EUROPEAN **S**YNCHROTRON **R**ADIATION **F**ACILITY

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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:** In-situ X-ray Scattering Studies of Ordered Oxides and CO on Pt(*hkl*) Electrodes Modified by Irreversibly-Adsorbed Metal Monolayers	**Experiment number**: SI-596
Beamline: ID03	**Date of experiment**: from: 12/11/00 to: 20/11/00	**Date of report**: 1/9/01
Shifts: 21	**Local contact(s)**: Paul Steadman	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Christopher Lucas* University of Liverpool

Nenad M Markovic* Lawrence Berkeley National Laboratory

Bridget Murphy* Daresbury Laboratory

Report:

Abstract from 2 papers submitted so far from this work. Another paper is in preparation.

Title: X-ray Scattering Studies of Irreversibly Adsorbed Bismuth on the Pt(111) Electrode Surface

Authors: M. Ball[1], C. A. Lucas[1,*], N. M. Markovic[2], B. M. Murphy[3], P. Steadman[4], T. J. Schmidt[2], V. Stamenkovic[2] and P. N. Ross[2]

Affiliations: [1] Oliver Lodge Laboratory, Department of Physics, University of Liverpool, Liverpool, L69 7ZE, UK. [2] Materials Science Division, Lawrence Berkeley National Laboratory, University of California, Berkeley, CA 94720, USA. [3] Institut für Experimentelle und Angewandte Physik, Christian-Albrechts-Universität Kiel, Olshausenstr. 40, 24098 Kiel, Germany. [4] ESRF, BP-220, 38043 Grenoble cedex, France.

Journal: Langmuir, in press (scheduled for October 2001)

Abstract text:

The surface electrochemistry of irreversibly adsorbed bismuth (Bi_{ir}) on the Pt(111) electrode in 0.1 M $HClO_4$ has been studied by *in-situ* surface x-ray scattering (SXS), in order to correlate the atomic structure with the voltammetric features. Bi_{ir} saturates at a coverage of ~1/3 of a monolayer and forms a disordered structure on the Pt(111) surface with some evidence that the Bi_{ir} is mobile. If the Bi_{ir} coverage is slightly increased, then there is a

reduction in the charge under the reversible peaks in the cyclic voltammetry (CV) at a potential of ~0.68 V (vs. Pd/H_2). The charge under the reversible peaks is most likely associated with the adsorption of oxygenated species at platinum sites adjacent to Bi_{ir} atoms. Saturation of the electrolyte with CO and subsequent potential cycling leads to displacement of Bi_{ir} from the surface and the appearance of an x-ray diffraction pattern due to a p(2x2)-3CO adlayer which has previously been observed on the unmodified Pt(111) electrode.

Title: Surface Relaxation at the Metal/electrolyte Interface

Author: C. A. Lucas

Affiliation: Oliver Lodge Laboratory, Department of Physics, University of Liverpool, Liverpool, L69 7ZE, UK.

Journal: Electrochimica Acta (special issue concerning x-ray studies of electrochemistry), submitted in July 2001.

Abstract text:

X-ray diffraction is an ideal technique for the *in-situ* study of single crystal metal surfaces in an electrochemical environment. In this paper, measurements of the low-index surfaces of Au and Pt are described, in particular with reference to surface expansion effects. Surface expansion can be probed potentiodynamically to correlate expansion with the adsorption/desorption of solution species. In general, the results are in good agreement with recent theoretical calculations. The x-ray technique can also give insight into electrocatalytic reactions as shown by the results for the adsorption and oxidation of CO on Pt(111).