the CuO_6 octahedra, the spins are canted 0.17° in the *cb* plane away from the **b** axis [4,5]. As a result, a weak ferromagnetic (FM) moment perpendicular to the CuO_2 plane appears in each layer. Below T_N, the directions of the FM moments are opposite in neighboring CuO_2 planes, so that the system as a whole is a three-dimensional (3D) AF [5].

Application of high enough magnetic field along the **c** axis causes a magnetic transition into a weak-ferromagnetic (WF) state, in which all canted moments are aligned along the field direction [5]. The transition is accompanied by a jump-like change in the resistivity [5]. The critical field H_c of the transition is temperature dependent. It goes to zero for T approaching T_N, but increases with decreasing temperature and amounts up to 5–6 T below 100 K. Hole doping of La_2CuO_4, leading to lower T_N, causes smaller H_c values as well (down to about 3 T at low temperature for samples with T_N about 100 K) [6]. Some magnetic transitions have also been found for field applied parallel to the *ab* plane [7]. In this case, for field parallel

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to **b** axis, a spin-flop transition was found at field H_1 about 10 T, and a transition to the FM state at a field H_2 of about 20 T. These transitions manifest themselves as weak knees (no jumps) in the MR curves [7]. It is believed that no magnetic transition should take place when field is applied parallel to the **a** axis, which is perpendicular to the staggered moments [7,8].

Doping with excess oxygen introduces charge carriers (holes) in the CuO_2 planes. At small enough $\delta(< 0.01)$, $La_2CuO_{4+\delta}$ remains insulating, althongh T_N is lowered considerably [9,10]. The excess oxygen atoms reside at interstitial sites between La–O planes [11]. Each such excess atom is surrounded by a tetrahedron of apical oxygen atoms. For layered cuprates, in which the CuO_2 planes are the main conducting units, a quasi-2D behavior is expected for the in-plane transport. This has actually been found in many cuprates [12] but not in $La_2CuO_{4+\delta}$. In this compound, the Mott's variable-range hopping (VRH), with temperature dependence of the resistance described by the expression

$$R \approx R_0 \exp\left(\frac{T_0}{T}\right)^{1/4}, \qquad (1)$$

is found [13,14] at low T for both the in-plane (current **J** parallel to the CuO_2 planes) and out-of-plane (**J**|| **c**) transport. The fractional exponent in Eq. (1) equal to 1/4 corresponds to 3D system (for 2D systems, it should be equal to 1/3) [15]. At the same time, the hopping conduction in $La_2CuO_{4+\delta}$ samples with fairly high crystal perfection shows a considerable anisotropy, so that the values of R_0 and T_0 in Eq. (1) are different for the in-plane and out-of-plane transport. The in-plane conductivity σ_{ab} is found to be considerably higher than the out-of-plane conductivity σ_c. The ratio σ_{ab}/σ_c is strongly temperature dependent. It is minimal (about 10) in the liquid-helium temperature range, but increases dramatically with temperature and saturates above 200 K to maximal values of the order of 100 [16–18].

The 3D character of VRH in $La_2CuO_{4+\delta}$ testifies that a hole transfer between CuO_2 is likely not only at **J**|| **c**, but at **J**|| **a**, **b** as well. In considering this question it is important to know the exact nature of the holes in $La_2CuO_{4+\delta}$. Although about 17 years has passed since the discovery of superconductivity in doped $La_2CuO_{4+\delta}$, the nature of the holes in it still cannot be considered completely clear. This in turn makes it hard to gain insight into the nature of the cuprate's superconductivity. In the undoped state, the CuO_2 planes present a lattice of d^9Cu^{2+} $(S = 0.5)$ and p^6O^{2-} $(S = 0)$ ions. Doping with excess oxygen causes (to ensure neutrality) the appearance of additional holes in the planes. This can be achieved in two ways: 1) some of the d^9Cu^{2+} ions change into the $d^8Cu^{3+}(S = 0)$ state, or 2) some of the in-plane oxygen ions p^6O^{2-} change into the p^5O^{1-} $(S = 0.5)$ state. In either case, the holes induce strong local perturbations of the AF order. In the known literature [19–25], both kinds of holes have been taken into account in theoretical models of fundamental properties of the cuprates. There is much speculation, however, that holes in $La_2CuO_{4+\delta}$ have a strong oxygen character [19–24], and this view has strong experimental support [20,22,26]. At the same time, due to the overlapping of the d and p orbitals and hybridization of the d and p bands, the d orbitals exert a significant influence on the hole motion.

According to Ref. 21, owing to the special the character of the excess oxygen as interstitial atoms [11] with weak oxygen—oxygen bonding, the holes can be delocalized from the CuO_2 planes onto the apical O atoms, i.e., into the $La_2O_{2+\delta}$ region between adjacent CuO_2 planes. This assures the 3D nature of VRH in $La_2CuO_{4+\delta}$. In this way the $La_2CuO_{4+\delta}$ differs drastically from the Sr-doped system, where the holes remain quasi-two-dimensional. In fact, the ratio σ_{ab}/σ_c in lightly doped $La_{1-x}Sr_xCuO_4$ crystals of good quality can be as high as several thousand [27].

In this communication we report the results of a study of the AF–WF transition by magnetoresistance (MR) measurements in a $La_2CuO_{4+\delta}$ single crystal. In the known previous studies [5,28–31] the MR investigations of AF–WF transition in $La_2CuO_{4+\delta}$ were done for the case when both the magnetic field and transport current are perpendicular to the CuO_2 planes (i.e., **H**|| **c** and **J**|| **c**). Under these conditions a rather sharp decrease in the resistance has been found as the critical field H_c was approached from below. The amplitude of the relative change in resistance ($\Delta R/R_n$, where R_n is the resistance in the AF state) due to the AF–WF transition depends on temperature. It is maximal in the range 20–30 K, where it can amount to 0.30–0.50 in fairly perfect crystals [5,7,29–31].

It is known that the enhancement of spin order usually leads to a decrease in resistivity of metallic systems. For example, a considerable decrease in resistivity can occur at transitions from the paramagnetic to the FM or AF state in some metals, alloys or even in some FM perovskite oxides, like mixed-valence manganites [32–34]. This is usually attributed to a decrease in the scattering rate of quasi-free charge carriers on disordered local spins as a result of the above-mentioned magnetic transitions. The situation is rather different in the case of insulating $La_2CuO_{4+\delta}$. Here the transition to the 3D AF state produces hardly any noticeable change in the hopping conductivity at T_N (apparently for the reason that 2D AF correlations in the CuO_2 planes persist up to temperatures far above T_N [1–3]). But transition to the 3D WF state increases the conductivity enormously. Since VRH in $La_2CuO_{4+\delta}$ has a pronounced 3D character, it can be expected that the AF–WF transition would manifest itself in resistivity in field $\mathbf{H} \| \mathbf{c}$ not only for the transport current perpendicular to the CuO_2 planes, as was found in Refs. 5, 29–31, but for the in-plane hole transport as well. In this study this effect has been actually revealed, as described below.

2. Experimental

A single-crystal $La_2CuO_{4+\delta}$ sample with dimensions of 1.3×0.3×0.39 mm is investigated. This sample was studied previously in Ref. 18, where it was indicated as sample No. 1 with T_N = 188 K. After that study, the sample was annealed additionally in an oxygen atmosphere (700 °C, 5.5 days) in the hope that oxygen content (that is, δ) would be increased. It turned out, however, that the thermal treatment caused only a slight decrease in T_N (down to 182 K) and in the resistivity. The T_N value was determined from magnetic susceptibility measurements.

The crystallographic orientation of the sample was determined from an x-ray diffraction study. This reveals that the sample has a quantity of twins, which inevitably appear in $La_2CuO_{4+\delta}$ crystals when cooled through tetragonal-to-orthorhombic structural transition at $T \approx 530$ K [3]. As a result, a peculiar domain structure is developed. The orientation of the **c** axis is the same in each domain, but the orientations of the **a** and **b** axes are switched (or reversed) in a fixed way between two possible orientations upon crossing the domain (twin) boundaries. In this connection, although we will speak conventionally in the following about **a** or **b** directions of transport current in the sample studied, they should be taken, first of all, as the two in-plane current directions in the twinned crystal, which are perpendicular to each other. In a heavily twinned crystal no significant anisotropy in the in-plane conductivity can be expected even assuming that some intrinsic conductivity anisotropy within the CuO_2 planes is present. We have found, however, a pronounced anisotropy in the conductivity (and a rather significant one in the MR) for these two in-plane directions. This matter will be touched upon in the next Section of the paper. In contrast, we can speak about the *c* directions in the sample studied without any reservation or possible misunderstanding.

In this study, the dc resistance in the directions parallel to CuO_2 planes was measured by the Montgomery method [35], which is appropriate for systems with a pronounced anisotropy of the conductivity. Contacts between the measuring wires and the sample were made using a conducting silver paste. The measurements were done in field $\mathbf{H} \| \mathbf{c}$ in a helium cryostat with a superconducting solenoid. Although the maximum field in the cryostat (about 6 T) has appeared to be quite sufficient in most cases to reveal manifestations of the AF–WF transition in MR of the sample studied, a somewhat higher field is needed to study the transition more thoroughly, especially for the study of hysteretic phenomena in the $R(H)$ curves in the vicinity of the critical field H_c [5,29–31]. This hysteretic behavior is considered as an indication of a first-order transition. For this reason, a part of the dc resistance measurements in this study were done in pulsed magnetic field with amplitude up to 15 T. The nearly sinusoidal pulse has a duration about 33 ms, during which the field is swept from zero to a maximum amplitude and back to zero. For these measurements the field $\mathbf{H} \| \mathbf{c}$ and transport currents, $\mathbf{J} \| \mathbf{c}$ and $\mathbf{J} \| \mathbf{a}$, were used. The rate of variations in magnetic field was up to 10^3 T/s. Other essential details of the pulse measuring technique employed can be found in Ref. 36.

3. Results and discussion

The temperature dependences of the resistivity ρ_a measured along the **a** axis ($\mathbf{J} \| \mathbf{a}$) is shown in Fig. 1 for different magnitudes of the measuring current. It is seen that the $\rho(T)$ behavior does not depend essentially on current in the whole measuring temperature

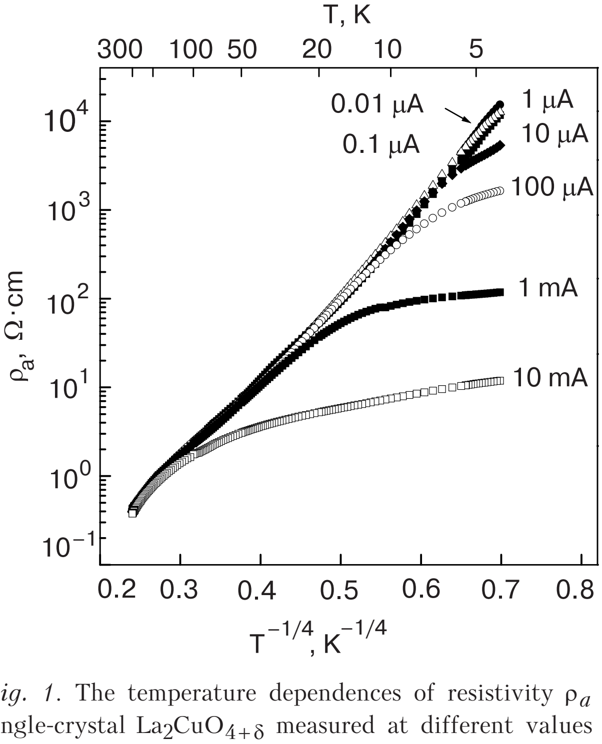

Fig. 1. The temperature dependences of resistivity ρ_a of single-crystal $La_2CuO_{4+\delta}$ measured at different values of transport current. In all cases the current was directed parallel to the crystallographic axis **a**. The dependences are presented as $\lg \rho_a$ versus $T^{-1/4}$.

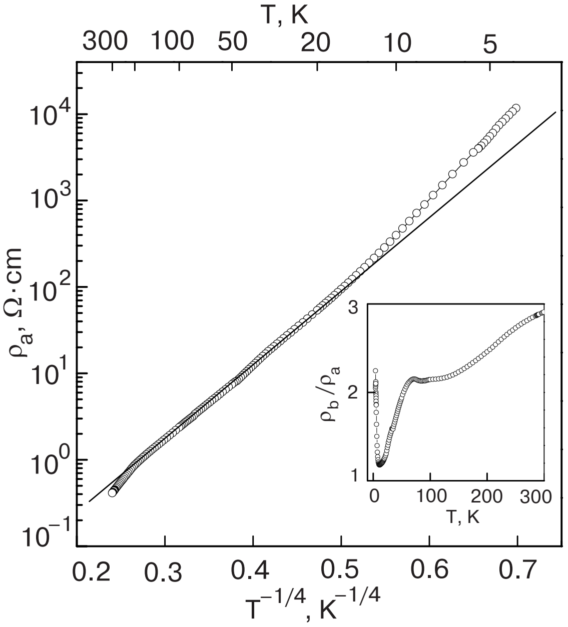

Fig. 2. The temperature dependence of resistivity ρ_a of single-crystal $La_2CuO_{4+\delta}$ measured for transport current equal to 1 μA. The current was directed parallel to the crystallographic axis **a**. The inset shows temperature behavior of the ratio of the resistivities ρ_b and ρ_a for measuring currents directed along the crystallographic axes **b** and **a**.

range, $4.2\ K \le T \le 300\,K$, for current magnitude less than about 1 μA; that is, Ohm's law holds in this case. For better consideration, one of these ohmic $\rho(T)$ curves (at $J = 1$ μA) is presented separately in Fig. 2. It can be seen that Mott's law [Eq. (1)] is obeyed fairly well in the range $20\ K \lesssim T \lesssim 200$ K. In the range $T < 20$ K, a steeper [as compared to Eq. (1)] increase in R with decreasing temperature is found. This deviation from Mott's law at low temperature is rather typical for $La_2CuO_{4+\delta}$ and was observed earlier in Refs. 14, 31. In Ref. 14, a possible reason for this behavior is suggested: the presence of superconducting inclusions in the insulating sample due to phase separation in $La_2CuO_{4+\delta}$.

Magnetic structure of $La_2CuO_{4+\delta}$, according to neutron diffraction data [2,4], is anisotropic for all three orthorhombic axes. The same can be expected, therefore, for transport and magnetic properties. In the presence of twins, however, the measured transport and magnetic properties usually show quite definite anisotropy solely for directions parallel and perpendicular to the CuO_2 planes. Recently, in untwinned $La_2CuO_{4+\delta}$ crystals, a clear in-plane anisotropy of the magnetic susceptibility χ was found [37]. A similar phenomenon may be expected in the transport properties of $La_2CuO_{4+\delta}$ samples without twins.

In a sample with multiple twins, no considerable in-plane anisotropy could be expected. The measured ratio ρ_b/ρ_a in the sample studied (see inset in Fig. 2) reveals, however, a rather distinct anisotropy. The ratio is close to unity at $T \approx 11$–12 K, but it increases with temperature and approaches value of about 3 at room temperature. A similar behavior was found in the previously studied sample with somewhat higher $T_N \approx 188$ K [18]. The *a–b* anisotropic conductivity behavior in a twinned sample (in the case that the conductivities σ_a and σ_b are inherently different) can be observed only when, *first*, the existing twins are few in number (so that the measured resistivity is not properly averaged between the two possible crystal orientations), and, *second*, a given current direction is really parallel to the **a** (or **b**) axis in most of the crystal. The results of this study therefore give evidence that the intrinsic conductivity anisotropy in the CuO_2 planes of $La_2CuO_{4+\delta}$ is quite credible.

We found that the MR behavior of the sample studied depends significantly on the magnitude of the measuring current, especially at low temperature. The upper panel of Fig. 3 presents the MR curves recorded at $T = 5$ K for the case $\mathbf{J} \| \mathbf{a}$. It can be seen that for low currents (that is in the Ohmic regime) the MR is positive, but for high enough currents ($J \ge 1$ μA) the MR becomes negative and strongly increases above $H \simeq 5$ T. Positive MR was observed only at low temperature ($T < 20$ K) for both the in-plane current

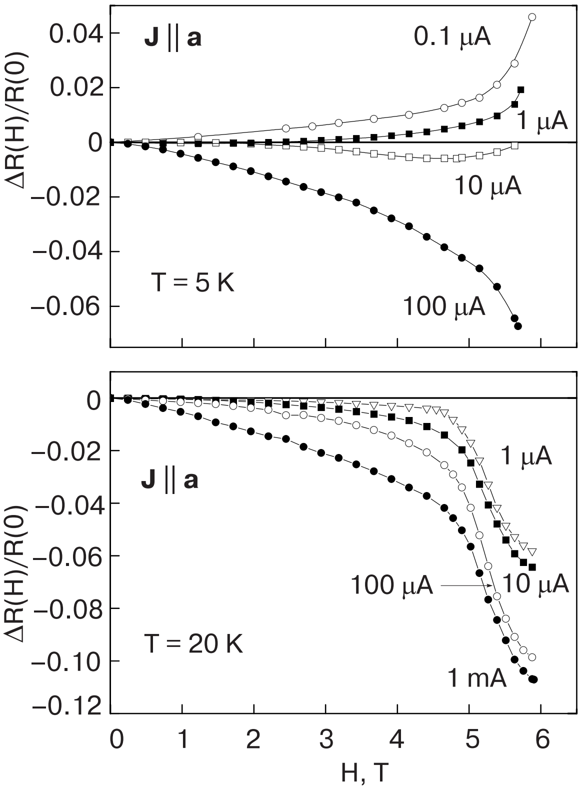

Fig. 3. Magnetoresistance curves at $T = 5$ and 20 K measured for single-crystal $La_2CuO_{4+\delta}$ in the out-of-plane dc magnetic field ($\mathbf{H} \| \mathbf{c}$) for different amplitudes of measuring current directed along the crystallographic axis **a**.

directions used, $\mathbf{J} \| \mathbf{a}$ and $\mathbf{J} \| \mathbf{b}$. At fairly high temperature, $T \geq 20$ K, only negative MR is observed, which increases profoundly above $H \simeq 5$ T, as well (lower panel of Fig. 3). We have attributed this rather sharp increase to an influence of the AF–WF transition, as will be discussed in more detail below. As to the positive MR at low temperature ($T < 20$ K), this could be attributed to the presence of superconducting inclusions due to phase separation, as was mentioned above. For example, in Ref. 38, positive MR attributed to superconducting inclusions has been found in the low-temperature range ($T < 10$ K) in even more resistive $\mathrm{La_2CuO_{4+\delta}}$ with higher T_N.

For all of the temperature range in which the MR was measured in this study ($4.2\ \mathrm{K} \leq T \leq 90$ K), the MR magnitude is strongly dependent on the measuring current (as illustrated by Fig. 3). For this reason, to compare MR curves with an evident effect of AF–WF transition at different temperatures we have used only data for rather high currents, that is for the non-Ohmic conduction regime. Some examples of the MR curves at $\mathbf{H} \| \mathbf{c}$ for the cases $\mathbf{J} \| \mathbf{a}$ and $\mathbf{J} \| \mathbf{b}$ and current $J = 100$ μA are shown in Figs. 4 and 5 for certain selected temperatures. It is obvious from the curves that a rather sharp decrease in resistance occurs when H exceeds some critical magnitude (in the range 5–6 T). All main features of this resistive transition are quite identical to those found in MR behavior of $\mathrm{La_2CuO_{4+\delta}}$ at the AF–WF transition for the case $\mathbf{H} \| \mathbf{c}$, and the out-of-plane current direction ($\mathbf{J} \| \mathbf{c}$), when mainly interplane hopping is affected by the transition [5,28–30]. The results obtained show that the AF–WF transition influences hopping conduction in the directions parallel to $\mathrm{CuO_2}$ planes as well. This effect, although being anticipated (as is indicated above), have never been seen previously in $\mathrm{La_2CuO_{4+\delta}}$, to our knowledge.

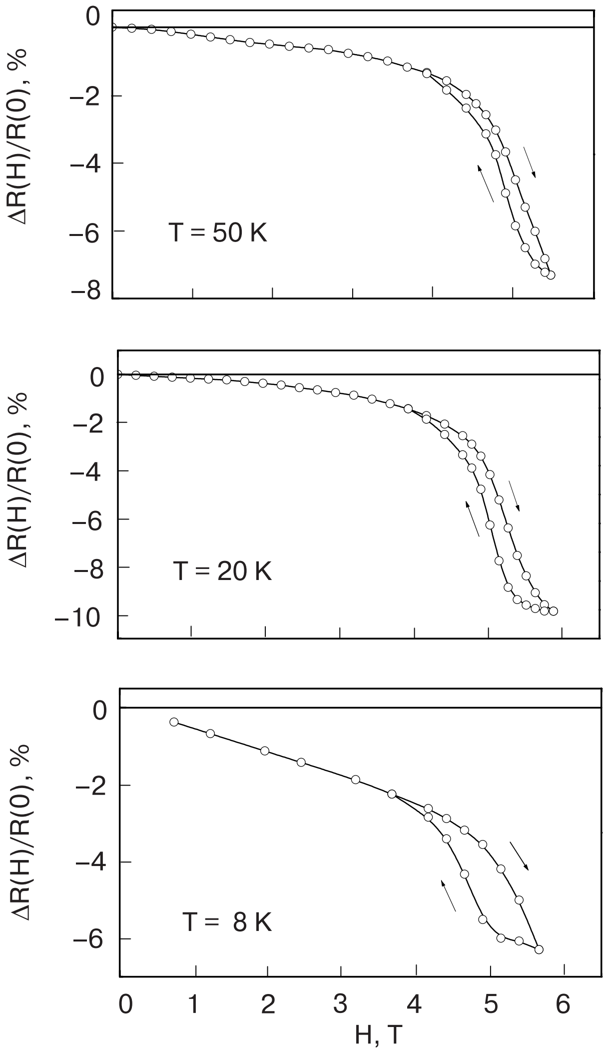

Fig. 4. Magnetoresistance curves at various fixed temperatures measured for single-crystal $\mathrm{La_2CuO_{4+\delta}}$ in the out-of-plane dc magnetic field ($\mathbf{H} \| \mathbf{c}$) for measuring current (100 μA) directed along the crystallographic axis **a**.

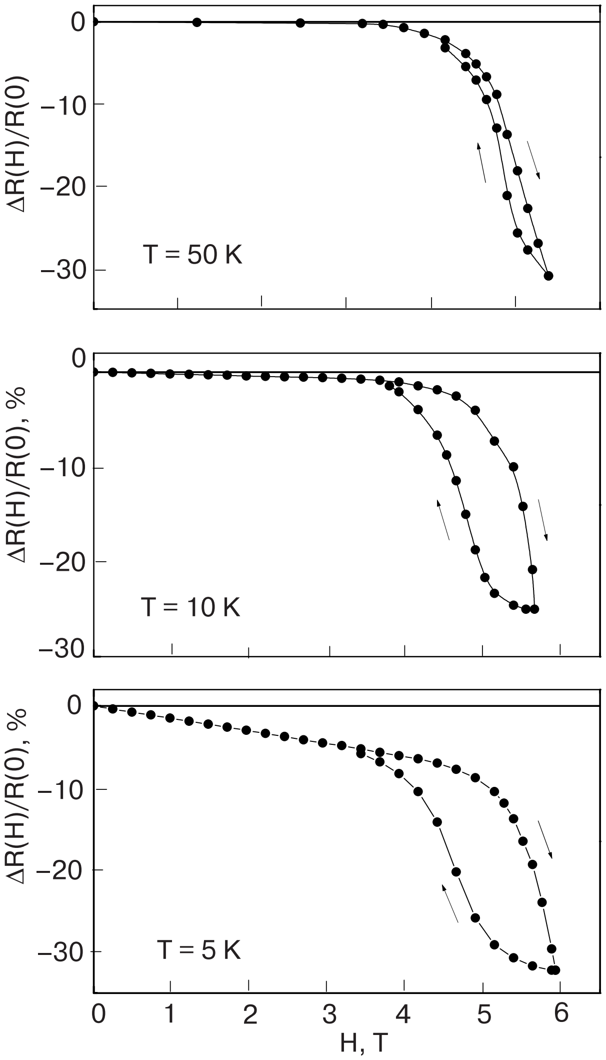

Fig. 5. Magnetoresistance curves at various fixed temperatures measured for single-crystal $\mathrm{La_2CuO_{4+\delta}}$ in the out-of-plane dc magnetic field ($\mathbf{H} \| \mathbf{c}$) for measuring current (100 μA) directed along the crystallographic axis **b**.

The following features of the resistive transition can be pointed out. First, the transition is sharper and the relative changes in resistance, $\Delta R/R_n$, are larger for the **b** direction of the transport current than those for **a** direction (compare Figs. 4 and 5). Second, the MR curves are hysteretic in the field range of the transition, as expected. The hysteresis becomes more pronounced for decreasing temperature. The latter feature of the MR curves is quite consistent with that found previously at the AF–WF transition for $\mathbf{J} \| \mathbf{c}$ [5,29–31]. Third, a considerable negative MR in the low-field range below the magnetic transition can be observed (Figs. 4 and 5). This contribution to the total MR is not hysteretic and, maybe, has little if any relationship to the magnetic transition. For a given current (for example, for $J = 100\ \mu\text{A}$, as in Figs. 4 and 5) the contribution of this type of MR increases with decreasing temperature and is more pronounced for the **a** direction of the measuring current. It is found as well that the negative MR at low field increases with current magnitude (Fig. 3) and, therefore, with an applied voltage, so it is much more pronounced in the non-Ohmic regime of hopping conductivity (compare Fig. 1 and 3). In previous studies, negative MR in the AF $La_2CuO_{4+\delta}$ for the case of both the current and field parallel to CuO_2 was found and discussed to a certain degree [14,18]. The nature of the negative MR in rather low field $\mathbf{H} \| \mathbf{c}$ and $\mathbf{J} \| \mathbf{a}, \mathbf{b}$ revealed in this work is not clear and is worthy of additional study.

It is evident from Figs. 4 and 5 that the maximal dc field about 6 T, used for these measurements, is not high enough to accomplish the magnetic transition in full measure. To overcome this disadvantage, measurements were done in pulsed magnetic field with magnitude up to 15 T. The MR curves were recorded at temperatures $T = 4.2$ K, 20.4 K, and 77 K for both the in-plane and out-of-plane directions of the transport current. Examples of MR curves for pulsed field at $T = 4.2$ and $T = 77$ K are shown in Figs. 6 and 7.

The pulsed MR measurements enabled us to see the magnetic transitions in full measure. The MR curves for low temperature region were found to be quite similar for both methods (compare Figs. 4 and 6). It is also seen that the resistive transition for the out-of-plane

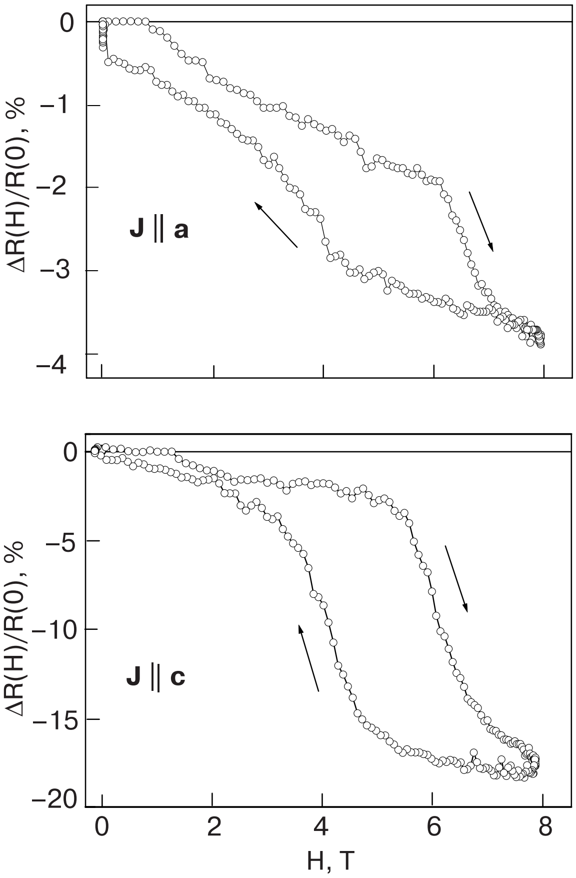

Fig. 6. Magnetoresistance curves registered for single-crystal $La_2CuO_{4+\delta}$ in the out-of-plane pulsed magnetic field $(\mathbf{H} \| \mathbf{c})$ at $T = 4.2$ K for the in-plane and out-of-plane current directions $(\mathbf{J} \| \mathbf{a}$ and $\mathbf{J} \| \mathbf{c})$ with current magnitudes 6 μA and 7.4 μA, respectively.

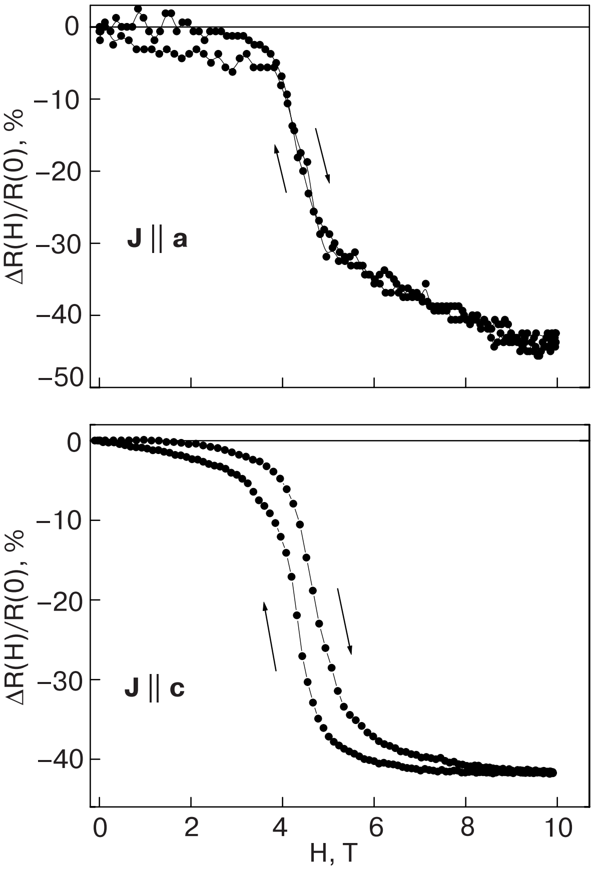

Fig. 7. Magnetoresistance curves registered for single-crystal $La_2CuO_{4+\delta}$ in the out-of-plane pulsed magnetic field $(\mathbf{H} \| \mathbf{c})$ at $T = 77$ K for the in-plane and out-of-plane current directions $(\mathbf{J} \| \mathbf{a}$ and $\mathbf{J} \| \mathbf{c})$ with current magnitudes 5.93 mA and 178 μA, respectively.

current direction is sharper, and the relative changes in resistance, $\Delta R/R_n$, are generally larger than those for in-plane direction. The MR curves in pulsed magnetic field at $T = 77$ K are less hysteretic than those at $T = 4.2$ K, as expected (Fig. 7). The maximum values of $\Delta R/R_n \approx 50$ % found in this study for pulsed magnetic field agree well with those found in previous studies in dc magnetic field [7].

In conclusion, we have found that the AF–WF transition in $La_2CuO_{4+\delta}$ clearly manifests itself in the in-plane hopping conductivity. This supports the 3D nature of hopping conduction in this compound.

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