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Research Article

Crystallization features of $YBa_2Cu_3O_{7-\delta}$ single crystals in $2YBa_4Cu_3O_{9-\delta} + BaCu_2O_2 + CuO_2$ system

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Abstract

In this article, a consistent study of phase transformations during the crystallization of $YBa_2Cu_3O_{7-\delta}$ compound was carried out using XRD, thermogravimetric and differential thermal analyzes, as well as optical microscopy. When studying the microstructure and elemental composition in the reaction zone in the process of obtaining single crystals by the crucible-less method, the products of chemical reactions were identified depending on the composition of the reacting components and synthesis conditions. It has been established that the use of precursors Y_2BaCuO_5, $YBa_4Cu_3O_{9-\delta}$ and $BaCu_2O_2$ as initial reagents has made it possible to carry out the direct synthesis of $YBa_2Cu_3O_{7-\delta}$ single crystals without the formation of intermediate phases. The superconductor has been synthesized at 1270 K on single-crystal MgO substrates with the (001) orientation, since their surface is poorly wetted by the melt solution and stimulates the $YBa_2Cu_3O_{7-\delta}$ nucleation process. This ensures the minimum loss of the liquid fraction formed in the sample. The growth conditions for $YBa_2Cu_3O_{7-\delta}$ single crystals have been studied and optimized. It has been experimentally revealed that the use of combined cooling conditions leads to an increase in the size of single crystals and a reduction in the time of their growth without changing the quality and crystal structure. The investigation showed that the largest volume (50 mm^3) was achieved for single-phase $YBa_2Cu_3O_{7-\delta}$ single crystals grown at a cooling rate of 0.5 deg/h in the temperature range 1260–1240 K and at a rate of 1.2 deg/h in the range 1240–1210 K. An analysis of the Laue rotation lines obtained in this work indicates the presence of blocks in single crystals cooled in the temperature range 1243–1193 K at a cooling rate of 1.5 deg/h and their absence in crystals cooled at 1.2 deg/h. An assessment of the degree of perfection of the structure by the width of the rocking curves at half-height of the X-ray reflection (006) showed that the width of the rocking curves of 0.36 deg indicates the absence of structural defects, such as twins, blockiness, and shear defects.

Keywords

high-temperature superconductivity, $YBa_2Cu_3O_{7-\delta}$ single crystals, oxygen nonstoichiometry, crucible-less synthesis method, thermogravimetric analysis, differential thermal analysis

1. Introduction

The advent of high-temperature superconductors (HTSC) with a transition to the superconducting state at temperatures exceeding the temperature of liquid nitrogen has opened up new possibilities for creating devices with unique characteristics. Such superconductors can function with simpler and more affordable cooling systems, instead of expensive equipment using helium. An extremely wide range of applications of HTSC materials is due to the absence of losses in direct current and small losses in alternating current, shielding of magnetic and electromagnetic fields, and the possibility of transmitting signals with minimal distortion [1].

Of greatest interest is the $YBa_2Cu_3O_{7-\delta}$ family, which transition temperature to the superconducting state (T_c) is about 93 K. These compounds have a number of properties such as, for example, a layered structure, electrically conductive copper-oxygen flat layers, and a pronounced anisotropy of electrical parameters [1–4]. For the practical use of $YBa_2Cu_3O_{7-\delta}$ materials, it is necessary to ensure high values of the critical current density (not less than $J_c \sim 10^4$ A/cm^2). Due to the anisotropy of conductivity and the small coherence length of yttrium-barium cuprate, defects associated with dislocations and stacking faults significantly reduce the critical current density J_c. Because of this, as confirmed by numerous studies, the use of the $YBa_2Cu_3O_{7-\delta}$ material is possible only for textured bulk products and single crystals [1–4].

An urgent problem in the field of high-temperature superconductivity remains the improvement of the technology for obtaining high-quality samples, including the $YBa_2Cu_3O_{7-\delta}$ compound, with reproducible superconducting properties and the investigation of their physico-chemical properties.

Currently, in order to obtain single crystals and textured $YBa_2Cu_3O_{7-\delta}$ ceramics, mainly the melt methods of synthesis are used. There are a fairly large number of technologies for obtaining textured ceramics and single crystals, $YBa_2Cu_3O_{7-\delta}$ using a liquid fraction. The basic methods for growing a textured compound $YBa_2Cu_3O_{7-\delta}$ are two main ones: MTG (Melt–Textured–Growth) – the method is based on the growth of textured ceramics from a molten initial charge of composition $YBa_2Cu_3O_{7-\delta}$ [2–5]; QMG (Quench–Melt–Growth) method is based on the growth of textured ceramics from a molten initial charge of $YBa_2Cu_3O_{7-\delta}$ composition with an additional stage consisting in melt quenching [2–4, 6, 7]. On their basis, all other methods for obtaining a high-temperature superconductor are formed [8–14].

However, their capabilities are limited by the high temperatures of the process (1300–1223 K), the high aggressiveness of the solution-melt, and the low growth rate (~10 μm/h) of $YBa_2Cu_3O_{7-\delta}$ crystals. Obtaining dense, textured ceramics and structurally perfect single crystals of yttrium-barium cuprate is difficult due to the peritectic nature of crystallization, the active interaction of the solution-melt with the material of technological equipment, the lack of oxygen in the liquid phase, the crystallization of satellite phases, etc. [2–7]. In this case, individual simple oxides, such as, for example, Y_2O_3 and BaO, form, upon interaction with other reagents, chemically stable refractory compounds Y_2BaCuO_5, $BaCu_2O_2$ and $BaCuO_2$. They do not completely react during the formation of $YBa_2Cu_3O_{7-\delta}$, and therefore are present in barium yttrium cuprate as separate inclusions, which significantly impairs its superconducting properties. In this regard, traditional methods for obtaining $YBa_2Cu_3O_{7-\delta}$, which use Y_2O_3, BaO, and CuO simple oxides, turned out to be inefficient [2–14]. The study of the sequence of phase transformations using Y_2BaCuO_5, $BaCu_2O_2$, and $BaCuO_2$ oxides as starting components can allow direct synthesis of $YBa_2Cu_3O_{7-\delta}$ without intermediate reactions.

Therefore, the search for new methods for obtaining single crystals and textured $YBa_2Cu_3O_{7-\delta}$ ceramics with a minimum content of impurities and having high physico-chemical characteristics is an urgent task.

2. Experimental

For the synthesis of the $YBa_2Cu_3O_{7-\delta}$ compound, precursors Y_2BaCuO_5, $Y_2Cu_2O_5$ and $BaCuO_2$ have been used, which were obtained from Y_2O_3, $BaCO_3$ and CuO oxides. The samples were prepared by conventional ceramic technology [16–19]. To remove crystallization moisture, the initial oxides were kept in a resistive thermal unit for 10 hours at a temperature of 573 K, barium carbonate at 1273 K. Mixing and grinding of the mixture of initial oxides with alcohol were carried out in a vibrating mill for 3 hours. The resulting mixture was dried at a temperature of 320 K until the alcohol was completely removed and pressed at 10 Pa into tablets 10 mm in diameter and 5 mm high. Pre-calcination was carried out in air at a temperature of 973 K for 18 hours. To increase the homogenization of the charge, secondary dry grinding was used in a PM 100 vibrating mill Retsch GmbH (Germany) for 2 hours and the resulting powder has been sieved. By sieving through a set of sieves with a given aperture size, powder fractions were obtained, consisting of grains with certain sizes. The powder was then compressed into tablets 12 mm in diameter and 5 mm thick. Samples were synthesized by heating in air to temperatures of 1223 K, 1273 K, and 1223 K for Y_2BaCuO_5, $Y_2Cu_2O_5$ and $BaCuO_2$, respectively, holding in a thermal device for 17 hours, followed by cooling in the switched off thermal device mode [18–20]. The temperature in the thermal set-ups has been maintained using a RIF-101 temperature controller and monitored using a Pt–Pt/Rh(10%) thermocouple with an accuracy of ±0.5 K.

The phase composition and crystal lattice parameters were determined by the Rietveld method using the ICSD-PDF2 database (Release 2000) and the PowderCell software [20] based on X-ray diffraction data obtained on

a DRON-3 set-up in CuK_α radiation. Diffractograms were taken at room temperature at a rate of 60 deg/h in the range of angles θ = 10–90°.

The powders were characterized by thermogravimetry (TGA) and differential thermal analysis (DTA) using a Setaram Labsys TG-DSC16 measuring complex at various heating rates in the range of 300–1300 K. The samples were kept until thermodynamic equilibrium with the gaseous medium has been established, and then cooled to room temperature in a continuous flow of a 5% H_2/Ar gas mixture. The sign of the achievement of thermodynamic equilibrium was the absence of a change in the mass of the sample at a fixed temperature of the samples. The weight of the samples was controlled by weighing with an accuracy of $\pm 3 \cdot 10^{-5}$ g.

Microstructure of the obtained samples has been investigated by the atomic force microscopy (NT-206 setup).

3. Results and discussion

Optimization of the composition of Y_2BaCuO_5 + $3BaCuO_2$ + xCuO samples, in which the maximum geometric dimensions of $YBa_2Cu_3O_{7-\delta}$ (S_{cryst}) crystallites are formed during superconductor synthesis, was carried out according to the data of X-ray phase and microstructural analysis.

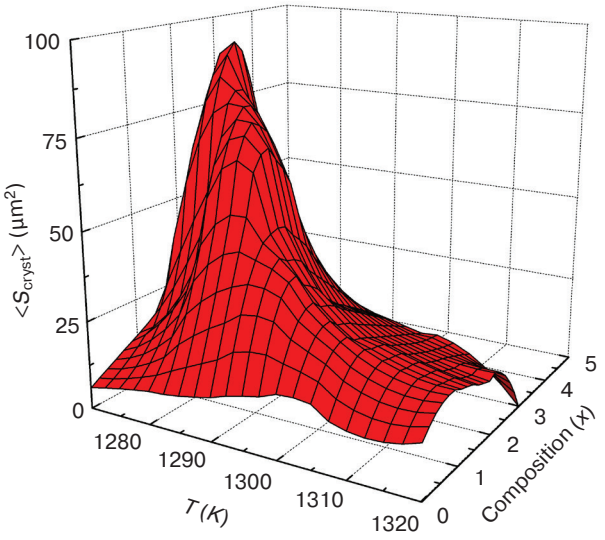


Figure 1. Dependence of the change in the area S_{cryst} of $YBa_2Cu_3O_{7-\delta}$ crystallites on the temperature of the beginning of synthesis (T) and the composition (x) of samples of the Y_2BaCuO_5 + $3BaCuO_2$ + xCuO system

It was found that as the sample synthesis temperature increased to 1320 K, the crystallite size increased, reaching a maximum value. With a subsequent increase in the synthesis temperature, the size of the crystallites decreased (Fig. 1) [16–18, 21]. From the graph of dependence $S_{cryst} = f(x)$ it was determined that samples with x = 0.6 had the maximum crystallite size.

Therefore, it has been established that at a cooling rate of 1 deg/h for samples of the Y_2BaCuO_5 + $3BaCuO_2$ + xCuO system from a temperature of 1305 to 1170 K, the largest fraction occupied by the maximum geometric dimensions of $YBa_2Cu_3O_{7-\delta}$ crystallites located in textured macrograins was observed in samples of the composition Y_2BaCuO_5 + $3BaCuO_2$ + 0.6CuO at a cooling rate of 1 deg/h from a synthesis temperature of 1320 to 1170 K [16–18, 21].

Optimization of the conditions for the growth of crystallites was carried out by studying the sequence of phase transformations in a mixture of compositions Y_2BaCuO_5 + $3BaCuO_2$ + 0.6CuO (a) at temperatures of the beginning of synthesis of 1305 and 1320 K, respectively, followed by cooling at a rate of 1 deg/h and quenching to room temperature.

Based on the data of TGA, DTA, XRD and microstructural analyzes for samples of composition (a) heated to T = 1320 K and cooled in the temperature range of 1320–1280 K with their subsequent quenching at room temperature, the presence of compounds Y_2BaCuO_5, $BaCuO_2$ and liquid phase has been confirmed (Table 1). Here, L is the liquid phase.

With a further decrease in the cooling temperature from 1280 to 1260 K, the content of the Y_2BaCuO_5 phase decreases, and the compound $YBa_4Cu_3O_{9-\delta}$ is found in the melt solution. In the lower cooling temperature range of 1260–1240 K, the solution-melt increases the intensity of reflections of the $YBa_4Cu_3O_{9-\delta}$ compound and the appearance of traces of $YBa_2Cu_3O_{7-\delta}$ [16–18, 21]. DTA fixes the presence of an exothermic effect, and TGA indicates an increase in the mass of the mixture with a decrease in temperature from 1260 to 1240 K (Fig. 2).

Based on these data, the formation reaction of the $YBa_4Cu_3O_{9-\delta}$ compound can be represented as: $Y_2BaCuO_5 + L + zO_2\downarrow \rightarrow 2YBa_4Cu_3O_{9-\delta}$ [16–18, 21, 22]. Intense crystallization of $YBa_2Cu_3O_{7-\delta}$ in the temperature range 1240–1210 K is accompanied by an

Table 1. Phase composition of Y_2BaCuO_5 + $3BaCuO_2$ + 0.6CuO samples heated to T = 1320 K and cooled to different temperatures followed by their quenching

T_{ann} (K)	Mixture of powders Y_2BaCuO_5 + $3BaCuO_2$ + 0.6CuO
1320–1280	Y_2BaCuO_5, $BaCuO_2$ and L (solution-melt)
1280–1260	Y_2BaCuO_5, $YBa_4Cu_3O_{9-\delta}$ (traces), L (solution-melt)
1260–1240	Y_2BaCuO_5, $YBa_4Cu_3O_{9-\delta}$, L (solution-melt) and $YBa_2Cu_3O_{7-\delta}$ (traces)
1240–1210	$YBa_2Cu_3O_{7-\delta}$, Ba_2CuO_3, $BaCuO_2$ and L (solution-melt)

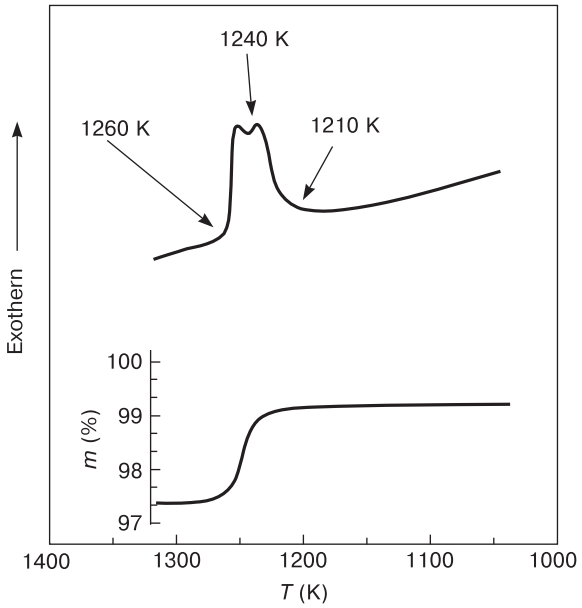


Figure 2. Temperature dependences of thermogravimetric and differential thermal analyzes of the system Y_2BaCuO_5 + $3BaCuO_2$ + 0.6CuO

exothermic effect without a change in mass. In this case, the growth of large single-crystal $YBa_2Cu_3O_{7-\delta}$ blocks occurs in a solution-melt far from the pores and sample surface (Fig. 3 *a*, *b*). Due to the fact that no change in the mass of the samples was found in this temperature range, the growth process of $YBa_2Cu_3O_{7-\delta}$ proceeds without oxygen absorption: $YBa_4Cu_3O_{9-\delta} + Y_2BaCuO_5 + L \rightarrow YBa_2Cu_3O_{7-\delta}$.

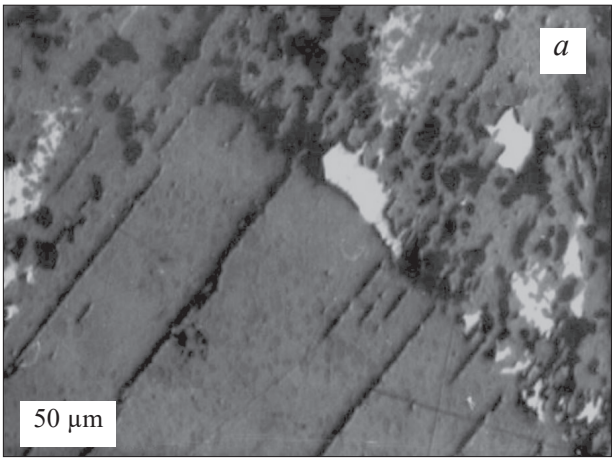
Thus, in a system with a high content of barium for the crystallization of $YBa_2Cu_3O_{7-\delta}$ the required amount of oxygen is supplied not only from the solution-melt, in which there is always its deficiency, but also from the dissolution of the solid phases $YBa_4Cu_3O_{9-\delta}$ and Y_2BaCuO_5. In this case, the absence of restrictions on the delivery of oxygen to the crystallization zone allows single crystals of the $YBa_2Cu_3O_{7-\delta}$ phase to increase their size constantly upon cooling.

For a mixture of the Y_2BaCuO_5 + $3BaCuO_2$ + 0.6CuO composition, the process of crystallization of $YBa_2Cu_3O_{7-\delta}$ occurs according to the peritectic reaction $Y_2BaCuO_5 + YBa_4Cu_3O_{9-\delta} + L \rightarrow YBa_2Cu_3O_{7-\delta}$ without oxygen uptake [16–18, 21–25]. The main absorption of oxygen falls on the crystallization period of $YBa_4Cu_3O_{9-\delta}$ in the cooling temperature range of 1260–1240 K.

To obtain single crystals, complex oxides Y_2BaCuO_5, $YBa_4Cu_3O_{9-\delta}$ and $BaCu_2O_2$ have been used as initial reagents, which made it possible to carry out direct synthesis without intermediate phases, eliminate the nonequilibrium of the $YBa_2Cu_3O_{7-\delta}$ crystallization process, control the dispersion and distribution of Y_2BaCuO_5 particles in the sample volume and, accordingly, increase the values of critical current densities dissipatively passing through textured $YBa_2Cu_3O_{7-\delta}$ ceramics.

In order to obtain $YBa_2Cu_3O_{7-\delta}$ single crystals with high J_c values, we used the initial Y_2BaCuO_5 particle size controlled powder, which after milling contained at least 70% Y_2BaCuO_5 grains with a size of $d_{av} \sim 10$ μm. After obtaining a homogeneous mixture of Y_2BaCuO_5, $YBa_4Cu_3O_{9-\delta}$, $BaCu_2O_2$ and CuO powders, pellets were formed at a pressing pressure of ~0.34 GPa using oleates.

Let us consider the features of growing $YBa_2Cu_3O_{7-\delta}$ single crystals by the crucible-less method [16–18]. In this case, the interest in growing single crystals of large geometric dimensions is primarily due to the possibility of obtaining high values of critical current densities and studying the conductivity anisotropy. Single-crystal MgO plates with the (001) orientation were chosen as the substrate on which the sample was placed, since their surface is poorly wetted by the melt solution, stimulates the nucleation of $YBa_2Cu_3O_{7-\delta}$, and ensures the minimum loss of the liquid fraction formed in the sample [9, 16–18, 24, 25]. The synthesis of single crystals in a pellet consisting of a mixture of $2YBa_4Cu_3O_{9-\delta}$ + $BaCu_2O_x$ + CuO_x powders and placed on a single-crystal MgO substrate was started at 1260 K after holding the pellet for 2 hours. To reduce the number of $YBa_2Cu_3O_{7-\delta}$ nuclei in the temperature range of 1260–1240 K, the sample was cooled at a rate of 0.5 deg/h. When cooling from T = 1240 K



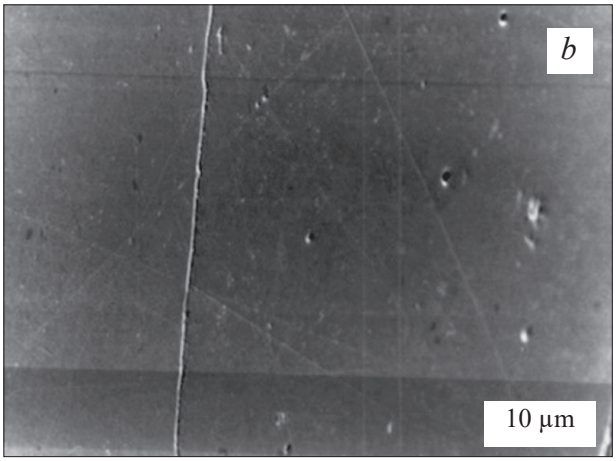


Figure 3. Microstructure of the reaction zone of samples of the Y_2BaCuO_5 +$3BaCuO_2$ + 0.6CuO system quenched from temperatures: (*a*) 1235 K, (*b*) 1200 K

Table 2. Effect of cooling rate on the maximum sizes of $YBa_2Cu_3O_{7-\delta}$ single crystals

Powders mixture	Cooling rate (deg/h)	Temperature range of cooling (K)	Maximal sizes of crystals (mm^3)
$2YBa_4Cu_3O_{9-\delta} + BaCu_2O_2 + CuO_2$	0.8	1240–1210	23.6
	1.0		37.4
	1.2		50.0
	1.4		39.2
	1.8		28.3
	2.5		13.1

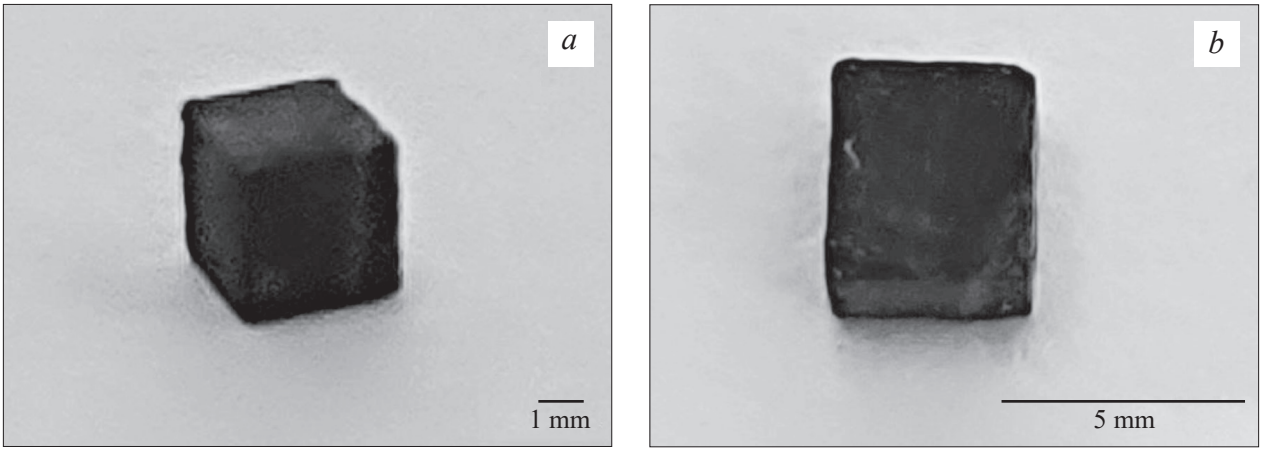


Figure 4. $YBa_2Cu_3O_{7-\delta}$ single crystals, obtained from the $2YBa_4Cu_3O_{9-\delta} + BaCu_2O_2 + CuO_2$ mixture at combined cooling rates: (*a*) υ = 1.2 deg/h, (*b*) υ = 1.5 deg/h

to $T = 1210$ K, the cooling rate has been increased to 1.2 deg/h (Table 2).

According to the microstructural analysis, it was found that single crystals had the largest volume ~50 mm^3 when cooled in the temperature range of 1240–1210 K at a rate of 1.2 deg/h (Fig. 4). Layer-by-layer XRD analysis and electron probe microanalysis revealed no inclusions of the melt solution and impurity phases in $YBa_2Cu_3O_{7-\delta}$ single crystals.

Layer-by-layer XRD analysis and electron probe microanalysis revealed no inclusions of the melt solution and impurity ions in $YBa_2Cu_3O_{7-\delta}$ single crystals. An analysis of the Laue rotation lines obtained in this work indicates the presence of blocks in single crystals cooled in the temperature range of 1243–1193 K at a cooling rate of 1.5 deg/h and their absence in crystals cooled at 1.2 deg/h (Fig. 5 *a*). An assessment of the degree of perfection of $YBa_2Cu_3O_{7-\delta}$ crystals cooled at a rate of 1.2 deg/h along the width of the rocking curves (*w*) and

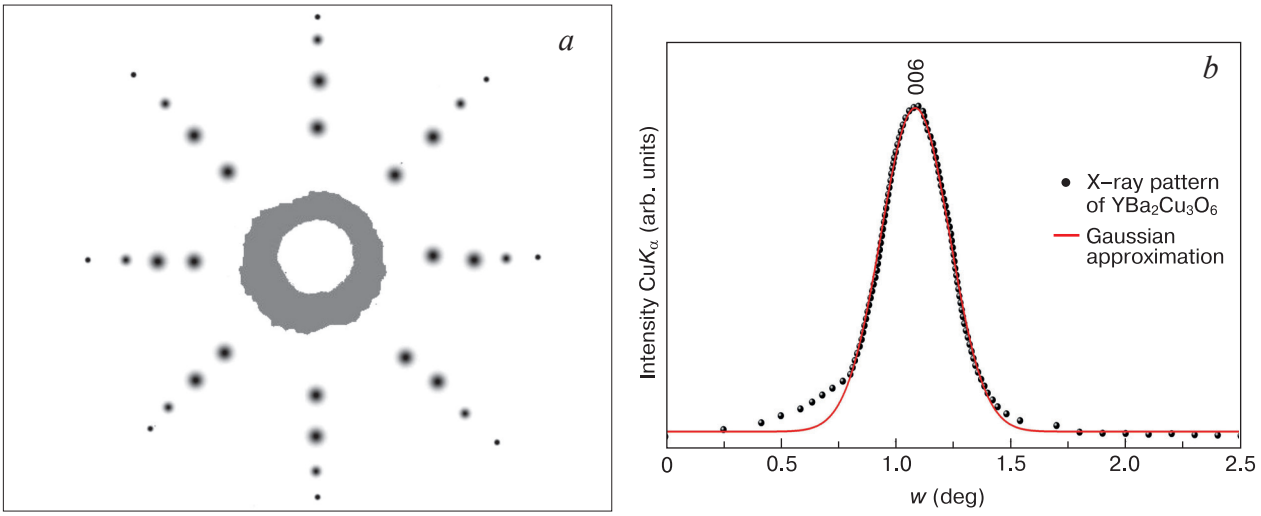


Figure 5. Laue rotation lines (*a*) and width of the X-ray reflection (006) at half-height of the rocking curve (*b*) of the $YBa_2Cu_3O_{7-\delta}$ single crystal

the half-height of the X-ray reflection (006) showed that the value w = 0.36 deg, indicating the absence of twins, blockiness, shear defects, and other structural defects (Fig. 5 *b*) [16–18].

An analysis of the Laue rotation lines indicates the presence of a block structure in single crystals cooled in the temperature range of 1240–1210 K at a cooling rate of 1.5 deg/h, and the absence of block structure at a cooling rate of 1.2 deg/h.

4. Conclusion

Therefore, it has been found that Y_2BaCuO_5, $YBa_4Cu_3O_{9-\delta}$ and $BaCu_2O_2$ precursors have been used as initial reagents to obtain $YBa_2Cu_3O_{7-\delta}$ single crystals. This ensured direct synthesis without intermediate phases. The synthesis of the $YBa_2Cu_3O_{7-\delta}$ superconductor at 1270 K was carried out on single-crystal MgO substrates with the (001) orientation, since their surface is poorly wetted by the melt solution and stimulates the nucleation process.

It was experimentally found that the use of combined cooling conditions has made it possible to increase the size of single crystals, as well as to reduce the time of their growth without deteriorating the quality and changing the crystal structure. It has been established that single-phase $YBa_2Cu_3O_{7-\delta}$ single crystals cooled in the temperature range 1260–1240 K at a rate of 0.5 deg/h, and in the range T = 1240–1210 K at a rate of 1.2 deg/h, had the largest volume ~50 mm^3.

Evaluation of the degree of perfection according to the Laue rotation lines, as well as the width of the rocking curves (w) at half-height of the XRD reflection (006) showed that the value w = 0.36 deg, indicates the absence of twins, blockiness, shear defects and other structural defects.

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