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Dependence of Oxygenation Temperature on Critical Temperature and Current for $Tl_2Ba_2Ca_2Cu_3O_x$

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Thallium based $Tl_2Ba_2Ca_2Cu_3O_x$ superconductors were synthesized in double silver foil at 880 °C for 30 min under flowing oxygen gas. Additional oxygenations, each lasting 20 h, were performed at temperatures varying from 700 °C to 820 °C to increase the critical temperature and the critical current of the superconductor. Optimal temperature of the oxygenation was of 760 °C.

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PACS/topics: thallium, cuprate, critical current, critical temperature

1. Introduction

Thallium-based superconductors are known from having the highest critical temperatures among cuprates although their properties, especially in the range of transition to the superconducting state, are still poorly recognised. These superconductors, particularly these in the form of thin layers due to their critical temperatures higher than the boiling point of liquid methane (about 112 K), can be used for current transmission lines combined with the liquid natural gas transmission lines cooled with this gas.

The case of Tl-2223 compound was previously studied by Kaneko [1] and Nabatame [2]. In the former work (encapsulated or not) Tl-2223 has been heated at 760 °C in various times, achieving 127 K of zero resistance transition temperature. In the latter work thin films of Tl-2223 on MgO substrate have been studied. SEM images of the film surface were confronted with critical current density and heating conditions.

Zalecki et al. [3] studied bulk Tl-2223 sintering in silver foil [4] consisted of two steps for 0.2 h at 925 °C, and for 10 h at 910 °C. These steps corresponded, respectively, to the synthesis and the oxygenation steps. There, from AC magnetic susceptibility T_c was determined as the critical temperature of separated polycrystalline grain.

Thallium based superconductors in the 2223 system after short (30 min) synthesis in oxygen atmosphere showed low critical temperature of 107.1 K and current of 37.5 A/cm^2. Thermal treatment in oxygen atmosphere can considerably change properties of a thallium cuprate. Possible processes that might be involved are oxygen interpolation [5], thallium loss [6], and change of chemical content [7]. Oxygenation is carried out according to temperature versus time relation.

In this work, we present a detailed study of the dependence of critical temperature and critical current density on the oxygenation conditions of thallium based superconductors.

2. Experimental

To obtain $BaCuO_2$ and Ca_2CuO_3 precursors, the appropriate amounts of the starting powders were sintered at 840 °C for 24 h, and then at 940 °C for 48 h. Next, the materials were reground and placed into furnace for 48 h at 940 °C. To obtain the Tl-2223 superconductor, appropriate amounts of $BaCuO_2$, Ca_2CuO_3, and Tl_2O_3 were ground and pressed into pellets which were wrapped in silver foil. The pellets were heated at 880 °C for 30 min in a furnace in flowing oxygen gas. The samples were cut into pieces, and they were oxygenated for 20 h at various temperatures. For the pieces of the first sample (sample A) the oxygenation temperatures were 700–760 °C. For the other sample (sample B) the oxygenation temperatures were 760-820 °C. The AC susceptibility measurements were done using mutual inductance bridge technique at the AC magnetic field ranging from 0.022 Oe to 10.9 Oe. A Stanford SR 830 lock-in nanovoltmeter was serving as a source of the AC current for the primary coil and as the nanovoltmeter of the bridge. The temperature was measured with the accuracy of 0.05 K by a chromel-gold-0.07% iron thermocouple working with a Lake Shore temperature controller. The samples investigated had shapes of a flat cuboid and the magnetic field direction was parallel to their longest edge. The working frequency was set to 189 Hz. The X-ray powder diffraction (XRD) was performed with Empyrean Panalytical diffractometer (CuK_α) at 300 K in 2θ range from 4° to 80°. SEM images were obtained with JEOL 5900LV apparatus.

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3. Results and discussion

The XRD pattern of the sample A oxygenated at 760 °C is shown in Fig. 1. The majority phase of this sample is the Tl-2223 superconductor. The minority phases are residuals of $BaCO_3$, $BaCuO_2$, CaO and CuO, which are not superconducting. The microstructure of this sample was observed in SEM, and the image is shown in Fig. 2. Flat crystals with typical sizes 30×3 μm and dark spaces between them in the microstructure are visible. The crystals contact each other and often penetrate the neighbours. No preferred direction along which the crystals are oriented can be distinguished.

The dispersive and absorption parts of AC susceptibility of the samples after the oxygenation process are shown in Fig. 3a and 3b, respectively. Both the intra- and inter-grain critical temperatures of the samples were determined from the dispersive parts of AC susceptibility by the method described in [8]. The results are shown in Fig. 4. Additionally, Fig. 4 shows the critical temperatures as fitted parameters of (2). The intra-grain critical temperature raises with increasing oxygenation temperature in the whole range. Both, the inter-grain critical temperature and the critical temperature as fitting parameter have local minimum for the sample oxygenated at 780 °C. The highest intra-grain critical temperature was achieved for the 760 °C oxygenated sample, however, the highest inter-grain critical temperature and the critical temperature from fitting has the sample oxygenated at 800 °C. The critical current value for given temperature was obtained from the Bean formula [9]:

$$j_c = \frac{2H_{\mathrm{AC}}}{d}, \quad (1)$$

where H_{AC} is the AC magnetic field amplitude, and d is the sample thickness. The critical current calculation for the sample A and B oxygenated at temperatures ranging from 700 °C to 800 °C are shown in Fig. 5. For high temperature superconductors (HTS), in order to describe the dependence of the critical current on temperature one can used the Ginzburg–Landau strong coupling limit approach [10, 11], which is expressed by:

$$j_c = j_{c0}\left(1 - \frac{T}{T_c}\right)^n, \quad (2)$$

where j_{c0} can be interpreted as critical current at 0 K, T_c is the critical temperature, and n is the exponent, which characterises pinning strength. Above Eq. (2) that was used to fit temperature dependence of critical current

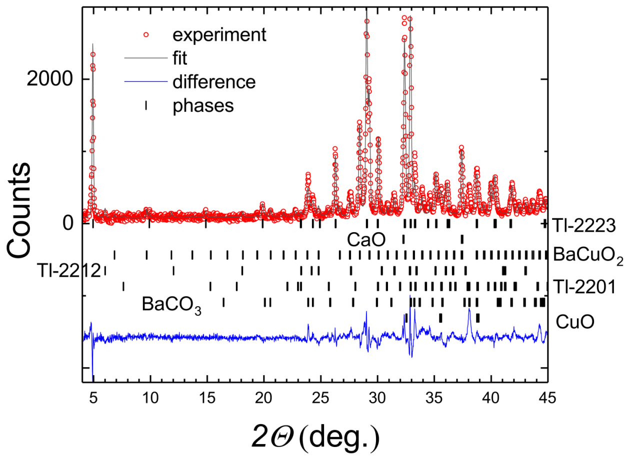

Fig. 1. XRD pattern of the sample A oxygenated at 760 °C.

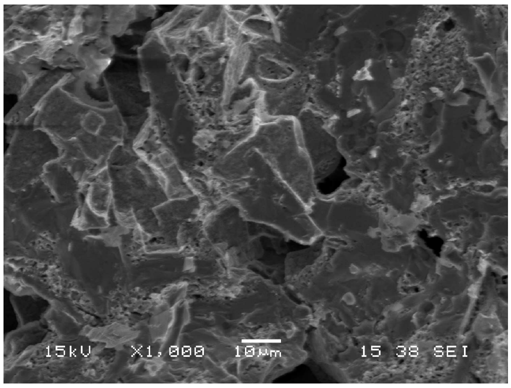

Fig. 2. SEM image of the sample A oxygenation at 760 °C.

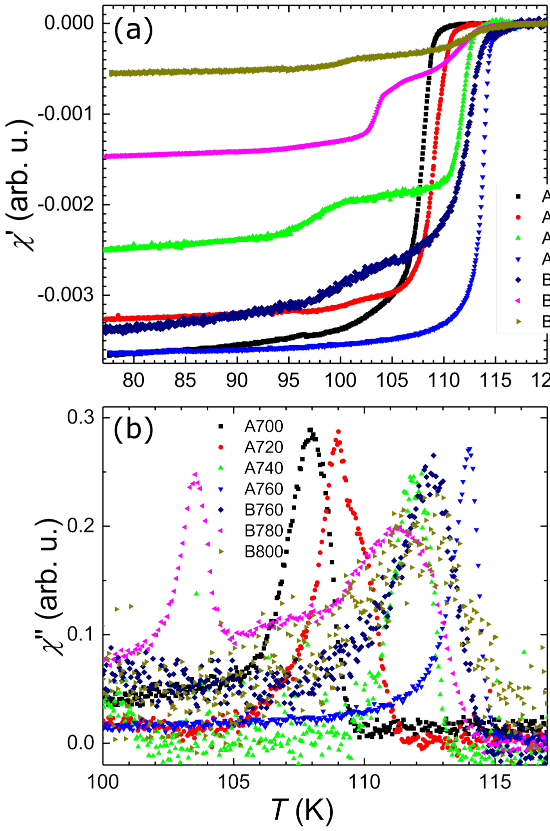

Fig. 3. Dispersive (a), and absorption (b) parts of the AC susceptibility for samples heated at 700–800 °C.

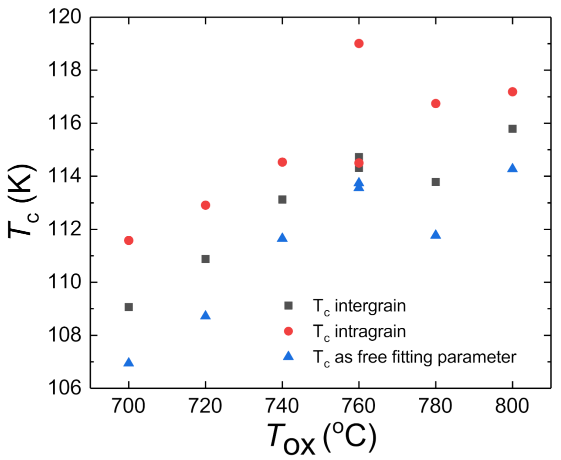

Fig. 4. The values of critical temperature for samples oxygenated in the range from 700 °C to 800 °C.

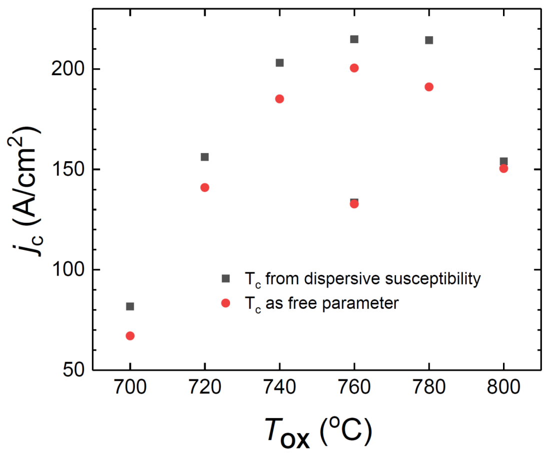

Fig. 5. The critical currents for samples oxygenated at temperatures from 700 °C to 800 °C.

density, it relates to two cases. In the first case there are two fit parameters: j_{c0} and n, but the third parameter T_c is taken from experiment as the inter-grain critical temperature (see Fig. 6a). In the second case three parameters are taken in fitting procedure (see Fig. 6b). Fit parameters are collected in Table I. Taking advantage of the all fitted parameters the critical current densities at the liquid nitrogen temperature were calculated for all the oxygenated samples, and they are shown in Fig. 5. To derive the critical current it was assumed that the value of inter-grain critical temperature is taken from the experiment (black squares in Fig. 5), or T_c is treated as a free-fit parameter (red circles in Fig. 5). It can be observed that the critical current density reaches the highest value for both variants.

Strong pinning regime typical for HTS [12] was found to be the case for the most of the studied samples because exponents n were greater than unity. The samples exhibited a vortex glass structure. Only samples A700 and A720 have n lower or greater than unity, depending on fitting procedure details.

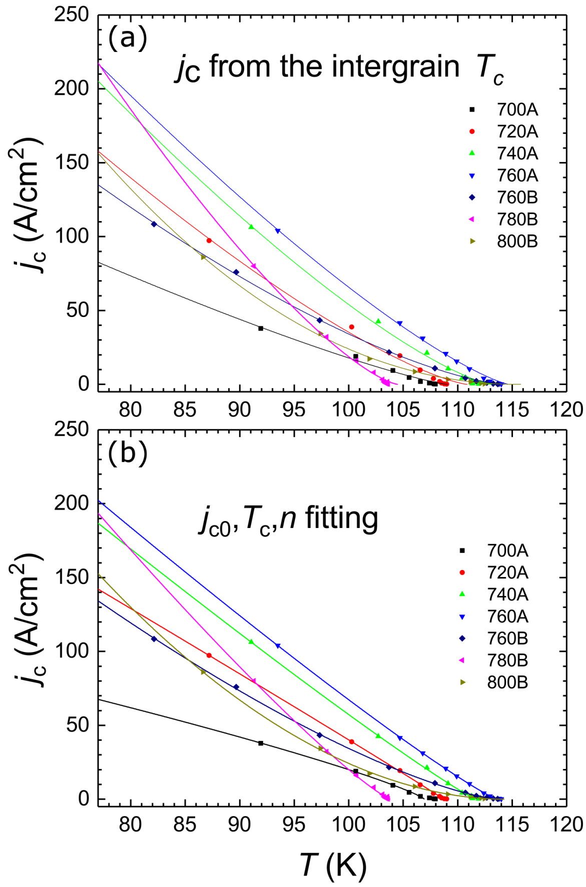

Fig. 6. The critical current versus temperature for samples oxygenated at 700 °C to 800 °C. (a) T_c as inter-grain temperature, and (b) T_c is free fitting parameter.

TABLE I

Parameters of j_c vs. T curves.

Sample	j_c [A/cm^2] with 77.3 K	j_{c0} [A/cm^2]	T_c [K]	n [1]
j_c from inter-grain temperature				
A700	82	360	109.07	1.21
A720	156	750	110.88	1.32
A740	203	920	113.12	1.32
A760	215	890	114.72	1.27
B760	134	675	114.31	1.437
B780	214	1330	104.50	1.35
B800	154	1540	115.79	2.09
Three fitting parameters				
A700	67	195	106.9	0.83
A720	141	475	108.72	0.979
A740	185	660	111.65	1.08
A760	200	711	113.56	1.109
B760	133	640	113.74	1.39
B780	191	930	103.8, 111.8	1.16
B800	150	1310	114.27	1.92

Two oxygenations performed at 760 °C for A and B series indicate different critical temperatures and critical currents. Thus, T_c and j_c determinations made here should be treated as qualitative.

Our study showed that oxygenation increases both the critical temperature and the critical current of the material. An exception was oxygenation at the highest temperature used (820 °C) where the sample lost its superconductivity related diamagnetism or its critical temperature was lower than 77 K.

4. Conclusions

The thallium based $Tl_2Ba_2Ca_2Cu_3O_x$ superconductor compounds oxygenated at temperatures from 700 °C to 820 °C for 20 h were obtained. Maximal j_c has been achieved for A760. The inter-grain, free fitting parameter T_c was able to raise with the oxygenation temperature up to B800. The highest intra-grain temperature was for A760.

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