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

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:** Optimising electrical poling processes for single crystal piezoelectrics	**Experiment number**: MA5625
Beamline: ID15A	**Date of experiment**: from: 10th May 2023 to: 13th May 2023	**Date of report**: 09-10-23
Shifts: 9	**Local contact(s)**: Gavin Vaughan	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

John Daniels[1*]
Luke Giles[1*]
Stefano Chechia[2*]

1. School of Materials Science and Engineering, UNSW Sydney, Australia 2052
2. European Synchrotron, Grenoble, France

Report:

Diffuse scattering data was collected on a series of relaxor ferroelectric single crystal samples both $Pb(Mg_{1/3}Nb_{2/3})O_3–PbTiO_3$ (PMN-PT) and $Pb(In_{0.5}Nb_{0.5})O_3-Pb(Mg_{1/3}Nb_{2/3})O_3-PbTiO_3$ (PIN-PMN-PT). Crystal cut orientations and electrical poling history/*in situ* field application were chosen to investigate two pemomena, changes in nanoscale structure upon field-induced phase transformation along each of the three possible transition pathways (monoclinic a (M_a), monoclinic b (M_b) and monoclinic c (M_c)) and application of alternating current poling (AC poling) cycles. In each case the diffuse scattering data are analysed by resampling scattering intensity into 3D reciprocal space, and identifying the direction and distribution of diffuse scattering streaks in the vicinity of Bragg reflections. These streaks are indicative of the nano domain structure in the relaxor ferroelectric single crystals.

Previously, this method has been used to look into the rhombohedral (R) to orthorhombic (O) transition (M_b pathway) [1,2]. Here data from all three possible pathways have been collected which are matched to macroscopic property data from the *in situ* electric field application (see Figure 1). From these experiments diffuse streaking was observed in the (110)-type family of directions around the observed Bragg reflections. Where dissapearance of the diffuse streaking from samples undergoing the R-O (M_b) transition indicate a change from polydomain initial state to an essentially monodomain final state, the changes in streak intensity observed from the other transition pathways as well as changes in intensity observed stepwise along the transition will allow us to extract information about the way in which each of these transitions proceed.

Similarly, macroscopic property data and diffuse scattering from (001)-cut crystals through AC poling cycles have been collected. These AC poling methods have previously been shown to promote long-range ordering of ferroelectric domains and correspondingly improve the measured piezoelectric coefficient (d_{33}) up to around 10 cycles, however the structural changes on the nanoscale occuring throughout this transion are not known. The diffuse scattering collected collected through this experiment will allow us to understand the changes occuring on this shorter length scale throughout this process and therefore move closer to understanding the underlying reasons why the piezoelectric performance of the system is improved.

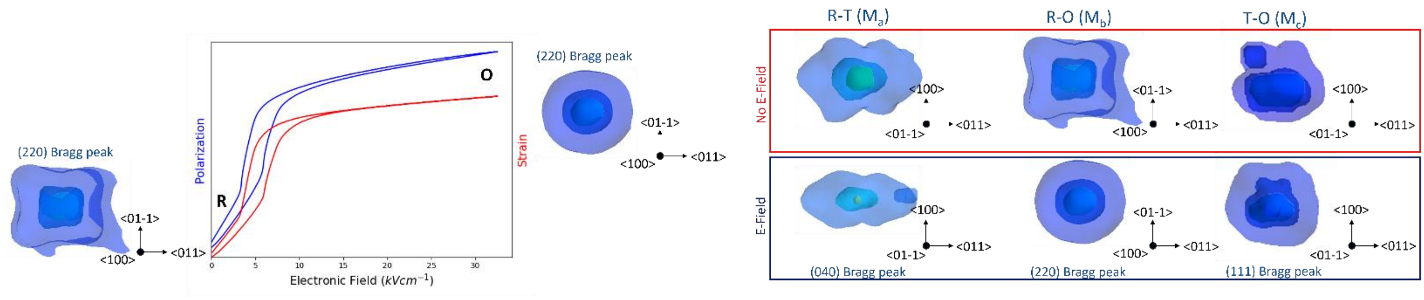

Figure 1. Left-hand side: isosurface plots of the diffuse scattering around the 220 bragg reflection and corresponding macroscopic property data from a sample that undergoes an R-O (M_b) phase transition. Right-hand side: isosurface plots of the diffuse scattering around selected Bragg reflections from samples that undergoe each of the M_a, M_b, and M_c transition pathways shown at their start and end states throughout the transition.

[1] Finkel, P., Cain, M.G., Mion, T., Staruch, M., Kolacz, J., Mantri, S., Newkirk, C., Kavetsky, K., Thornton, J., Xia, J., Currie, M., Hase, T., Moser, A., Thompson, P., Lucas, C.A., Fitch, A., Cairney, J.M., Moss, S.D., Nisbet, A.G.A., Daniels, J.E., Lofland, S.E., 2022. Adv. Mater. 34, 2106827.
[2] Finkel, P., Staruch, M., Amin, A., Ahart, M., Lofland, S.E., 2015. Sci. Rep. 5, 13770.