EUROPEAN SYNCHROTRON RADIATION FACILITY

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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

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:** Structure of TiO2(110) – Impact of UV-induced hydrophobic/hydrophilic transition	**Experiment number**: 28-01-972
Beamline: BM28	**Date of experiment**: from: 13/06/2012 to: 19/03/2012	**Date of report**: 21/03/2013
Shifts: 18	**Local contact(s)**: Oier Bikondoa	*Received at ESRF:*

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Report:

It is well established that under ambient atmospheric conditions UV irradiation induces a reversible hydrophobic to hydrophilic transition on the surface of titania [1-5]. However, rigorous mechanistic understanding of this phenomenon is lacking. Many pertinent details remain uncertain, including knowledge of the impact of UV-light on the nanoscale surface structure of titania. The aim this experiment was to address this key issue through determining the structure of a model titania surface, TiO_2(110), in presence/absence of UV radiation. Surface X-ray diffraction (*SXRD*) was employed for this study, as this approach facilitates quantitative surface structure determination without the need for an ultra high vacuum (*UHV*) sample environment.

The TiO_2(110)(1x1) surface was prepared using a *wet-chemical* approach, involving annealing in air (~1000 K) and chemical cleaning through immersion in acid (HCl/HNO_3 mixture). This procedure has been developed in Manchester, and is known to produce a well-ordered surface, as characterised by both low energy electron diffraction (sharp (1x1) pattern) and atomic force microscopy (> 200 nm terrace widths) [6]. Such a surface is known to be hydrophobic from contact angle measurements, but can easily be switched to hydrophilic through exposure to UV-light (contact angle tends to 0°).

For the *SXRD* measurements the sample was inserted into a simple environmental chamber, in which the gaseous atmosphere can be controlled, and both UV-light and X-rays can enter. This chamber was mounted on the diffractometer in the *XMaS* experimental hutch. Diffraction data were collected ($h\nu$ = 15 keV) using a 2D photon detector to acquire scattered X-ray intensity. For a given *(h,k)*-integer, data were measured as a function of *l*, enabling profiles of scattered intensity versus perpendicular momentum transfer, known as crystal truncation rods (*CTR*s), to be compiled. In addition, reciprocal space was searched further at low values of *l* to look for fractional order reflections.

Initially, diffraction data were collected in the absence of UV-light to assess the structure of the 'hydrophobic' surface. These measurements were performed with the sample in a He atmosphere to avoid any surface damage due to X-ray induced ozone. Unfortunately, due to a technical problem with the diffractometer, three days were spent trying to align the sample correctly, as no diffracted intensity was apparent away from the Bragg peaks along the *CTR*s. Following resolution of this issue by beam line staff, we were able to pursue our measurement programme. However, given the limited amount of beam time remaining, it did not prove possible to acquire data with sufficient signal-to-noise ratios to be useful for analysis. Swapping to another TiO_2(110) sample did not improve the data quality.

Subsequently, similar measurements have been performed at I07 on the Diamond Light Source. The increased flux enabled suitable data to be acquired, which is currently undergoing analysis. It is interesting to note that even in a flow of He in the environmental cell, the X-ray beam switches the TiO_2(110) surface into a hydrophilic state, without the need for UV irradiation.

References

1. R. Wang *et al*, Nature 388, 431 (1997).
2. A. Fujishima *et al*, Surf. Sci. Rep. 63, 515 (2008).
3. T. Zubkov *et al*, J. Phys. Chem. B 109, 15454 (2005).
4. N. Sakai *et al*, J. Phys. Chem. B 107, 1028 (2003).
5. R. Wang *et al*, Adv. Mater. 10, 135 (1998).
6. M.H.M. Ahmed *et al*, *to be published.*