https://doi.org/10.31489/2024PH3/61-70
UDC 538.945(045)

Received: 17.05.2024
Accepted: 29.06.2024

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Critical current density of high-temperature superconducting ceramics BSCCO Bi-2223

In the paper the results of the study on the synthesis of high-temperature superconducting ceramics of nominal composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ by various methods were presented based on amorphous phases using glass-ceramic technology and solid-phase method. For comparison, amorphous phases were obtained in two ways. In the first case, a heating furnace of a special design was developed to obtain an amorphous phase, which provides melting without using a crucible. The heating of the initial samples for melting is carried out due to the combined effect of the convection heat flux and the radiation of heating elements, which consists of the IR region of the spectrum at a melting temperature in the spectral range of 1300–1350 nm. In the second case, melting is carried out under the influence of broadband optical radiation, including UV, visible and IR spectral regions. The production of glassy precursors is carried out by draining the melt onto a quenching device in the form of a propeller made of stainless steel. Studies of the formation rate of the superconducting high-temperature phase Bi-2223 were carried out in the same temperature conditions at 848–850 °C with intermediate grinding every 24 hours and the study of the phase composition by X-ray diffraction method. Studies showed that the glass phase-based method ensures the completeness of the formation of the high-temperature phase Bi-2223 and the rate of its formation is significantly higher than by the solid-phase method (2.5–3 times). Studies of the critical density of the transport current have shown that the current value is 7.05×10^3 mA/cm^2, (measured by the criterion of 1 μV/cm), which is significantly higher compared to other methods.

Keywords: superconductivity, glass phase, microstructure, ceramics, IR radiation, diffractogram, melt, crystallization.

Introduction

High-temperature superconductors (HTS) are one of the promising materials used in various advanced areas of industry, science and technology. Their field of application covers such diverse areas as energy, electronics and communications, space technology, medicine, metallurgy, instrument engineering and many others. To date, a small group of HTS systems was developed, in which metal cuprates are the basis [1–3]. Among them, the following superconducting compounds are actively used in practice — compounds based on bismuth $(Bi, Pb)_2Sr_2CaCu_2O_8$ (Bi-2212) and $(Bi, Pb)_2Sr_2Ca_2Cu_3O_{10}$ (Bi-2223), as well as yttrium $YBa_2Cu_3O_{7-x}$ (Y123). It is known that the main parameters of HTS materials are critical temperature, critical current and critical magnetic field. The main factors determining the parameters of these HTSPS are: the creation of the necessary phase composition, increased textures, densities, the presence of pinning centers and others, which depend on the methods and technological conditions for their production.

Currently, various methods are used for the synthesis of HTSP — they are widespread: solid-phase synthesis, chemical and cryochemical, as well as various melt methods, etc [4–7]. The results of numerous studies show that obtaining the required critical parameters is not an easy task. A definite solution to the problem of creating HTS materials of a given phase composition with the necessary critical parameters (especially current ones) is the use of a glass-ceramic method. This method has certain advantages over others and allows you to obtain high-density ceramics with the required morphology (texture) and controlled grain size. In [8–11], the authors obtained, using the glass-ceramics method based on amorphous precursors, HTSC samples with increased density (the density value reached closer to the theoretical 99.5 % and higher), as well as with a high crystallite texture (97–98 %) according to direction of the crystal plane [00l].

The glass-ceramic method can only be used for compositions having glass-forming properties during melt quenching, which is characteristic of the bismuth system. The method was first developed in the 1990s, but we were unable to evaluate its advantages over the solid-phase method due to the not fully elucidated processes during crystallization from the amorphous state of the phase with high Tc (Bi-2223). The synthesis of bismuth-based HTSP has been carried out by many researchers [12–18]. The analysis of these studies

showed that if the synthesis of the low-temperature superconductor Bi-2212 using glass-ceramic technology is not very difficult, then the synthesis of the high-temperature single-phase Bi-2223 based on glass phase is not an easy task. Traditionally, to obtain glassy precursors, the melting of the initial charge is carried out in a crucible made of corundum or platinum. During melting, there is a violation of the stoichiometric composition, especially significantly in oxygen (oxygen vacancy), as a result, oxygen deficiency negatively affects the kinetics and dynamics, as well as the completeness of the formation of a superconducting high-temperature phase 2223. The generalized results of the work on glass-ceramic technology were discussed in detail in the work of Abe [19], which is also noted about the lack of oxygen. In this case, cations of variable valence are reduced to a low-valent state, especially copper $Cu^{++} \rightarrow Cu^{+}$ [20, 21]. Compensation of oxygen deficiency during thermal annealing during the formation of HTS is difficult, since the rate of oxygen diffusion in dense structures of glassy precursors is very low, therefore long annealing (150–400 hours) is required. In this regard, the use of a method for producing bismuth-containing superconductors based on a glass phase that does not lead to a significant violation of the component composition (especially oxygen) could positively affect the completeness of the formation of the high-temperature superconducting phase Bi-2223, as well as a significant increase in the critical current density and shorten the synthesis time.

In this regard, this paper considers a method for obtaining amorphous precursors without using a crucible and synthesizing high-temperature superconducting ceramics based on them and studying their critical parameters.

The purpose of this work was to study the formation of a superconducting high-temperature phase Bi-2223 and the critical parameters of superconducting ceramics of nominal composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ synthesized on the basis of a glass phase using an optical light flux and the study of their critical parameters.

Experimental

For the synthesis of HTSP, reagents of the "extra clean" brand with a content of the main component of at least 99.9 % were used: Bi_2O_3, PbO_2, $SrCO_3$, CaO and CuO. Before preparing the mixture of components, the reagents were calcined at a temperature of 120 °C for 2 hours.

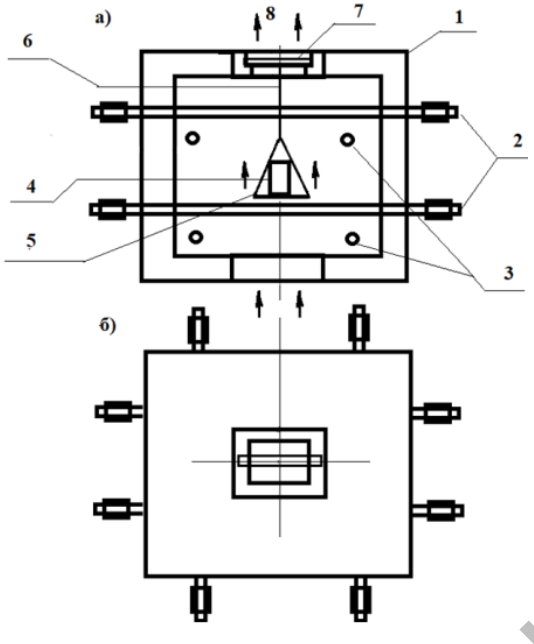
The phase compositions of superconductor samples were determined using Bruker D8ADVANCEECO and XPertPRO X-ray diffractometers (Netherlands). Microstructural and elemental analyses of the samples were carried out on scanning electron microscopes JEOL-6490LA (Japan) with an energy dispersion analyzer system "OXFORD Instruments Analytikal Limited" (Great Britain) and JSM-6390LV (Japan) with an integrated energy dispersion X-ray analyzer (EDS) (acc. voltage — 20 kV, probe current — 1.0 nA). The critical temperature and the density of the critical transport current of the sulfur-conducting samples were measured by a four-point probe method using a CryoIndustry REF-1808-ACS cryocamera cooled with helium gas, a LakeShoreModel 340 temperature meter and a microvoltmeter. The density of the critical transport current (Jc) was determined by the criterion of 1 μV/cm. The critical temperature was measured, also by a non-contact method, by studying the temperature dependence of magnetic susceptibility. This method is highly sensitive and can detect traces of HTS phases with higher Tc in superconducting samples.

Results and discussions

The synthesis of HTSP composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ was carried out in three ways. Synthesis based on glassy precursors obtained in a specially designed resistive furnace, which provides melting of a mixture of samples in the form of tablets without using a crucible and simultaneous quenching of the melt by gradually draining the melt into a quenching device. The general diagram of the device for producing glassy precursors from a melt is shown in Figure 1.

A platinum substrate was used as a support for the initial samples. The melting of the samples was carried out due to the influence of a thermal convection flow and under the influence of an IR radiant flux in the wavelength range of 1300–1350 nm, which is emitted from heating elements at a temperature of 1200–1250 °C. The distance of the sample from the heating elements is 10–15 mm (the method of melting by resistive heating).

For comparison, there is also a synthesis based on glassy precursors obtained by melting the initial mixture under the influence of a radiant flux (electric arc lamp) and simultaneous quenching of the melt by flowing onto the rotating propeller of the quenching device. The spectral composition of the radiant flux consists of intense continuous radiation from the ultraviolet, visible and infrared regions of the spectrum. In addition, the IR region of the spectrum, along with continuous radiation, contains intense linear radiation in the wavelength range from 800 to 1000 nm (a method of melting by a radiant flux).



a) sectional melting devices: *1* — insulating housing; *2* and *3* — heaters; *4* — initial workpiece; *5* — substrate (platinum) with workpiece; *6* — substrate holder; *7* — ceramic rod for hanging the substrate; *8* — convection air flow; b) top view of the melting device

Figure 1. A device for producing initial glassy precursors by melt quenching

Also, the synthesis of HTSP was carried out according to the traditional solid-phase method by heat treatment of samples in the form of tablets in an isothermal mode at a temperature of 850 °C with intermediate grinding in a planetary mill every 24 hours.

The synthesis of HTSP based on glassy precursors was carried out, subsequently, according to the solid-phase synthesis mode, which is described above according to the scheme: grinding – pressing – thermal annealing.

For the synthesis of HTS ceramics based on glass phase, glassy precursors were previously obtained using the two methods described above. Visually, the precursors did not differ in appearance. Basically, they consisted of plates with a glassy luster (Fig. 2).

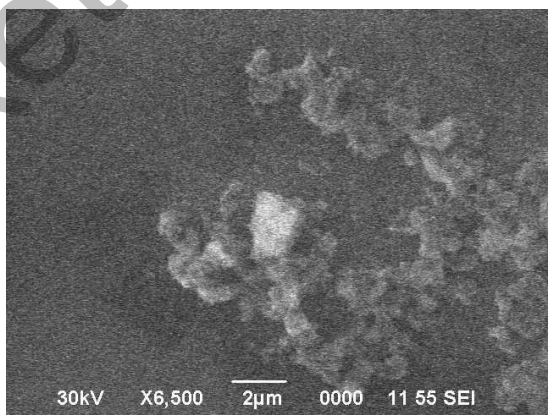
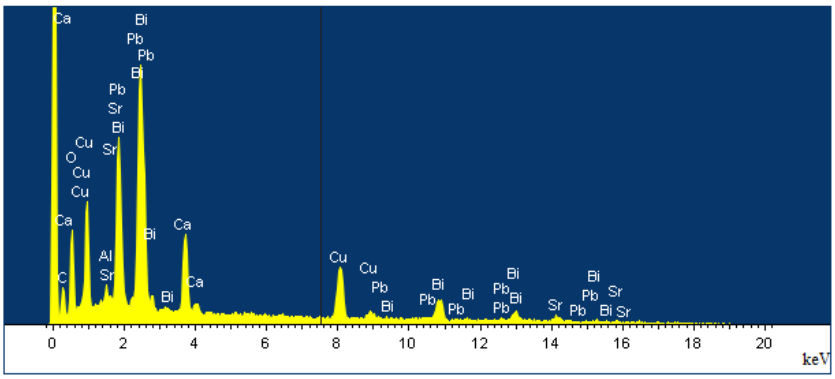
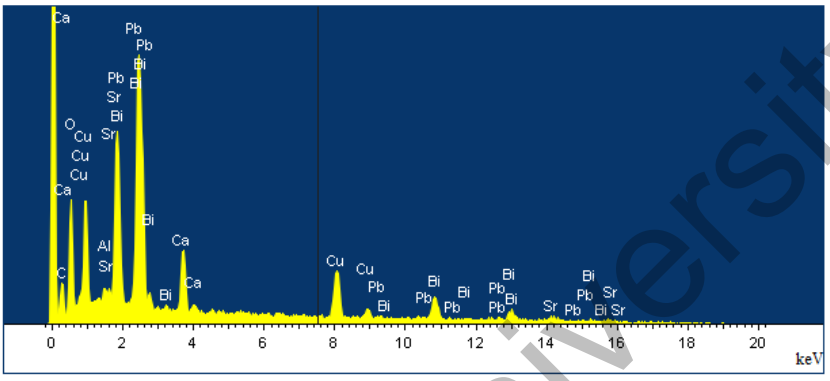


Figure 2. Microstructure of precursors of plates of the composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ obtained by quenching the melt by melting by resistive heating without using a crucible

The glassy appearance of the precursors was confirmed by X-ray studies, the absence of peaks on diffractograms. The study of the elemental composition of glassy precursor samples showed a slight deviation from the stoichiometric composition, i.e., a decrease in the content of calcium and lead was observed (Fig. 3, Table).



a)



b)

a — spectrum of a plate obtained in a furnace; *b* — spectrum of a plate obtained by a radiant flow

Figure 3. Energy-dispersive spectral electron microscopy studies of plate samples obtained by ultra-fast quenching of the melt

Table

Elemental composition of precursors

Name of the samples	O	Ca	Cu	Sr	Pb	Bi	Sum
Melted in the oven, wt.%	22.72	5.25	17.22	16.43	5.15	33.23	100
Fused by a radiant stream, wt.%	24.34	3.96	16.88	16.51	4.08	34.23	100

In the samples obtained by melting with a radiant flux, the deviation is slightly greater. The large decrease in the calcium and lead content in the samples obtained using the radiant flux is apparently related to the increased melt temperature associated with the anisotropic, one-sided effect of the radiant flux. Visually, it can be observed that the surface of the molten sample reaches the boiling point and above. The temperature of the boiling melt (sample surface), as measured by a pyrometer, exceeds 1500–1600 °C. The melting time of the surface of the initial samples under the action of a radiant flux is no more than 5 seconds. As for the melting temperature mode of the initial samples in a device with resistive melting, it can be strictly controlled with a difference of up to 2-3 degrees. At a melting temperature of 1250 °C, the melt begins to drip off in the form of droplets about 10 seconds after loading the initial samples inside the melting device onto a platinum substrate. Therefore, no significant change in the cationic composition was observed. An increase in oxygen content was observed in all precursor plates, since in both methods the melting processes are carried out in an oxidizing atmosphere.

The synthesis of superconducting phases was carried out according to the above technological regime. Previously, the precursors of the plate were annealed at a temperature of 750–800 °C for 10–12 hours for recrystallization. After that, they were crushed in a planetary mill and prepared samples in the form of tablets with a diameter of 15 mm and a thickness of 2.0–2.5 mm by pressing at a pressure of 6000 kgf/cm^2.

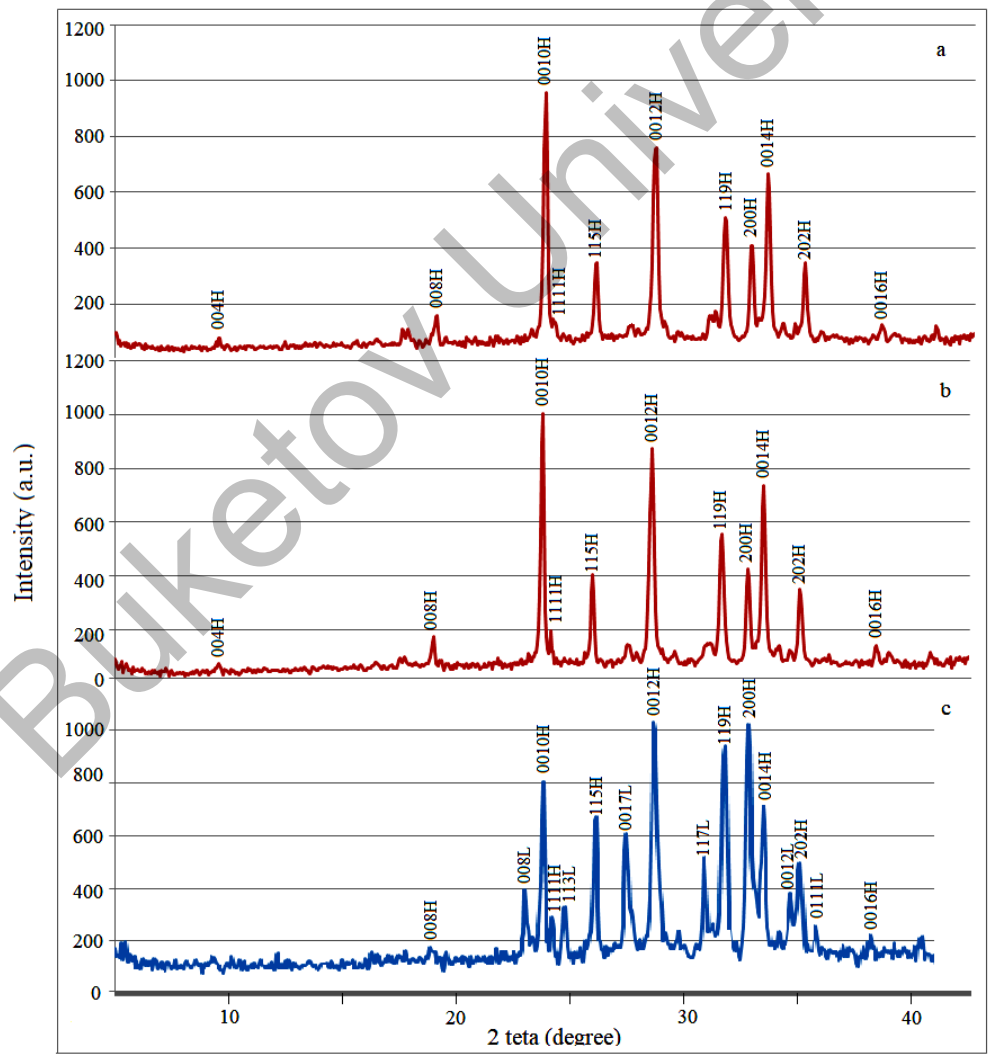
The synthesis of HTSP samples of all three types (using a radiant flux, resistive heating and solid-phase synthesis) was carried out in the same temperature regime and technological conditions. The dynamics of the

formation of the superconducting high-temperature phase Bi-2223 was traced by studying the phase composition of the samples in every 24 hours of heat treatment. Analysis of diffraction patterns revealed the presence of superconducting phases by comparison with data from the international ASTM database. The quantitative assessment of the superconducting high-temperature phase Bi-2223 (K_H) was determined by the ratio of the intensities of X-ray peaks [0010] (H[0010]) and the superconducting low-temperature phase Bi-2212 [008] (L[008]) according to the formula

$$K_H = (H[0010]/(H[0010]+L[008])\cdot 100\ \%.$$

After 24 hours of heat treatment, no peaks of the high-temperature phase Bi-2223 were observed in the solid-phase synthesis sample. And after 48 hours of heat treatment, all samples consisted of superconducting phases.

After 48 hours of heat treatment, X-ray reflections of the high-temperature phase Bi-2223 and traces of the low-temperature phase Bi-2212 were observed on the diffractogram of the sample obtained by melting in the furnace. In the samples obtained by the radiant method, the content of the Bi-2212 phase was about 20 %. After 72 hours of heat treatment, only reflexes related to the high-temperature phase of Bi-2223 were present in the samples obtained in the furnace. In the samples obtained by the radiant flux, the complete formation of the high-temperature phase occurred after 96 hours of heat treatment. Whereas in the samples obtained by the solid-phase method, the content of the low-temperature phase was about 25–30 %. Diffractograms of the samples after 96 hours of annealing are shown in Figure 4.



a — melting in a furnace; *b* — radiant melting: *c* — solid-phase method

Figure 4. Diffractograms of HTSP samples of composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ after heat treatment at 850 °C, 96 hours

The study of the microstructure showed that the crystallites have a lamellar shape with a plate thickness of up to 350 microns (Fig. 5).

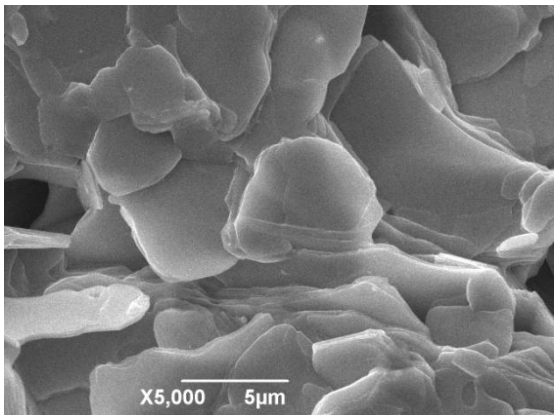
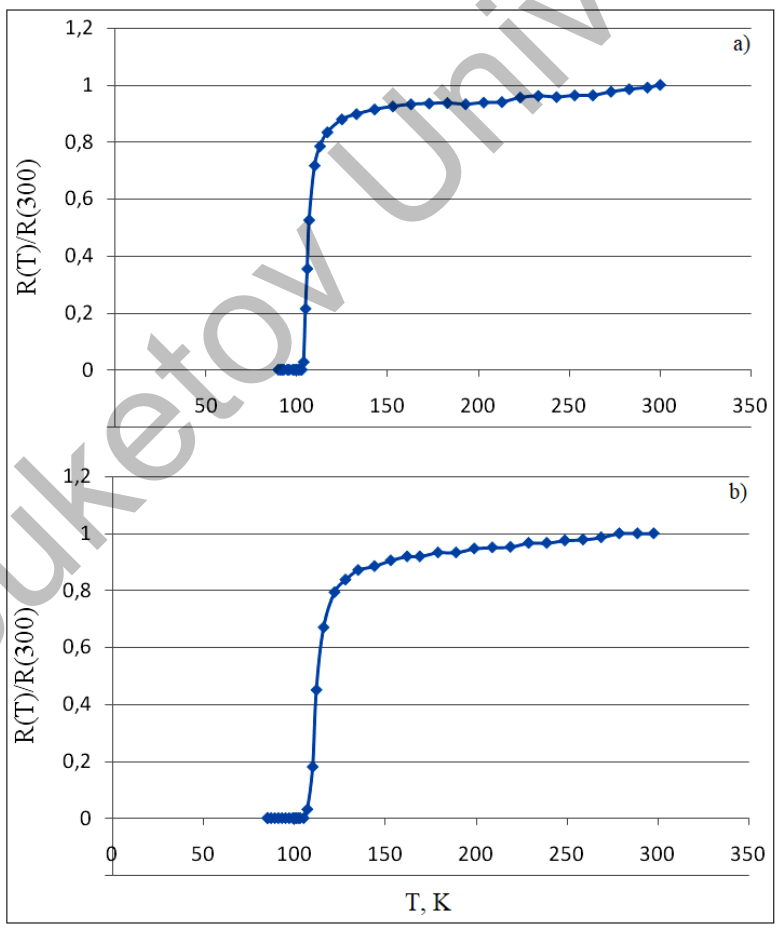


Figure 5. Microstructure of the HTSP sample of the composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ obtained on the basis of the glass phase

The study of the critical temperature showed that the temperature of the beginning of the transition to the superconducting state corresponds to 115–117 K. The critical temperature of the T_{zero} corresponds to 103 K and 105 K for samples, respectively, obtained by melting in a furnace and radiant flux (Fig. 6).



a — melting in a furnace; *b* — melting by a radiant stream

Figure 6. Curves of temperature dependence of resistance of HTSP samples of composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ obtained on the basis of glass phase

Studies of the critical temperature by the method of measuring the temperature dependence of the magnetic susceptibility of samples obtained by a radiant flux showed that microphases with higher T_c up to 150 K are present in the samples.

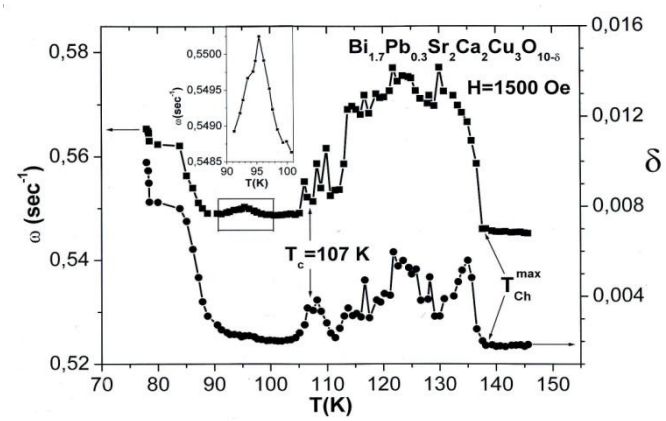


Figure 7. Curves of the temperature dependence of the magnetic susceptibility of the HTSP sample of the composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ obtained by a radiant flux

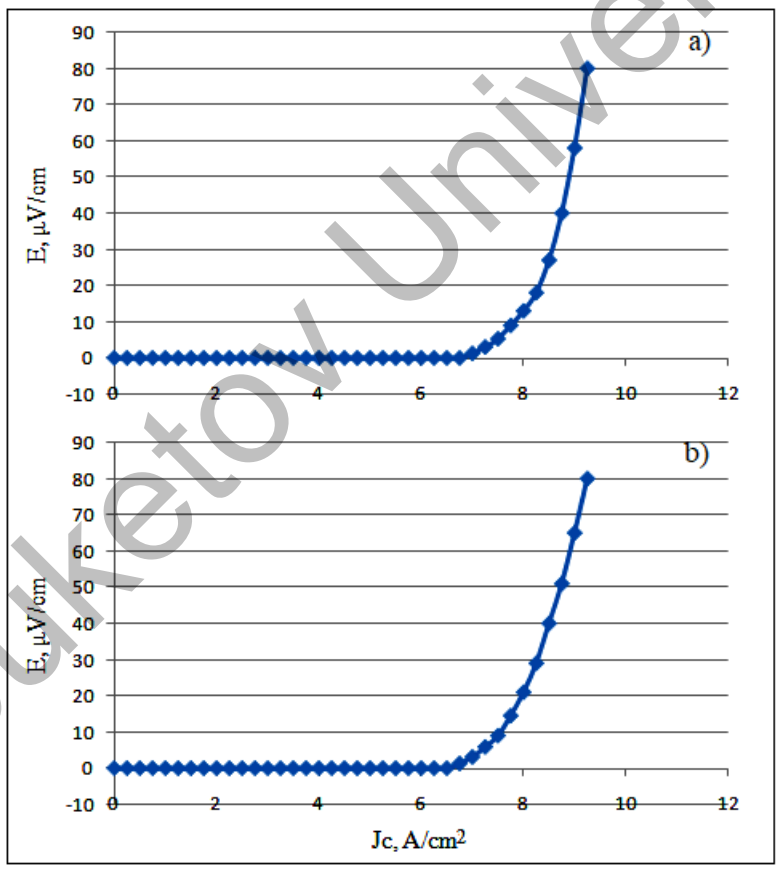


Figure 8. Critical density of the transport current of samples of the composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ obtained on the basis of the glass phase

Analysis of the results of a study on the synthesis of HTS ceramics of the composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$, obtained on the basis of amorphous precursors by quenching the melt during melting in a furnace without using a crucible and using a concentrated radiant flux, as well as by the traditional solid-phase method, show that the formation rate of the superconducting high-temperature phase Bi-2223 differs significantly. The completeness of the formation of the high-temperature phase Bi-2223 and the increased

rate of formation when using the glass phase can be explained by the following: firstly, the homogeneous distribution of the components; secondly, during melting and quenching of the melt, bonds between atoms are formed during the melting process and ultrafast quenching stabilizes the amorphous phase (presence of a short-range order), which has increased internal energy, which ensures increased reactivity; thirdly, melting and quenching of the melt is carried out in an oxidizing atmosphere, precursors contain excessive oxygen content, cations of variable valence (Cu. Bi, Pb) of which are oxidized to a highly valent state; Fourth, it is necessary to take into account the effects of high-density light flux, which can contribute to the formation of defects, changes in the energy state of atoms, etc. During crystallization of the glass phase, depending on the temperature, the formation of the Bi-2212 or Bi-2223 structure occurs due to the ordering of atoms. At temperatures below 840 °C, the Bi-2212 phase stabilizes. At a temperature of 850 °C, the Bi-2223 phase is formed, which means that the temperature range of phase formation is narrow. A slight difference in the rate of formation of the Bi-2223 phase based on the glass phase obtained by various methods can be explained by the fact that the melting temperature in the furnace can be strictly controlled. When melting by a radiant flux, the initial samples are significantly overheated (above the boiling point), which can lead to a violation of the composition due to evaporation. In this case, cations can replace each other. In the synthesis of HTSP based on the glass phase obtained by the above methods, the distribution of components in precursors is more homogeneous.

If we compare the glass-ceramic method, when the melt is obtained in crucibles, then melting occurs under equilibrium conditions and the homogeneity of the components is not ensured due to the difference in the mass of atoms, there is a significant decrease in the oxygen content. This has a negative effect on the rate of formation of the Bi-2223 phase.

As for solid-phase synthesis, the bond between atoms is formed during heat treatment, the distribution of components is heterogeneous, phase formation will occur due to the diffusion of atoms and the formation of a superconducting structure, the processes are relatively slow compared to glass-ceramic methods using the above methods.

Conclusion

Based on amorphous precursors, HTSP ceramics of the composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ were synthesized. For comparative analysis, the synthesis of superconducting ceramics was carried out on the basis of glass phases obtained by melt production without using a crucible in a furnace, melting under the influence of broadband optical radiation and synthesis of HTSP by a solid-phase method. Studies of the dynamics of the formation of the Bi-2223 superconducting phase have shown that the rate of its formation is significantly higher than in solid-phase synthesis. The critical temperature of superconductors is 103 K and 105 K for samples obtained in a furnace and water under the action of a radiant flux, respectively. It was also found that the critical density of the transport current is significantly higher than that of superconductors obtained by co-deposition, solid-phase synthesis, etc.

Acknowledgments

The work was carried out under grant AP23490836 with the financial support of the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan.

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Е.А. Ногай, А.С. Кудусов, Б.Е. Хамзина

BSCCO Bi-2223 жоғары температуралы асқын өткізгіш керамиканың критикалық ток тығыздығы

Жұмыста $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ номиналды құрамындағы жоғары температуралы асқын өткізгіш керамиканы шыны керамикалық технология бойынша аморфты фазалар негізінде және қатты фазалық әдіспен синтездеу бойынша зерттеу нәтижелері келтірілген. Салыстыру үшін аморфты фазаларды алу екі жолмен жүзеге асырылды. Бірінші әдісте, аморфты фазаны алу үшін тигельді пайдаланбай балқуды қамтамасыз ететін арнайы технологиялық жылыту пеші жасалды. Балқу үшін бастапқы үлгілерді қыздыру конвекциялық жылу ағынының және 1300–1350 нм спектрлік аймақта балқу температуралық режимі кезінде спектрдің ИҚ аймағынан тұратын қыздыру элементтерінің сәулеленуінің бірлескен әсерінен жүзеге асырылады. Екінші әдісте балқу спектрдің ультракүлгін, көрінетін және ИҚ аймақтарын қамтитын кең жолақты оптикалық сәулеленудің әсерінен орындалады. Шыны тәрізді прекурсорларды алу тот баспайтын болаттан жасалған винт түріндегі сөндіру құрылғысына балқыманың ағуы арқылы іске асырылады. Bi-2223 асқын өткізгіш жоғары температуралық фазаның түзілу жылдамдығын зерттеу 848–850°C температурада бірдей температуралық режимдерде әр 24 сағат сайын аралық ұнтақтаумен және фазалық құрамын рентгендік дифракциялық әдіспен зерттеумен жүргізілді. Зерттеулер көрсеткендей, шыны фазасына негізделген әдіс жоғары температуралы Bi-2223 фазасының толық қалыптасуын қамтамасыз етеді

және оның түзілу жылдамдығы қатты фазалық әдіске қарағанда едәуір жоғары (2,5-3 есе). Критикалық тогының тығыздығын зерттеу көрсеткендей, токтың мөлшері $7{,}05 \cdot 10^3$мА/см^2 (1 мкВ/см өлшемімен өлшегенген), бұл басқа әдістермен салыстырғанда айтарлықтай жоғары екендігін білдіреді.

Кілт сөздер: асқын өткізгіштік, шыны фазасы, микроқұрылым, керамика, ИҚ сәулелену, дифрактограмма, балқу, кристалдану.

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Плотность критического тока высокотемпературной сверхпроводящей керамики BSCCO Bi-2223

В работе приведены результаты исследования по синтезу высокотемпературной сверхпроводящей керамики номинального состава $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ различными способами, на основе аморфных фаз по стеклокерамической технологии и по твердофазному способу. Сравнение получения аморфных фаз осуществлялось двумя способами. В первом случае для получения аморфной фазы разработана нагревательная печь специальной конструкции, которая обеспечивает плавление без использования тигля. Нагрев исходных образцов для плавления осуществлялся за счет совместного влияния конвекционного теплового потока и излучения нагревательных элементов, которые состоят из ИК-области спектра при температурном режиме плавления в спектральной области 1300–1350 нм. Во втором случае плавление реализовывалось под действием широкополосного оптического излучения, включающего УФ-видимый и ИК-области спектра. Получение стеклообразных прекурсоров осуществлялось за счет стекания расплава на устройство закалки в виде пропеллера, изготовленного из нержавеющей стали. Исследования скорости образования сверхпроводящей высокотемпературной фазы Bi-2223 осуществлялись в одинаковых температурных режимах при 848–850 °С с промежуточным измельчением каждые 24 ч и исследованием фазового состава рентгеновским дифракционным методом. Исследования показали, что способ на основе стеклофазы обеспечивает полноту формирования высокотемпературной фазы Bi-2223 и скорость ее образования существенно выше, чем по твердофазному методу (2,5–3 раза). Исследования критической плотности транспортного тока показали, что величина тока (измеренная по критерию 1 μВ/см) составляет $7{,}05 \cdot 10^3$ мА/см^2, что значительно превышает показания по сравнению с другими методами.

Ключевые слова: сверхпроводимость, стеклофаза, микроструктура, керамика, ИК излучение, дифрактограмма, расплав, кристаллизация.

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