EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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: **Local Lattice Distortions in IV-VI compound Topological Crystalline Insulators**	**Experiment number**: HC2636
Beamline: ID11	**Date of experiment**: from: 29.11.2016 to: 5.12.2016	**Date of report**: 28.2.2017
Shifts: 18	**Local contact(s)**: Vadim Dyadkin	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Oliver Rader, *Helmholtz Zentrum Berlin, Germany*

Gunther Springholz, *J. Kepler University Linz, Austria*

Guenther Bauer, *J. Kepler University Linz, Austria*

*Václav Holý, *Charles University, Prague, Czech Republic*

*Milan Dopita, *Charles University, Prague, Czech Republic*

*Petr Mikulík, *Masaryk University, Brno, Czech Republic*

Report:

The aim of this proposal is to investigate local lattice distortions in epitaxial layers of the topological crystalline insulators (TCIs) SnTe, $Pb_{1-x}Sn_xTe$ and $Pb_{1-x}Sn_xSe$ by the measurement and analysis of the temperature dependence of diffracted Bragg peaks in order to assess the impact of structural perturbations on the topological properties of these materials.

Recently, the existence of *local* electric dipoles in SnTe was reported well above the ferroelectric-paraelectric phase transition [1]. Similar observations were also reported for PbTe [2], for which an order-disorder transition was proposed to exist at 250K, based on the analysis of pair-distribution functions (PDFs) of *polycrystalline* PbTe samples and the temperature dependence of the first PDF peak. This was interpreted as evidence for a displacement of the atoms from their lattice positions by ~0.24 Å along the ⟨100⟩ directions. Our calculations based on kinematical diffraction theory show that such small atom displacements affect the structure factor and hence the integrated intensities of the diffractions with large hkl's.

In order to detect the mentioned atomic displacements, we have measured x-ray diffraction intensities in reciprocal lattice points *hhh* starting from $h = 2$ up to $h = 14$ using a high photon energy. We have investigated four samples with 1 μm thick layers of $Pb_{1-x}Sn_xTe$ with different concentrations x ranging from 0.0 to 0.6, on BaF_2 (111) substrate. The experimental conditions were the following. We have used fixed energy of 69 keV (0.17833 Å) in order to detect as many diffraction spots as possible from the layer and from the substrate. Diffractometer was set-up to allow low and high angles of diffraction from planar samples and azimuthal rotation of the sample. As a detector, we have used FReLoN camera 2k (2048×2048 pixels) with pixel size of 47.5 μm, thus the field of view being slightly less than 10×10 mm. It was mounted on a vertical arm behind the sample which allowed linear scanning of the detector plane from low to high positions (angular motor for two-theta was not available).

Different types of scans were performed for substrate and layer diffraction peaks, mainly omega-scans and azimuthal scans. Field of view of the FReLoN camera allowed us to measure symmetric diffractions from 222 to 14 14 14. Additionally to the integrated intensities of diffraction maxima we measured also three-dimensional intensity maps of diffuse scattering between the diffraction maxima; these measurements allow us to detect possible correlatios of the occupations of the Pb/Sn lattice places. The tree-dimnesional reciprocal space maps have been measured by performing omega-scans through diffraction maxima and large azimuthal scans over a 120deg range, so that large three-dimensional regions of reciprocal space were scanned. All measurements were carried out at 10 temperatures between 100K and 300K, the samples were cooled by a nitrogen cryostream.

During the beamtime we obtained a huge amount of experimental data, the analysis of which is not finished yet. In this report we present the following examples of measured data. In Fig 1 we show the intensity distribution [panel (a)] and the maximum intensities [panel (b)] around the 222 maxima of the $Pb_{1-x}Sn_xTe$ layer (x=0.4, broad maximum) and the BaF_2 substrate (sharp peak). The image in panel (a) was obtained by integration of about 150 detector frames obtained during an eta scan (incidence angle scan) at 100K. The curve in panel (b) was obtained by taking the maximum intensities from individual detector frames at various icidence angles eta. Figure 2 shows the reciprocal-space image and the diffraction curve of the same sample in diffraction 888.

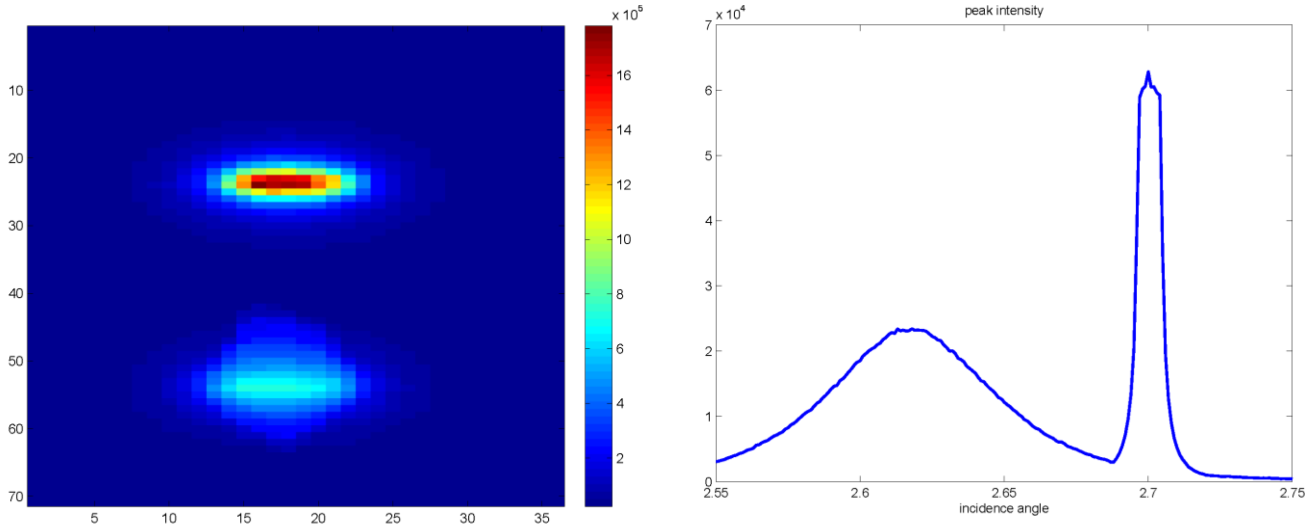

Fig. 1 (left) Three-dimensional intensity distribution around the 222 maxima of substrate (sharp peak) and layer (diffuse peak) integrated over many incidence angles, the measurement was taken at 100K. (right) The maximum intensites in the detector frames at various incdence angles.

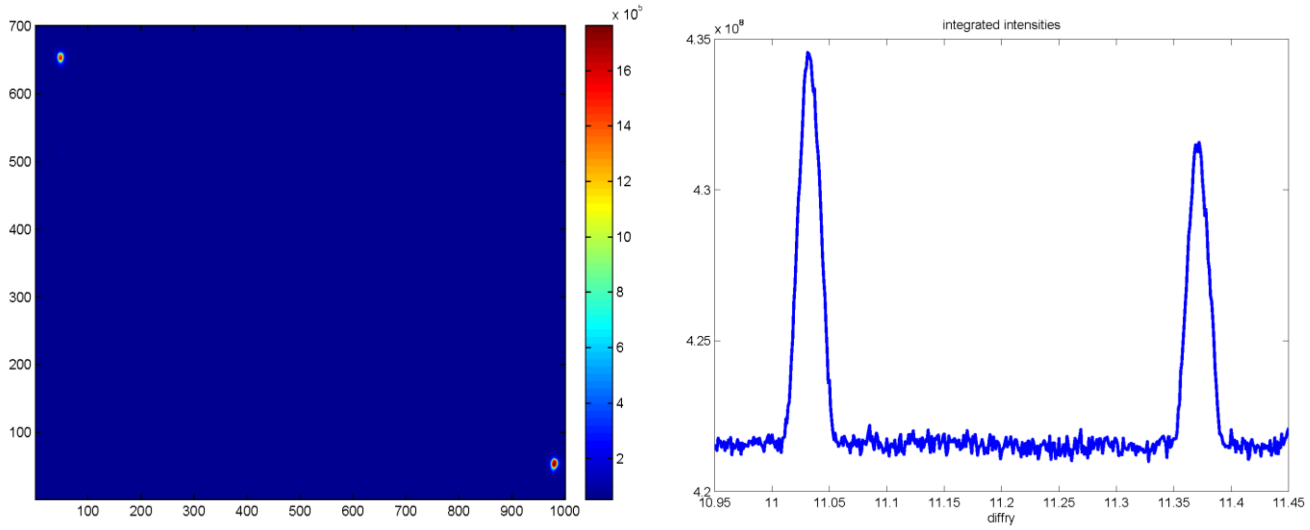

Fig 2. The same situation as in Fig.1, diffraction 888.

From the integrated intensities we determine the magnitudes of the layer structure factors and the mutual displacements of the Pb/Sn and Te sublattices as a function of temperature.

[1] K. R. Knox et al., Phys. Rev. B **89**, 014102 (2014); K. V. Mitrofanov et al., ibid B **90**, 134101 (2014).

[2] E. S. Bozin et al., Science **330**, 1660 (2010).