EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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.

ESRF	**Experiment title:** Single crystal diffuse X-ray scattering methods	**Experiment number**: 86599
Beamline: BM01	**Date of experiment**: from: 07.05.2021 to:10.05.2022 from: 08.06.2021 to:09.06.2022	**Date of report**: 06.2022
Shifts: 9	**Local contact(s)**: Dr. Dmitry Chernyshov	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):
*Dr. Thomas Weber, Department of Materials Science, ETH Zurich
*Joohee Bang, Department of Materials Science, ETH Zurich

Report:

Ferroelectric oxide thin films were first measured at a conventional single crystal diffraction beamline BM01, which showed promising results. The measurement plan was that we conduct grazing-incident step-scan experiments in reflection mode using the pixel area detector PILATUS 2M installed at SNBL and vary the incident angle of the beam to optimize the surface sensitivity of the beam and the coverage of the reciprocal spatial volume. The plan was successfully executed and we observed diffuse rings around crystal truncation rods, indicating isotropic arrangement of in-plane domain structures arising from the strain effect of cubic strontium titanate substrate (right panel of Figure 1). In addition to these diffuse rings, we found periodic patterns of sharp candle-shaped figures in every half h, k, and l directions, potentially arising from an antiferroic order (middle panel of Figure 1). These patterns were not previsouly reported or studied and will thus help us gain deeper insights on structure-property correlations of the films.

Nevertheless, the measurements were met with limitations. In particular, the relatively large incident angle of the beam (2 – 3°) created a shadow of the crystal and prevented us from clear observation of in-plane scatterings. The collected 0^{th} layer evidently shows that it lacks intensities (left panel of Figure 1). This was a major problem, because the 0^{th} layer is the projection of the structure, from which we can learn about both the in-plane and out-of plane polarization structures of the sample. The measurements also suffered from the large footprint of the beam, which resulted in bad resolution at higher angles. Hence, the acquired data will be complemented with high energy experiments to minimize shadow of the crystal as well as allow for small scattering angle and reduce the smearing effect.

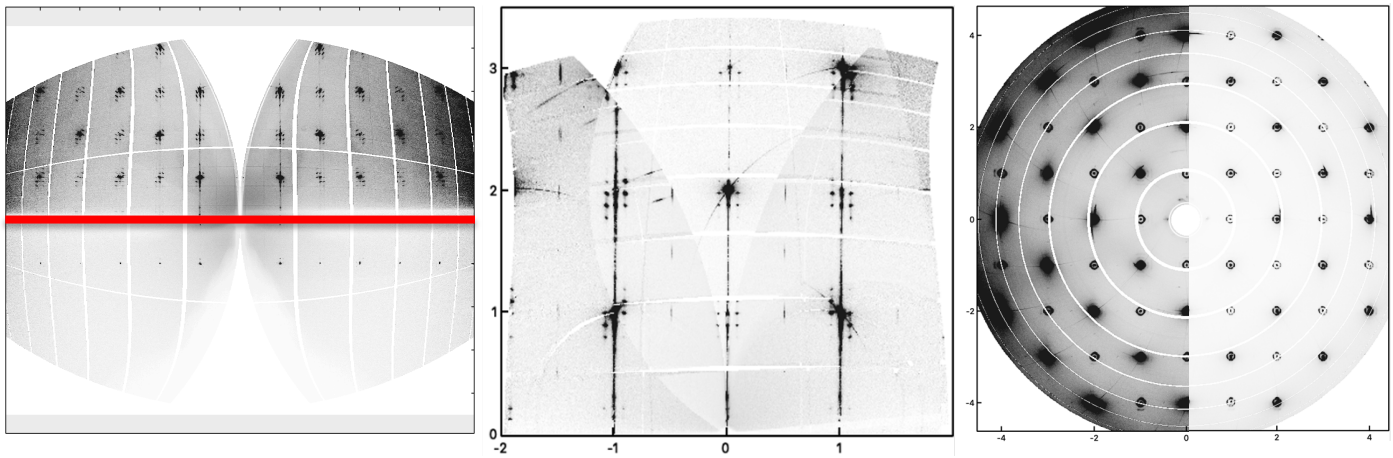

Figure 1. Reconstructed reciprocal space layers of 0kl, h0l, and hk2 collected at 19 keV, 12keV, and 16 keV respectively from left to right at BM01. The red line on 0kl layer indicates the 0^{th} layer of hk-plane and the hk2 layer shows two different color contrast. The h0l layer is a merged reconstruction of omega and phi scan.

We would like to note that the submission of this experimental report was delayed due to challenges met in the analysis of the data. The lack of proper software for analyzing the three-dimensional diffuse scattering data of thin film structures left us with the only option to write our own software. We will further update the report in the case of publication. Lastly, we would like to thank the SNBL staff, especially Dr. Dmitry Chernyshov, who helped our experiment throughout the beamtime and supported us to explore experimental possibilities. We also appreciate the access to the SNBL toolbox software, which was a great help in preprocessing the data for the analysis.