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Synthesis of nanostructured materials based on $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$ and BiFeO_3

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Abstract. Methods for creating new nanostructured materials based on $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ and BiFeO_3 , results of a study of their structure, morphology and electrical properties are present. Morphology studies show that the micron-sized grains of ceramics are agglomerates of nanoparticles of about 50 nm average size for both BiFeO_3 and $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$. These nanoparticles are responsible for weak ferromagnetism in bismuth ferrite, and for increase of the critical temperature up to 96 K in $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ nanoceramics.

1. Introduction

We report on synthesis of new nanostructured materials based on compounds of $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$ (YBCO) and BiFeO_3 (BFO) and discuss the methods, improving their composition, structure and morphology during the preparation of both the primary powders and ceramics. The structure and morphology of nanopowders and nanostructured ceramics, based on YBCO, were studied by the scanning probe microscope «LEO-1450» with EDX-analyzer «INCA Energy», X-ray diffractometer «PANalytical Empyrean series 2», ASPEX Express with EDX-analyzer. The electrical resistivity of HTS samples was measured by four-probe method, the dielectric constant BiFeO_3 was measured using LCR-78110G meter company Goodwill Instrument Co.

2. Results and discussion

2.1. Nanostructured materials based on $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$

The nanostructured superconducting materials with different densities were prepared from nanopowders. The preparation method differs from reported elsewhere [1] both in the burning of nitrite-organic precursors and thermal treatment, and in method of their compactification. Different density of ceramic achieved without additives of organic binders. An optimal oxygen saturation of ceramics in the process of their sintering was reach due to the high specific surface of nanopowders.. The new method of nanopowder fabrication is characterized by the possibility to control the rate of

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combustion of thickened sediment arising after evaporation of the liquid solution of precursors. This technology provides definite size distribution of particles in the powder by selecting the combustion rate.

Figure 1 shows the diffraction patterns for two HTSC ceramics samples containing 95% of $\text{YBa}_2\text{Cu}_3\text{O}_{6.9}$ phase, produced from powders, thermally treated at 350 °C for 1 hour (Fig. 1a) and at 910 °C for 20 hours (Fig. 1b), respectively. After pressing powders were sintered at 920 °C for 1 hour. There is also shown the diffraction pattern of $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$ from database PAN-ICSD (Fig. 1c).

Copper oxide phase (5%) in the crystalline state (Fig. 1a and 1b), indicates that the Y_2BaCuO_5 oxide in X-ray amorphous state may also exist. After prolonged the heat treatment a recrystallization of this phase (211) observed. Morphology of nanostructured ceramics $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$ with densities of 2.5 g/cm³ and 6 g/cm³ shown in Figure 2a and 2b, respectively. Both highly porous and dense ceramics consist of micron-size grains, which are agglomerates of nanoparticles. According to diffraction studies, the average size of nanoparticles is about 50 nm. Agglomerates in the dense ceramics (Fig. 2b) have grown together. The uniform distribution of the nanoparticles of Y_2BaCuO_5 in a matrix of $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$ promotes pinning of the magnetic flux [2] and thus increases the critical current density.

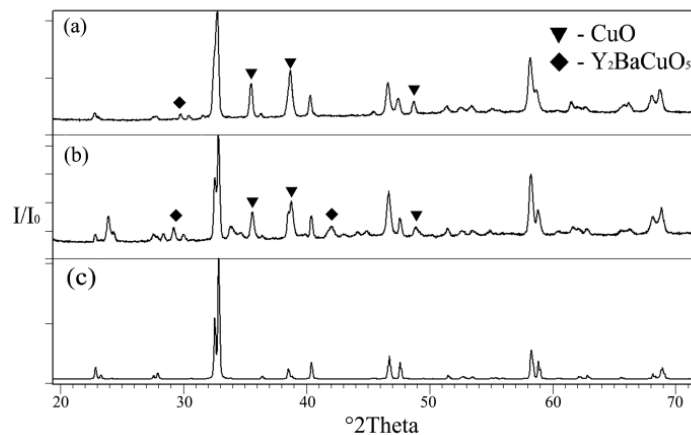


Figure 1. Diffraction patterns of ceramics made of thermally treated powders: (a) at 350 °C; (b) at 910 °C; (c) structure $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$ from the database PAN-ICSD.

Appropriate thermal treatment of nanopowder, in one step, allows to produce nanostructured ceramics samples with different densities up to X-ray density. We used the same technique, but different quantity of glycerol, to synthesize ceramics samples with densities of 3 g/cm³ and 5.7 g/cm³ [3].

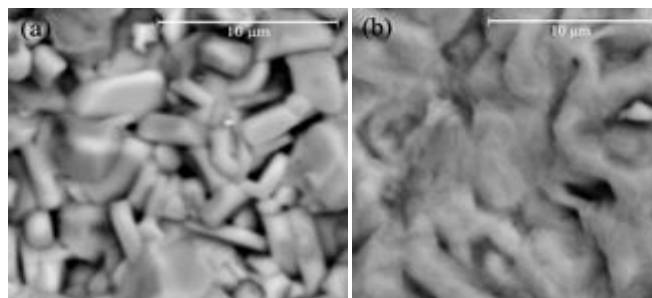


Figure 2. Morphology of nanostructured ceramics $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$: (a) porous; (b) dense.

Various glycerol content leads to different combustion rates of a “gel” which remains after evaporation of aqueous solutions of nitrates Y, Ba and Cu. As one can see, the amount of glycerol in the initial solution, other conditions being equal, allows us to control the value of nanostructured ceramics density. To obtain microcrystalline ceramics in one step it is enough to increase the temperature and duration of nanopowder heat treatment, but the increase in particle size reduces the oxygen content in the ceramics. Optimal oxygen concentration may be achieved by preliminary destruction of nanopowder agglomerates before sintering. In this case, the sintering temperature of the powder increased by about 5 degrees. Figure 3 shows the temperature dependence of the resistivity of nanostructured ceramics $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$ made from nanopowders, heat treated at 350 °C - (\blacktriangle) and 910 °C - (\bullet) respectively. It turned out that the transition into the superconducting state, both for dense and porous ceramics (Fig. 3) starts at $T_c \sim 96$ K. The absolute values of the electrical resistance of high-porous ceramics are an order of magnitude greater. For this ceramics, the temperature coefficient of resistance is also higher, and the superconducting transition stretched up to 85 K, apparently due to the small area of contacts between the grains. Because the increasing T_c does not depend on the porosity this effect may be explained by the fact, that the size of the nanoparticles is the same for the dense and porous ceramics. Similar results were obtained for the nanostructured ceramics with the densities of 3 g/cm³, and 5.7 g/cm³ [3] with a particles sizes about 60 nm.

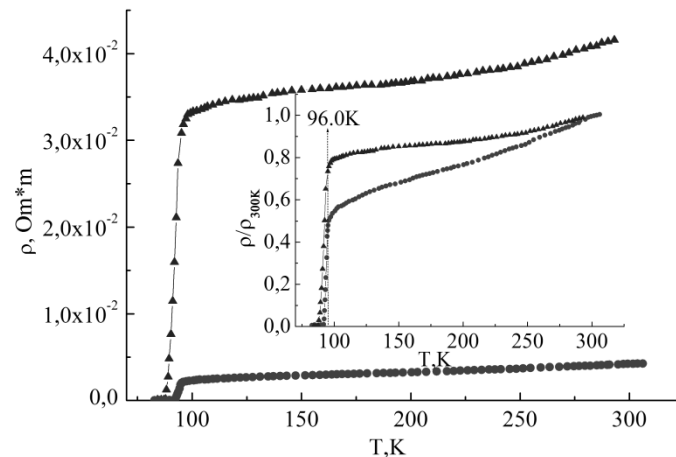


Figure 3. The electrical resistance of nanostructured ceramics made from $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$ nanopowders, precalcined at 350°C - (\blacktriangle) and 910 °C - (\bullet)

2.2. Nanostructured materials based on BiFeO_3

To implement the linear magnetoelectric effect in bismuth ferrite the crystallite size should not exceed the size of the space-modulated spin cycloid with a period of 62 nm. Rabadanov et al. [4] developed a method (burning of bismuth nitrate and iron nitrate with the addition of glycine), which allows one to obtain the nanopowder, consisting of stoichiometric BiFeO_3 crystals only (Fig. 4a). Thermal treatment of the powder at temperatures higher than 600 °C gives rise to side phases, which are undesirable in the ceramics manufacture [5]. Optimization of this technology allows to obtaining powders having a particle size from 35 nm to 45 nm without increase of side phase after calcination at 600°C (Fig. 4b). Phase analysis of powder before thermal treatment shows the presence of about 6% impurities, representing probably a mixture of $\text{Bi}_{25}\text{FeO}_{39}$ and $\text{Bi}_2\text{Fe}_4\text{O}_9$. After calcination of the powder at temperature 600°C, the amount of secondary phases reduced to 4%, and the crystallite size calculated from Scherrer-Wulff formula, increases by approximately 10-15%.

The morphology of nanopowder obtained by optimized technology (Fig. 5a) indicates that powder consists of agglomerates of bismuth ferrite nanocrystals. Figure 5c presents diffraction pattern of ceramics obtained from this powder. As can be seen, all the peaks of side phases are suppressed. The density of the synthesized ceramics is close to the theoretical value and is approximately of 7.8 g/cm³.

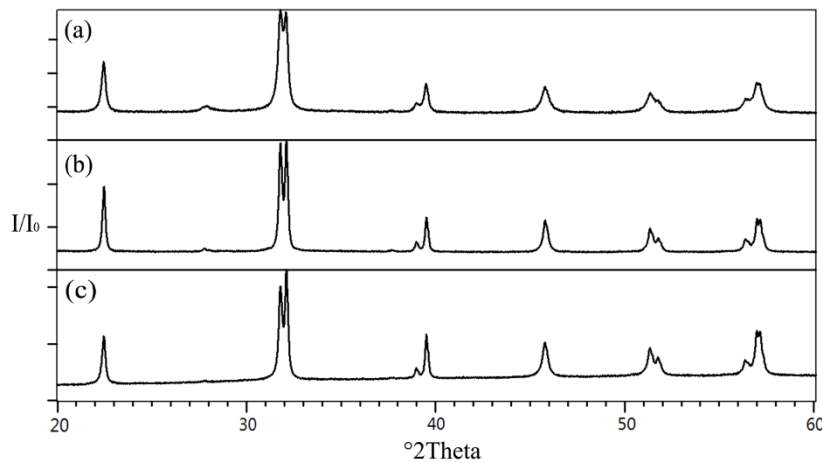


Figure 4. Diffraction patterns of BiFeO_3 nanopowders: (a) initial; (b) after thermal treatment at 600 °C; (c) nanoceramics

The morphology analysis shows that ceramics is dense and homogeneous (Fig. 5b). There are also clearly seen pores of faceted shape in ceramics grains. This indicates that grains are agglomerates of nanoparticles. This fact agrees with crystallite size evaluation from diffraction data (Fig. 1c) which gives size of ~ 50 nm, while the large grains are of micron size. Faceting agglomerates probably, acquire during their compression. High level of porosity in the ceramic BiFeO_3 is a serious obstacle for most practical applications of this material. According [6] the grain growth and compaction occur simultaneously during the sintering, so it is important to find the sintering process mode, in which the compacting will prevail over the increase of the crystallite size.

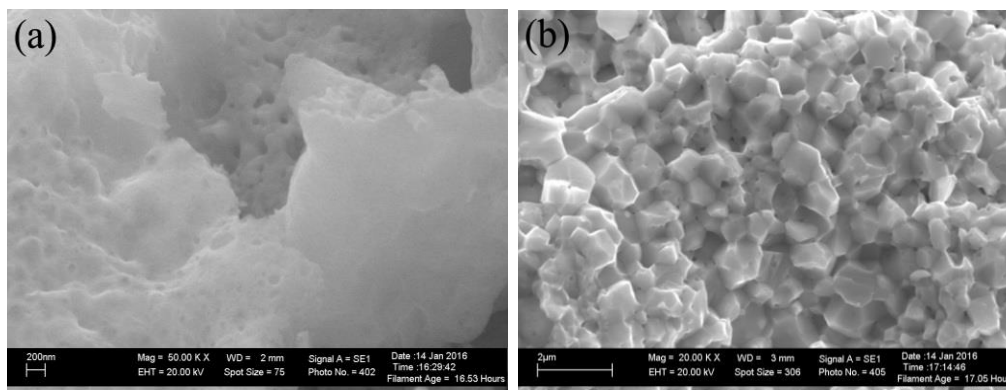


Figure 5. The surface morphology studied by the scanning electron microscope LEO - 1450: a) - nanopowder; b) - nanoceramics

Figure 6 shows the frequency and temperature dependences of the dielectric constant for nanoceramics and cold-pressed sample of the same powder. The anomaly on the $\epsilon = f(t)$ dependence in the temperature range of 20 – 150 °C (Fig. 6b) is probably due to the presence of adsorbed moisture. Abnormal behavior of heat capacity at the same temperatures [7] also indicates the presence of hydrated moisture in the cold-pressed samples. This anomaly is absent in a ceramic sample. The values of ϵ' for ceramics are slightly higher, this may be caused by increased interaction between the nanoparticles which are in the ferroelectric phase and by the absence of moisture as well. Hydrated moisture may improve the electrical conductivity [6], which complicates the use of bismuth ferrite as a ferroelectric.

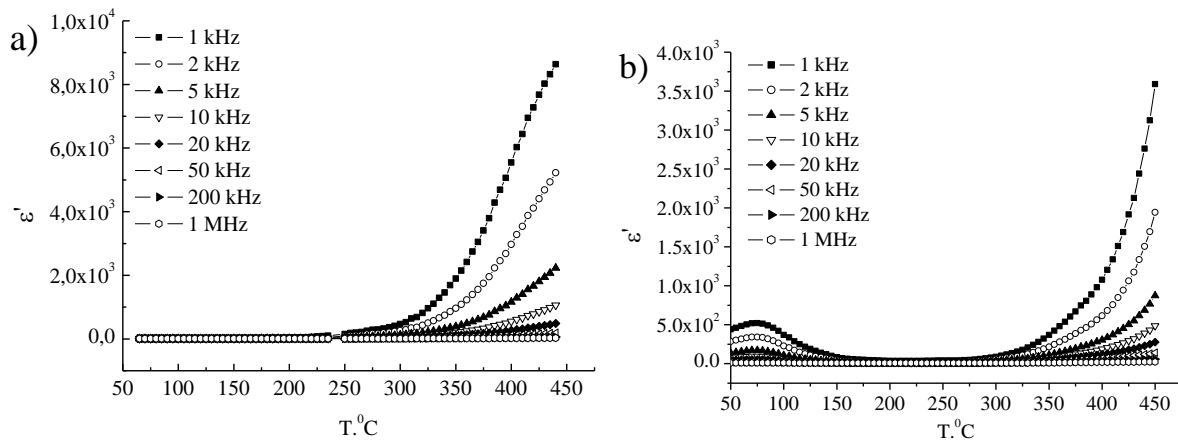


Figure 6. The real part of the dielectric constant: a) nanoceramics; b) cold pressed

Acknowledgments

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