

RO1A38B1-0008N

BL38B1

RO1A38B1-0009N

BL38B1

Total Reflection XAFS at Aqueous Solution Surface by Fluorescence Technique

Hajime Tanida (1275), Tomoya Uruga (182)
Materials Science Division,
Japan Synchrotron Radiation Research Institute,
1-1-1 Kouto, Mikazuki, Sayo, Hyogo 679-5198, Japan

1. Introduction

Total Reflection X-ray absorption spectroscopy is a good tool for air/aqueous solution surface. In the previous study, the absorption has been detected by total conversion He ion-yield method under helium gas. The total conversion yield method has several advantages over the fluorescence method; ultimately simple design of only a collecting electrode, its wide detection area, nearly 100 % charge collection efficiency, and two orders of gas amplification to the signal current owing to the high energy of Auger electron. However, the detection limit of the total ion yield method is much larger than that of the fluorescence one. Therefore, the fluorescence technique is needed to be developed because of the study on diluted samples.

2. Experimental

The sample solution was prepared from stearyltrimethylammonium bromide (STAB). The spectra were obtained with the use of a Si (111) double crystal monochromator, the first Rh-coated mirror at the grazing incidence angle of 3.0 mrad, and the second Si mirror at 2.5 mrad. Therefore the incident angle to the solution surface is 1 mrad. The I_0 chamber was 17 cm long filled with N_2 , and the fluorescence ionization chamber, so-called Lytle detector, as I chamber was filled with Ar. The remodeling cell cover has the two kapton windows on the top and by the side. The Lytle detector obtains the fluorescence from the solution surface through the windows. The solution is at room temperature ($25 \pm 3^\circ\text{C}$).

3. Results and Discussion

Fig. 1 shows the Multi Channel Analysis (MCA) spectra of fluorescence, which are

obtained with a silicon drift detector (SDD). The signal intensity with the vertical detection is 4 times as large as that with horizontal one, while the ratio of intensity between the signal and the scattering with the former is worse than that with the latter. Fig. 2 shows x-ray absorption spectra with the Lytle detector. The spectrum with the vertical detection shows more clearly than that with the horizontal.

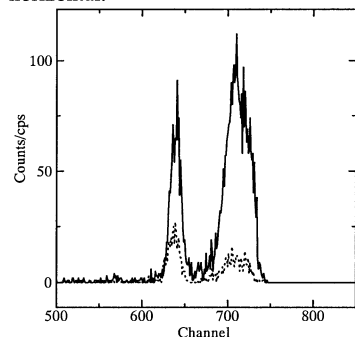


Fig. 1. MCA spectra with the vertical (solid line) and horizontal (dashed line) detections of fluorescence from Br in 0.1 mM STAB aqueous solution with SDD.

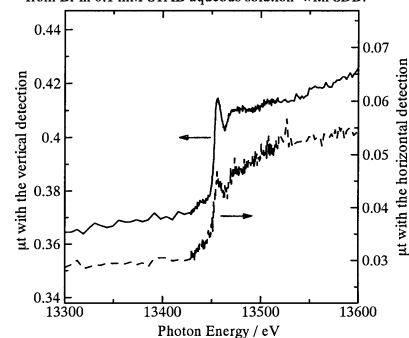


Fig. 2 XAFS spectra with the vertical (solid line) and horizontal (dashed line) detections of fluorescence from Br in 0.1 mM STAB aqueous solution with SDD.

Development of Quick Scan XAFS

Hajime Tanida (1275), Shigeru Yokota (1451)

Japan Synchrotron Radiation Research Institute,
1-1-1 Kouto, Mikazuki, Sayo, Hyogo 679-5198, Japan

1. Introduction

Time-resolved XAFS technique is a good tool for a study on a chemical reaction. It is known to be mainly two methods, one is a quick scan method with a monochromatized x-ray by scanning a monochromator rapidly, another is an energy dispersive method with a white x-ray, a polychromator after the sample, and a position sensitive detector. The quick scan technique has several advantages over the energy dispersive method; the incident x-ray intensity can be monitored simultaneously with the absorbance of the sample, several detection modes can be used, for example, a fluorescence mode for a dilute sample. In this study, the quick scan method is tried.

2. Experimental

The SPring-8 standard monochromator was used at BL38B1. Usually, the θ axis is moved simultaneously with Y_1 axis leading to the fixed exit of the x-ray beam. In this study, the Y_1 position is fixed at the middle of the scan range in the XAFS spectrum. The drift of the beam position with the fixed Y_1 did not affect to measure the XAFS spectrum for the large size of the sample. The θ was moving from the start angle to the end in the scan region, while the I_0 and I signals were collected continuously.

The spectra were obtained with the use of a Si (111) double crystal monochromator, a Rh-coated mirror at the grazing incidence angle of 2.2 mrad. The I_0 and I chambers were 17 and 31 cm long filled with Ar. A current amplifier amplified the currents from the ion chambers, and the converted voltage signals were collected through an analog-digital converter in a personal computer. The sampling rate was 6660 Hz. The angle of monochromator moved from 4.91195

degree to 4.86545, and 2000 points were sampled in 7.277 seconds. The angle of the monochromator was monitored at the start and the end of the each scan by an encoder. Every 15 seconds, the spectrum was measured.

Samples were set in the In-situ cell and treated with 10 % H_2/He gas flow at 500 $^\circ\text{C}$. Rh K-edge XANES was measured at the same temperature and atmosphere through the quick-scan method.

3. Results and Discussion

The results were shown in Fig.1. Each point in the spectra was averaged 10 points in both sides of the point. In Fig.1, a structural change had taken place around rhodium under 10 % H_2/He gas flow at 500 $^\circ\text{C}$ for about twenty minutes. It was shown that Rh^{2+} changed to Rh metals.

EXAFS was also tried. However, it took 21.729 seconds to move the angle from 4.93595 degree to 4.78975, and the signal/noise ratio is not enough to analyze the spectra obtained.

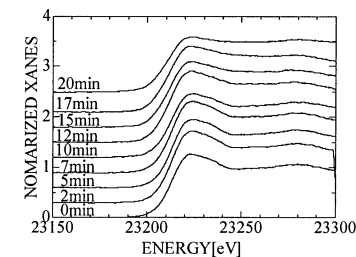


Fig. 1 Rh K-edge XANES spectra of 0.5%Rh/AI2O3 under 10% H_2/He gas flow at 500 $^\circ\text{C}$ by quick-scan method