Installation of Fluorescence X-ray Detectors for Upgrade of BL-10

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Abstract

Two kinds of fluorescence X-ray detectors were installed in BL-10; a silicon drift detector and a micro channel plate detector, and their performances were studied by measuring X-ray absorption spectra of two prototypical samples; α-Al_2O_3 powder and a thermal oxide film on Si wafer. Obtained spectra demonstrate that the fluorescence yield method gives high S/B ratio and combined use of electron yield and fluorescence yield modes provides information of the depth profile.

1. Introduction

XAFS measurements in the soft X-ray of photon energy range from about 1000 to 4000 eV have been performed at BL-10. So far, we have exclusively employed the total electron yield (TEY) mode by monitoring the sample current. However, we sometimes encountered difficulties when we tried to measure reliable spectra for insulator samples and surface contaminated samples. The former is due to charging up and the latter is due to high surface sensitivity of the electron yield (EY) mode.

Recently, we installed two kinds of fluorescence X-ray detectors; the silicon drift detector (SDD) and the micro-channel plate (MCP) detector at BL-10. In general it is known that the influence by charge up of a sample is low, and the S/B ratio of a spectrum is high in the fluorescence yield (FY) mode. Moreover the FY mode is bulk sensitive measurement than the EY mode.

In this report, we demonstrated the performances of these newly installed fluorescence X-ray detectors, and show several preliminary spectra.

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2. Experiments

This work was performed at the BL-10, which consists of a pre-focusing toroidal mirror, a Golovchenko-type double-crystal monochromator, an I_0 monitor of a tungsten mesh evaporated with copper, and a sample chamber. InSb(111) in the Si K-edge X-ray absorption near edge structure (XANES) measurement and KTP(011) in the Al K-edge XANES were used as monochromatizing crystals.

The SDD was purchased from RONTEC. Inc. (X Flash Detector, 1201 series). Active area of the SDD is 5 mm^2, covered with an 8 μm thick Be window. It is cooled down to -20°C with a Peltier device to avoid temperature noise from the field effect transistor of the preamplifier. Maximum of the count rate is about 100000 cps. The signal of the SDD detector is transferred to an amplifier (Spectroscopy Amplifier 672, EG&G ORTEC Inc.), a single channel analyzer (SCA 550A, EG&G ORTEC Inc.), and then counted with a Counter (Quad Counter/Timer 974, EG&G ORTEC Inc.). Since the active area of the detector is small, we get the detector head close to the sample position, as close as 10 mm.

Another detector consists of a chevron type micro channel plate (MCP) with a diameter of 25 mm, attached with metal anode (Photonics USA Inc.), and two grids for a retarding bias and an earth voltage, all of which are surrounded by a metal cover to avoid electric noises. By applying a retarding voltage higher than the electron kinetic energies, we can detect only fluorescence X-rays. Thus, we can use this MCP detector both for (partial) electron yield and fluorescence yield (FY) modes by changing an applying voltage to the retarding grid. Typical applied voltage of the MCP is 1.7 kV, and bias voltage is -2 kV for the FY mode. Te signal form the MCP detector is transferred to a preamplifier (System Electrometer 6514, Keithley Inc.), a V-F converter (Dual V/F Converter DS-VFC2, EG&G ORTEC Inc.), and then to the counter (Quad Counter/Timer 974, EG&G ORTEC Inc.). The MCP detector is also set close to the sample position, as close as 30 mm. The detectors and the sample holder are mounted in the sample chamber, as shown in Figs. 1 and 2.

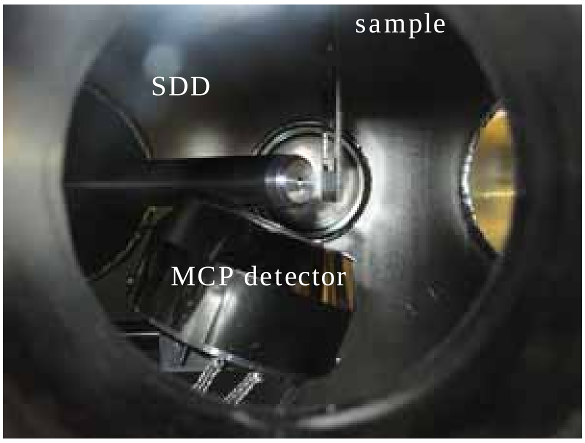

Figure 1. The photograph inside the sample chamber

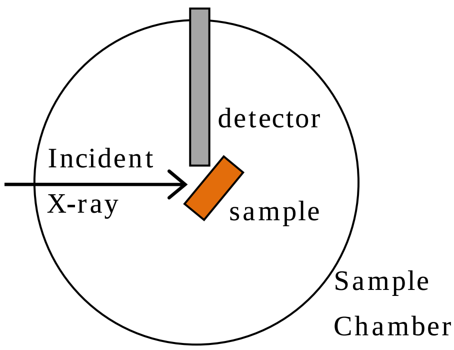

Figure 2. Top view of the schematic diagram of the sample chamber

As samples to check the performance of the detectors, we chose α-Al_2O_3 powder for Al K-edge XANES and a Si wafer with a 100 nm thick thermal oxide for Si K-edge XANES.

TEY by the sample current was also measured for comparison with the FY measurements.

3. Results and Discussion

Figure 3 shows raw Al K-edge XANES spectra (I/I_0) of α-Al_2O_3 by the TEY and the FY modes obtained simultaneously. As clearly shown, the signal to background ratio (S/B ratio) of the FY spectrum is much higher than that of the TEY spectrum: the S/B ratios at 1600 eV were 0.43 in the TEY, and 2.35 in the FY, respectively. The difference is more than 5 times. High S/B ratio is especially important for a sample with low concentration.

However, it should be noted that the use of the FY mode sometimes results in unreliable XANES spectra. Figure 4 shows the spectra which obtained by background subtraction of the spectra in Figure 3 and then normalized by the edge jump height. As shown in Fig. 4, the peak intensity is much different from each other, although the peak positions are same. The peak height at the white line (about 1569 eV) is 2.32 in the FY mode and 5.45 in the TEY mode when each edge jump is normalized to 1. Significant reduction of the FY spectrum is known to be due to the self-absorption effect, since there are considerably high amount of Al atoms in the sample. Figure 4 demonstrates that the FY mode should be carefully selected.

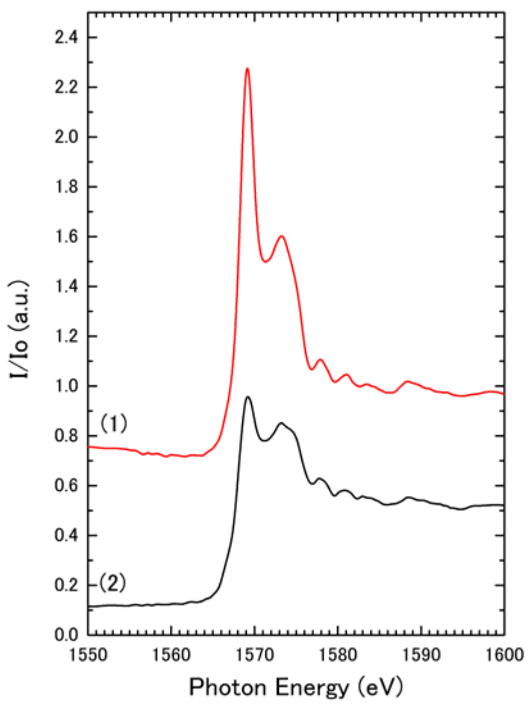

Figure 3. Raw Al K-edge XANES spectra of α-Al_2O_3. (1) TEY mode, (2) FY mode

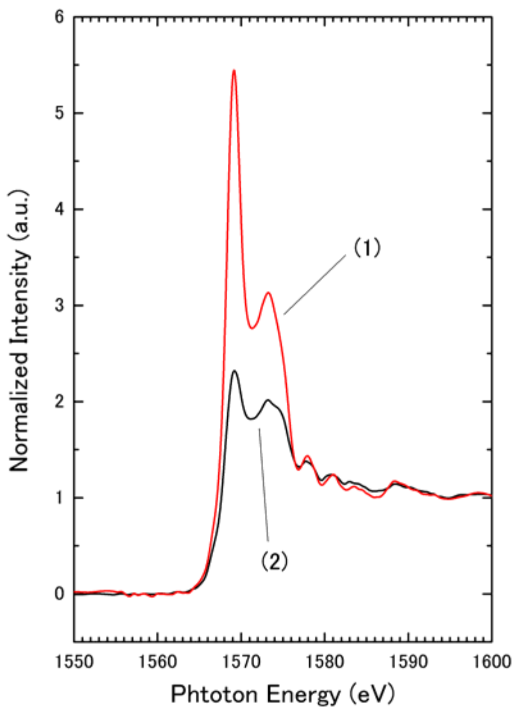

Figure 4. Normalized Al K-edge XANES Spectra of α-Al_2O_3. (1) TEY mode, (2) FY mode

Figure 5 shows Si K-edge XANES spectra with TEY (1) and FY (2) modes from a 100 nm thick thermal oxide film on a Si wafer. The MCP detector was used with -2 kV bias voltages in this FY spectrum. Even if it takes the self-absorption effect into account, shapes of these spectra are greatly different from each other, such as peak positions and peak intensities. The spectrum of (1) typically stands for SiO_2, as shown in a sharp peak at about 1847 eV and a broad peak at about 1864.5 eV. On the other hand, the spectrum of (2) is a typical one of Si wafer, as shown in the peak at about 1840 eV, derived from bulk Si and the peak at about 1847 eV derived from SiO_2 by naturally contaminated surface.

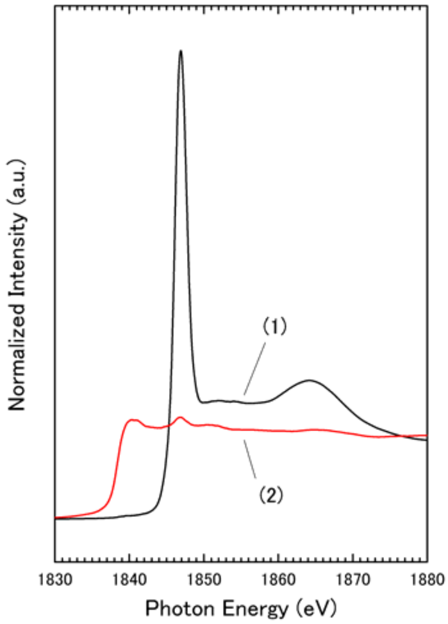

Figure 5. Normalized Si K-edge XANES spectra Of 100 nm thick thermal oxide film on a Si.

This difference mainly comes from the different probing depth between the FY and the TEY modes. The signals in Figure 5 (1) are mostly from surface of the sample, since the escape depth of electrons derived from bulk Si is about 66.1 nm [2]. On the other hand, the signals of Figure 5 (2) are almost from the Si wafer since Si Kα fluorescence X-rays come from the deep part of the sample. In fact, 50 % of Si Kα fluorescence X-rays transmit 1 μm thickness Si [3]. Figure 5 demonstrates that simultaneous measurements of the FY and TEY spectra provide useful information of the depth profiles.

4. Summary

We newly installed fluorescence X-ray detectors for the FY at BL-10, and performed preliminary measurements. We found it possible to perform high S/B ratio measurement from the result of Al K-edge XANES of α-Al_2O_3. In addition we found it is possible to obtain information of the depth profile from the result of Si K-edge XANES of a thermal oxide film on a Si wafer.

References

[1] J. Stöhr, NEXAFS Spectroscopy, Springer-Verlag, Berlin (1992).

[2] M. Kasrai, W.N. Lennard, R.W. Brunner, G.M. Bancroft, J.A. Bardwell, K.H. Tan, Appl. Surf. Sci **99** (1996) 303-3 12

[3] Center for X-ray Optics, X-ray Interaction with Matter Calculator, http://www-cxro.lbl.gov/optical-constants/filter2.html
We used it for calculating transmittance.