Magnetoresistive study of antiferromagnetic–weak ferromagnetic transition in single-crystal La$_2$CuO$_{4+\delta}$

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The resistive measurements were made to study the magnetic field-induced antiferromagnetic (AF) - weak ferromagnetic (WF) transition in La$_2$CuO$_4$ 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$_2$CuO$_4$ single-crystal in the parallel - to - CuO$_2$ layers direction. The results provide support for the 3-dimensional nature of the hopping conduction of this layered oxide.

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I. INTRODUCTION

Transport and magnetic properties of cuprate La$_2$CuO$_{4+\delta}$ attract up to now a 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$_2$CuO$_4$ ($\delta = 0$) is an antiferromagnetic (AF) insulator with Néel temperature, $T_N$, about 320 K, but doping it with bivalent metals (such as Sr) or with excess oxygen ($\delta \neq 0$) leads to violation of long-range AF order and decrease in $T_N$. A fairly high doping results in transition to metallic state.

Perovskite crystal lattice of La$_2$CuO$_4$ is an orthorhombic (below about 530 K) consisting of CuO$_2$-layers separated by La$_2$O$_2$ layers (the latter consisting of two buckled La-O layers). In Bmab space group, the CuO$_2$ layers are perpendicular to the $c$ axis and parallel to $a$-$b$ plane. The CuO$_6$ octahedra are tilted in a staggered way; the tilting is uniform in a given $c$-$b$ plane. The AF state is strongly connected with crystal lattice features. The magnetic state is determined by $d^9$Cu$^{2+}$ ions with spin $S = 0.5$. In the CuO$_2$ planes, the magnetic structure is characterized by simple two-dimensional (2D) AF array with nearest neighbors having antiparallel moments. Due to the above-mentioned tilting of the CuO$_6$ octahedra, the spins are canted 0.17° in the $c$-$b$ plane away from the $b$ axis. As a result, a weak ferromagnetic (FM) moment perpendicular to CuO$_2$ plane appears in each layer. Below $T_N$, the directions of FM moments are opposite in neighboring CuO$_2$ planes, so that the system as a whole is three-dimensional (3D) AF.

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. The transition is accompanied by a jump-like change in the resistivity. The critical field $H_c$ of the transition is temperature dependent. It goes to zero for $T$ approaching $T_N$, but increases with temperature decreasing and amounts up to 5-6 T below 100 K. Hole doping of La$_2$CuO$_4$ leading to smaller $T_N$ causes smaller $H_c$ values as well (down to about 3 T at low temperature for samples with $T_N$ about 100 K). Some magnetic transitions have also been found for field applied parallel to $a$-$b$ plane. In this case, for field parallel to $b$ axis, a spin-flop transition was found at field $H_1$ about 10 T, and a transition to FM state at field $H_2$ about 20 T. These transitions manifest themselves as weak knees (no jumps) in MR curves. It is believed that no magnetic transition should take place when field is applied parallel to axis $a$, which is perpendicular to the staggered moments.

The doping with excess oxygen introduces charge carriers (holes) in CuO$_2$ planes. At small enough $\delta$ ($< 0.01$), La$_2$CuO$_{4+\delta}$ remains insulating, even if $T_N$ reducing considerably. The excess oxygen atoms reside at an interstitial sites between LaO planes. Each of such excess atom is surrounded by tetrahedron of apical oxygen atoms. For layered cuprates, in which the CuO$_2$ planes are the main conducting units, a quasi 2D behavior to be expected for the in-plane transport. This has really been found in many cuprates, but not in La$_2$CuO$_{4+\delta}$. In this compound, the Mott’s variable-range hopping (VRH) with temperature dependence of resistance described by expression,

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

is found at low $T$ for both, the in-plane (current $J$ parallel to CuO$_2$ planes) and out-of-plane ($J//c$) transport. Fractional exponent in Eq. $\dagger$ is equal to 1/4 corresponds to 3D system (for 2D systems, it should be
The 3D character of VRH in La$_2$CuO$_{4+\delta}$ testifies that a hole transfer between CuO$_2$ is likely not only at $J \parallel c$, but at $J \parallel \{a, b\}$ as well. At considering this question it is important to know the exact nature of holes in La$_2$CuO$_{4+\delta}$. Although about 17 years has passed since the discovery of superconductivity in doped La$_2$CuO$_{4+\delta}$, the nature of holes in it still cannot be considered as completely clear. This in turn hampers to gain an insight into the nature of cuprate’s superconductivity. In the undoped state, CuO$_2$ planes present a lattice of $d^9$Cu$^{2+}$ ($S = 0.5$) and $p^6$O$^{-2}$ ($S = 0$) ions. Doping with excess oxygen causes (to ensure neutrality) appearance of additional holes in the planes. This can be achieved in two ways: 1) some of the $d^9$Cu$^{2+}$ ions change into $d^8$Cu$^{3+}$ ($S = 0$) state, or 2) some of the in-plane oxygen ions $p^6$O$^{-2}$ change into $p^5$O$^{-1}$ ($S = 0.5$) state. In either case, the holes induce strong local perturbations of AF order. In known literature [19, 20, 21, 22, 23, 24, 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$_2$CuO$_{4+\delta}$ have a strong oxygen character [19, 20, 21, 22, 23, 24, 25], and this view has a strong experimental support [21, 22, 23]. At the same time, due to overlapping of $d$- and $p$-orbitals and hybridization of $d$- and $p$-bands, $d$-orbitals exert a significant influence on the hole motion.

According to Ref. [21], owing to special character of the excess oxygen as interstitial atom [11] with weak oxygen-oxygen bonding, the holes can be delocalized from CuO$_2$ planes onto the apical O atoms, i.e. into La$_2$O$_{2+\delta}$ region between adjacent CuO$_2$ planes. This assures the 3D nature of VRH in La$_2$CuO$_{4+\delta}$. In this way the La$_2$CuO$_{4+\delta}$ differs drastically from Sr doped system, where the holes remain quasi two-dimensional. Really, the ratio $\sigma_{ab}/\sigma_c$ in lightly doped La$_{1-x}$Sr$_x$CuO$_4$ crystals of good quality can be as high as several thousands [27].

In this communication, we report the results of study of AF-WF transition by magnetoresistance (MR) measurements in a La$_2$CuO$_{4+\delta}$ single crystal. In known previous studies [1, 2, 3, 4, 5, 6, 7] the MR investigations of AF-WF transition in La$_2$CuO$_{4+\delta}$ were done for the case when both the magnetic field and transport current are perpendicular to the CuO$_2$ planes (i.e., $H \parallel c$ and $J \parallel c$). Under these conditions a rather sharp decrease in the resistance has been found as the critical field $H_c$ was approached from below. Amplitude of the relative change in resistance ($\Delta R/R_n$, where $R_n$ is the resistance in the AF state) due to AF-WF transition depends on temperature. It is maximal in the range 20-30 K, where it can amount up to 0.30-0.50 in fairly perfect crystals [1, 2, 3, 4, 5, 6, 7]. It is known that the enhancement of spin order usually leads to decrease in resistivity of metallic systems. For example, a considerable decrease in resistivity can occur at transitions from paramagnetic to FM or AF state in some metals, alloys or even in some FM perovskite oxides, like mixed-valence manganites [32, 33, 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$_2$CuO$_{4+\delta}$. Here the transition to 3D AF state produces hardly any noticeable change in the hopping conductivity at $T_N$ (apparently for the reason that 2D AF correlations in CuO$_2$ planes persist up to temperatures far above $T_N$ [1, 2, 3]). But transition to the 3D WF state increases the conductivity enormously. Since VRH in La$_2$CuO$_{4+\delta}$ has a pronounced 3D character, it can be expected that AF-WF transition would manifest itself in resistivity in field $H \parallel c$ not only for the transport current perpendicular to the CuO$_2$ planes, as it was found in Refs. [3, 4, 5, 6, 7], but for the in-plane hole transport as well. In this study this effect has been actually revealed, as described below.

II. EXPERIMENTAL

A single-crystal La$_2$CuO$_{4+\delta}$ sample with dimensions 1.3$\times$0.3$\times$0.39 mm is investigated. This sample was studied previously in work [18], where it was indicated as a sample No. 1 with $T_N = 188$ K. After that study, the sample was annealed additionally in oxygen atmosphere (700°C, 5.5 days) in the hope that oxygen content (that is, $\delta$) will be increased. It turns out, however, that the thermal treatment has 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$_2$CuO$_{4+\delta}$ crystals when cooled through tetrahedral to orthorhombic structure transition at $T \approx 530$ K [3]. As a result, a peculiar domain structure is developed. Orientation of $c$-axis is the same in each domain, but orientations of $a$ 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 Cu$_2$O$_2$ planes takes place. 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 c-directions in the sample studied without any reservation or possible misunderstanding.

In this study, the DC resistance in the directions parallel to Cu$_2$O$_2$ planes was measured by the Montgomery method [32], 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 $H||c$ in a helium cryostat with a superconducting solenoid. Although maximum field in the cryostat (about 6 T) has appeared to be quite sufficient in most cases to reveal manifestation 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 study of hysteretic phenomena in $R(H)$ curves in vicinity of critical field $H_c$. This hysteretic behavior is considered as an indication of the first-order transition. For this reason, a part of DC resistance measurements in this study were done in pulse magnetic field with amplitude up to 15 T. A 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 $H||c$ and transport currents, $J||c$ and $J||a$, 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].

III. RESULTS AND DISCUSSION

The temperature dependences of resistivity $\rho_a$, measured along axis $a$ ($J||a$), is shown in Fig. 1 for different magnitudes of measuring current. It is seen that $\rho(T)$ behavior does not depend essentially on current in the whole measuring temperature range, 4.2 K $\leq T \leq$ 300 K, for current magnitude lesser than about 1 $\mu$A; that is Ohm’s law holds in this case. For better consideration, one of these ohmic $R(T)$ curves (at $J = 1 \mu$A) 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 $\leq T \leq$ 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$_2$CuO$_{4+\delta}$, and was observed earlier in Refs. [14, 31]. In Ref. [14], a possible reason for this behavior is suggested to be determined by the presence of superconducting inclusions in insulating sample due to phase separation in La$_2$CuO$_{4+\delta}$.

Magnetic structure of La$_2$CuO$_{4+\delta}$, according to neutron diffraction data [2, 3], 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 show usually quite definite anisotropy solely for directions parallel and perpendicular to the Cu$_2$O$_2$ planes. Recently, in untwinned La$_2$CuO$_{4+\delta}$ crystals, a clear in-plane anisotropy of the magnetic susceptibility $\chi$ was found [37]. A similar phenomenon may be expected in transport properties of La$_2$CuO$_{4+\delta}$ samples without twins.

In a sample with multiple twins, no considerable in-plane anisotropy could be expected. The measured ratio $\rho_b/\rho_a$ 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 $\sigma_a$ and $\sigma_b$ are inherently different) can be observed only when, first, available twins are few in number (so that measured resistivity is not properly averaged between the two possible crystal orientations), and, second, a given current direction is really parallel to axis $a$ (or $b$) in the most part of the crystal. The results of this study give, therefore, an evidence that the intrinsic conductivity anisotropy in Cu$_2$O$_2$ planes of La$_2$CuO$_{4+\delta}$ is quite credible.

We found that MR behavior of the sample studied depends significantly on magnitude of measuring current, especially at low temperature. Upper panel of Fig. 3 presents the MR curves recorded at $T = 5$ K for the case $J||a$. It can be seen that for low currents (that is in ohmic regime) the MR is positive, but for high enough currents ($J \geq 1 \mu$A) the MR becomes negative and strongly increases above $H \approx 5$ T. Positive MR was observed only at low temperature ($T < 20$ K) for both the in-plane current directions used, $J||a$ and $J||b$. At fairly high temperature, $T \geq 20$ K, only negative MR is observed, which increases profoundly above $H \approx 5$ T, as well (lower panel of Fig. 3). We have attributed this rather sharp increase to an influence of the AF-WF transition, as it will be discussed in more detail below. As for the positive MR at low temperature ($T < 20$ K), this could be attributed to the presence of superconducting inclusions due to phase separation as it was mentioned above. For example, in Ref. [36], positive MR attributed to superconducting inclusions has been found at low temperature range ($T < 10$ K) in even more resistive La$_2$CuO$_{4+\delta}$ with higher $T_N$.

For all temperature range, where MR is measured in this study (4.2 K $\leq T \leq$ 90 K), the MR magnitude was strongly dependent on 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 non-ohmic conduction regime. Some examples of the MR curves at $H||c$ for the cases $J||a$ and $J||b$ and
current \( J = 100 \, \mu A \) 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 MB behavior of \( \text{La}_2\text{CuO}_{4+\delta} \) at the AF-WF transition for the case \( H \parallel c \), and the out-of-plane current direction \( (J \parallel c) \), when mainly inter-plane hopping is affected by the transition \( \parallel \text{CuO}_2 \) planes as well. This effect, although being anticipated (as it is indicated above), have never been seen previously in \( \text{La}_2\text{CuO}_{4+\delta} \) to our knowledge.

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 \( b \)-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 \( J \parallel c \) \([5, 28, 29, 30]\). Third, a considerable negative MR in 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 a little if any relationship to the magnetic transition. For a given current (for example, for \( J = 100 \, \mu A \), as in Figs. 4 and 5) the contribution of this type of MR increases with temperature decreasing and is more pronounced for \( a \)-direction of 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 non-ohmic regime of hopping conductivity (compare Fig. 1 and Fig. 3). In previous studies, negative MR in the AF \( \text{La}_2\text{CuO}_{4+\delta} \) for the case of both the current and field parallel to \( \text{CuO}_2 \) was found and discussed to a certain degree \( [14, 15] \). The nature of the negative MR in rather low field \( H \parallel c \) and \( J \parallel a, b \) 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, the measurements in pulse magnetic field with magnitude up to 15 T were done. 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 pulse field at \( T = 4.2 \, K \) and \( T = 77 \, K \) are shown in Figs. 6 and 7.

The pulse MR measurements have enabled 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 Fig. 4 and Fig. 6). It is also seen that resistive transition for out-of-plane current direction is sharper, and the relative changes in resistance, \( \Delta R/R_n \), are generally larger than those for in-plane direction. MR curves in pulse magnetic field at \( T = 77 \, K \) are less hysteretic than those at \( T = 4.2 \, K \), as expected (Fig. 7). 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 \( \text{La}_2\text{CuO}_{4+\delta} \) clearly manifests itself in the in-plane hopping conductivity. This supports the 3D nature of hopping conduction in this compound.

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Figure captions

Figure 1. The temperature dependences of resistivity $\rho_a$ of single-crystal La$_2$CuO$_{4+\delta}$ 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 $\lg \rho_a$ vs $T^{1/4}$.

Figure 2. The temperature dependence of resistivity $\rho_a$ of single-crystal La$_2$CuO$_{4+\delta}$ measured for transport current equal to 1 $\mu$A. The current was directed parallel to the crystallographic axis $a$. The inset shows temperature behavior of the ratio of the resistivities $\rho_a$ and $\rho_b$ for measuring currents directed along the crystallographic axes $b$ and $a$.

Figure 3. Magnetoresistance curves at $T = 5$ K and $T = 20$ K measured for single-crystal La$_2$CuO$_{4+\delta}$ in the out-of-plane DC magnetic field ($H \parallel c$) for different amplitudes of measuring current directed along the crystallographic axis $a$.

Figure 4. Magnetoresistance curves at various fixed temperatures measured for single-crystal La$_2$CuO$_{4+\delta}$ in the out-of-plane DC magnetic field ($H \parallel c$) for measuring current (100 $\mu$A) directed along the crystallographic axis $a$.

Figure 5. Magnetoresistance curves at various fixed temperatures measured for single-crystal La$_2$CuO$_{4+\delta}$ in the out-of-plane DC magnetic field ($H \parallel c$) for measuring current (100 $\mu$A) directed along the crystallographic axis $b$.

Figure 6. Magnetoresistance curves registered for single-crystal La$_2$CuO$_{4+\delta}$ in the out-of-plane pulse magnetic field ($H \parallel c$) at $T = 4.2$ K for the in-plane and out-of-plane current directions ($J \parallel A$ and $J \parallel c$) with current magnitudes 6 $\mu$A and 7.4 $\mu$A, respectively.

Figure 7. Magnetoresistance curves registered for single-crystal La$_2$CuO$_{4+\delta}$ in the out-of-plane pulse magnetic field ($H \parallel c$) at $T = 77$ K for the in-plane and out-of-plane current directions ($J \parallel A$ and $J \parallel c$) with current magnitudes 5.93 mA and 178 $\mu$A, respectively.
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