ESRF	**Experiment title:** Grain scale structural and micro-structural evolution within piezoelectric morphotropic Pb(Zr0.52Ti0.48)O3 (PZT) thin films during electric excitation	**Experiment number**: 32-02 770
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Report:

Please notice that this is a preliminary report due to the experimental dates (06/02/15-09/02/15) close to deadline for new proposal submission. The final report will be provided later on.

The aim of this experiment was to probe at the grain scale the structural and microstructural evolution within the piezoelectric morphotropic Pb(Zr0.52Ti0.48)O3 (PZT) thin films during electric excitation.

The measurements were made using the standar setup for with beam Laue microdiffraction with a 300 × 500 nm beam. For in-operando conditions we have used a power supply available from the instrumentation pool (KEPCO). This made possible to control the voltage supply from the SPEC session and automatise the electric cycles.

During the allocated beam time three in situ measurements were done under in-operando conditions. Three functional devices (parallelepiped capacitors of 0.3 mm thick x 5 x 5 mm2 surface) with different Zr-Ti compositional gradients were studied. We have also validated the possibility to reposition the sample using the fluorescence of W markers deposed at the surface of the sample.

Our measurements include :

Sample 1 (gradient free) Hysteresis loop up to +/- 25 V measured at different x,y positions
Sample 2 (standar gradient) Hysteresis loop up to +/- 25 V measured at different x positions
Sample 3 (amplified gradient) Fast cycling between +/- 15 V and +/- 20 V measured at different x positions

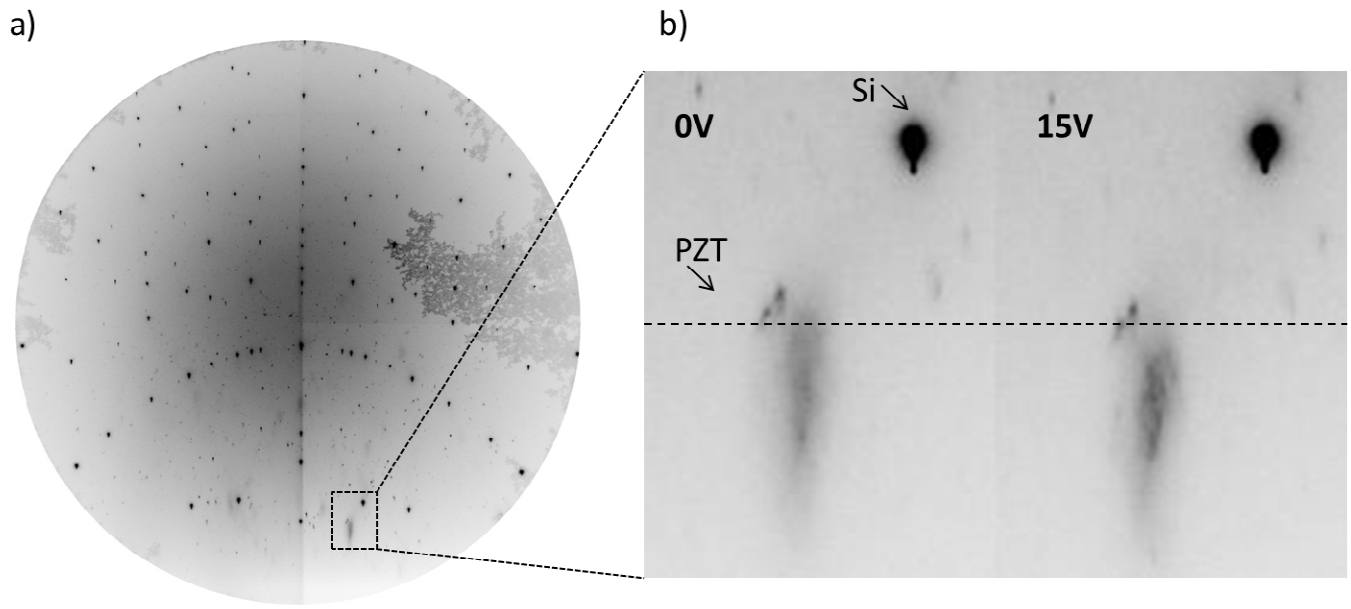

Figure 1. a) Laue pattern showing the spots from the Si substrate, the Pt electrode and the PZT sample. b) zoom on single grain of PZT showing change on the position and rotation of the Laue spot when cycled from 0V to 15V. The horizontal dashed line in is a guideline for the eyes.

Measurements at different sample positions allow to follow the microstructural behaviour of the films grain-by-grain. The analysis of the position and shape of the Laue spots will determine weather the piezoelectric effect is driven by domain switching or phase transition from tetragonal P4mmm to rhombohedral R3m. In figure 1.b) the measure shows the rotation of the PZT grains and probably also grain deformation, this second parameter is to be confirmed.

These first and preliminary results prove that white beam micro-diffraction allows following at the grain scale the structural modifications induced by an electrical excitation in PZT piezoelectric material. This outstanding observation opens the door towards many experiments aiming at better understand the microstructure evolution of such material during their time of life, and thus help understand the mechanism at work before breakdown.

We have noticed that during the week of experiments a variation of the temperature in the experimental hutch (±0.4 deg) was reflected as a fast drift in the beam position over the time. For this type of experiment where the size of the PZT grains (250-300 nm) is similar to the nanometric beam spot, this drift is critical. As consequence, a modification of the data collection strategy was required for Sample 3 in order to keep the measure within the beam stability. Further data analysis will confirm if the data needs to be remeasured under better stability conditions.

During this experimental time, we have also determined a number of improvements that our team will develop for future experiments: (a) design a dedicated sample holder to ensure fixation stability of the sample (b) modify patterning of W reference used to recenter the sample by measuring a fluorescence signal.

The results of this in-operando runs will be the object of a future publication.