High Energy X-Ray Diffraction Investigation of Ferroelectric Constitutive Behavior

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Introduction

High energy X-ray diffraction was employed to probe the complex multiaxial constitutive behavior of ferroelectrics. $BaTiO_3$ and several $Pb(Zr,Ti)O_3$ (PZT) ceramics were subjected to cyclic electrical field during diffraction. Using transmission geometry and a two-dimensional detector, lattice strain and texture evolution (domain switching) were measured in multiple sample directions simultaneously. The results offer a unique coupled strain/domain switching dataset on the multiaxial constitutive behavior of ferroelectric ceramics. It is seen, for instance, the lattice strain data are highly anisotropic resulting in large differences between *hkl*-specific strains. In addition, texture analysis suggests that non-180° domain switching is coupled with lattice strain evolution during electrical loading. In one case, the electric field induced an antiferroelectric-to-ferroelectric phase transformation. These investigations confirmed the unique advantages of high energy XRD in quantifying the constitutive behavior of ferroelectrics and yielded rigorous data for mechanics model validation.

Methods and Materials

High energy (~80 keV) X-rays were employed in transmission geometry at beamline 6-ID-D (MU-CAT) of the Advanced Photon Source (APS). $BaTiO_3$ and PZT specimens measured about 1x1.5x10 mm, where the thickness (1 mm) was along the X-ray beam and the electric field was applied parallel to the width (1.5 mm). Using a 0.5x1 mm spot size, the data were collected in macrodiffraction mode by a Mar345 digital image plate detector placed ~1 m from the sample so that complete Debye-Scherrer rings were captured. CeO_2 powder was employed as an internal standard. This experimental geometry allowed the complete chracterization of the multiaxial specimen response perpendicular to the beam. The diffraction data were analyzed by both single peak fitting and the whole-pattern Rietveld method.

Results and Discussion

Fig. 1 exhibits lattice strain evolution in a tetragonal PZT specimen under electric field. The strain data show the expected tensile and compressive behavior along the directions parallel and perpendicular to the electric field, respectively, due to the piezoelectric effect. However, there is significant discrepancy in the strains along the *a* and *c* axes of the lattice suggesting high strain anisotropy. One reason for this is the 90º domain switching above the coercive field (~4 kV). The texture evolution that accompanies this domain switching is shown in Fig. 2.

In another specimen, $(Pb_{0.99}Nb_{0.02}[(Zr_{0.57}Sn_{0.43})_{0.94}Ti_{0.06}]0_3)$ or PZST, the electric field induced a phase transition from an antiferroelectric (recognized by satellite peaks around the 210 peak in Fig. 3) to a ferroelectric where the satellites disappear. It was found that the transformation proceeds with aligning the Pb-cation displacements, which resembles the process of 90° domain switching and 180° reversal in normal ferroelectrics.

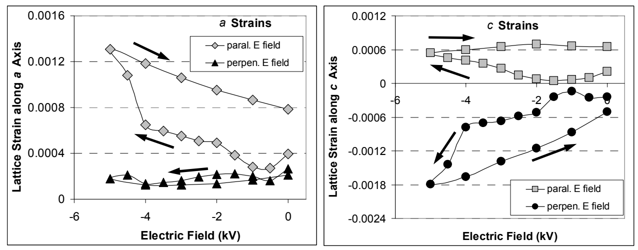

Fig. 1. Lattice strain evolution along and perpendicular to the applied electric field as measured by the changes in the lattice constants of a tetragonal PZT specimen.

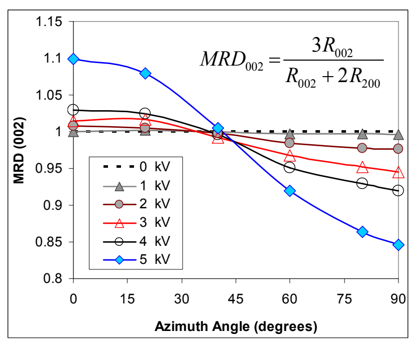

Fig. 2. Texture evolution induced by the electric field parallel to the 0° azimuth. The MRD (multiples of random distribution) value was obtained from the ratio of the integrated peak areas (relative to an unpoled sample) as shown in the inset.

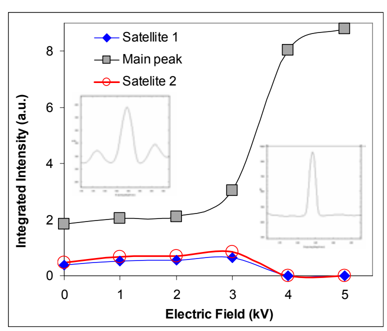

Fig. 3. Intensity changes in the 210 peak of PZST and its satellites as it transforms from an antiferroelectric (at low field values) to a ferroelectric.

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