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 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:** Grazing incidence small angle X-ray scattering from heterogeneous alloy films prepared by a novel pulse electrodeposition method	**Experiment number**: HS-2343
Beamline: ID-01	**Date of experiment**: from: 18/05/2004 08:00 to: 22/05/2004 08:00	**Date of report**: 01/03/05
Shifts: 12	**Local contact(s)**: Dr Bruno Jean	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr Walther Schwarzacher*

H. H. Wills Physics Laboratory, Tyndall Avenue, Bristol BS8 1TL, U.K.

Dragomir Tatchev*

Institute of Physical Chemistry, Bulgarian Academy of Sciences, 1113 Sofia, Bulgaria

Report:

The aim of this experiment was to use grazing incidence small-angle anomalous X-ray scattering (GISAXS) to study the heterogeneity of electrodeposited Cu-Ni alloy films prepared by alternating between two deposition potentials. At the more positive potential only Cu was deposited, while at the more negative potential mostly Ni was deposited. The quantity of Cu and Ni deposited per cycle was varied from less than a monolayer up to several monolayers, so that the transition between heterogeneous alloy and conventional multilayer could be observed. Automatic compensation for e.g. Ni dissolution was made during growth. Samples were characterized magnetically and by scanning force microscopy prior to the ESRF experiment.

This experiment was the first with samples of this kind, and a major objective was to establish what information could be obtained from GISAXS measurements of the alloys with the smallest quantities of Cu and Ni deposited per cycle. Magnetic data for similar samples suggested that they would consist of disk-like Ni-rich clusters in a Cu or Cu-rich matrix. Although we lost some experimental time due to loss of beam following a thunder storm and problems at the station, we were able to study several samples at different energies close to the Ni absorption edge, at different sample-detector working distances and for a variety of angles of incidence. The quality of the data was high, with data (corrected for absorption and refraction effects) taken at different angles of incidence superposing quite well when plotted as a function of scattering vector q. Plotting $\ln (q^2 I(q))$, where I is the scattered intensity, against q^2 gave a reasonably good straight line, as expected if the films may be approximated as an array of disks - see Figure 1. However, the fitted disk thickness did not depend on the amount of Cu and Ni deposited per cycle, which was unexpected, and at present we are still unsure as to the significance of this result. There was also no evidence for any in-plane anisotropy in the small angle-scattering.

From the GISAXS data of the films in which several monolayers of Cu and Ni were deposited per cycle we were able to identify Bragg peaks, enabling us to measure the films' superlattice repeat distance - see Figure 2. This was a very useful result, which we expect to publish at some point.

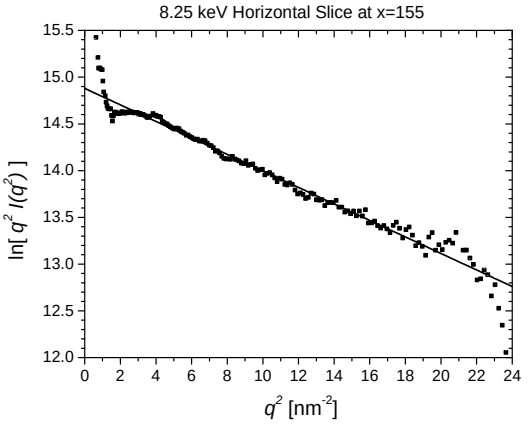

Figure 1: Guinier-type plot for electrodeposited Cu-Ni alloy. 2400 cycles, each consisting of the deposition of 1Å (equivalent thickness) Cu and 1Å Ni.

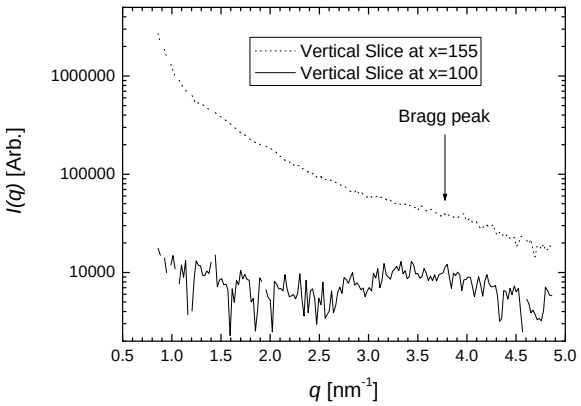

Figure 2: *I*(*q*) data for electrodeposited Cu-Ni alloy. 300 cycles, each consisting of the deposition of 8Å (equivalent thickness) Cu, and 8Å Ni. Bragg peak gives repeat distance as ~ 19Å.