

**PS10.01.13 EFFECT OF HIGH-PRESSURE ON POLYTYPE AND STACKING DISORDER IN SINTERED SiC.** S. Stel'makh, S. Gierlotka, B. Palosz, Murali Mohan\*, C. Divakar\*, S.K.Bhaumik\* & A.K.Singh\*. High Pressure Research Center, UNIPRESS, Warsaw, POLAND, \*National Aerospace Laboratories NAL, Bangalore, INDIA

Isostatic high pressure technique was used for sintering of SiC polycrystals. Belt apparatus (pressure up-to 8 GPa) and cubic anvil cell (3 GPa) in the temperature range up-to 2200°C were used. The following SiC polycrystals were examined:  $\alpha$ -SiC (STARCK),  $\beta$ -SiC powder obtained by Self-propagating High-temperature Synthesis (SHS), High Pressure self-Combustion Synthesis (HPCS) and also a series on nanocrystalline SiC powders. Comparative structural studies of these materials sintered without external pressure and with application of high isostatic pressure were performed. The structural analysis was performed with the use of a method of computer modelling of disordered structures and simulation of corresponding diffraction patterns. This method permitted us for identification of stacking faults that occur in starting materials and to follow the structural changes induced under high temperature and high pressure. The SiC polycrystals synthesized by SHS method under high pressure (3-6 GPa) contain only very little number of stacking faults. Very strong inhomogeneous stresses applied to starting powders (compacted without liquid phase) generate microtwins and stacking faults at low temperature. At high temperature, the thermally activated dislocation motions lead to pressure-induced structural changes in the stacking of the layers. These changes always reduce one-dimensional disorder present in starting polycrystals. They are not however inter-polytype transformations that originate from thermodynamic conditions and relative stability of cubic 3C and  $\alpha$ -type polytypes: They obviously follow changes of the dislocation network forced by pressure and activated at high temperature.

**PS10.01.14 BaTiO<sub>3</sub> SYNTHESIS PARAMETER DETERMINATION VIA X-RAY POWDER DIFFRACTION ANALYSIS.** C. Thompson<sup>1</sup>, G.J.Kruger<sup>1</sup> and J.D. van Wyk<sup>2</sup>, <sup>1</sup>Department of Chemistry and Biochemistry, <sup>2</sup>Research Group for Industrial Electronic Technology, Rand Afrikaans University, Johannesburg-Auckland Park 2006, South Africa.

X-ray powder diffraction was used to study the effects of certain parameters in a revised sol-gel synthesis of barium titanate. This method involved the formation of a sol consisting of barium acetate and titanium isopropoxide dissolved in glacial acetic acid. Gelation was obtained by the addition of water to the sol. In order to determine the influence of certain parameters within the procedure, these were each individually adjusted. The gel products were dried and calcined at 900°C to yield the final products and analysed for composition by x-ray powder diffraction. The effect of the parameters were determined by studying the composition of the product. For instance, addition of an organic base DBU (diazabicycloundecene) instead of water to obtain gelation yielded a final product consisting of several undesirable phases, instead of the required tetragonal BaTiO<sub>3</sub>. Further, if no addition of water was made to institute gelation, the formation of a colloidal precipitate still occurred, although from the XRD analysis it was seen to contain several unwanted phases. However, if water was subsequently added to the colloidal gel, the calcined product was shown to be pure tetragonal barium titanate. Increasing dilution of the acetic acid by water before the addition of titanium isopropoxide was found to correspond with an increase in contaminating phases in the final product, as determined by x-ray diffraction. These and other facts obtained made it possible to determine the parameters necessary to obtain pure tetragonal barium titanate via the revised sol-gel process.

**PR10.01.15 FERROELASTIC DOMAIN SWITCHING AND PHASE TRANSFORMATIONS IN Ce-TZP.** E.H. Kisi, Department of Mechanical Engineering, The University of Newcastle, Callaghan, NSW 2308, Australia; S.J. Kennedy and C.J. Howard, Australian Nuclear Science and Technology Organisation, Menai, NSW 2234, Australia

A ceria-tetragonal zirconia polycrystal (Ce-TZP) ceramic has been studied, by neutron powder diffraction, under uniaxial compressive stresses to 1.6 GPa. At stresses above about 1.2 GPa, ferroelastic switching of tetragonal material and the tetragonal to monoclinic phase transformation were both observed. The stress-strain plot was simultaneously recorded making use of a strain gauge parallel to the applied stress. Neutron diffraction patterns were analysed by the Rietveld method, the parameters of interest being the lattice parameters (responding to the stress), the preferred orientation parameter for the tetragonal phase (a measure of the ferroelastic switching), and the fraction of monoclinic phase. At stresses to 1.2 GPa, the stress-strain plot was essentially linear, and the sample remained entirely in the tetragonal phase. There was some deterioration in the Rietveld fit with increasing stress, attributed to elastic anisotropy in the tetragonal phase. At stresses above 1.2 GPa, there was a change in slope of the macroscopic stress-strain curve, a change in the value of the preferred orientation parameter (onset of ferroelastic switching), and the appearance of monoclinic phase. Lattice parameters showed anomalous variation in the same stress range. Both ferroelastic switching and tetragonal to monoclinic transformation were partly reversed when the stress was removed. It is interesting that tetragonal ferroelastic switching and the tetragonal to monoclinic transformation both occur in Ce-TZP. It may be that one of these processes triggers the other, but it is not clear from the measurements reported here which occurs first. A similar study of Y-TZP (Cain, Bennington, Lewis and Hull, *Philos. Mag.*, 1994, B 69, 499-507) has indicated a higher threshold stress for ferroelastic switching and no monoclinic phase, suggesting that the tetragonal to monoclinic phase transformation in Ce-TZP may act as the trigger for the switching.

**PS10.01.16 CORRELATION BETWEEN THE STRUCTURE AND PROPERTIES OF RARE-EARTH TITANATES WITH FORMULA (LnM)TiO<sub>3</sub>.** A.G. Belous. Department of Electrophysical Materials, Institute of General and Inorganic Chemistry, Kiev, Ukraine

The paper examines the synthesis conditions for heterosubstituted titanates with perovskite structure with formula (LnM)TiO<sub>3</sub>, where Ln is the rare-earth element and M the alkali or alkaline-earth metal.

It was shown that it is possible to stabilize perovskite structure in rare-earth titanates, (Ln<sub>2/3-x</sub>M<sub>3x</sub>)TiO<sub>3</sub>, by the partial substitution of rare-earth ions and filling of vacancies by alkali metal ions. Rare-earth ions play the main role in perovskite structure formation, and alkali metal ions fill vacancies and stabilize them thereby. It was found that perovskite structure in (Ln<sub>2/3-x</sub>M<sub>3x</sub>)TiO<sub>3</sub> compounds is formed at  $1/24 < X < 1/6$ .

A lithium-ion conducting ceramic based on lanthanum and lithium titanates with perovskite structure has been obtained for the first time. It was shown that the partial substitution of rare-earth ions by sodium and potassium ions in compounds with formula Ln<sub>2/3</sub>TiO<sub>3</sub> leads to the formation of MW dielectrics with temperature-stable dielectric constant.