	Experiment title: Microstructural evolution of PZT thin films under applied electric field	**Experiment number**: MA 2448
Beamline:	**Date of experiment**: from: 12-09-2014 to: 14-09-2014	**Date of report**:
Shifts:	**Local contact(s)**: Thanh Tra NGUYEN	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

GERGAUD Patrice (*), CEA-LETI DTSA/SCMC Grenoble

NGUYEN Thanh Tra (*), ESRF Grenoble

KOVACOVA Veronika (*), CEA-LETI/DRT/DCOS/SCMS/LCMA Grenoble

Report:

Summary

The aim of the present experiment was to probe a full micro-structural evolution within the piezoelectric morphotropic Pb(Zr0.52Ti0.48)O3 (PZT) thin films during electric excitation. This goal was achieved using in-operando X-ray diffraction on Leti prepared capacitors with PZT active layer. This experiment is providing complementary information to the PZT phase transition under electric field observed in the Leti laboratory [1].

Experimental method

The experiment was carried out on BM02 line during 16 bunch filling mode. The beam was monochromatized at E = 8.33 KeV and the vertical and horizontal beam size was respectively 140µm and 240µm. The 560x960px XPad 2D camera was placed about 20cm from the sample. In order to make a complete 3D RSM considering the PZT (100) fiber texture the 2θ angle of camera and the ω and the chi angles of sample were modified. 6 positions of 2θ : 21°, 35°, 43°, 68°, 82°, 94° and 6 positions of ω : 10.5°, 17.5°, 21.5°, 34°, 41°, 47° corresponding to 2θ/2, so as to have the symmetric mode of acquisition, were performed. Each of 2θ/ω acquisitions were performed under several chi in order to map the out-of-plane crystallographic plans from chi =0° to at least chi =75°. For higher chi we could not obtain any information because of the sample shadowing on the camera. As the 2D camera has some dead rows and columns, we did each acquisition at ν=0° and 2° (in-plane rotation of the detector). In order to calibrate the setup, a LaB6 powder was mesured as well.

The samples were previously prepared using equipments available in Leti. 1µm of PZT was deposited on 200 mm SOI wafer by sol-gel method on 100nm of Pt and recovered by 100nm of Ru. Then square capacitors of 5 x 5 mm were prepared by photolithography. Three types of capacitors were prepared differing by PZT gradient : gradient free, standand gradient and amplified gradient PZT. Samples were then glued to a piece of PCB to report the contacts from the device.Each type of PZT capacitor was made in double. The first was the reference and the second one was poled at 20V and at 200°C, during 30min. It

was observed that this poling process enhances the e31 piezoelectric coefficieng of PZT thin films of nearly 80%.
PZT capacitors were stimulated with different voltages going from -25V to 25V in order to describe the whole hysteresis cycle caracteristic for ferroelectric materials.

Results

We identified the PZT phases peaks and their modification under electric field.
We observed a transition under electric field in capacitor containing gradient free PZT active layer, as shown in figure 1. The tetragonal (400) reflections seem to transit to the rhombohedral (400) reflections. Moreover, other less abundant textures as (211), (301), (222), (321) and (411) seems to undergo the same transition process. It is interesting to add, that we observed an apparition of the (311) and (322) planes. Interpretation of the results is still in process.
In order to have a better global view on the datasets corresponding to one particulat voltage condition, we achieved to merge several datasets on one image. The figure 2 is showing this merged image for gradient free sample under 0V.

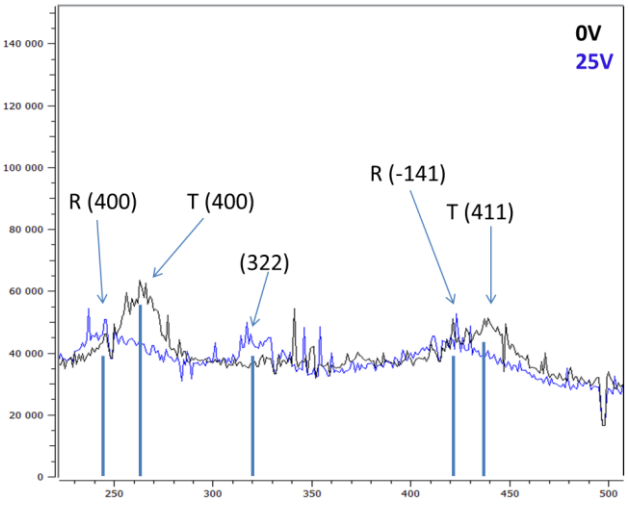

Fig. 1: The superposition of two sections, under 0 and 25V, of image 2ϑ=94° and chi=0° for gradient free sample.

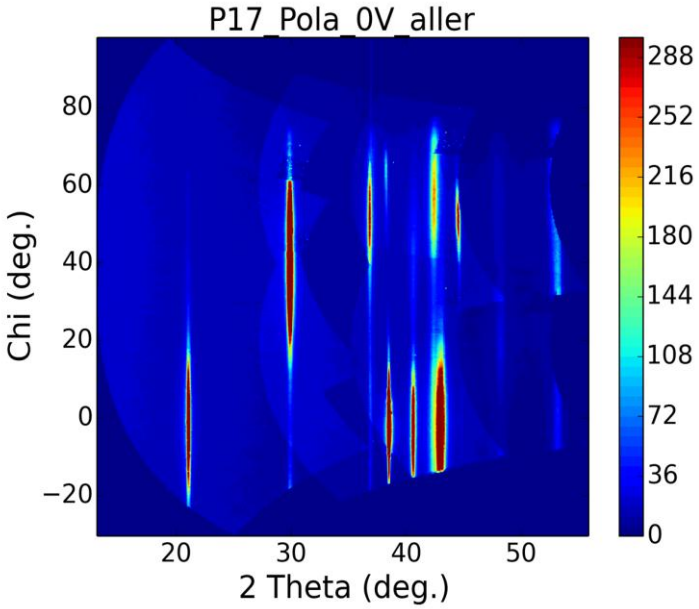

Fig.2: The merge of images corresponding to 2ϑ=21; 35; 43° , v=0;2° and chi= 20; 60° for gradient free sample at 0V.

We are still running data exploitation, so we can compare samples between each other and explain the 200°C poling effect on samples.

Conclusion

The experiment was a success. The data exploitation on first samples seem to confirm the PZT phase transition from tetragonal to rhombohedric phase under electric field and revealed other phenomena which is the apparition of two in-plane crystallographic planes the (322) and the (311). The interpretation as well as other data exploitation is still in progress.

References:

[1] V. Kovacova et all, Phys. Rev. B **90**, 140101(R) (2014)