Modern Physics Letters B
Vol. 32, No. 4 (2018) 1850102 (9 pages)

DOI: 10.1142/S0217984918501026

Microscopic study of the $YBa_2Cu_3O_{7-x}$ crystal

S. I. Bondarenko*, Qiudong Guo[†,‡,§], J. D. Fan[†,‡], A. G. Sivakov*,
A. V. Krevsun* and S. I. Link*

*Institute for Low Temperature Physics and Engineering of NASU,
Kharkov, Ukraine
†Chongqing Academy of Science and Technology,
Chongqing 401123, China
‡JD Duz Academy for Superconductivity, Chongqing 401120, China
§frd33@qq.com

Received 6 January 2018
Accepted 8 January 2018
Published 31 January 2018

The form and parameters of the superconducting temperature transition (STT) of microscopic parts of a quasi-single crystal domain of $YBa_2Cu_3O_{7-x}$ depend on their locations in the volume domain and they are different from the superconducting transition of a bulk sample. This difference can be explained by the presence of low temperature and normal phases of the Y–Ba–Cu–O compound.

Keywords: Superconductivity; YBCO; cuprate.

1. Introduction

32 years ago, in 1986 Swiss physicists Bednorz and Muller published a paper[1] about the experimental discovery of a new type of superconductor $(La,Ba)_2CuO_4$ with the transition temperature (T_c) in the superconducting state of about 35 K. This temperature exceeded the record of the transition temperature for that time in the compound Nb_3Ge ($T_c \approx 23$ K). The surprise was that the new superconductor was made of three non-superconducting oxides of lanthanum, barium and copper and was a typical ceramics. It was unusual and very interesting from the point of view of physics of superconductivity, but not from the point of view of superconductivity applications. The emergence of a superconductor with significantly higher transition temperature than from a compound that originally is a ceramics needs a revolution in the theory of superconductivity in order to really understand its mechanism.[2] A breakthrough was made by a group of researchers from the University of Houston in the U.S. led by Paul Chu. In January, 1987, he patented a new superconducting compound $YBa_2Cu_3O_{7-x}$ in the form of oxide ceramics with $T_c^{\text{onset}} \approx 90$ K. This

type of superconductor is known as a high-temperature superconductor (HTSC). This type of superconductor has transferred from the category of expensive and, in many cases, a unique "helium" technology, since the discovery of superconductivity in 1911, at a temperature of about 4 K to the category of significantly cheaper "nitrogen" technology in use of liquid nitrogen as a refrigerant with a boiling point of 77 K at normal pressure.

In the same year, a paper of Paul Chu's group[3] with a revolutionary compound (Fig. 1) was published and the results of the first experimental studies were about the properties of this type of compound.

From the given dependences, it is visible that the beginning of a sharp drop in the resistivity of the sample occurs at a temperature T_c^{onset}, which is close to 90 K. The authors, in particular, reported[3] a record value at $T = 0$ K, the second critical magnetic field H_{c2} of this compound, which they estimate could reach 180–200 T. Currently, these estimates are confirmed by direct measurements of this critical field of a single crystal of this compound.[4] Therefore, at a temperature of about 4 K in the direction along the field and perpendicular to the plane a–b were the values of the field of $H_{c2} = 240$ T and 128 T, respectively, achieved. This allows, in the future, the creation of a DC magnetic field of unprecedented magnitude that, in turn, would bring the technical revolution in the field of compact high-power electric machines, magnetic separators, transports based on magnetic levitation and electricity transmission lines without resistive energy losses.

For growing single crystals of HTSC with large dimensions (in particular, in the form of discs with a diameter of 10 mm to 150 mm and thickness up to 15 mm[6–8]) in most cases, the Czochralski method[5,6] is used. Figure 2 shows one such single crystal after a cycle of growing from a seed of the same composition and the same crystal structure.

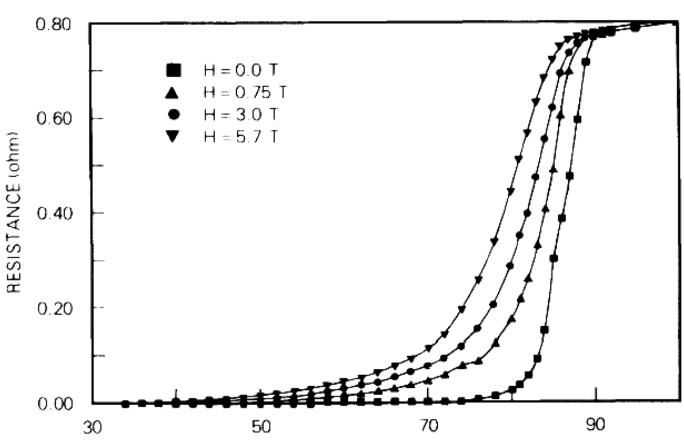

Fig. 1. First temperature dependences of the electrical resistivity of the ceramic sample at different values of external magnetic field with induction $H = 0 - 5.7$ T was published in 1987.[3]

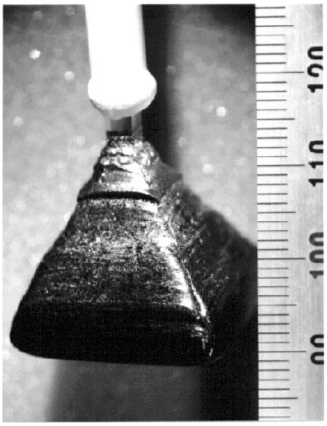

Fig. 2. One of the large single crystals having a size of 24×24 mm^2 in the "a–b" plane and 21 mm in the direction of the "c"-axis.[6]

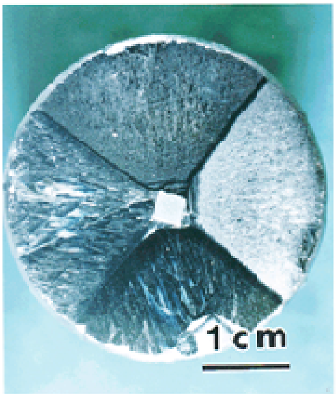

Fig. 3. Appearance of quasi-single crystal disk with typical domains in view off our sectors with the boundaries between them.[7]

In the pictures of a quasi-single crystal disk (Fig. 3), our domains in the form of the sectors can be seen clearly. Neighboring domains in the plane "a–b" are greatly misaligned and a place of a contact with each other forms the cross-domain boundaries. The direction c coincides with the axis of symmetry of the disk. In the center of the disk, there is a visible trace of the seed. Hitherto, it was assumed that the superconducting properties of such domains of the material are homogeneous and the critical temperature of transition to superconducting state T_c^{onset} is the same for any parts of such a sample. In this regard, one could expect that the dependence of the sample resistance R on temperature T in the Earth's magnetic field for any part of the crystal will be similar to the dependencies shown in Fig. 1.

The aim of this work is an experimental verification of this assumption.

2. Description of the Experiments

The appearance of the investigated disk is shown in Fig. 4.

The disc diameter is 30 mm and the thickness is 15 mm. The disk was made by the General Research Institute of Nonferrous Metals of China (Beijing).[9] Photos on the disk are clearly visible on four cross-domain boundaries.

In one of the main superconducting parameters of the disks, the repulsive force, F, directly from the magnet surface at temperature 77 K is 90 N. This force value means that the disk has good superconducting properties.

The next steps are about the preparation of microscopic samples of the material. First of all, a flat disk in the same diameter and with a thickness of 1 mm was cut off the big disk (Fig. 4) by a mechanical metal cutter. Figure 5 shows the optical micrograph of the disk surface.

The X-ray analysis of the disc material shows (Fig. 6) that this disk is a good single crystal with orthorhombical lattice, although, as shown in Fig. 5, the surface of the disk is heterogeneous.

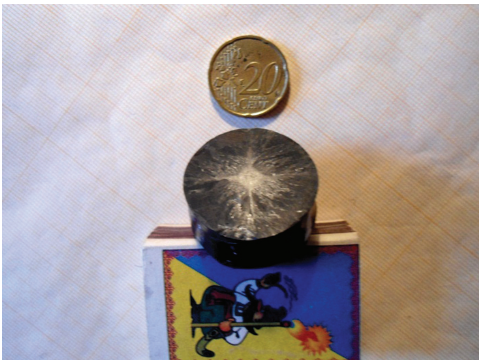

Fig. 4. External view of the $YBa_2Cu_3O_{7-x}$ quasi single crystal disk.

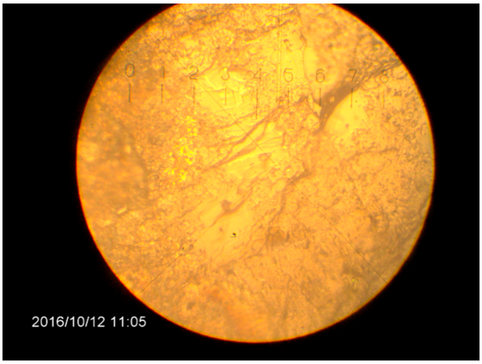

Fig. 5. Optical microscope picture (with diameter 3 mm) of the external view of the disk surface.

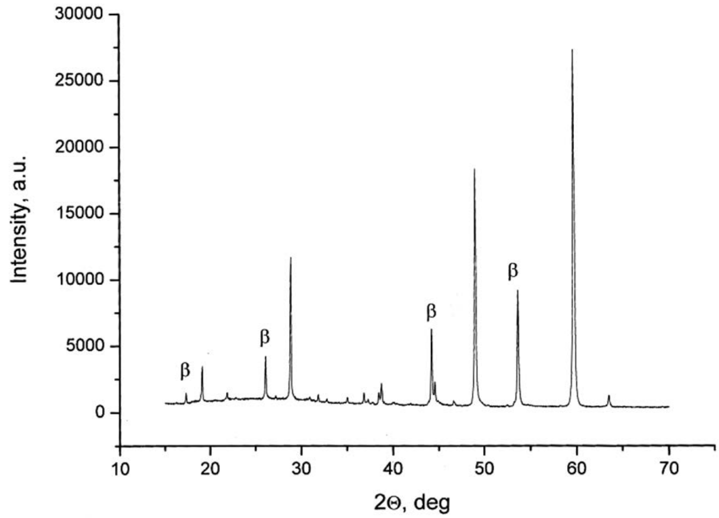

Fig. 6. X-ray diffraction picture of our $YBa_2Cu_3O_{7-x}$ disk.

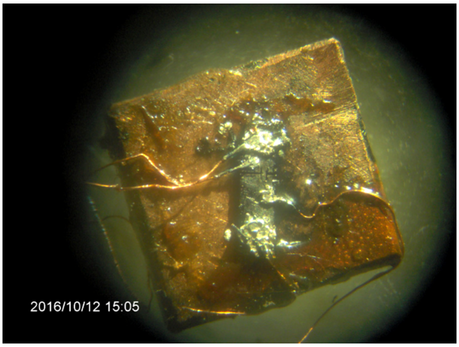

Fig. 7. Photo of one of the experimental samples in the view of micro-bridge on a copper substrate for the measurements of STT.

Structures in the form of micro-bridge were cut by the laser beam[10] of one domain of this disc. One of these bridges on the surface of a copper substrate is shown in Fig. 7.

A bridge connects the two “shores” of the disk material. The dark lines in the region of the structure with a width of about 30 μm represent places where the $YBa_2Cu_3O_{7-x}$ material was removed by laser beam. The width of the bridge is 160 μm and the length and thickness is 30 μm. The bridge is glued to the copper substrate measurements of the resistance bridge were carried out by the four-probe method, one can see the contact surfaces and the measuring conductors. The measurements were performed at the facility developed by and located in the Institute for Low Temperature Physics and Engineering of the National Academy of Sciences of Ukraine. The installation allows for the measurements of electric current–voltage characteristics of superconducting samples of various materials in weak magnetic fields and in the temperature range of 2–300 K. The measurement results are processed in a computer and reproduced on the monitor screen.

3. Results of the Experiments and Their Discussions

Figures 8 and 9 show the temperature dependence of the resistivity $R(T)$ of the two bridges, cut from two places of one domain of a disk, spaced from each other at a distance of about 2 mm.

Three sections of dependence $R(T)$ with different steepness are in the superconducting transition on first of them (Fig. 8). The middle section corresponding to the temperature interval 85–88 K is more gentle. We will call it a non-ordinary "step". The beginning temperature of the superconducting transition $T_c^{\text{onset}} = 93$ K corresponds to the value for bulk ceramics $YBa_2Cu_3O_{7-x}$, observed by most researchers. Full-width transition to the superconducting state is equal to 11 K. The bridge becomes fully superconducting at $T_{c0} = 82$ K.

Figure 9 shows the dependence $R(T)$ of the second bridge.

In Figs. 8 and 9, we can see a significant difference between temperature dependencies of the superconducting transition for bridges of different places of the

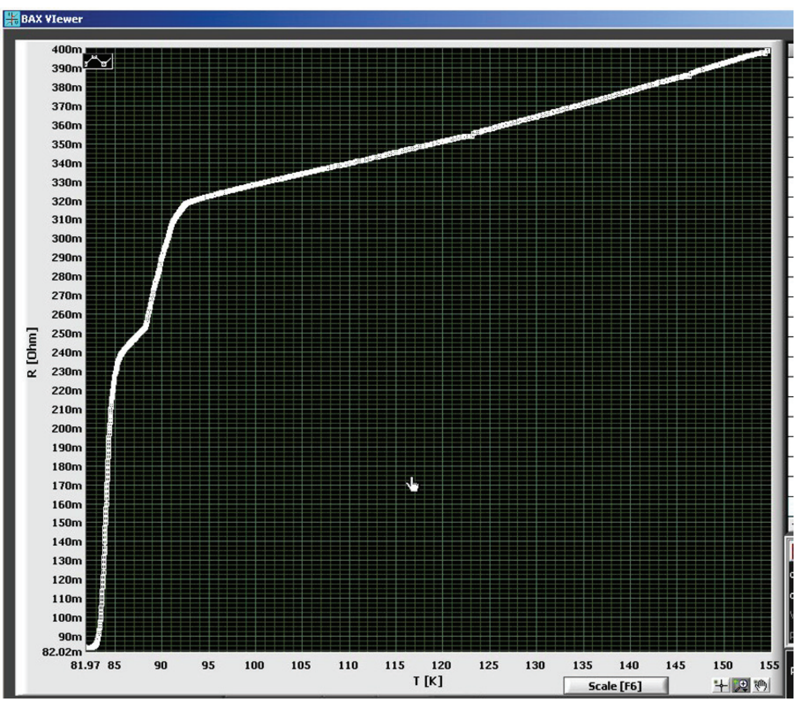

Fig. 8. Temperature dependence of resistivity of one of the bridges with $T_c^{\text{onset}} = 93$ K with non-ordinary "step" at $T = 88$ K.

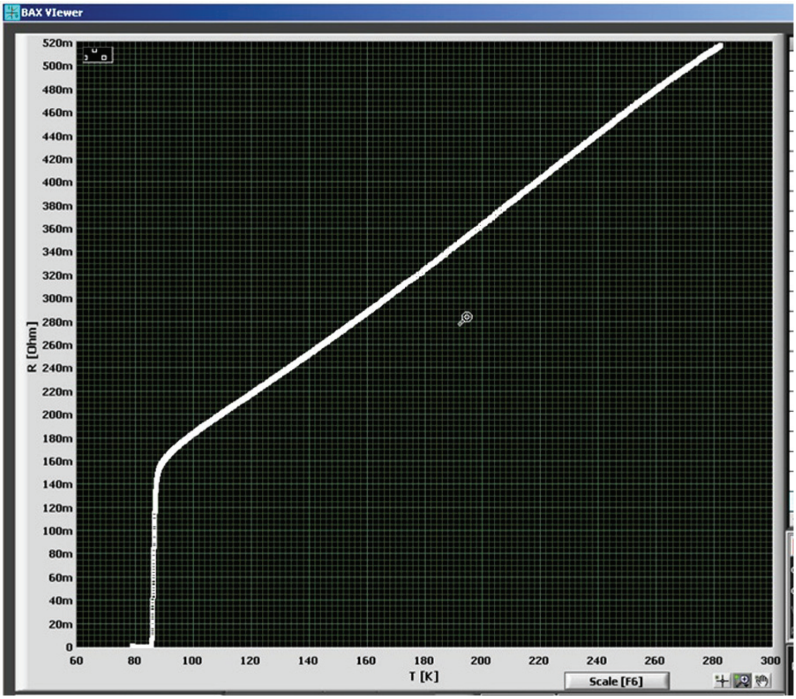

Fig. 9. Temperature dependence of resistivity of other bridge with $T_c^{\text{onset}} = 83$ K without the "step".

same disk. Dependence $R(T)$ in Fig. 9 is monotonous in contrast to Fig. 8 but it has $T_c^{\text{onset}} = 83$ K. It is 10 K lower than it was known earlier for bulk ceramics of $YBa_2Cu_3O_{7-x}$. The dependence width of the second bridge is substantially less and is located within 1–1.5 K. The bridge also becomes fully superconducting at $T_{c0} = 82$ K.

An explanation of the strange features of the dependences can be done if we inspect the structure and superconducting properties of the bulk YBa_2Cu_3 O_{7-x} crystal in the beginning. The bulk single crystal of the material has highest $T_c^{\text{onset}} = 93$ K if it has the orthorhombical structure of the crystal lattice, but it has no admixtures and a content of oxygen corresponding to the formula $YBa_2Cu_3O_{6.93}$. Modern superconducting bulk crystals of Y–Ba–Cu–O compound have a small quantity of special inclusions for creating different pinning centers, increasing critical current and critical magnetic field of the compound samples.

The difference in the dependences $R(T)$ of the two micro-bridges can be explained by the presence of micro-inclusions with different properties. First of all, other superconducting and non-superconducting phases (mainly hexagonally non-superconducting phase)[11] are the inclusions. The superconducting phase inclusions with less oxygen content have lower values of T_c^{onset}. The phases containing less than six atoms of oxygen in a molecule of this compound are non-superconducting.

The domains of large crystals consist mainly of the optimum phase and contain a small amount of inclusions of other phases. The predominance of high-temperature orthorhombical phase in the large volume of the bulk sample crystal is confirmed by the result of X-ray measurements (Fig. 6). As a result of the dependences, $R(T)$ of larger samples are monotonic and similar to dependence in Fig. 1 with $T_c^{\text{onset}} = 93$ K. The dependence of $R(T)$ of the sample can have more complex shape and a different value of T_c^{onset}, if the magnitude of the cross-section of the investigated sample in view of the micro-bridge is approximately equal to or less than the size of the inclusions. For the studied bridges that correspond to the cross-section of the bridge, $s = w \cdot t$. In our case $w = 1.6 \times 10^{-2}$ cm, $t = 0.3 \times 10^{-2}$ cm, then $s = 0.48 \times 10^{-4}$ cm^2 and the characteristic size $d = s^{0.5}$ is about $d \approx 0.7 \times 10^{-2}$ cm $= 70$ μm. Thus, the characteristic size of inclusions is tens of microns. The first bridge contains both the optimal compound phase (orthorhombical phase with optimal oxygen content) and low-temperature phase. The second bridge consists of the entire low-temperature phase. Structures with smaller characteristic size can largely differ in its properties when compared with the superconducting properties of the bulk crystal. Thus, the big crystals measured by Paul Chu[3] similarly have a monotonous dependence (RT) with $T_c^{\text{onset}} \approx 90$ K but their microscopic parts can have non-usual and non-monotonous dependence $R(T)$ with $T_c^{\text{onset}} \leq 90$ K. Therefore, one can find the sizes of crystal parts where there is the transition from the non-usual form of $R(T)$ to the usual monotonous form of a dependence $R(T)$ with $T_c^{\text{onset}} \cong 90$ K. An interesting aim of further research is to find the volume (v_g) of a material in which the dependence of $R(T)$ of the sample becomes independent of its location inside the crystal with big sizes. The value of v_g can be related to the distribution density of inclusions in the crystal.

The same temperature T_{C0} of achieving full superconductivity in the different bridges and the similar value of T_{c0} for the bulk sample in Fig. 1 may indicate that samples with different sizes of this big crystal will become fully superconducting at $T_{c0} = 82$ K.

4. Conclusion

We had investigated the temperature dependence of the resistivity $R(T)$ of the local microscopic parts of a bulk $YBa_2Cu_3O_{7-x}$ crystal domain for the first time and discovered that the form and parameters of the curves $R(T)$ of micro-bridges are strongly dependent on their locations in the crystal volume. In particular, the onset temperature of the superconducting resistive transition T_c^{onset} of one of these parts is 10 K below known T_c^{onset} of bulk samples at optimal oxygen doping ($T_c^{\text{onset}} = 93$ K). The strong temperature dependence can be explained by the crystal inhomogeneity in the form of microscopic inclusions of low-temperature and non-superconducting phases into the Y–Ba–Cu–O compound. An estimation of the inclusion sizes affecting the shape and parameters of the superconducting transition curves shows that they are equal to tenth microns in the crystal.

A comparison of the $R(T)$ dependences of microscopic parts of the investigated crystal and a bulk samples of this compound suggests that the temperature of the full transition of samples of this material in the superconducting state in a magnetic field of the Earth equals to $T_{c0} = 82$ K.

An actual task for further research of local properties of HTSC domains of crystals is to determine the minimal material volume, where the form of the $R(T)$ curve does not depend on its position in the bulk crystal.

The proposed method for the study of the $R(T)$ dependences of local microscopic parts of a high-temperature superconductor may be useful in improving the technology of production of bulk crystals to achieve the necessary superconducting parameters.

Acknowledgments

This work was funded by the Minstry of Science and Technology of China with the grant of 2015DFR0120. We are thankful to A. I. Prokhvatilov, A. S. Pokhila and V. P. Koverya for their help in measurements of the sample parameters.

References

1. J. G. Bednorz and K. A. Müller, *Z. Phys. B* **64**(1) (1986) 189.
2. J. D. Fan, *Endless Quests: Theory, Experiments and Applications of Frontiers of Superconductivity* (Peking University Press, China, 2014).
3. M. K. Wu, J. R. Ashburn, C. J. Torng, P. H. Hor, R. L. Meng, L. Gao, Z. J. Huang, Y. Q. Wang and C. W. Chu, *Phys. Rev. Lett.* **58** (1987) 908.
4. T. Sekitani, N. Miura, S. Ikeda, Y. H. Matsuda and Y. Shiohara, *Physica B* **346–347** (2004) 319.
5. Yu. D. Tretyakov, E. A. Gudilin, D. V. Perishkov and D. M. Itkis, *Prog. Chem.* **73**(9) (2004) 954.
6. X. Yao and Y. Shiohara, *Supercond. Sci. Tech.* **10** (1997) 249.
7. M. Murakami, N. Sakai, T. Higuchi and S. I. Yoo, *Supercond. Sci. Tech.* **9** (1996) 1015.
8. A. Murakami, H. Teshima, M. Morita and A. Iwamoto, *Physica C* **496** (2014) 44.
9. M. Zheng and Y. Jiao, General Research Institute of Nonferrous Metals of China (Beijing, China).
10. A. P. Zhuravel, A. G. Sivakov, O. G. Turutanov and I. M. Dmitrenko, *Appl. Surf. Sci.* **106** (1996) 321.
11. M. Murakami (ed.), *Melt Processed High-Temperature Superconductors* (World Scientific Publishing, Singapore, 1992).