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:** Domain and polarisation switching in hybrid improper ferroelectrics	**Experiment number**: HC3530
Beamline:	**Date of experiment**: from: 04/04/18 to: 08/04/18	**Date of report**:
Shifts:	**Local contact(s)**: Stefano Checchia	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Stefano Checchia*

ESRF

Mark Senn*

Gabriel Clarke*

Warwick University, UK

John Daniels*

University of New South Wales

Report:

Proposal Summary:

The aim of this work is to study the mechanism of polarisation switching in polycrystalline hybrid improper ferroelectrics by time-resolved diffraction under electric field. Ruddlesden-Popper (RP) oxides are prototypical hybrid improper ferroelectrics, a promising class of materials for room-temperature cross-coupled multiferroicity, in which electrical polarisation couples to non-polar structural distortions. The mechanism of ferroelectric switching in RP hybrid improper ferroelectrics has been described until now mostly through theoretical analysis; in this experiment, time-resolved diffraction would give insights into microstructure, symmetry, and dynamic response of two Ruddlesden-Popper oxides $(Ca,Sr)_3Ti_2O_7$ at various stages of the switching process

Results obtained during beamtime:

Nine sintered pellets were measured as a function of applied field over ten cycles. In total twenty-six data sets were collected on Ruddlesden-Popper oxides $Ca_{2.15}Sr_{0.85}Ti_2O_7$. Data was collected every 5000, 3750, 2500 and 1250 ms using the Pilatus detector. Each run consisted of between 100-300 frames. These frames were radial intergrated to extract powder diffraction patterns.

Analysis of the data has involved performing batch Pawley refinements of the field dependent data to extract the variation of the superstructure peak intensities (that are related to the improper ferroelectric mechanism) and to ascertain if there is any strain coupling (lattice parameter variation). Modulations of intensities are observed at the same frequency of the applied electric field (See Figure 1). These have been Fourier transformed to extract only the component that oscillates at the same frequency of the applied electric field. To validate the significance of the observed modulations, results have been compared across a series of different pellets and applied frequencies to produce a list of robust

intensities. The modelling of the modulation of these intensities will allow us to extract information on the switching pathway in these hybrid improper ferroelectric materials.

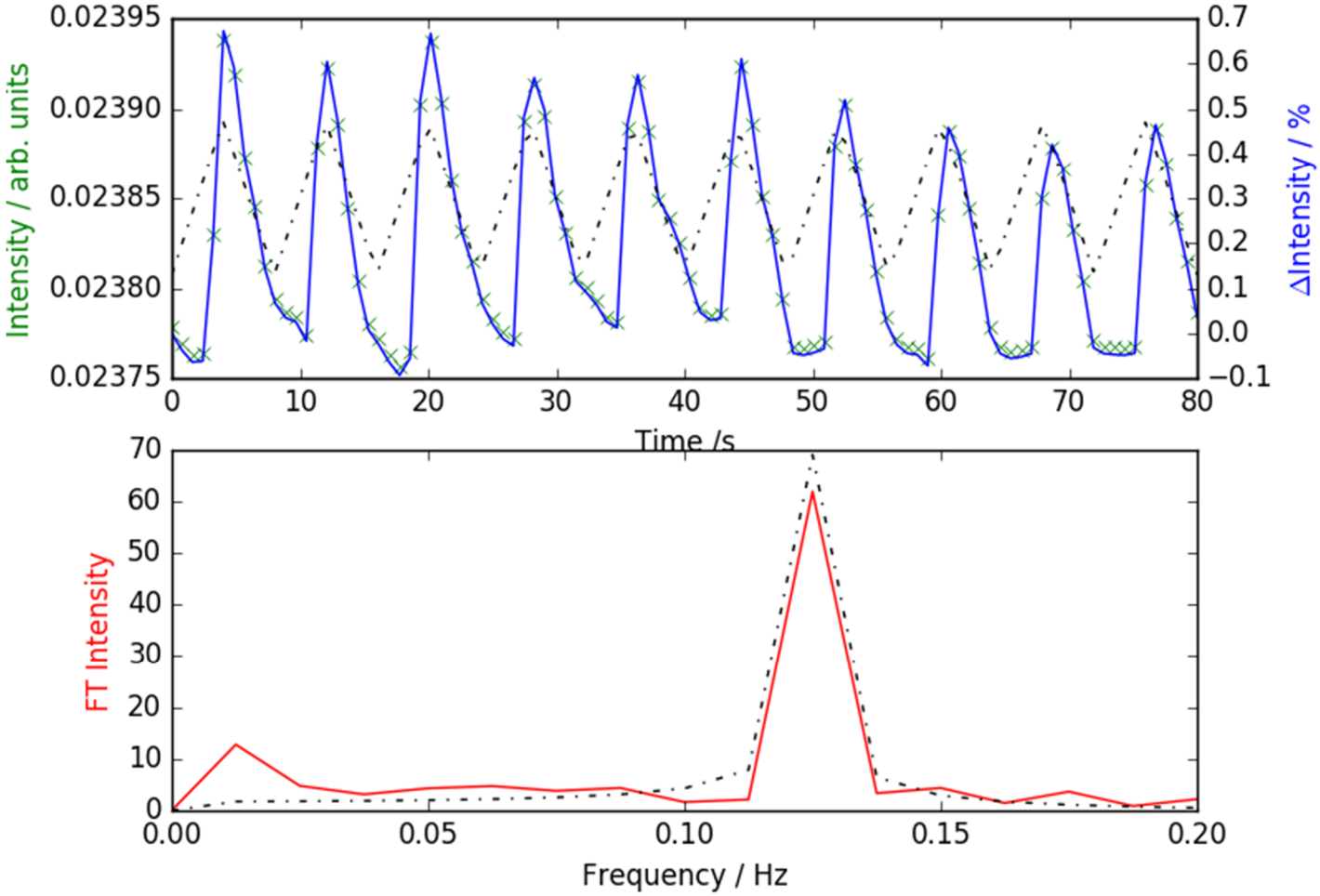

Figure 1. Time-resolved data from in-situ experiment showing variation in intensity of (1 2 4) reflection; Top: Intensity change expressed in terms of intensity extracted from TOPAS Academic (green crosses) and percentage change (blue line); Bottom: Fourier transform of intensity shift. Black dotted lines in both plots represent the applied voltage.