EUROPEAN SYNCHROTRON RADIATION FACILITY

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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

	Experiment title: Non-ambient lattice dynamics in PbZrO3: flexoelectricity-diven interplay between competing instabilities	Experiment number: HC 1113
Beamline: ID28	**Date of experiment**: from: 12/02/2014 to: 18/02/2014	**Date of report**: 01/03/2014
Shifts: 18	**Local contact(s)**: Roman Burkovsky	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The sample was PbZrO3 single crystal with dimensions approximately 100x100x25 microns. It was put in the high-temperature DAC with neon filling. The DAC was put into a specially built chamber which allows to heat the whole cell up to 700 K. We performed the measurements of the lattice dynamics by ID28 spectrometer and in-situ measurements of the diffraction by the Pilatus 300 detector from the instrument pool of ESRF. The pressure was kept close to P=13kbar and we made the measurements at several temperatures above and below the phase transition.

We start with orthorhombic phase where only superstructures of the form (H+-0.25, 0, L+-0.25) exist. There are no domains in other planes. In this phase there is a flat dispersion of the lowest energy mode along the sigma directions that do not have superstructures. The spectra for directions with superstructures are different. The spectra in the plots correspond to the q-points of the form **q**=(a,+-a,0) with a=0.1, 0.2, 0.3, 0.4. On heating above the phase transition temperature the superstructures disappear and the butterfly-shaped diffuse scattering emerges. The wings of the butterfly appear only in those sigma directions that previously had superstructures. This means that the symmetry of the phase is not really cubic. The diffuse scattering is not centered at Bragg positions but rather around q=0.2. This fits well with the absence of the near-transition dielectric anomaly in PbZrO3 at high pressures. The IXS spectra demonstrate an excitation with far not strictly resonant character and apparently nearly-flat dispersion. On heating further the diffuse scattering becomes weaker and becomes centered at Bragg positions. Apparently the cubic symmetry is restored at high temperatures.

Apparently we see that at high pressure the paraelectric phase is not really cubic as was expected before, but of symmetry lower than cubic. This is clearly indicated by the absence of cubic symmetry in diffuse scattering distributions. We suggest that the careful diffraction characterization is needed to fully assess the origin of observed phenomena.

From the lattice-dynamical point of view the phases above and below the phase transition at T~465K differ mostly in the damping of excitations. In the low-temperature phase the mode is flat and resonant. The diffuse scattering manifests itself in the form of sharp quasielastic line in corresponding IXS spectra. In the high-temperature phase the frequency of the excitation, if fitted by a DHO model demonstrates about the same dispersion as in the low-temperature phase. However the mode here is not resonant but rather overdamped tunneling mode. In contrast with ambient pressure measurements, HP IXS spectra nor HP DS distributions do not demonstrate any critical growth of the zone center diffuse intensity on approaching the phase transition from above.