Single-Crystal Study of the 55 K Superconductor $TlSr_2CaCu_2O_{7-\delta}$

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Single crystals of the TlSr₂CaCu₂O₇ superconductor have been grown for the first time. The structures of an as-grown crystal ($T_{\rm c} = 55$ K) and an oxygen pressure annealed one ($T_{\rm c}$ = 15 K) have been refined. The results are compared to those reported for the polycrystalline phase. The magnetic study (H||c) of a $T_c = 55$ K as grown crystal allows us to compare the superconducting properties of TISr₂CaCu₂O₇ with those of other thallium 1212 single crystals.

The thallium cuprates form a large family (see for review ref 1) whose superconducting properties, although very promising, are so far not completely understood. An important issue deals with the relationships between the pinning properties of these materials and the anisotropy of their structure. To answer this question, a systematic investigation of the single crystals of these cuprates is absolutely necessary. In fact, the majority of superconducting properties for single crystals have been obtained in the system Tl-Ba-Ca-Cu–O.^{2–11} More recently, superconductive parameters have also been established for single crystals of the strontium 1212 cuprates $Tl_{0.5}Pb_{0.5}Sr_2CaCu_2O_{7-\delta}^{12}$ and $(Tl,Bi)Sr_2CaCu_2O_{7-\delta}$.¹³ This is not the case for the "pure" thallium 1212 cuprates $TlSr_2CaCu_2O_{7-\delta}$, for which only polycrystalline data are available.¹⁴⁻¹⁶ To determine the part played by the normal layers-

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thickness, nature of the cations (Pb, Tl, Bi)-in the pinning properties of the 1212 thallium cuprate, an effort has been made to grow single crystals of the phase $TlSr_2CaCu_2O_{7-\delta}$. We report herein on the crystal growth, structural characterization, and superconducting properties of single crystals of these 1212 cuprates that exhibit a T_c of 55 K for the optimized phase and a $T_{\rm c}$ of 15 K for the overdoped cuprate.

Crystal Growth

The crystals were grown from mixtures of oxides and strontium carbonate of nominal compositions corresponding to the molar ration Tl:Sr:Ca:Cu = 1:2:1:2. Appropriate amounts of SrCO₃, CaO and CuO were first mixed and heated at 900 °C in air for 24 h to obtain the precursor. The adequate amount of Tl₂O₃ was then added to the mixture, and the resulting powder (2 g)was introduced into an alumina crucible and sealed in an evacuated silica tube (inner diameter 0.9 cm). The ampule was then installed vertically in a vertical tubular furnace and heated in 6 h at 1050 °C and slowly cooled to 900 °C at a rate of 1.7 °C/h, then quickly cooled to 400 °C at a rate of 12 °C/h, and finally cooled to room temperature in 10 h. Black shiny plateletlike crystals were then extracted of the preparation. Two kinds of crystals have been selected with different dimensions for crystal structure characterization $(0.1 \times 0.1 \times 0.02)$ mm³) and for magnetic and electrical study (typically 1 \times 1 \times 0.2 mm³).

Structural Characterization

Although the structure of this phase has been established previously by neutron diffraction,¹⁴ a structure determination has been performed here, from single crystals, to check the cationic and the positional parameters, which may vary with the method of synthesis because of possible nonstoichiometry on thallium and oxygen.

The X-ray diffraction study was performed with a CAD 4 Nonius diffractometer using Mo Ka radiation with a graphite monochromator, scanning mode $\omega - \theta$, on a 154.2 \times 77.1 \times 25.7 μ m³ single crystal. 2981 reflections were registered, i.e., 1395 reflections with I $> 3\sigma(I)$, corresponding to 246 independent reflections. After absorption corrections ($\mu_{Mo} = 447.5 \text{ cm}^{-1}$), the

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Table 1. TlSr₂CaCu₂O_{7 $\pm \delta$} Atomic Parameters (Space Group P4/mmm)^a

$\begin{array}{c} {\rm Tl:} x0 \; 0 \\ {\rm Ca:} {}^{1\!/_{2}}, {}^{1\!/_{2}}, {}^{1\!/_{2}} \\ {\rm Sr:} {}^{1\!/_{2}}, {}^{1\!/_{2}}, z \end{array}$		$\begin{array}{c} O(1): \ 0, \ \frac{1}{2}, \ z\\ O(2): \ 0, \ 0, \ z\\ O(3): \ \frac{1}{2}, \ \frac{1}{2}, \ 0\end{array}$								
· · ·		$T_{\rm c} = 55 \ {\rm K}$	$T_{\rm c} = 15 \ {\rm K}$							
$\begin{array}{c} a (\mathring{A}) \\ c (\mathring{A}) \\ c/a \\ V (\mathring{A}^3) \end{array}$		3.7966(3) 12.092(1) 3.1850 174.29	3.7808(5) 12.0855(12) 3.1965 172.75							
Ϋ́Ι	x B	0.0793(7) 2.08(6)	0.0701(5) 1 34(4)							
Ca/Tl	$\stackrel{D_{ ext{eq}}}{B_{ ext{eq}}}_{ au}$	0.56(2) 81/19	0.46(2)							
Sr	z Bea	0.2165(1) 0.74(1)	0.2130(1) 0.58(1)							
Cu	$z = \frac{z}{B_{eq}}$	0.3642(2) 0.39(1)	0.3622(2) 0.34(1)							
O(1)	$z B_{eq}$	$0.3714(6) \\ 0.8(1)$	0.3709(6) 0.77(9)							
O(2)	$z B_{eq}$	0.166(1) 1.1(2)	0.1667(8) 0.6(1)							
O(3) R R _w	$B_{\rm eq}^{\rm eq}$	$1.9(4) \\ 0.035 \\ 0.040$	1.8(3) 0.032 0.038							

^{*a*} τ : occupancy factor (0.81Ca + 0.19Tl).

reliability factor was lowered to R = 0.035 and $R_w = 0.040$ for the as-synthesized single crystal ($T_c = 55$ K) and to R = 0.032 and $R_w = 0.038$ for the same crystal annealed under an oxygen pressure of 100 bar ($T_c = 15$ K).

The atomic parameters of this single crystal, very similar to those obtained from powder neutron diffraction data for the "same" $TlSr_2CaCu_2O_7$ obtained either as nonsuperconducting or as 58 K superconductor,¹⁴ are given in Table 1.

The main difference between the two studies deals with the occupancy of calcium sites by thallium which is of 12% in the powder work¹⁴ and of 19% in our single crystal. This can easily be explained by the different method of synthesis, since our crystal growth is performed in silica ampoule and consequently avoids thallium losses. In any case both studies converge to the existence of a thallium over stoichiometry according to the formula $Tl_{1+x}Sr_2Ca_{1-x}Cu_2O_{7-\delta}$, with $x \approx 0.12-0.19$. It is remarkable that the positional parameters are not significantly affected by the variation of the thallium content.

The disordered character of the $[TIO]_{\infty}$ layer observed by neutron diffraction¹⁴ is confirmed on the single crystal. One observes no splitting of O(3) and a very similar splitting of the Tl sites (x00). In both studies, the splitting seems to decrease slightly as $T_{\rm c}$ decreases.

It is not possible to detect any oxygen deficiency on the O(3) site from our single-crystal X-ray diffraction study. But taking into consideration the results obtained by neutron diffraction, we can admit that this site is partly occupied and the occupancy factor varies only weakly with the critical temperature, since Izumi et al.¹⁴ evidenced 14% oxygen vacancies on O(3) for the nonsuperconducting phase and 22% oxygen vacancies for the 58 K superconductor.

The interatomic distances (Table 2), which vary in the same way in both studies, attest to the reproducibility of the effect of oxygen nonstoichiometry in polycrystalline as well as in single crystals. Clearly the Ca-O distances do not vary significantly with the oxygen



Figure 1. $\chi(T)$ curves of a $T_c = 55$ K as-synthesized crystal (a) and of an oxygen pressure annealed one ($T_c = 15$ K) (b).

Table 2. TlSr₂CaCu₂O_{7 $\pm\delta$} Interatomic Distances (Å)

	$T_{\rm c} = 55 \ {\rm K}$	$T_{\rm c} = 15 \ {\rm K}$
$T1-O(2) \times 2$	2.034(9)	2.032(8)
$-O(3) \times 2$	2.481(2)	2.493(2)
$-O(3) \times 2$	2.905(2)	2.867(2)
$Ca/Tl-O(1) \times 8$	2.454(4)	2.451(4)
$Sr-O(1) \times 4$	2.667(5)	2.686(4)
$-O(2) \times 4$	2.752(2)	2.731(2)
$-O(3) \times 1$	2.618(1)	2.574(1)
$Cu-O(1) \times 4$	1.9003(3)	1.8931(3)
$-O(2) \times 1$	2.392(9)	2.362(8)
Cu-Cu	3.285(3)	3.332(3)

stoichiometry, i.e, remain constant whatever the T_c . In the same way, the mean Tl-O distances that form the TlO₄ tetrahedra are not affected by the annealing. This is not the case for the CuO₅ pyramids which show a significant decrease as the T_c decreases, especially for the Cu-O apical bond, in agreement with the increase of oxygen content found by neutron diffraction. Note also that the Cu-Cu distance between two [CuO₂]_∞ basal planes increases as T_c decreases. The average Sr-O distance remains unchanged, even if the Sr-O distances vary, particularly the Sr-O(3) distances.

Superconducting Properties

Critical Temperatures. The T_c 's of the as-prepared crystals (55 K) and oxygen-pressure annealed ones (15 K) are in the range of T_c 's observed by Kubo et al. for ceramics of $TlSr_2CaCu_2O_{7-\delta}$ which can be either nonsuperconducting on the overdoped side or superconducting with an optimized T_c of 68 K.¹⁶ The $\chi(\hat{T})$ curves of our two types of crystal is reported in Figure 1; after a zero field cooling, a magnetic field of 10 G was applied and the measurements were then performed when Twas increased. To compare the doping levels of our crystals to those of Kubo's study, $\rho(T)$ measurements were performed on two large crystals of dimensions ${\sim}1$ \times 1 \times 0.2 mm³ with T_c's of 55 and 15 K. Four contacts were painted using silver paste on the crystal surfaces and 50 μ m diameter gold wires were used as electrical leads. With respect to the high doping level of $TlSr_2$ - $CaCu_2O_{7-\delta}$ superconductors, the T dependence of the normalized resistance could be fitted by using a power low-temperature dependence $\rho = \rho_0 + AT^n$. The logarithms of the corresponding data are reported in Figure



Figure 2. Normal parts of the $\log[(\varrho - \varrho_0)/\varrho_{300K}]$ vs log *T* plots for the 55 K (a) and 15 K (b) single crystals. The straight lines correspond to n = 1 and n = 2 in the relation $\varrho = \varrho_0 + BT^n$.

2. The exponent *n* value is close to 1.1 for our as-grown crystal and 1.5 for the oxygen pressure annealed one; these values are quite similar to those reported for ceramics where the exponent *n* was found to vary between n = 1.3 for the 68 K superconductor and n = 1.7 for the $T_c < 2$ K as-prepared ceramic. The increase of *n* when the doping level increases has also been evidence for the Tl₂Ba₂CuO_{6+ δ} overdoped superconductor.¹⁷ Finally, it appears that our 55 K crystal exhibits a $n \cong 1$ value, which seems to indicate that its doping level is close to be optimal and that consequently the 15 K superconductor with $n \cong 1.5$ is overdoped due to an oxygen uptake during the oxygen pressure annealing.

Magnetic Study. To compare the superconductivity of TlSr₂CaCu₂O₇ to that of Tl_{0.5}Pb_{0.5}Sr₂CaCu₂O₇ and Tl_{2/3}Bi_{1/3}Sr₂CaCu₂O₇,^{12,13} a similar study has been performed to extract its H|c properties.

The magnetization was recorded with a SQUID magnetometer with magnetic fields ranging from 0 to 5 T. One crystal with $T_c = 55$ K was chosen for these measurements with dimensions $0.770 \times 0.750 \times 0.205$ mm³. A Weissenberg photography of this crystal has revealed a misorientation along its c axis of less than 1°. The crystal was directly glued to the sample holder in order to align the magnetic field (H) direction parallel to its c axis.

The $\chi_i(T)$ curve of this crystal registered at low field, $\mu_0 H = 1$ mT, after zero-field cooling is reported in Figure 3. The internal susceptibility (χ_i) has been calculated from the relation $\chi_i = \chi_{ext}/(1 - \eta\chi_{ext})$, where χ_{ext} is the apparent external susceptibility and η the demagnetization factor. From the Osborn tables,¹⁸ an $\eta = 0.69$ value was deduced for the crystal. A $\chi_i(5 \text{ K}) = -1$ value is obtained which confirms the perfect shielding of the crystal. The T_c (onset) and T_c (midpoint) are found to be respectively 60 and 55 K. These T_c 's have been found to be reproducible for several crystals of quite similar dimensions.

In the following, all the magnetization data and superconducting properties correspond to the same crystal of dimensions $0.770 \times 0.750 \times 0.205 \text{ mm}^3$. The



Figure 3. *T* dependence of the internal susceptibility χ_i for the 0.770 × 0.750 × 0.205 mm³ single crystal registered in the geometry H||c|(H = 1 mT) after a zero-field cooling.



Figure 4. Half magnetization loop registered at 35 K. H_{sp} indicates the magnetic field value of the second peak.

M(H) magnetization half-loops have been recorded in the range 5 K $\leq T \leq$ 47.5 K. A typical plot is reported in Figure 4, it exhibits a "fishtail" shape as it has also been evidenced for other 1212 crystals of $Tl_{0.5}Pb_{0.5}Sr_2$ - $CaCu_2O_7$ and $Tl_{2/3}Bi_{1/3}Sr_2CaCu_2O_7$.^{12,13} In a systematic way, the fishtail effect of different Tl-based single crystals has been studied by Hardy et al.;¹⁹ the location of the second peak (noted $H_{\rm sp}$ on Figure 4) in the magnetization loops vs T/T_c has been found to be essentially dependent on the nature of the compound (structure and cationic composition). In Figure 5a, the $H_{\rm sp}(T/T_{\rm c})$ curves are reported for several 1212 thallium based crystals with the H_{sp} line of the title compound $TlSr_2CaCu_2O_7$. It can be observed that the H_{sp} lines of all three strontium 1212 cuprates are higher than that of the barium 1212 superconductor of formula TlBa₂- $CaCu_2O_7$. The values of electronic anisotropy (γ) were extracted on the basis of the work of Hardy et al.,¹⁹

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Figure 5. (a, top) $H_{sp}(T/T_c)$ curves for 1212 thallium based single crystals: TlSr₂CaCu₂O₇ (\bullet), Tl_{0.5}Pb_{0.5}Sr₂CaCuO₇ (\bullet), Tl₂₃Bi_{1/3}Sr₂CaCu₂O₇ (\blacksquare), and TlBa₂CaCu₂O₇ (\blacktriangle). (b, bottom) Irreversibility lines $H^*(t)$ of the corresponding 1212 crystals; the symbols are unchanged.

considering a 3D-2D transition or a 3D melting to explain the existence of H_{sp} . Thus, it appears that for TlSr₂CaCu₂O₇ the electronic anisotropy is very similar to that of Tl_{0.5}Pb_{0.5}SrCaCu₂O₇ and Tl_{2/3}Bi_{1/3}Sr₂CaCu₂O₇ and smaller than that of TlBa₂CaCu₂O₇. This point concerning γ is in agreement with the locations of the irreversibility lines H^* ($t = T/T_c$) of these 1212 crystals. The $H^*(t)$ line of TlSr₂CaCu₂O₇ has been determined from the closing points of the M(H) loops by using a $J_{\rm c}$ = 50 A cm⁻² criterion. Here again, one can observe (Figure 5b), that the $H^*(t)$ lines of strontium based 1212 superconductors are higher than that of the corresponding barium cuprate, TlBa₂CaCu₂O₇. In previous studies of Tl_{0.5}Pb_{0.5}Sr₂CaCu₂O₇ and Tl_{2/3}Bi_{1/3}Sr₂CaCu₂O₇ crystals,^{12,13} these higher $H^*(t)$ lines for strontium based 1212 cuprates were correlated to some structural features via the Kim et al. model:^{20,21} the $H^*(t)$ line is displaced toward high t and H when the thickness of the rock salt layer is decreased corresponding to a decrease of the *c*-axis resistivity (ρ_c) . The present result



Figure 6. Reversible magnetization, M vs H, at the temperatures indicated on the graph.



Figure 7. T dependence of B_{c2} with the fit (straight line); in the insert, the corresponding κ_c values are also reported.

for $TlSr_2CaCu_2O_7$ reinforces this hypothesis since all these three strontium based 1212 cuprates exhibit a smaller Cu-Cu distance via the rock salt type layers than that of $TlBa_2CaCu_2O_7$. This also rules out explanations based on Tl/Pb (or Tl/Bi) substitutions.

To extract basic parameters of superconductivity of our TlSr₂CaCu₂O₇ crystal of dimensions 0.770×0.750 \times 0.205 mm³ and $T_{\rm c}$ = 55 K, an analysis similar to that used for a $Tl_{0.5}Pb_{0.5}Sr_2CaCu_2O_7$ crystal,¹² has been performed. The reversible parts of the M(H) loops (Figure 6) have been fitted with the London equation (straight lines in Figure 6): $-\mu_0 M = (\phi_0/8\pi\lambda_{ab}^2) \ln[\beta B_{c2}/\beta M_{c2}]$ *B*], with $\beta = 1.16$, which is valid in the region $B_{c1} \ll B$ $\ll B_{c2}$. The $B_{c2}(T)$ values extracted from the fits are reported in Figure 7. From these values, $\kappa_c(T)$ is calculated by means of the equation $B_{c2}(T) = \kappa^2 {}_c \phi_0 /$ $(2\pi\lambda_{ab}^2(T))$ (insert of Figure 7). This average value, κ_c = 90, indicates the strong type II character of $TlSr_2$ -CaCu₂O₇, similarly to other superconducting cuprates. The $B_{c2}(T)$ values have been fitted with the WHH equation:²² $B_{c2}(0) = -0.69 T_c (dB_{c2}/dT)_{T_c}$. The slope obtained is $-dB_{c2}/dT = 1.5T \text{ K}^{-1}$ (straight line in Figure 7) which yields $B_{c2}(0) = 60$ T. The coherence length

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 $\xi_{ab}(0)$ is calculated by using the relation $\xi_{ab}(0) = \phi_0/2\pi B_{c2}(0)$; by using the $\kappa_c(T)$ values, $B_c(0)$ and $B_{c1}(0)$ are also calculated from the relations $B_c(0) = B_{c2}(0)/(\sqrt{2}\kappa_c)$ and $B_{c1} = B_c \ln(\kappa_c)/(\sqrt{2}\kappa_c)$. The other parameter of the M(H) fits is $\lambda_{ab}(T)$; in order to extrapolate the $\lambda_{ab}(0)$ value, the data were fitted with the two fluids model relation $\lambda_{ab}(T) = \lambda_{ab}(0)[1 - (T/T_c)^4]^{-1/2}$. All these superconducting parameters are summarized in Table 3.

The comparison of these parameters with those of $Tl_{0.5}Pb_{0.5}Sr_2CaCu_2O_7$ shows that the values derived for the title compound $TlSr_2CaCu_2O_7$ are rather similar. The $\lambda_{ab}(0)$ value is higher for $TlSr_2CaCu_2O_7$, 250 nm against 175 nm for $Tl_{0.5}Pb_{0.5}Sr_2CaCu_2O_7$ and is rather

Table 3. Basic Superconducting Parameters of the TlSr_2CaCu_2O_7 Single Crystal of Dimensions 0.770 \times 0.750 \times 0.205 mm^3

T _c midpoint (K)	$-\mathrm{d}B_{\mathrm{c}2}/\mathrm{d}T$ (T/K)	$B_{c2}(0)$ (T)	κc ^a	$\substack{\xi_{ab}(0)\\(nm)}$	$\lambda_{ab}(0)$ (nm)	<i>B</i> _c (0) (T)	<i>B</i> _{c1} (0) (mT)
55	1.5	60	90	2.34	250	0.47	16
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^a Average value.

similar to that of Tl₂Ba₂CaCu₂O₈ ($\lambda_{ab}(0) = 250 \text{ nm}$).¹⁹ The coherence length $\xi_{ab}(0) = 2.34 \text{ nm}$ is higher than that of Tl_{0.5}Pb_{0.5}Sr₂CaCu₂O₇, $\xi_{ab}(0) = 1.92 \text{ nm}$; this may be explained by the difference of T_c , 55 K for the former and 82 K for the latter, since roughly speaking, $T_c \alpha \Delta \alpha \xi^{-1}$, where Δ is the superconducting gap.

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