

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://www.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: Investigation of the interaction of water with natural anatase TiO ₂ (101) and (001) and brookite TiO ₂ (210) surfaces at ambient conditions with GIXRD	Experiment number: MA-2246
Beamline: BM25	Date of experiment: from: 21.01.2015 to: 27.01.2015	Date of report:
Shifts: 18	Local contact(s): German R. Castro	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Andreas Bürger, Ruhr-Universität Bochum*; Hermann Gies, Ruhr-Universität Bochum*; Xavier Torrelles, Laboratory Institut de Ciencia de Materials de Barcelona ICMAB*		

Report:

Anatase is one of the three crystalline TiO₂ modifications (rutile, anatase and brookite) which are in the focus of recent research, due to the widely used technological applications. The experiment was motivated by the importance of the interface between the different TiO₂ surfaces with liquid water in relation to their use as photo catalysts for water splitting. We proposed to measure the (001), (101) and (210, brookite) surfaces or the (001) and (101) surfaces at ID03 or BM25B, respectively. All surfaces should be measured in dry and water covered conditions. This set-up allows correlating the structure of the dry surfaces with the structural interface in wet environment conditions.

Rutile surfaces have been studied most intensively, compared to anatase and brookite, due to the easy access to large rutile single crystals [1]. The fewer studies of anatase surfaces deal with cleaved, cut, sputtered or etched surfaces [2] or with anatase nanocrystals or anatase powder [3]. Large natural single crystals with adequate surface quality are difficult to get. We possess a natural, large anatase single crystal from Hardangervidda, Norway, with the typical tetragonal bipyramidal shape without (001) surfaces on top and with a flat (101) surface of about 20 mm x 10 mm in size. A second anatase sample is a small tetragonal bipyramidally shaped single crystal from Baluchistan, Pakistan, with a (001) surface of 3 mm x 2 mm in size. During the beamtime we were able to measure a large dataset of CTRs and Lscans for both surfaces in dry and water covered conditions. The dataset for the dry (101) anatase surface contains 9 CTRs and the dataset for the water covered surface contains 18 CTRs. The

dataset for the dry (001) anatase surface contains 20 CTRs and the dataset for the water covered surface contains 15 CTRs. From this dataset, it will be possible to determine the relaxation and the resulting structure of the (001) and (101) single crystal samples very accurately. Especially the influence of water on the relaxation of both surfaces should be able to investigate, as been shown for different systems before [4, 5].

Due to strong problems with the beam during the beamtime we were not able to measure all proposed systems. Unfortunately we failed to measure the natural brookite single crystal (210) surface for both conditions.

Altogether we were able to measure the anatase (001) as well as the anatase (101) surfaces of the natural single crystals in dry and water covered conditions. First refinement of the datasets shows the differences of the surfaces under dry and wet conditions.

References:

- [1] Diebold, U., Surf. Sci. Rep. **2003**, 48, 53-229
- [2] Aschauer, U., He, Y., Cheng, H., Li, S., Diebold, U., Selloni, A., J. Phys. Chem. C **2010**, 114, 1278-1284
- [3] Deiana, C., Fois, E., Coluccia, S., Martra G., J. Phys. Chem. C **2010**, 114, 21531-21538
- [4] Magdans, U., Torrelles X., Angermund, K., Gies, H., Rius, J., Langmuir **2007**, 23, 4999-5004
- [5] Pareek, A., Torrelles, X., Angermund, K., Rius, J., Magdans, U., Gies, H., Langmuir **2008**, 24, 2459-2464