An increasing interest in electrolyte materials for advanced energy applications demands understanding of their real structure and its influence on the physical properties. It is therefore relevant to investigate both the crystal- and micro-structures, since the considered properties depend on domain walls and their distribution (twin structures) [1]. ZrO2 doped with Sc, and LaGaO3 doped Sr and Mg, are considered as prospective solid electrolytes for application in solid oxide fuel cells (SOFCs). The present work is devoted to structure investigations of ZrO2 doped with 10 mol % Sc2O3 (ZSO-10) and La0.95Sr0.05Ga0.95Mg0.05O3-δ (LSGM-05) and determination their twin structures in their ferroelastic phases. In order to study the thermal evolution of their crystal structures were carried out at the synchrotron powder diffractometer B2 (HASYLAB/DESY). High-temperature diffraction data were collected in the Debye-Scherrer capillary geometry using the on-line image plate detector OBI and the STOE furnace. Data analysis was carried out by the Rietveld method using the WinCSD program package. Domain orientations were determined by the Laue method. The white beam synchrotron experiments have been carried out using the Kappa-diffractometer at F1 (HASYLAB) equipped with a MAR CCD system and a gas-stream heating device. The powder diffraction examinations revealed that the rhombohedral structure (space group R3̄) of ZSO-10 transforms into the high-temperature cubic structure (space group Fm̅3 m) at 873 K, while LSGM-05 transforms at 500 K from orthorhombic to monoclinic and at 670 K to rhombohedral phase. Analysis of the Laue patterns confirms that the ZSO-10 crystal was twinned relatively to intersecting (101) and (100)/(001) mirror planes in the rhombohedral phase, while LSGM-05 was twinned relatively to intersecting (011) and (211)/(211) mirror planes in the orthorhombic phase. In both crystals the twin structure tends to form typical “chevron-like” wall configurations that allow for a stress-free co-existence of four different orientation states. These four orientation states occur because they perfectly match geometrically and no additional stress occurs at the intersections of domain walls throughout the full temperature range of the corresponding phases [2]. The work was supported by WTTZ (UKR 07/009) and Ukrainian Ministry of Science (project “Segnet”).

Keywords: crystal structure; ferroelastic; electrolytes; solid oxide fuel cells; structure investigations.
Samples of the (1 - x) BaSc1/2Ta1/2O3 solid solution were prepared from high purity BaCO3, TiO2, Sc2O3 and Ta2O5 powders using solid state methods. All these materials were previously dried at 120°C for 15 h, weighed for 1 h and calcined at 1200°C for 15 h. After calcinations, powders were mixed for 1 h and pressed under 100 MPa into 8mm diameter and about 1 mm thick. The pellets were then sintered in oxygen atmosphere at 1350°C for 4 h. Room temperature powder XRD patterns were recorded on a Philips diffractometer X'Pert Pro MPD using CuKα radiation (5° ≤ 2θ ≤ 80°). The X-ray diffraction pattern for ceramics with compositions x = 0.025 and 0.05 were investigated. The results obtained suggest that these compositions have respectively tetragonal and cubic symmetry at room temperature. The dielectric measurements were performed on ceramic discs after deposition of gold electrodes on the circular faces by cathodic sputtering. The dielectric permittivity discs after deposition of gold electrodes on the circular faces by cathodic sputtering. The dielectric permittivity peaks have been observed and correspond at 102 - 210 Hz. For composition close to BaTiO3, three types of micro- and nanodomains [2]. The tetragonal to cubic phase transition in BaSc1/2Ta1/2O3 ceramics is associated with a change in the microstructure and a decrease in the dielectric permittivity. The observed superstructure reflections reveal new structural aspects of the low temperature phases. A study of combined neutron and synchrotron high resolution powder diffraction data refinement (MS beamline at the SLS in Villingen) gives a detailed overview of the structural changes across the MPB. Temperature dependent measurements at the beamline B2 in Hamburg provide an insight into the phase composition in the vicinity of the MPB and will be compared with undoped PZT. These results are complemented by high resolution neutron powder diffraction data collected at SPODI at the FRM II in Munich at low temperatures that describe the compositional dependent structural evolution from the rhombohedral to the tetragonal side of the phase diagram. The observed superstructure reflections reveal new structural aspects of the low temperature phases. A study of combined neutron and synchrotron high resolution powder diffraction data refinement (MS beamline at the SLS in Villingen) gives a detailed overview of the structural changes across the MPB. Results will be discussed together with transmission electron microscopic observations. The authors appreciate the financial support of the German Research Foundation (DFG) through the Sonderforschungsbereich 595 „Electric Fatigue in Functional Materials“. [1] Noheda B., Gonzalez J.A., Cross L.E., Guo R., Park S.E., Cox D.E., Shirane G., Phys. Rev. B, 2000, 61. [2] Schmitt L.A., Schönau K.A., Theissmann R., Fuess H., Kungl H., Hoffmann M.J., J. Appl. Phys., 2007, 101, 074107.

Keywords: synchrotron powder diffraction; neutron high-resolution diffractometry; piezoelectric ceramics

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Combined Refinement of High Resolution Neutron and Synchrotron Data of PLZT. Manuel Hinterstein, Roland Schierholz, Markus Hölzel, Anatoliy Senyshyn, Jens Kling, Ljubomira Ana Schmitt, Hans-Joachim Kleebe, Hans Kungl, Michael Knapp, Hartmut Fuess. *Institute for Materials Science and Geosciences, University of Technology Darmstadt, Germany. †Institute for Ceramics in Mechanical Engineering, University Karlsruhe, Germany, ‡CELLS, Barcelona, Spain.

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Lead containing oxides with perovskite structure like PbZr1-xTi1-xO3 (PZT) are widely used as sensors and actuators. Especially, solid solutions near the rhombohedral-tetragonal morphotropic phase boundary (MPB) possess eminent piezoelectric characteristics and are widely used in a donor or acceptor doped modification with improved electrical properties. Despite extensive studies the microstructure of the morphotropic phase boundary (MPB) in ferroelectric PZT is still under discussion. Whereas some groups (Noheda et al [1]) fitted diffraction data by monoclinic symmetry, other groups describe the MPB as composed of a complicated system of micro- and nanodomains [2]. Extensive studies have been performed on donor doped Pb0.985La0.015(Zr0.21Ti0.79)O3 (PLZT) across the entire compositional range of the MPB. Temperature dependent measurements at the beamline B2 in Hamburg provide an insight into the phase composition in the vicinity of the MPB and will be compared with undoped PZT. These results are complemented by high resolution neutron powder diffraction data collected at SPODI at the FRM II in Munich at low temperatures that describe the compositional dependent structural evolution from the rhombohedral to the tetragonal side of the phase diagram. The observed superstructure reflections reveal new structural aspects of the low temperature phases. A study of combined neutron and synchrotron high resolution powder diffraction data refinement (MS beamline at the SLS in Villingen) gives a detailed overview of the structural changes across the MPB. Results will be discussed together with transmission electron microscopic observations. The authors appreciate the financial support of the German Research Foundation (DFG) through the Sonderforschungsbereich 595 „Electric Fatigue in Functional Materials“. [1] Noheda B., Gonzalez J.A., Cross L.E., Guo R., Park S.E., Cox D.E., Shirane G., J. Appl. Phys., 2000, 61. [2] Schmitt L.A., Schönau K.A., Theissmann R., Fuess H., Kungl H., Hoffmann M.J., J. Appl. Phys., 2007, 101, 074107.

Keywords: synchrotron powder diffraction; neutron high-resolution diffractometry; piezoelectric ceramics

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Probing the Giant Piezoelectric Effect at the Atomic Scale in PbZn1/3Nb2/3O3. Jérôme Rouquette, Ali Al-Zein, Julien Haines, Philippe Papet, Claire Leulent, Hichem Dammak, Olivier Mathon, ICG UMR CNRS 5253, PMOF, Montpellier, France. *LCVN UMR CNRS 5587, Montpellier. †SPMS UMR CNRS 5580, ECP, Châtenay-Malabry, France. ‡ESRF, Grenoble, France. E-mail: Jerome.Rouquette@univ-montp2.fr