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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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:** The effect of S and Cl poisoning on the active phase and its distribution in Cu/Zn based catalysts	**Experiment number**: CH-3076
Beamline: ID15b	**Date of experiment**: from: 07/07/2010 to: 12/07/2010	**Date of report**: 16/03/2011
Shifts: 12	**Local contact(s)**: Marco Di michiel	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr Andrew michael Beale *

Dr Simon Jacques *

prof. Bert Weckhuysen

Dr Matthew O'brien*

Report:

The aim of this project was to examine the effect of both chlorine and sulphur poisoning processes on a series of industrial sized CuZnO methanol synthesis and water gas shift production catalysts. Utilizing the high energy X-rays of ID15 allows us to penetrate the large sample environment whilst still maintaining a high quality signal and very fast measurement speeds. Using these advantages the aim was to be able to reconstruct 2 and 3D tomographic diffraction and fluorescence images from the sample.

The experiment was largely successful and large amount of high quality diffraction data was recorded which has now been reconstructed into ‘slices’ of the catalyst sample. Some examples of this are given in Figure 1 where we show how for a single pellet exposed to 200 ppm sulphur poisoning in the gas stream (performed offline and followed by a passivation procedure) results in a high concentration of metal oxide and metallic species on the inside of the pellet and the production of numerous poison species on the outside of the pellet. From these data and on-going additional laboratory experiments we should be able to begin to understand the poisoning processes in these full size, industrially relevant materials and we hope to produce a peer reviewed publication on this work as the project proceed in 2011.

Unfortunately, whilst the diffraction imaging has proved very successful, the Compton florescence mapping of the sample was not as successful as we have found that the density of the samples results in strong self-reabsorption making it very difficult to reconstruct the images correctly. In addition we note that the work to process the data for PDF is on-going, however the quality of the data for the time resolution required may still be too low, something which can be resolved in the future with the recent addition of the tranforcator mirrors on the beam line (allowing more flux for the same spatial and time resolution).

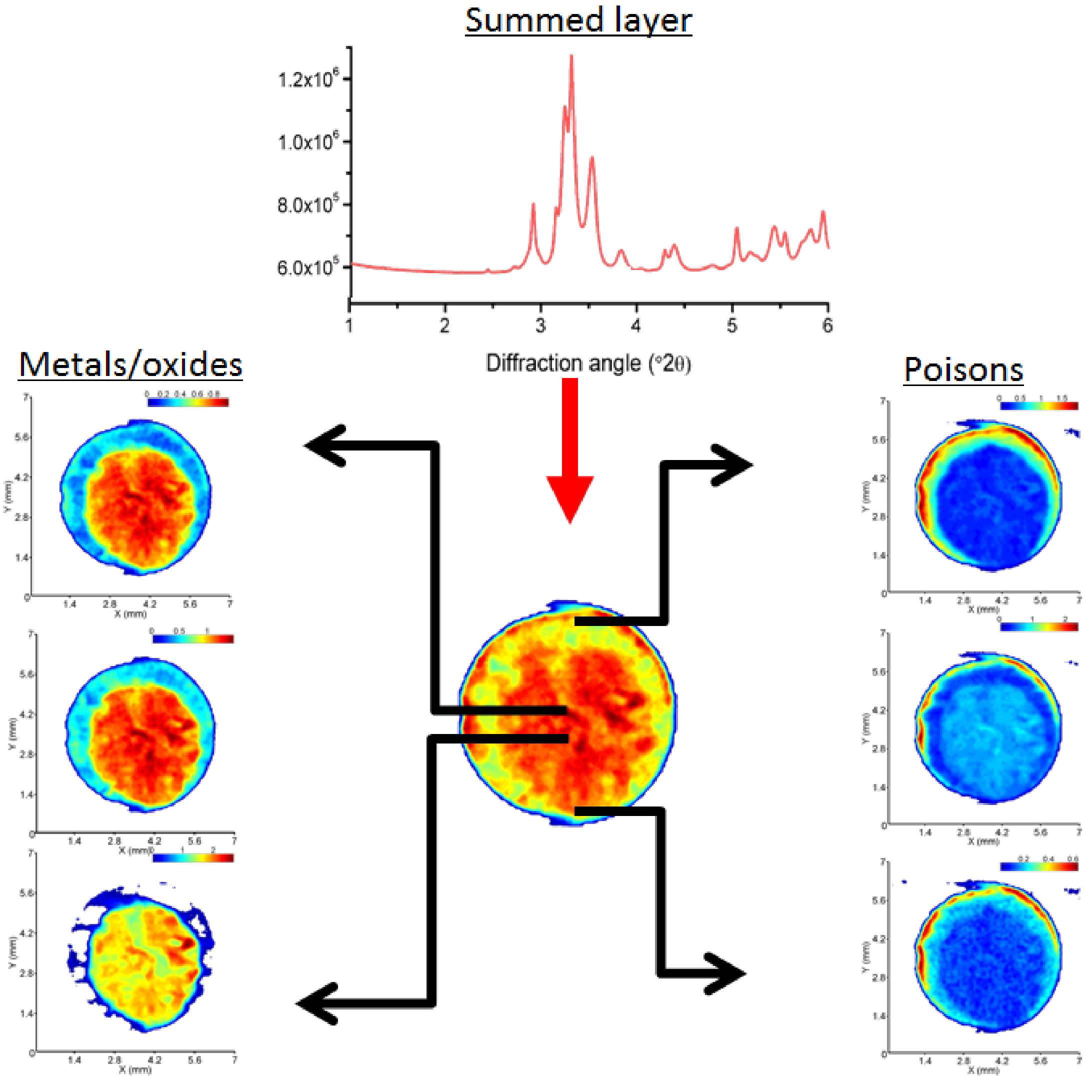

Figure 1: Demonstration of some of the data obtained from this beam time. The image shows how the spatial details form the summed diffraction image of the entire layer can be obtained using back projection algorithms, allowing us to observed the distribution of various metals, oxides and poisons within an cylindrical industrial catalyst body.