EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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 via the User Portal:

https://wwws.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.

ESRF	**Experiment title:** Photo-Electro-Chemistry of Anatase TiO_2 (101): Understanding the water splitting reaction	**Experiment number**: CH 4767
Beamline: ID03	**Date of experiment**: from: 01/02/17 to: 07/02/17	**Date of report**: 06/03/17
Shifts: 18	**Local contact(s)**: Francesco Carla	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Prof. Geoff Thornton (University College London)

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Report:

Our original experiment consisted of photo-electro-chemical surface science of anatase TiO_2 (101) where we planned to prepare anatase TiO_2 (101) samples in utra-high vacuum (UHV) and use our custom built photo-electro-chemical droplet cell for the photo-electro-chemical measurements. At ID03, it was possible to prepare our samples in UHV, however, the ID03 UHV set up was not compatible with our droplet cell set up rendering it impossible to transfer our sample from the ID03 UHV set up to our droplet cell under UHV conditions. For this purpose, we changed our sample from UHV prepared anatase TiO_2 (101) to 'in-air' prepared Nb-doped rutile TiO_2 (110). Additionally, we encountered shipping problems from the UK to France which resulted in our droplet cell being misplaced by the shipping company - our cell arrived mid-way through our beamtime. Due to this, we decided to use an electrochemical Myler cell (at ID03) for our experiment to allow us to take electro-chemical measurements of the Nb-doped rutile TiO_2 (110) surface.

We used ID03's Myler cell to understand the Nb-doped rutile TiO_2 (110) interface with 0.1 M NaOH and the evolution of the surface under electrochemical control. During our experiment, we encountered a number of problems with the Myler cell. This was the first time that our group used the Myler cell and we needed to explore the different geometries (thick film, mid-film, thin film etc) in which to acquire the data. During our initial attempts at using the cell we encoutered a number of problems such as 'liquid leaks' in the cell and sample stage movement which we negotiated and optimised. The Nb-doped rutile TiO_2 (110) crystals were prepared in the UK and bought to the ESRF, the surface quality was checked with LEED and the surfaces were UV/Ozone treated before the experiment to remove adventigious carbon. We firstly measured our Nb-doped rutile TiO_2 (110) in an N_2 environment. We were able to collect 8 crystal truncation rods (CTRs) of the surface under an N_2 environment. This data is currerntly in the process of being analysed.

Subsequently, we introduced 0.1 M NaOH electrolyte into the Myler cell to probe the Nb-doped rutile TiO_2 (110) and 0.1 M NaOH interface. Initally we set up the Myler cell such that the surface had ~2 mm (thick film geometry) of electrolyte over it (i.e surface to Myler distance was ~2 mm). Under these conditions, the back ground (associated with the electrolyte) was masking the CTR intensity making it difficult to obtain the necessary results. We subsequenlty kept reducing the surface to Myler distance until the Myler film was lying on top of the sample (thin film geomtery) with the Myler to sample distance expected to be on the μm scale. Under these conditions we collected 5 CTRs. However, during the 'thin-film' geometry measurements the electrolyte associated background continued to mask the CTR intensity. Our attempts at electrochemistry were laregly hampred by beam loss and electrolyte associated background staurating the detectors.