

ULTRASOUND MODIFICATIONS OF PHYSICAL PROPERTIES OF BaTiO₃ POWDERS

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Abstract

The starting BaTiO₃ powder was prepared by solid state reaction, and then modified by ultrasound into powders with different physical characteristics. The prepared barium titanate powders were characterized by X ray powder diffraction, laser particle size analyzer, also, specific surface area and powders density were determined. The studies indicate that physical properties of BaTiO₃ powders were changed by sonochemical treatment. However, tetragonal crystal structure was maintained. It is shown that sonochemical method can be used for easy modification of barium titanate powder characteristics.



Experimental

The BaTiO₃ (BT) starting powder was prepared by conventional solid state reaction. Mixture of BaCO₃ and TiO₂ was homogenized for 24 h in ethanol, dried and calcined at 1100 °C for 2 h. After calcinations, starting barium titanate powder was modified by ultrasound treatment. The powder was dispersed in isopropanol and treated for 10, 60 and 180 minutes, with high-intensity ultrasound radiation using a direct-immersion titanium horn (Sonics VCX – 750, 20 kHz, 750 W). After the ultrasound process was stopped, the powders were filtered and dried.

The crystal structure of the barium titanate powders were investigated by XRD measurements. The FullProf program was used for structural refinement. The average particle size and particle size distribution were determined in isopropanol using a laser particle size analyzer (Mastersizer 2000, Malvern Instruments Ltd, UK). The specific surface area (BET) and pore size of powders were measured by N₂ adsorption-desorption isotherms at -195.8 °C on a Micromeritics® analyzer, while powders density were measured by pycnometry in hexane.

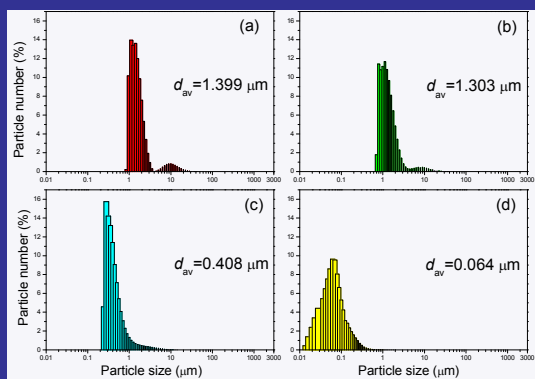
Introduction

The importance of the starting powders characteristics on the properties of the final ceramics is very well known from the literature. The classical route to prepare barium titanate powder is the solid state reaction between BaCO₃ and TiO₂ at temperature interval 1000-1200 °C. This is a widely used method for large bath processing for barium titanate based powders. Powders prepared by solid state reaction are agglomerated and aren't highly reactive toward sintering. A narrow particle size distribution and a small average size may be achieved by powders milling before pressing and sintering. During milling impurities can be introduced into system. Instead milling, sonochemical treatment can be used for powders activation.

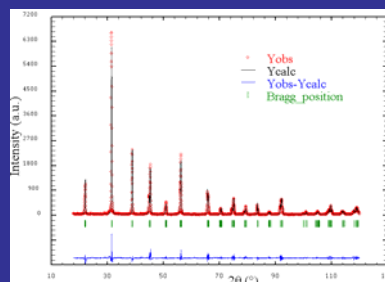
The particle size distribution of the starting barium titanate powder, prepared by solid state reaction, was very narrow, and average particle size was 1.399 μm. It is noticeable that during ultrasound treatment particles are deagglomerated, and average particle sizes decreases, while, the width of the particle size distribution is broadened.

Results and Discussion

All powders were high crystalline, pure barium titanate with tetragonal symmetry. The crystallite size in the BT powders were calculated from the half width of the XRD peaks of (002) and (200) planes, using by the Scherrer's equation. It can be noticed that these values are very similar, i.e. negligibly dependent on duration of ultrasound treatment.



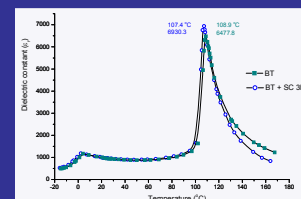
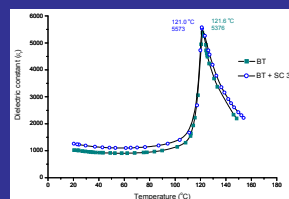
Particle size distribution of BaTiO₃ powders.
Sonication time: (a) 0; (b) 10; (c) 60 and (d) 180 min.



Observed, calculated and residual XRD of starting BaTiO₃ powder.

Characteristics of BaTiO₃ powders

Sonication time (min)	<i>d</i> _v	Crystalite size (Å)	Density (g/cm ³)	Average particle size (μm)	Specific surface area (m ² /g)
0	1.0077	(002) 318.6 (200) 695.7	5.58	1.399	1.01
10	1.0077	(002) 307.4 (200) 635.1	5.54	1.303	4.61
60	1.0077	(002) 327.4 (200) 645.3	5.63	0.408	4.79
180	1.0079	(002) 293.3 (200) 562.1	5.06	0.064	1.31



Temperature dependence of ε', in BT ceramics (BT and BT+SC 3h) sintered at 1370 and 1420 °C, 2h.

Results

of our investigations indicated that using by ultrasound irradiation we could improve physical characteristics of BaTiO₃ powder previously prepared by solid state reaction, without changing of powder's crystal structure. After three hours of ultrasound treatment of tetragonal BaTiO₃ we obtained nanometer sized, uniformly distributed particles, suitable for preparation of high density ceramics, and consequently, for ceramics with good dielectric properties.