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Accelerated modes of obtaining Ti(Bi)-HTSC ceramic samples and its record characteristics in the Hubbard model

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Accelerated modes of synthesis of thallium-containing samples (tablets) of HTSC ceramics with record superconducting transition temperatures ($T_c = 124$ K or more) and with critical current densities (J_c) up to thousands of A/cm^2 were found. A technology of stable standard (two-stage) accelerated synthesis (less than a day instead of 48 hours) of thallium HTS ceramics has been developed. The synthesis process has been significantly reduced in time (compared to the known two-stage technologies). According to the fractures of the conduction curves $\sigma(T)$ and magnetic susceptibility $\chi(T)$, possible values of T_c up to 205 K are determined. In the Hubbard model, the dependences of $T_c(x)$, magnetic susceptibility $\chi(T)$ and conductivity $\sigma(T)$ on the concentration of x cations Me_x^{2+}, Me = Pb or Bi, and temperature T are determined. The results of the relevant calculations are consistent with the experimental data.

Keywords: Tl(Bi)-HTSC ceramics, record superconducting transition temperatures, accelerated synthesis, magnetic susceptibility, electrical conductivity.

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1. Introduction

The value $T_c$equal to 120 K in the Tl-Ba-Cu-O system was first reported in 1988 jcite1. This result was repeated in 1989 [2] and in 1990 [3,4]. The value $T_c = 124-125$ K was achieved by the authors in 1989, 1990 [5,6]. The issues of obtaining HTSC ceramics, which attracted interest at that time, faded into the insignificance due to attention to the properties of HTSC single-crystals, films and tapes. However, the issues of obtaining massive samples of Tl-HTSC ceramics, including in the form of tablets, again became relevant at the present time in connection with the prospects for their application. So, for example, massive samples of HTSC materials and power electronic elements based on silicon carbide are used in energy-loaded circuits of combined laser systems with a power of up to one megawatt [7]. In [5,6], based on the breaks in the dependences $\sigma(T)$, the possibility of increasing T_c to values in the ranges 145–150, 172–175, 185– 190, 203–205 K was shown, which is higher than the temperature of „dry ice“ (solid carbon dioxide CO_2, sublimation temperature at atmospheric pressure is 194.7 K).

This paper examines the results of search and optimization of the technological mode parameters for the accelerated synthesis of samples of thallium and bismuth HTSC ceramics with unprecedented characteristics. The essence of the found accelerated method is determined by the degree of grinding (50–150 μm) of the pre-annealed mixture, by annealing temperatures and times, and by the intensity of oxygen purging in the chamber. This refers to competitive bulk ceramics samples that make it possible to launch the production of HTSC products of commercial importance [7,8–10]. If 7–9 years ago the length of HTSCs produced using sandwich technology was limited to one meter (Tl, Pb-1223 wire from J_c to 10^4 A/cm^2 at 77 K [9,10]), then currently these are tapes (Tl, Bi-1223 HTSC wires of the second generation) with high current-carrying capacity, more than 1000 m long and with average linear critical current $I \sim 500$ A per 1 cm of width (Super Power and American Superconductor (AMSC) companies [7,10]). In particular, the technologies for manufacturing second-generation HTSC tapes are also being developed at the Faculty of Chemistry of Lomonosov Moscow State University with the participation of the Institute of Metal Physics of the Ural Branch of the Russian Academy of Sciences, Kurchatov Institute [7,9], as well as other Scientific and Research Institutes and enterprises [10].

The possibility of increasing the transport critical current in second-generation HTSC conductors today is mainly associated with an increase in the thickness of the superconducting layer without a significant decrease in the texture sharpness. In a three-layer HTSC coating 2 μm thick, the linear critical current was achieved till up 660 A/cm of width. Another way to increase the critical current is to increase the concentration of intentionally introduced pinning centers with a high potential [8]. Methods for deposition of epitaxial YBCO films and other initial HTSC materials onto prepared substrates play an important role.

The choice of substrate and method of deposition of HTSC ceramics films for second-generation cables is of key importance, since this determines the price and operation characteristics of the HTSC cable. Interest in Tl and Bi macro-HTSC samples suitable for these purposes, as well as their potential possibilities motivated the authors to resume work on optimizing the accelerated synthesis of HTSC ceramics with unprecedented physical characteristics.

2. Values of obtained unprecedented characteristics

We reviewed various technologies for the synthesis of thallium and bismuth ceramics and implemented the synthesis technology that produces stable samples of HTSC ceramics with optimal and unprecedented characteristics. As a result, using ceramics samples of various stoichiometric compositions, we obtained superconducting transition temperatures from 93 to 125 K with transport critical current density of more than 1000 A/cm^2. These values J_c were obtained by the method of electrical transport measurements at zero magnetic field and a temperature about T_c. X-ray diffraction spectra were studied, the Meissner effect was measured, and magnetic and resistive measurements were carried out for samples with different superconducting transition temperatures [5,6].

The authors used standard physical equipment described in many articles on experiments related to the synthesis of HTSC materials. The author's development is exactly the technological accelerated modes of synthesis, into which a number of „know-how„ were introduced, which ultimately give the results presented here.

During the studies, the density of the transport critical current was measured by the method of voltage drop across the samples. The specific cross section of transport HTSC harnesses on the samples was considered. The transport critical current density was calculated through the cross section of the sample, not the HTSC harness. And since, according to X-ray diffraction spectra (Figure 1), the volume fraction of HTSC harnesses in the body of HTSC ceramics samples was estimated at the level of 25–30%, there is reason to state that the actual density of the transport critical current in the HTSC harnesses of the obtained samples reached several thousand A/cm^2. That is, if you increase the specific density of HTSC harnesses in the material, then you can increase the density of transport critical currents by several times.

In the paper, in addition to single-phase samples with HTSC transition at 125 K, on which the synthesis technology was mainly tested, the individual samples were obtained containing two, three, or more phases, with abnormally high temperatures of superconducting transitions at 145–150 K,

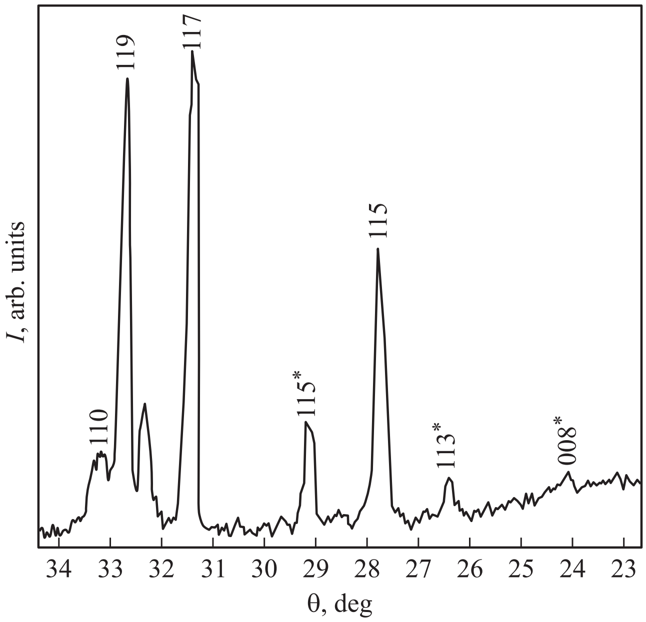

Figure 1. X-ray diffraction spectrum of sample $Tl_2Ba_2Ca_{n-1}Cu_nO_{2n+4}$, representing is a mixture of two superconducting phases — 2223 and 2212.

170–175 K, 190–205 K, and even 270–275 K. This indicates an actual real possibility of increasing the critical temperature of the transition to the superconducting state up to reaching the temperature range of „dry ice" and above.

In 1989–1990 we developed the technology for stable accelerated synthesis of thallium HTSC ceramics. The synthesis process was significantly reduced in time (compared to known two-stage technologies). This technology, after some updating, can be transformed into a factory technological process.

3. Structure and temperature dependence of properties of obtained HTSC samples

Tl-HTSC-materials are described by two general chemical formulas $Tl_2Ba_2Ca_{n-1}Cu_nO_{2n+1}$ or $Tl_{1-x}M_xA_2Ca_{n-1}O_{2n+1}$. Here A = Ba, Cr or their combination, and M = Pb, Bi or their combination, $n = 1, 2, 3, 4, 5$. First one contain double thallium layers called „Tl-bilayers" — these are Tl — 2201, Tl — 2212, Tl — 2223 and Tl — 2234 with $n = 1, 2, 3$ and 4. The latter are called „Tl monolayers" and contain the compositions Tl — 1201, Tl — 1223 and Tl — 1234. The layered structure of phases for various samples of Tl-HTSC ceramics is shown in Figure 2.

Measurements of the properties of previously obtained massive samples (tablets) of HTSC ceramics were carried out in Grozny (ChISU) and Rostov-on-Don (RSU) in 1989–1990, at the physics and chemistry departments of Moscow State University in 2011–2012 and 2022.

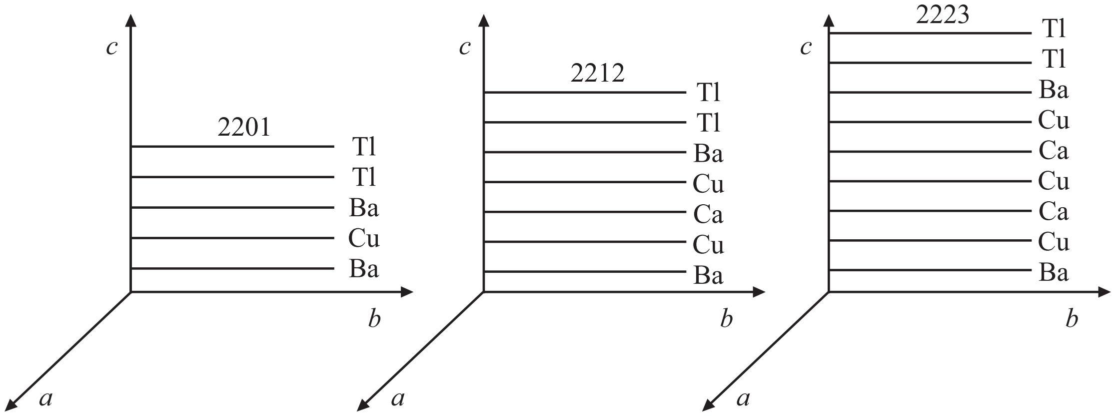

Figure 2. Scheme of the layered structure of various phases of thallium superconductors of type $Tl_2Ba_2Ca_{n-1}Cu_nO_{2n+4}$.

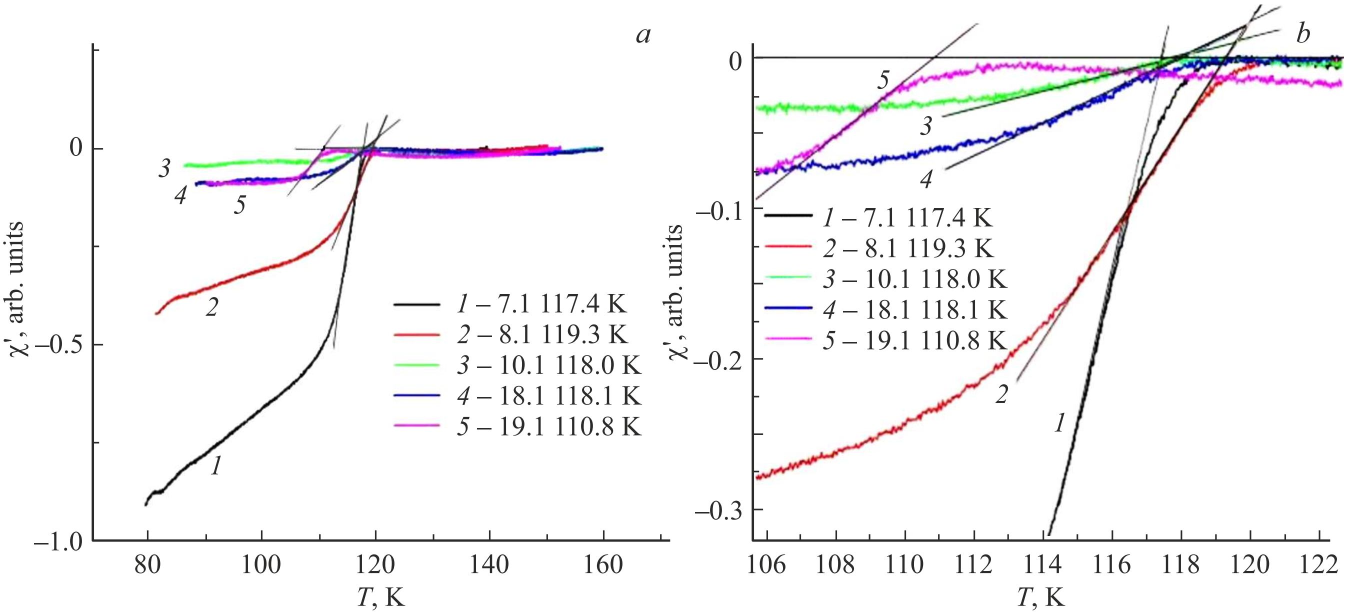

Figure 3. magnetic susceptibility of Tl-ceramics vs. temperature: *a*) for five samples of different composition „y" with T_c of 110 to 120 K; *b*) same results with zoom of temperature range of basic transition.

The experimental results obtained (Figure 1, *b*) confirm that HTSC-samples have standard high and unprecedented characteristics: temperature of superconducting transition $T_c = 125$ K and higher, transport critical currents up to 10000 A/cm^2 and other characteristics. The values of the parameters obtained in the paper are consistent with the results of other authors [11–13].

The results obtained using the proposed synthesis technology for thallium and bismuth samples of HTSC-ceramics were partially published in the regional mass media and conference proceedings [5]. The results of these studies for thallium HTSC-ceramics still remain unprecedented.

Figure 3 shows the results of measurements of the temperature dependence of the real part of the magnetic susceptibility χ of Tl-HTSC ceramics of five samples with different composition „y": (*1* — 7.1; *2* — 8.1; *3* — 10.1; *4* — 18.1; *5* — 19.1% O_2). Measurements of magnetic susceptibility (Figures 3 and 4) were made at alternating current of low frequency (50 Hz) on samples (tablets: $R \approx 4.0$ mm and $h \approx 2.5$ mm) with temperature of superconducting transition $T_c \leq 125$ K, this excludes the possibility of significant effect of Foucault currents.

When measuring the electrical resistivity of the samples, we observed both traditionally high temperatures of superconducting transitions from 93 to 125 K, and abnormal high temperatures of superconducting transitions of individual phases at 145–150, 170–175 and 193–205 K for multiphase samples, as curves in Figure 4 and Figure 5 show.

Characteristics of HTSC ceramics samples obtained previously (1989–1990) [4–13], were re-measured in 2012 at the chemical and physical faculties of Moscow State University for degradation over time. In January 2022 Measurements of the Meissner effect were again carried out with samples studied in 1990 and in 2012. Experiments showed that all samples retained their HTSC properties within 33 years when stored in the open air.

During experiments in 2022 It was observed that when heated to a critical temperature T_c the sample containing one phase immediately returns to the magnet. Samples with a multiphase structure return to the magnet in steps as each phase reaches the critical temperature T_c. Table 1 shows the values of the critical temperature T_c for various phases of Tl-HTSC samples.

Abnormal high values T_c of individual phases of multiphase samples, synthesized by authors, confirm the previous suppositions on possibility of existence of superconducting transitions at temperatures much larger then 125–127 K, up to temperature of „dry ice" and higher. The determined by us modes for HTSC ceramics synthesis make this possibility quite real.

Table 1. Value of critical temperature of T_c samples pf Tl-HTSC-ceramics by breaks in curves of the electrical resistance $R(T)$ and magnetic susceptibility $\chi(T)$

No. of sample; measured property	Temperature T_c for various phases of Tl-HTSC samples by measurements of 1989–90, (K)	New T_c, identified in graphs by measurements of 2012 and 2022, (K)
10.1; $R(T)$	113; 123; 145; 205	263
10.1; $\chi(T)$	113; 150; 190	205
7.1; $\chi(T)$	113; 143	183
18.1; $R(T)$	103; 123; 133; 190	250
18.1; $\chi(T)$	90; 103; 123; 153; 173	203
19.1; $R(T)$	103; 115; 121; 143; 153; 180	243; 270

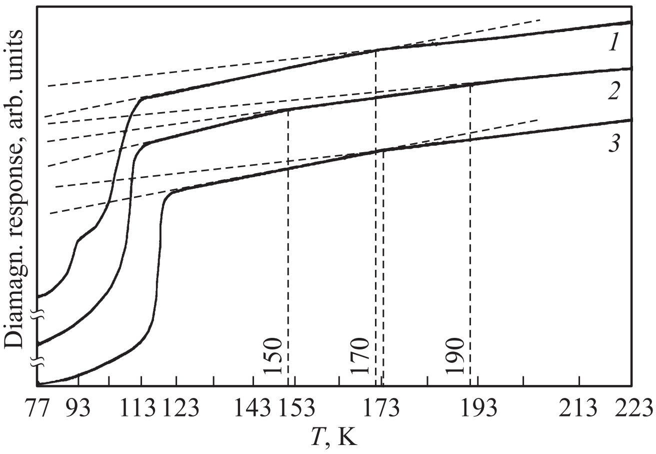

Figure 4. Magnetic susceptibility (Meissner effect) in multiphase samples of Tl-HTSC-ceramics with characteristic breaks in the curves at temperatures significantly exceeding T_c of basic transition. Concentration of cations Me^{2+} increases in sequence $x_1 < x_2 < x_3$.

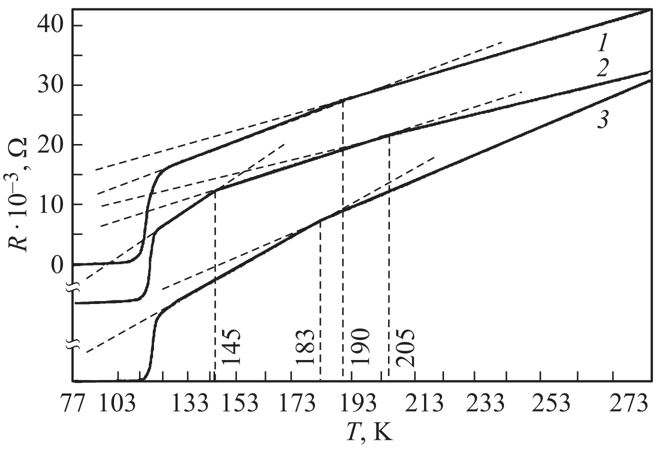

Figure 5. Temperature graph of electrical resistance (four-contact method) of Tl-HTSC ceramics samples with breaks at temperatures significantly exceeding T_c of basic transition: Concentration of cations Me^{2+} increases in sequence $x_1 < x_2 < x_3$.

In the future, the technological problem is reduced to separating individual phases with the required T_c from multiphase samples of Tl-HTSC ceramics with further obtaining from these monophases of HTSC materials with abnormal high temperatures of the superconducting transition. The concept of such phase separation was developed by the authors already in 2012.

4. Hubbard model of superconducting transition in Tl(Bi)-HTSC ceramics

The discovery of high-temperature superconductivity (HTSC) without any significant isotope effect indicates the existence of non-phonon mechanisms of superconductivity. One of these mechanisms is implemented in the Hubbard model in infinite repulsion ($U \to \infty$) and the hopping integral t. In this case, the Hamiltonian of the system is considered in the form [11–13]:

$$H = \frac{U}{2}\sum_{l\sigma}[\hat{n}_{l\sigma}\hat{n}_{l\bar{\sigma}} - \mu\hat{n}_{l\sigma}] + {\sum_{l,l',\sigma}}' t(l-l')\alpha^{+}_{l\sigma}\alpha_{l'\sigma}, \quad (1)$$

$$\hat{n}_{l\sigma} = \alpha^{+}_{l\sigma}\alpha_{l\sigma}, \quad (1a)$$

here l — electron position in lattice cell (atom) with spin σ or $\bar{\sigma} = -\sigma$, U — maximum repulsive force in a given cell (l), μ — chemical potential, t — overlap integral, α^{+}, α — creation and destruction operators of elementary excitation.

4.1. Superconducting transition temperature $T_c(x)$.

Superconductivity is realized in the lower Hubbard subband with half-width ω and filling number $n = 1 - x$,

Table 2. Calculated temperature values T_c for given concentrations x

x	0.025	0.050	0.100	0.150	0.200
T_c, K	99.9	128.5	140.4	119.0	71.4

where x — degree of band underfilling due to the presence of substitutional impurities in the sample M_x with concentration x. For $n < 1$ consideration of diagrams for the vertex part [11,13] with a single-particle Green's function

$$G_\omega(p) = (i\omega - \xi_p)^{-1}, \ \xi_p = ft_p - \mu, \ f = (2-n)/2 \quad (2)$$

gives an equation for determining the superconducting transition temperature

$$\sum_p \xi_p^{-1} t_p th[\xi_p/2T] = 1, \quad t_p = \sum_e t(e)e^{-p}. \quad (3)$$

In this case, the chemical potential μ is specified by the average occupation number of electrons in the sublattice site of the cation M_x

$$n = 2(1-n/2)\sum_p n_F(\xi_p). \quad (4)$$

Here n_F — Fermi distribution, and the factors 2 and $(1-n/2) = f$ take into account the double spin degeneracy and the infinite repulsion ($U \to \infty$) of electrons at the value

$$n_F = 1 - x, \quad f = f_x = (1+x)/2. \quad (4a)$$

From the condition $T_c \to 0$ at $x_c = 1/3$ according to (3) we obtain that $T_c > 0$ in the region of electron concentrations $n > n_c = 3/2$ or $0 < x < x_c$. For two-dimensional sublattice of copper cations with a weakly varying density of states, the latter can be replaced by a rectangular function θ with the half-width of the Hubbard subband ω:

$$\theta(\varepsilon) = \left(\frac{1}{2}\omega\right)\Delta(\omega^2 - \varepsilon^2), \quad (5)$$

where $\omega = \max t_p$.

In this simple but actual model according to (2) and (3), we obtain

$$\frac{n(2+n)}{2-n} = \frac{\mu}{\omega}\ln\frac{4\gamma^2(\omega^2-\mu^2)}{\pi^2 T_c^2}, \quad (6)$$

$$\frac{3n-2}{2-n} = \frac{1}{T_c\omega}\ln\frac{\mathrm{ch}(\omega+\mu/2T_c)}{\mathrm{ch}(\omega+\mu/2T_c)}, \quad (7)$$

where $2\gamma/\pi = 1.14$ — known constant of the Bardeen–Cooper theory, ω — the half-width of the lower Hubbard subband. Excluding μ from (6) and (7) under condition $n > n_c = 3/2$, we obtain an explicit expression for the superconducting transition temperature

$$T_c = \frac{2\gamma}{\pi} 3\omega \sqrt{\frac{n(1-n)}{2}} \exp\left[-\frac{n(2+n)}{6(n-n_c)}\right]. \quad (8)$$

For samples of Tl-HTSC ceramics T_c initially increases with increasing of $n > n_c$, then returns to zero at $x \to x_c = 1/3$ and increases, according to (8), at $x \to 1$ under the exponential law of the Bardeen–Cooper type (Figure 6, *a*):

$$T_c = \frac{2\gamma}{\pi}\omega\exp\left[\frac{-8}{27(x_c - x)}\right]. \quad (9)$$

Thus, in the range $0 < x < x_c$ the calculated phase transition temperature T_c as a function of the concentration x of divalent cations Me_x^{2+} has a maximum, which is consistent with the results of our experiments for samples of thallium superconducting ceramics of various concentrations (y), (Table 2, Figure 6). The transition temperature T_c initially also increases with increase in number of layers Cu_{n_1} ($n_1 = 1, 2, 3, 4, 5$) for $n_1 < 3$, then decreases at $n_1 > 3$ (Figure 6, *b*) and further passes via minimum.

The family of thallium-based high-temperature superconductors turned out to be very promising both from the point of view of the critical transition temperature increasing and from the point of view of resistance to variations in manufacturing process modes. Optimization of manufacturing conditions and variation of composition made it possible o obtain samples of HTSC ceramics from family $T1_mBa_2Ca_{n-1}Cu_nO_{2n+m+2}$, where $m = 1, 2$ and $n = 1, 2, 3, 4$ (sometimes 5), with transition temperatures 90, 110, 115 and 125 K and higher, and with critical currents up to 3000–10000 A/cm^2 and higher. For the values of transition temperatures with realistic width of the lower subband $2\omega = 0.12$ eV of the family (2223) of TI-Ba-Ca-Cu-O-ceramics, using formulas (8) and (9) we obtain the values T_c, given in Table 2.

4.2. Magnetic susceptibility.

Magnetic permeability $\varkappa = \chi + 1$(χ — magnetic susceptibility) can be obtained by considering the magnetization $\mathbf{M} = \chi\ \mathbf{H}$ of superconductor in weak magnetic field H. The magnetic field splits each Hubbard subband into two, which contain n_+ and n_- electrons with spin along and against the magnetization of $M = n_+ - n_-$ electrons (in Bohr magneton units). In „Hubbard 1" approximation [13], the electron bands with spin projection σ have the form

$$\xi_p^{(\sigma)} = \frac{t_p}{2} + \frac{U}{2} - \sigma H - M \pm \frac{1}{2}\left[(t_p - U)^2 + 2Ut_p(n - \sigma M)\right]^{1/2}. \quad (10)$$

Using the formulas for n_+ and n_- [13], integrating with the model density of states (5), we obtain magnetization $\mathbf{M}$:

$$\mathbf{M} = (\varkappa - 1)\mathbf{H} = -\left\{1 - (2/w)\,\mathrm{th}\left[(1-x)w/(2T)\right]\right\}\mathbf{H}. \quad (11)$$

Dependences (11) for $\varkappa_x(T)$on $x = 1 - n$ and T are consistent with the experimental results shown in Figures 3 and 4.

4.3. Conductivity

The calculation of the conductivity tensor $\sigma_{\alpha\beta}$ is based on the use of the general Kubo formula [10]. In the case of low temperatures $T \ll \omega$, $\sigma_{\alpha\beta}(T)$ has the form

$$\sigma_{\alpha\beta}(T) = \frac{2e^2}{V} \sum_p \frac{\partial e}{\partial p_\alpha} \frac{\partial e}{\partial p_\beta} D_+(p) D_-(p), \qquad (12)$$

where $D_\pm(p)$ — normal Green's functions [13] and e — excitation energy.

Integrating near the Fermi surface $\varepsilon_F = f t_p^{\max}$ when $t_p^{\max} - \mu/f = 2\mu/(1+x)$, using formula (12) we obtain

$$\sigma_{\alpha\beta}(T) = \frac{2\pi e^2 \rho}{3m_{\alpha\beta}} \Phi[Z(T)], \qquad (13)$$

$$\Phi(Z) = \frac{1+Z^\tau}{Z} \pi \nu_0 \rho_F K_1 \frac{\mu}{f}, \qquad (13a)$$

$$f = 2 - n, \quad \rho_F = \rho(\varepsilon_F), \quad \rho(\varepsilon) = \frac{1}{V} \sum_p \delta(\xi_p). \qquad (13b)$$

Here $K_1 = K_1(T)$ — spin fluctuation correlator, ρ_F — electron density of states at the Fermi level, ν_0 — constant in the formula for the square of the inverse screening length

$$L^{-2} = 4\pi e^2 \frac{\partial m}{\partial \mu} / \nu_0.$$

If $Z \ll 1$

$$\sigma_x(T) = \frac{2\pi e^2 \rho_F}{3m} \frac{1}{Z_x(T)}, \qquad (14)$$

$$Z_x(T) = \pi \nu_0 \rho_F K_1(T) \mu / f_x. \qquad (14a)$$

Here the mass tensor is approximately expressed through the scalar mass m. At $T \ll \omega$ spin fluctuation correlator is $K_1(T) \approx 11 \cdot T/3\omega$, and for electrical resistance $\rho = 1/\sigma$ we obtain

$$\rho_x(T) = B_x T, \qquad (15)$$

where $T_c < T \ll \omega$,

$$B_x = \frac{11 \cdot m v_0^2}{3e^2 \omega} \frac{1}{f_x}, \quad f = f_x = \frac{1+x}{2} = 1 - \frac{n}{2}. \qquad (15a)$$

According to (14a) $Z = 1$ at $T_0 = 3f\omega/(11\pi\nu_0\rho_f\mu)$, and at $Z > 1$ we have $\rho_x(T) \propto (1/T)$. Dependence (15) for $\rho_x(T) \propto T$ at $Z \ll 1$ is consistent with the experimental results (Figure 5).

5. Conclusions

1. Accelerated modes of synthesis of thallium-containing samples (tablets) of HTSC ceramics with unprecedented superconducting transition temperatures ($T_c = 124$ K and more) and with critical current densities (J_c) up to thousands A/cm^2 were found. A technology of stable standard (two-stage) accelerated synthesis (less than a day instead of 48 h) of thallium HTSC ceramics was developed. The synthesis process was significantly reduced in time (compared to known two-stage technologies) [7]).

2. Based on the breaks in the curves of conductivity $\sigma(T)$ and magnetic susceptibility $\chi_x(T)$, possible values of T_c up to 205 K were determined. The Hubbard model determines $T_c(x)$, magnetic susceptibility $\chi_x(T)$ and conductivity $\sigma_x(T)$ versus the concentration x of cations Me_x^{2+}, Me = Pb or Bi, and temperature T. The results of the corresponding calculations are consistent with experimental data.

3. Operating temperatures (T_c) of new generation of Tl(Bi)-HTSC materials exceed the current level of 125−127 K and, according to experimental data Figure 4, 5 and Table 1, reach temperatures of „dry ice" (195 K) and higher. This makes it possible to switch to a qualitatively different temperature mode for the operation of superconductors and all technical devices created on their basis.

4. The density of the transport critical current in the resulting Tl-HTSC materials reaches over 1000 A/cm^2. Similar results were previously obtained only on HTSC films, but not on macrosamples. However, „sandwich" technologies turned out to be unpromising [7].

5. Despite the unprecedented physical parameters and relatively accessible technology for producing macrosamples of Tl(Bi)-HTSC ceramics, the production of products based on it is less developed than on the basis of other HTSC materials. In particular, this circumstance increases interest in samples and products specifically based on Tl(Bi)-HTSC materials.

Thus, the technology proposed by the authors, with appropriate updating, will make it possible to create the production of HTSC materials of new generation, which have favorable prospects in comparison with the known HTSC materials produced using „sandwich technology„.

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Conflict of interest

The authors declare that they have no conflict of interest.

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