Quick XAFS System using Quasimonochromatic Undulator Radiation at SPring-8

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Abstract. A time-resolved quick x-ray absorption fine structure (QXAFS) system was designed and constructed using high flux but low power quasimonochromatic undulator radiation at a helical undulator beamline, BL40XU [1, 2] at SPring-8. At 12 keV, a flux of 10^{13} – 10^{14} photons/sec/0.1 % bw. was generated over an energy range of 1 keV with a total power of 7 – 15 W. A compact Si(111) channel cut monochromator having no active cooling was utilized. The monochromator was mounted on a galvano-scanner stage within an experimental hutch. The stage could be oscillated at frequencies up to 100 Hz over a 1.4-degree range. The Pt L_3-edge (11.6 keV) EXAFS spectra of Pt foil was measured in transmission and fluorescence modes up to k = 15 A^{-1} in a period of 50 ms/scan. In this report, the design of the QXAFS system and some preliminary experimental results are presented.

Keywords: Synchrotron radiation instrumentation, XAFS, Quick XAFS, Helical undulator
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INTRODUCTION

The time-resolved quick scanning x-ray absorption fine structure (QXAFS) technique is a powerful tool for investigating local structure and chemical states during physical and chemical reaction processes and has been used worldwide at synchrotron facilities (see ref. [3]). QXAFS can be adapted for transient process with measurements taken in fluorescence mode or in total electron yield mode, which is used to measure dilute or thin film samples. This is an advantage of QXAFS as compared with energy dispersive XAFS.

For better time resolution, a higher photon flux is needed on the sample. QXAFS has been coupled with undulator radiation by Frahm *et al* [3] using a cryogenically cooled channel-cut monochromator system to withstand the high heat load and has achieved a 40-Hz measurement cycle.

To achieve even faster QXAFS, we used helical undulator radiation in BL40XU at SPring-8, which gives a high flux but low power quasimonochromatic x-ray beam. The smaller head load allowed the monochromator to be downsized and the cooling devices to be removed. We developed a compact channel-cut monochromator and repeatedly oscillated the Bragg angle at 10 Hz in the EXAFS range at around 12 keV using a galvano scanner stage. In this report, we describe the design of the QXAFS system and some preliminary experimental results.

BEAMLINE AND INSTRUMENTATION

The radiation source was a helical undulator in BL40XU at SPring-8 [1]. This undulator generates on-axis fundamental radiation in the energy range from 8 – 17 keV. The energy bandwidth is about 1.5 – 2.5 % full width at half maximum of the peak energy. At 12 keV, a flux of 10^{13} – 10^{14} photons/sec/0.1 % bw. was obtained over an energy range of 1 keV. The total power was reduced to 7 – 15 W by eliminating off-axis higher harmonics at the front end without significant loss of the fundamental intensity. This greatly mitigated the heat load on the optics.

Two Si mirrors were located in an optics hutch in a Kirkpatrick-Baez arrangement [2]. Both mirrors were coated with stripes of Rh and Ni. A Rh coated mirror was used at glancing angle of 3.5 mrad in the preliminary test around 12 keV to remove high energy x-rays. The beam was collimated and focused at the sample position in the vertical and horizontal directions, respectively, by the mirrors.

The quasimonochromatic beam reflected from the mirrors was monochromatized with a compact Si(111) channel cut monochromator (27 (l) x 13 (w) x 14 (t) mm). The monochromator has no active cooling due to the low heat load. The gap between the reflecting surfaces was 3 mm. The inertia of a monochromator crystal was about 10 gcm^2. The monochromator crystal was mounted on a galvano scanner stage (Cambridge

Technology, 6900) (Fig.1). Detectors used were ionization chambers for the transmission mode and a photomultiplier (Hamamatsu Photonics, H7195) with a plastic scintillater (Saint-Gobain, BC420) for the fluorescence x-ray detection. The current signal from the ionization chambers was amplified and converted to voltage by a current amplifier (Keithley, 428). The output signal of the detectors and the angle position of the galvano scanner were digitized with an ADC board (Yokogawa, WE7272) and stored in the ADC's memory. The stored data were transferred to a PC after one series of measurements.

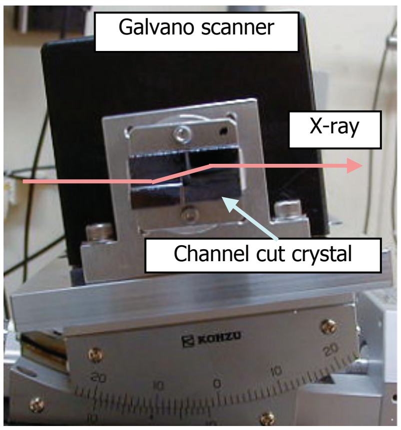

FIGURE 1. Si(111) channel cut monochromator mounted on the galvano scanner stage.

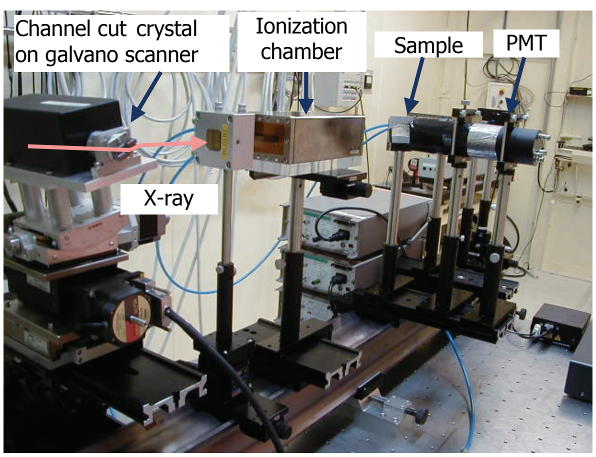

FIGURE 2. Layout of monochromator, sample and detectors on experimental stage.

BL40XU is a multipurpose beamline dedicated for experiments using high flux quasimonochromatic radiation. The monochromator, detectors and a sample were arranged on an experimental stage (Fig.2) temporarily placed in the experimental hutch during the QXAFS experiment.

The energy at each data points was calculated from the angle of the galvano scanner, and the calculated energy was corrected by simultaneously measuring the XAFS spectrum of a standard sample using the beam transmitted through the target sample.

PRELIMINARY EXPERIMENTAL RESULTS

This section contains some preliminary results obtained around 12 keV. The beam divergence incident on the mirrors was set to 5 (vertical) x 30 (horizontal) μrad^2 by a slit at the front end. The total power loading on the monochromator was estimated by calculation to be about 15 W. The beam size at the sample was about 0.2 (vertical) x 0.2 (horizontal) mm^2. Figure 3 shows the angle of the monochromator on the galvano scanner as it was oscillated with a triangular wave in the frequency range of 1 – 100 Hz. The stage could be oscillated at frequencies up to 100 Hz over a 1.4-degree range, which corresponds to an energy range of 1.5 keV around 12 keV.

The height of the exit beam from the channel cut monochromator changed during an energy scan but was reduced to about 12 μm at around 12 keV due to the small gap (3 mm) between the reflecting surfaces of the crystal. A refocusing mirror in the vertical direction should be placed downstream from the monochromator in experiments where a fixed beam position is required on the sample.

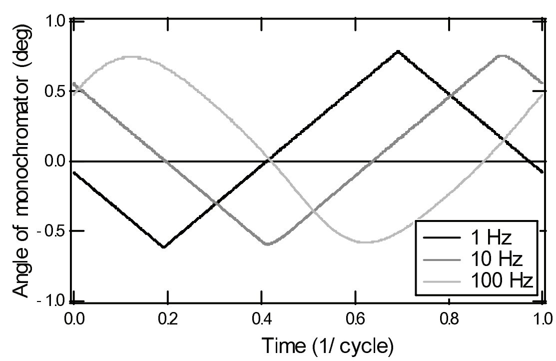

FIGURE 3. Angle of galvano scanner stage oscillated with triangular wave of 1 - 100 Hz.

Figure 4(a) shows an XAFS spectrum around the Pt-L_3 edge (11.6 keV) of a Pt foil in the transmission mode together with the incident beam intensity. The $k^3\chi(k)$-XAFS spectrum of the Pt foil is shown in Fig.4(b) in comparison with that measured at the bending magnet beamline, BL01B1. The collection time of the spectrum shown was 50 ms (10 Hz). The peak of the fundamental radiation from the undulator can be set to the target energy region, such as the

XANES or EXAFS region. We set the measurement at 12.5 keV to measure data with a high S/N ratio up to k = 15 A^{-1}. The intensity of the incident beam gently decreased toward the lower energy region but was still higher than that of the bending magnet source in SPring-8. Such an incident beam profile of the helical undulator is adequate for XAFS measurements because the EXAFS spectrum in the high k region requires a high incident photon flux.

Figure 5 shows a $k^3\chi(k)$-XAFS spectra of Pt foil in the fluorescence mode measured in BL40XU and BL01B1. The data were not corrected for self-absorption. Good quality spectra were obtained in 50 ms (10 Hz) for the concentrated samples.

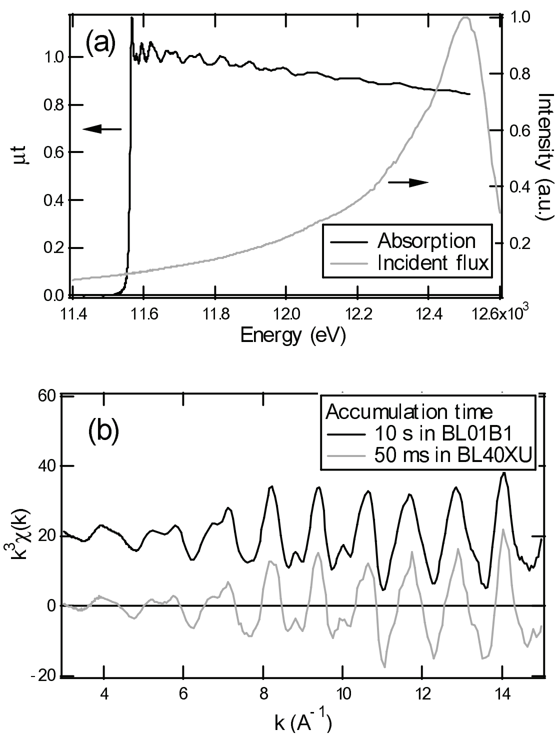

FIGURE 4. (a) Pt-L_3 edge (11.6 keV) XAFS spectrum of Pt foil in transmission mode together with incident beam intensity, and (b) $k^3\chi(k)$-XAFS spectra measured in BL40XU and BL01B1.

CONCLUSION

A new QXAFS measurement system was designed and constructed using helical undulator radiation at SPring-8. Using a galvano scanner stage with a compact Si(111) channel cut monochromator, we obtained EXAFS spectra for standard samples in the transmission and fluorescence modes in 50 ms.

We plan to improve the system in the fluorescence mode to achieve a time resolution of tens milliseconds for actual dilute or thin film samples.

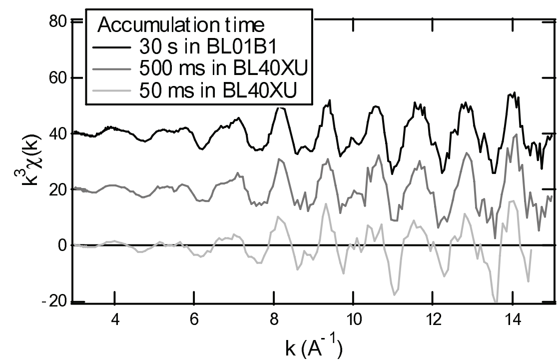

FIGURE 5. $k^3\chi(k)$-XAFS around Pt-L3 edge (11.6 keV) of Pt foil in fluorescence mode measured by scintillation counter in BL40XU and BL01B1.

ACKNOWLEDGMENTS

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