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Improvement in XAFS beamline BL01B1 at SPring-8

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Abstract. Beamline BL01B1 was constructed for conventional XAFS measurements using a bending magnet light source at SPring-8 in 1997 [1]. BL01B1 covers a wide energy range from 3.8 to 113 keV using an adjustable inclined double-crystal monochromator and a double mirror system. Here, we describe a newly developed apparatus and the current status of the BL01B1. A time-efficient QEXAFS method was developed to measure high quality spectra up to high-k regions in the shortest possible time. The method involves sweeping the monochromator continuously at a variable angular speed depending on the k-region using a VME stepping motor controller. During scanning, a pair of crystals of the monochromator rotates without translation motion like a channel-cut crystal. This QEXAFS method was applied to the fluorescence mode using a 19-element Ge detector combined with a digital X-ray processor system. A new conversion electron yield detector having a rotational sample stage was developed for single crystalline samples. Angular oscillations of 4 degrees in the sample rotation stage around the axis normal to the sample surface successfully remove diffraction noise in the XAFS spectra.

1. Introduction
A bending magnet light source at SPring-8 affords a high flux of photons of more than 40 keV. The beamline BL01B1 is dedicated to conventional XAFS measurements in a wide energy range from 3.8 to 113 keV for various research objects [1]. It offers ways to study XAFS spectra in a high-energy range covering the K-edges of most heavy elements. The research objects requiring special features concerning X-ray beam, such as high flux beam and/or microbeam, are targets of the undulator beamlines, BL37XU, BL39XU, and BL40XU.

So far, BL01B1 has been improved to achieve users’ demands, e.g., a better quality XAFS measurement in a wider energy range with shorter measurement time using more user-friendly systems. The main technical improvements achieved are an in-situ time-resolved quick scanning EXAFS (QEXAFS) method [2] and a high-quality XAFS measurement method for thin films and dilute samples. Both measurement methods are very common and occupy about 30% and 40% of the total beamtime, respectively. In addition to installing an in-situ experimental system, such as an exhaust gas treatment system, we developed a new time-efficient QEXAFS method to measure high quality spectra up to high-k regions in the shortest possible time. This QEXAFS method is applicable not only for the transmission mode but also for the fluorescence mode using a Lytle detector or multi-element Ge detector for thin films and dilute samples.

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The conversion electron yield (CEY) mode’s XAFS is useful for studies of thin film samples. Single crystalline thin films and/or substrates make a significant amount of noise in the XAFS spectra due to diffractions. We developed a new type of CEY detector having a rotational sample stage to remove the diffraction noise in the spectra. This report describes the current status of the BL01B1 with its newly developed apparatus.

2. Instrumentation and Results

2.1. Time-efficient QEXAFS technique using variable speed continuous energy-scanning

2.1.1. QEXAFS technique in a wide energy range. The X-ray optics and their arrangement in BL01B1 are shown in figure 1 [1]. The main X-ray optics consist of a first vertical collimating mirror, a fixed-exit double-crystal monochromator, and a second vertical refocusing mirror. The components downstream of the first mirror are installed on a deflection stage or on an elevation stage to follow the reflected beam from the mirrors [3]. The monochromator is a fixed-exit adjustable inclined double-crystal type with a single pair of Si(311) crystals [4]. The net plane of the crystals can be switched between Si(111), Si(311), and Si(511) by rotating the crystals around the [011] axis, which is in the scattering plane, to cover a wide energy range without breaking the vacuum chamber. The mirrors can be inserted into the transport channel below 60 keV. Due to the slope error from the ideal surface of the first collimating mirror, 50% of the central part of 1 m long mirrors was used for XAFS experiments to achieve sufficient energy resolution.

The QEXAFS measurements of BL01B1 were achieved by rotating a pair of crystals of the monochromator without a translation motion of the first crystal like a channel-cut crystal. The minimum measurement time of QEXAFS is many seconds depending on the energy range, which is limited by the maximum rotation speed of the monochromator of 0.17 degrees in Bragg angle per second. The height of the exit beam from the monochromator changes during scanning. The maximum change is 2.2 mm during scanning from 4 to 5.5 keV. However, the downstream refocusing mirror can maintain the beam height at the sample, which is crucial for measuring thin films in a glancing angle geometry. The second mirror can reflect an incident beam regardless of its change in height because 50% of the central part of the mirrors are used as described earlier. The beam height at the sample measured using a position sensitive ionization chamber (PSIC) changed by less than 20 μm during XAFS measurements from 3.8 to 60 keV. Figure 2 shows the change in the beam height on the sample around 4 keV with and without translation motion of the first crystal of the monochromator. The slightly larger beam height fluctuation during quick scanning was due to the slope error from the ideal surface at the off-center part of the second refocusing mirror.

Figure 1. Layout of main X-ray optics of BL01B1.

Figure 2. Changes in beam height of sample during scanning with (black) and without translation of first crystal (grey) around 4 keV measured using PSIC.
2.1.2. **Time-efficient QEXAFS technique.** In most cases, the amplitude of the EXAFS oscillation is smaller in the higher \( k \)-region. Thus, XAFS measurements are usually conducted with longer measurement time in the higher \( k \)-region to obtain spectra having a better signal to noise ratio. We developed a new time-efficient QEXAFS method to measure high quality spectra up to high-\( k \) regions in the shortest possible time. The method involves sweeping the monochromator continuously at a variable angular speed depending on the \( k \)-region using a VME stepping motor controller. Actually, users can divide an XAFS measurement region into several blocks and set the sweep speed for each block. Figure 3 shows the improvement in the quality of the EXAFS spectra in the high \( k \)-region using the time-efficient QEXAFS method.

2.1.3. **QEXAFS technique for thin films and dilute samples.** The time-efficient QEXAFS method under fluorescence mode for ultra dilute samples and thin films was developed using a 19-element Ge detector combined with a digital X-ray processor system (XMAP, XIA LLC). This method achieved time-resolved studies in the XANES region at a resolution of tenths of a second. Figure 4 shows the Ge K-XANES spectra of Ge\(_2\)Sb\(_2\)Te\(_5\) thin film measured over 45 sec.

![Figure 3. Pd K-edge \( k^3 \chi(k) \) XAFS spectra of PdO measured at constant (black) and variable (grey) angular speeds. Measurements were performed in a transmission mode, and the measurement time was 60 sec.](image)

![Figure 4. Ge K-edge XANES spectra of 40-nm-thick Ge\(_2\)Sb\(_2\)Te\(_5\) film. Measurements were performed in a fluorescence mode using a 19-element Ge detector, and the measurement time was 45 sec.](image)

2.2. **Conversion electron yield XAFS method with continuous sample rotation**

The CEY mode is more suitable than the fluorescence mode for the XAFS of thin film samples having a high density of core-hole atoms due to its high detection efficiency. The He CEY mode has been successfully applied for sub-nanometer thin film samples from 3.8 to 40 keV in BL01B1.

Many thin film samples have a single crystalline structure in the thin film and/or substrate, such as silicon and sapphire, which causes unwanted diffraction noise spikes in the XAFS spectra. Erbil et al. [5] developed a TEY detector that removes glitches in spectra of Si wafers by rocking the sample. We developed a new CEY detector that removes these noise spikes by a similar procedure. The detector’s sample stage, which is 50 mm in diameter, could be continuously rotated around the normal axis of the surface during the XAFS measurement (shown in figure 5). Angular oscillations 4 degrees in the sample rotation stage around the axis normal to the sample surface successfully remove the diffraction noise in the spectra. The principle of this method is based on reducing the time period under a diffraction condition to make it negligible compared with the total measurement time. The small rotation angle in this method minimized the effect of the sample nonuniformity, such as the sample shape and thickness of the film, which is of intrinsic importance for measuring high-quality XAFS spectra. The signal cable of the CEY detector was connected to the sample stage (= signal electrode)
along the rotation axis to minimize the mechanical stress on the cable connector, which reduced the electrical noise from the connector due to the rotation motion.

Figure 6 shows the Sr K-edge XAFS spectra of a (Ba,Sr)TiO$_3$ thin film coated on a sapphire substrate. Almost all the diffraction noise disappeared due to continuous sample rotation. Additionally, the continuous sample rotation method was successfully adapted for the fluorescence XAFS measurement of the single crystalline samples.

**Figure 5.** Schematic drawing of the CEY detector having a sample rotation stage.

**Figure 6.** Sr K-edge XAFS spectra of (Ba,Sr)TiO$_3$ film of 150 nm thick coated on single crystalline sapphire measured by CEY mode with sample rotation (black) and no rotation (grey). Arrows show noise due to diffractions of sapphire. Measurement time is 1300 sec.

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**References**


