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Local Symmetry in $PbZr_xTi_{1-x}O_3$-Ceramics

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The symmetry of $PbZr_xTi_{1-x}O_3$ in the region of the morphotropic phase boundary is still under debate. Noheda *et al.* [1] claimed the existence of a monoclinic phase. In contrast to this Jin *et al.* [2] showed for relaxor ceramics that, if the width of tetragonal microdomains is smaller than the diffraction coherence length, the measured crystal lattice constants are of monoclinic symmetry.

With convergent-beam electron diffraction (CBED) very small volumes can be examined. So crystal symmetry can be investigated on single domains. To distinguish the most probable phases with space group symmetry P4mm, R3m and Cm, just one zone-axis is needed. At most two projected CBED-patterns of neighbouring domains are necessary. The method will be explained by the use of simulated and experimental CBED-patterns.

[1] Noheda B., Gonzalo J.A., Cross L.E., Guo R., Park S.E., Cox D.E., Shirane G., *Phys. Rev. B*, 2000, **61** 8687. [2] Jin Y.M., Wang Y.U., Khachaturyan A.G., Li J.F., Viehland D., *Phys. Rev. Lett*., 2003, **91**, 197601.

Keywords: convergent-beam electron diffraction, crystal symmetry, microdomains

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Local Order in the PSN, PST and PSNT Ferroelectric Relaxors

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Diffuse X-ray scattering studies were performed for as grown $Pb(Sc_{0.5}Nb_{0.5})O_3$ - PSN, $Pb(Sc_{0.5}Ta_{0.5})O_3$ - PST and $Pb(Sc_{0.5}Nb_{0.2}Ta_{0.3})O_3$ - PSNT which belong to the ferroelectric relaxors family of complex $Pb(B^{3+}_{0.5}B^{5+}_{0.5})O_3$ perovskites in which the degree of order could be controlled by the thermal history [1]. In the disordered state the ferroelectric relaxors consist of a nonpolar matrix (*Pm3m* space group) that contains nanosize chemical domains and polar nanodomains [2,3]. The crystal structures of PSN, PST and PSNT were studied on single crystals at several temperature points in the range from 300 to 550 K by means of a four-circle single crystal diffractometer equipped with CCD area detector. Diffuse streaks and superstructure reflections were found in PSN, PST and PSNT crystals. The diffraction patterns of the disordered structures for the relaxors of the 1:1 composition were simulated using the DISCUS program [4] and compared with those obtained experimentally.

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In-situ Electric Field Synchrotron Diffraction of $PbZr_{(x)}Ti_{(1-x)}O_3$ Microdomains

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The structure of $PbZr_xTi_{1-x}O_3$ at its morphotropic phase boundary (MBP) and the influence of electric field on the domain structure in this area have been the subject of controversy. While Noheda et al. [1] proposed a monoclinic "bridging" phase at the MPB without considering the real structure, Jin et al. [2] describe an adaptive phase of tetragonal microdomains in relaxors, which are not resolvable with X-rays and therefore only appear to be of monoclinic symmetry.

In this work, a correlation is drawn between these two models, and the variation of the domain structure with composition and electric field is investigated. Rietveld refinement and analysis of ex-situ and in-situ electric field high-resolution synchrotron X-ray diffraction data of polycrystalline pellets (~60µm), measured in transmission geometry, and TEM studies indicate various changes in domain structure across the MPB. A lowering of the domain wall energy by a decrease in c/a-ratio of the tetragonal phase with lower Ti fraction leads to a simultaneous increase in content of microdomains – the adaptive phase, which has a tremendous influence on the poling behaviour of the material.

[1] Noheda B., Gonzalo J.A., Cross L.E., Guo R., Park S.E., Cox D.E., Shirane G., *Phys. Rev. B*, 2000, **61**, 8687. [2] Jin Y.M., Wang Y.U., Khachaturyan A.G., Li J.F., Viehland D., *Phys. Rev. Lett*., 2003, **91**, 197601.

Keywords: high-resolution X-ray diffraction, in-situ electric field, ferroelectric microdomains

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Analysis of the Crystalline Phases of PLZT doped with Nd, Ho, Er, Tm, Yb

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The addition of La to PZT improves densification, transparency and electro-optical properties. To further improve those qualities samples of PLZT doped with Yb, Ho, Tm, Er and Nd were produced and characterized by XRD (Rigaku-Denki rotating anode source RU-200 B, λ= 1.5408, i=100mA, V=50kV, θ-2θ scan from 15 to 120^o, step 0.02^o, time = 5sec, room temperature, Rietveld Method, GSAS package of programs) Samples of PLZT 65/35 and 9% La were produced with 1, 2, 4% of Yb and syntherized at room temperature. They presented systematically a mixture of Rhombic (R3cH) and tetragonal (P4/mm) phases. It was observed that less than 1% Yb incorporated with the excess forming a third phase of $Zr_2Yb_4O_{12}$. For this reason PLZT samples containing only 1% of rare earth were prepared and syntherized at high temperature. It was observed the formation of only the rhombohedral phase. The rare earth ions occupied both sites A and B of the perovskyte structure with sof respectively of 0.099 and 0.0232 for Nd, 0.0102 and 0.0130 for Ho, 0.0179 and 0.0164 for Er, 0.058 and 0.0215 for Tm and 0.00 and 0.0028 for Yb.

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In-situ Study of Residual Strain in Solid Oxide Fuel Cells

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Solid Oxide Fuel Cells (SOFC) are electrochemical devices converting the chemical energy of a fuel directly to electricity. Due to the high efficiency of the process they are considered as one of the most promising power production technologies of the future.

Among various designs of SOFCs, the so called flat cell design is subject to extensive research at the Risø National Laboratory and presently produced there in a pre-pilot cell production plant. The flat cell is a 3-layer structure consisting of a ~300 µm thick supporting anode layer of NiO-YSZ (Yttria Stabilized Zirconia, YSZ), a ~10 µm thick solid YSZ electrolyte, and a ~24 µm thick LSM-YSZ cathode (Strontium doped Lanthanum Manganite, LSM). The NiO in the anode is reduced to Ni under operating conditions.