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:** Resolving the microscopic nature of electronic phase separation in the CMR manganites via 3DXRD	**Experiment number**: HC 5185
Beamline: ID11	**Date of experiment**: from: 04/05/23 to: 09/05/23	**Date of report**: 18/09/23
Shifts: 12	**Local contact(s)**: Dr Jon Wright	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Mark Senn, University of Warwick

Evie Ladbrook, University of Warwick

Matt Edwards, University of Warwick

Arkadiy Simonov, ETH Zurich

Summary of Proposal:

Colossal magnetoresistance (CMR) in the manganite perovskites is understood to arise from intrinsic electronic phase separation (EPS) occurring because of the competition between ferromagnetic (FM) conducting and antiferromagnetic (AFM) orbitally ordered (OO) insulating phases, at specific doping levels. However, the very nature of the EPS state has frustrated any attempts over the past 3 decades to accurately characterise the microscopic structures of its constituents. Here we propose a diffraction resolved tomography experiment with nano-focused beam on ID11 to tackle this long-standing problem. The results will allow for accurate microscopic structural models of the competing states to be ascertained
for the first time and will also provide a 3-dimensional map of strain fields within the electronically separated phases on a 0.1-100 μm length scale. Our insights will lead to a broader understanding of how these functional properties are linked with EPS phenomena so that these can be systematically tuned and enhanced.

Summary of Data collected:

A significant number of 3DXRD scans were collected on 5 different crystallites of LPCMO at ~10 K intervals between 100 and 200K, and at room temperature. Crystalites were in the size range 15 – 30 microns. Initial variable temperature scans were collect using the "half" scan approach in which a forward/backwards 180 degree scans (with 0.2 degree steps) was collected followed by successive rastering across the sample in 200 nm steps. Since the aims of the experiment hinged on being able to reconstruct the domain and phases coexisting structure on this ~200 nm length scale, establishing the resolution of the tomographic reconstructions under the in situ conditions (with cryostream operational) was a key part of the experiment. Early on it was noticed that there was a significant systematic shift between forward and back scans, on the length scale of ~0.5 micron, which ultimately results in a significant blurring of the reconstructions. A significant amount of time during the experiment was given over to reducing these effects, including changing the angle of the cryostream, building a shield to deflect the crystostream gas away from the diffractometer and tyring to optimise the stiffness of the sample older. Ultimately from the data quality it seemed like none of these adjustments the experimental setup yielded the desired increase in stability. Devlopements are currenlty ongoing with the sample enviroment team at the ESRF.

An alternative data collection strategy was finaly employed in the later half of the experiments which involved a continuous omega rotation and rastering of the sample, which seems promising with respect to evening out the effect of the uneven thermal expansion of the sample holder that possibly contributes to the loss of experimental data resolutions.

Summary of data analysis progress and results so far

Reconstructions of all data sets revealed that a single grain / crystallite had been studied in all cases but with multiple domains of the Pbnm perovskite structure always present. In principle 6 domains are possible by symmetry (with respect to the Pm-3m aristotype). Selecting sub-classes of superstructure reflections only present in certain Pbnm domains, allows the reconstruction of the domain structure (Fig. 1). At low temperatures (at and below 160 K), additional reflections due to the orbital order appear that in principle allow for a total of 12 domain state. Again, in most instance enough of these reflections are observed experimentally for the selective reconstruction of the domain structure. Significantly, from this it is found that, a given Pbnm domain only ever host one possible domain with respect to orbital ordering. This implies the coherence length of the orbital order is greater than that of the typical length scale of Pbnm domains (observed to be ~1-10 microns in the current experiment). Provisional results are shown below for one crystal at one temperature. More work is needed to develop data analysis routines to correct for sample drift during the experiment. This will allow for greater spatial resolution in the reconstruction allowing the subtle phase coexistence structure in associated with the orbital ordering and metallic phase to be probed. Data analysis is ongoing.

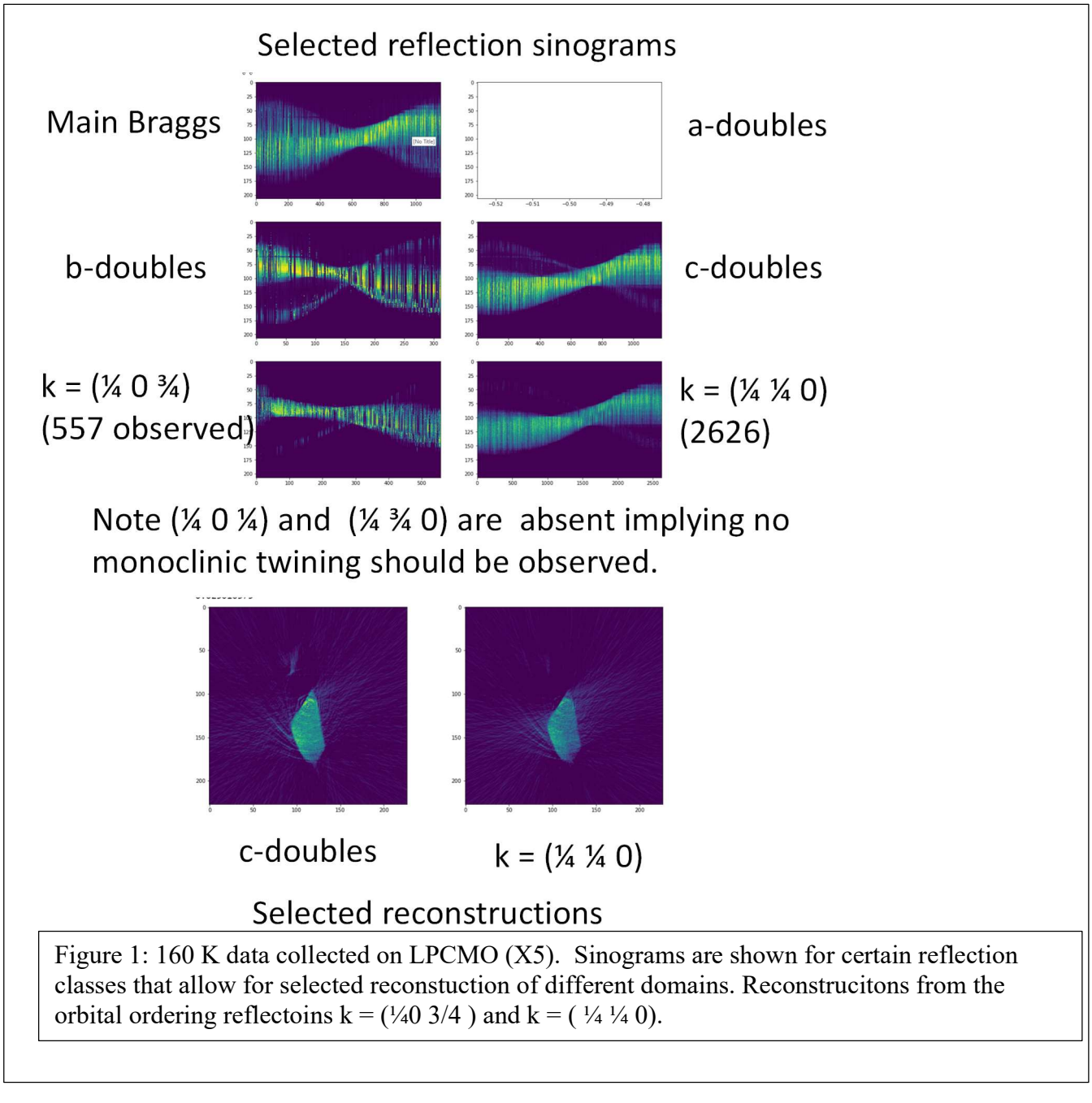

Figure 1: 160 K data collected on LPCMO (X5). Sinograms are shown for certain reflection classes that allow for selected reconstuction of different domains. Reconstrucitons from the orbital ordering reflectoins k = (¼0 3/4) and k = (¼ ¼ 0).