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ELECTRON YIELD DETECTORS FOR NEAR SURFACE EXAFS AT ATMOSPHERIC PRESSURE

K.I. PANDYA, K. YANG, R.W. HOFFMAN, W.E. O'GRADY* and D.E. SAYERS**

Department of Physics, Case Western Reserve University, Cleveland, OH 44106, U.S.A.
Brookhaven National Laboratory, Upton L.I., NY 11973, U.S.A.
Department of Physics, North Carolina State University, Raleigh, NC 27650, U.S.A.

Abstract - Simple He-filled electron detectors have been developed by Kordesch and Hoffman as a method for near-surface EXAFS investigation using a reflection geometry. We report our experience on beam line X11 at NSLS in Brookhaven National Laboratory for the gold L and nickel K edges. Thin foils were mounted such that the transmission and electron yield data could be obtained simultaneously. The X11 monochromator was operated in the two-crystal mode and a series of scans was taken primarily as a function of helium flow rate, and to a lesser extent, the positive voltage applied to the electron collecting gold wires. Minor changes were made in the geometry of the electron detector. The electron detector output was connected directly to a Keithley Model 427 current amplifier. Preliminary data analysis in the form of background subtraction and transformation to k (the wave vector) was done on-line.

Typical data in the form of background subtracted relative yield vs. k for both transmission and electron detection indicate agreement within the linewidth of the plots. We anticipate that the interatomic spacing and coordination number determined from both the techniques will agree well. Electron-yield EXAFS data were successfully obtained from a Ni surface covered with a thin layer of H₂O. This illustrates the potential of these detectors to study in-situ wet electro-chemistry. A sharp white line was observed in both transmission and electron-yield EXAFS for Ni(OH)₂, and in electron yield EXAFS for NiO.

INTRODUCTION

Electron-yield EXAFS (Extended X-Ray Absorption Fine Structure) technique is a useful tool in understanding the structure of the thin films and the interactions taking place at surfaces[1,2]. A simple helium filled electron detector has been developed as a method for near-surface EXAFS measurements[3]. Our electron detector is an ionization chamber filled with helium at atmospheric pressure. The sample is mounted inside the detector the same way as in CEMS (Conversion Electron Mossbauer Spectroscopy). The electron yield due to the relaxation of the core hole is measured as a function of the incoming photon energy. Electron yield EXAFS are inherently surface sensitive since the electrons can only escape from near-surface region of the sample.

A detailed comparison of transmission EXAFS and reflection electron yield EXAFS has been done by Guo and denBoer [4]. A significant point in their study in the case of a nickel foil was that, for photon energies just above the edge, the electron detected amplitudes were considerably smaller than the transmission amplitudes.
This led to less than satisfactory agreement between the amplitudes and hence coordination number, although yielding excellent agreement in bond length (\(<0.005\ \text{Å}\) difference) between the two techniques.

We report our experience with gold L\(_2\) and nickel K edges. These data were obtained with the goal of optimizing the operating conditions of the electron detector and to test its application in an aqueous environment.

**EXPERIMENTAL**

A 17.8 \(\mu\text{m}\) thick gold foil and a 3.8 \(\mu\text{m}\) thick nickel foil were used for the EXAFS measurement. The samples were mounted such that the transmission photon and reflection electron yield data could be obtained simultaneously. Detailed description of the electron detector can be found elsewhere [3]. The photon beam was incident perpendicular to the surface of the sample. The electron collecting gold wires were at +67 Volts with respect to the aluminum foil electrode. The electron detector output was then amplified by a Keithley 427 current amplifier. Typical electron currents were in nanoampere range and increased by about a factor of 5 above the edge.

Scans were taken as a function of i) the He flow rate, ii) positive voltage applied to the gold wires, iii) the distance between the sample and the gold wires. Electron-yield EXAFS data were also obtained for NiO and Ni(OH)\(_2\). Preliminary data analysis in the form of background subtraction and transformation to \(k\) (the wave vector) was done on-line.

We used a rotating cell geometry [5] to obtain electron-yield EXAFS data from a nickel surface covered with a thin layer of water. The sample was a 45 mm diameter and .5 mm thick nickel disk which was mechanically polished. The bottom half of the cell was filled with water and an electron detector was mounted on the open top of the cell. The nickel disk made about 9 rotations during the 12 minutes of data collection.

One of the detectors was modified by inserting a copper grid (100 lines/inch, 80% transmission) between the sample and the gold wires. By adjusting the potential of the grid, the low energy electrons contribution to the signal could be enhanced or suppressed. By monitoring the signal while varying the grid potential, a fair idea about the energy distribution of the electrons contributing to the signal, and hence the depth probed, may be gotten. We used a 3.8 \(\mu\text{m}\) thick nickel foil for this experiment. Voltage of the grid was varied from -50 V to +50 V with respect to the aluminum foil.

These data were obtained at the beam line X-11A of the National Synchrotron Light Source (NSLS) in the Brookhaven National Laboratory. Two Si(111) crystals were used for monochromatization.

**RESULTS AND DISCUSSION**

In fig.1, typical transmission and electron-yield data of Ni foil in the form of background subtracted relative yield \(k^2\times\chi(k)\) vs. \(k\) and the Fourier transform of \(k^2\times\chi(k)\) as a function of \(r\) (interatomic distance) are shown. Unlike the experience of Guo and denBoer for nickel, electron detected amplitudes just above the edge are not reduced. It is clear that the signal to noise is sufficient to obtain usable data from a single scan covering the range in \(k\) from 2 to 16 inverse angstroms.

Fig.2 (a) shows transmission and electron-yield data of Au foil in the form of \(k^2\times\chi(k)\) vs. \(k\). Fig.2(b) shows electron yield EXAFS data of NiO. A sharp white line is observed for NiO and Ni(OH)\(_2\).

As we increased the He flow to the detector, signal to noise improved initially. The optimal flow rate was found to be ~1 cm\(^3\)/sec. No significant change in the quality of the data was observed when the distance between the sample and the gold wires was varied from 3 to 10mm. The electron-yield signal showed a gradual saturation for voltages applied to the gold wires in excess of 50 volts.

Fig.3 shows electron-yield data of the rotating nickel disk in the dry and wet state. It can be seen that the presence of moisture...
Fig. 1. (a) Transmission and, (b) electron-yield EXAFS data of nickel. (c) and (d) corresponding magnitude of Fourier transforms.

Fig. 2 (a) Transmission and electron-yield EXAFS data of gold. (b) Electron-yield EXAFS data of NiO.
Fig. 3. Electron-yield EXAFS on the rotating nickel disk (a) without and (b) with water in the cell.

in the detector and a thin liquid layer on the nickel surface does not interfere with the electron-yield EXAFS measurements.

Electron-yield data obtained with the grid are in general noisier than the data obtained without the grid. This may be related to the new configuration of the detector. The best spectrum was obtained at the grid voltage +20 Volts. Further experimentation will characterize the energy distribution in more detail and give an idea about the surface sensitivity.

Conclusions

It is encouraging to us that electron-yield data obtained with our detector are in agreement with transmission data. These electron detectors offer near surface sensitivity without requiring ultrahigh vacuum (UHV) and can be used to examine wet and thick samples.

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