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Resistance and Susceptibility of Ceramic $Tl_2Ba_2Ca_2Cu_3O_x$ Superconductor in the Critical Region

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Bulk $Tl_2Ba_2Ca_2Cu_3O_x$ superconductor was synthesized and oxygenated. The critical temperature was determined to be 116.5 K from resistivity, 117.5 K from AC susceptibility (intra-grain) and 115.0 K from AC susceptibility (inter-grain). The critical current at 77.3 K, utilizing a non-contact Bean's method, is 189 A/cm^2. After oxygenation, the sample contained 60% mass of the desired compound (X-ray diffraction measurement). The critical exponent in the closest vicinity of the critical temperature occurred to be 0.112 (AC susceptibility) and 0.259 (resistance). Further, from the critical temperature the susceptibility exponent was determined to be 0.779 and the resistance critical exponent was equal to 1.13.

topics: thallium, cuprate, critical current, critical temperature

1. Introduction

There are six critical exponents describing, for example, a superconductor. They are connected by four relations hence only two of them are independent [1–5]. The square of the order parameter $|\psi|^2$ scales with reduced temperature $\varepsilon = (T - T_c)/T_c$ in the vicinity of the critical point as $\varepsilon^{-\gamma}$. The critical point for superconductors exists at the temperature of phase change from normal to superconducting state. The order parameter manifests experimentally as sample diamagnetism [6, 7] or sample enhanced conductance [7, 8] (the difference of experimental conductance and predicted from linear $R(T)$ dependence).

$Tl_2Ba_2Ca_2Cu_3O_x$ superconducting material was chosen because of its high critical temperature of 125–127 K [9–12]. The critical temperature of this compound is one of the highest among cuprates and depends on oxygen content [11] which is the result of synthesis and oxygenation conditions. The sample can contain unreacted substrates, other oxides as CaO or even other superconductors as Tl-1223, beside the desired Tl-2223 phase after the synthesis [13–15].

AC susceptibility and resistivity measurements are able to detect the critical temperature and critical current of the main phase [16–19]. Enhanced conductivity is visible [20] but it is difficult for a bulk polycrystal to separate the Aslamazov–Larkin and Hikami–Larkin (Maki–Thomson) term. There is no resistance peak at T_c vicinity connected to the density of states (DOS) term.

In this article, Tl-2223 bulk superconductor is presented. Resistivity and AC susceptibility measurements are performed to determine the critical temperature and critical current and to approximate the value of the γ critical exponent from two kinds of measurements. In the case of resistivity measurement, it is necessary to introduce enhanced conductivity $\Delta\sigma = \frac{1}{R} - \frac{1}{R_R}$ which is used to determine the critical exponent $\Delta\sigma = K\,\varepsilon^{-\gamma}$. The modulus of magnetic AC susceptibility is another quantity determining the critical exponent: $|\chi| = K'\,|\varepsilon|^{\gamma}$. The critical exponent is a value of a regression slope of the function $\log(\Delta\sigma)$ (or $\log(|\chi|)$) versus the logarithm of the reduced temperature ε.

2. Experimental

Firstly, $BaCuO_2$ and Ca_2CuO_3 oxides were synthesized using a solid state reaction method. Then, the oxides and Tl_2O_3 were mixed in a mortal to obtain the resulting Tl-2223. After mixing, the material was pressed under pressure of 0.59–0.52 GPa. The pellet of 13 mm diameter was wrapped in a double silver layer. The wrapped sample was heated in flowing oxygen gas at 880 °C for 30 min.

A not wrapped sample was oxygenated at 760 °C for 20 h. The heating and cooling rate was 5 °C/min when the temperature was below 750 °C and 1 °C/min in the temperature range 750–760 °C.

X-ray diffraction (XRD) patterns of the sample of Tl-2223 were achieved on Empyrean Panalytical diffractometer at room temperature (CuK$_\alpha$ radiation). The microstructure was analysed on a scanning electron microscope (SEM) type JEOL 5900LV. Four-point method was applied to measure resistance versus temperature and versus magnetic field up to 2.1 kGs dependence. The contacts with resistance smaller than 7 Ω at room temperature were made using Leitsilber 200 silver paint. The sample was supplied with 1 mA current with frequency of 29 Hz from SR 830 Stanford Lock-in nanovoltmeter. Both dispersion and absorption parts of AC magnetic susceptibility versus temperature and versus AC magnetic field (amplitudes 0.02 Oe to 11 Oe) were measured by a standard mutual inductance bridge working at frequency of 189 Hz. Stanford SR-830 Lock-in nanovoltmeter was a source of AC current, as well as voltmeter of the bridge. A Lake Shore temperature controller connected to a chromel-gold–0.07% thermocouple controlled the temperature with accuracy of ±0.05 K.

3. Results and discussion

Both before and after the oxygenation, Tl-2223 is a dominating phase. With the use of XRD method, also other oxides after the oxygenation (Fig. 1) were detected. The sample critical temperature and current density before the oxygenation are not very high (102.0 K and 20.4 A/cm^2). As a result of the oxygenation, the value of T_c increased to 117.5 K and the critical current at 77.3 K achieved the value of 189 A/cm^2 despite the fact that the mass content of the dominating superconductor fell. The last effect might be the result of thallium loss during the oxygenation.

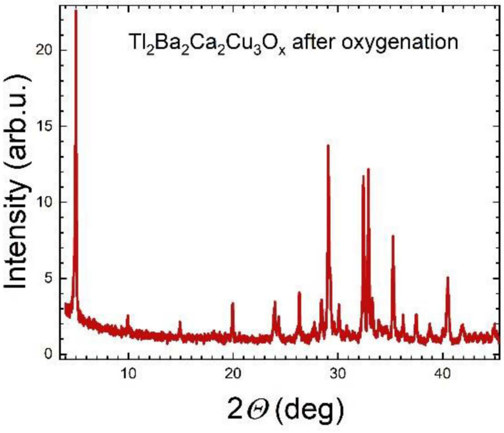

Fig. 1. XRD spectrum of the sample after oxygenation.

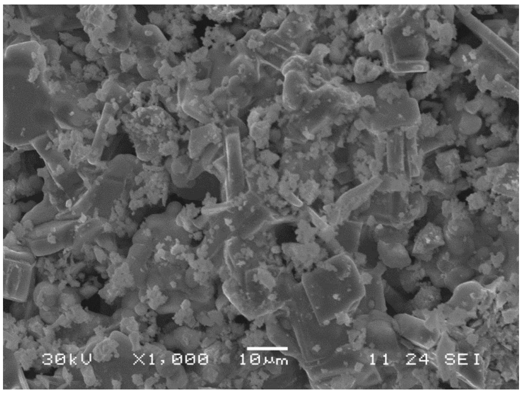

Fig. 2. SEM image of the cross-section of Tl-2223.

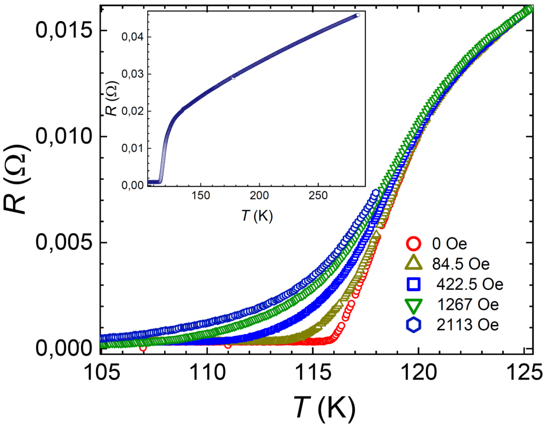

Fig. 3. Resistance of Tl$_2$Ba$_2$Ca$_2$Cu$_3$O$_x$ superconductor in various magnetic fields. Inset: resistance in the temperature range from 77 K to 290 K.

The SEM picture (Fig. 2) shows well-developed superconducting grains in the form of rectangular plates and bars with dimensions up to 20 µm. Besides the superconductor parts (recognized by energy distribution of X-ray, EDX) the impurities having irregular shapes (light shade) are visible. The pictures were taken on the cross-section, not the surface of the pellet before oxygenation.

The resistance versus temperature dependence of the oxygenated sample is shown in Fig. 3. The onset critical temperature (127.4 K) is insensitive to the applied DC magnetic field. The transition to a superconducting state, as it is for HTS, is not sharp. The DC magnetic field shifts the transition towards lower temperatures and broadens it. Zero resistance temperature T_{c0} varies from 116.5 K without the DC magnetic field to 110.7 K for 2.1 kGs. In the case of $T_{c50\%}$, it changes from 119.4 K to 119.0 K, respectively. Transition width ΔT is 8.0 K without the field and 12.0 K for the maximal DC magnetic field.

The susceptibility versus temperature curves are shown in Fig. 4. A dispersive part of AC susceptibility serves to determine the critical temperature. One can also utilize Bean's method [21, 22]

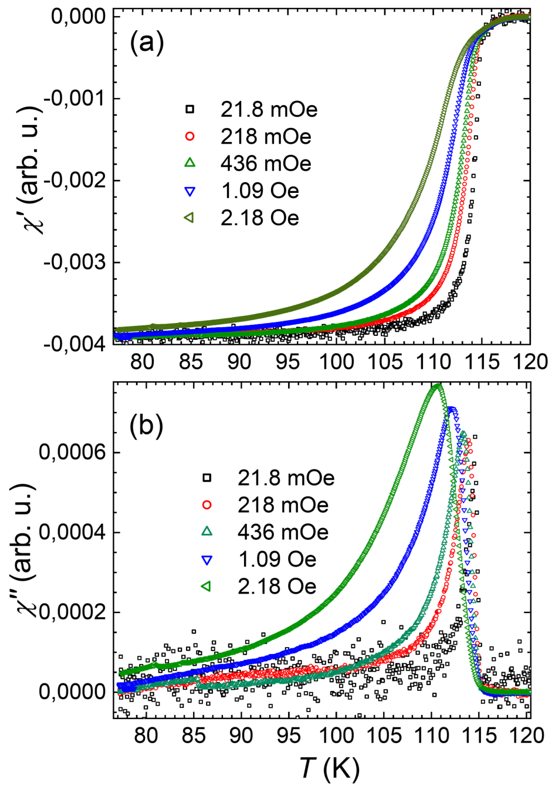

Fig. 4. Dispersion (a) and absorption (b) part of AC susceptibility as a function of temperature of thallium-based 2223 superconductor for selected amplitudes AC magnetic field.

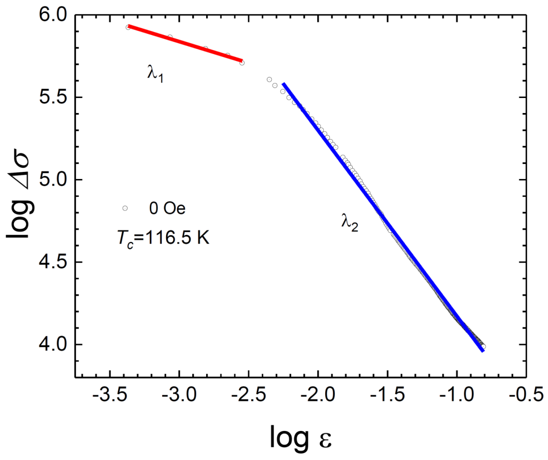

Fig. 5. Log–log chart from resistance measurement in zero DC magnetic field.

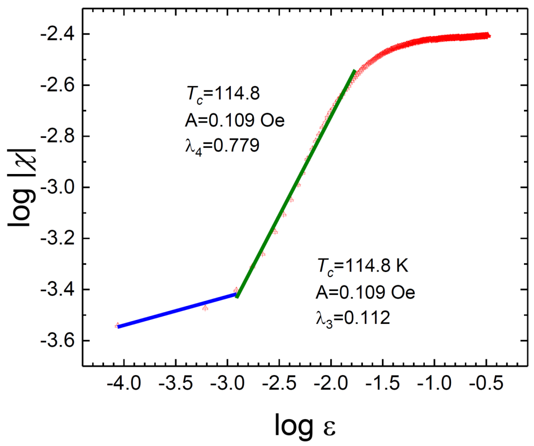

Fig. 6. Log–log chart from AC susceptibility measurement at 0.109 Oe AC magnetic field.

and the Ginzburg–Landau strong coupling limit approach [23, 24] to determine a critical current curve from the absorption part of AC susceptibility.

Both AC susceptibility and resistivity data can be used to approximate the critical exponent γ, e.g., from log–log plot (Figs. 5 and 6, respectively). The mentioned critical exponent describes how the square of the order parameter $|\psi|^2$ changes with temperature. Both the enhanced conductance and diamagnetic response are proportional to $|\psi|^2$. From the AC susceptibility measurement, one can obtain the value of the critical exponent below the transition. Resistivity provides data to determine the exponent above the transition.

The critical temperature read from the AC susceptibility measurement at $H_{ac} = 0.022$ Oe of the oxygenated sample (see Fig. 4a) is 117.5 K (intra-grain, deviation) or 115.0 K (inter-grain, cross-section [19]). The absorption susceptibility part has a peak whose position goes towards lower temperatures as a higher AC magnetic field is applied (Fig. 4b). The critical current at 77.3 K, assuming the critical temperature of 115.0 K, is 189 A/cm^2 as estimated from the Ginzburg–Landau strong coupling limit fit.

The critical exponent close to the critical temperature was calculated. The exponent values are $\lambda_3 = 0.112$, just below the critical temperature, and $\lambda_1 = 0.259$, just above the critical temperature (Figs. 5 and 6, respectively). For temperatures further from the critical one, higher values are obtained ($\lambda_4 = 0.779$ from AC susceptibility, Fig. 5, and $\lambda_3 = 1.13$ from resistance, Fig. 6). Susceptibility exponents were calculated from measurement at $H_{ac} = 0.109$ Oe. The values of critical exponents are — to our knowledge — a new result for the Tl-2223 bulk system. Exponents for the Tl-1223 bulk system were presented for instance in [25]. The Tl-2223 bulk system turned out to be (i) 3D (assuming critical fluctuations $d = 7/2–3\lambda/2$) in the vicinity of T_c, and (ii) 2D further from T_c (assuming Gaussian fluctuations $d = 4–2\lambda$), both above and below T_c.

4. Conclusions

Thallium-based $Tl_2Ba_2Ca_2Cu_3O_x$ superconductor has a zero resistance temperature $T_{c0} = 116.5$ K, the critical temperature $T_{c50\%} = 119.4$ K and the transition width $\Delta T = 8.0$ K at the zero applied magnetic field. The critical current density obtained by the non-contact method at liquid nitrogen temperature is $J_c = 189$ A/cm^2. After the oxygenation process, the critical temperature grew by 15.4 K (AC susceptibility) and critical current increased nine times. At the same time, the mass content of Tl-2223 superconductor decreased. Resistivity measurement enables to determine critical exponents $\lambda_1 = 0.259$ and $\lambda_2 = 1.13$. The critical exponents from susceptibility data are $\lambda_3 = 0.112$ and $\lambda_4 = 0.779$. The exponents λ_1 and λ_3 are close to T_c ones. The presented values of the critical exponents are new results for the Tl-2223 bulk system.

Acknowledgments

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