



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: Comparing silicon anode compositions for Li-ion batteries with Operando Total Scattering CT	Experiment number: CH-5682
Beamline:	Date of experiment: from: 8/6/21 to: 15/6/21	Date of report: 12/9/22
Shifts: 18	Local contact(s): Stefano Checcia	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): David Wragg* , Helmer Fjellvåg , Alok Mani Tripathy - University of Oslo Anders Brennhagen* - University of Oslo (not an original proposer)		

Report:

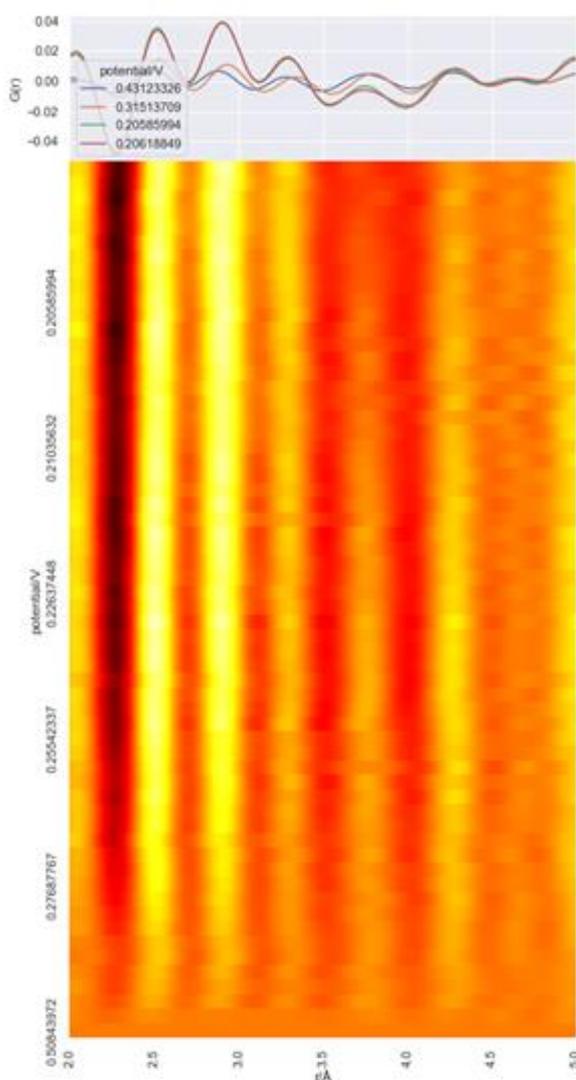


Figure 1 Difference PDF contour plot for lithiation of amorphous silicon, obtained using operando TSCT

Due to the COVID-19 situation, closure of the ESRF electrochemistry lab and rules preventing us from travelling by air with experimental lithium ion batteries, we had to bring assembled cells for total scattering CT (TSCT) from overland from Oslo to Grenoble. Unfortunately, by the time we arrived at the ESRF none of the cells was in good working condition and we could not assemble new ones. None of the cells went beyond the initial lithiation of the silicon, and only minimal lithiation (under 10 %) was observed.

Despite the experimental problems we were able to validate TSCT as a highly sensitive method for studying silicon lithiation. By subtracting the initial PDF of pristine amorphous silicon from the PDFs obtained during the operando experiment we can see changes in the structure, even with minimal amounts of lithium alloyed (figure 1).

The almost complete disappearance of the 3.8 Å peak in the difference PDF and the growth of the 2.85 Å peak agree with the literature measurements of ex situ PDFs. The difference method allows us to pick out trends which are almost invisible in the untreated PDF series. The number of data points in the operando experiment confirms that these observations are real trends, not just noise.

To conclude, we believe this experiment has shown the high sensitivity of TSCT for a weakly scattering material with a complex mechanism in a battery which did not perform very well. The less than 10 % lithiation is still observable. A manuscript based on the results is in preparation.