



COST 539 Action - ELENA



**Programme
&
Book of abstracts**

NanoMetro Workshop

January 31, 2008
Leysin, Switzerland



Programme and Book of abstracts of the NanoMetro Workshop COST 539

Editors

Dr. Paul Bowen

Dr. Nathalie Jongen

Press

Atelier de Reprographie de l'EPFL, Lausanne, Switzerland

3. Programme of the NanoMetro Workshop

Morning Session

08:30 - 09:00 Invited talk 1

CHARACTERISATION OF NANO- AND MICRODISPERSIONS USING
MULTISAMPLE ANALYTICAL CENTRIFUGATION: INTERPARTICLE FORCES,
DEFORMABILITY AND PARTICLE SIZE

Dietmar Lerche, Torsten Detloff, Titus Sobisch

*Dispersion Stability & Particle Analysis, L.U.M. GmbH, Rudower Chaussee 29, 12489
Berlin, Germany*

09:00 - 09:15 Short talk 1

PREPARATION AND CHARACTERIZATION OF LaNiO_3 NANOCRYSTALLINE
POWDER

Přemysl Vaněk¹, Jan Drahokoupil¹, Radmila Krupková¹, Karel Maca²,
Jiří Plocek³, and Petr Wandrol⁴

¹*Institute of Physics ASCR, v.v.i., Na Slovance 2, CZ-18221 Praha 8, Czech Rep.*

²*Brno University of Technology, Faculty of Mechanical Engineering, Department of
Ceramics, Technická 2896/2, CZ-61669 Brno, Czech. Rep.*

³*Institute of Inorganic Chemistry ASCR, v.v.i., CZ-25068 Řež, Czech Rep.*

⁴*Institute of Scientific Instruments ASCR, v.v.i., Královopolská 147, CZ-61264 Brno,
Czech Rep.*

09:15 - 09:30 Short talk 2

X-RAY STUDIES OF PbTiO_3 NANOPOWDER PRODUCED BY MECHANICAL
SYNTHESIS

Izabela Szafraniak-Wiza¹, Adam Pietraszko², Wiktor Walerczyk²

¹*Institute of Molecular Physics, Polish Academy of Sciences, M. Smoluchowskiego 17,
60-179 Poznań, Poland*

²*Institute of Low Temperature and Structure Research, Polish Academy of Sciences,
Okólna 2, 50-422 Wrocław, Poland*

09:30 - 09:45 Short talk 3

PREPARATION OF FINE BaCeO_3 AND $\text{BaCe}_{0.9}\text{Y}_{0.1}\text{O}_{3.8}$ POWDERS

A. Bassano^{a,b}, M. Viviani^b, V. Buscaglia^b, M. T. Buscaglia^b, P. Nanni^{a,b}

^a*Dept. of Process and Chemical Engineering, University of Genoa, P.le Kennedy Pad. D
16129, Genoa, Italy*

^b*Institute for Energetics and Interphases, Dept. of Genoa, National Research Council, Via
De Marini 6, 16149 Genoa, Italy*

09:45 - 10:00 Short talk 4

MECHANO-SYNTHESIS OF SODIUM NIOBATE AT DIFFERENT BALL-IMPACT ENERGIES

Tadej Rojac, Marija Kosec, Barbara Malič, Janez Holc

Jozef Stefan Institute, Jamova cesta 39, 1000 Ljubljana, Slovenia

10:00 - 10:30 COFFEE BREAK AND POSTERS

10:30 - 11:00 Invited talk 2

CRYSTALLITE SIZE BY X-RAY POWDER DIFFRACTION

Frank Kubel

Institut für Chemische Technologien und Analytik, Fakultät für Technische Chemie, TU Wien, Getreidemarkt 9, 1070 Wien, Austria

11:00 - 11:15 Short talk 5

CHARACTERIZATION OF BiFeO₃ OBTAINED BY MECHANOCHEMICAL SYNTHESIS

Zorica Marinković Stanojević, Biljana Stojanović

Institute for Multidisciplinary Research, Kneza Višeslava 1a, 11030 Belgrade, Serbia

11:15 - 11:30 Short talk 6

PRODUCTION OF COMPLEX PROTON CONDUCTING OXIDE POWDERS BY SPRAY PYROLYSIS

Kjell Wiik, Tommy Mokkelbost, Trine Øyås, Mari-Ann Einarsrud, Tor Grande, Rune T. Barland

Department of Materials Science and Engineering, Norwegian University of Science and Technology (NTNU), 7491 Trondheim, Norway

11:30 - 11:45 Short talk 7

CHARACTERIZATION OF BARIUM TITANATE POWDERS PREPARED BY POLYMERIC PRECURSORS METHOD

M.M. Vijatović¹, J.D. Bobić¹, B.D. Stojanović¹, P. Bowen²

¹*Institute for Multidisciplinary Researches, Kneza Višeslava 1, 11000 Belgrade, Serbia*

²*Powder Technology Laboratory, Ecole Polytechnique Fédérale de Lausanne, Station 12, 1015 Lausanne, Switzerland*

11:45-12:00 Short talk 8

ONE DIMENSIONAL TITANIA-BASED MATERIALS

Marija Maletin¹, Ružica R. Đenadić^{1,2}, Ljubica M. Nikolić¹, Vladimir V. Srdić¹

¹*Department of Materials Engineering, Faculty of Technology, University of Novi Sad, Bul. Cara Lazara 1, 21000 Novi Sad, Serbia*

²*Nanoparticle Process Technology, Department of Engineering Science, University Duisburg-Essen, Lotharstr. 1, Duisburg, Germany*

Short Talk 7**Characterization of Barium Titanate Powders Prepared by Polymeric Precursors Method**M.M. Vijatović¹, J.D. Bobić¹, B.D. Stojanović¹, P. Bowen²¹*Institute for Multidisciplinary Researches, Kneza Višeslava 1, 11000 Belgrade, Serbia*²*Powder Technology Laboratory, Ecole Polytechnique Fédérale de Lausanne, Station 12, 1015 Lausanne, Switzerland***Abstract**

Barium titanate is the first ferroelectric ceramics and a good candidate for variety applications due to its excellent dielectric, ferroelectric and piezoelectric properties. Barium titanate is a member of large family of compounds with general formula ABO_3 which is called perovskite [1]. Barium titanate is the first discovered ferroelectric perovskite. Doping of $BaTiO_3$ ceramics is very important for obtaining very interesting characteristics for various applications. Barium titanate is normally an insulator but after doping with trivalent donors such as La it becomes semiconductive.

Synthesis method depends of the desired characteristics for the end application and it was distinguish a significant influence of used method on structure and properties of barium titanate materials.

The aim of this work is to characterize pure and doped barium titanate powders prepared by polymeric precursors method. Pechini process is described below. Firstly, titanium citrate and barium citrate solutions were prepared, as a source of titanium was used titanium iso-propoxide and as source of barium was used barium acetate. Solutions of titanium citrate and barium citrate were mixed, with constant stirring. For doping barium titanate, in this mixture was added $La(NO_3)_3$ solution (0.1 mol% La and 0.3mol% La). Temperature was raised up to 120 - 140 °C, when the solution becomes solidified into a dark – brown glassy resin. Decomposition of the most organic part was performed in the oven with special caution. The temperature regime was 200 °C for 4h. When the resin incinerated, and becomes black solid mass, then material was pulverized. Material was thermally treated at 500°C for 4h and 700 °C for 4h. The heating rate was 2 °C/min. After drying at room temperature and querying the barium titanate powders were obtained. The advantage of this method is based on the fact of its simplicity for obtaining powders of high purity and possibility to hold the initial stoichiometry [2].

Particle size distributions (PSDs) were measured using laser diffraction (Malvern Mastersizer S), specific surface areas (SSA) were measured by nitrogen adsorption (Gemini 2375, Micromeritics) and average particle diameters (D_{BET}) were calculated from the SSA ($6/\rho_{theoretic} * SSA$) and agglomeration factor, F_{agg} calculated by dividing the D_{V50} with the D_{BET} . Density of pure and doped barium titanate powders were measured using Pycnometer (Micromeritics AccuPyc 1330). The microstructure and morphology of nanopowders were investigated using X-ray diffraction and scanning electron microscopy. Obtained results are given in the table below.

Table I. Results for pure and doped barium titanate powders

Sample	D _{V10} (nm)	D _{V50} (nm)	D _{V90} (nm)	Span	SSA (m ² /g)	D _{BET} (nm)	F _{agg}	Density (g/cm ³)	D _{SEM} (nm)
pure BT	1920	6530	13690	1.80	13.47	74.07	88.16	5.7186	40
BT 0.1% La	2110	7080	14610	1.76	10.98	90.85	77.93	5.7388	40
BT 0.3%La	2580	7820	16570	1.79	13.48	74.00	105.677	5.7172	26

From the experimental results it can be concluded that powders of barium titanate obtained by Pechini process are nanosized. SEM observation indicates that higher percentage of La inhibits grain growth in doped barium titanate.

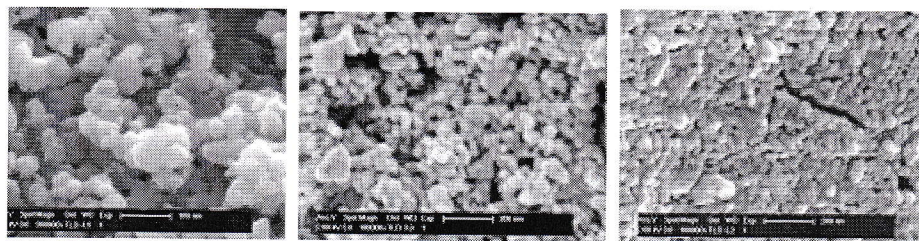


Figure 1. SEM photographs of pure, doped with 0.1 mol% La and 0.3 mol% La barium titanate powders, respectively

Particle size distribution measurement pointed that the powders are highly agglomerated. Figure 2. shows particle size distribution for pure barium titanate powder. The next step in our work will be addressed to reducing the particle size by attrition milling with zirconia media and these results will be presented and discussed.

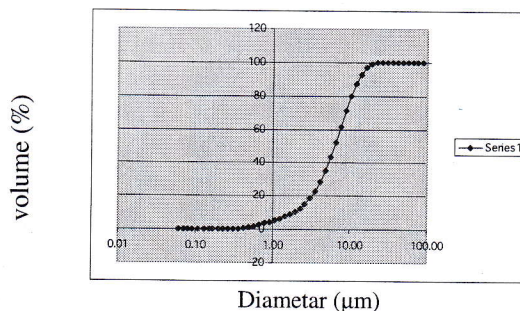


Figure 2. Particle size distributions for pure barium titanate

- [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] "Materiais Ferroeletricos com Estrutura Perovskita: Sistemas de titanato de bario comportamento de PTCR" projeto de CMDMC (LIEC, UNESP – Instituto de Quimica, Araraquara e LIEC, UFSCar, Sao Carlos), 2003.