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Processing, phase transitions and functional properties of BSPT Ceramics

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Strong demand for piezoelectric materials, capable of working at high temperatures, drives the development of new piezoelectrics with high Curie temperatures T_C [1]. Ceramics based on solid solutions (1-*x*)BiScO₃ - *x*PbTiO₃ (BSPT) was intensively studied in recent years because its compositions close to the morphotropic phase boundary (MPB) revealed rather high T_C values > 700 K and high piezoelectric activity [2 - 5]. However, functional properties of the BSPT-based compositions are not optimized up to date, and wide variations in ceramics properties may be observed depending on preparation conditions.

In this work, influence of the initial powder preparation conditions on phase content, the unit cell parameters, dielectric and piezoelectric properties of compositions in the BSPT system close to the MPB (with x = 0.635 - 0.65) were studied. Ceramic solid solutions were prepared using the powders obtained from Bi, Sc, Pb and Ti nitrate solutions following the procedure described in [5]. The powders were calcined at 700 - 950°C (2 - 6 h), pressed into pellets and sintered at 1130-1150°C (2 - 4 h) in the PbO-enriched atmosphere, then slowly cooled to the room temperature. Small amounts of Cr₂O₃ were added to some compositions.

Phase composition and crystal structure parameters were determined using the X-ray diffraction (DRON-3M with $Cu_{K\alpha}$ -beam). Microstructure of the powders was checked by scanning electron microscopy (SEM) (JEOL JSM-7401F, Analysis Station JED-2300). Dielectric properties were studied by the dielectric spectroscopy method (Agilent 4284 A, 1 V) in the temperature range of 300 – 1000 K at frequencies 100 Hz – 1 MHz. The d_{33} piezoelectric coefficients were measured using YE 2730A d_{33} meter (APC products) with preliminary poling of samples in electric field. The electromechanical coupling coefficient k_t was measured by the standard resonance – antiresonance method.

According to the X-Ray diffraction data, powders obtained at temperatures 750 - 950°C possessed Perovskite-type structure (Figure). The convolutions of the diffraction peaks with $h^2+k^2+l^2=4$ (shown in inserts) demonstrate strong dependence of relative content of tetragonal and rhombohedral phases, typical for the compositions in the vicinity of MPB, on both sintering temperature and composition. Content of tetragonal phase increases with *x* value. Small displacements of the diffraction peaks indicate small changes in unit cell parameters of the samples caused by changes in composition.

According to the SEM data, average size of synthesized particles varies in a range $0.1 - 1 \mu m$, and grain size in ceramics sintered at $1130 - 1150^{\circ}C$ does not exceed 2 - 3 μm . Small grain size may be related to the neighboring of the tetragonal and rhombohedral phases in obtained ceramics, which hinders grain growth by inducing strain during sintering.

Dielectric measurements revealed the 1st order sharp ferroelectric phase transitions marked by peaks in dielectric permittivity $\varepsilon(t)$ and corresponding minima in dielectric loss $tg \delta(t)$ curves at temperatures ~ 700 K. Slight shift of the $T_{\rm C}$ values determined by both x value and relative content of tetragonal and rhombohedral phases was revealed. It should be noted that strong variations in ε and σ values, more than to 2 times and 1 order, respectively, were observed. High piezoelectric coefficients d_{33} values up to 430 and 340 pC/N were measured in ceramics with compositions, corresponding to x=0.64 and x=0.645, respectively. Additional quenching of poled ceramics [6] allowed us to increase the d_{33} value up to 520 pC/N, which is close to the top values reported for BSPT ceramics [7].

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Figures:



Fig.1. X-Ray diffraction patterns of the samples with x=0.64 annealed at 750°C (*a*), 850°C (*b*), 950°C (*c*). In inserts convolutions of the peaks with $h^2+k^2+l^2=4$ are shown.

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