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 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.

	Experiment title: Structure of ZnO(10-10) – H_2O interface as a function of water partial pressures using SXRD	**Experiment number**: SI-2189
Beamline: ID32	**Date of experiment**: from: 01/09/2010 to: 15/09/2010	**Date of report**: 12/07/2011
Shifts: 18	**Local contact(s)**: Parasmani Rajput	*Received at ESRF:*

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Report:

The adsorption of water on the ZnO(10-10) surface has been extensively studied both experimentally and theoretically [1] and is important in connection with a wide range of technologies (eg. Light harvesting, heterogeneous catalysis and methanol synthesis) [2,3]. Wang et al [4] used a combination of High Resolution Electron Energy Loss Spectroscopy (*HREELS)* and Thermal Desorption Spectroscopy *(TDS)* to investigate the interaction of water with this surface by exposing the clean, Ultra High Vacuum *(UHV)* prepared surface to up to 10^{-6} mbar water at room temperature. Their experimental data indicates, through new losses in the *HREELS* at 0.1 L water exposure compared to the clean surface, that there is partial dissociation at the surface. With increasing water exposure the losses increase in intensity whereas the frequency remained constant. This finding is in agreement with another combined investigation using He Atom Scattering *(HAS)*, Low Energy Electron Diffraction *(LEED)*, Scanning Tunneling Microscopy *(STM)*, and *He-TDS* [5]. We proposed to investigate the adsorption of water with Surface X-ray Diffraction *(SXRD)* as a function of water partial pressure, particularly focusing on higher partial pressures to probe a more technologically relevant regime.

The ZnO(10-10) surface was prepared *in situ* by repeated cycles of argon ion sputtering and annealing until a sharp 1×1 *LEED* pattern and characteristic *STM* images were obtained. Auger Electron Spectroscopy *(AES)* showed no signs of contamination within the detection limits. The sample was prepared in the *ESRF's* Surface Characterisation Lab *(SCL)*, and then tranferred to a *Baby* chamber [6] under vacuum. *SXRD* measurements were performed on

ID32 where the *Baby* chamber is mounted on the diffractometer in *EH1*. Data were collected at room tempertaure, employing a photon energy of 17.7 keV, using conventional rocking scans, in which the sample is rotated about its surface normal while scattered x-ray intensity is measured. For a given *(h,k)* these were performed at different *l*, enabling Crystal Truncation Rods *(CTRs)* to be compiled.

Initially, 23 *CTRs* were recorded for the ZnO(10-10) substrate in *UHV* to ensure sample integrity and to easily identify structural changes due to the presence of water. Next the surface was exposed to 10 L of H_2O ($5x10^{-8}$ mbar for 200 s) and a further 12 *CTRs* were obtained. Further diffraction data were recorded for two partial pressures of water, $5x10^{-7}$ mbar (10 *CTRs*) and 8 mbar (6 *CTRs*). *Figure 1* shows the (2,0,*l*) and (2,1,*l*) scans measured for the ZnO(10-10) surface in *UHV*, the 10 Langmuir of H_2O exposed surface and the two different partial pressures, demonstrating that there is some variation in the diffracted profiles. Analysis is currently underway to quantitatively determine the structure of each of the surfaces.

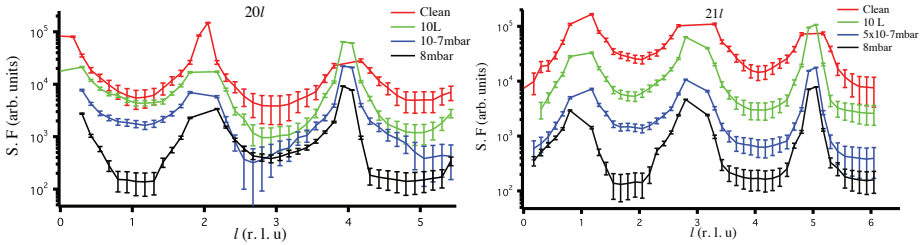

Figure 1 - (2,0,l) and (2,1,l) scans aquired from the ZnO(10-10) surface in UHV (Red) and the ZnO(10-10) - H_2O interface at 10 Langmuirs (Green) and partial pressures of $5x10^{-7}$ mbar (Blue) and 8 mbar (Black). Graphs are offset vertically for clarity.

References

[1] C. Woll, *Surf. Sci*, 2007, **82**, 55 – 120.
[2] T. S. Askgaard *et al, J. Catal*., 1995, **156**, 229.
[3] Martinson, A.B.F *et al*, *Phys. Chem. Chem. Phys.*, **8**, 4655.
[4] Wang *et al*, *Phys. Chem. Chem. Phys*., 2006, **8**, 1521 – 1524.
[5] B. Meyer *et al*, *Angew. Chem. Int. Ed.,* 2004, **43**, 6642 – 6645.
[6] http://www.esrf.eu/UsersAndScience/Experiments/StructMaterials/ID32/Preparation_Lab