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# Synthesis of Titanates Phases via Sol-Gel Method

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**Abstract**
The titanate phase synthesis ($BaTiO_3$, $Ba_2TiO_4$) was carried out by the sol-gel method in the laboratory, thermal treatment of the experimental compositions up to 850ºC and applied restraints up to 24 hours. In preparing the compositions, the following precursors were used: barium acetate and titanium butoxide in the presence of surfactants. The assayed samples were examined by X-ray phase analysis (XRD). Registered titanium phases are obtained at lower temperatures than the required heat treatment applicable to a standard high temperature synthesis method. At 830ºC and retention for 30h, a sample with predominant presence of $BaTiO_3$ phase and presence of concomitant phase of $Ba_2TiO_4$ was obtained.

**Keywords**: sol-gel method, $BaTiO_3$, phase-forming, nanoparticles

## 1. Introduction

Zol-gel materials have a variety of applications [1-2] in electronics, electrotechnics, optics, photonics, chemical technologies, high temperature technologies, energy, biosensors and biochemistry. This method is quickly applied in a universal technique for preparing powders, coatings, massive monolithic ceramics and glasses of the same initial composition.[2] Advantages of the sol-gel technique include fine particle size, easy composition control and low processing temperature [3]. Titanium ceramics are one of the most promising materials widely used in various industries. Barium titanate is a well-known material present in tetragonal ferroelectric form at 130°C with high dielectric permeability and is converted to a paraelectric cubic structure at above 130°C. [4-5] In the literature, there are a number of methods for the synthesis of titanium ceramics such as high temperature solid phase synthesis, co-precipitation, hydrothermal, mechano-chemical activation, supercooled melt synthesis, etc., as a solid phase synthesis method. [6-8] It has a number of drawbacks, which leads to the search for suitable methods of synthesis. In the present work, attention is directed to the production of lead-free ceramics by the sol-gel synthesis method. The use of suitable surfactants to reduce the temperature of subsequent caltination leads to the search for alternative methods for obtaining the material of desirable properties. [9-10] Other advantages of this process include net shape casting, film coating and fiber pulling [11]. Even small quantities of dopants, such as organic dyes and rare earth elements, can be introduced in the sol and end up uniformly dispersed in the final product [1]. The use of new precursors aims at switching to certain synthesis methods to improve performance parameters and lower the incineration temperature. The low-temperature sol-gel process has a number of positive properties, such as low cost apparatus, low temperatures and small particle sizes. [12-13]

## 2. Materials and methods

Sol-gel synthesis of ceramic phases was carried out using barium acetate and titanium butoxide as starting materials in certain stoichiometric ratios. The process is carried out by adding 10 ml of ethyl alcohol to 10 ml of barium acetate. The obtaining solution was homogenized with a

KERN PCB version 1.8 magnetic stirrer for 30 minutes at 60$^{O}$C, with the revolutions being continuously monitored. Titanium butoxide is used in the same procedure. The quantitative ratio is as 10 ml of ethyl alcohol is added to 15 ml of titanium butoxide and homogenized for 15 minutes. The solutions thus obtained were mixed and homogenized at 20°C for 4 hours. After gelling, the mixture is dried at 80°C for 24 h. The resulting barium titanate powders were ground in an agate mortar followed by calcination at 830°C with a 30 hour rest in a laboratory furnace. Characterization of the resulting barium titanate phases was performed by X-ray analysis using a Bruker D8 Advance automatic X-ray diffractometer with CuKα radiation (Ni filter) and a LynxEye solid detector. The X-ray spectrum is recorded in the angular range of 10 to 80° 2θ in a step of 0.02°2θ and a counting time of 17.5 sec./step. The dimensions of the obtained ceramic powders are of the order of 10-20nm.

## 3. Results and discussions

In Fig. 1 the results of the study of a sample of barium titanate powders obtained by low-temperature sol-gel method with subsequent calcination at a temperature of 850°C and a retention of 24h are shown. The outline phases $BaTiO_3$, $BaTi_2O_5$ are clearly visible. There is also barium carbonate, which does not favor the conditions for further use of the material.

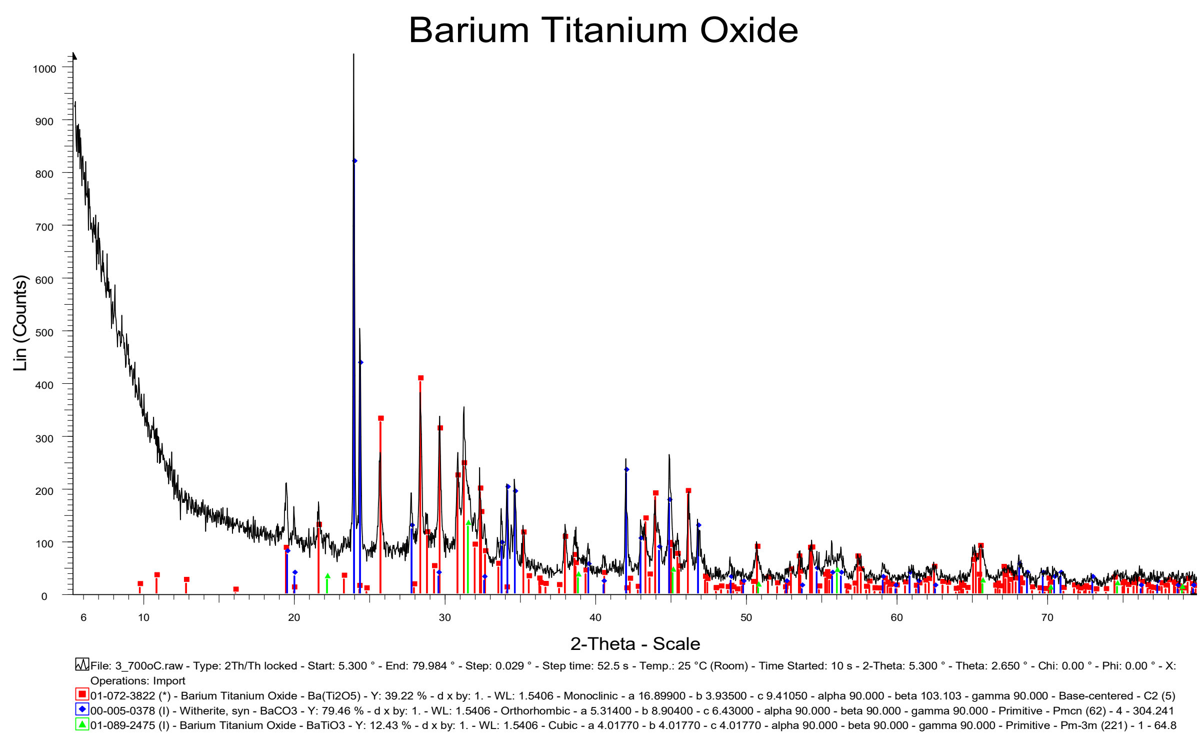


**Fig.1 The phase of $BaTiO_3$ at a temerature 850°C and retention from 24h.**

In the subsequent sample treatment and tempering at lowering the temperature to 830°C and increasing the retention time 30°C the presence of barium titanate in two variants of the crystal lattice of the monoclinic and rhombic phase was established (Fig.2).

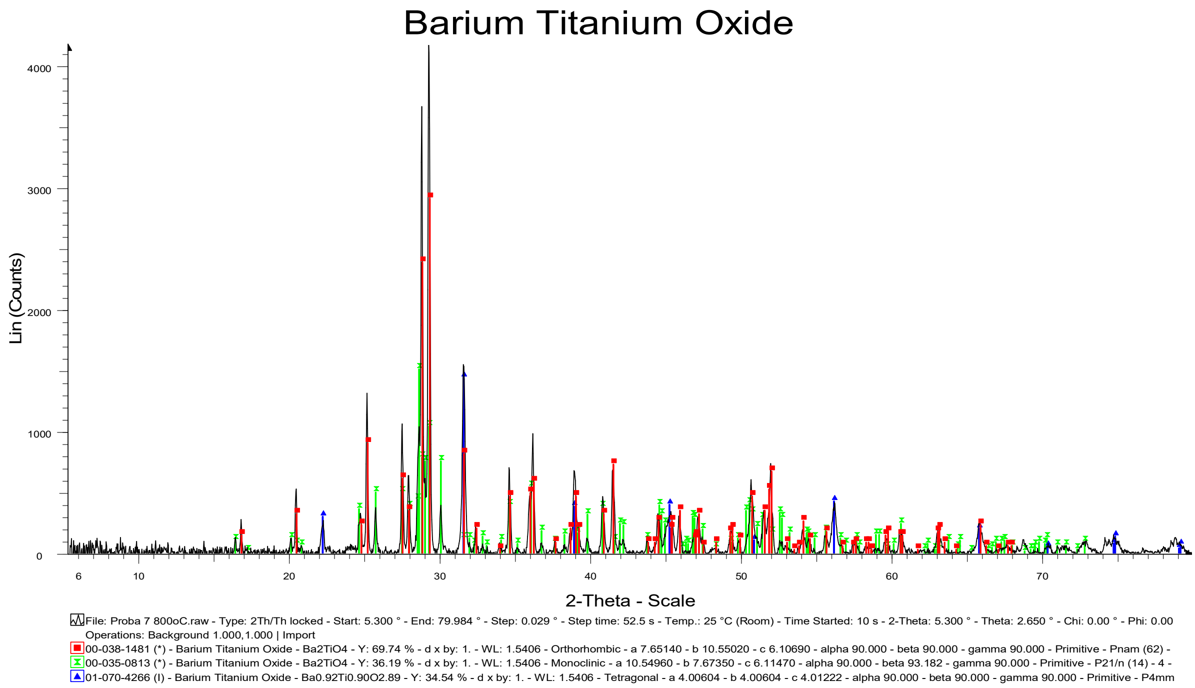


**Fig.2 The phases of $Ba_2TiO_4$ at a temerature 830°C and retention from 30h.**

## 4. Conclusion

There were synthesis titanium ceramic powders by sol-gel technology. The precursors were used suitably selected to provide compositions with desirable properties. The stoichiometric entered components were calculated in order to improve proper process control. The resulting phases are $BaTiO_3$ and $BaTi_2O_5$ by a sol-gel method and subsequent incineration at 850°C. The presence of an undesired phase of $BaCO_3$ leads to long-term studies and experiments on the synthesis of $BaTiO_3$. For continuous treatment of the sample, $BaTiO_3$ is synthesized in two forms: rhombic and monoclinic. This difference in crystal lattice parameters is due to the increased presence of $Ba^+$ in the feedstock. The subsequent stage of the research is aimed at achieving clean phases at a reduced temperature, modifying the samples in order to obtain materials applicable in the electronics.

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