EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

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:** Why are advanced Pt-alloyed ORR catalysts much better on paper than standard Pt NPs but fail in real devices?	**Experiment number**: CH-6234
Beamline: ID31	**Date of experiment**: from: 28/04/2022 to: 02/05/2022	**Date of report**:
Shifts: 12	**Local contact(s)**: Jakub Drnec	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The first shift was dedicated to beamline alignment and preparation of the setup. We faced a sudden technical issue when we tried to run the experiment. The fuel cell (FC) test station at ID31 had a malfunctioning gauge in the bubbler required for gas humidification. This caused severe flooding of the whole tubing system in the FC test station. We spent two shifts repairing it, and one shift was required just to let the entire system dry out. Due to that, we already started the actual X-ray measurements with one day delay.

This is a continuation of MA-5017. From the previous experiment, we reported samples: (i) commercial Pt/C with 0.6 V SW-AST, (ii) oh-PtNiIr with 0.6 V SW-AST, and (iii) oh-PtNiIr with 0.7 V SW-AST. To get a set for publication, we started the CH-6234 experiment with a commercial Pt/C with 0.7 V SW-AST. Due to the experiences acquired during the previous beamtime, we successfully reached high-current densities (~1.2A/cm2 at 0.45 V) and measured stable cyclic voltammetry. However, there were issues with the beam in the storage ring the first night, which caused data loss and delay. Thus, this sample took four shifts to measure. Despite this, we were able to finalize the dataset successfully, and we are now in preparation for the manuscript, see Figure 1. Clearly, the lower potential limit of the fuel cell operation significantly impacts the stability of the catalyst regardless of its composition.

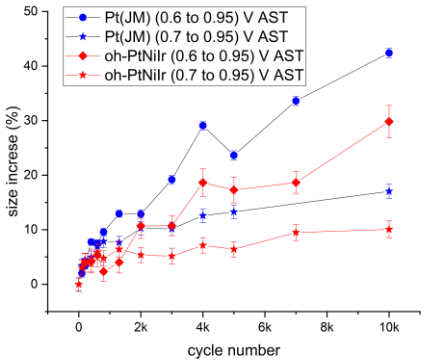

Figure 1: Completed dataset ready for publication – crystallite size evolution of Pt/C and oh-PtNiIr catalysts during two types of ASTs.

At this point, we already had just four shifts left. That is why we decided to try a shorter type of accelerated stress test (AST) in which we hold a constant potential for several hours. The first is with the potential hold at 0.6 V, and the second at 0.7 V. We acquired those datasets for two oh-PtNiIr samples. These data will be analyzed in the upcoming months.

One additional observation was made. When we compare the high-current operation of the commercial Pt/C and oh-PtNiIr catalyst, we see that oh-PtNiIr performs worse. Despite its higher electrochemical activity, we reached about 0.2 A/cm^2 lower current density at 0.45 V compared to the commercial Pt/C. This might be a consequence of the contamination of the Nafion membrane with Ni ions that leach out during the AST. To confirm this, we designed a new PEM fuel cell optimized for XAS fluorescence, with which we apply for beamtime at ID26.

Despite the initial hurdles, this beamtime was a success as we finalized the dataset for publication and acquired new quality data that will provide complementary information once analyzed. Generally, XRD experiments at ID31 ESRF are well suited to enlighten the knowledge gap between laboratory conditions and industrial applications.