

BiFeO₃ Synthesized on the Solar Furnace

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Abstract

It is shown that ferrite of bismuth synthesized on the basis of precursors - iron and bismuth oxides melted on a solar furnace has a denser structure, a low coefficient of thermal expansion, in comparison with the traditionally synthesized bismuth ferrite.

Introduction

As the analysis shows, the materials of perovskite (Ti (Sr) BaO₃) and pyroxene (CaMgSi₂O₆) compositions synthesized from melt in the Big Solar Furnace (BSP) have a stable structure and increased mechanical and dielectric properties [1-3]. However, the synthesis of perovskite structures based on bismuth ferrite BiFeO₃ with magnetic properties using BSP has not been studied.

Synthesis and properties of bismuth ferrite BiFeO₃ are investigated quite widely [1-16]. However, the preparation of a single-phase sample of BiFeO₃ still presents a serious problem. For example, a material synthesized from a mixture of Bi₂O₃ + Fe₂O₃ always contains impurities Bi₂₅FeO₃₉ and Bi₂Fe₄O₉ not depending on the synthesis method [5-8]. The use of a mixture with a large excess of Bi₂O₃ also did not lead to a single-phase BiFeO₃ ferrite [9]. In [10] it is noted the difficulty of preparation of single-phase BiFeO₃, which is associated with the features of the system state diagram Bi₂O₃-Fe₂O₃ (the presence of three compounds), volatility Bi₂O₃ above the point of their melting point [11] and thermodynamic BiFeO₃ instability in air in the absence of Bi₂O₃ equilibrium molten solution - Fe₂O₃ [12]. Analysis of the literature data shows the impossibility of obtaining a single-phase compound BiFeO₃ by solid-phase synthesis [13].

Thus existing thermal (solid-phase reaction at high temperatures (T_{sin} < T_m) and chemical (reaction solution) methods do not allow to obtain a single-phase ferrite bismuth. In this aspect, the scientific interest is the use of solar technology, i.e. synthesis of a melt obtained Exposure to concentrated high-density light radiation at which compounds are formed during reactions in melts,

and then this state is fixed by quenching.

This work is aimed at studying the synthesis of BiFeO₃ from a mixture of bismuth (Bi₂O₃) oxides and iron (Fe₂O₃) fused on the solar furnace.

Methodology

At the first stage of the experiments, the oxides of bismuth (Bi₂O₃) and iron (Fe₂O₃) were melted on the focal plane of the solar furnace under the action of a concentrated light flux of 450 W / cm² density and held in the melting state for 15 minutes. The melts are quenched in water (104 deg / s). Molten melts in a ball mill by a wet method (material: water: grinding media = 1: 1: 1) for 10 hours. Sieved through a sieve of 0.05. On the basis of fused oxides, a mixture of Bi₂O₃ + Fe₂O₃ was produced in a stoichiometric ratio. Imagery-tablets are pressed by pressing at an effort of 1 ton on a C-100 press. The firing was carried out in an electric resistance furnace with silicate heaters at various temperatures. The samples were designated A-type.

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In the second stage, bismuth ferrite was synthesized from a mixture of oxides without melting on a solar furnace (B-type samples) was used as control.

The thermogravimetric analysis of the Bi₂O₃ + Fe₂O₃ mixture was carried out in the temperature range 100-1000°C on a Q-1500D derivatograph at a heating rate of 15°C / min.

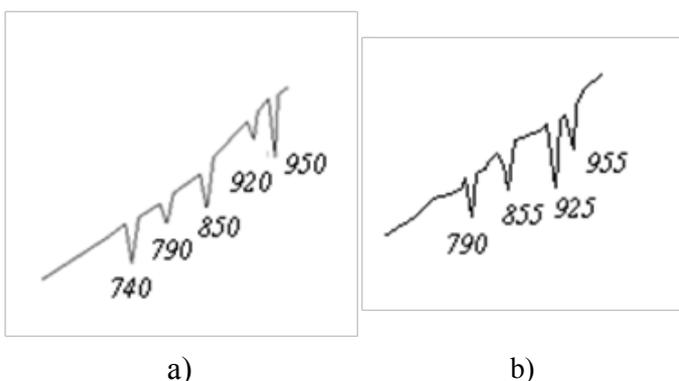
X-ray diffraction patterns were taken on powders using a DRON-3M diffractometer with copper anode. The determination of the apparent density of the samples, ρ , was carried out by hydrostatic weighing in octane, the calculation of the X-ray density, the ρ_{pent} , was carried out according to the formula: $\rho_{\text{pent}} = 1.66 \times M / V$ (M weight of the formula unit in grams, V-volume of the perovskite cell in Å³), $\rho_{\text{pot}} = (\rho_{\text{ж}} / \rho_{\text{прент}}) \times 100\%$. The structural looseness was determined by the formula: $\omega = M / (n\rho)$; Where M is the molecular weight equal to the sum of the atomic weights of the elements of the compound, n is the number of structural units (atoms, ions, complexes or radicals) in the formula unit of the compound, ρ is its density.

The coefficient of linear thermal expansion was determined on a cathetometer in the temperature range 25-600°C. Micro structural studies were carried out on transparent (in transmitted light) and polished sections (in reflected light).

Results and Discussion

Figure 1 shows the differential-thermal analysis curve for the Bi₂O₃ + Fe₂O₃ mixture in the temperature range 100-1000°C.

Figure 1: The Thermo Gravimetric Analysis of the Bi₂O₃ + Fe₂O₃ Mixture in the Temperature Range 100-1000°C: a) with Components not Melted in the Solar Furnace, and b) With Components Melted in the Solar Furnace

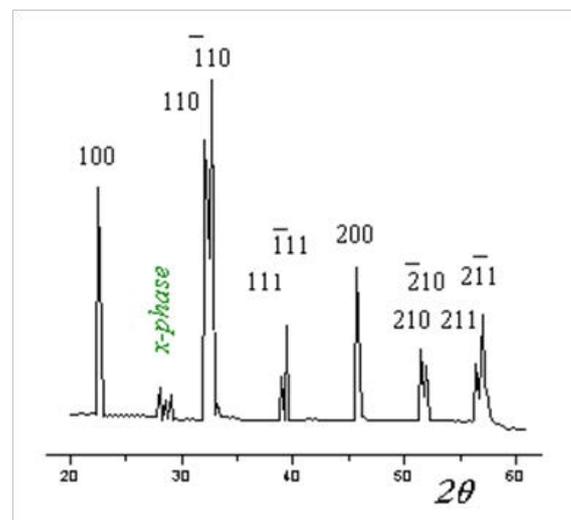


The thermogravimetric analysis of the Bi₂O₃ + Fe₂O₃ mixture in the temperature range 100-1000°C reveals 5 endothermic effects. It is clear that at 740°C a polymorphic Bi₂O₃ transformation occurs, the peak at 790°C is due to the melting of the eutectic in the Bi₂O₃-Fe₂O₃ system at which the synthesis of BiFeO₃ begins. As the analysis of the DTA curve shows, BiFeO₃ begins to decompose at 920°C and 950°C. Analysis shows that on the DTA curve of a mixture of oxides melted on a solar furnace, there is no peak at 740°C (Figure 1b). This indicates that the preliminary melting of the Bi₂O₃ and Fe₂O₃ components on a solar furnace leads to an increase in the chemical activity of the oxides. In addition, the endothermic peaks are shifted toward high temperatures by 50°C.

Figure 2 shows X-ray diffraction patterns of calcined samples at different temperatures.

An analysis of the X-ray diffraction pattern of the calcined sample at a temperature of 8850°C showed that this picture describes a BiFeO₃ compound with a rhombic lattice with lattice parameters $a = 3.958$, $b = 3.78$, $c = 4.08$ Å (ITSM No. 20-169). There is also an impurity phase Bi₂Fe₄O₉ (solid Fe₂O₃2BiFeO₃ solution) (ITSM No. 25-90).

Figure 2: The X-ray Diffraction Pattern of Bismuth Ferrite BiFeO₃ Synthesized at Various Temperatures (°C): a) 700; (B) 800; C) 885. X-phase Corresponds to a Solid Solution of Bi₂Fe₄O₉ = [Fe₂O₃ 2BiFeO₃]



As is known, bismuth ferrite is characterized by orthorhombic distortion of the perovskite cell [18] and temperature-dependent non-stoichiometry [19, 20], which complicate the synthesis of single-phase bismuth ferrite, as well as compounds containing BiFeO₃.

The x-ray density of bismuth ferrite was $\rho = 8.39 \text{ g/cm}^3$.

Figure 2 shows that, depending on the type of samples, the size and shape of the grains of ferrite of bismuth ceramics change. Thus, in the case of A-type samples, the grain size varies between 5-20 μm and 10-30 μm for B-type.

Table 1 shows the values of porosity, apparent density and coefficient of linear thermal expansion of the samples, depending on the synthesis method. It can be noted that the sinter ability of bismuth ferrite is somewhat improved when it is synthesized from fused oxides.

Figure 1(A&B): Microstructure of Bismuth Ferrite a) A- and b) B-types

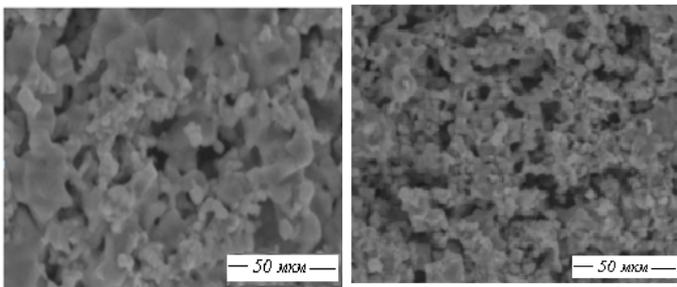


Table 1: The Values of Apparent Density (ρ density), Porosity (Π), Relative Density (ρ_{rel}), Structural Looseness (ω) and Coefficient of Linear Thermal Expansion (α) of Ceramic Bismuth Ferrite Samples, Depending on the History of the Components.

Samples	$\rho, \text{g/cm}^3$	$\Pi, \%$	$\rho_{\text{rel}}, \%$	ω	$\alpha \cdot 10^6, \text{K}^{-1}$
A-type	5.40	36	64,36	7,36	13.4
B-type	4.87	42	58,04	11,44	11.9

The coefficient of linear thermal expansion was $13 \times 10^{-6} \text{K}^{-1}$ for A-type samples with a predominance of rhombic phase and $11 \times 10^{-6} \text{K}^{-1}$ for B-type samples in which the orthorhombic phase prevailed. The difference between the structural looseness of materials is due to the fact that preliminary melting of oxides on a solar furnace promotes the synthesis of bismuth ferrite with a denser structure.

Table 2 presents the results of the synthesis of bismuth ferrite as a function of the prehistory of the Fe₂O₃ and Bi₂O₃ components. It can be seen that bismuth ferrite, obtained from fused oxides, is characterized by a small content of impurity phases. It can be assumed that the fused oxides are a more active form of the reagent, as a result of which the formation of impure Bi₂Fe₄O₉ begins at a lower temperature than the formation of bismuth ferrite.

Table 2: Results of Synthesis of Bismuth Ferrite as a Function of the Prehistory of Fe₂O₃ and Bi₂O₃ Components.

Prehistory of oxides Fe ₂ O ₃ Bi ₂ O ₃	Technological mode	Phase structure, %	
		BiFeO ₃	Bi ₂ Fe ₄ O ₉
Not melted on the solar furnace	885°C, 5 h	88	12
Melted on the solar furnace		97	3

Thus, bismuth ferrite, synthesized on the basis of precursors - iron and bismuth oxides, melted on a solar furnace has a denser structure, a low coefficient of thermal expansion, in comparison with the traditionally synthesized bismuth ferrite. The technology of creating ceramic materials based on bismuth ferrite requires careful regulation of the physico-chemical state of the starting materials.

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