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Extensive characterization of anisotropic conductors in the Montgomery geometry

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Résumé. — Dans le cas de matériaux très anisotropes, les propriétés de transport d'un monocristal peuvent être équivalentes à celle d'une longue barre de section quasi carrée. Dans cette géométrie, nous avons analysé en détail les erreurs introduites par l'emploi de la technique de Montgomery [1]. Nous proposons une extension de cette méthode. Dans ces conditions, nous montrons qu'une analyse complète peut aisément permettre de choisir une géométrie de test conduisant à la détermination de l'anisotropie réelle du matériau et de sa variation thermique. La méthode est utilisée pour montrer que, dans le composé $\text{La}_2\text{CuO}_{4+0.018}$, la conductivité est activée dans les plans Cu-O alors que, perpendiculairement aux plans, nous avons montré précédemment qu'elle résulte d'un processus de sauts.

Abstract. — In very anisotropic materials the transport properties of a single crystal may be equivalent to those of a long thin bar. In this geometry we show that an extension of the Montgomery method [1] allows a detailed discussion of the experimental results. It is then possible to establish the correct value of the anisotropy of the resistivity tensor. The method is used to show that in $\text{La}_2\text{CuO}_{4+0.018}$, the conductivity is activated in the Cu-O planes while it results from variable range hopping process perpendicular to the planes as previously established.

1. Introduction.

The knowledge of the components of the resistivity tensor is an important step to discuss the electronic properties of a material. This three by three second-rank tensor is symmetrical and diagonal in the lattice axis system, in zero magnetic field, for lattice types different from triclinic and monoclinic. The number of unknown elements depends on the crystal symmetry (one, two and three for the cubic, the tetragonal/hexagonal and the orthorhombic symmetries respectively) [2].

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The resistivity of some materials is very sensitive to minor variations of their composition. Therefore the anisotropy of the resistivity tensor must be determined by connecting up the sample successively in geometries allowing, at each time, the determination of one component of the resistivity tensor. However, this conventional technique may fail if any variation is induced during the wiring process. Thus, the best way is the use of Montgomery's method [1] which allows to determine the temperature variation of two tensor components in one wiring process.

The original Montgomery's method is difficult to apply to small, very anisotropic crystals. Thus, in a first part, we are going to develop an extension of this method, and, in discussing the various uncertainties, to show how it is possible to test the validity of the results. In a second part, we will apply our analysis to measurements performed on a La_2CuO_4 crystal.

2. Measurement technique.

Montgomery's method applies to materials with two distinct components of the resistivity tensor (ρ_{ab} in the ab plane and ρ_c in the c direction, perpendicularly to the plane). A single crystal is cut in the form of a prism, a face has edges in the directions of the two resistivity components to be measured. Small electrodes are attached at the four corners of this face. The ratio of the voltage measured between two adjacent electrodes to the current flowing between the other two electrodes is determined, then a similar measurement is made with all connections rotated by 90° . Let R_{ab} and R_c be these ratios measured as described in figure 1. As the current lines are distributed all over the sample, each of these two ratios depends on the two components of the resistivity tensor (ρ_{ab} and ρ_c). In order to calculate these two components, Montgomery uses: i) Wasscher's equations [3] which map the anisotropic sample with dimensions A' , B' , C' along a , b , c axes respectively on its isotropic equivalent of resistivity $\rho = (\rho_{ab}^2 \rho_c)^{1/3}$ and with dimensions $A = A' \times (\rho_{ab}/\rho)^{1/2}$, $B = B' \times (\rho_{ab}/\rho)^{1/2}$, $C = C' \times (\rho_c/\rho)^{1/2}$, ii) the Logan *et al.* [4] calculation of the voltage-current ratios on the faces of a rectangular isotropic block.

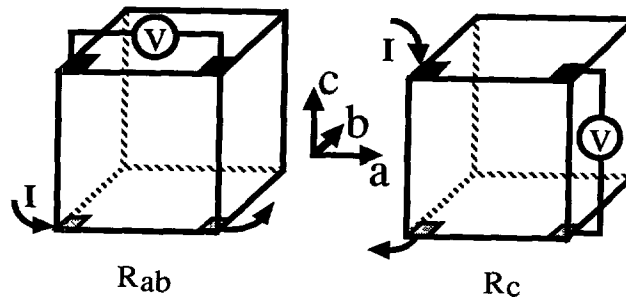


Fig. 1. — Classic Montgomery's configuration for the measurement of R_{ab} and R_c .

Montgomery's method requires only four electrical connections. However, for small crystals of very anisotropic materials, the measured value R_{ab} is very small. That's why it was proposed to use six connections in order to measure R_{ab} on a (a, b) face and R_c on a (a, c) face [5]. Then, as described in figure 2, there are for a given R_c measurement two possible R_{ab} measurements: $R_{ab\parallel}$ and $R_{ab\perp}$. A very interesting limiting case is obtained when the equivalent isotropic sample looks like a long thin bar. Then the sample is very thin when measurements are made in the bar axis and very thick normally to this axis. The two components of the resistivity tensor are easily obtained by the application of the Logan *et al.*

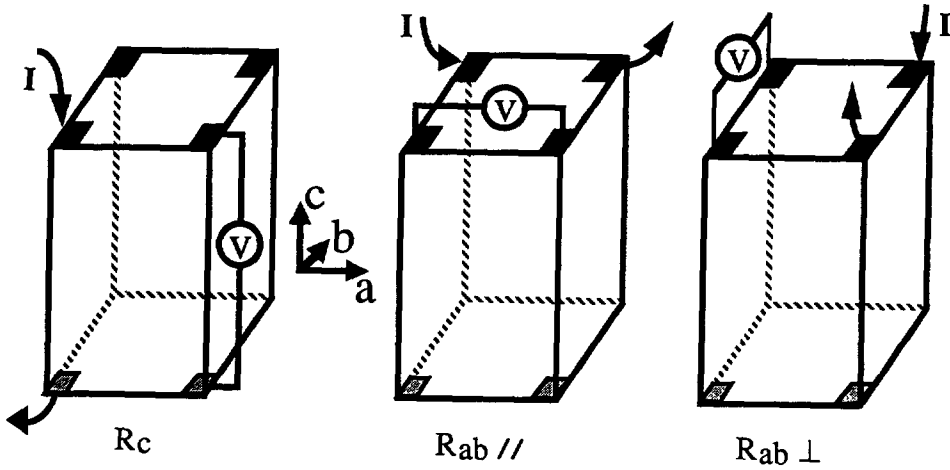


Fig. 2. — The two configurations (\parallel and \perp) for the measurement of R_{ab} at a given R_c .

formulas :

$$\rho_{ab} = \rho_{ab,0}(1 + \Delta)^{-1} \tag{1}$$

$$\rho_c = \rho_{c,0}(1 + \Delta) \tag{2}$$

where

$$\rho_{ab,0} = \frac{X^2 C'}{A' B'} R_{ab} \frac{R_{ab}}{R_c} \left[\frac{\pi 1}{4 Q} \right]^2$$

$$\rho_{c,0} = \frac{A' B'}{C'} R_c$$

and

$$\Delta = \frac{R_{ab} X}{R_c B'} \frac{\ln(2) - 4 K_0 \left(\frac{\pi A'}{B'} \right)}{Q}$$

K_0 is the modified Bessel function and Q is series of K_0 tabulated in [4] as « aM_∞ ».

When $R_{ab} = R_{ab \parallel}$, then $X = A'$ and $Q = Q(B'/A')$. When $R_{ab} = R_{ab \perp}$, then $X = B'$ and $Q = Q(A'/B')$.

An even easier formulation is obtained in the limit $[0.67 < (A'/B') < 1.5]$. Then,

$$K_0(\pi A'/B') \approx 1.09 \times \exp(-3.59 \times A'/B') \tag{3}$$

$$Q(A'/B') \approx 5.5 \times (B'/A')^{1/2} \times \exp(-\pi A'/B'). \tag{4}$$

The relative errors induced by (3) and (4) are less than $\pm 2\%$ and $\pm 0.6\%$ respectively.

One must notice that the formulas (1) and (2) restrict Logan *et al.*'s series to the first order corrections (Δ) with regard to the ideal situations : sample infinitely thin, sample infinitely thick. The errors induced by this approximation will be discussed later. The second order corrections depend on the anisotropy (ρ_c/ρ_{ab}). Their use would require a self consistent calculation which is out of our present project.

The setting-up of the Logan *et al.* series implies : first, that the electrical connections are small and localized at the prism vertices, and second, that the single crystal is cut along the

directions of the resistivity tensor. Two types of uncertainties may appear during the experimental process.

The first type of uncertainties arises even in a perfect single crystal cut with its edges parallel to the axes of the resistivity tensor. They would have the same importance in both isotropic or anisotropic materials. They can be referred as quasi-isotropic uncertainties. This includes uncertainties due to i) the misplacement of the contacts, ii) the use of the limiting case formulas, iii) the size measurements. As shown in [6], it is easy to fit the contacts so as to reduce the effect of the first i) to a relative uncertainty on ρ less than $\pm 2\%$. The use of the formulas suited for long thin bar induces relative errors that are less than $\pm 3\%$ as long as the large to small dimension ratio is higher than 1.6 in the equivalent isotropic solid. On the whole, the effect of the two first is lower than $\pm 5\%$. The importance of the uncertainties on length measurement increases when sample dimensions decrease, with small samples the relative uncertainties may reach $\pm 10\%$. At first, we limit the analysis to the zero order terms (the $\rho_{c,0}$ and $\rho_{c,0}$ terms), the length measurements uncertainties have an effect on : $(A'B'/C')$ and $[(X^2 C')/(A'B')] \times Q^{-2}$ respectively for the $\rho_{c,0}$ and the $\rho_{ab,0}$ determination. As long as $A'/B' \geq 0.6$, we have :

$$\text{if } R_{ab} = R_{ab\parallel} : [(X^2 C')/(A'B')] \times Q^{-2} \alpha C' \times \exp(2 \pi B'/A')$$

$$\text{if } R_{ab} = R_{ab\perp} : [(X^2 C')/(A'B')] \times Q^{-2} \alpha C' \times \exp(2 \pi A'/B').$$

This exponential variation leads to a very high sensitivity to the length measurements uncertainties. Let us suppose $A' = B'$ and a $\pm 10\%$ uncertainty on each length measurement. Then the exact value of a component of the resistivity tensor is equal to the calculated value times (1 ± 0.3) and $[(1 \pm 0.1) \times \exp(\pm 0.4 \pi) \approx 3.9^{\pm 1}]$ for $\rho_{c,0}$ and $\rho_{ab,0}$ respectively. Within the previous condition on A'/B' , the complementary term Δ varies exponentially with $(A'/B')^{\pm 1}$. As a result, the uncertainties on length measurements may have on $(1 + \Delta)$ a strong effect, this effect may be of the same order of magnitude as the uncertainties on $\rho_{c,0}$. If $A' \approx B'$ in $[\rho_{(ab,0\parallel)} \times \rho_{(ab,0\perp)}]$ the product of the Q terms reduces the uncertainty induced by length measurements δA because the first order terms in $\delta A/A$ cancel each other out. Then : $\rho_{(ab,0)} \approx [\rho_{(ab,0\parallel)} \times \rho_{(ab,0\perp)}]^{1/2}$ within uncertainties of the same order of magnitude as uncertainties on $\rho_{c,0}$. In this expression $\rho_{(ab,0\parallel)}$ and $\rho_{(ab,0\perp)}$ are connected to R_{ab} measurements performed on the same face. All these uncertainties distort the quantitative determination of the resistivity tensor components but not their temperature variations because they develop on the equivalent isotropic solid.

A second type of error appears when the sample is not cut exactly along the resistivity tensor axes or has defects, i.e. when the link between the macroscopic conduction in the sample and the symmetry of the conductivity tensor is broken. In that case, the voltage-current ratios (R_{ab} and R_c) do not contain ρ_c and ρ_{ab} in the way leading to the formulations (1) and (2). They can be referred as anisotropic uncertainties. This type of error is of importance when measurements are performed in the high conductivity direction. With regard to macroscopic defects, the result is obvious, a diversion of the current flow exploring the low conductivity direction increases strongly the measured voltage/current ratio. The effect of a badly cut sample can be understood on the equivalent isotropic solid. Let us take as an example a quasi cubic crystal with an anisotropy of 400, where the face on which R_{ab} is measured is not exactly parallel to ab planes. Figures 3 shows the single crystal and the equivalent solid. As it clearly appears the A/B ratio is strongly modified. In the pictured configuration, the R_{ab} ratio will be increased strongly as compared to a perfectly cut sample. With all connections rotated by 90° , R_{ab} will be reduced. These variations will be strongly dependent on the anisotropy. A similar analysis shows that the same kind of error does not affect as much the R_c ratio. Therefore, in

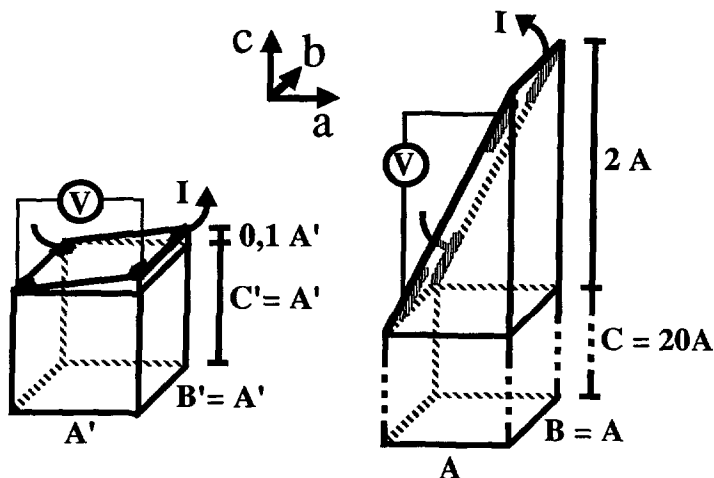


Fig. 3. — A single crystal with a badly cut (*a*, *b*) face and the equivalent solid.

this paragraph, we can restrict the discussion to the effect of a R_{ab} variation on the calculated resistivities. To calculate ρ_c , the values of R_{ab} and Q are used in the correcting term, as a result, the effect of an incorrect determination should be small. To calculate ρ_{ab} , the value of R_{ab} and Q are used not only in the correcting term, but in the zero order term too, the effect of an improper determination should be very strong. At a given temperature, the effect may be of the same order of magnitude as the uncertainties on length measurements, but the anisotropic effect should be temperature dependent, in the same way as the anisotropy is. As a result, the deduced temperature variation of the component of the resistivity tensor of lowest value is qualitatively wrong.

A way to determine whether or not the set-up is correct will be given in the present paragraph. It is possible to carry out four R_{ab} measurements (two on each face parallel to the a and b axes) and four R_c measurements (one on each face parallel to the c axis) on the crystal by using eight contacts (one at each vertex). Combining each R_{ab} measurements with each R_c measurements the ρ_c and ρ_{ab} values are obtained from sixteen different ways. These sixteen values should be within the discussed uncertainties but, if the uncertainties due to the size measurements are very important, it should be difficult to determine whether or not these values are true determinations of the components of the resistivity tensor. Nevertheless, this can be done by studying the ratios of the calculated values : $V_c = [\rho_c(T_1)/\rho_c(T_2)]$ and $V_{ab} = [\rho_{ab}(T_1)/\rho_{ab}(T_2)]$. Within a ratio, R_{ab} and R_c have been measured in the same way but at two different temperatures (i.e. for two different anisotropies) [7]. If these ratios do not suffer from anisotropic errors then :

$$V_c = [R_c(T_1)/R_c(T_2)] \times \{ [1 + \Delta(T_1)]/[1 + \Delta(T_2)] \} \tag{5}$$

$$V_{ab} = [R_{ab}^2(T_1)/R_{ab}^2(T_2)] \times [R_c(T_2)/R_c(T_1)] \times \{ [1 + \Delta(T_2)]/[1 + \Delta(T_1)] \} . \tag{6}$$

In these formulas the quasi isotropic errors affect the Δ terms only. Therefore the values of V_c and V_{ab} must be examined in the following way. At first, the scattering of the V_c and V_{ab} values must be of the same order of magnitude. If not, the sample suffers from anisotropic defects, formulas (1) and (2) cannot be used, and thus it is not possible to determine the resistivity tensor components. Second, formula (5) allows to test the effect of Δ in looking at the scattering of the values of V_c obtained from a given R_c and the various

R_{ab} . Third, the scattering should be the same for the whole set of values of V_c ; if not this means that uncertainties on R_c are not negligible (in case of a strong temperature dependence of R_c , this may be due to a bad temperature regulation).

In any case, the scattering is due to the temperature variation of the anisotropy. By the way, as soon as the anisotropy is temperature dependent, the comparison of the ratios allows to check the quality of the achieved deconvolution, and to choose, if any, a correct set of measurements.

3. Application to a real crystal : $\text{La}_2\text{CuO}_{4+y}$

This material shows a metallic behaviour and a superconducting phase ($T_c \leq 40$ K) when $y > 0.03$ [8]. When $y < 0.03$, this material is a very anisotropic semi-conductor. As the anisotropy of the electronic properties plays a major role in the discussion of the high temperature superconducting state it is of importance to determine the intrinsic anisotropy of the electrical resistivity. To obtain such a result a detailed discussion of the experimental results is needed. We are going to illustrate our method in the following.

The results discussed below come from a single crystal cut in the form of a rectangular prism of ($A' \times B' \times C'$) along ($a \times b \times c$): ($A' \times B' \times C'$) = $(260 \times 240 \times 40) \mu\text{m}^3$. This small crystal is a part of a slab of $(2 \times 4 \times 1) \text{mm}^3$ thinned by polishing down to $40 \mu\text{m}$. The uncertainty on length measurements is $\pm 10 \mu\text{m}$. Thus, the relative uncertainty on A' , B' and C' are respectively $\pm 3.8 \%$, $\pm 4.2 \%$ and $\pm 25 \%$. As $A'/B' = 1.1$, we may use formula (4) to calculate Q . As a result of the uncertainties on length measurements the value of the exponential term in Q is uncertain within a factor $1.3^{\pm 1}$ (in Sect. 2 such a formulation is explained). Such an anisotropic crystal maps to an equivalent isotropic solid looking like a long thin bar as long as the anisotropy (ρ_c/ρ_{ab}) is higher than 100. Magnetoresistance measurements [9] show three-dimensional magnetic order sets in at $T_N = 200$ K; this is consistent with an oxygen doping $y = 0.018$ [10]. The eight electrodes are $17 \mu\text{m}$ diameter silver wire at first attached with silver epoxy glue, then cured at 400°C [11]. The corresponding silver dots on the sample are smaller than $40 \mu\text{m}$ in diameter. Such large dots as compared to sample size cause a relative uncertainty on the determination of ρ less $\pm 2 \%$ [12]. The contact resistances are few ohms.

An ac four probes conventional technique is used to make each R measurements. Instrumentation introduces relative uncertainties which are small with regard to uncertainties discussed in this paper and we can over-estimate these ones at $\pm 1 \%$. The temperature sensor is a platinum resistance which allows a measure with an error lower than ± 0.1 K. During the 8 measurements of R the temperature was supposed to be stabilised at ± 0.5 K.

The ρ_c and ρ_{ab} obtained in the sixteen different ways are displayed in table I. Parameter i refers to values of R_c measured on successive edges. Parameter j refers to values of R_{ab} . When $j = 1$ or 2 they were measured on a given (a, b) face. When $j = 3$ or 4 the same measurements were done on the other (a, b) face. The relative uncertainties expected on ρ_c , from quasi-isotropic uncertainties, are $[(\pm 33 \%) + (\pm 9 \%) + (\pm 5 \%)]$, respectively for the zero order term, the complementary term (written as Δ in Sect. 2) and the approximation used. At 295 K, $[177 < \rho_c < 219] \text{m}\Omega \text{m}$; this $\pm 11 \%$ scattering is within the quasi-isotropic uncertainties. The uncertainties on ρ_{ab} are the previous $\pm 47 \%$ added to the factor $1.7^{\pm 1}$ due to the exponential factor: which means that $\rho_{ab} \times 0.3 < [\text{right value}] < \rho_{ab} \times 2.5$. At 295 K, $[0.247 < \rho_{ab} < 0.325] \text{m}\Omega \text{m}$. The factor 3.8 between the lowest and the highest value is within the quasi-isotopic uncertainties. Thus, the values calculated at 295 K do not allow to determine whether or not the sample has macroscopic defects or is badly cut. As $A' \approx B'$, we can reduce the ρ_{ab} scattering by combining values calculated for a given i on the same (ab) face: $\rho_{(ab)} = [\rho_{(ab\parallel)} \times \rho_{(ab\perp)}]^{1/2}$; we find $[0.247 < \rho_{ab} < 0.325] \text{m}\Omega \text{m}$.

Table I. — Values in $m\Omega\ m$ of the resistivity in the CuO_2 planes (ρ_{ab}) at 295 K, values in $m\Omega\ m$ of the resistivity perpendicular to the planes (ρ_c) at 295 K, and the ratios : $V_c = [\rho_c(295\ K)/\rho_c(65\ K)]$ and $V_{ab} = [\rho_{ab}(295\ K)/\rho_{ab}(65\ K)]$ of their values at two different temperatures (295 K and 65 K).

(i, j)	$\rho_{ab}(295\ K)$	$\rho_c(295\ K)$	V_{ab}	V_c
(1, 1)	0.391	216	0.135	0.175
(2, 1)	0.403	210	0.124	0.191
(3, 1)	0.421	201	0.120	0.196
(4, 1)	0.420	202	0.129	0.183
(1, 2)	0.229	206	0.707	0.195
(2, 2)	0.241	196	0.605	0.227
(3, 2)	0.248	191	0.607	0.227
(4, 2)	0.252	188	0.627	0.219
(1, 3)	0.131	199	0.334	0.186
(2, 3)	0.140	186	0.292	0.213
(3, 3)	0.143	183	0.289	0.215
(4, 3)	0.147	177	0.305	0.204
(1, 4)	0.465	219	0.256	0.187
(2, 4)	0.480	210	0.227	0.210
(3, 4)	0.498	205	0.224	0.213
(4, 4)	0.493	207	0.236	0.203

We can use the median values of ρ_{ab} and ρ_c to calculate the anisotropy at ambient temperature : 700, the uncertainty is smaller than 50 %. The influence of the complementary term may be tested by comparing the values of ρ_c obtained with a given R_c , but with the two R_{ab} measured on the same face of the sample (2 couples $j = 1$ and $j = 2$ or $j = 3$ and $j = 4$). From the calculated $\rho_{ab}(i, j)$ we know that the largest variation of the ratio $[R_{ab}/Q]^2$ (by a factor 3.5) is established when j is changed from 3 to 4. Thus the biggest difference is expected between $\rho_c(i, 3)$ and $\rho_c(i, 4)$. This is actually observed. The observed scattering of the calculated values is within the expected uncertainties.

In order to choose a correct set of measurement, we have done (as explained previously) the whole set of measurements at $T_1 = 295\ K$ and $T_2 = 65\ K$. This allows the determination of the ratios V_c and V_{ab} to test whether or not the measurements suffer only isotropic uncertainties. First, the results are much more scattered for the V_{ab} than for the V_c values : anisotropic uncertainties are of importance in this sample. Second, a factor more than 5 is observed between $V_{ab}(i, 1)$ and $V_{ab}(i, 2)$, so the (a, b) face of the prism corresponding to $j = 1$ or 2 is either near macroscopic defects or has not the right orientation. So we shall consider the other (a, b) face ($j = 3$ or 4). As previously, the effect of the complementary terms is tested by comparing the V_c values obtained from a given i and the two j . For $i = 1$ to 4, as expected, the relative variation ($\pm 1.5\ %$) is small. Nevertheless, the scattering of the whole set of the calculated V_c values is much higher, it is necessary to go more deeply into the analysis. In the set of rows corresponding to $j = 3$ the scattering is the same on V_c as on V_{ab} values, the sign of the variations is reversed. The same results are observed for $j = 4$. Certainly, the uncertainties on the values of R_c induce the scattering. As the uncertainty on the measurements at a given temperature is much lower than the $\pm 7.2\ %$ of these relative scatterings, either this comes from macroscopic defects, or the temperature was badly controlled... For a given i , the difference between $V_{ab}(i, 3)$ and $V_{ab}(i, 4)$ is lower than $\pm 13\ %$, this difference is induced by uncertainty on R_{ab}^2 which means that the uncertainty on R_{ab} is of the same order of magnitude as on R_c . Although we cannot claim that such an effect is

not due to macroscopic defects, it is easier to find such a result with a temperature drift of few K during the measurements at low temperature. Anyway, the scattering is fairly small : a study of both ρ_{ab} and ρ_c may be carried on thanks to R_{ab} measurements performed on this (a, b) face. If the scattering is due to a temperature drift a right quantitative determination is expected, on the opposite, at least a correct qualitative behaviour will be established.

We have measured R_{ab} and R_c corresponding to $i = 2$ and $j = 4$ in order to determine the in plane and out of plane conduction mechanism. Figures 4 and 5 respectively display $\log(\rho_{ab}/T)$ plotted against T^{-1} and $\log(\rho_c)$ plotted against $T^{-1/2}$. The inset shows the

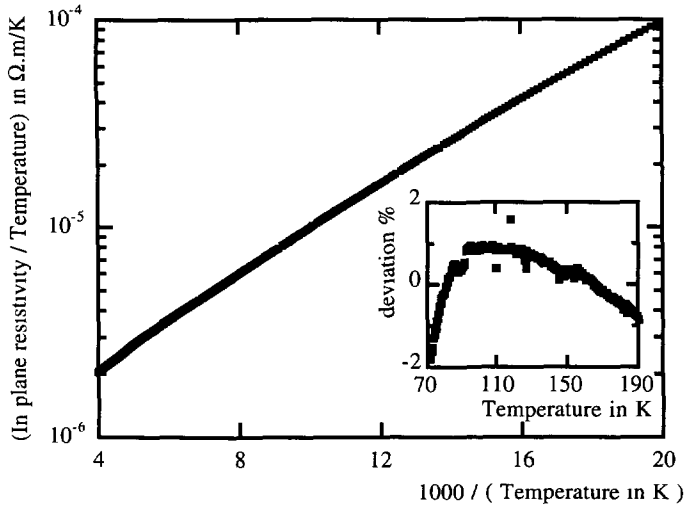


Fig. 4. — $\log(\rho_{ab}/T)$ as a function of T^{-1} . Inset : relative deviation ($d\rho/\rho$) of the experimental points from $\rho_{ab,t} = 8.13 \times 10^{-7} \times T \times \exp(249/T) \Omega m$ as a function of temperature.

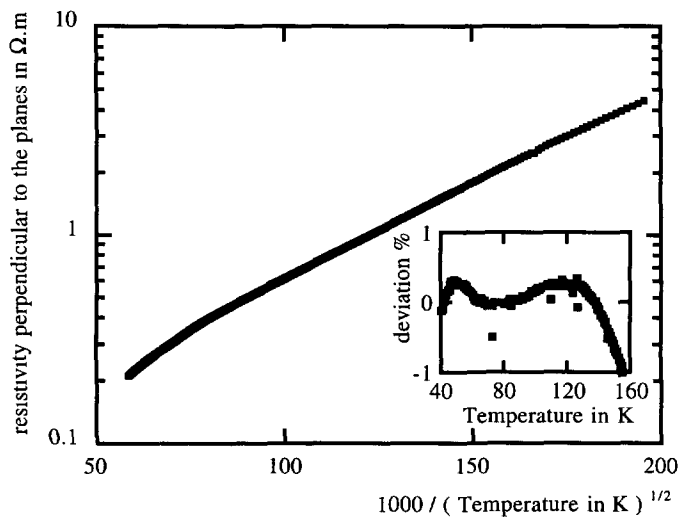


Fig. 5. — $\log(\rho_c)$ as a function of $T^{-1/2}$. Inset : relative deviation ($d\rho/\rho$) of the experimental points from $\rho_{c,t} = 7.33 \times 10^{-2} \times \exp[(455/T)^{-1/2}] \Omega m$ as a function of temperature.

relative difference between the experimental values and respectively :

$$\rho_{ab,t} = 8.13 \times 10^{-7} \times T \times \exp(249/T) \Omega\text{m}$$

and

$$\rho_{c,t} = 7.33 \times 10^{-2} \times \exp[(455/T)^{-1/2}] \Omega\text{m} .$$

These differences are less than $\pm 1\%$ respectively from 75 K to 194 K for $(\rho_{ab} - \rho_{ab,t})/\rho_{ab}$ and from 40 K to 155 K for $(\rho_c - \rho_{c,t})/\rho_c$. At 50 K while the anisotropy is reduced by a factor 2 [9] the relative difference between $\rho_{ab,t}$ and the experimental values ρ_{ab} is less than 5%. $\rho_{ab,t}$ is the temperature variation expected from a two-dimensional extrinsic semiconductor and it was previously established that $\rho_{c,t}$ comes from a one-dimensional variable range hopping process [12]. These laws fits the experimental data much better over a larger temperature range than others. Is the experimental technique overestimated when such a quantitative analysis is performed? Even then the main result remains : the conduction process is not the same within the CuO_2 planes as perpendicular to these planes [13]. We have shown previously that the conductivity is by a variable range hopping process perpendicular to the planes. Here we show that the conductivity is activated within the planes. This material is a set of weakly coupled semiconducting planes. The method developed in the present paper allows to understand why previously published results were misinterpreted [14].

4. Conclusion.

By using electrodes glued at each vertex of a prismatic single crystal it is possible to perform a complete set of resistivity measurements in the Montgomery geometry. If these measurements are performed at various temperatures an extensive discussion may allow to find a set of test points to determine the right anisotropy.

As a result we have been able to establish that between the Neel temperature and 75 K, the conductivity in $\text{La}_2\text{CuO}_{4+0.018}$ is activated in the Cu-O planes while we have previously shown that in the same sample it results from a variable range hopping perpendicular to the planes.

Acknowledgments.

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