

DEPENDENCE OF THE CRITICAL TEMPERATURE ON THE SYNTHESIS
PARAMETERS OF $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_n\text{Cu}_{n-1}\text{O}_y$ CUPRATES.

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Abstract. This article describes the preparation of BSCCO cuprate based on bismuth, the most common representative of high-temperature superconductors, and what should be paid attention to when preparing it.

The advantages and disadvantages of obtaining BSCCO cuprate by the solid-phase reaction method are presented. At the same time, it was pointed out why the phase of the transition of this type of cuprates to superconductivity manifests itself in several phases, and not in one, and these reasons can be explained simply. BSCCO has several cuprate extraction technologies. The article sequentially presents the stages of obtaining by the method of solid-phase reaction.

Key words: Bismuth, high-temperature superconductor, cuprate, calcination, sintering, solid-state reaction, phase, macromolecule, crystal lattice.

Introduction. Since the discovery of high- T_c superconductors in the cuprate family, considerable effort has been devoted to improving the synthesis process using the solid-state reaction technique. There are various technologies for obtaining high-temperature superconductors; however, the solid-state reaction method—based on mixtures of solid precursor materials such as oxides, carbonates, and others—is the most widely used approach for synthesizing polycrystalline bulk superconducting materials. To prepare superconducting materials, the solid powders are thoroughly mixed and heated, then mixed again. Subsequently, they are heat-treated in a furnace at temperatures ranging from 800 °C to 950 °C for several hours [3].

The preparation of cuprate superconductors generally follows the procedure described above. However, in much of the literature on this technology, the terms *calcination* and *sintering* are frequently used. Calcination refers to the thermal treatment of a solid chemical compound (for example, mixed carbonates or oxides). This process serves several purposes, ranging from the removal of chemically bound (crystalline) water to the volatilization of impurities from the precursor materials, thermal decomposition, and even phase transformations. Calcination enables manufacturers to control the properties of the final product, minimize contamination from waste or recycled materials, and achieve several other processing advantages. Sintering, on the other hand, occurs as part of the manufacturing process for metals, ceramics, plastics, and other materials. During sintering, atoms in the material diffuse along particle boundaries, bonding the particles together and forming a single solid body. The sintering temperature must be very close to the melting point of the material.

Bismuth-based superconductors (BSCCO) were discovered around 1988. The BSCCO superconductor family comprises three phases with the generalized chemical formula $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{2n+4+x}$, where $n = 1, 2,$ and 3 (here, n denotes the number of CuO_2 layers in the crystal structure, which undergo superconducting phase transitions at approximately 20 K, 85 K, and 110 K, respectively) [2].

Research Object and Methods.

BSCCO is a cuprate superconductor and represents an important class of high-temperature superconductors characterized by a two-dimensional layered structure. Superconductivity occurs

within the copper-oxide (CuO_2) planes. BSCCO and YBCO are among the most extensively studied cuprate superconductors.

In the course of the present study, cuprates based on the formulas $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_y$ and $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_y$ (where $n = 5, 9, 15, 30$) were synthesized as research objects. To obtain the superconducting state in these compounds, the solid-state reaction method—one of the widely used preparation techniques—was employed.

When Pb is added to the precursor mixture during the preparation of bismuth-based cuprates, modifications occur in the crystal lattice. The macromolecular structure of $\text{B}(\text{Pb})\text{SCCO}$ is illustrated in the corresponding figure. Only when the elements are arranged in this specific lattice configuration does the resulting crystal undergo a superconducting phase transition at the critical temperature (T_c).

For the oxide and carbonate mixture to form such a crystal lattice, the mixture is gradually heated to a high temperature and subjected to calcination. Once this process is sufficiently completed, and taking into account the melting temperatures of each constituent, the mixture is sintered at a temperature close to its melting point for a certain period of time.

Results Obtained and Their Analysis.

In the $\text{Bi}(\text{Pb})\text{-Sr-Ca-Cu-O}$ system, the sample was prepared by the solid-state synthesis method. High-purity powders of Bi_2O_3 (99.9%), PbO (99.9%), SrCO_3 (99.9%), CaO (99.9%), and CuO (99.9%) were taken in appropriate proportions. Based on the elemental composition corresponding to the formula $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_y$, the mass fractions of the components in a 5 g mixture are presented in Table 1.

Table 1

	Elem ent n=5	Mas s fraction (gr)	Eleme nt. n=9	Ma ss fraction (gr)	Eleme nt n=15	Ma ss fraction (gr)	Ele ment n=30	Mas s fraction (gr)
	Bi_2O_3	2,24 007	Bi_2O_3	1,6 8935	Bi_2O_3	1,2 342	Bi_2O_3	0,73 747
	PbO	0,26 825	PbO	0,2 023	PbO	0,1 478	PbO	0,08 831
	CaO	0,67 397	CaO	1,0 1655	CaO	1,2 9967	CaO	1,60 866
	CuO	1,19 503	CuO	1,6 2221	CuO	1,9 7526	CuO	2,36 056
	SrCO_3	0,88 714	SrCO_3	0,6 6904	SrCO_3	0,4 8879	SrCO_3	0,29 206

Similarly, the mass fractions of the constituent elements for the mixture based on the formula $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_y$ were calculated and are presented in Table 2.

Table 2

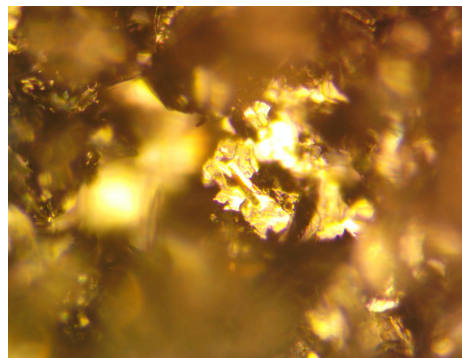
	Elem ent n=5	Mas s fraction (gr)	Eleme nt. n=9	Mas s fraction (gr)	Ele ment n=15	Mas s fraction (gr)	Ele ment n=30	Mas s fraction (gr)
	Bi_2O_3	2,34 5853	Bi_2O_3	1,77 5398	Bi_2O_3	1,30 0883	Bi_2O_3	0,77 9821
	PbO	0,19 8298	PbO	0,15 0077	PbO	0,10 9965	PbO	0,06 5919
	CaO	0,66	CaO	1,00	CaO	1,28	CaO	1,60

		4278		5484		9305	O	0967
	CuO	1,17 7845	CuO	1,60 4559	CuO	1,95 9507	Cu O	2,34 9274
	SrCO	0,87 4388	SrCO	0,66 1758	SrC O ₃	0,48 4888	Sr CO ₃	0,29 0669

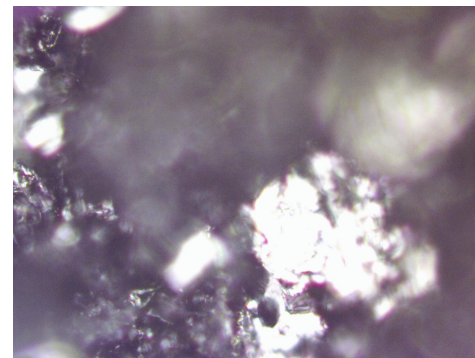
The components with the masses indicated in the above tables were first thoroughly mixed. The mixture was then pressed into disc-shaped pellets using a specially prepared mold with a diameter of $d = 15$ mm and a thickness of $h = 2.5$ mm, each pellet having a mass of $m = 1$ g. The pressing was carried out at a pressure of $P = 3.922$ MPa. To prevent air layers from remaining within the sample, the pressure was gradually increased during the pressing process. The prepared samples of specific shape were then arranged in a designated order for placement in the furnace for sintering.

During the sintering process, the mixture must be heated to temperatures very close to its melting point. This is necessary because the elements in the mixture rearrange and form a common macromolecular crystal lattice.

When the pressed samples were heated at a temperature of $t = 845$ °C for 16 hours, it was observed that the melting process had begun in the samples with $n = 5$, and the heating was therefore stopped. Subsequently, the samples were tested to determine whether they had transitioned into the superconducting state. For this purpose, the prepared samples were examined at the boiling temperature of liquid nitrogen, $T = 77$ K. No Meissner effect was observed in the samples; therefore, to determine the reason for its absence, the samples were examined using an electron microscope.



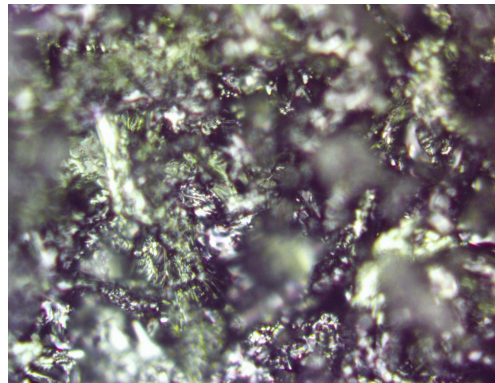
a) Front view of the sample



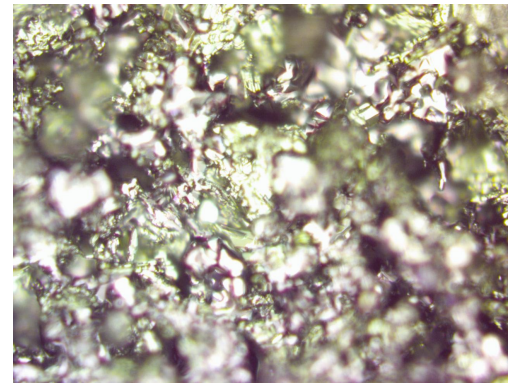
b) Rear view of the sample.

1-Fig. 50× magnified image of the $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_7$ ($n = 5$) sample obtained using a Euromex iScope microscope.

Examining the image obtained with the microscope, it can be seen that the elements in the mixture are not completely homogenized; the elements remain partially separate. Since this sample has $n = 5$, the amounts of Ca and Cu are relatively low, which is why the sample began to melt at $T = 845$ °C. Because the melting process had started, the constituent elements partially melted and accumulated in certain regions. Let us now consider the other samples as well.



a) Front view of the sample



b) Rear view of the sample.

Fig. 2. 50× magnified image of the $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_y$ ($n = 30$) sample obtained using a Euromex iScope microscope.

Since $n = 30$ in this sample, the amounts of Ca and Cu constitute a larger fraction of the mixture. Therefore, this sample did not begin to melt at $T = 845^\circ\text{C}$. In the microscope image, the individual elements of the mixture can still be observed, but unlike the $n = 5$ sample, the elements remain in the state in which they were originally mixed, without significant segregation.

Conclusion.

After reviewing the literature on high-temperature superconductor synthesis technologies and performing the actual preparation of BSCCO cuprates, the following conclusions were drawn. Producing BSCCO cuprates is somewhat more difficult and delicate compared to other cuprates, such as YBCO. The challenges arise because these superconductors contain three or more phases with similar layered structures. During synthesis, structural interconnections and defects—such as imperfect bonding—occur, making it difficult to isolate a single superconducting phase. Poor compositional control, chemical inhomogeneity, time-consuming procedures, coarse particle sizes, the incorporation of mixtures during grinding, and high-temperature requirements ($>1000^\circ\text{C}$) are inherent limitations of the ceramic method. Neglecting any one of these factors can prevent achieving the desired superconducting properties.

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