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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:** In situ investigation of formation metal oxide catalyst during hydrothermal synthesis	**Experiment number**: 26-01-881
Beamline:	**Date of experiment**: from: 03/03/2010 to: 08/03/2010	**Date of report**:
Shifts:	**Local contact(s)**: Sergey Nikitenko	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Wim Bras

Sergey Nikitenko

Gopinathan Sankar

Vladimir Martis

Report:

This report is on an experiment carried out at DUBBLE from 3rd of March till 8th of March 2010.We used combined EXAFS/XRD technique to investigate the structural changes that took place during isothermal hydrothermal reaction of multicomponent mixed metal molybdates .

Here are presented only data from hydrothermal reaction of Bi_2MoO_6 at 150 ^{0}C, though an experiment was carried out at another 3 temperatures.
The precursor Bi_2MoO_6 was synthesised by precipitation method. The resulting gel was loaded into hydrothermal cell and then heated at 150 ^{0}C for several hours, until it completely crystallized.

In Figure 1 are shown EXAFS data of formation γ-Bi_2MoO_6 from amorphous precursor gel at 150 ^{0}C collected at Mo K-edge. It is known that intensities of the pre-edge peak and white line are indicators for determining the coordination environment of Mo (IV) species.

Initially the EXAFS spectrum was collected from freshly prepared gel at room temperature in order to identify species present in the starting gel. As we can see in Figure 1 the EXAFS spectrum in red suggests that tetrahedral species are present in the initial precursor gel because of the strong pre-edge peak (marked as A) and weak shoulder on the top of the edge. The pre-edge peak is higher due to 1s -4d transition in tetrahedrally coordinated system compared to octahedral one (1s-5p transition is lower for tetrahedral compared to octahedral one). During isothermal crystallisation over period of ~ 240 min we can notice the decrease in pre-edge intensity and increase in the intensity of the shoulder on the top of the edge. This corresponds to changes in coordination of Mo(IV) from tetrahedral to octahedral. The EXAFS spectra in blue illustrates Mo(IV) in octahedral coordination.

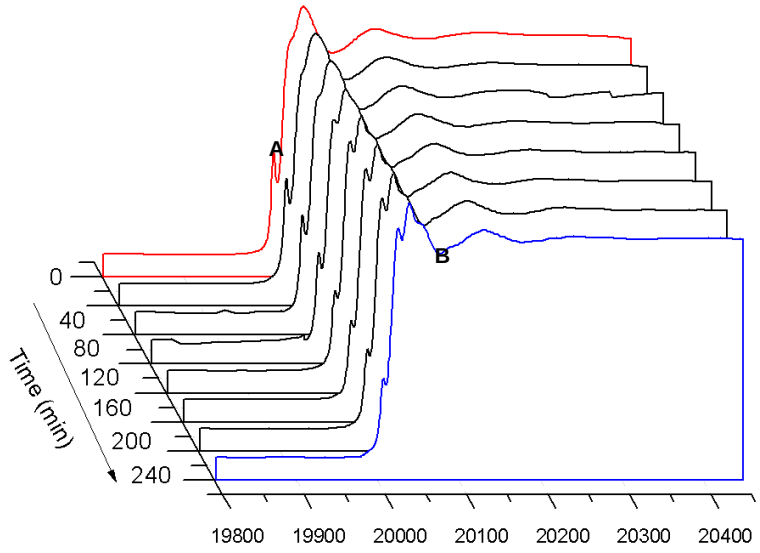

Figure 1 Mo K-edge EXAFS data of γ-Bi_2MoO_6 collected during crystallisation at 150 ^{0}C. Red EXAFS spectrum was collected from freshly prepared gel at room temperature. The feature marked as A is characteristic of Mo (IV) in tetrahedral environment. Blue EXAFS spectrum was collected after 240min at 150 ^{0}C. The feature B is characteristic of Mo (IV) in octahedral environment.

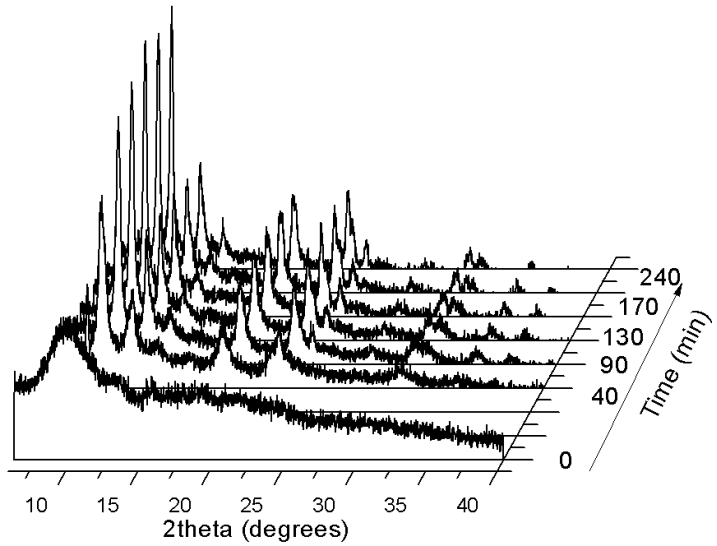

Figure 2 Stacked XRD pattern recorded during hydrothermal synthesis of precursor gel of Bi_2MoO_6 at 150^{0}C. Diffraction pattern at time 0 was taken from freshly prepared Bi_2MoO_6 gel at room temperature. Data were recorded at wavelength of 0.625 Å.

Inel detector was used for collecting diffraction patterns. XRD patterns were noisy with a poor angular resolution but sufficient for following changes taking place during isothermal crystallisation of amorphous gels. In Figure 2 is illustrated formation of γ-Bi_2MoO_6 during hydrothermal reaction at 150 ^{0}C. At time 0 was taken XRD pattern from freshly prepared gel which was amorphous. After 40 min of hydrothermal reaction at 150 ^{0}C the initial gel crystallized and formed the γ-Bi_2MoO_6.

Sum up

We were able to collect high quality EXAFS data during isothermal crystallisation of precursor gels. These EXAFS spectra are suitable for complete EXAFS analysis which gives us information about interatomic distances and coordination numbers. We used diffraction measurements for following phase transformation during isothermal crystallisation of gels.

Following changes during the isothermal crystallisation of $NiMoO_4$ at various temperatures didn't work well, as sample never fully crystallized.

The rest of data have been analysed and assessed.