Методом кислотного растворения был проведен анализ на содержание аморфной фазы в полученных осадках TiO_2, высушенных при 120˚С. Установлено, что увеличение температуры осаждения диоксида титана позволяет увеличить содержание кристаллической фазы. Данный факт может быть использован для получения гидрозолей диоксида титана, разного фазового состава, что может быть востребовано для ряда областей применения.

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UDK 539.2

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SYNTHESIS AND CHARACTERIZATION OF LOW LOSS DIELECTRIC CERAMICS PREPARED FROM COMPOSITE OF TITANATE NANOSHEETS WITH BARIUM IONS

Ceramic nanoparticles of barium titanate are characterized by increased value of dielectric permeability (ε) and low loss tangent. These dielectric oxide ceramic can be classified as a modern functional nanomaterials, while synthesis, grains morphology (i.e., shape, size or size dispersion) desirable dielectric properties are crucial in dynamic developing modern electronics devices. These materials are widely studied due to their importance in electronic components such as capacitors, gate dielectrics, memories or power-storage devices. Nowadays, the main objection of research on nanoceramics is the improvement of already known systems (i.e. reducing dielectric loss and increasing dielectric permittivity) or development of new materials. Fast improvement of electronic devices required miniaturization of used components. Nanoceramics synthesized by solid state methods may not fulfill with the current material requirements.

Numerous soft or wet chemical methods of obtaining nanomaterials, such as co-precipitation, sol-gel processes as well as modified Pechini method are commonly used and products of their synthesis are widely investigated. This approach of synthesis has significant advantages compare to conventional solid-state reactions, such as better compositionalcontrol, nano-sized precursor powders and resulting lower crystallization temperature, avoiding the grinding step in synthesis procedure and possible contamination.

In this work [1], the organic-inorganic nanocomposite reaction of titanate nanosheets [2] with barium ions, has been obtained using the reaction of titanate nanosheets with barium ions and further used as a precursor of barium titanate. Gradual calcination of precursor leads to formation of titanates with $BaTi_4O_9$ or $Ba_2Ti_9O_{20}$ stoichiometry.

Therefore, the obtained barium titanate precursors were subjected to the structural research (X-ray Diffraction, Raman spectroscopy), grain morphology (scanning electron microscopy) and electrical properties (broadband dielectric spectroscopy), which were carried out simultaneously with step sintering in the temperature range of 900–1300 °C. The rapid contraction of calcinated sample was found in the temperature range 1000–1050 °C. It was observed that up to temperature of 1050 °C barium titanate exhibits fine ceramics grain size. At higher temperatures grain morphology radically changes simultaneously with crystallographic phase composition change revealed by XRD and Raman studies.

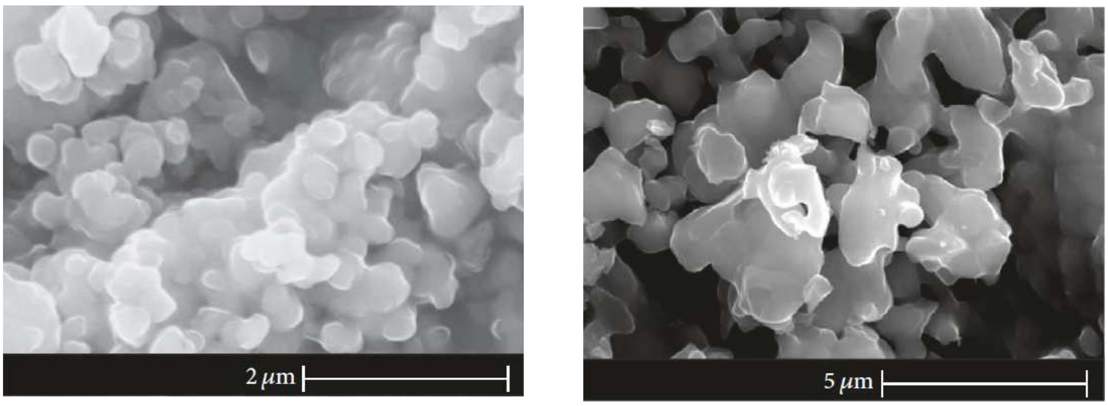

Figure 1 – SEM images of Ba-TN after sintering at (a) 1050 °C, (b) 1150 °C collected with accelerating voltage 25 kV.

It was noticed an increase of the dielectric constant (ε) of ceramic material with applied gradual calcination. Such behavior of material is associated with the well-known connection between density of the sample and its dielectric properties, according to which higher density results in a higher dielectric permittivity. Lower porosity of ceramic pallets also improves quality factor value ( ) and results in the lower value of loss tangent.

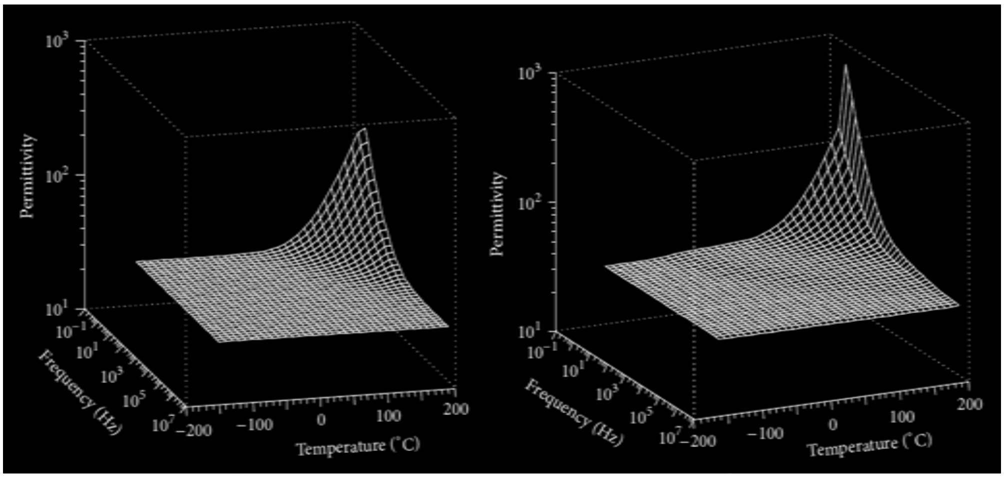

Figure 2– Temperature-frequency representation of dielectric permittivity Ba-TN sintered at: a) 900 °C and b) 1200 °C.

The temperature coefficient can be tuned from -32 ppm/°C (for the sintered precursor (at T = 900 °C) to +37 ppm / °C (after calcination at T = 1300 °C). It is an effect of transformation of compound to titanate of different stoichiometry, $BaTi_4O_9$ changed to $Ba_2Ti_9O_2$ at temperature higher than 1100 °C, that was confirmed by Raman and XRD analysis.
Consider the widespread trend of the miniaturization of electronic devices used nanosized objects is required. From this reason temperatures in the range of 900–1100 °C were selected as the optimal calcination conditions for precursor, which guarantees satisfactory electrical permittivity and nanomeric size of grains.

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