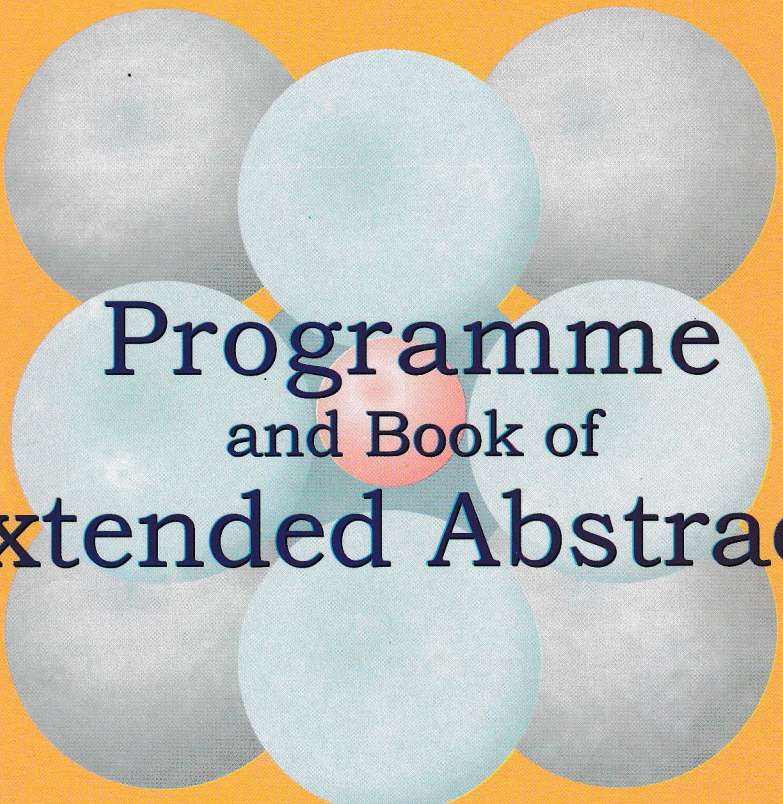


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Programme
and Book of
Extended Abstracts

3rd Workshop

*Nanostructured Materials:
Processing and Application*

September 2, 2007
Bled, Slovenia

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1. Introduction

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EFFECT OF LANTHANUM ON MICROSTRUCTURE OF BaTiO₃ PREPARED BY POLYMERIC PRECURSORS METHOD

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Due to its high dielectric constant and low loss characteristics, barium titanate has been used in many applications, such as capacitors, piezoelectric devices, positive temperature coefficient thermistors, semiconductors and etc. Doping of BaTiO₃ ceramics is very important for obtaining very interesting characteristics for various applications [1].

In this paper it was investigated the effect of doping BaTiO₃ with La on microstructure of BaTiO₃ ceramics.

Powders of pure barium titanate and barium titanate doped with 0.3 mol % La, in the presence of small amount of manganese (0.01 mol% Mn), were prepared by polymeric precursors method through Pechini process (soft chemistry) which was carried out as a three stage process from organometallic complex. Synthesized powders were pressed into a pallets using a cold isostatic press. Sintering was performed at 1300°C for 8h (Lenton furnace, UK). The heating rate was 10°C/min, with nature cooling in air atmosphere [2].

The X-ray results of samples sintered at 1300°C for 8h prepared from pure BaTiO₃ powder and doped BaTiO₃ are given on Fig.1. and Fig.2., respectively. It was approved that pure and doped BaTiO₃ were formed as tetragonal crystal structure.

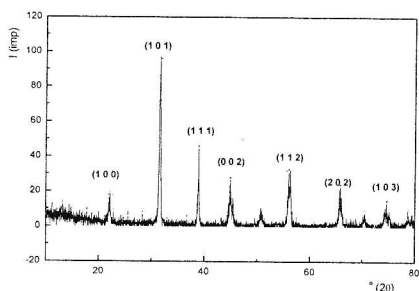


Figure 1. X-ray diffraction of sintered sample pure BaTiO₃

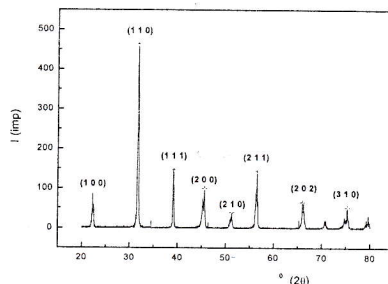


Figure 2. X-ray diffraction of sintered sample of doped BaTiO₃

Scanning electron microscopy observation was carried out to identify microstructure of the sintered samples. Sintered samples were prepared for observation by thermal etching at 1200°C for 20 min. Figs. 3. and 4. represent SEM photographs of BaTiO₃ sintered samples at 1300°C for 8h, pure and doped BaTiO₃, respectively.

It can be observed that La³⁺ inhibits grain growth and densification. The obtained microstructure possesses rather high uniformity of microstructure and rounded grain morphology [3]. Sintered samples of pure BaTiO₃ consists of polygonal grains with

different dimensions. Average doped 0.2 - 1 μm.



Figure 3. The microstructure of sintered sample

Reference

- [1] B.D. Stojanovic, M.A. Zec, Properties of donor doped BaTiO₃ (2002) 15-20.
- [2] W-S. Cho, E. Hamada, "Synthesis, structure and surface properties of BaTiO₃" (2002) 15-20.
- [3] M.T. Buscaglia, V. Buscaglia, "Effect of La³⁺ on the crystal structure of BaTiO₃" (2002) 15-20.

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different dimensions. Average particle size of pure BaTiO₃ is about 1.5 - 3.5 μm and for doped 0.2 - 1 μm.

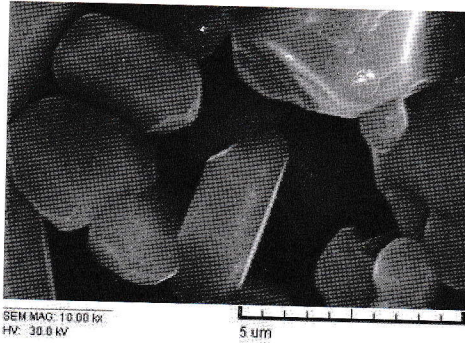


Figure 3. The microstructure of pure BaTiO₃ sintered sample

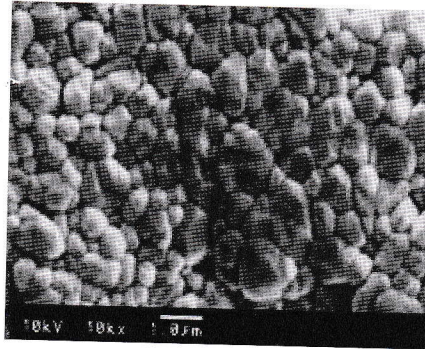


Figure 4. The microstructure of La doped BaTiO₃ sintered sample

Reference

- [1] B.D. Stojanovic, M.A. Zaghete, C.R. Foschini, F.O.S. Vieira, J.A. Varela, "Structure and properties of donor doped barium titanate prepared by citrate process", *Ferroelectrics*, **270** (2002) 15-20.
- [2] W-S. Cho, E. Hamada, "Synthesis of ultra fine BaTiO₃ particles from polymeric precursor: their structure and surface property", *J Alloys and Compounds*, **266** (1998) 118-122.
- [3] M.T. Buscaglia, V. Buscaglia, M. Viviani, P. Nanni, M. Hanuskova, "Influence of foreign ions on the crystal structure of BaTiO₃", *J. Eur. Ceram. Soc.*, **20** (2000) 1997-2007

COST-P-15

MICROSTRUCTURE DEVELOPMENT OF $\text{Bi}_4\text{Ti}_3\text{O}_{12}$ AND $\text{BaBi}_4\text{Ti}_4\text{O}_{15}$ CERAMICS PREPARED BY MECHANOCHEMICAL SYNTHESIS

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Several bismuth-layered crystal structure and their properties have been investigated in detail. However, a lot of aspects of the preparation and properties of barium bismuth titanate unexplored, whereas being promising candidate for memory applications.

In present work barium-bismuth titanate ($\text{BaBi}_4\text{Ti}_4\text{O}_{15}$ -BBT) was prepared from stoichiometric quantities of barium titanate and bismuth titanate obtained via mechanochemical synthesis. Barium titanate ($\text{BaTi}_3\text{O}_{12}$ -BT) has been synthesised from mixture of BaO and TiO_2 and bismuth titanate ($\text{Bi}_4\text{Ti}_3\text{O}_{12}$ -BIT) was prepared starting from Bi_2O_3 and TiO_2 , commercially available. Mechanochemical synthesis was performed in air atmosphere in a planetary ball mill, for BT during 60 min and for BIT during 360 min. Milling conditions were: zirconium oxide jars and zirconium oxide balls, ball-to-powder weight ratio 20:1 and determined basic disc and disc with jars rotation speed. The powder mixture of BT and BIT was homogenized for 30 min and after that sintered at 1100°C for 4h. Separatly, BIT was sintered at 1000°C for 12h, in both cases without pre-calcination step and by conventional sintering technique.

The Fig. 1. shows the phase formation and crystal structure of BIT, BT and mixture of this powders. Pattern of BBT powder sintered at 1100°C for 4h was analysed by XRD analysis revealing the existance of tetragonal phase (Fig. 2.).

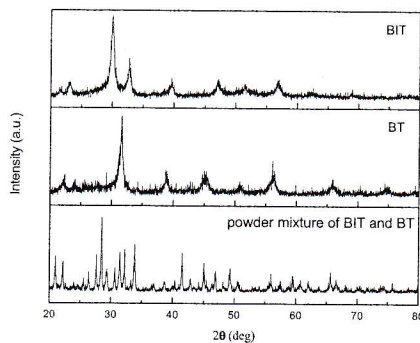


Figure 1. XRD powder of BIT, BT and mixture of BIT and BT

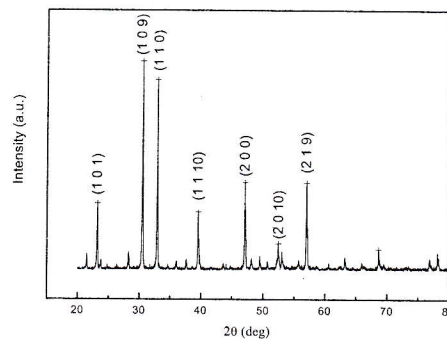


Figure 2. XRD pattern of BBT powder sintered at 1100°C for 4h

The morphology of obtained powders was examined by SEM and TEM method (Fig. 3. and Fig. 4.). It can be conclude that pattern of BIT consists of nanoparticles which size is less than 20 nm. Mixture of BIT and BT consists of agglomerates of varying size and morphology which size is about 250 nm.



Figure 3. SEM micrograph of powder BIT and BT

The microstructure development... From Fig. 5. and Fig. 6. it can be seen that in the case of BBT beside plate-like morphology... addition leads to the change in morphology.



Figure 5. SEM micrograph of BBT powder sintered at 1100°C

Reference

- [1] Z.S. Macedo, M.H. Lente, *J. Appl. Phys.*, **81** (1997) 2811-2818.
- [2] Ismunandar, T. Kamiyama, *J. Solid State Chem.*, **177** (2004) 41-48.



Figure 3. SEM micrograph of powder mixture of BIT and BT

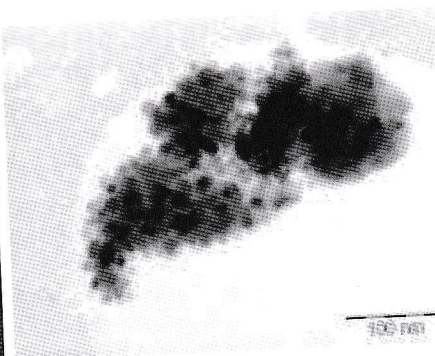


Figure 4. TEM image of crystalline/amorphous $\text{Bi}_4\text{Ti}_3\text{O}_{12}$ powder obtained after milling of 6h

The microstructure development of BIT and BBT ceramics was followed by SEM. From Fig. 5. and Fig. 6. it can be observed that BIT consists of plate-like grains but in the case of BBT beside plate-like grains exist and spheric grains, also. It is evident that Ba^{2+} addition leads to the change in the microstructure development.

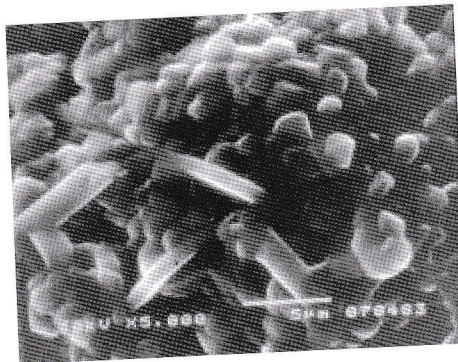


Figure 5. SEM micrograph pattern of BBT powder sintered at 1100°C for 4h

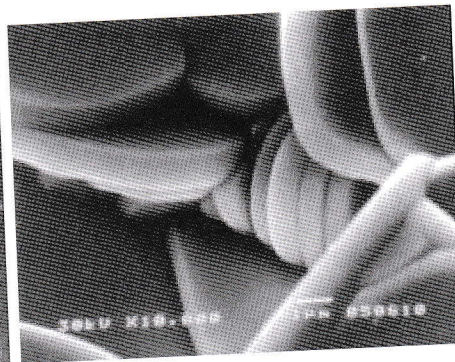


Figure 6. SEM micrograph pattern of BIT powder sintered at 1000°C for 12h

Reference

- [1] Z.S. Macedo, M.H. Lente, J.A. Eiras, A.C. Hernandez, *J. Phys. Condens Matter.*, **16** (2004) 2811-2818.
- [2] Ismunandar, T. Kamiyama, A. Hoshikawa, Q. Zhou, B.J.Kennedy, Y. Kubota, K. Kato, *J. Solid State Chem.*, **177** (2004) 4188-4196.