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Investigation of the Phase Change during Solid Phase Synthesis of $Bi_{1.7}Pb_{0.3}Sr_2Ca_{n-1}cu_no_y$ Bismuth-Based Cuprates

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Abstract: There are different preparation technologies of any kind of cuprates. This article talks about the technology of obtaining bismuth-based cuprates and the aspects that need to be paid attention to in this process. The advantages and disadvantages of obtaining BSCCO cuprate by solid state reaction method are presented. At the same time, it was mentioned why cuprates of this type of superconducting phase appear in several phases instead of one, and these reasons are explained in a simple way. The steps for obtaining BSCCO cuprate by the solid state reaction method are listed in sequence. The constituents of the BSCCO composite material, the role of each element in the mixture was studied under an optical microscope. The formation of the overall structure near the melting temperature was analyzed by microscopic images.

Keywords: Solid Phase Synthesis, Investigation.

Introduction BSCCO as a new class of superconductor was discovered around 1988 by Hiroshi Maeda and colleagues at the National Research Institute for Metals in Japan, though at the time they were unable to determine its precise composition and structure [1]. The BSCCO family of superconductors includes three phases with the generalized chemical formula $Bi_2Sr_2Ca_{n-1}Cu_nO_y$, where n=5 (where n is the number of CuO_2 layers in the crystal structure, they transition to superconducting phases at 20, 85, and 110 K, respectively) [4].

Since the discovery of high-Tc superconductors in the cuprate family, many efforts have been made to improve the synthesis process through solid-state reaction technology. There are various technologies for obtaining high-temperature superconductors, and the solid state reaction method, polycrystalline casting from a mixture of solid starting materials such as oxides, carbonates, etc., is the most common method for the synthesis of superconducting materials.

In the process of preparing superconductors, cuprate is prepared in the same order as mentioned above. But in many literatures about this technology, the terms calcination and sintering are often used. Calcination refers to heat treatment of a solid chemical compound (for example, mixed carbonate, oxides). It helps to achieve a number of goals, from the removal of chemically bound (crystalline) water to the volatilization of contaminants from the source material, thermal decomposition, and even phase change. Calcination allows industrial manufacturers to control the properties of the final product, minimize the contamination of waste materials or processing products, and many other possibilities. Sintering occurs as part of the manufacturing process used with metals, ceramics, plastics, and other materials [3]. Atoms in the material spread along its boundaries, unite the particles and form one solid piece. The sintering temperature should be very close to the melting point of the material.

Scientific novelty of research.

During the research, many scientific works in this field were studied. In our research, it is aimed to observe the change in the critical temperature Tc when lead is added to bismuth-based cuprates. At the same time, the mechanism of placement of lead in the cuprate macromolecule was studied.

Molecular structure of BSCCO

BSCCO is a cuprate superconductor, an important class of high-temperature superconductors with a two-dimensional layered structure. Superconductivity occurs in the plane of copper oxide [3]. BSCCO and YBCO are the most studied cuprate superconductors. During the scientific research, cuprates based on the formula $Bi_{1,7}Pb_{0.3}Sr_2Ca_{n-1}Cu_nO_y$ and $Bi_{1,6}Pb_{0.4}Sr_2Ca_{n-1}Cu_nO_y$ (where n=5;9;15;30) were taken as the object of research, and to make this mixture a superconducting state state reaction method was used [3].

The macromolecule of B(Pb)SCCO is depicted in the figure below, when Pb is added to the mixture in the preparation of bismuth-based cuprate. Only if the elements are placed in this order in the lattice, the formed crystal will pass to the superconducting phase at the critical temperature T_C (Fig. 1).

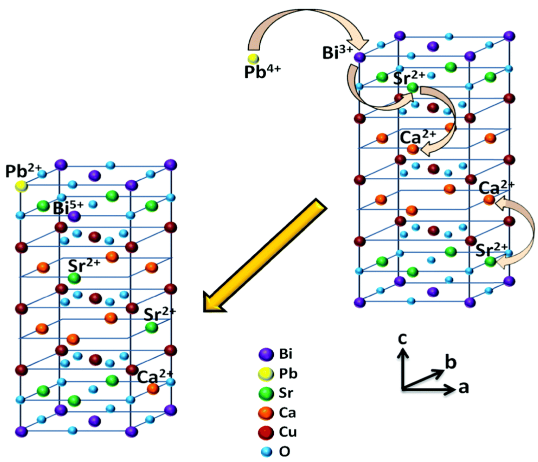


Fig. 1. Polycrystalline lattice structure and arrangement of element atoms of B(Pb)SCCO. Mechanism of placement of Pb in BSCCO crystal lattices.

Important aspects in the formation of polycrystal

In order for the mixture of oxides and carbonates to form such a crystal lattice, the mixture is slowly heated to a high temperature and calcined. After this process is sufficiently carried out, knowing the melting temperature of each composition, the mixture is sintered at a temperature close to the melting temperature for a certain time [2].

Mixing each element in the order of increasing mass is a very important factor in the preparation of composite material, otherwise the elements of the mixture may not form a complete macromolecule.

Experimental Method

There are many methods in the world experience of obtaining superconductors. We decided to take samples using the solid phase method and used the technology of Turkish scientists who achieved results in this method. A. Coşkun et al.'s research and its results are presented using the technology in the article titled Structural, Magnetic, and Electrical Properties of $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10+x}$ Superconductor Prepared by Different Techniques [6].

As mentioned above, the solid state reaction method was used to obtain the samples. The constituent elements of the selected composite material were selected according to the Bi(Pb)-Sr-Ca-Cu-O system. For this purpose, powders of high purity Bi_2O_3 (99.9%), PbO (99.9%), $SrCO_3$ (99.9%), CaO (99.9%) and CuO (99.9%) were taken in appropriate proportions. The mass fractions of elements in 5 g of the mixture based on the formula $Bi_{1,6}Pb_{0.4}Sr_2Ca_{n-1}Cu_nO_y$ are given in Table 1 below [6].

Table 1. ***The mass fraction of the elements in the mixture***

№	Element n=5	Mass fraction (gr)	Element n=9	Mass fraction (gr)	Element n=15	Mass fraction (gr)	Element n=30	Mass fraction (gr)
1	Bi_2O_3	2,24007	Bi_2O_3	1,68935	Bi_2O_3	1,2342	Bi_2O_3	0,73747
2	PbO	0,26825	PbO	0,2023	PbO	0,1478	PbO	0,08831
3	CaO	0,67397	CaO	1,01655	CaO	1,29967	CaO	1,60866
4	CuO	1,19503	CuO	1,62221	CuO	1,97526	CuO	2,36056
5	$SrCO_3$	0,88714	$SrCO_3$	0,66904	$SrCO_3$	0,48879	$SrCO_3$	0,29206

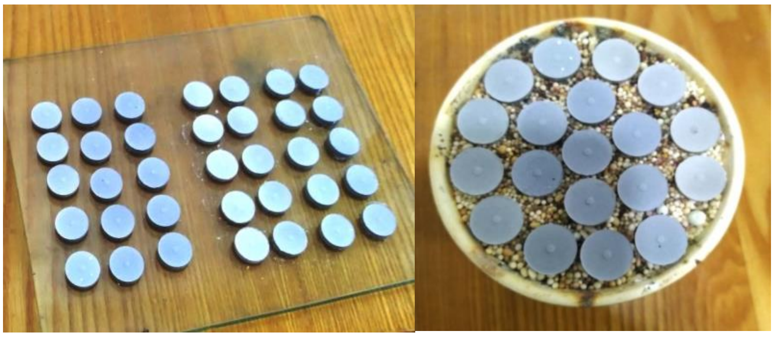
In the same way, the mass fractions of the elements of the mixture based on the formula $Bi_{1,7}Pb_{0.3}Sr_2Ca_{n-1}Cu_nO_y$ were calculated (Table 2).

Table 2. ***The mass fraction of the elements in the mixture***

№	Element n=5	Mass fraction (gr)	Element n=9	Mass fraction (gr)	Element n=15	Mass fraction (gr)	Element n=30	Mass fraction (gr)
1	Bi_2O_3	2,34585	Bi_2O_3	1,77539	Bi_2O_3	1,30088	Bi_2O_3	0,77982
2	PbO	0,19829	PbO	0,15007	PbO	0,10996	PbO	0,06591
3	CaO	0,66427	CaO	1,00548	CaO	1,28931	CaO	1,60096
4	CuO	1,1778	CuO	1,6045	CuO	1,9595	CuO	2,3492

		4		5		1		7
5	$SrCO_3$	0,87438	$SrCO_3$	0,66175	$SrCO_3$	0,48488	$SrCO_3$	0,29067

The elements in the mass given in the above tables were first thoroughly mixed. The mixture was pressed in a specially prepared mold with a diameter of d=15mm, a thickness of h=2.5mm, and the mass of each disc was m=1g. Pressing was carried out at P=3.922 MPa. In order not to leave an air layer in the sample, the pressure was gradually increased during the pressing process. The ready-made samples of special shape were placed in the specified order in order to put them in the oven for sintering.



a **b**

Fig. 2. ***The appearance of samples pressed in a special mold (a) and the condition prepared for placing it in the oven (b).***

In the process of sintering, it is necessary to heat the mixture to values very close to the melting temperature. Because only then the elements in the mixture are rearranged and form a general macromolecular crystal lattice.

Results and disscusion

When the pressed samples were heated for 16 hours at a temperature of t=845°C, it was found that the melting process started in the samples with n=5, and the heating was stopped. Later, it was checked whether the sample will pass into the state of superconductivity or not. The sample prepared for this purpose was tested at the boiling temperature of liquefied nitrogen T=77 K. In this case, the Messner effect was not observed in the sample. In order to find out why it was not observed, the samples were examined under an electron microscope.

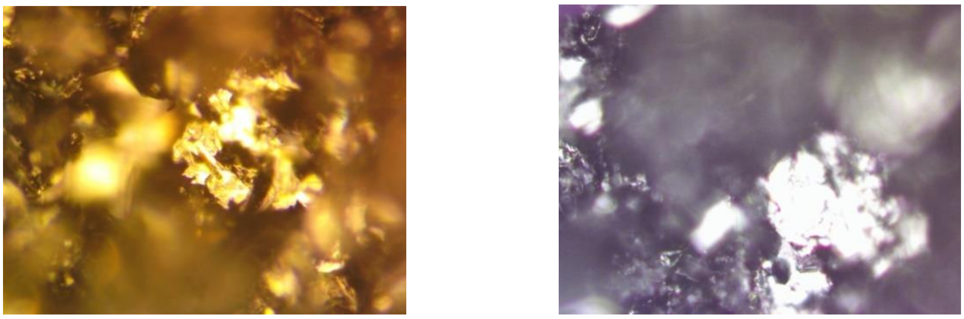


Fig. 3. ***500x magnification image of*** $Bi_{1,7}Pb_{0.3}Sr_2Ca_{n-1}Cu_nO_y$ ***(n=5) sample on Euromex iScope microscope.***

If you pay attention to the image taken under the microscope, you can see that the elements in the mixture are not completely mixed, the elements are separate. Since n=5 in this sample, the amount of Ca and Cu is relatively low, so the sample started to melt at T=845°C. Due to the process of melting, the elements of the composition melted separately and gathered in certain parts. Now let's look at other examples.

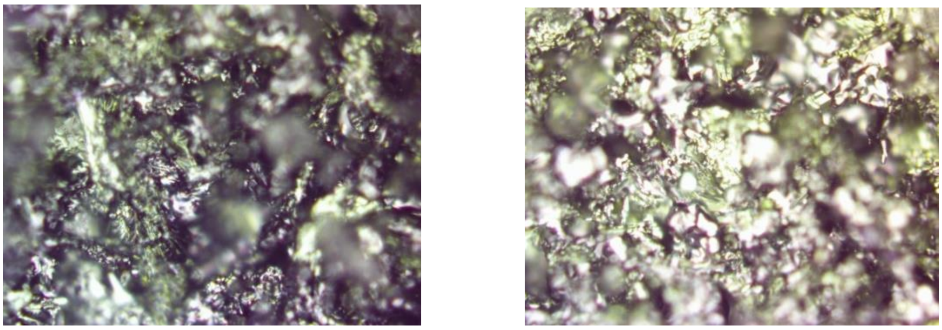


Fig. 4. ***500x magnification image of*** $Bi_{1,7}Pb_{0.3}Sr_2Ca_{n-1}Cu_nO_y$ ***(n=30) sample on Euromex iScope microscope.***

Since n=30, the amount of Ca and Cu in the mixture is more. Therefore, this sample did not melt at T=845^{o}C. It is also possible to observe the separate states of the elements of the mixture in its photo under the microscope, but the difference from the n=5 sample is that the elements remain as they are in the mixed state.

Conclusions

This conclusion was reached after researching the sources of high-temperature superconductor extraction technologies and following the BSCCO cuprate extraction process in practice. It turned out that the extraction of BSCCO cuprate is somewhat difficult and delicate compared to other types of cuprates, such as YBCO. In these superconductors, problems arise due to the presence of three or more phases with a similar layered structure. Syntactic interdependencies and defects, such as fusion faults, occur during synthesis and it is difficult to isolate a single superconducting phase. Poor compositional control, chemical uniformity, time-consuming process, coarse particle size, inclusion of impurities during grinding, and high temperature (>1000^{o}C) requirements are the specific disadvantages of the ceramic method. Because if any of these is neglected, the intended goal cannot be achieved.

In order to form the above polycrystalline grid, these samples were ground again and heated again in the oven. In the following articles, we will mention the observation of the Messner effect in the sample and the advantages of this type of cuprates over others. Our scientific research continues.

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