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: Operando Total Scattering Studies Of Amorphous Silicon For Lithium Ion Batteries Using A Multi-Channel Collimator To Probe Coin Cells**	**Experiment number**: CH6550
Beamline: ID15a	**Date of experiment**: from: 27 June 2023 to: 30 June 2023	**Date of report**: 28/02/2023
Shifts: 9	**Local contact(s)**: Stefano Checcia	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

David Wragg*, Alexey Koposov*, Casper Skautvedt*, Erlend T. North* - University of Oslo

Report:

Preliminary report: The experiment was partly successful. A test measurement with graphite proved the viability of "looking inside" a working coin cell using the multi channel collimator (MCC) and obtaining high quality XRD data. Unfortunately, the 30x18 micron focussed beam (focused with X-ray lenses) was so intense it shut down the electrochemistry at the illuminated spot and no changes were occurred despite clear electrochemical evidence of full lithiation of graphite. Scanning slightly away from the area illuminated during the operando test showed lithiation. We intend to publish this as a short study of beam deactivation during operando battery XRD (especially at very bright sources). The influence of the beam is not limited to the directly illuminated area.

For the main study on silicon and silicon nitride we used a sample holder to scan over 5 cells, allowing time for each to "cool" between XRD scans. We also made a line scan across each cell, averaging the data afterwards. This not only prevented the beam from stopping electrochemistry in specific areas, but also gave us different regions to choose from. Unfortunately, it was difficult to ensure that the electrodes inside the cells were aligned parallel to the beam, and this combined with the long footprint of the beam gauge volume and relatively thin electrodes meant that significant amounts of copper current collector were sampled along with the active material. This caused problems with the PDF analysis as the crystalline copper dominates the PDF, we are working on methods for improving this, including use of difference PDF. For future experiments a smaller beam is suggested. KB mirrors could be a possibility, but this level of focussing has apparently not been tried with the MCC before. For crystalline silicon the amorphization process during lithiation is clearly observed, from inside a normal coin cell (fig. 1). This kind of data collection previously proved impossible with XRDCT due to multiple scattering from different regions of the sample, leading to multiple overlapped Bragg peaks.

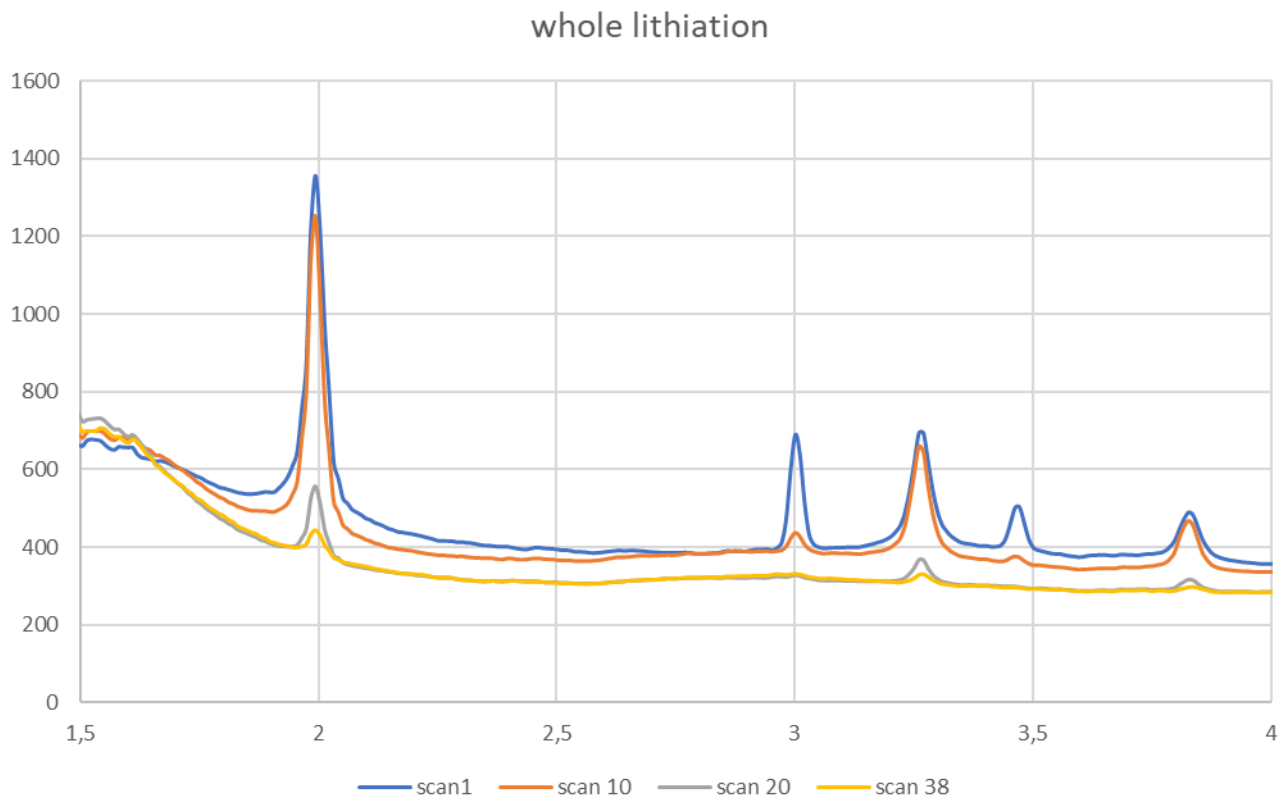

Figure 1. Lithiation of crystalline silicon inside a steel coin cell observed operando with MCC-XRD.

Due to the small area of the Pilatus 2M detector covered by the MCC, effects from the sensor chip boundaries caused significant glitches in the integrated 1D scattering patterns- this introduced noise at low radial distances into the PDFs. We combatted this to some extent by collecting four images in each sample position, each with a slightly different detector position to cover the chip edges. In future we suggest using more detector scans or a taller MCC to further improve the results.