Crystallization features of YBa$_2$Cu$_3$O$_{7-\delta}$ in the Y$_2$BaCuO$_5$-BaCuO$_2$-CuO and Y$_2$Cu$_2$O$_5$-BaCuO$_2$ systems


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Based on the data of X-ray phase and microstructure analysis, the sample composition was optimized in order to provide maximum size of the textured macrograins of YBa$_2$Cu$_3$O$_{7-\delta}$ and of the crystallites in the Y$_2$BaCuO$_5$-BaCuO$_2$-CuO, Y$_2$Cu$_2$O$_5$-BaCuO$_2$ systems. The growth rate has been studied and the YBa$_2$Cu$_3$O$_{7-\delta}$ growth activation energy has been calculated for the samples of Y$_2$BaCuO$_5$+3BaCuO$_2$+2.3CuO, Y$_2$BaCuO$_5$+3BaCuO$_2$+0.6CuO, and Y$_2$Cu$_2$O$_5$+3.5BaCuO$_2$ compounds in the temperature range of 1240-1270K for the case of use of the Y$_2$CuO$_5$ and Y$_2$BaCuO$_5$ precursors with an average grain diameter of 10 μm and 1mm. A crystallization mechanism of YBa$_2$Cu$_3$O$_{7-\delta}$ in the Y$_2$BaCuO$_5$-BaCuO$_2$-CuO and Y$_2$Cu$_2$O$_5$-BaCuO$_2$ systems in the case of different sizes of Y$_2$BaCuO$_5$ and Y$_2$Cu$_2$O$_5$ precursor grains was proposed and validated.

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1 Introduction

Despite of the huge amount of work performed up to now [1], it is difficult to obtain high-quality ceramics and yttrium-barium cuprate (YBa$_2$Cu$_3$O$_{7-\delta}$) single crystals because of crystallization peritectic character, active interaction of solution-melt with material of technological equipment, lack of oxygen in the liquid phase, crystallization of satellite phases, etc. [2-4]. At that, some reagents such as Y$_2$O$_3$, BaO and CuO form the chemically stable refractory compounds, which do not react completely, and, thus, are present in yttrium-barium cuprate in the form of separate inclusions, significantly worsening its superconductive properties. Due to that, the traditional techniques of getting YBa$_2$Cu$_3$O$_{7-\delta}$ in which feeds containing simple oxides like Y$_2$O$_3$, BaO and CuO are used are not effective [5,6]. The use of feeds containing Y$_2$BaCuO$_5$ and Y$_2$Cu$_2$O$_5$ precursors provides the YBa$_2$Cu$_3$O$_{7-\delta}$ synthesis without reactions intermediate with oxygen change in solution-melt [5,7,8]. Since the YBa$_2$Cu$_3$O$_{7-\delta}$ crystallization rate and its mechanism depend on the grain size of Y$_2$BaCuO$_5$ and Y$_2$Cu$_2$O$_5$ precursors, the study of the grains dispersity on peculiarities of crystallization processes of yttrium-barium cuprate becomes significant.

This work is devoted to investigation of the influence of the Y$_2$BaCuO$_5$ and Y$_2$Cu$_2$O$_5$ precursor grain size on the crystallization rate and mechanism as well as on the microstructure of YBa$_2$Cu$_3$O$_{7-\delta}$ in the Y$_2$BaCuO$_5$-BaCuO$_2$-CuO and Y$_2$Cu$_2$O$_5$-BaCuO$_2$ systems.

2 Experimental

To obtain Y$_2$BaCuO$_5$, Y$_2$Cu$_2$O$_5$ and BaCuO compounds the Y$_2$O$_3$, BaO and CuO (99.99%) oxides were used. Oxides together with ethyl alcohol were grinded and mixed in vibrating mill for 3 h, dried at T=320 K and

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pressed into tablets. Preliminary annealing was carried out in air at \( T = 970 \text{ K} \) (BaCuO) and \( T = 1070 \text{ K} \) (Y\(_2\)BaCuO\(_5\), Y\(_3\)CuO\(_5\)) for 24 h. The second grinding was used in order to increase the homogeneity. Final anneal of oxide mixtures was carried out in air at \( T = 1240 \text{ K} \) (for BaCuO) and \( T = 1270 \text{ K} \) (for Y\(_2\)BaCuO\(_5\), Y\(_3\)CuO\(_5\)) for 24 h.

The furnace temperature was maintained by an RIF-101 precision temperature controller and monitored by Pt-Pt/Rh10 thermocouple with an accuracy up to ±0.5 K. X-ray radiometrical (XRA), X-ray phase (XRD) analyses were performed, and methods of electron microscopy, X-ray spectral microanalysis as well as optical microscopy were used to determine the mechanism of the YBa\(_2\)CuO\(_{7-\delta}\) single crystals formation. Growth kinetics of yttrium-barium cuprate is determined by the change of the normalized intensity of the (103) X-ray line of YBa\(_2\)CuO\(_{7-\delta}\) compound. For detachment of the Y\(_2\)BaCuO\(_5\) and Y\(_3\)CuO\(_5\) grains with average size \( d_{av} \sim 1 \text{ mm} \), the sieving separation was used. The furnace thus contained not less than 90% of grains of Y\(_2\)CuO\(_5\) and Y\(_2\)BaCuO\(_5\) with \( d_{av} \sim 1 \text{ mm} \). The fine grinding of the Y\(_2\)BaCuO\(_5\) and Y\(_3\)CuO\(_5\) furnace was carried out in a ball mill, using ethyl alcohol as the grinding medium. After fine grinding, the furnace contained grains of Y\(_2\)BaCuO\(_5\) and Y\(_3\)CuO\(_5\) with \( d_{av} \sim 10 \mu\text{m} \) in amount not less than 70%.

3 Results and discussion

The study of influence of the geometric dimensions of grains of Y\(_2\)BaCuO\(_5\) and Y\(_3\)CuO\(_5\) precursors on YBa\(_2\)CuO\(_{7-\delta}\) crystallization kinetics and mechanism was carried out on the samples with the following compositions: Y\(_2\)BaCuO\(_5\)-BaCuO-\(_2\)CuO and Y\(_3\)CuO\(_5\)-BaCuO\(_5\). Three systems have been investigated: (I) - Y\(_2\)BaCuO\(_5\)+3BaCuO\(_2\)+2.3CuO, (II) - Y\(_2\)BaCuO\(_5\)+3BaCuO\(_2\)+0.6CuO, (III) - Y\(_2\)CuO\(_5\)+3.5BaCuO\(_2\). A MgO monocrystalline plate oriented in the (001) plane was chosen as a substrate. MgO is poorly wetting by the solution – melt. It also stimulates the nucleation of the YBa\(_2\)CuO\(_{7-\delta}\) phase and provides minimum loss of the melt forming in a diffusion pair [9]. The composition of the samples synthesized in the temperature range 1270–1320 K and having maximum size of the textured macro grains and YBa\(_2\)CuO\(_{7-\delta}\) crystallites, was determined by the phase and microstructure analysis of the surfaces of five sections of each tablet [8]. For estimation of composition with the greatest size of both textured YBa\(_2\)CuO\(_{7-\delta}\) macrogains and crystallites, the samples were heated up to the temperatures 1270 < \( T < 1320 \text{ K} \) and after two hours they were cooled down with the rate of 1 K/h to the temperature of 1170 K. Below this last temperature, all the chemical processes are frozen [7]. The calculation of the normalized area \(<S_{m gran}>/<S_{max}>\), where \(<S_{m gran}>\) is the average area of the surface of five sections of the sample occupied by the textured YBa\(_2\)CuO\(_{7-\delta}\) macrogains, \(<S>\) is the average area of the total surface of five sections of the sample, was carried out based on results of the phase and microstructure analysis of the surface of the above mentioned five sections of the synthesized sample [8].

It was established that at synthesis of Y\(_2\)BaCuO\(_5\)+3BaCuO\(_2\)+xCuO samples with \( x = 0 \), \( x = 0.5 \), \( x = 1.0 \), \( x = 1.5 \), \( x = 2.0 \), \( x = 2.5 \), \( x = 3.0 \), with rising start temperature of the synthesis \( T_s \) from 1270 K to 1305 K with the step of 5 K, the normalized areas \(<S_{m gran}>/<S>\) and the average areas \(<S_{m gran}>\) increase, while for \( T_s \) from 1305–1320 K they decrease (Fig. 1a,b). From the plotted dependencies \(<S_{m gran}>/<S> = f(x, T)\) it was established that the highest values of \(<S_{m gran}>/<S>\) and of \(<S>\) was observed in compositions Y\(_2\)BaCuO\(_5\)+3BaCuO\(_2\)+2.3CuO and Y\(_2\)BaCuO\(_5\)+3BaCuO\(_2\)+0.6CuO, respectively (Fig. 1 a,b).

It was found that the highest value of the function \( f(y, T) = (<S_{m gran}>/<S>) \) where \( y \) was changed step-like, namely \( y_1 = 2.0 \), \( y_2 = 2.5 \), \( y_3 = 3.0 \), \( y_4 = 3.5 \), \( y_5 = 4.0 \), \( y_6 = 4.5 \), in the samples consisting of the phase mixture Y\(_3\)CuO\(_5\)+\(_{y}Y\(_2\)BaCuO\(_5\) corresponds to the composition Y\(_3\)CuO\(_5\)+3.5BaCuO\(_2\) (Fig. 2). It should be noted that for all compositions of the Y\(_3\)CuO\(_5\)+YBaCuO\(_2\) samples, after their synthesis, the structure of the textured macrogains was fine-grained, and, thus, the function \(<S_{c}> = f(y, T)\) was not built. Thus, based on the above described results, for the study of the kinetics and mechanism of the YBa\(_2\)CuO\(_{7-\delta}\) crystallization, the samples with the following compositions were chosen: (I) - Y\(_2\)BaCuO\(_5\) + 3BaCuO\(_2\) + 2.3CuO, (II) - Y\(_2\)BaCuO\(_5\) + 3BaCuO\(_2\) + 0.6CuO, (III) - Y\(_3\)CuO\(_5\) + 3.5BaCuO\(_2\).

Using the data of the change of the relative intensity of the (103) X-ray reflex, it was established that at the use of the Y\(_2\)CuO\(_5\) and Y\(_2\)BaCuO\(_5\) precursors with the average grain size \( d_{av} \sim 10 \mu\text{m} \), the growth rate of YBa\(_2\)CuO\(_{7-\delta}\) in the systems (I) to (III) can be well described by the parabolic dependence \( (I_{max})^2 = k(t-t_0) \), where \( k \) is the constant of growth rate of YBa\(_2\)CuO\(_{7-\delta}\), \( t_0 \) is the intensity of the (103) reflex measured through time intervals \( \Delta t = 1h \), \( I_{max} \) is the maximal intensity of the (103) reflex line.
YBa$_2$Cu$_3$O$_{7-\delta}$, $t_0$ is the time from the start of the furnace annealing to the appearance of YBa$_2$Cu$_3$O$_{7-\delta}$ in the furnace (Fig 3). It was established that the highest growth rate of YBa$_2$Cu$_3$O$_{7-\delta}$ was in the system (II). The values of the growth activation energy of YBa$_2$Cu$_3$O$_{7-\delta}$ were calculated by the formula $k = k_0 \exp(-E/RT)$, where $k_0$ is the constant of growth rate at $t = t_0$, R is the gas constant (8.31 J$\cdot$K$^{-1}$$\cdot$gram-molecule$^{-1}$). They are 31, 39 and 54 K/J$\cdot$m for the systems (II), (I) and (III), respectively.

Fig. 1 a) Plot of $\langle S_{mg} \rangle / \langle S \rangle$ vs $T$, $x - (Y_2BaCuO_5 + 3BaCuO_2 + xCuO)$ for YBa$_2$Cu$_3$O$_{7-\delta}$. B) Plot of $\langle S_{cryst} \rangle$ vs $T$, $x - (Y_2BaCuO_5 + 3BaCuO_2 + xCuO)$ for YBa$_2$Cu$_3$O$_{7-\delta}$.

Fig. 2 Plot of $\langle S_{mg} \rangle / \langle S \rangle$ vs $T$, $y - (Y_2CuO_5 + yBaCuO_2)$ for YBa$_2$Cu$_3$O$_{7-\delta}$.

Fig. 3 Plot of $(I/I_{max})^2$ vs time for YBa$_2$Cu$_3$O$_{7-\delta}$ in the systems (I) to (III) (a – grains size $<d>\sim 10 \mu$m, b – $<d>\sim 1$ mm).

To establish the causes of different values of YBa$_2$Cu$_3$O$_{7-\delta}$ growth rates in the systems (I) to (III) with the average grain size of Y$_2$BaCuO$_5$ and Y$_2$Cu$_2$O$_5$ $d_{av}\sim 10 \mu$m, let us consider the peculiarities of the crystallization mechanism of yttrium-barium cuprate in these systems. Based on the data of the local X-ray spectral microanalysis of the samples (I) - (III) tempered from $T=1240$-$1270$ K, it was established that the average concentration of yttrium ($<C_Y>$) in the solution-melt does not exceed 3 at. %. One may suppose that the maximal concentration of yttrium (max $C_Y$) will be on the border between the Y$_2$BaCuO$_5$, Y$_2$Cu$_2$O$_5$ grains and a solution-melt, and the heterogeneous nucleation and growth of YBa$_2$Cu$_3$O$_{7-\delta}$ should occur on the grain surfaces of the above mentioned solid phases [8,10]. However, during the investigation of the microstructure of the tempered samples, the formation of the YBa$_2$Cu$_3$O$_{7-\delta}$ compound was found to be not on the border between the grain surfaces and the solution-melt, but rather in the ellipsoid regions of the crystallized solution-melt (fig.4). In this case, the peritectic transformation reaction can be presented as: L + Y$_2$BaCuO$_5$ or (L + Y$_2$Cu$_2$O$_5$) $\rightarrow$ L + YBa$_2$Cu$_3$O$_{7-\delta}$ $\rightarrow$ YBa$_2$Cu$_3$O$_{7-\delta}$, L + Y$_2$BaCuO$_5$ $\rightarrow$ L + YBa$_2$Cu$_3$O$_{7-\delta}$ $\rightarrow$ YBa$_2$Cu$_3$O$_{7-\delta}$.
Y$_2$BaCuO$_5$+L $\rightarrow$ L $\rightarrow$ YBa$_2$Cu$_3$O$_7$-$\delta$. In the same conditions, in the II-system both the Y$_2$BaCuO$_5$ and the YBa$_4$Cu$_3$O$_9$-$\delta$ compounds were discovered. Since the content of Y$_2$BaCuO$_5$ and YBa$_4$Cu$_3$O$_9$-$\delta$ decreases during the crystallization of YBa$_2$Cu$_3$O$_7$-$\delta$, hence, the formation to the YBa$_2$Cu$_3$O$_7$-$\delta$ compounds should take place according to the scheme: L + Y$_2$BaCuO$_5$ $\rightarrow$ L + Y$_2$BaCuO$_5$ + YBa$_4$Cu$_3$O$_9$-$\delta$ $\rightarrow$ L + YBa$_2$Cu$_3$O$_7$-$\delta$ $\rightarrow$ YBa$_2$Cu$_3$O$_7$-$\delta$.

Thus, the YBa$_2$Cu$_3$O$_7$-$\delta$ crystallization in the (I)-(III) systems with the grain size of the Y$_2$BaCuO$_5$ and Y$_2$Cu$_2$O$_5$ precursors <d>~10 $\mu$m occurs in the solution-melt. In this case, the growth rate of YBa$_2$Cu$_3$O$_7$-$\delta$ in the systems (I), (III) is limited by the Y intake speed from the Y$_2$BaCuO$_5$ grains to the YBa$_2$Cu$_3$O$_7$-$\delta$ growing grains through the liquid, because of the relatively low value of <C$_Y$> ~ 3 at.% in the solution-melt. In the system (II), the growth rate will be limited by the Y intake speed also from the grains of the intermediate phase YBa$_4$Cu$_3$O$_9$-$\delta$ additionally. The obtained experimental results coincide well with the theory.

It is known that density of yttrium (J$_Y$) flow to the YBa$_2$Cu$_3$O$_7$-$\delta$ grains surface is proportional to the gradient of its concentration ($\Delta$C$_Y$) in the liquid phase, which can be conditionally divided into two areas with various concentration Y [3]. The first area (C$_{Y1}$) is located on border of L/Y$_2$BaCuO$_5$ Y$_2$BaCuO$_5$/L grains. The second area (C$_{Y2}$) is located on border of L/YBa$_2$Cu$_3$O$_7$-$\delta$ YBa$_2$Cu$_3$O$_7$-$\delta$/L grains. Suppose that yttrium concentration in a solution-melt is a constant (C$_{Yo}$). It is well known (Fick's first law) that substance stream from unit of Y$_2$BaCuO$_5$ grains surface is described as: J$_{Y1}$ = k$_1$D$_{Y1}$/$\delta_1$A$_{C_1}$, where D$_{Y1}$ is the chemical diffusion-factor of yttrium through boundary layer, $\delta_1$ is the thickness of boundary layer between Y$_2$BaCuO$_5$ grains surface and conventional border outside of which the solution-melt structure is constant. At crystallization YBa$_2$Cu$_3$O$_7$-$\delta$ the gradient stream on unit of crystal surface makes J$_{Y2}$ = k$_2$D$_{Y2}$/$\delta_2$A$_{C_2}$, where D$_{Y2}$ is the chemical diffusion-factor of yttrium through boundary layer, $\delta_2$ is the thickness of boundary layer between Y$_2$BaCuO$_5$ crystals surface and conventional border outside of which the solution-melt structure does not change. Streams J$_{Y1}$ and J$_{Y2}$ are equal in (I) and (III) systems at quasi-equilibrium conditions of YBa$_2$Cu$_3$O$_7$-$\delta$ crystallization. The growth rate equation can be presented as: v$_{2(cryst.)}$ = v$_{(dec.)}$A$_{C_2}$/A$_{C_1}$, where v$_{2(cryst.)}$ = kD$_{Y2}$/$\delta_2$ is the growth rate
of YBa$_2$Cu$_3$O$_{7.6}$, $v_{\text{dec.c}} = k_{\text{D}/L}/\delta$ – is the decomposition rate of YBa$_2$Cu$_3$O$_{7.6}$ grains. Growth rate of YBa$_2$Cu$_3$O$_{7.6}$ is characterized by yttrium streams from YBa$_2$CuO$_5$ – (J$_3$), YBa$_2$Cu$_3$O$_{8.4}$ – (J$_4$) grains through a phase liquid to YBa$_2$Cu$_3$O$_{7.6}$ surface in (II) system. The growth rate equation of YBa$_2$Cu$_3$O$_{7.6}$ can be formulated as: $v_{\text{cryst.c}} = \Delta C_2[\delta_{\text{3}}/\Delta C_3 + \delta_{\text{4}}/\Delta C_4]$, where $v_{\text{3}}$, $v_{\text{4}}$ is the decomposition rate of Y$_2$BaCuO$_5$ and YBa$_2$Cu$_3$O$_{8.4}$, $\Delta C_3$, $\Delta C_4$ – is the gradient of yttrium concentration on section border of L/YBa$_2$CuO$_5$ Y$_2$BaCuO$_5$/L and L/YBa$_2$Cu$_3$O$_{9.4}$/YBa$_2$Cu$_3$O$_{8.4}$/L. Thus, growth rate of YBa$_2$Cu$_3$O$_{7.6}$ in (I), (III) systems is determined rate of Y$_2$BaCuO$_5$ grains decomposition and supersaturation degree on interface section a solid phase. Growth rate of YBa$_2$Cu$_3$O$_{7.6}$ in (II) system was determined rate of decomposition of YBa$_2$Cu$_3$O$_{8.4}$ grains additionally. Growth rate of YBa$_2$Cu$_3$O$_{7.6}$ in (I), (III) systems was determined Y mobility in solution-melt at absence of artificial homogenization. The greatest growth rate of YBa$_2$Cu$_3$O$_{7.6}$ in (II) system is caused by two streams of Y to a crystal surface. Formation and subsequent decomposition of YBa$_2$Cu$_3$O$_{9.4}$ provides, in our opinion, chemical homogenization of reagents in a solution-melt. In (III) system formation and growth of YBa$_2$Cu$_3$O$_{7.6}$ grains occurs, as well as in (I), but at greater growth rate that can be explained by the chemical homogenization caused by peritectic reaction of L + Y$_2$Cu$_3$O$_5$ → L + YBa$_2$Cu$_3$O$_{7.6}$ → YBa$_2$Cu$_3$O$_{7.6}$, Y$_2$CuO$_3$ + L → Y$_2$BaCuO$_5$. At increasing size of the grains of Y$_2$BaCuO$_5$ and Y$_2$CuO$_3$ and Y$_2$BaCuO$_5$ up to $d_{\text{av}}$ ~ 1 mm, the change of the growth rate of YBa$_2$Cu$_3$O$_{7.6}$ in the systems (I)-(III) can be divided into two stages (α, β), which can be described by the quasi-linear and parabolic dependencies, respectively (Fig. 3).

The greatest growth rate of YBa$_2$Cu$_3$O$_{7.6}$ was observed in the (I) system. Growth activation energy of YBa$_2$Cu$_3$O$_{7.6}$ increased up to 183, 197 and 201 kJ/mol at the second growth stages for (I), (III) and (II) systems accordingly. To explain these peculiarities, the following additional experiment was carried out. The polycrystalline high-density ($\rho$–0.94$\rho_{\text{cm}}$) tablets of Y$_2$BaCuO$_5$ and Y$_2$CuO$_3$ 8 mm in diameter and 3 mm thick were immersed in L(BaCu$_{1.2}$O$_5$), L(BaCu$_2$O$_3$), and L(BaCu$_3$O$_5$) liquid at 1240-1290 K for 2 h. Then the tablets were cooled with the rate of 1 K/h down to the different temperatures in the range of 1240-1290 K, with the subsequent tempering. It was established by the X-ray spectral and X-ray radiometrical (XRA) analyses that Y was absent in liquid phase at 1240-1270 K, but the Y was detected in the vicinity of the Y$_2$BaCuO$_5$ and Y$_2$CuO$_3$ tablets at 1270-1290 K. Microstructure analysis showed that Y$_2$BaCuO$_5$ tablet tempered from melt L(BaCu$_{1.8}$O$_3$) at 1240-1270 K has a textured layer of YBa$_2$Cu$_3$O$_{7.6}$ (Fig. 5).

Fig. 5 Diffusion layers of Y$_2$BaCuO$_5$ + L (BaCu$_{1.8}$O$_3$)

Fig. 6 Diffusion layers of Y$_2$BaCuO$_5$ + L (BaCu$_{1.8}$O$_3$) system.

At the use of L (BaCu$_{1.8}$O$_3$), a textured layer YBa$_2$Cu$_3$O$_{9.4}$, was formed initially on the tablet of Y$_2$BaCuO$_5$, then the YBa$_2$Cu$_3$O$_{9.4}$ was formed (Fig. 6). The YBa$_2$Cu$_3$O$_{9.4}$ and YBa$_2$Cu$_3$O$_{8.4}$ crystallites were grown perpendicularly to the frontal surface of the sample. For the Y$_2$CuO$_3$ tablet placed into BaCuO$_5$ melt at $T=1270$ K, the sequence of crystallization of layers Y$_2$CuO$_3$/Y$_2$BaCuO$_5$/YBa$_2$Cu$_3$O$_{7.6}$/L was established (Fig. 7).
During the analysis of the growth rate of yttrium-barium cuprate in systems (I), (II) and (III), where the peculiarities of the crystallization of yttrium-barium cuprate at using of peritectic reaction from the intermediate phase 

At increasing grain sizes of solid phases of 

Hence use of firm phases grains of 

Conclusions

1. To obtain the yttrium-barium cuprate with large sizes of textured macrograins and crystallites the following compositions of furnace have been proposed: 

2. The peculiarities of the crystallization of yttrium-barium cuprate at using of peritectic reaction from the furnace of systems (I), (II) and (III), containing precursors 

3. During the analysis of the growth rate of yttrium-barium cuprate in systems (I), (II) and (III), where the average grain size of solid phases of 

4. At increasing grain sizes of solid phases of 

5. By the analysis of the microstructures of hardened samples it was established that the mechanisms of crystallization of yttrium-barium cuprate in systems (I) (II), and (III) have differences at different sizes of 

Fig. 7 Diffusion layers of 

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