11th International Conference
on Advanced Materials

Rio de Janeiro Brazil
September 20 - 25

ICAM2009

Synthesis of $Ba_xSr_{1-x}TiO_3$ nanoparticles obtained by Microwave-Assisted Hydrothermal Method

G.T.A. Santos[(1)*], A. E. Souza[(1)], R. A. Silva[(1)], M.L. Moreira[(2)], D.P. Volanti[(2)], S.R. Teixeira[(1)] and E. Longo[(2)]

(1) UNESP – Presidente Prudente, FCT/DFQB, e-mail: tadeu_gtas@hotmail.com
(2) UNESP – Araraquara, LiEC/CMDMC, UFSCAR – São Carlos
* Corresponding author.

Abstract – **$Ba_xSr_{1-x}TiO_3$** with x= (0, 0.25, 0.50, 0.75 and 1) were obtained by microwave-assisted hydrothermal method (MAH). The obtained powders were characterized by X-ray diffraction (XRD) and FT-Raman spectroscopy. The X-ray patterns confirm the $Ba_xSr_{1-x}TiO_3$ formation and show peaks dislocation with the growth of barium concentration changing the lattice parameter. Although, it is not clear the tetragonal structure of barium titanate in the X-ray pattern the Raman spectrum confirms this structure and shows the change from cubic symmetry (strontium titanate) to tetragonal symmetry (barium titanate). Scherrer's equation was used to calculate the average grain size which decreases with increasing Ba concentration.

Nanostructures can be obtained by sol-gel hydrothermal, solid state reactions, etc. The Microwave-Assisted Hydrothermal (MAH) method is an alternative synthesis process developed recently to prepare this material. It is a low-temperature and high reacting rates method which permit the synthesis of powdered ceramic materials in short times with uniform microstructure. Due to the short time and temperature reactions this technique allow to control unwanted grain growth and the final particle-size [1,2]. $Ba_xSr_{1-x}TiO_3$ (BST) is an important material which shows extensive applications in DRAM capacitors, phase shifters, phase array antennas, thermistors and pyroelectric detectors [3]. In this work we obtained BST with different barium concentration by using MAH.

The $Ba_xSr_{1-x}TiO_3$ (BST) nanoparticles were synthesized using a solution of barium chloride ($BaCl_2.2H_2O$) and strontium chloride ($SrCl_2.6H_2O$) in deionized water under constant stirring and nitrogen flowing out to avoid carbonate formation. Titanium (IV) isopropoxide ($C_{12}H_{28}O_4Ti$), 3.1 ml, and KOH (50 ml) were added to the solution. The reaction mixture was placed into a Teflon® autoclave which was sealed and placed in the microwave equipped with inventor technology MAH system (2.45 GHz and maximum power of 800 W). The solution was heated to 140°C (at 140°C/min) and was maintained at this temperature for 40 min under a pressure between 3 and 4 atm.

Figure 1 illustrates the X-ray diffraction patterns of all samples where can be identified barium (PDF 83-1878) and strontium (PDF 84-0444) titanates, intermediate compounds and strontium (PDF 84-1778) carbonate (impurity). It is observed that increasing barium concentration the peaks position shift to the left (low angles) increasing the lattice parameter. The average grain sizes were calculated via Scherrer's equation $D = k\lambda/(\beta \cos\theta)$, where k is the constant (shape factor about 1.0), λ the X-ray wavelength (1.542 Å), β the FWHM of the (110) diffraction line and θ its peak position. The average grain size decreases with increasing Ba concentration. Although, X-ray data did not show any evidence of tetragonal structure to these compounds the Raman data **(Fig. 2)** show a peak nearby 304 cm^{-1} an intrinsic peak for tetragonal $BaTiO_3$. In **Fig. 2** it can be observed that this peak appear with the increasing of Ba concentration i. e., when the structure changes from a cubic symmetry ($SrTiO_3$) to the tetragonal $BaTiO_3$. According the literature the tetragonal distortion (δ =(c – a)/a) is very low (1%) limiting the angular resolution of peak-splitting in particular for nanocrystalline materials. These results show that nanoparticles of pure $Ba_xSr_{1-x}TiO_3$ can be synthesized at low temperature in a short reaction time by using the MAH method.

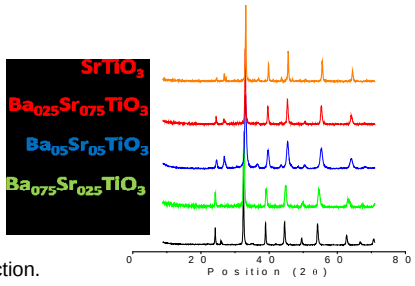

Figure 1: X-ray diffraction.

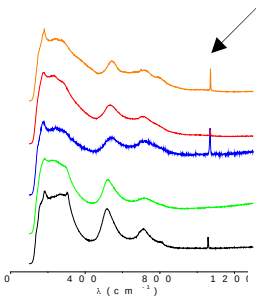

Figure 2: Raman spectrum.

[1] M. L. Moreira et. Al., Hydrothermal Microwave: A New Route to Obtain Photoluminescent Crystalline BaTiO3 Nanoparticles. Chem. Mater. 20 (2008) 5381–5387.
[2] R. R. Menezes, et al., Microwave sintering of ceramics. Part I: Fundamental aspects. Cerâmica. 53 (2007) 1-10.
[3] C. T. Lee et al., Structural and vibrational properties of $Ba_xSr_{1-x}TiO_3$ nanoparticles, J. Mater. Sci. 40 (2005) 1277-1279.