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:** Characterization of a novel technical catalyst for oil refinery by Spectral-Ptychography	**Experiment number**:
Beamline: ID16B	**Date of experiment**: from: 04/03/2021 to: 08/03/21	**Date of report**:
Shifts: 12	**Local contact(s)**: Jaime SEGURA RUIZ	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):
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Report:

The objective of this beamtime was to investigate a novel technical catalyst with spectral-ptychography. This technique is not yet well established at the ID16B beamline, but the feasibility has been shown in the former beamtime (MI-1346). This beam time served both to further develop the method on the beamline and to use the knowledge already gained from the feasibility study to obtain valuable information about the catalysts under investigation.

The studies include 2D spectral-ptychographic scans and complementary low-resolution X-ray fluorescence scans of the samples. The chosen samples are 150-nm thin slices of fluid catalytic cracking (FCC) particles deposited in Si_3N_4 membranes. Three different particles in different states (fresh, spent, and regenerated) were investigated. The samples are composed of zeolites, an alumina binder, kaolin clay, SiC, and a metal-oxide additive, in this case, Mn_2O_3. The goal was to localize the Mn in the catalyst and determine the oxidation state of the active metal site. Therefore, the ptychographic scans were repeated at 40 different energies around the Mn K-edge (6539 eV) including the resonant (edge) energy.

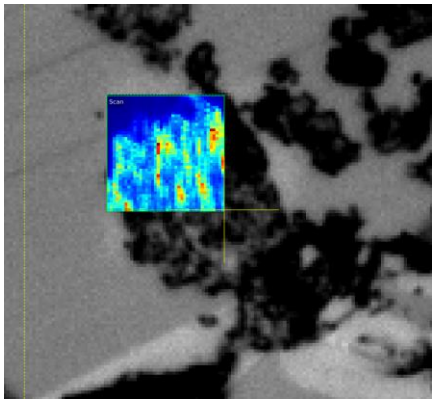

Figure 1 - XRF map (Mn K alpha) of a part of the regeneretated FCC catalyst particle.

The first two shifts were used to prepare the beamline for the experiments. The preparation included the installation of a vacuum flytube to avoid the absorption of the scattered radiation in the air between the sample and the detector, which has to be as far away as possible for the proper sampling of the Fourier space. As a detector, we used a maxipix with a pixel size of 55x55 µm², which had to be installed at the correct height. For the energy calibration, we performed scans of a Mn foil. The flux had to be adjusted to ensure the linearity of the detector. To make sure that the detector pixels and the scanning direction of the sample stage are parallel, the first scans were performed with an InGaN nanofiber sample.

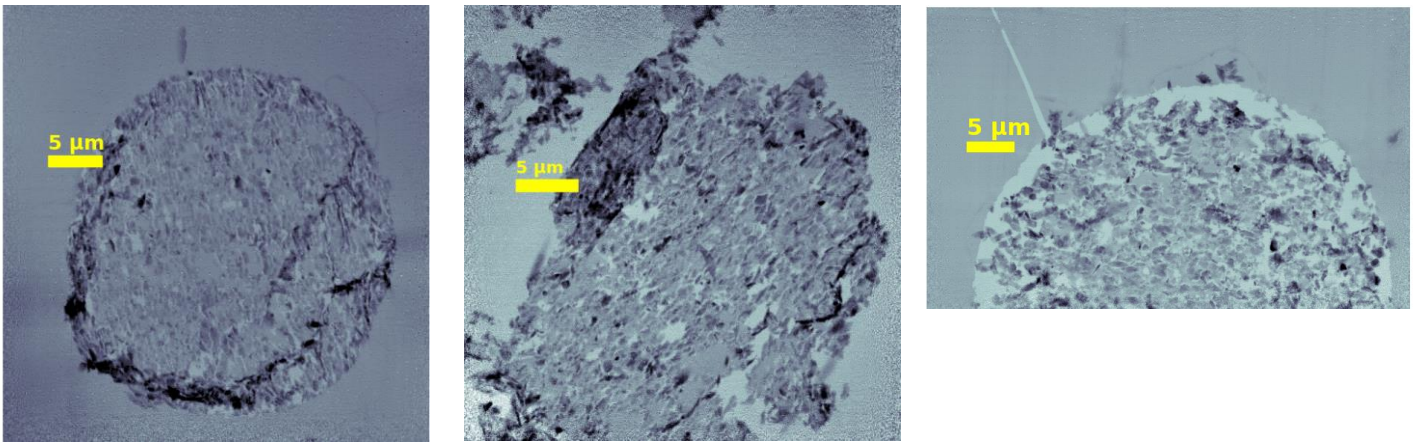

Figure 2 - Reconstructed phase images of a FCC catalyst particle at different lifetimes: fresh (left), spent (middle) and regenerated (right)

The following shifts were used for the resonant ptychography experiment. For each sample, test scans were performed to find the best scan parameters, that is, the scan region, the number of scan points, the step size and the acquisition time per scan point. The number of scan points per sample was around 900, the step size between 1 µm and 1.3 µm, and the counting time around 1 ms. The pixelsize in the reconstructed phase images is around 35 nm. One ptychographic scan at one energy took around 15 minutes, and hence the pure measurement time for one sample was around 10 hours (without alignment and test scans).

We could successfully reconstruct the phase images of the three catalyst samples for almost all energies. At some energies, we had problems that could be related to instabilities of the beam and a loss of coherence, which could be observed especially directly after injections. We could also observe changes in the resin around the samples. Nevertheless, for the first time, the microstructure of an FCC catalyst particle is revealed by ptychography at the beamline ID16B. The experiments and first results have already been presented in an oral presentation at the IUCr congress 2021. We also plan to publish the results.

The spectral analysis of the data turns out to be difficult due to several reasons, including the instabilities of the beam, but also the very thin sample with a low concentration of Mn. Data analysis is still in progress.