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Perovskite compounds containing cation inducing probable disorder is very attractive owing to the fact that positional disorder is often at the origin of the relaxor phenomena. The complex perovskite AA'(BB')O_3 are known to exhibit normal and/or relaxor ferroelectric behaviour. Many investigations have been devoted to the study of relaxation parameters in such materials in order to achieve their use in satisfactory capacitors and actuators [1,2]. However, most of these materials are lead-based ceramics which present a disadvantage due to the toxicity of PbO. So, for environmental and health reasons, manufacture of such materials are more and more constrained to eliminate the lead content from these compounds. In this way, we have investigated new lead free compositions in $BaSc_{1/2}Ta_{1/2}O_3$-$BaTiO_3$ systems. We present here the dielectric study and correlate the results obtained with the structure of these new lead-free materials.
Samples of the (1-x) $BaSc_{1/2}Ta_{1/2}O_3$- x $BaTiO_3$ solid solution were prepared from high purity $BaCO_3$, TiO_2, Sc_2O^3 and Ta_2O_5 powders using solid state methods. All these materials were previously dried at 120°C for 15 h, weighed, mixed for 1 h and calcined at 1200°C for 15 h. After calcinations, powders were mixed for 1h 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 ($5° \leq 2\,\theta \leq 80°$). 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 of the sample was measured under helium atmosphere as a function of both temperature (80–500 K) and frequency (10^2–2.10^5 Hz). For composition close to $BaTiO_3$, three dielectric peaks have been observed and correspond at cubic paraelectric to tetragonal ferroelectric, and then to an orthorhombic ferroelectric and finally to a rhombohedral ferroelectric similar to those of pure BaTiO3. For the relative high values of x (incorporation of Sc^{3+} and Ta^{5+} cations) only one peak occurs with weak frequency dispersion. The tetragonal and cubic symmetry are related to the classical or relaxor ferroelectric behaviour respectively.

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Keywords: lead-free; ferroelectric; XRD

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Combined Refinement of High Resolution Neutron and Synchrotron Data of PLZT. Manuel Hinterstein[a], Roland Schierholz[a], Markus Hölzel[a], Anatoliy Senyshyn[a], Jens Kling[a], Ljubomira Ana Schmitt[a], Hans-Joachim Kleebe[a], Hans Kungl[b], Michael Knapp[c], Hartmut Fuess[a]. [a]*Institute for Materials Science and Geosciences, University of Technology Darmstadt, Germany.* [b]*Institute of Ceramics in Mechanical Engineering, University Karlsruhe, Germany,* [c]*CELLS, Barcelona, Spain.*
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Lead containing oxides with perovskite structure like $PbZr_{1-x}Ti_xO_3$ (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 $Pb_{0.985}La_{0.01}(Zr_{1-x}Ti_x)O_3$ (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".

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

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Probing the Giant Piezoelectric Effect at the Atomic Scale in $PbZn_{1/3}Nb_{2/3}O_3$. Jérôme Rouquette[a], Ali Al-Zein[a,b], Julien Haines[a], Philippe Papet[a], Claire Levelut[b], Hichem Dammak[c], Olivier Mathon[d]. [a]*ICG UMR CNRS 5253, PMOF, Montpellier, France.* [b]*LCVN UMR CNRS 5587, Montpellier.* [c]*SPMS UMR CNRS 5580, ECP, Châtenay-Malabry, France.* [d]*ESRF, Grenoble, France*
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"*Ferroelectric-relaxors*" can be distinguished from "*classic ferroelectrics*" by a diffuse ferroelectric-paraelectric phase transition where ε'_r reaches a broad high peak value with a strong frequency dispersion (the temperature at the maximum T_m increases with frequency). Additionally, the spontaneous polarization P_s is not suddenly lost at T_m, but decays more gradually to zero (at T_{Burns}) with increasing temperature. On cooling from high temperatures, $PbZn_{1/3}Nb_{2/3}O_3$ relaxors (PZN) transform from a paraelectric state to a state with ferroelectric nanodomain fluctuations in a paraelectric matrix and, to a disordered low-temperature polar phase. The existence of polar Nano-Regions in these materials, which can be associated with local lattice distortions with respect to the average structure can be linked to the presence of diffuse scattering in diffraction experiments. From a vibrational viewpoint, a ferroelectric polarization is found to exist based on the recovery of the soft optic mode without a rhomboedral distortion. From a technological viewpoint, PZN exhibits giant electromechanical coupling with a relatively modest applied electric field along the [001] direction. Many studies are related to the competition between short-range and long-range order in these materials, which is supposed to be responsible for the giant piezoelectric response. In this contribution, we present the first local "in-situ" investigation as a function of $\vec{E}\vec{E}$ to really understand what occurs at the atomic scale.
X-Ray Absorption Spectroscopy was performed at the 3 absorption-edges of interest in PZN (i.e., $Pb(L_{III})$, Zn(K), Nb(K)) and collected in the fluorescence mode as a function of the electric field up to 10 kV/cm along the [001] direction on BM29 at the ESRF. Carefull analyses of our EXAFS data linked with an entire dielectric/piezoelectric characterization have clearly enabled us to determine the polarisation process, i.e. between the unpoled and poled states; above a critical value of $\vec{E}\vec{E}$ (the coercitive field here is about 3 kV/cm), one can observe the persistence of the polarization even when the $\vec{E}\vec{E}$ is removed. Additionally, our data permited us to follow the local distortion of the Zn and Nb atoms in their oxygen octahedra cage, giving rise to the piezoelectric effect, to field reaching 10 kV/cm!!!

Keywords: ferroelectric-relaxor; X-ray absorption spectrocopy; electric field

FA2-MS05-P07

Domain Structure in $PbZr_{1-x}Ti_xO_3$. Roland Schierholz[a], Hartmut Fuess[b]. [a]*Institute for Materials Science, Technische Universität Darmstadt, Germany.*
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The *Cm* phase of $PbZr_{1-x}Ti_xO_3$ (PZT) for compositions at the morphotropic phase boundary (MPB) has been proposed based on X-ray diffraction results [1]. Also nanodomains were observed in this compositional region [2]. We observed monoclinic symmetry within single domains of $PbZr_{0.54}Ti_{0.46}O_3$. by Convergent-Electron Beam Diffraction (CBED) [3].
The monoclinic phase allows 24 polarization directions. In PZT the low temperature monoclinic phase is a subgroup of a tetragonal phase at higher temperatures. Due to group-subgroup relations four domains with <uuv> polarization can form within one tetragonal domain. In former 90° micro domain walls domains with <uuv> and <uvu> polarizations meet. This leads to restrictions in lattice parameters in the (110) plane for polarizations that cannot be transformed by a single twin operation.
On the rhombohedral site of the MPB within one rhombohedral micro domain at most three different domains can be formed. In experiments a finer nanodomain contrast is observed with orientations of the nanodomain walls, that differ from those on the tetragonal site. These nanodomain walls can be explained as domain walls of the Cm phase as a subgroup of *R3m* instead of *P4mm*.
An *in situ* study revealed a change from a tetragonal/monoclinic shape of the 110_{pc} reflection in X-ray diffraction to a more rhombohedral appearance with cycling [4]. For a sample cycled *ex situ* wide monoclinic domains and rhombohedral appearing domain configurations exist. Those rhombohedral domains show an inner contrast that is consistent with nanodomains. So we discuss the possibilities for a rearrangement of monoclinic nanodomains formed on the *P4mm* to *Cm* transition under an electrical field.

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Electron Diffuse Scattering in epitaxially grown SrTiO3 thin film, Jérôme Pacaud[a], Frédéric Pailloux[a]. [a]*Laboratoire PhyMat Université de Poitiers, France.*
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The main requirement for many devices is the growth of a high purity and high structural quality thin film. The perovskite structure is extremely sensitive to the deposition condition and particularly to the temperature and the partial pressure of oxygen. Changes in deposition conditions may lead to a large deviation of the dielectric properties of thin films from those of bulk materials. The chemistry of defects is often proposed as an explanation of this deviation.
Another important parameter for thin perovskite films is the geometrical constraint imposed by the substrate. Most of the time, perovskite exhibit excellent epitaxy on each other and the films are tied to the substrate so the in-plane parameters are not free to reach their bulk equilibrium values. For materials as sensitive to phase transition as perovskite this effect and the associated relaxation processes can be extremely important for the fine tuning of the physical properties of the film.
For perfect crystal, diffuse scattering is mostly inelastic due to phonons, plasmons and other processes. Thermal