Report from the experiment **HC-2141**:
Searching for local structure footprints of antiferroelectric phase transition in $PbZrO_3$ using anomalous diffuse scattering
carried out at BM01 beamline on 30 September 2015 - 03 October 2015

Initial plan

The aim of the experiment was to measure diffuse scattering signal across the antiferroelectric phase transition in perovskite material $PbZrO_3$. The plan was to approach phase transition (PT) temperature ~335ºC from above and collect data at several temperatures and energies. Different energies close to absorption edges of Pb (LIII) and Zr (K) were supposed to allow for an extraction of anomalous diffuse scattering data to be used in direct 3DPDF analysis without the need of solving of the phase problem.

Experimental proceeding

Experiment started with test runs on the prepared samples. We wanted to figure out whether the mounting/gluing will withstand the high temperature. Here we came across first problems – larger platelet sample cracked when crossing PT, most probably due to strain that developed in the relatively large gluing area. For a smaller, needle shaped sample of approximate diameter of 50 µm (one of the samples that were initially supposed to be of the desired shape and size) we realized that the mounting is not stable against hot air blow at high temperatures. We consecutively decided to reglue the samples.

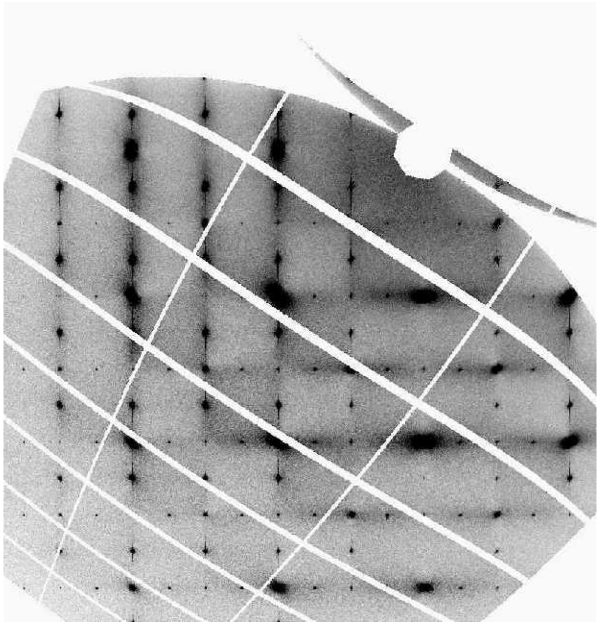
After choosing one of the needle shaped samples we proceeded with some test runs at the energy of 17.6keV (with the threshold at 14keV):

- RT, 360º rotation with $\Delta\varphi$=0.1º
- 180-210ºC tests for measuring in periods and binning
- 220-246ºC ramp up, data collected every 2ºC (360º, $\Delta\varphi$=0.1º)

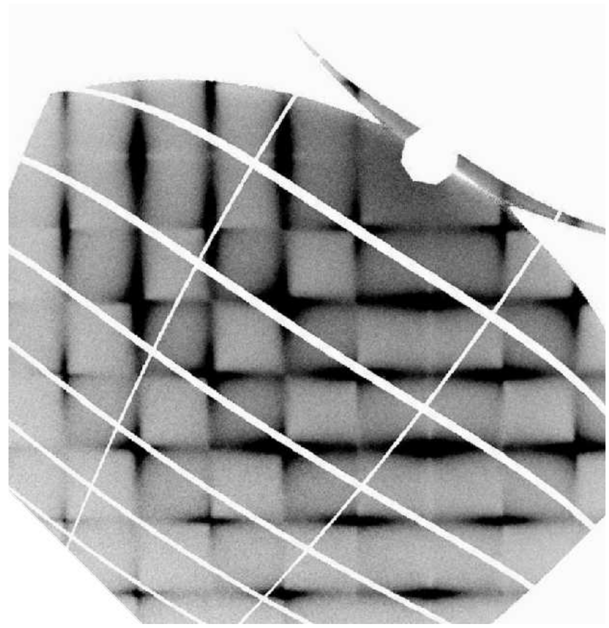
At temperature above the PT we realized that we are not obtaining strong diffuse scattering characteristic for the cubic phase as known from the other experiments. We tested for the beam position, changed vertical slits, tried increasing number of periods acquired at the same position. None of the above could improve the diffuse scattering signal which prompted the decision to abandon needle shaped samples and continue with much larger platelet samples (thickness of ~250 µm).

With the platelet sample chosen it was reasonable to reduce the range of φ scan as signal with the sample plane parallel or close to parallel to the beam was affected by huge absorption. Structure scanning upon heating showed the phase transition to be at somewhat lower temperature (225ºC) than expected. Planned overnight measurement designed to collect data upon slow cooling towards the PT failed due to two main reasons: the script governing the run failed to separate data into two different folders depending on the sample detector distance (unknown reason), the sample mounting eventually did not withstand a constant air blow on a relatively large sample area.

With the limited time left for the experiment we decided to continue with another sample collecting data at just one temperature (230ºC) and two detector distances (146 mm and 176 mm) as well as 4 different incident beam energies (17.6keV, 17.83keV, 12.93keV and 12.7keV). For each distance and energy combination we collected 1200 images (20periods * 0.5s) at $\Delta\varphi$=0.1º.



hk0 plane 224ºC



hk0 plane 232ºC

Conclusions

Due to different reasons we were unable to proceed with the initial plan for this experiment. We overestimated the intensity of the beam and the samples of a proper shape (needle-like) for this experiment were too small. For big platelet samples we experienced problems of sample stability under the heat flow as well as uneven temperature distribution. While we collected data suitable for anomalous diffuse scattering analysis at one temperature just above the phase transition, data processing needs some non-standard procedures taking care of anisotropic absorption which we still need to develop. A single piece of information that might prove the most useful is that coming from the pictures above – there is a clear evidence of scattering coming from antiphase boundaries in the low-temperature phase.