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Investigation of the Effect of Laser and Optical Radiation on the Formation and Properties of High-Temperature Superconducting Compounds

[1]**NOGAY Adolf,** *Dr. Phys. and Math. Sci., Professor, nogay06@mail.ru,*
[1]**USKENBAEV Daniyar,** *PhD, Associate Professor, usdan01@mail.ru,*
[1]***TATKEYEVA Galina,** *Dr. Tech. Sci., Head of Department, tatkeeva@mail.ru,*
[2]**ALEKSANDROVSKY Vladimir,** *Senior Researcher, alexandrovsky@inbox.ru,*
[1]**ZHETPISBAEV Kairatbek,** *Senior Lecturer, 79kairatbek@mail.ru,*
[1]**USKENBAEV Alisher,** *master student, Д.alish_us@mail.ru,*
[1]*S. Seifullin Kazakh Agrotechnical University, Kazakhstan, 010011, Nur-Sultan, Zhenis Avenue, 62,*
[2]*MIREA – Russian Technological University, Russia, 119454, Moscow, Vernadsky Avenue, 78,*
**corresponding author.*

Abstract. *The paper presents the results of the synthesis of bismuth superconductors of the compositions $Bi_{1.6}Pb_{0.4}Sr_2Ca_{n-1}Cu_nO_y$ (n = 1, 2) from amorphous and crystalline starting materials obtained from the melt. Laser and optical radiation of a wide spectral range containing ultraviolet, visible and near-infrared regions of the spectrum were used as a heating source. The elemental composition of the starting materials and the effect of the starting materials on the formation of superconducting phases, as well as on the critical parameters of superconducting ceramics, are studied. It is established that, depending on the conditions for obtaining the initial materials from the melt, there is a slight violation of the stoichiometric composition of the samples in terms of cation composition and a significant change in oxygen, which in turn affects the formation of superconducting phases and the properties of superconducting ceramics.*

Keywords: *high-temperature superconductivity, laser radiation, structure, ceramics, morphology, diffractogram, phase.*

Introduction

Currently, high-temperature superconductors (HTSC), due to their unique properties, are becoming one of the promising materials used in various advanced areas of science and technology – energy, electronics, communications, medicine, space technology and other fields of application [1-3]. On their basis, such elements and devices are manufactured as: conductive cables, current limiters, power transformers, electromagnetic shields, modulators, antennas, switches and filters of microwave and pulse signals, bolometers of millimeter, submillimeter and infrared radiation ranges, schematic diagrams of ultra-high-speed computers, elements.

The main factors that determine the critical parameters (critical current, critical temperature, and critical magnetic field) of an HTSC material are to create the required phase composition and structure, microstructure and morphology, density, etc. parameters, depending on the methods of obtaining, the optimal combinations of these factors.

At present, various methods have been developed for the synthesis of HTSCs: solid-phase synthesis, chemical and cryotechnology, various melt methods, etc. Among these methods, the most common method is solid-phase synthesis. But the analysis of the results of numerous studies shows that the required critical characteristics (especially current) are difficult to obtain by solid-phase synthesis.

The creation of HTSC materials of a given phase composition with the necessary critical parameters consists in the development of melt methods and their modifications [4, 5]. In particular, methods have been developed aimed at obtaining amorphous-crystalline and glassy (amorphous) states, which have certain advantages over the traditional solid-phase method and make it possible to obtain high-density ceramics with a controlled grain size and required morphology. In addition, there are opportunities for obtaining the required configuration and dimensions of the product in practical application.

Although at present several classes of HTSC compounds are known: yttrium, thallium, mercury, bismuth, etc., when implementing amorphous-crystalline and amorphous methods, bismuth containing HTSCs are widely used, which is the only representative of the above classes of superconducting compounds, which, when quenching the melt the glassy state is stabilized.

As is known, bismuth-containing superconducting compounds are a homologous series $Bi_2Sr_2Ca_{n-1}Cu_nO_y$ (n = 1, 2 … …), among which the most stable and stable are compounds with n = 2 and 3, respectively, having a critical transition temperature to the superconducting state of about 85 K and 107 K, which are currently finding wide practical applications.

Obtaining materials of a given composition with high critical parameters is a serious problem. The aim of this work was to study the effect of laser and optical radiation on the initial precursor materials obtained from the melt on the phase composition and properties of bismuth-containing HTSC ceramics.

Experimental technique

To obtain the initial glassy and amorphous-crystalline samples of HTSC ceramics in the Bi_2O_3-PbO-SrO-CaO-CuO system of nominal compositions $Bi_{1.6}Pb_{0.4}Sr_2CaCu_2O_x$ and $Bi_{1.4}Pb_{0.4}Sr_2Ca_2Cu_3O_x$ using the melt method, the following reagents were used: Analytical grade, lead (II) oxide, grade, strontium carbonate, grade, as well as calcium oxide and copper (II) oxide, grade. Before preparing the charge, the reagents were calcined at a temperature of 150°C for 3 hours, after which the carefully displaced charge was pressed under a pressure of 180 MPa into tablets with a diameter of 15 mm and a thickness of 3 mm. Before the melting process, the samples were preliminarily annealed in a muffle furnace at a temperature of 750-800°C for 2 hours. For comparative analysis, precursor starting materials from the melt were obtained by the following methods: by melting in a muffle furnace on a corundum crucible; direct action of UV laser radiation on the surface of tablet samples; as well as high-density optical radiation of a wide spectral composition containing UV, visible and IR spectral regions. The melt was quenched between two oppositely rotating copper rolls and also by quenching on a rotating stainless steel disk.

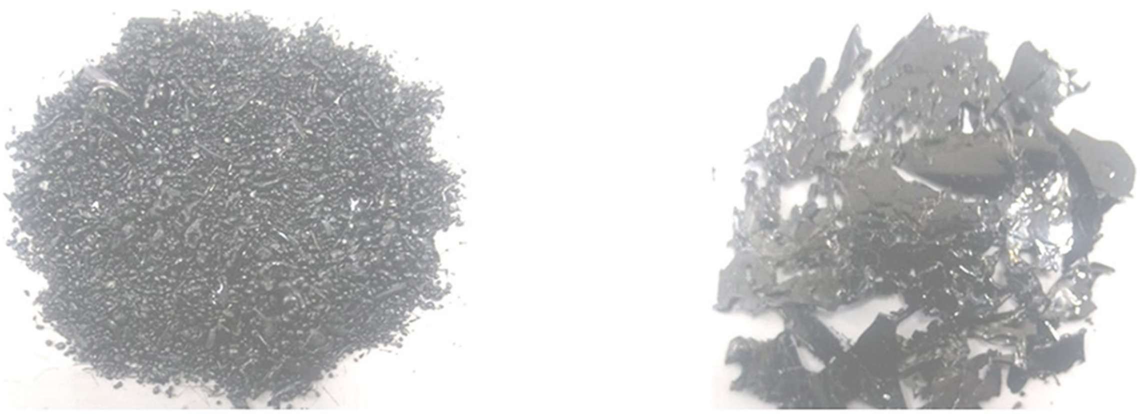
Investigations of the phase composition were carried out by the X-ray diffraction method on a DRON-6 diffractometer, CuK_α-radiation. The microstructure of the samples was investigated in a Jeol JCM-6490LA scanning electron microscope. Elemental analysis of the samples was carried out on an Optima 2000 DV inductively coupled plasma atomic emission spectrometer. The critical parameters of the samples were determined by measuring the temperature dependence of the resistivity using the four probe method and the temperature dependence of the magnetic susceptibility.

Results and Discussions

To identify the influence of laser and optical radiation on the initial precursors, on their phase formulations and the properties of superconducting ceramics, melting and hardening of the melt were carried out in various ways. In the first case, for comparative analysis, the melting of the initial mixture was carried out in the corundum crucible at a temperature of 1050-1100°C for 4 hours, and the melt should be carried out by draining the melt on the rotating water-cooled disc, made of stainless steel. As a result of quenching the melt, the following samples were obtained: when quenching the melt between copper rolls, plates were obtained with a thickness of about 0.1 mm, and when the spray washed, the precursors were mainly obtained in the form of a ball-shaped form (sphere) with a diameter of up to 1.5 mm (about 80%), as well as in the form of pieces of arbitrary shape and dimensions up to 3 mm, the plates with a thickness of 01-0.3 mm, a needle up to 10 mm (Figure 1).

The initial precursors presented: plates with a thickness of about 0.1 mm; The needles were a glassy phase, and pieces of samples consisted of crystallites.

In the second case, the sample melting was carried



a) b)

Figure 1 – Photo of the original precursors obtained by hardening melt: a) Chigue precursors obtained by the melt in the form of a sphere; b) pieces of arbitrary shape and sizes up to 3 mm, plates with a thickness of 0.1-0.3 mm, a needle up to 10 mm long

out by the UV laser beam. The diameter of the laser radiation beam was 2 mm, due to this, the melting was carried out locally. In this case, an intensive evaporation of the sample components was observed. The study of the phase composition of the molten part of the sample showed that reflexes of crystalline phases were manifested on diffractograms, as peaks, which are shown on the X-ray diffractograms (Figure 2). In this case, traces of the amorphous phase are observed on the X-ray diffractograms.

In the future, the sample melting was carried out with heating in a tubular furnace with a nichrome heater and the simultaneous impact of low-power UV laser radiation. The sample was placed inside the vertical tubular furnace and was heated to the intake temperature (840-850°C). Then, under the action of laser radiation, the lower part of the sample was melted and spread on the hardening device, i.e. spray. After hardening the precursors, also acquired a form in the form of plates, needles, puskets and fine balls. X-ray diffractograms of samples showed a similar phase composition as when melting in a muffle furnace.

In the third case, the sample melting was carried out under the action of a highly discovered radiant optical range flow, including UV, visible and near IR spectrum. The beam was directed to the surface of the edge of the sample and the sample melting, it was raised on the hardening device. Practical in all experiments, all challenged samples acquired a similar configuration. On diffractograms of crystal phases (pieces, thick-walled plates, large balls), various X-ray intensity were observed, and differences were observed due to the manifestation of additional low-intensity reflexes. The study of the elemental composition of the samples was shown that in the precursor materials obtained by melting in the muffle furnace and tempered on the cooling devices, a small deviation (decrease) from the stoichiometric composition of calcium was observed and a decrease in 10-15% oxygen content.

On the sample molten under the action of UV laser radiation, an intensive evaporation of the sample components was observed. Because of the small size of the beam diameter (2 mm), the complete melting of the sample for quenching could not be obtained, the sample melting was carried out only locally. The elemental composition of the cations in the melted part of the sample was not noticeably different from the stoichiometric. There was an increase in oxygen content by 3-5%. On the precursor materials obtained by melting and hardening, with the help of heating in a muffle furnace and exposure to a low-power UV laser radiation, the element formulations of the samples were not noticeably different from the stoichiometric. The oxygen content also turned out to be within the normal range.

On the precursors materials obtained by the melt chain under the action of high-definite radiation of the optical range, comprising a UV, visible and IR spectrum region, there were small deviations on the

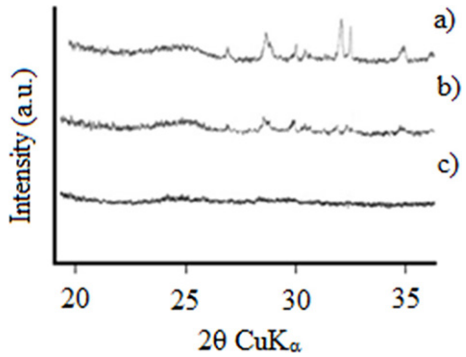


Figure 2 – The X-ray diffractograms of the initial precursor materials, poured from the melt: a) the surface of the melted sample with laser radiation; b) precursors in the form of a sphere; c) plates

cationic composition, according to the composition of calcium and lead. As for oxygen, it was observed an increase in its content by 15-20% relative to the stoichiometric composition.

In the future, for the synthesis of superconducting ceramics, the initial precursors based on amorphous precursors (plates, needles, balls) were flashed to dispersion of less than 2 microns and pressed into a tablet with a diameter of 10 mm and a thickness of 2.5 mm. The synthesis of superconducting phases was carried out by heat treatment (in isothermal mode) in the temperature range from 700°C to 850°C, with a shutter speed from 25 hours to 160 hours (with intermediate mistress).

For the nominal composition $Bi_{1,7}Pb_{0.3}Sr_2CaCu_2O_\delta$ (2212), the superconducting phase 2212 started to form at temperatures above 700°C. The temperature of the maximum rate of formation of the superconducting phase 2212 was in the temperature range of 850-850°C. At the same time, the maximum formation rate was manifested for samples prepared on the basis of the initial precursor materials obtained under the action of laser radiation and a radiant flux of the optical range (Figure 3a). After 25-30 hours of thermal annealing on the diffractogram, reflexes belonging to only phase 2212 were manifested. In samples prepared on the basis of precursor materials obtained in the crucible, the content of HTSC P phase 2212 was only about 35-40%. The total formation of the phase 2212 was carried out by prolonged thermal annealing (90-100 hours).

For nominal composition $Bi_{1,7}Pb_{0.3}Sr_2Ca_2Cu_3O_\delta$ (2223), the mechanism for the formation of HTSC phases passed according to homologous series, according to Scheme 2201 → 2212 → 2223. In all the samples under study, the superconducting phase 2212 began to form at temperatures above 700°C. At a temperature of 780-800°C, on samples obtained on the basis of precursor materials using UV laser radiation and a radiant flux of the optical range began to form a Phase Phase 2223. In samples obtained on the basis of precursor materials melted in the muffle furnace appeared only traces 2223 phases. With increasing

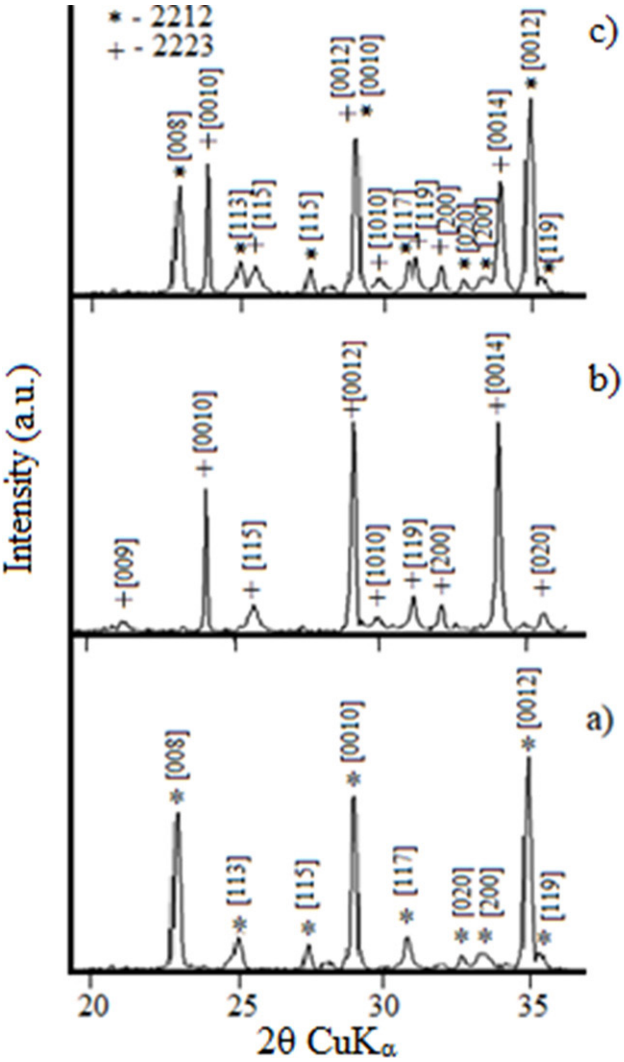


Figure 3 – X-ray diffractograms of samples of Bi-HTSC ceramics: a) Composition 2212, synthesized with the use of UV laser and optical radiation range; b) Composition 2223, synthesized using UV laser and optical radiation range; C – composition 2223, synthesized on the basis of precursors in a muffle furnace. Temperature dependences of the resistance of samples of HTSC; c) Sostables 2212 (1) and 2223 (2), synthesized from melts under the action of UV laser and optical radiation range

temperature to 846-848°C, the intensity of phase formation 2223 increased dramatically. Under the exposure time 25 hours, the phase of 2223 in samples using the UV laser radiation and the radiant flux of the optical range was about 40-50%, and at 50 hours of thermal annealing, the phase 2223 reached 70-80% and single-phase samples were obtained at annealing duration 85 hours and 70 hours, respectively (Figure 3b). As for the formation of Ceramics HTSC, synthesized on the basis of the initial precursors, obtained by the melt chain in the muffle furnace, even with a long annealing (more than 120 hours), the sample consisted of superconducting phases 2212 and 2223 (Figure 3c).

According to the intensity of X-ray reflections of the compositions: 2212 (008; 0010; 0012) and 2223 (0010; 0012; 0014) It can be judged that HTSC ceramics have a high texture (80-85%) in the direction of the [00I] plane, which may be positive Influence the current characteristics of superconducting ceramics. Studies of critical parameters of HTSC ceramics of nominal compounds 2212 and 2223 shows that the transition temperature to the superconducting state for sample 2212 is 89 K, and for sample 2223 is 107 K (Figure 4a).

As for the sample on the basis of precursor materials obtained by melting in a muffle furnace, the phase formation rate was much slower. With the duration of the synthesis of the sample120 hours, on the diffractogram, in addition to the main superconducting phase 2223, the reflexes of the low-temperature superconducting phase 2212 were present, as well as low-constracy reflexes of impurity non-top conduction phases. With an increase in exposure time up to 160 hours, the main superconducting phase was phase 2223, and the phase traces of 2212 were present. Measurement of the temperature dependence of the heat-treated sample obtained from the melt in the muffle furnace at a temperature of 848°C and the duration of annealing 120 hours shows the presence of a mixture of superconducting phases 2212 and 2223 (Figure 4b).

Comparative analysis of the results of the synthesis of HTSC compounds based on precursor materials obtained in various ways showed that the rate of formation of superconducting phases with high critical parameters (2212 and 2223 phases) 2-2.5 exceeds in the samples of Ceramics synthesized on the basis of precursor materials, The laser radiation obtained under the influence of the UV laser radiation and the radiant flow of the spectral composition of the optical ranges compared with the Ceramics SHA, synthesized by the standard solid-phase method or synthesized based on the precursor materials obtained from the melt in the muffle furnace. The studies of the elemental composition according to cations show that a strong deviation from the stoichiometric composition during melting of the nominal compositions 2212 and 2223 was not detected, and according to the authors [7-10], the formation of wide areas of homogeneity by cationic substitution for 2212 and 2223 compositions is observed. Consequently, non-essential disorders of the cationic composition cannot affect the rate of phase formation. In this case, the speed can affect the oxygen content in precursor materials. In the work of [8], it is noted that in the process of melting materials in molded methods in the synthesis of bismuth-containing HTSC Ceramics glass-crystalline, the oxygen content in the melt occurs, and monovalent copper is $R(Cu^{+})=Cu^{+}/(Cu^{2+}+Cu^{+})=0.7-0.8$. Therefore, in samples of precursors obtained by melting in a muffle furnace, the oxygen content was less than 10-15%, compared with the stoichiometric composition and the rate of formation of superconducting phases

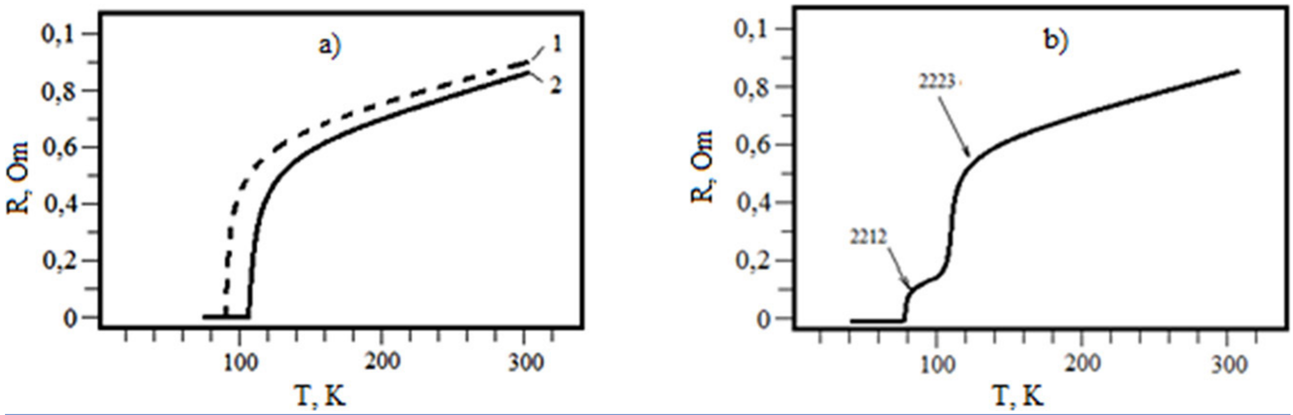


Figure 4 – Temperature dependences of the resistance of samples of HTSC: a) Stables 2212 (1) and 2223 (2), synthesized from melts under the action of UV laser and optical radiation range; b) composition 2223, synthesized from melt in a muffle furnace at a temperature of 848°C, 120 hours

2212 and 2223 were 2 or more times slower. Perhaps a strong deficiency of oxygen in dense precursor materials reduces the rate of the process of forming a superconducting structure, which is associated with a slowdown oxygen diffusion into the structure of a dense sample during thermal processing. As for the precursor materials obtained under the influence of the UV laser radiation and a highly discovered radial flux of the optical range, then the oxygen is observed in them. Perhaps an excess of oxygen in precursor materials is associated with the following factor: first, under the action of anisotropic effects of radiation, the melting process occurs on the thin surface of the sample in the atmospheric oxygen medium, therefore the solubility of oxygen in the melt increases. Secondly, under the action of high-intensity laser and optical radiation, the melt overheating to a temperature of 1250-1300°C, as well as the ionization of atmospheric oxygen to form ozone, which can lead to a more increase in oxidative properties, and may also increase the solubility of ozone in the melt. In the presence of ozone, the cations of variable valence can move to a state with a higher valence, respectively, the rate of formation of superconducting phases can increase.

Conclusion

To identify the influence of laser and optical radiation on the composition and properties of the initial precursors, bismuth HTSC of ceramics obtained under the action of the UV laser radiation and the emission energy of a highly determined optical radiation spectrum containing the UV, visible and near IR region, and were also investigated, synthesized source Materials-precursors of a glassy state. The studies of the elemental composition of the precursors were shown that there were no significant differences in the cationic composition, while the oxygen deficiency was observed in the precursor materials obtained in the muffle furnace, and the oppositely of this, in the precursor materials obtained under the action of Laser radiation And the energy of high-tender radiation was observed excess oxygen. Synthesis of HTSC Ceramics based on the precursors obtained showed that the rate of formation of superconducting phases increases in Ceramic HTSC synthesized on the basis of precursor materials with overpressure of oxygen 2-2.5 times, compared with the materials of the precursors obtained in the muffle furnace. On diffractograms of the samples, particles are observed, with the predominant texture along the crystallographic plane in the direction of [00i].

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Жоғары температуралы асқын өткізгіш қосылыстардың түзілуіне және қасиеттеріне лазерлік және оптикалық сәулеленудің әсерін зерттеу

[1]**НОГАЙ Адольф Сергеевич,** *ф.-м.ғ.д., профессор, nogay06@mail.ru,*
[1]**УСКЕНБАЕВ Данияр Есанкулұлы,** *PhD, қауымдастырылған профессор, usdan01@mail.ru,*
[1]***ТАТКЕЕВА Галина Галимзяновна,** *т.ғ.д., кафедра меңгерушісі, tatkeeva@mail.ru,*
[2]**АЛЕКСАНДРОВСКИЙ Владимир Владимирович,** *ғылыми қызметкер, alexandrovsky@inbox.ru,*
[1]**ЖЕТПИСБАЕВ Қайратбек Уристембекұлы,** *аға оқытушы, 79kairatbek@mail.ru,*
[1]**УСКЕНБАЕВ Алишер Даниярұлы,** *магистрант, Д.alish_us@mail.ru,*
[1]С. Сейфуллин атындағы қазақ агротехникалық университеті, Қазақстан, 010011, Нұр-Сұлтан, Жеңіс даңғылы, 62,
[2]МИРЭА – Ресей технологиялық университеті, Ресей, 119454, Мәскеу, Вернадский даңғылы, 78,
**автор-корреспондент.*

Аңдатпа. *Жұмыста балқымадан алынған аморфты және кристалды бастапқы материалдардан* $Bi_{1.6}Pb_{0.4}Sr_2Ca_{n-1}Cu_nO_y$ *(n = 1, 2) қосылыстарының висмут өткізгіштерін синтездеу нәтижелері келтірілген. Қыздыру көзі ретінде ультракүлгін, көрінетін және спектрдің жақын инфрақызыл аймақтары бар кең спектрлі диапазондағы лазерлік және оптикалық сәулелер қолданылды. Бастапқы материалдардың элементтік құрамы және бастапқы материалдардың өткізгіш фазалардың пайда болуына, сондай-ақ өткізгіш керамиканың критикалық параметрлеріне әсері зерттелді. Балқымадан бастапқы материалдарды алу жағдайларына байланысты катиондық құрамы бойынша үлгілердің стехиометриялық құрамының шамалы бұзылуы және оттегінің айтарлықтай өзгеруі байқалады, бұл өз кезегінде өткізгіш фазалардың пайда болу процесіне және өткізгіш керамиканың қасиеттеріне әсер етеді.*

Кілт сөздер: *жоғары температуралы өткізгіштік, лазерлік сәуле, құрылым, керамика, морфология, дифрактограмма, фаза.*

Исследованние влияния лазерного и оптического излучений на образование и свойства высокотемпературных сверхпроводящих соединений

[1]**НОГАЙ Адольф Сергеевич,** *д.ф.-м.н., профессор, nogay06@mail.ru,*
[1]**УСКЕНБАЕВ Данияр Есанкулович,** *PhD, ассоциированный профессор, usdan01@mail.ru,*
[1]***ТАТКЕЕВА Галина Галимзяновна,** *д.т.н., зав. кафедрой, tatkeeva@mail.ru,*
[2]**АЛЕКСАНДРОВСКИЙ Владимир Владимирович,** *научный сотрудник, alexandrovsky@inbox.ru,*
[1]**ЖЕТПИСБАЕВ Кайратбек Уристембекович,** *старший преподаватель, 79kairatbek@mail.ru,*
[1]**УСКЕНБАЕВ Алишер Даниярович,** *магистрант, Д.alish_us@mail.ru,*
[1]Казахский агротехнический университет им. С. Сейфуллина, Казахстан, 010011, Нур-Султан, пр. Женис, 62,
[2]МИРЭА – Российский технологический университет, Россия, 119454, Москва, пр. Вернадского, 78,
**автор-корреспондент.*

Аннотация. *В работе приведены результаты по синтезу висмутсодержащих сверхпроводников* $Bi_{1.6}Pb_{0.4}Sr_2Ca_{n-1}Cu_nO_y$ *(n = 1, 2) из аморфных и кристаллических исходных материалов, полученных из расплава. В качестве источника нагрева были использованы лазерное и оптическое излучения широкого спектрального диапазона, содержащие ультрафиолетовую, видимую и ближнюю инфракрасную область спектра. Исследованы элементный состав исходных материалов и влияние исходных материалов на образование сверхпроводящих фаз, а также на критические параметры сверхпроводящей керамики. Установлено, что в зависимости от условий получения исходных материалов из расплава происходит незначительное нарушение стехиометрического состава образцов по катионному составу и существенное изменение по кислороду, что в свою очередь влияет на процесс образования сверхпроводящих фаз и свойства сверхпроводящей керамики.*

Ключевые слова: *высокотемпературная сверхпроводимость, лазерное излучение, структура, керамика, морфология, дифрактограмма, фаза.*

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