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Short Communication

Formation of Spherical Nanoparticles $BaTiO_3$ by Peroxide Method

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Structure of barium titanate particles obtained by peroxide method was studied by SEM, X-ray phase analysis, IR spectroscopy and Raman scattering. Found, that particles have preferably a spherical shape with a diameter of 20-200 nm and contain both cubic and tetragonal phases.

Keywords: Barium titanate, Peroxide method, Cubic and tetragonal phases.

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1. INTRODUCTION

Barium titanate is a well-used material for the production of components of microwave technology, such as phase shifters, delay lines, parametric amplifiers and harmonic generators. Improvement of the parameters of devices is possible with increasing purity requirements, the number of defects and the dielectric properties of this material. Well known that the structure and dielectric properties of the ferroelectric, including barium titanate, essentially depend on the particle size [1]. Miniaturization particle size leads to improved electrical and mechanical properties of ceramics based on the compound. Analysis of the methods of obtaining $BaTiO_3$ shows that the most promising is its preparation by the thermal decomposition of the inorganic precursor.

2. EXPERIMENTAL SECTION

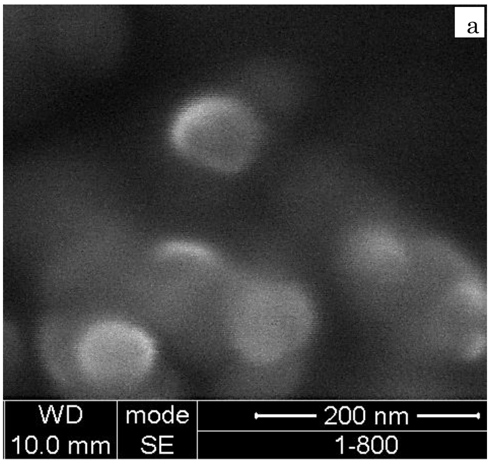
$BaTiO_3$ precursor was synthesized according to reaction:

$$TiCl_4 + BaCl_2 + 2H_2O_2 + 6NH_4OH \rightarrow$$
$$BaO_2O_2TiO{\cdot}2H_2O\downarrow + 6NH_4Cl + 3H_2O$$

The resulting precipitate was filtered off, washed out and dried. Thermal treatment of the precursor was performed in air during one hour at temperatures of 700, 800 and 900 °C. Method of the scanning electron microscopy (Quanta 650 FEG) shows that the obtained particles have a spherical shape with a diameter of 50 to 200 nm and agglomerated into clusters of up to several micrometers (Fig. 1a).

Microprobe analysis shows a uniform distribution of barium, titanium and oxygen on the surface of particles with an aspect ratio typical of BaTiO3, which follows from the data element mapping. Particles produced in the decomposition of the precursor at a temperature of 700 °C, a peak is observed and the peak of carbon, chlorine indicative of partial decomposition of the precursor. Further reduction of the precursor products is achieved by annealing at 800 °C (Fig. 1b).

This fact is also confirmed by X-ray diffraction data (GBC EMMA, CuK_α), as seen in X-rays (Fig. 2a), on which there are reflexes typical of the cubic phase of $BaTiO_3$ (Fig. 2a). Reflexes had broadening, indicating



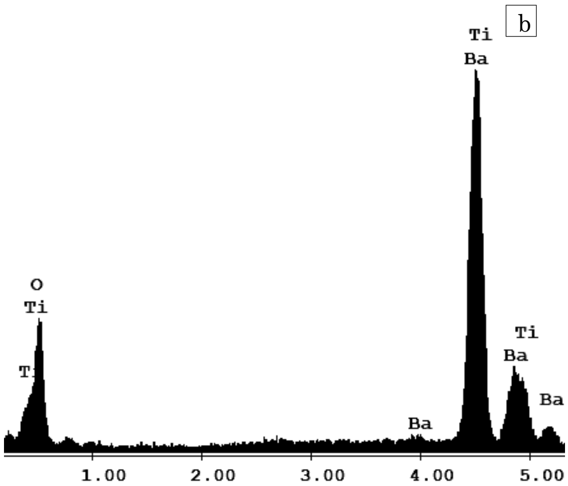


Fig. 1 – SEM-images of nanoparticles $BaTiO_3$ (a), spectrum of X-ray microanalysis (b)

incomplete decomposition of the precursor. For the particles annealed at 700 °C there is a splitting of the reflex (200/002), characteristic of the tetragonal phase (Fig. 2b).

During the heat treatment, peroxide synthesis and also noted the formation of $BaTiO_3$ in the cubic structure distortion, for example, in the form of inclusions $BaTi_2O_5$ at angles $2\theta = 26°$ and 70°. The ratio of the

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intensities for reflections I_{101} / I_{002} also marks the formation of the cubic phase.

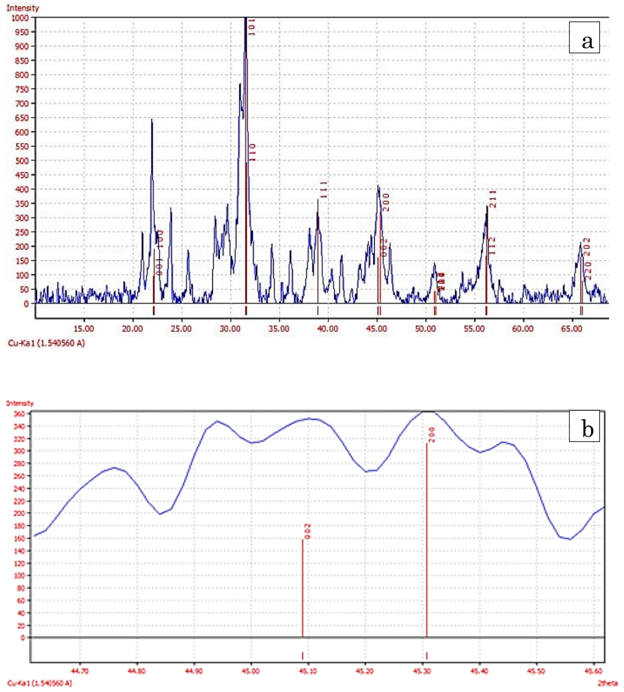

Fig. 2 – X-ray patterns of $BaTiO_3$ after annealing at 800 °C (a), splitting reflex (200 / 002) after annealing at 700 °C (b)

The sizes of $BaTiO_3$, can be evaluated (the coherent scattering areas (L) of X-ray radiation) from the Scherer equation: $L = k\lambda/(B\cos\theta)$. The reflection width was considered by the level 0.5 (B), θ is the Bragg angle, $\lambda = 0.154178$ nm for CuK_{α}, k is a constant equal to 0.9. Considering the arising during synthesis microstrain and instrument broadening their sizes are about 20 nm.

Raman scattering spectra samples $BaTiO_3$ annealed at temperatures 700 and 800 °C lines marked appearance as an amorphous halo in the range of 170-320 cm^{-1}, ~ 520 and 720 cm^{-1}, characteristic of the cubic structure [2]. Comparison of the Raman spectra with increasing annealing temperature is characterized by both a decrease in the intensity of all the lines, so they shifted to lower frequencies, indicating a decrease in the size of nanoparticles and the increase in the cubic phase.

IR spectra and peroxide precursor powders after heat treatment similar to the spectra of the powders obtained by mechanochemical synthesis of oxalate precursor [2]. Peaks of absorption 1356 and 1415 cm^{-1} for the untreated and ~ 1344 and 1413 cm^{-1} for the treated precursor at 700 °C of impurities caused by the presence of carbonates.

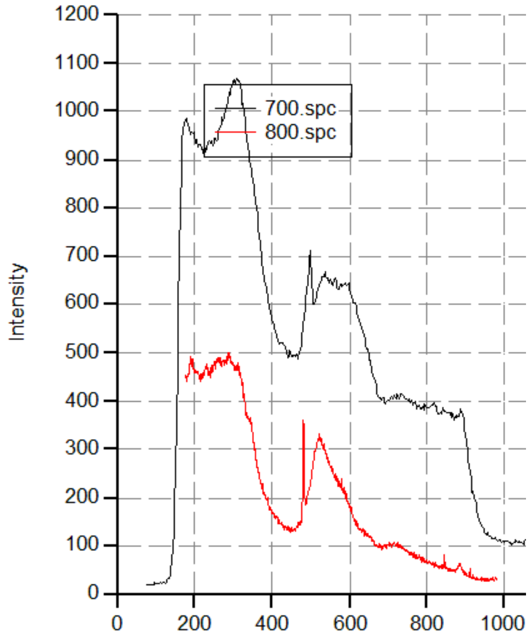

Fig. 3 – Raman scattering spectra samples $BaTiO_3$ annealed at temperatures 700 and 800 °C

3. RESULTS AND DISCUSSION

The difference between the SEM and X-ray analysis relative to the size of the particles may be explained by the significant variation in shape and size of the synthesis products. In the SEM images at different magnifications indicated forming cubic, whiskers, cluster structures of different phase composition.

4. CONCLUSIONS

Thus, the peroxide synthesis by the of barium titanate powder according to SEM are nanosized particles of 50 to 200 nm by XRD are cubic and tetragonal phases with sizes of about 20 nm nanoparticles. With increasing annealing temperature of the precursor from 700 to 800 °C promotes structural perfection of the lattice, and the reduction of the content of the tetragonal phase.

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