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

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.

	Experiment title: Time-resolved 3D XRD-CT of low-temperature water-gas shift catalysts during activation; effects of catalyst and gas composition on crystalline phase and deactivation processes.	**Experiment number**: CH-5143
Beamline: ID15a	**Date of experiment**: from: 14/06/17 to: 19/06/17	**Date of report**: 13/02/18
Shifts: 10	**Local contact(s)**: Marco di Michiel	*Received at ESRF:*

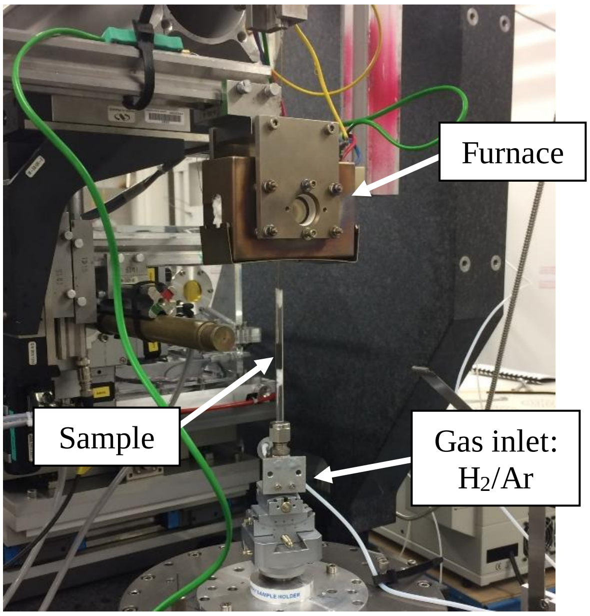
Names and affiliations of applicants (* indicates experimentalists):

Andrew Beale*, Daniela Farmer*, Jennifer Herbert* (UCL), Antony Vamakeros*, Simon Jacques* (Finden), Dorota Matras* (UoM)

Report:

The catalysts investigated consisited of an industrially relevant (**60/30/10**) and high copper loading (**80/10/10**) **$Cu/ZnO/Al_2O_3$** catalyst which had also been doped with **1wt.% Cs_2O**. A series of reductions, as presented in the table below, were performed on each of these catalysts on the ID15a beamline at the ESRF. For each experiment, 0.3 g of sieved catalyst was packed into a 5 mm quartz capillary (see photography on page 2) and 10 mL min^{-1} reducing gas was flowed.

Exp. #	Sample	Gas [H_2]/Ar (%)	GHSV (h^{-1})	Temperature Programme
1	60/30/10	5	200	RT to 160°C at 5°C min^{-1} 160°C to 230°C at 1°C min^{-1} Hold at 230°C till reduced
2	60/30/10-Cs	1	1985	
3	60/30/10	1	1985	
4	60/30/10	5	1985	
5	60/30/10-Cs	5	1985	
6	60/30/10	2.5	1985	
7	60/30/10-Cs	2.5	1985	
8	80/10/10	5	1985	
9	80/10/10-Cs	5	1985	



XRD-CT data was collected across 3 planes of the catalyst bed (bottom, middle, top) at room temperature, when 230°C (reduction temperature) was reached and at the end of reduction; point measurements were also collected along the bed throughout the whole reduction process. We are currently in the process of analysing the data, however, initial analysis shows that there is a difference in the reduction behaviour depending on the position in the bed and inclusion of the Cs-dopant.

Included is an example of the information we can gain from these experiments. An advantage of collecting the point measurements is that we are able to follow the evolution of the active Cu phase along the bed. Figure 1 shows that there is no real difference in reduction behaviour as a function of bed height for the 60/30/10 Cu/ZnO/Al_2O_3 catalyst in 5% H_2/Ar. However, when we consider the XRD-CT data (Figure 2), we are able to see that there is a clear distribution of stacking fault probabilities across the bed. This then gives us a new insight into possible deactivation mechanisms occurring in industrial reactors.

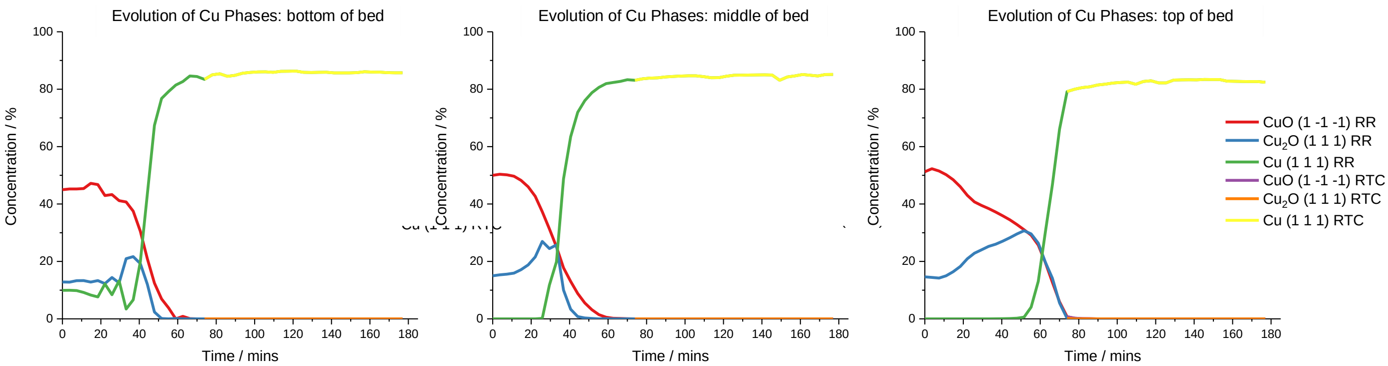


Figure 1: Evolution of the Cu phases dependent on bed height in the 60/30/10 sample at 5% H_2/Ar (RR = reduction ramp, RTC = reduction temperature constant)

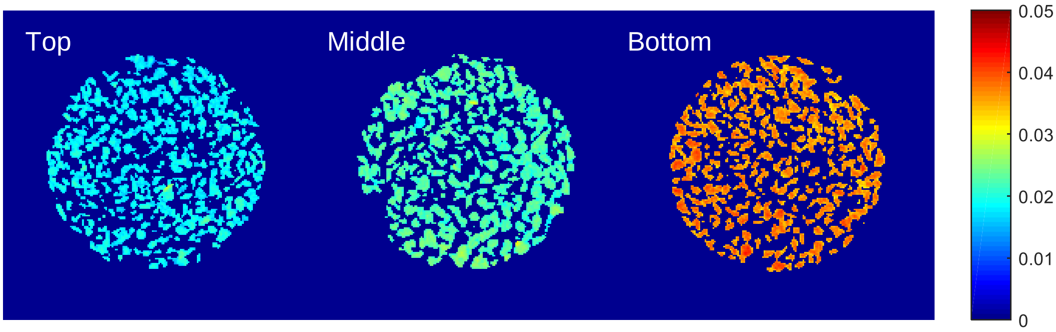


Figure 2: XRD-CT images showing the distribution of stacking fault probabilities across the catalyst bed at the end of reduction