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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 using the **Electronic Report Submission Application:**

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

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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.

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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.

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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:** Depth resolved electron yield EXAFS on model catalysis systems	**Experiment number**: 26-01-880
Beamline:	**Date of experiment**: from: 30/03/2010 to: 04/04/2010	**Date of report**:
Shifts:	**Local contact(s)**: Sergey Nikitenko	*Received at ESRF:*

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Nikitenko Sergey

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Martis Vladimir

Report:

This is preliminary report on an experiment carried out at DUBBLE from 30th of March till 4th of April 2010.
This report cover a description of the experimental setup and conclusion on data obtained from e-yield measurements using the electron yield detector.

The e-yield detector was mounted on a goniometer the way that a sample was on the centre of rotation. During measurements the sample was inclined at 2.5^0 with respect to the incoming X-ray beam. Using slits, the X-ray beam size was set to 3 mm (width) by 0.8 mm (height). This beam size produced a beam spot covering an area of ~45.8 mm^2. The detector was operated with standard gas mixture of 25 % isobutene and 75% Helium; drift voltage of – 1 500 V, cathode potential -550 V and with anode held at ground.

As a reference sample to test detector was used NiO/Ni thin file.
The structure of NiO/Ni consisted of 150 nm layer of Ni covered by layer NiO of thickness 25 nm deposited onto a Si (100) substrate.

In
Figure *1* is shown the typical Auger electron spectrum collected with the e-yield detector at the Ni K-edge. This electron spectrum is divided into 4 different electron regions. After integrating the number of counts within each region, the EXAFS spectrum for 4 energy regions are obtained. Before performing full EXAFS analysis, the EXAFS spectra have to be normalised on incoming X-ray beam.

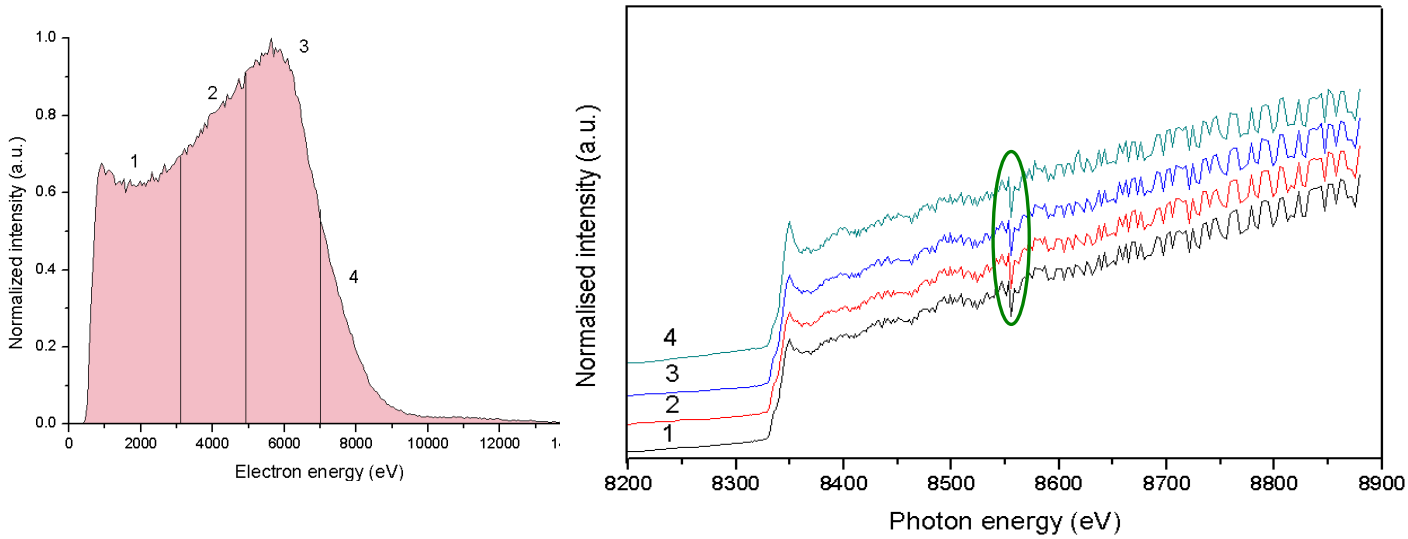

Figure 1 Left -Typical PHD recorded at Ni K-edge showing sampled 4 different energy regions. Right - four energy resolved EXAFS spectra corresponding 4 different electron energy windows.

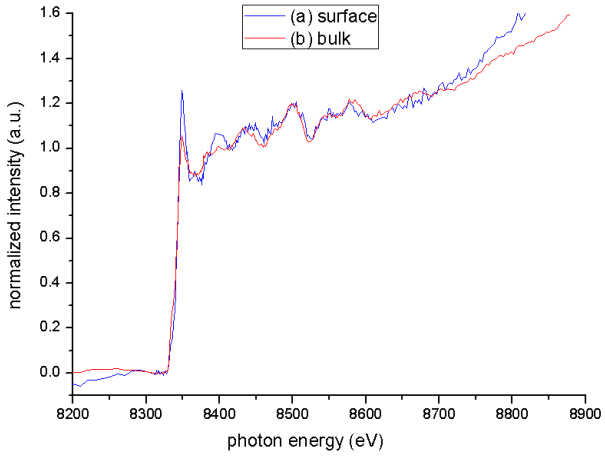

Figure 2 *3* Energy resolved EXAFS spectra of the NiO/Ni using low energy window referring to bulk and high energy window referring to surface.

In order to see clearly changes in magnitude of the sharp edge, in **Figure 2** are shown EXAFS spectra of the same NiO/Ni sample corresponding to low and high Auger electron energy windows. The low Auger electron energy refers to electrons coming from the bulk (b) (Ni) and high Auger electron energy is assigned to electrons escaping from the surface (a) (NiO). Increase in magnitude of the sharp edge (~8341 eV) from 1.05 to 1.25 is evidence of NiO contribution to EXAFS spectra at different Auger electron energies.

We also found out that sample holders made of alumina, bras, copper can be used for powder samples e.g. $CoMoO_4$, Fe_3MoO_4, Fe_3O_4 deposited on conductive carbon tape. Data obtained from powder samples are being analysed.

We also carried out test experiment with the GMSD on Ti K-edge. Aim of the experiment was to obtain information on the structure of the surface layer as well as to see different between bulk and surface of TiO_2/Ti and TiO_2/SS treated at 120/550 ^{0}C and 550 ^{0}C respectively . The surface layer of TiO_2 can be in a form of rutile or anastase. The preliminary results on this work are presented below.

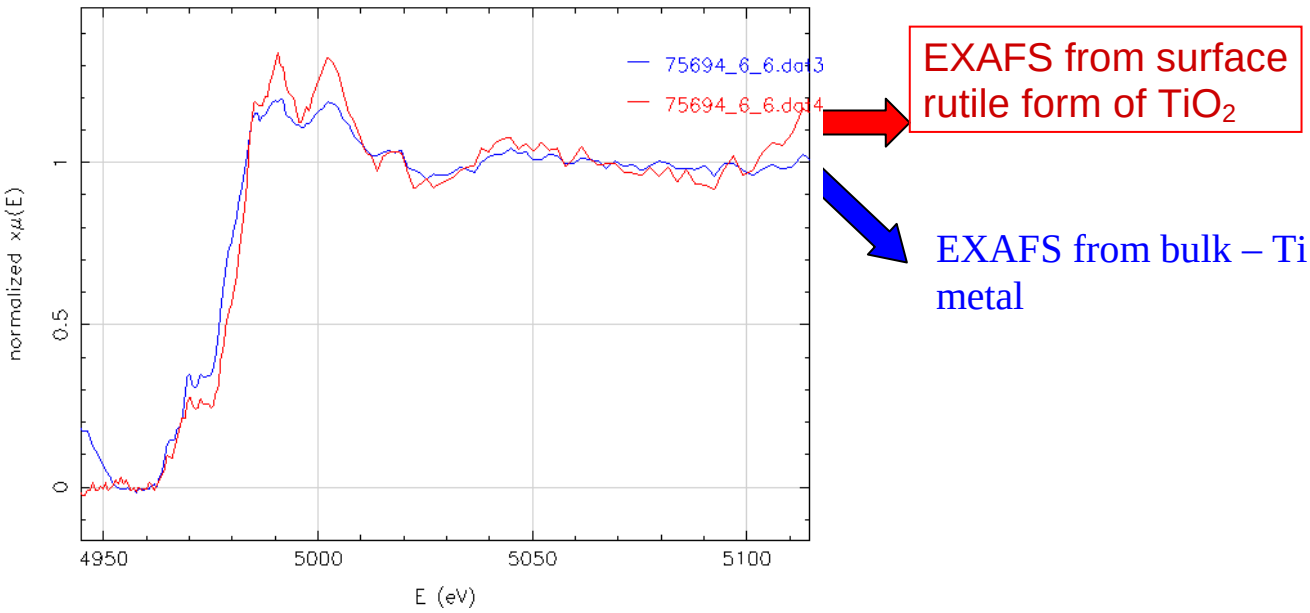

Figure 4 Energy resolved EXAFs of TiO_2/Ti_120 measure by e-yield detector.

In ***Figure 4*** are shown EXAFS spectra obtained by dividing PHD spectra into two electron energy regions; high energy window for electrons emerging close to the surface and low energy window for electrons that originated in the bulk.
The same TiO_2/Ti_120 sample was measured in fluorescence mode which gave us the EXAFS spectrum of Ti metal as can be seen in ***Figure 5***.

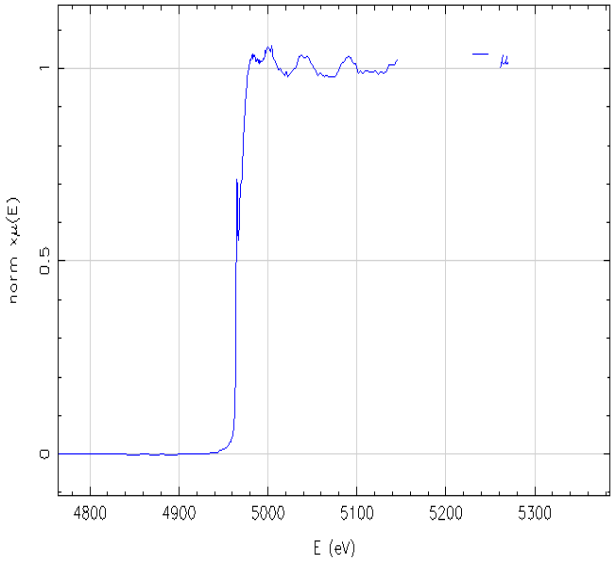

Figure 5 Fluorescence EXAFS of TiO_2/Ti_120 collected at Ti K-edge.

Conclusion

Our preliminary results on the energy resolved XAS collected by e-yield detector show that it is possible to distinguish the structure of the surface from that of the bulk material, as we demonstrated on preliminary results on NiO/Ni and TiO_2/Ti_120 samples.