Synthesis and Properties of Bi-HTSC synthesized on the basis of precursors obtained by melt quenching

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Abstract. The article presents the research results on the synthesis of HTSC ceramics using concentrated radiant radiation. It was established that all synthesized samples of the nominal composition Bi₁₋ₓPbxSr₂Caₙ₋₁CuₙOₚ (n = 3, 4, 5) the main phase was the superconducting phase Bi₁₋ₓPbxSr₂Ca₂CuₙOₚ, which has a critical temperature of the transition to the superconducting condition of 107 K. Also in samples of nominal compositions Bi₁₋ₓPbxSr₂Caₙ₋₁CuₙOₚ (n = 4, 5) showed traces of phases with higher Tc.

1. Introduction

After the discovery of high-temperature superconductivity (HTSC), various compositions (yttrium, bismuth, thallium, mercury, etc.) and methods for obtaining (superphase, melt, glass-crystalline, chemical, etc.) superconducting materials have been developed to increase the critical parameters (Jc) and the critical temperature (Tc). Among the above mentioned compositions, superconducting materials based on the Bi-Sr-Ca-Cu-O system are recognized as one of the most promising, since they are characterized by high critical characteristics, much less degradation, and consistency of the composition.

Analysis of the synthesis effect for conditions on the phase composition and properties of Bi-HTSC revealed certain advantages of melt technology. Synthesis in the liquid phase presence allows to obtain more dense and low-porous target materials [1]. When melt quenching, it is possible to obtain a material in the amorphous state [2, 3], which determines the prospect of grain size adjustment, a higher rate of interfacial interactions due to the contribution of the metastable initial condition, and the material increased density. In addition, such a method is more likely than a solid phase method to assume the possibility of obtaining a textured material [4, 5].

The results comparative analysis of the use of different methods for the synthesis of oxide materials (including oxide superconductors) in the melt shows certain advantages of using a concentrated radiant flux as the heating source [6–9]. With respect to the fulfillment of the requirements determined by the specificity of the peritectic reaction for the formation of HTSC compositions 2223, 2234, 2245, a positive argument of this approach is the possibility of maintaining and rapidly adjusting the stationary mode of synthesis in the presence of the liquid phase. The positive contribution is: the purity of the process, carrying out the process in a crucibleless way, the completeness of the reactions...
and the best homogenization in the melt, carrying out the synthesis at an equilibrium oxygen partial pressure, the inertialess control of the amount of the liquid phase, the possibility of creating a texture due to a sharp temperature gradient at the melt-solid interface, directed crystallization at a given rate of cooling of the melt, the realization of the glassy state and control of the amount of the crystalline phase of the irovaniem cooling rate [8].

The article purpose is to investigate the effect of melting, quenching and heat treatment conditions on the completeness of the formation of BSCCO nominal compositions 2223, 2234, 2245.

2. Experimental method

As a heating source, a concentrated radiant flux, created by a 10 kW xenon lamp, was used.

The initial charge of nominal compositions corresponding to Bi$_{1.3}$Pb$_{0.3}$Sr$_2$Ca$_{n-1}$Cu$_n$O$_y$ ($n = 3, 4, 5$) was prepared from oxides of Bi$_2$O$_3$, PbO, SrCO$_3$, CaO, CuO. Beam samples melting was carried out in a crucibleless manner on a water-cooled substrate. For quenching, the melt dispersion technique was used.

The phase composition was monitored by X-ray diffraction using a DRON-UM1 diffractometer, CuK$_\alpha$ radiation and Diffractometer Model Rigaku Co, Ltd., Tokyo, Japan. The microstructure was examined with Neophot and TEM microscopes, Model JEM-1200EX11, JEOL Co., Ltd., Tokyo, Japan). The critical temperature was measured by a four-contact method.

3. The obtained results and discussion

As a result of quenching, samples of three types were obtained: pieces measuring about 0.5 cm$^3$, plates 0.1 – 0.3 mm thick; Needles 1 - 10 mm in length and 0.1 – 0.4 mm in diameter. In the initial state after quenching, the phase composition of the pieces and plates was represented by amorphous and crystalline phases. The needles were completely amorphous.

The hardened precursors were heat treated at a temperature of 840–845 °C, 18–25 hours. After that, complete crystallization of the glass phase occurred in all the samples and the predominant formation of a phase of composition 2212 with unit cell parameters $a = 0.5427$ nm, $c = 0.5412$ nm, $c = 0.3135$ nm. From the described precursors, ceramic samples were prepared using traditional ceramic technology.

The samples microstructure of nominal composition 2223 (as an example) is shown in Figure 1a,b. The nature of crystallization of phases in samples-pieces and plates was similar. Their structure was characterized by the formation of blocks of about 120 – 140 μm in size, consisting of tightly packed plates (Figure 1a). Needles had a slightly different structure due to the formation of blocks of much smaller sizes, equal to 5 - 8 μm (Figure 1b).

![Figure 1. Samples microstructure of the initial composition 2223; plates (a) and needles (b) obtained by ultrafast quenching of the melt and heat treated at 845 °C, 25 hours.](image)

Synthesis of high-temperature superconducting phases was carried out by thermal treatment of ceramics samples in the form of a cylinder in the isothermal regime in the temperature range 845 – 850 °C in the interval 40–150 hours. According to X-ray analysis of samples [10], the main phase is 2223 (Figure 2). In addition to X-ray reflections related to phase 2223, additional reflexes are present in samples 2234 and 2245.
The parameters of elementary cells are determined and the features of the crystal lattice formation in dependence on the temperature-time parameters are investigated. The optimum time for formation of the superconducting phase in all the samples studied was 60–120 hours.

![Figure 2. Samples diffractogram of a high-temperature superconductor of the nominal composition Bi$_{1.7}$Pb$_{0.3}$Sr$_2$Ca$_{n-1}$Cu$_n$O$_y$ ($n = 3, 4, 5$).](image)

In the nominal composition samples corresponding to phases 2234 and 2245, a high-temperature modification is established, characterized by an identical crystalline structure, presumably tetragonal. These results allow one to assume a continuous mutual solubility of these phases at high temperatures [11].

The low-temperature form in the BSCCO samples of composition 2234 and 2245 corresponded to the ICDD data for 2234 with a monoclinic crystal structure.

All samples with a well-formed crystal structure were observed by the Meissner effect. The critical transition temperature for BSCCO samples of composition 2223 is determined to be 119 °K (Figure 3).

![Figure 3. The result is the measurement of the critical temperature of the transition to the superconducting state of a sample of a superconducting ceramics of the nominal composition Bi$_{1.7}$Pb$_{0.3}$Sr$_2$Ca$_2$Cu$_3$O$_y$ in an electromagnetic field H = 250 Oe.](image)

For the samples of superconducting ceramics of nominal compositions Bi$_{1.7}$Pb$_{0.3}$Sr$_2$Can-1Cu$n$O$y$ ($n = 4, 5$), the main phase was the superconducting phase of 2223, also had a transition to the superconducting condition of 107–110 K. However, traces of high-temperature superconducting phases up to 150 K were detected.
4. Conclusions

Based on amorphous precursors, a high-temperature superconducting ceramics of the nominal composition \( \text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_{2}\text{Ca}_{n-1}\text{Cu}_{n}\text{O}_{y} \) \((n = 3, 4, 5)\) was synthesized under the influence of a concentrated radiant flux. With an increase in the content of Ca and Cu, the temperature regime of the synthesis of superconducting phases decreases. It is established that for all synthesized samples the main phase is the superconducting phase \( \text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_{2}\text{Ca}_{2}\text{Cu}_{3}\text{O}_{y} \), which has a critical temperature of the transition to the superconducting condition of 107 K. It is assumed that in samples of the composition \( \text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_{2}\text{Ca}_{n-1}\text{Cu}_{n}\text{O}_{y} \) \((n = 4, 5)\) is present phase with \( T_{c} \) exceeding 107 K.

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