XMaS the UK-CRG	**Experiment title:** Surface x-ray diffraction studies of Au electrodes: the electrodeposition of Nickel	**Experiment number:** 28-01-42
Beamline: **BM 28**	**Date of experiment:** from: 24 / 11 / 99 to: 30 / 11 / 99	**Date of report:** April 2000
Shifts: 18	**Local contact(s):** Dr. Simon Brown	*Received at XMaS:*

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Report: Introduction

In previous experiments on the Ni / Au (111) system, X-ray scattering features were observed which corresponded to the $22 \times \sqrt{3}$ "herringbone" surface reconstruction of Au. Following electrochemical deposition of Ni, changing the energy of the incident X-rays revealed that there was a step-change in the intensity of the feature at the Ni K-edge (at 8.36 kV). This result showed that some Ni is included within the $22 \times \sqrt{3}$ reconstruction on initial deposition. It is the detail of this structure which we have addressed in the current experiments.

Experimental Method

The experimental methods involved in these measurements are described in detail in ref. [1] and in the original application for beamtime [proposal number 28-01-17]. The Au(111) surface was prepared by flame annealing and placed into an electrochemical cell. The cell was then covered with a polypropylene window held in place with a rubber o-ring, and filled with modified Watt's electrolyte (containing H_3BO_3, HCl, and $NiSO_4$). The polypropylene film could be deflated using a syringe attached to the liquid feedthroughs. Deflating the film traps a thin layer of electrolyte on the crystal surface, and allows X-ray measurements to be made.

Results

Figure 1 shows the energy dependence of the (0 0 1.8) peak, which is sensitive to the $22 \times \sqrt{3}$ surface reconstruction on Au (111). Following electrochemical deposition of submonolayer quantities of Ni, the increase in intensity of this peak at the energy of the Ni K-edge confirms that Ni adopts the same lattice, forming an ordered surface alloy. Further deposition of Ni resulted in the observation of bulk Ni features due to a Ni overlayer. Even with a Ni overlayer, the reconstruction features persisted, revealing that the ordered AuNi alloy is a stable and abrupt interface.

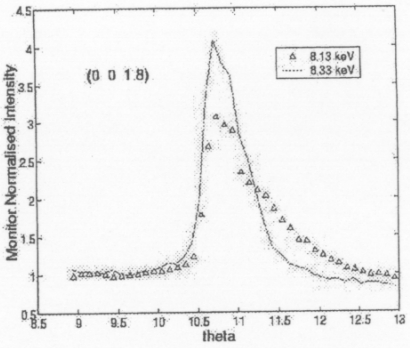

Fig. 1

Comparison of the x-ray signal at (0 0 1.8), which is sensitive to the $22 \times \sqrt{3}$: Ni reconstruction, both close to and far from the Ni K-edge

At submonolayer coverages of Ni on the surface, in the presence of the reconstruction, the (0 0 L), (0 1 L) and (1 0 L) crystal truncation rods were measured using photon energies both close to, and far from, the Ni K-edge. The (1 0 L) is shown in figure 2, which displays a large difference in intensity in the region of the anti-Bragg position between the data sets measured at the two energies.

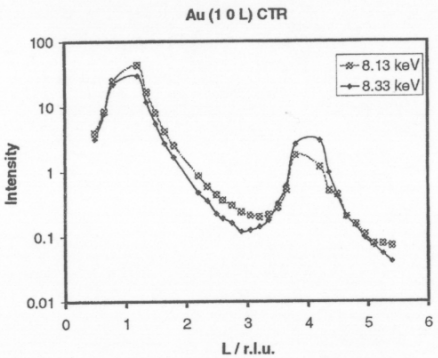

Fig. 2

Comparison of Au (1 0 L) CTRs from the $22 \times \sqrt{3}$: Ni reconstruction both close to and far from the Ni K-edge

Detailed analysis of these data will reveal the extent of distortion to the reconstructed surface brought about by the substitution of Ni into the structure. The transition from submonolayer inclusion to the development of a Au/Ni interface will also be addressed by the data collected from greater Ni deposition.

References

[1] I. M. Tidswell, N. M. Marcovic, C.A. Lucas and P. N. Ross, Physical Review B, **47**, *16542* (1993).