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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:** Structural characterisation of surface modified materials by using REFLEXAFS (II): Cu/Cr multilayers	**Experiment number**: ME-737
Beamline: BM29	**Date of experiment**: *(Long term project)* from: Dec-2003 to: Jun-2005	**Date of report**: Feb-2007
Shifts: 72	**Local contact(s)**: Silvia Ramos; Gianluca Ciatto	Received at ESRF:

Names and affiliations of applicants (* indicates experimentalists):

Adela Muñoz Páez; Víctor López-Flores *Institute of Materials Science of Seville.*

Stuart Ansell; Daniel T. Bowron *ISIS, Rutherford Appleton Laboratory.*

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Report:

To develop, test, optimize and finally obtain the first analyzable ReflEXAFS spectra at BM29, a set of simple samples was prepared and measured at BM29 beamline. Multilayers of metallic Cu and Cr over Si (100) wafers were grown by DC Magnetron Sputtering Physical Vapor Deposition. The samples are made of a periodic (CuCr)xN structure, where N = 2, 4 and 8, being the period a Cu/Cr bilayer. Deposition time was controlled in order to keep the total thickness around 200 Å, as well as to keep the Cu:Cr thicknesses ratio equal to 2:1.

Prior to the ReflEXAFS measurements, sample layers composition and absolute atomic concentration were measured by Rutherford Backscattering Spectrometry (RBS). Layers thicknesses and roughnesses were measured by X-ray reflectometry (XRR). Surface roughnesses were measured by Atomic Force Microscopy (AFM). A micrograph of the sample CuCrx8 surface is shown in Fig. 1b.

In the standard procedure followed to perform the ReflEXAFS measurements, the sample and the reflectometer has to be accurately aligned in a first instance, using the protocol described in [1].

Then, a XRR spectrum at low angles (from 0 to 1.5 deg) was recorded in order to choose the best angles for ReflEXAFS spectra. These are the ones that allow different penetration depths while maintaining a high reflectivity due to the interference effects that these layered samples show. Once the angles were chosen, an EXAFS-like energy scan was recorded.

The samples were measured by ReflEXAFS at angles selected, at the Cu-K absorption edge. The

spectra for all the angles for CuCrx4 sample are shown in Fig. 1a. Both the background with the typical interference pattern due to the multilayers reflection and the EXAFS fine structure can be seen.

The lowest workable angle spectrum for each sample were analyzed using the total reflection approximation[2], and the FEFF 6 and FEFFIT 2.54 codes. The Fourier transform of the spectra for sample CuCrx2 with its fit is shown in Fig. 1c.

The results show that the Cu metal structure is maintained. However, the high Debye-Waller factors show that there is an increasing structural disorder for decreasing thicknesses of the samples. The AFM images and the Fourier transform of the EXAFS spectra also show that some surface oxidation has taken place in the case of sample CuCrx8.

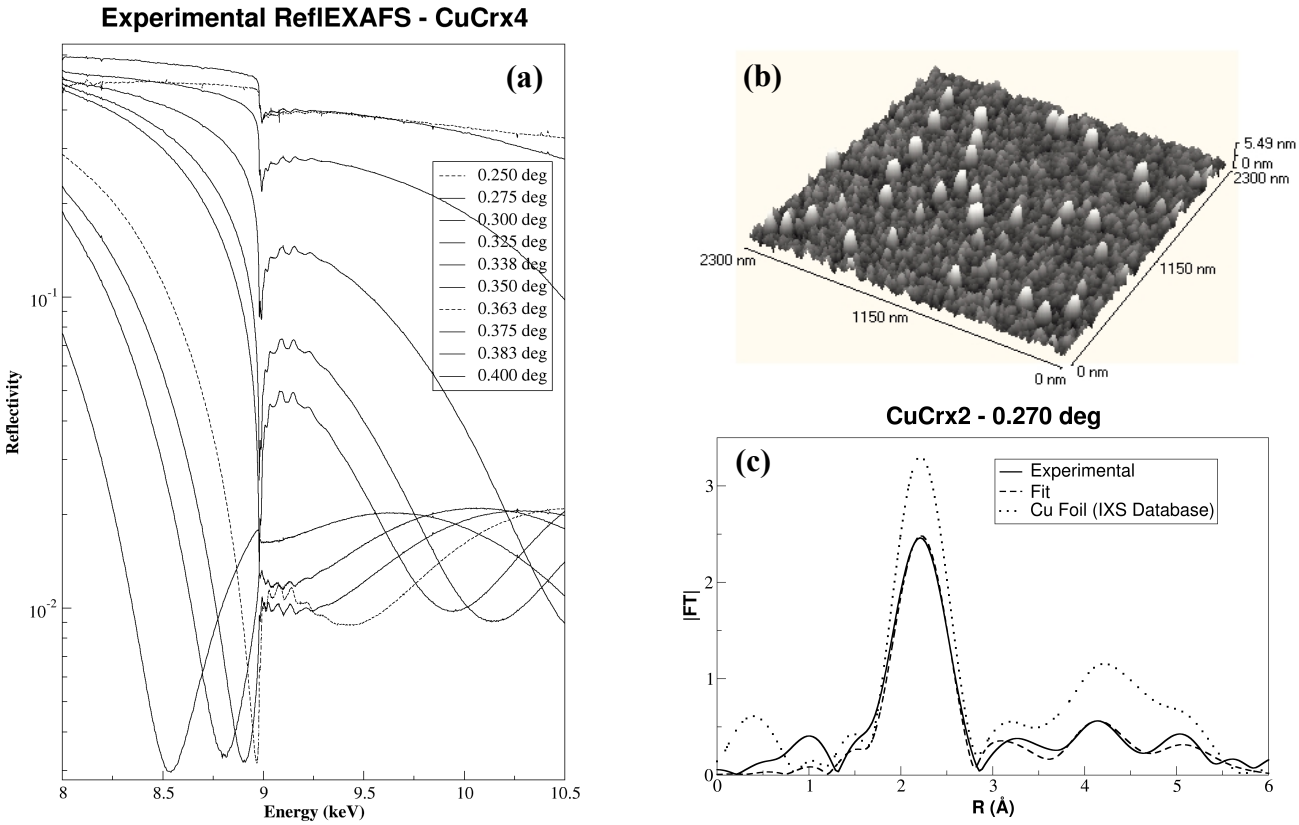

Fig. 1: *(a)* Experimental ReflEXAFS spectra for sample CuCrx4 for all the incidence angles. *(b)* AFM image of sample CuCrx8 surface. *(c)* Fourier transform of the EXAFS spectrum recorded at 0.275 deg, and best fit.

[1] V. López-Flores, S. Ansell, D. T. Bowron, S. Díaz-Moreno, S. Ramos, and A. Muñoz-Páez. *Rev. Sci. Instrum.* ***78****, 013109 (2007)*

[2] G. Martens and P. Rabe. *J. Phys. C: Solid St. Phys* ***14****, 1523 (1981)*