DOI 10.31489/2023PH3/163-170

UDC 621.384.327

D.E. Uskenbaev, A.S. Nogai*, A.D. Uskenbaev, E.A. Nogai

S. Seifullin Kazakh Agrotechnical Research University, Astana, Kazakhstan,
*(*E-mail: nogay06@mail.ru)*

Effects of conditions on the synthesis and properties of Bi-2234 HTSC ceramic produced from the melt

The article presents the results of a study aimed at obtaining the formation of superconducting phases and the formation of a high texture of particles that affect the current-carrying characteristics in ceramics of the composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_3Cu_4O_y$ and the study of their properties. For the synthesis of ceramics, precursors from the glass phase were used, which were obtained by melting the starting material under the influence of radiant flux (IR radiation) and quenching the melt in a facility rotating at a speed of 3000 rpm. Platinum wire was used as a substrate. During the heat treatment of samples in the temperature range of 845–850 °C and a holding time of 72 hours (with intermediate grinding every 24 hours), the superconducting high-temperature phase Bi-2223 crystallized in the studied samples. Critical temperatures and resistances of superconducting samples were measured by the four-probe method by measuring the dependence of resistance on the temperature in the range from 300 K to 60 K.

Keywords: superconductivity, critical temperature, electrical resistance, diffractogram, glass phase.

Introduction

Superconductors are among the promising materials with special electrical properties for use in various industries. The superconductors developed to date have made it possible to create prototypes of various electronic devices — SQUID magnetometers [1], microwave generators [2], protective devices for sensitive semiconductor elements [3], elements of quantum computers [4, 5], optical sensors [6] and elements of high-speed electronics [7], etc. Due to their high current carrying capacity, they occupy a special place in the power industry for the creation of various high-current elements and devices such as motors and electric generators [8], power cables [9-11], transformers [12, 13], motors [14, 15], current limiters for short circuits [16-18].

With the establishment of superconductivity in the La-Ba-Cu-O oxide system [19], the science of superconductivity moved to a new stage of development, which developed families of a number of superconducting cuprate systems with a critical temperature exceeding the temperatures of cheap liquid nitrogen — yttrium [20], bismuth [21], mercury [22], thallium [23], etc.

Among these superconducting systems, bismuth-containing superconductors can be attributed to one of the most promising.

It is known that in the Bi–Sr–Ca–Cu–O bismuth system, three stable superconducting compounds of the homologous series $Bi_2Sr_2Ca_{n-1}Cu_nO_y$ (n = 1, 2, 3) are established with a transition temperature to the superconducting states of 30-35 K, 80-90 K and 107 — 110 K, respectively. With an increase in the number of Ca and Cu layers, the critical temperature also increases. But on the other hand, with an increase in the critical temperature, the requirements for the condition for obtaining (if phase 2201 crystallizes from the melt, then the temperature interval for the formation of phase 2212 lies in the region of 750-850 C and the synthesis time is 80-1100 hours, and for the phase, the existence interval lies in a narrow temperature region of 848-850 C and long annealing from 150-400 hours). If phase 2201 crystallizes from the melt, then the synthesis of phase 2212 requires several tens of hours of heat treatment at a temperature of 800 — 850 hours. And for the synthesis of phase 2223, more stringent conditions are required — heat treatment in a narrow temperature range with intermediate grinding involving the liquid phase with a time of 100-350 hours of exposure, depending on the approach and method. In [24], when synthesizing HTS of the composition $Bi_{1.65}Pb_{0.35}Sr_2Ca_4Cu_5O_y$by the solid-phase method using the matrix method ($Bi_{1.65}Pb_{0.35}Sr_2Ca_2Cu_3O_y$and Ca_2PbO_4 were used as a matrix), along with phase 2223, a low-temperature superconducting phase 2212 and Ca_2PbO_4 were present. In [25], during the synthesis of HTSP compositions $Bi_{1.7}Pb_{0.3}Sr_{2+x}Ca_{4-x}Cu_5O_y$ (x = 0.2; 0.4; 0.6) with partial substitution of calcium for strontium during heat treatment for 150 h at a temperature of 840-850 °C, a superconducting 2223 phase with Tc =107 K was obtained. In [26], when

synthesizing the composition $Bi_{1.9}Pb_{0.4}Sr_2Ca_{3.1}Cu_{4.2}O_x$ by ceramic technology, the dominant phase was 2223 (about 80 %). Pb(Sr, Ca)$_2O_4$, Ca_2CuO_3, CuO oxides and superconducting phases 2201 and 2212 were also present. In [27], the synthesis of compositions (Bi, Pb)$_4Sr_3Ca_3Cu_{4-m}Fe_mO_x$ (m = 0 — 0.06) was carried out from the glass phase. Melting was carried out in a platinum crucible and tempered by flapping between two massive plates. After annealing at 840 °C for 40 hours, the presence of a low-temperature superconducting phase 2212 (the main phase was 2223) was found in the samples. In [28], superconducting ceramics of the composition $Bi_{1,6}Pb_{0,4}Sr_2Ca_3Cu_4O_y$ were synthesized by casting from a melt. As a result of prolonged annealing (150 hours), the samples consisted of phases 2212 and 2223. In [29-31], the synthesis of HTS was carried out using laser radiation. In [29], new layered bismuth cuprates were obtained by laser ablation in the form of films of the compositions $Bi_2(La, Ca)_2Ca_{n-1}Cu_nO_z$(n = 3, 4, 5, 6, and 7). Measurements of the lattice constant showed that "c" is 3.66 nm; 4.31 nm; 4.94 nm; 5.60 nm and 6.25 nm corresponding to phases 2223, 2234, 2245, 2256 and 2267. But in all samples, the effect of superconductivity was not detected above 4.2 K, which may be related to the defects of crystal structures. In [30, 31], a textured superconductor with a critical current of 5000 A/cm^2 was synthesized by zone melting under the influence of laser radiation.

The analysis of numerous studies shows that in the synthesis of HTS with increased content of calcium and copper by various methods, obtaining a high-temperature monophase sample with 2223 is a difficult task, because low-temperature superconducting phase 2212 is always present in the samples. In many cases, the samples also contain non-superconducting intermediate phases. And in the synthesis of HTS using radiant energy, it can be assumed that the formation of superconducting phases with an increased content of calcium and copper, also obtain superconducting materials with high critical parameters. In this regard, the synthesis of HTS phases in the Bi(Pb)–Sr–Ca–Cu–Os system using IR radiation was of interest.

Materials and methods

The following components were used for the synthesis of HTSP composition $Bi_{1,6}Pb_{0,4}Sr_2Ca_3Cu_4O_y$: Bi_2O_3, $Bi(NO_3)_3 \cdot 5H_2O$, PbO, PbO_2, $SrCO_3$, CaO, CuObrands no worse than "PA". The initial precursors and HSTP samples were annealed in a muffle furnace SNOL –8.2/1100. The phase compositions of amorphous precursors and superconducting samples were controlled by X-ray diffraction using Bruker D8ADVANCEECO and XPertPRO diffractometers (Netherlands). Microstructural and elemental analyses of samples were carried out on scanning electron microscopes JEOL-6490LA (Japan) with an energy dispersive analyzer system "OXFORD Instruments Analytical Limited" (UK) and JSM-6390LV (Japan) with an integrated energy dispersive X-ray analyzer (EDS). The critical temperature was determined by measuring the temperature dependence of the resistance in the temperature range from 300 K to 60 K by a four-probe method using a closed cryo-camera "CryoIndustry REF-1808-ACS", cryo-camera cooled by helium gas, a LakeShoreModel 340 temperature meter and a microvoltmeter. As a contact between the sample and the electrodes, a contractor of the SCP brand based on a silver paste was used.

Results and discussion

This paper presents the results of the synthesis of Bi-HTS composition $Bi_{1,6}Pb_{0,4}Sr_2Ca_3Cu_4O_y$ based on glass phase, which is a kind of melt technique. When obtaining HTSC ceramics from a glass phase, the synthesis of superconducting phases is strongly influenced by the method and conditions for obtaining precursors, since in the bismuth system there are elements of variable valence Cu, Bi, Pb.

Depending on the receiving condition, they may be in different charge states.

And peculiarities of the influence of radiation, such as the electromagnetic field, which can affect the fine structure of the material (defect). Features of the influence of radiation, such as an electromagnetic field, which can affect the fine structure of the material (defect). To obtain precursors, a non-melting method was developed for obtaining a glass phase from a melt under the influence of IR radiation. For comparative analysis, the results obtained using dense optical radiation and glass phase obtained in a muffle furnace by melting in a crucible were also used.

By synthesizing HTSC ceramics with the composition $Bi_2Sr_2Ca_3Cu_4O_y$, it was found that the diffraction patterns of bulk HTSC precursors (glass-crystalline) obtained in a muffle furnace contain low-valence oxide Cu_2O.

On the contrary, Cu_2O oxides are absent in precursors obtained by heating with radiant energy (optical and IR radiation). Studies of the formed superconducting phases have shown that the superconducting phase 2223c T_{sn} = 107-110 K crystallizes in the composition of $Bi_2Sr_2Ca_3Cu_4O_y$ (Fig. 1, 2).

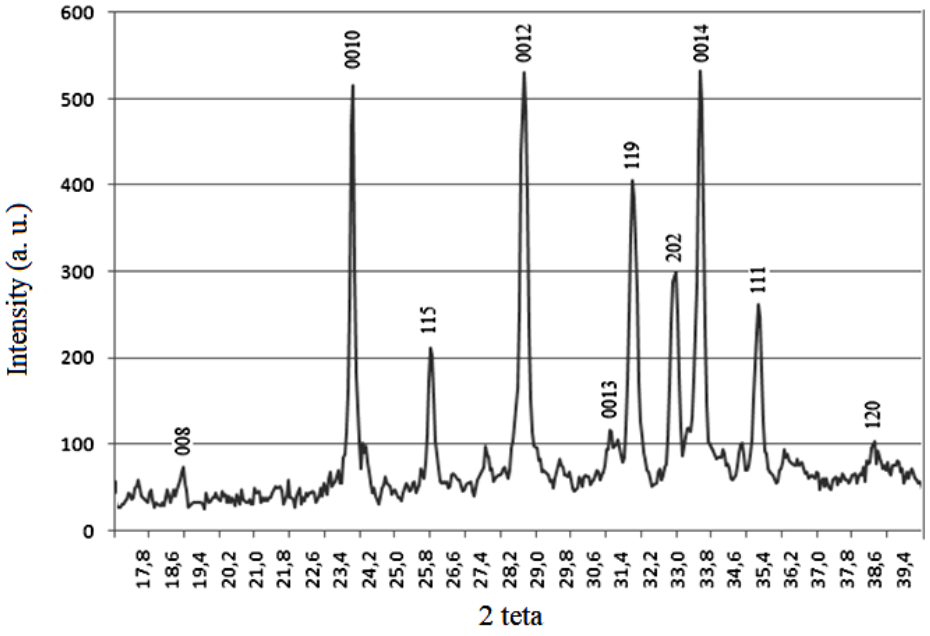
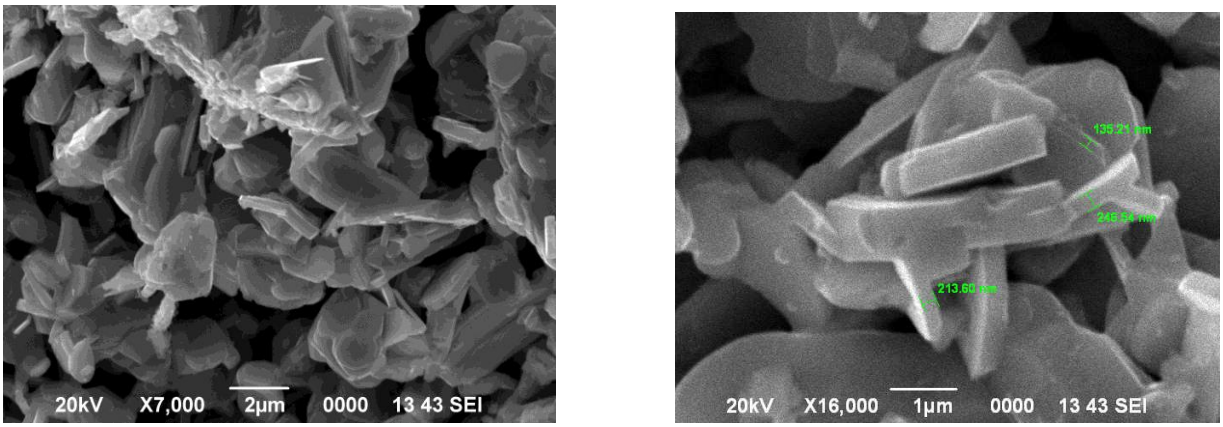


Fig. 1. X-ray diffraction pattern of an HTSC sample of composition Bi1.6Pb0.4Sr2Ca3Cu4Oy synthesized from amorphous precursors obtained by IR heating

The results of the X-ray phase analysis show that all X-ray reflexes are related to the high-temperature superconducting phase Bi-2223.

It follows from the X-ray data (hkl [0010], [0012], and [0014]) that the ceramic sample has a texture along the 00l crystallographic plane.

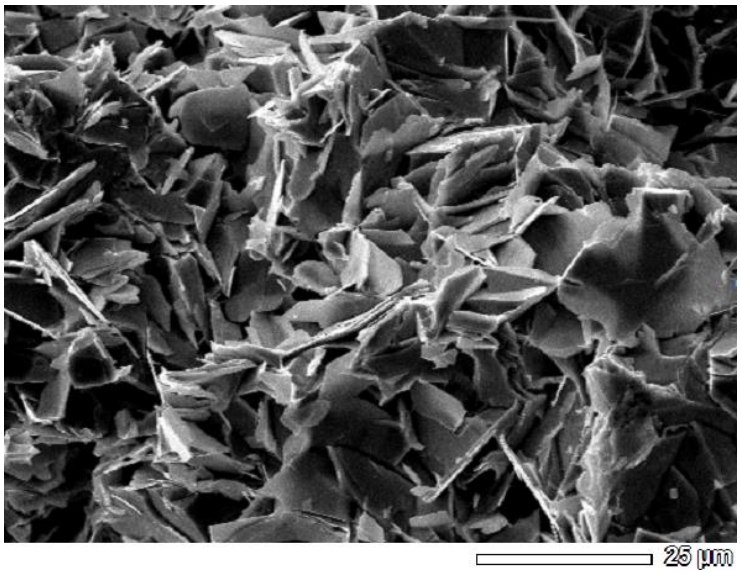
The results of the study of the electron microscopic analysis of HTSC ceramics show that the crystallites have a lamellar shape (Fig. 2)



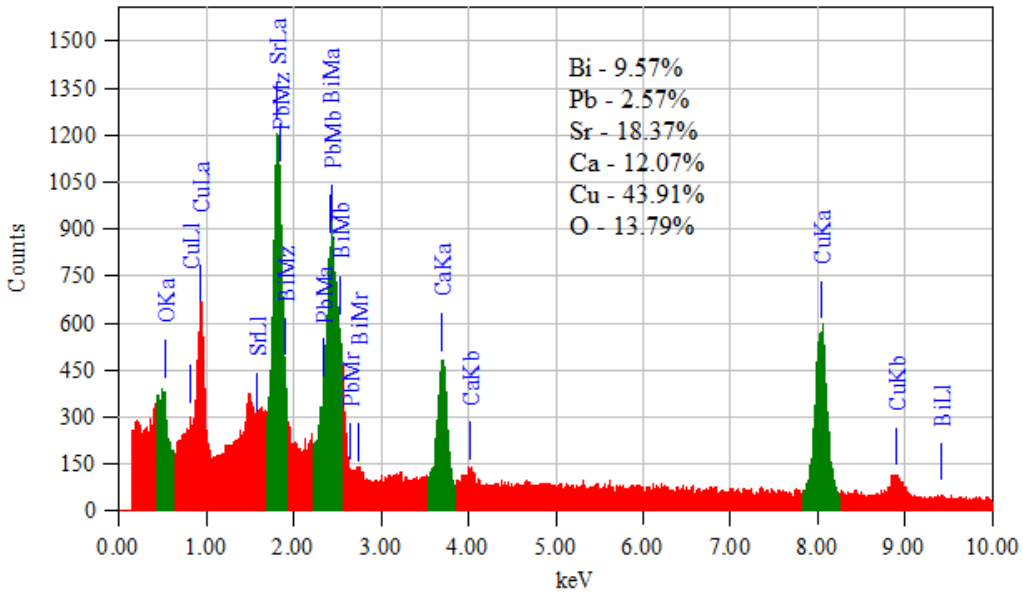
a) b)

Fig. 2. Microstructure of HTSC ceramics of composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_3Cu_4O_y$ synthesized from amorphous precursors obtained by IR heatinga) — x 7000, b) — x16000

The crystallite size is in the range of 2–8 μm (Fig. 2a), and the thickness is in the range of 0.2–0.25 μm. By analyzing the results of studying the content of elements in a sample of superconducting ceramics, it was found that metal cations do not undergo noticeable changes in their content (Fig. 3).



a)



b)

Fig. 3. Elemental analysis of $Bi_{1.6}Pb_{0.4}Sr_2Ca_3Cu_4O_y$ HTS ceramics synthesized from amorphous precursors obtained by IR heating: a) microstructure; b) elemental composition.

The critical temperature was determined in a cryogenic installation by changing the resistance of the sample when the sample temperature was cooled from room temperature (300 K) to 70 K. It was found that the transition to the superconducting state begins at 107 K. Such a transition is typical for the superconducting phase of Bi-2223 (Fig. 4).

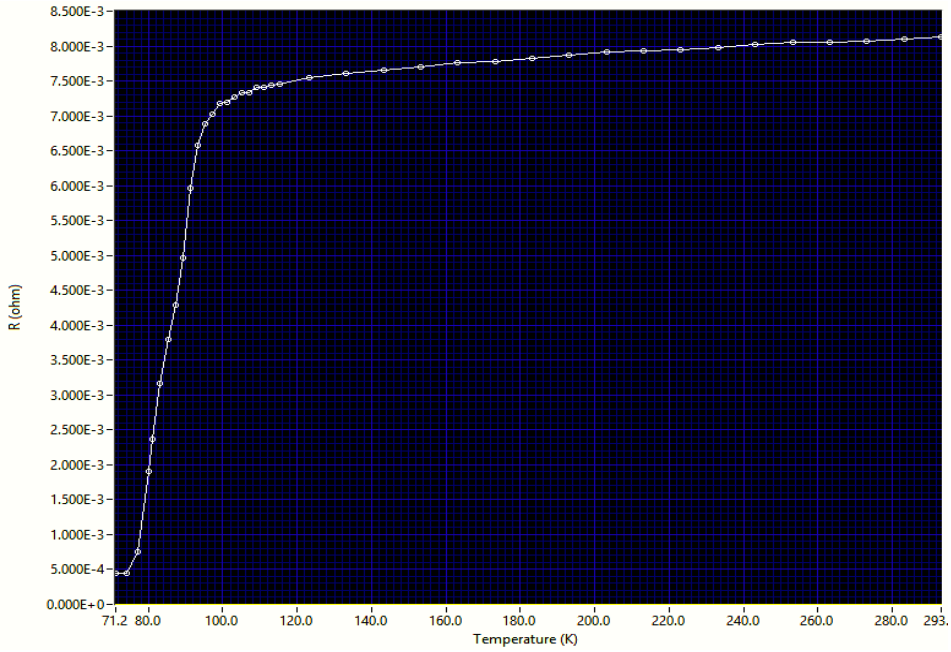


Figure 4. Critical temperature of a $Bi_{1.6}Pb_{0.4}Sr_2Ca_3Cu_4O_y$ sample synthesized from amorphous precursors obtained by IR heating

The complete transition to the superconducting state corresponds to a temperature of 75 K and the resistance of the HTSP sample is $4{,}7 \cdot 10^{-4}$ ohms. It is assumed that such a large transition width is associated with Ca and Cu cations, which can be located on the surface of crystallites and in grain boundaries. It is possible that with the growth of crystallites of the superconducting phase Bi-2223, excess Ca and Cu cations are displaced onto the surface of the crystallites and grain boundaries, forming a non-superconducting layer, which can affect the critical temperature of the superconducting sample, i.e. reduce the T_c.

When synthesizing HTSC samples from amorphous precursors using optical radiation with a high flux density, then single-phase samples 2223 are synthesized at a temperature of 846-848 C with an exposure time of 70-80 hours. In HTSC ceramics synthesized from a melt in a muffle furnace, even with a heat treatment time of more than 150 hours, the 2212 phase is present in the samples, which may be due to the influence of the atmosphere on the valence states of variable valence cations, as well as the peculiarity of the effect of radiation on precursors. When using amorphous precursors obtained under the influence of IR radiation, the synthesis time is reduced to 40-50 hours and the reliability of the formation of single-phase HTS materials increases (Fig. 5).

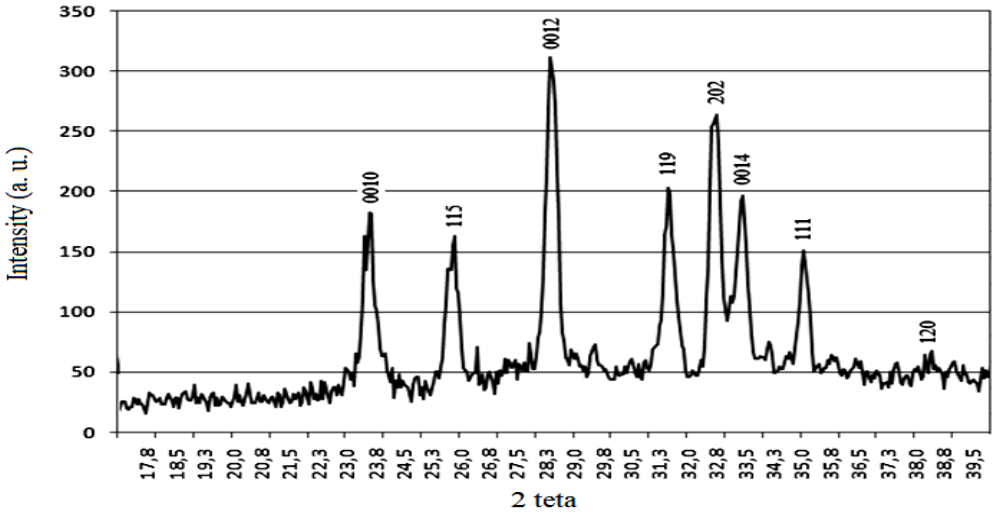


Fig. 5. X-ray diffraction pattern of the HTSC $Bi_{1.6}Pb_{0.4}Sr_2Ca_3Cu_4O_y$ sample synthesized at 850 °C for 48 h from amorphous precursors prepared by IR heating

Analysis of the results of the X-ray diffraction study of the HTS sample composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_3Cu_4O_y$ shows that all X-ray reflection reflexes belong to the high-temperature superconducting phase 2223. A particularly important task of the synthesis process is to achieve a high texture, which can directly affect the current-carrying characteristics of HTS ceramics. To achieve a high texture, it was necessary to increase the duration of the processes of grinding the charge, pressing under high pressure, as well as its heat treatment to improve the perfection of the structure and increase the size of the crystallites. After these successive HTS processes, the ceramics had a high texture with perfect X-ray reflection reflexes (Fig. 6).

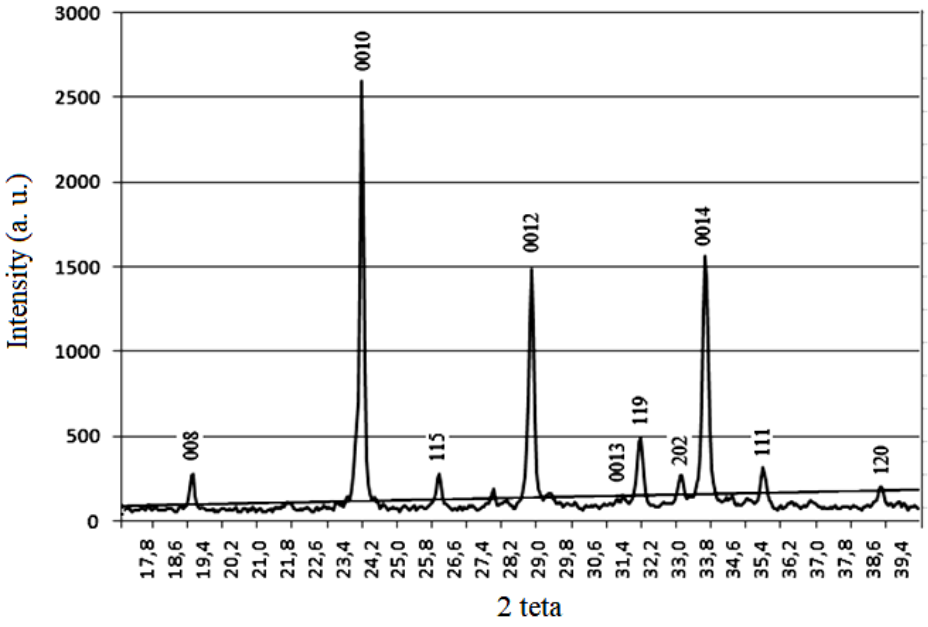


Fig. 6. X-ray diffraction pattern of the HTSC $Bi_{1.6}Pb_{0.4}Sr_2Ca_3Cu_4O_y$ sample synthesized at 850 °C for 120 h from amorphous precursors obtained by IR heating

All X-ray reflection reflexes belong to the superconducting phase 2223. The degree of uniformity of the texture is about 85-90 % and it is directed along the crystallographic plane *00l*.

Conclusions

Thus, based on the above, it can be concluded that in the synthesis of HTS composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_3Cu_4O_y$ based on amorphous precursors, the heating method and conditions for obtaining precursors are important. When precursors are obtained under oxidizing atmospheric conditions and under the influence of IR radiation, the formation time of the high-temperature superconducting phase Bi-2223 is reduced compared to samples obtained by melting (in a crucible) in a muffle furnace. The study of the temperature dependence of the resistance of samples of HTS ceramics found that on samples obtained with the use of IR radiation, the beginning of the transition to a superconducting state corresponds to a temperature of 107 K, and the critical temperature T_c = 75 K. It is assumed that such a wide transition to the superconducting state is associated with the formation of excess cations on the surface and in the grain boundaries, displaced from the volume during the growth of crystallites, having ordinary conductivity (not superconductor).

Acknowledgments

The work was carried of the grant of the Education and Science of the Republic of Kazakhstan AP09260251.

References

1 Keene, M. et al. (1993). Thin film HTS SQUID construction and characterization. *IEEE Transactions on Applied Superconductivity, Vol. 3,* No 1, P. 2430–2433.

2 An, D. et al. (2013).Terahertz emission and detection both based on high-Tc superconductors: Towards an integrated receiver. *Applied Physics Letters, Vol. 102,* No 9, P. 092601.

3 Fyk, O. Kucher, D., Kucher, L., Gonchar, R., Fyk, M., & Besedin, Y. (2018). Analysis of the technology to manufacture a high-temperature microstrip superconductive device for the electromagnetic protection of receivers. *Eastern-European Journal of Enterprise Technologies, Vol. 5,* No 12-95, P. 38—47.

4 Schulz, R.R. et al. (2000). Design and realization of an all d-wave dc π-superconducting quantum interference device. *Applied Physics Letters, Vol. 76,* No 7, P. 912–914.

5 Devoret, M.H., & Martinis, J.M. (2005). Implementing qubits with superconducting integrated circuits. *Experimental Aspects of Quantum Computing. Springer,* P. 163–203.

6 Jurbergs, D.C. et al. (1995). Electrochemical and optical devices based on molecule/high-Tc superconductor structures. *Electrochimica Acta., Vol. 40,* No 10, P. 1319–1329.

7 Xiong, W., Kula, W., & Sobolewski, R. (1994). Fabrication of High-T c Superconducting Electronic Devices Using the Laser-Writing Technique. *Advances in Cryogenic Engineering Materials. Springer,* P. 385–391.

8 Hull, J.R., & Strasik, M. (2010). Concepts for using trapped-flux bulk high-temperature superconductorin motors and generators. *Superconductor Science and Technology, Vol. 23,* No 12, P. 124005.

9 Hajiri, G., Berger, K., Trillaud, F., Lévêque, J., & Caron, H. (2023). Impact of Superconducting Cables on a DC Railway *Network. Energies, No 16*(2), P. 776.

10 Hamabe, M., Sugino, M., Watanabe, H., Kawahara, T., Yamaguchi, S., Ishigur, Y., & Kawamura, K. (2011). Critical current and its magnetic field effect measurement of HTS tapes forming DC superconducting cable. *IEEE Transactions on Applied Superconductivity, No 21,* P. 1038-1041.

11 Bulinski, A., & Densley, J. (1999). High voltage insulation for power cables utilizing high temperature superconductivity. *IEEE Electrical Insulation Magazine, No 15*(2), P. 14-22.

12 Kumar, M., Joshi, P., & Gupta, R.A. (2023). 50 kA Superconducting Transformer for the Upcoming High-Field High-Current Testing Station at the BNL. *IEEE Transactions on Applied Superconductivity, No 33*(5), P.5500305.

13 Orosz, T. (2022). FEM-Based Power Transformer Model for Superconducting and Conventional Power Transformer Optimization. *Energies, No 15*(17), P. 6177.

14 Lu, M., & Cao, R.A. (2021). Novel Double-Sided High Temperature Superconducting Linear Modular Flux-Switching Motor. *IEEE Transactions on Applied Superconductivity, No 31*(5), P. 9357980.

15 Ma, E., Wu, H., & Li, Y. (2021). Development and testing of a cryogenic induction motor with a novel Dewar structure. *Cryogenics, No 116,* P.103283.

16 Kozak, J. (2022). Forces and Stresses in the Windings of a Superconducting Fault Current Limiter. *Energies, No 15*(18), P. 6519.

17 Choi, Y., Song, S., Kim, J., Choi, Y.S., & Ko, T.K. (2022). Thermal and Electrical Characteristics of Hollow Former With Fault Current Limiting Function for Superconducting Power Cable. *IEEE Transactions on Power Delivery, No 37*(2), P. 1100-1108.

18 Lim, S.H. (2010). Study on current limiting characteristics of SFCL with two trigger current levels. *Physica C: Superconductivity and its Applications, No 470*(20), P. 1631-1635.

19 Bednorz, J.G., & Muller, K.A. (1986). Possible high Tc superconductivity in Ba-La-Cu-O system. *Z. Phys., Vol. 64B,* No 1, P.189-193.

20 Wu, M.K., Ashburn, J.R., & Torng, C.J. et. al. (1987). Superconductivity at 93 K in a New Mixed Phase Y-Ba-Cu-O Compound System at Ambient Pressure. *Phys. Rev. Lett., Vol. 58,* No 9, P. 908-910.

21 Takano, M., Takada, J., & Oda, K., et. al. (1988). High-Tc Phase Promoted and Stabilized in the Bi, Pb-Sr-Ca-Cu-O System. *Jpn. J. Appl. Phys., Vol. 27,* No 6, P. L1041-L1043.

22 Chu, C.W., Gao, L., & Chen, F., et. al. (1993). Superconductivity above 150 K in HgBa2Ca2Cu3O8-δat high pressures. *Nature, Vol. 365,* P. 323-325.

23 Sheng, Z.Z., & Hermann, A.M. (1988). Superconductivity in the rare-earth-free Tl–Ba–Cu–O system above liquid-nitrogen temperature. *Nature, Vol. 332,* No 6159, P. 55-58.

24 Ashoka, N.V., George, T.C., & Srinivasan, K. (2014). Investigation of Structural Formation of Starting Composition 2245 in the Bi-Pb-Sr-Ca-Cu-O System Superconductors. *Journal of Solid State Physics, V. Article ID 716032,* P. 1-7.

25 Meretliev, Sh., Sadykov, K.B., & Berkeliev, A. (2000). *Turk J. Physics,* No 24, P. 39.

26 Panova, G.P., Khlopkin, M.N., Chernoplekov, N.A., & Gorobchenko, V.D. (1990). *Superconductivity: physics, chemistry, technology. Vol. 3.*, No 3. P. 421.

27 Chatterjee, S., Bhattacharya, S., & Chaudhuria, B.K. (1998). *Journal of Chemical Physics, Vol. 108,* No 7, P. 2954.

28 Coskun, A., Ozcelik, B., & Kiymac, K. (2001). Physical Properties of Melt-Cast Annealed Bi{1.6}Pb{0.4}Sr{2}Ca{3}Cu{4}O{12}Compound. *Turk J. Phys., Vol. 25,* No 5, P. 473.

29 Yoshiba, K., Sasakura, H., Tsukuic, S., & Mizokawab, Y. (1999). New Bi-cuprate thin films of $Bi_2(La, Ca)_2Ca_{n-1}Cu_nO_z$ (n=3, 4, 5, 6 and 7) prepared by the laser ablation method. *Physica C: Supercond., Vol. 322,* Iss. 1-2, P. 25-30.

30 Mora, M., Gimeno, F., Angurel, L.A., & De la Fuente, G.F. (2004). Laser zone $Bi_2Sr_2CaCu_2O_{8+\delta}$ thick films on (100) MgO substrate. *Supercond. Sci. Technol. Philadelphia, No 17,* P. 1133-1138.

31 Mora, M., Lopez-Gascon, C., Angurel, L.A., & De la Fuente, G.F. (2004). The influence of support temperature on Bi-2212 monoliths textured by diode laser zone melting. *Supercond. Sci. Technol. Philadelphia, No 17,* P. 1129-1133.

Д.Е. Өскенбаев, А.С. Ноғай, А.Д. Өскенбаев, Э.А. Ноғай

Балқымадан өндірілген Bi-2234 висмут HTSC керамикасының синтезі мен қасиеттері

Мақалада $Bi_{1.6}Pb_{0.4}Sr_2Ca_3Cu_4O_y$ құрамды керамикадағы ток өткізгіштік сипаттамаларға әсер ететін асқын өткізгіш фазаларды алуға, бөлшектердің жоғары құрылымын қалыптастыруға және олардың қасиеттерін зерттеуге бағытталған зерттеу нәтижелері келтірілген. Керамика синтезі бұрын дайындалған бастапқы шыны фазасы үлгілері негізінде ИҚ-сәулелену әсерінен балқыту және өте жоғары жылдам сөндіру арқылы (3000 айналу/мин жылдамдықпен айналатын қондырғыда) жүзеге асырылды. Субстрат ретінде платина сымы қолданылды. Үлгілер 72 сағат ұстау уақытымен (аралығы әрбір 24 сағат сайын ұнтақтаумен) 845-850 температура аралығында термиялық өңдеу арқылы синтезделді. Барлық үлгілерде жоғары температуралы асқын өткізгіш Bi-2223 фаза кристалданды. Асқын өткізгіш үлгілердің критикалық температуралары мен кедергілерін өлшеу төрт зондты әдіспен 300 К-ден 60 К-ге дейінгі диапазондағы кедергінің температураға тәуелділігін өлшеу арқылы жүзеге асырылды.

Кілт сөздер: асқын өткізгіштік, критикалық температура, электр кедергісі, дифрактограмма, шыны фазасы.

Д.Е. Ускенбаев, А.С. Ногай, А.Д. Ускенбаев, Э.А. Ногай

Синтез и свойства висмутовой ВТСП керамики Bi–2234, полученной из расплава

В статье представлены результаты исследования, направленные на образование сверхпроводящих фаз и формирование высокой текстуры частиц, влияющих на токонесущие характеристики в керамике состава $Bi_{1.6}Pb_{0.4}Sr_2Ca_3Cu_4O_y$, и исследования их свойств. Синтез керамик осуществляли на основе стеклофазы, полученной под воздействием ИК излучения путем плавления и сверхбыстрой закалки расплава (на установке, вращающейся со скоростью 3000 об/мин) предварительно подготовленных исходных образцов. В качестве подложки была использована платиновая проволока. При термообработке образцов в интервале температур 845–850 °С и времени выдержки 72 ч (с промежуточным помолом в каждые 24 ч) в исследуемых образцах кристаллизовалась сверхпроводящая высокотемпературная фаза Bi–2223. Измерение критических температур и сопротивлений сверхпроводящих образцов осуществлялось четырехзондовым методом путем измерения зависимости сопротивления от температуры в интервале от 300 К до 60 К.

Ключевые слова: сверхпроводимость, критическая температура, электросопротивление, дифрактограмма, стеклофаза.