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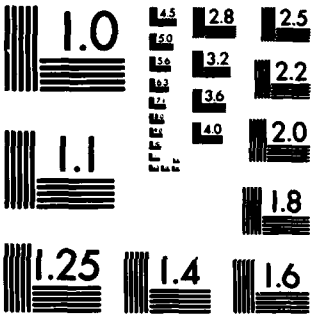

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During our latest run, November '83, at the Stanford Synchrotron Radiation Laboratory, we were able to detect x-ray fluorescence from a monolayer of Pb on single crystal silver film. The jump at the Pb edge represented increase in count rate of 150 eps on a 1500 cps background. This signal is too small for useful EXAFS to be extracted with a reasonable data collection time, but planned improvements in the electronic instrumentation and the experimental setup will achieve an edge jump greater than 1000 cps on a comparable background. Original Contains Color Plates

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TECHNICAL REPORT NO. 16

EXAFS OF MONOLAYERS ON SILVER ELECTRODES
1984 Progress Report for SSRL Proposal No. 717M

by

D. E. Reisner, O. R. Melroy, J. G. Gordon II,
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Chemistry

EXAFS OF MONOLAYERS ON SILVER ELECTRODES
1984 Progress Report for SSRL proposal No 717M

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Abstract:

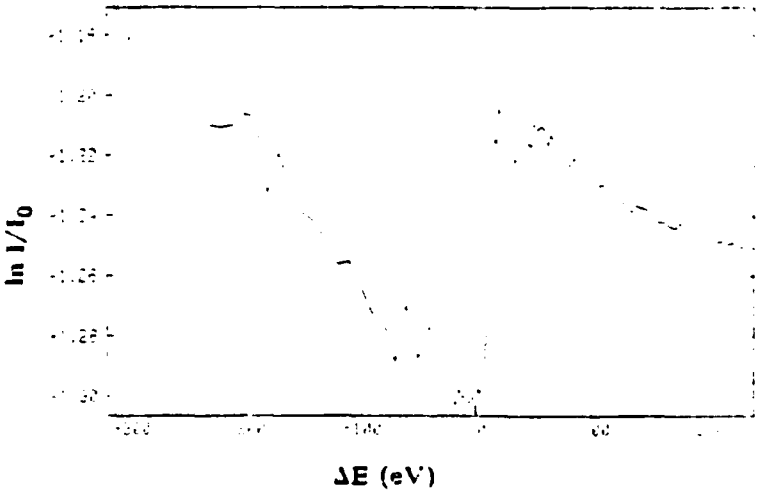
During our latest run, November '83, at the Stanford Synchrotron Radiation Laboratory, we were able to detect x-ray fluorescence from a monolayer of Pb on single crystal silver film. The jump at the Pb edge represented increase in count rate of 150 cps on a 1500 cps background. This signal is too small for useful EXAFS to be extracted with a reasonable data collection time, but planned improvements in the electronic instrumentation and the experimental setup will achieve an edge jump greater than 1000 cps on a comparable background.

Our current knowledge of the electrochemical interface is derived primarily from thermodynamic and kinetic measurements coupled with theoretical treatments based on highly idealized models which describe only the diffuse double layer, or space charge region, within 20-100 Å of the electrode surface. These types of measurements do not yield unambiguous information about the structure of the interface at the atomic or molecular level. This situation is not attributable to lack of interest but rather to the lack of structure sensitive techniques which can be used in solution.

Surface EXAFS is potentially such a technique since X-rays are able to penetrate condensed matter. Utilizing the intense radiation available from a synchrotron and fluorescence detection, it should be possible to observe EXAFS of a monolayer of atoms at a metal:liquid interface, without having to remove the electrode from solution.

As a demonstration experiment, we have chosen to study a monatomic layer of lead, prepared by electrochemical underpotential deposition (UPD), on silver. Electrochemical deposition of lead on silver occurs in stages. The first monolayer is deposited at a potential which is less negative (by several hundred mV - the underpotential) than the expected thermodynamic deposition potential and the lead coverage remains constant at one monolayer over a relatively wide potential range.

During our last run, November '83, we were able to detect fluorescence from a monolayer of Pb on single crystal silver film, but at a signal level too weak to permit meaningful analysis. The observed spectrum, normalized to incident x-ray flux is displayed in the Figure. This spectrum is a composite of 15 scans of 30 min duration each (45 sec/point). The edge jump corresponds to an increase in count rate of 150 cps on a 1500 cps background.



The substrate was a free standing 2 μm Ag(111) film prepared by epitaxial deposition on mica. The fluorescence data was collected using a 2" diameter NaI(Tl) scintillator preceded by an Se filter and Soller slits, and operated with a very narrow discriminator window. Despite its poor energy resolution, its large active area and high count rate

capability provided a better S/N than a smaller HPGe detector whose throughput was limited by the detection electronics (the total count rate, pre-discriminator, was > 100 Kcps).

Measurements made with the energy dispersive HPGe detector verified that the major contributions to background were elastic and Compton scatter from the substrate followed by impurity fluorescence and Se fluorescence from the filter. Planned improvements in the electronic instrumentation and the experimental setup, we believe, will achieve an edge jump of > 1000 cps on a comparable background.

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