



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: <i>In situ</i> PDF studies on the structure and crystallization of Co/Ni-B-oxide catalysts for oxygen evolution reaction	Experiment number: CH-6212
Beamline:	Date of experiment: from: 01.04.2022 to: 02.04.2022	Date of report: 09.08.2022
Shifts:	Local contact(s): Gavin Vaughan	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Claudia Weidenthaler, MPI Kohlenforschung *Haritha Cheraparambil, MPI Kohlenforschung *Hilke Petersen, MPI Kohlenforschung *Tolga Han Ulucan, MPI Kohlenforschung *Jan Ternieden, MPI Kohlenforschung		

Report:

We performed temperature dependent total scattering experiments starting with amorphous precursors to follow their structure development to crystalline materials. A system containing different ratios of Co/Ni-B oxides (Co-B oxide, and Ni-B oxide) was studied. To briefly describe the experiment; samples were filled into quartz glass capillaries and was subjected to heating from room temperature to 745 °C with continuous flow of synthetic air (10 °Cmin⁻¹). Heat blower which could reach a temperature of 745°C was given from the beamline. These experiments were performed in a specially designed *in-situ* cell coupled with a pressure sensor. Data were collected in temperature steps of 50 °C. We also performed ex-situ total scattering experiments on some reference materials like Co₃O₄, NiO, B₂O₃ and mixed oxides. All the obtained data were further evaluated by pair distribution function (PDF) analysis using PDFgetX3¹ within the package xPDFsuite².

Preliminary analysis on the total scattering data from Ni-B oxides enables us to follow the structure development from the amorphous sample obtained after preparation to the crystalline material at 463 °C as shown in Fig1a. From the preliminary understanding of the PDF refinements, the amorphous sample at room temperature is a phase mixture of nickle oxide, boron oxide and mixed nickel-boron oxies as shown. With the temperature increase, the PDF data change (as shown in Fig. 1a) which indicates a structural rearrangement with NiO being the crystalline phase at 463 °C . The location of boron within the structures at elevated temperatures is still under progress. Detailed evaluation of the *in situ* total scattering experiments is ongoing. Data are part of the PhD work of Haritha Cheraparambil.

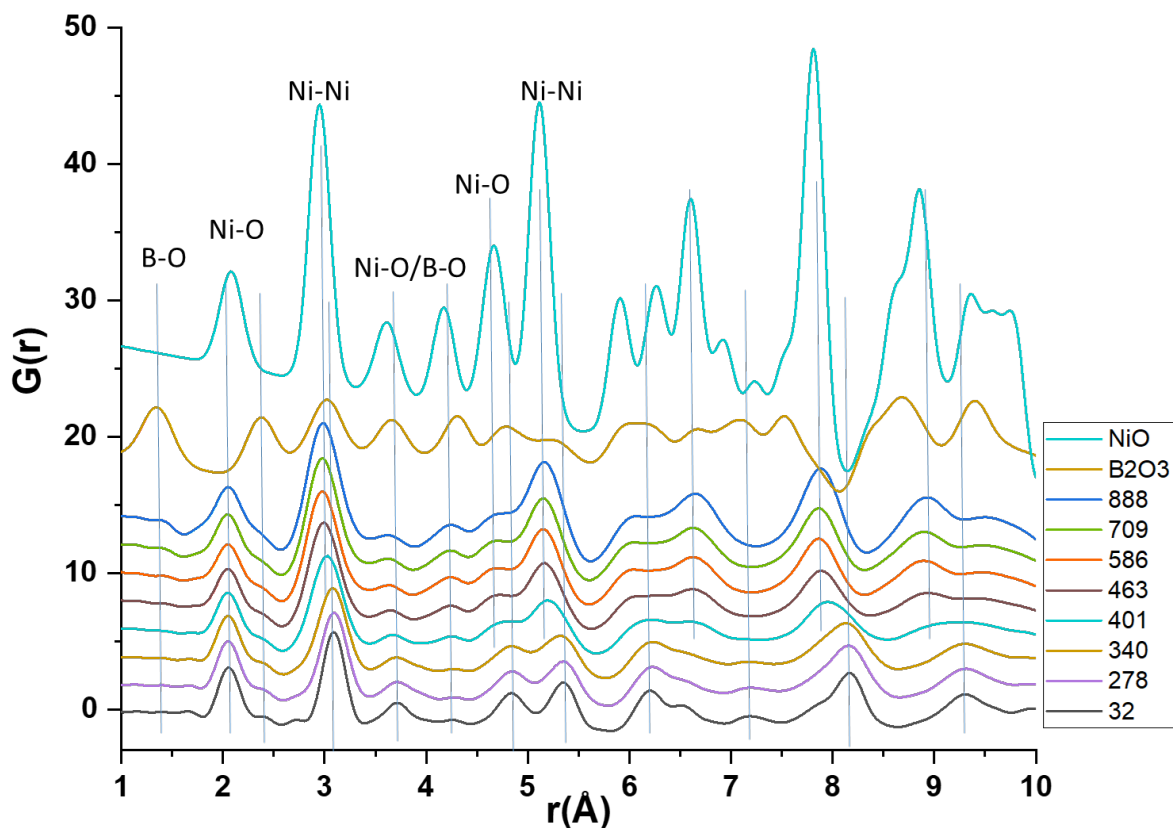


Fig1. *In-situ* PDF data collected at different temperatures compared with the simulated PDF of NiO and B₂O₃

Because of some general beamline problems, we lost more than one shift out of 2.5 shifts. We also encountered problems with calibration experiments as sensor for the heating setup was not working properly. We initially planned to perform temperature dependent experiments on six samples but we could only manage to perform two in situ total scattering experiments. One experiment on Co-B oxide failed because the capillary broke and we did not have enough time to arrange another experiment. Even though the beamtime got extended till 03.04.2022, we could not extend our stay due to our fixed travel schedule.

References

1. P. Juhás, T. Davis, C.L. Farrow and S.J.L. Billinge, *J. Appl. Crystallogr.*, 2013, 46, 560.
2. X. Yang, P. Juhás, C.L. Farrow and S.J.L. Billinge, 2015, arXiv 1402.3163v3.