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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

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

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.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal 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:** An in situ study of the formation of a IrO2(110) surface and its interaction with CH4	**Experiment number**: EV-293
Beamline: ID31	**Date of experiment**: from: 08.03.2018 to: 14.03.2018	**Date of report**: 22.05.2018
Shifts: 18	**Local contact(s)**: Maciej Jankowski	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The aim of this experiment was to investigate the pressure-dependent formation of iridium oxide phases - such as the IrO_2(110) surface - on an Ir(100) single crystal, and to study their interaction with gases under semi-realistic reaction conditions.
The experiment was performed in EH1 of beamline ID03, using an x-ray photon energy of 24 keV and a Maxipix 2D detector. To probe the sample under semi-realistic conditions, the ID03 in-situ catalysis chamber was used. The experiment was carried out at a constant total reactor pressure of 500 mbar and a sample temperature of 775 K.
In the first part of the experiment we investigated the time-resolved formation of the oxide structures by taking 2D snapshots in an reciprocal space area sensitive to Bragg peaks of the IrO_2(110) bulk oxide while adding a certain O_2 partial pressure to the pure Ar flow. Such a single snapshot, measured after the sample had been oxidized at a partial O_2 pressure of 5 mbar, is shown in Fig. 1 a). It reveals, in addition to the Ir(10L)-CTR of the single crystal surface, two Bragg reflections belonging to the IrO_2(110) bulk oxide. This in-situ oxidation study was carried out at the three different O_2 partial pressures of 2.5 mbar, 5 mbar and 10 mbar O_2. In each case, the oxidized sample was thoroughly characterized by scans along high symmetry directions, in-plane mesh scans and x-ray reflectivity. Fig. 1 b) shows such an in-plane mesh scan from the sample oxidized at 5 mbar O_2 partial pressure, a complex peak pattern stemming from the oxide phases can be seen.
In the second part of the epxeriment we studied the behaviour of the different oxide phases under operando conditions for CO oxidation. The measuring of 2D snapshots allowed once

again for a time-resolved investigation of the oxides in the transition to conditions of high catalytic activity.
We consider the beamtime as very successful. The observed oxide structures turned out to be more complex than expected and are currently being analyzed and compared to LEED measurements performed in our homelab. We think that future SXRD studies of these oxides might be needed to fully unravel their structure. In addition, we would like to study their interaction with methane in future experiments.

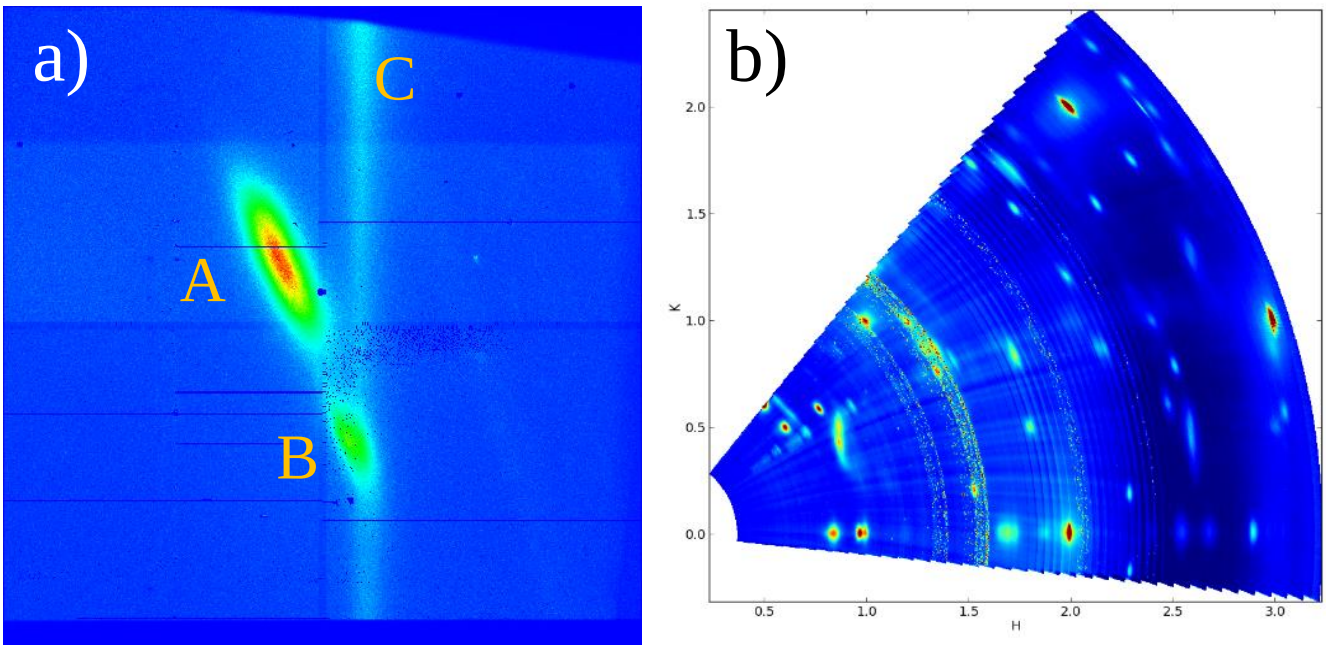

Fig. 1*: a): out-of plane reciprocal space map measured after oxidation at 5 mbar O_2 flow with the 2D detector around the Ir(10L)-CTR (C): Two iridium oxide peaks can be seen (A and B); b): in-plane mesh scan revealing a complex peak pattern that can be traced back to different iridium oxide phases.*