EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

	Experiment title: Time-Resolved Studies of Electric-Field-Induced Phase Transitions in Ferroelectric Materials	**Experiment number**: MA-2126
Beamline: ID22	**Date of experiment**: from: 12/11/2014 to: 17/11/2014	**Date of report**: 24/02/2015
Shifts: 15	**Local contact(s)**: Yves Watier	*Received at ESRF:*

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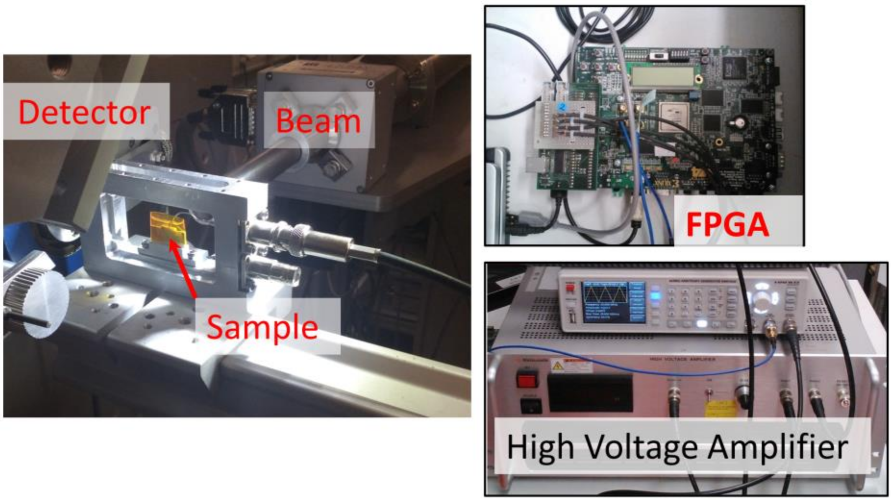
Report:

Brief Summary: The aim of the experiment was to study the kinetics of electric-field-induced phase transitions in the ferroelectric ceramics $Bi(Zn_{0.5}Ti_{0.5})O_3$-$BaTiO_3$ (BZT-BT). We proposed to implement the specially developed stroboscopic-based data acquisition system (realized with the field programmable gate array - FPGA) to record the diffraction intensity with the time resolution down to 100 ns. This technique has been previously successfully used for the in-situ time-resolved X-ray diffraction on single crystals of piezo and ferroelectrics [1,2]. This proposal posed a particular experimental challenge to implement the stroboscopic data aquisition system at the powder diffraction ID22 beamline. **The experiment was successfull: we collected the first time-resolved powder diffraction patterns from ferroelectric ceramics. The results of the experiment provided valuable information about the phase-switching related dielectric response in BZT-BT ferroelectric ceramics.**

Scientific background: Ferroelectric materials are used in variety of applications such as actuators and impact sensors. Their properties strongly depend on the structures, which can be transformed by several factors such as chemical composition, temperature, and electric field.

External electric field can change atomic position, lattice constant, and domain structure of ferroelectrics. The particular interest is attracted to the so-called electric field-induced phase transitions observed in ferroelectric solid solutions with compositions near the morphotropic phase boundaries (MPBs) or polymorphic phase transitions (PPTs). Although the field-induced phase transition of many ferroelectric materials has already been reported, there is a general lack of understanding of the kinetics of such transitions.

Experimental details: The ceramic samples of BZT-BT two different compositions (92BT-BZT and 94BT-BZT) of the 10×2×1 mm^3 size were prepared at North Carolina State University, including synthesis, polishing, and application of electrodes. The samples were be loaded to a specially designed electric field sample stage. The alternating high voltage (+/- 3 kV) of 100 Hz - 2 kHz frequency range was produced using a function generator and high-voltage amplifier and applied to the samples. The resulting electric field was aligned in a vertical direction (perpendicular to the beam). High-energy (E ~ 31 keV) powder diffraction patterns were collected in transmission mode. The detector signals were processed by the stroboscopic FPGA (Field Programmable Gate Array)-based data acquisition system providing adjustable time resolution (the best achievable time resolution is 100 ns).



Results: Figure 1 shows example output of the experiment. Here {111} powder diffraction profile from 94BT-BZT ceramics sample was stroboscopically measured under alternating 50 Hz bipolar electric field. The diffraction intensity as a function of scattering angle and time (0-20 ms) is shown in the form of a colormap in the Fig. 1a. The diffraction profiles, collected within selected time-channels are highlighted in the Fig 1bc. The exact time dependence of applied voltage is shown on the bottom of the Fig 1a.

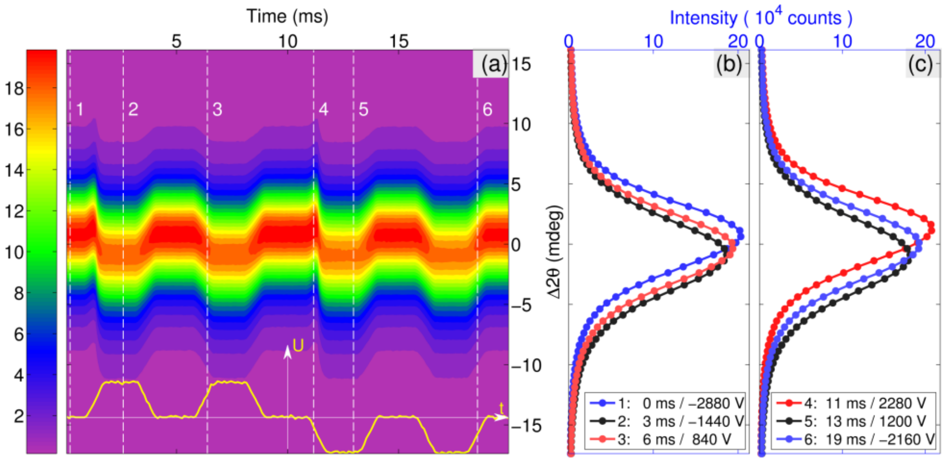


Figure 1. Example output of the experiment: Intensity of {111} Bragg peak profile as a function of time and scattering angle.

The systematic voltage-dependent angular shift of the diffraction profiles describes the intrinsic piezoelectric response - deformation of the lattice parameter, a_{111}, along the direction of the scattering vector. It is used to calculate intrinsic piezoelectric constants. The change of the integrated intensites are analysed using the structure factor formalism (the results are currently

being prepared for the publication). These results demonstrate the feasibility of application of the stroboscopic technique for X-ray diffraction studies.

Figure 2 demostrates the investigation of kinetics of electric field-induced phase transitions 92BT-BZT, previously studied under quasi-static electric field [3]. During the application of electric fields, intensity of the 200_{PC} decreases while intensities of 200_T and 002_T increase. This indicates field-induced phase transition between pseudocubic and tetragonal. The diffraction patterns were fit to extract intensity and calculate phase fraction. The phase fraction (Figure 2 middle plot) shows that field-induced phase transition of BT-BZT depends on frequency of applied fields. The phase fraction as a function of time is fit with Avrami-type equation to extract distribution of time constant for the field-induced phase transition. The time constant implies the kinetics of the field-induced phase transition and also the distribution of local fields.

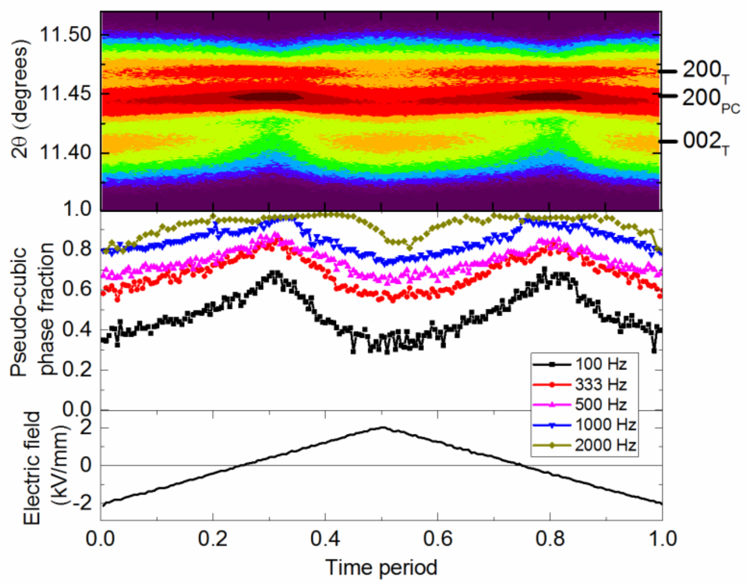


Figure 2. 200 reflections during the application of electric fields with frequency of 100-2000 Hz.

References:

[1] S. Gorfman. Crystallography Reviews, 20(3), 210- 232, (2014).

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