

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://www.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 from 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.



	Experiment title: Imaging the influence of dislocations on domain wall motion in KNbO ₃ using stroboscopic dark-field X-ray microscopy	Experiment number: MA4746
Beamline:	Date of experiment: from: 22/6-2021 to: 28/6-2021	Date of report: 4/3-2023
Shifts: 18	Local contact(s): Can Yildirim, Carsten Detlefs, Raquel Rodriguez lamas	<i>Received at ESRF:</i>

Names and affiliations of applicants (* indicates experimentalists):
Dr. Theodor Secanell Holstad (Technical University of Denmark)
Dr. Kristoffer Haldrup (Technical University of Denmark)
Prof. Martin meedom Nielsen (Technical University of Denmark)
Prof. Henning friis Poulsen (Technical University of Denmark)
Dr. Hugh Simons (Technical University of Denmark)

Report:

The aim of this experiment was to use dark-field X-ray microscopy to observe how the microstructure in a ferroelectric KNbO₃ single crystal changes when an alternating voltage is applied in a stroboscopic fashion. To this end mosaicity scans were performed while triangle pulses at 300 Hz were applied. Strain scans were attempted, but drift in the motors positioning the lens made this approach challenging. The experimental setup is sketched and outlined in **Figure 1**. An optical chopper with a 10% duty cycle was synchronized to the AC voltage of a signal generator. The AC voltage output is sent to a x20 amplifier and then applied to the sample. The chopper time delay (phase) is then scanned across the triangle pulses.

Scan setup

Fixed:
Voltage
Frequency
Image acquisition time
Scanning:
Phase

Wiring diagram

1. Signal generator sends:
Trigger -> Chopper control
Output -> Amplifier
2. Chopper synchronizes the trigger and the chopper wheel **at the disired Phase**
3. Amplifier multiplies the output X20 and forwards it to the sample

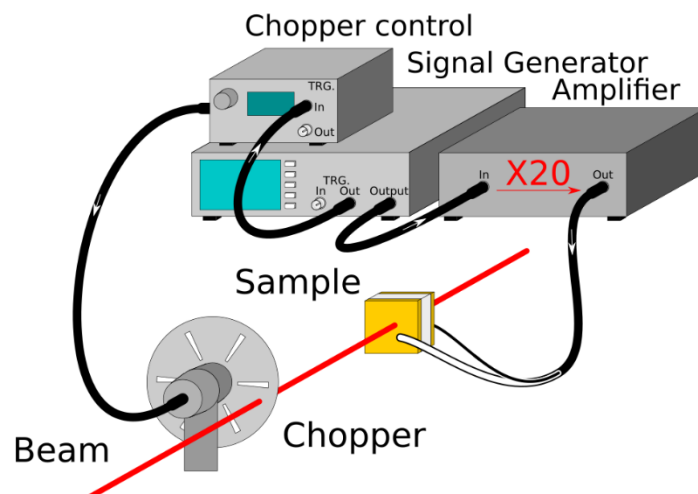


Figure 1

In the mosaicity scan the sample was rotated around two axes orthogonal to the Q-vector, ω and χ , where the axis of rotation for ω is also orthogonal to the beam. The KNbO_3 sample was poled along a (0,0,1) pseudocubic axis, resulting in four possible domain orientations, all of which were observed. Only one of the four pseudocubic (1,1,0) reflections were observed in any one mosaicity scan. The summed intensity is shown in **Figure 2a**. There is little variation in this while the voltage is cycled, suggesting that no non-180° switching occurs.

The center of mass (COM) in ω and χ is shown in **Figure 2b**. The COM varies as the electric field is cycled, both due to the piezoresponse but also due to switching of neighbouring domains. The experiment showed that this response is qualitatively different at 30 and 50 V, and there is significant variation in behaviour across the sample, but only at 50 V. At 30 V the behaviour is to a large extent homogeneous. **Figure 2d** shows how the average COM behaves during the voltage cycle. **Figure 2c** shows how the COM (center) deviates from the mean COM value of each pixel. There is a qualitatively different behaviour in the red and green parts of the figure. The selected mosaicity scan is from the rising edge in voltage, and the red part of the figure has switched, while the green part has yet to switch. This demonstrates the dynamical and inhomogeneous switching dynamics of an otherwise (nearly) defect-free single crystal.

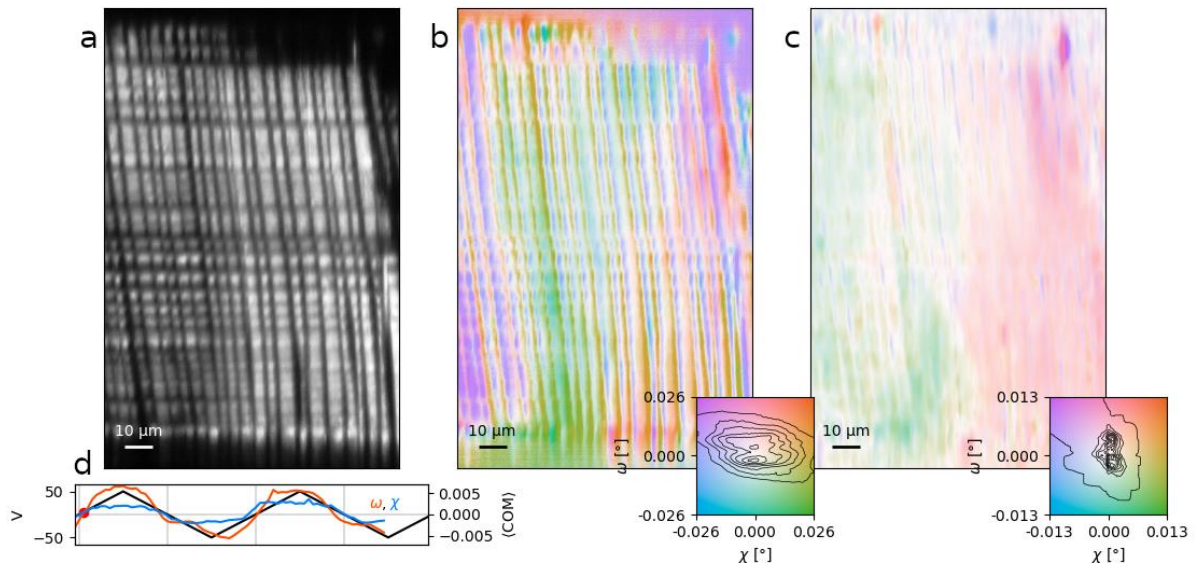


Figure 2