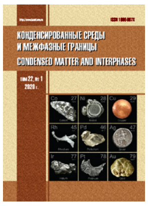

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Microwave Synthesis of $CaTiO_3$ Nanoparticles by the Sol-Gel Method

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Abstract

A technique for the microwave-activated synthesis of calcium titanate nanopowder was proposed. The microwave effect used in the synthesis of $CaTiO_3$ samples when using sodium carbonate as a precipitant allowed obtaining a chemically homogeneous nanopowder with a significant reduction of the process time.

Keywords: sol-gel, microwave synthesis, calcium titanate, nanoparticles.

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1. Introduction

In recent years, the development of methods for obtaining and studying the properties of nanoparticles and nanostructured materials of various chemical composition, structure, and morphology became a priority task in materials science.

Oxide compounds with a perovskite structure are widely used in the manufacture of solid oxide fuel cells, catalysts, magnetic materials, chemical sensors, and electrodes [1, 2].

Calcium titanate is known as a promising material with ferroelectric and paraelectric properties; it is used as an active element in piezoelectric transducers, optical modulators, ferroelectric memory devices, capacitors with a high dielectric constant, microwave devices, and photocatalysts [3].

Sol-gel technology has already proved to be efficient methods for the synthesis of powders of various compounds with particles of the nanometre range [4, 5].

The aim of this study was to establish the effect of microwave exposure on the synthesis of $CaTiO_3$ nanoparticles by the sol-gel method.

2. Experimental

The following precursors were used in this study: calcium nitrate crystallohydrate $Ca(NO_3)_2{\cdot}5H_2O$ (analytical reagent grade CAS 10035-06-0), titanium chloride $TiCl_4$ (special purity grade TU 6-09-4471-77), sodium carbonate Na_2CO_3 (reagent grade GOST 4201-79).

The synthesis of calcium titanate nanoparticles was carried out in two modes - in the presence and absence of microwave radiation. The rest of the process parameters were kept the same.

During the first stage, the initial solutions were prepared: aqueous solution $Ca(NO_3)_2$ and alcoholic solution $TiCl_4$. Then these solutions were mixed, boiled for a certain time, and cooled to room temperature. This process was accompanied by the formation of TiO_2. During the same stage, in the second series of experiments, microwave exposure was carried out (P_{max} = 700 W, operating frequency

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2450 MHz). The boiling time was 5 min in the presence of microwave exposure and 20 min in the absence of microwave exposure.

Sodium carbonate was used as a precipitant in an amount sufficient for the complete precipitation of the components. The gel formed by this method was filtered, dried in air, and then annealed for one hour at a temperature of 750 °C. The following reactions took place:

$$Ca(NO_3)_2 + Na_2CO_3 = CaCO_3 + 2NaNO_3 \quad (1)$$

$$CaCO_3 = CaO + CO_2 \quad (2)$$

$$CaO + TiO_2 = CaTiO_3 \quad (3)$$

For the investigation of the phase composition of the samples, we used the method of X-ray phase analysis, DRON-3 X-ray diffractometer with a Cu anode (λ = 0.71075 nm). The scanning was performed within an angle range of 2θ = 0–80° with a step of 0.1. Phases were identified using [6, 7].

3. Results and discussion

Thee diffraction patterns of the samples synthesized without (a) and in the presence (b) of microwave exposure are shown in Fig. 1.

For the sample obtained without microwave exposure, distinct peaks of unreacted starting substances: titanium and calcium oxides were observed. Reflexes corresponding to the target product – $CaTiO_3$ were also present, but they

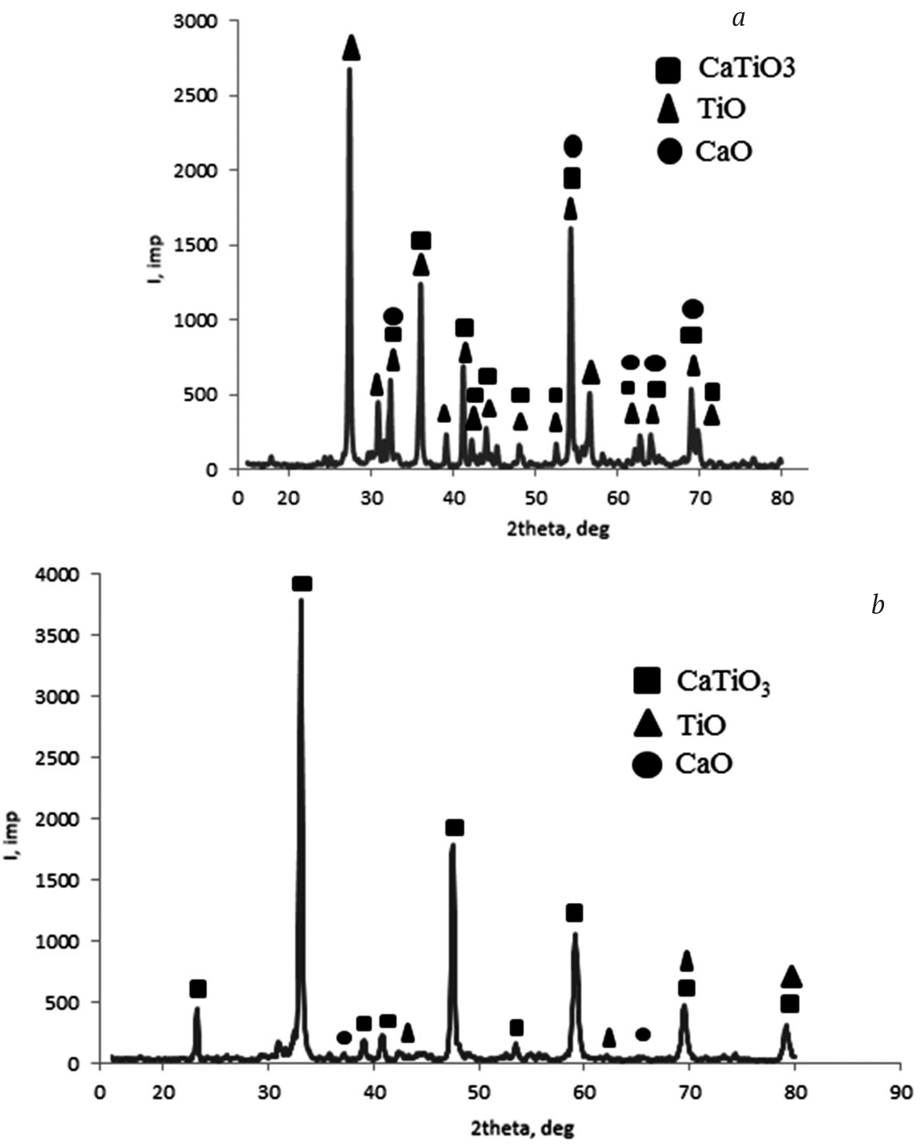

Fig. 1. Diffraction pattern of calcium titanate powder obtained in the absence (a) and presence (b) of microwave exposure

were not so distinct. This means that when the synthesis was carried out in the absence of microwave exposure, it was not possible to achieve the required degree of interaction between the reagents.

The diffractogram for the sample synthesized under the action of microwave radiation (Fig. 1b) was radically different. The peaks corresponding to calcium titanate were predominant and distinct, while the peaks related to the starting substances were present in small amounts and have low intensity, especially for calcium oxide.

4. Conclusion

Thus, the use of microwave exposure in the process of synthesis of nanocrystalline calcium titanate allows solving two problems at once: in the case of microwave synthesis, the powder had not only more homogeneous in composition, with a predominance of the required compound, but also significant intensification of the process, consisting in a decrease in the synthesis time was observed.

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Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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