EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: Diffuse scattering on dielectric material (KTN) under electric field	**Experiment number**: HC 4524
Beamline: ID28	**Date of experiment**: from: 31.10.2021 to: 02.11.2021	**Date of report**: 26.02.2022
Shifts: 12	**Local contact(s)**: Alexei Bosak	*Received at ESRF:*

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Report:

Twelve shifts were assigned to measure diffuse scattering on the perovskite ferroelectrics $KTa_xNb_{1-x}O_3$ (KTN), and on a similar oxide, $KTaO_3$ (KTO) sample with and without an applied electric field. The KTN sample was bought from NTT Advanced Technology Corporation (NTT-AT) to assure the high quality of both the chip and the electrodes. The chip has a size of 4x3x1 mm, with 4 sides optically flat polished and electrodes applied on the 2 sides of 4x3mm surface, made as KTN/Pt/Au.

The data were taken at the DS branch of ID28 beamline at the European Synchrotron Radiation Facility (ESRF, Grenoble - France). Data were acquired at energies: 21 keV for KTO and 17 keV for KTN. The size of KTN chip required the use of the beam in reflection instead of the transmission mode. For KTO, the sample was thin enough to permit the normal mode of measurement in ID28, in transmission. The measurements were particularly challenging because of the use of electric field and temperature. The first was applied through a set-up specifically made in ID28 for this purpose, used for both samples (as example KTN mounted on the set-up in figure 1a), and the second was done with a cryostream.

<u>KTN Result:</u>

The plan of action for this experiment was quite straighforward:

1. Find the temperature transition by x-ray diffraction (XRD);
2. Choose a T above the transition and apply the electric field and check for any change

From the producer, we know that the sample has a transition temperature of ~22-30 °C, passing from a paraelectric cubic phase to a ferroelectrics tetragonal phase. The transition temperature was found and we decided to measure above the transition, the chosen temperature was 315K. At this temperature, collections were

made with and without voltage. However, when a 300V positive and negative voltage were applied to the sample, no difference were found on the upper surface, as shown in figure 1b.

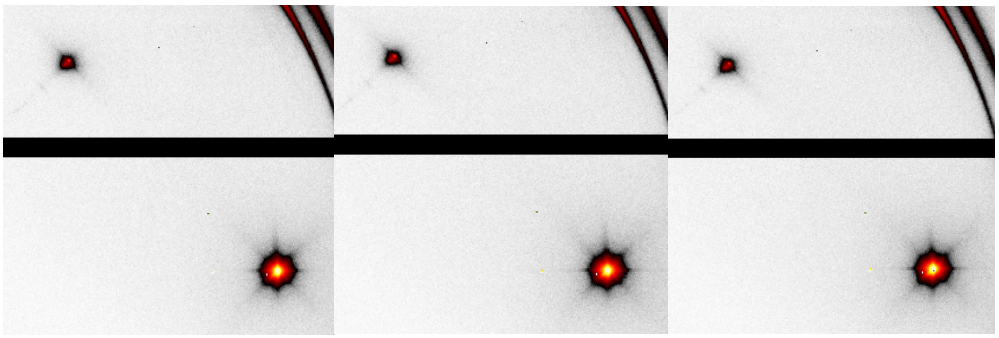
a)  b) 

Figure 1. a)KTN sample mounted on the set-up with two wires connected on the electrode in order to apply an electric field; b)the same frame at 315K with 0V, 300V and -300V voltages applied.

KTO Result:

For KTO our plan of action was different, since there is no phase transition in this material. We decided to create a loop with a step of ±100V going from 0 voltage to a maximum of 500V and let the detector collect frames continuosly. We observed a change in the diffuse scattering with and without the electric field. The difference is noticed through a torough check of every single frame. A difference on the frames with the same geometry orientation with/without voltage was found. In addition, thanks to a shut-down of the main ring, we noticed that the sample becomes a conductor not only for the applied voltage but also under x-rays irradiation. As a result, the sample transfers electric current and warms up. Obviously this two factors could not be deconvolved and we could not understand the effect of the sample heating with respect to the applied electric field. The figure 2 shows the voltage applied and the current in the sample during the measurement in ID28.

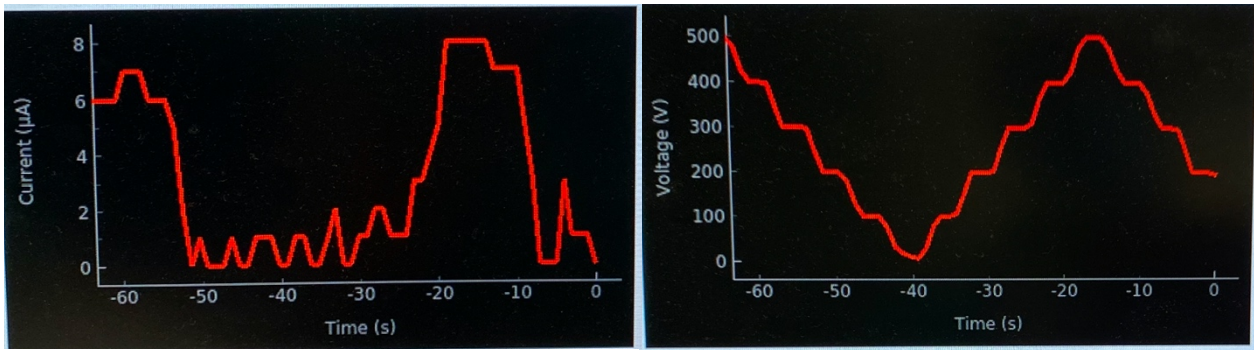


Figure 2. The current in the KTO sample with a maximum applied voltage of 500V, with steps of ±100V.

Conclusion:

The more interesting result obtained in this beamtime is the current obtained in the oxides KTO under x-rays illumination, where the sample becomes a conductor. This result shows that we should rethink the measurement for the KTN sample, since we did not considered the possible effect of a current given by the x-ray irradiation. Hence, the possible effect of x-rays on the sample and the temperature on the sample which could be varying during the measurement. The KTN clearly required more time to be measured and expecially a set-up that can avoid difference in the thermal load on the sample.