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Design and feasibility of quick EXAFS scans for a 'broomstick' double-crystal monochromator at PLS beamline

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A data-collection technique for quick extended X-ray absorption fine-structure spectroscopy (QEXAFS) was developed with a new 'broomstick' double-crystal monochromator, which has been installed for X-ray absorption fine-structure (XAFS) applications at the Pohang Light Source. The monochromator operates in a fixed-exit scan mode as the Bragg angle is varied from 8 to 80°, corresponding to 2–14 keV, using an Si(111) crystal. The monochromator scan capability was investigated by analysing EXAFS data quality from step-scan and from continuous rotation of the Bragg crystal reflection angle. In our fast continuous-scan design, the electronic pulsing speed of the step motor is adjustable to avoid the monochromatic beam instability caused by serious mechanical resonance. The feasibility of QEXAFS scanning is demonstrated by a typical EXAFS scan (*e.g.* 1 keV range) being taken within 1 min.

Keywords: XAFS beamlines; quick EXAFS; crystal monochromators.

1. Introduction

In EXAFS beamlines, operational stability of the monochromatic X-ray beam is required over the photon energy range scanned. In the case of the DCM (double-crystal monochromator) crystal-scan mechanism, the instrumental performance depends upon the crystal parallelism, the crystal tuning stability (Jones *et al.*, 1995) and the scanning speed. In a conventional step-scan monochromator, the main drawback is a lack of precise control of the crystal rotation. Since the motion of the crystal-carrier assembly involves an inertia momentum in translation and revolution, the positioning control error increases at each step interval control. On the other hand, the so-called quick-scanning EXAFS monochromator (Ramanathan & Montano, 1995) specifically requires that the crystal-carrier motion is stable during a continuous scan. Recently, many *in situ* experimental demands have stimulated synchrotron users to conduct fast EXAFS scans during chemical reactions.

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2. 'Broomstick' double-crystal monochromator

The prototype 'broomstick' DCM, similar to the well known 'boomerang' type (Yang *et al.*, 1992) in fixed-exit operation, is designed and manufactured by the PSL (Physical Science Laboratory) at the University of Wisconsin-Madison. In Fig. 1, the linear-motion unit, a scan-driver unit controlled by a stepping motor, is mechanically linked through a flexible shaft to an internal crystal-revolution body (UHV-compatible Wisconsin DCM) sitting in a vacuum vessel. For this DCM system project, our laboratory engineers fabricated an external scan-driver unit and a vacuum vessel. In this design, the translation distance of the second crystal along the linear sliding unit is 320 mm for the full Bragg angle range (8–80°) at a 50 mm height offset between the extracted and incident beams. Our autocollimator testing result indicates excellent control of the crystal parallelism error, which is within 4 arcsec throughout the full scan range. The electronic signal (a succession of pulses) to the stepping motor is the practical control signal that enables the driving motion in the linear scan unit. One pulse gives a translation motion of 0.2 µm *via* the harmonic reduction gear (50:1) attached to the stepping motor. The linear encoder (0.5 µm resolution) is attached to the linear driving unit. The experimental data confirm that the control error in rotating the monochromator crystals is not greater than 0.03 eV.

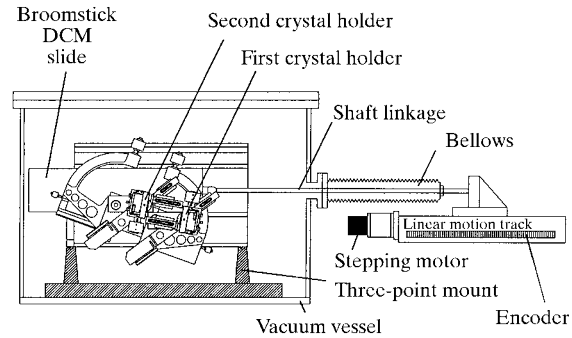

Figure 1
Schematic diagram of the 'broomstick' double-crystal monochromator.

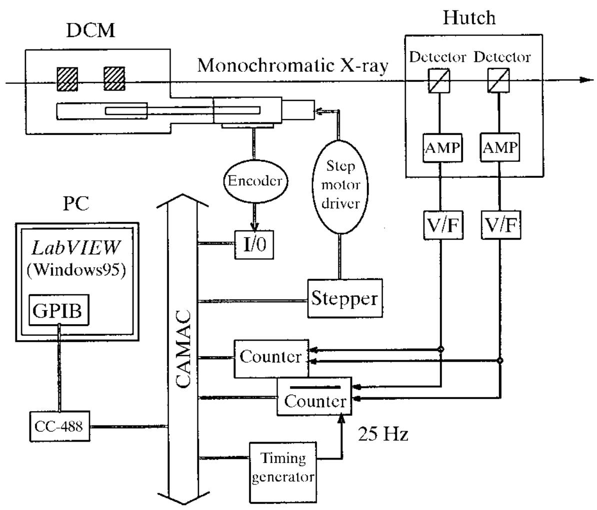

Figure 2
Electronic control diagram for QEXAFS data collection.

3. Quick EXAFS control and data acquisition

The EXAFS control software was developed using the commercial *LabVIEW* application code. Our QEXAFS technique is illustrated in Fig. 2. The stepper (E500) in the CAMAC module controls the scan-driver stepping motor, while the CAMAC digital register (IO-612) reads the digits from the linear position encoder (Mitutoyo KM-11) in the scan-driver unit. By scanning the photon energy, two ion-chamber X-ray detectors collect the photon flux in order to measure the transmission EXAFS signal. The pulse counter (KS-3610) obtains the number of pulses from a VFC (voltage to frequency converter) proportionally modulating the detector output signals. For this data collection, the VFC rate was chosen to be 100 kHz V^{-1} and the Keithley amplifier was set with a gain of 10^8 V A^{-1} and a time constant of 10 ms.

To configure the continuous-scan mode (fast-scan mode), our design has two counter modules receiving the inhibiting signal in turn. The 25 Hz square-wave signal from the timing generator (KS-3655) is divided into two lines of inhibiting signals, one from the internal data-way signal and another from the external inverted signal. Thus the data integration time at each counter becomes 20 ms, corresponding to the duration of the half cycle at 25 Hz. For each fixed 20 ms duration, while one counter performs the counting, the other counter completes data transfer from the KS-3610 memory register to a PC. We programmed various scan speeds, *i.e.* the translation velocity (v) of the linear sliding mechanism. The slew rate of the stepping motor, R_{slew}, is related to v by v (μm s^{-1}) = $0.2R_{slew}$ (pulse s^{-1}).

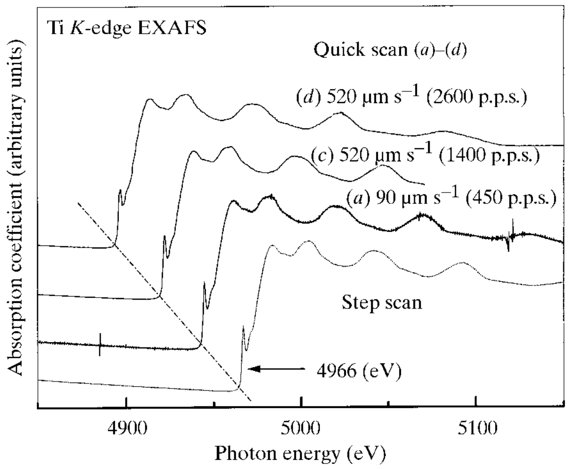

Figure 3
Comparison of QEXAFS signal measured at various speeds of continuous scan (*cf.* Table 1). p.p.s. = pulse s^{-1}.

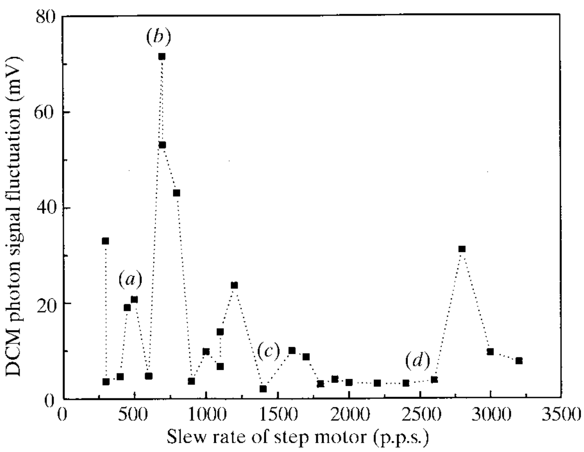

Figure 4
Overall fluctuation level of the DCM photon signal measured at various translation speeds of the scan sliding assembly. p.p.s. = pulse s^{-1}.

Table 1
Scan-setting parameters for QEXAFS.

Various stepping-motor slew rates are chosen for fast continuous-scan mode. Data for slow step-scan mode are given for comparison.

	Slew rate (pulse s^{-1})	Scan range (eV)	Number of data points	Total scan time (s)
Step scan	n/a	1200	300	1000
Fast scan (*a*)	450	380	2610	60
(*b*)	700	580	2548	59
(*c*)	1400	1200	2555	59
(*d*)	2600	1200	1376	32

4. Results and discussion

Table 1 gives the parameters and conditions for the EXAFS scan of the titanium *K*-edge (E_0 = 4966 eV). Scan conditions are denoted (*a*)–(*d*) for fast continuous scanning, with various scan speeds. In Fig. 3, the measured QEXAFS data are displayed, along with the slow step-scan case for comparison. At the stepping-motor slew rate 450 pulse s^{-1}, the type of data noise indicates transient behaviour caused by beam instability. Data quality of the QEXAFS spectra at 1400 and 2600 pulse s^{-1} is as good as in the step-scan case, but decreased sharpening of the near-edge peak is shown at faster slew rates. This distinction in energy resolution is less significant when the data-sampling rate is greater than the scan speed.

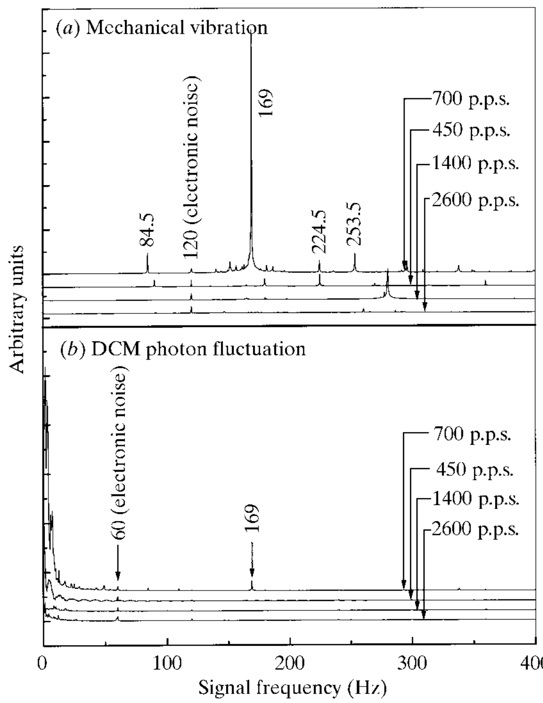

Figure 5
Frequency-mode analysis for mechanical vibration components and DCM photon fluctuation signal components. Electronic noise appears in the photon signal at 60 and 120 Hz. p.p.s. = pulse s^{-1}.

In QEXAFS operation, the relationship between the DCM beam instability (caused by the mechanical vibration of the monochromator crystals) and the slew rate of the stepping motor is shown in Fig. 4. When the electronic pulse rate to the stepping motor is resonant with the natural vibration frequency of the structure, the DCM photon fluctuation level increases. In Fig. 5, the frequency characteristics of the mechanical vibration signals around the scan-driver assembly and the photon fluctuation signals taken from the X-ray detector are compared. The strongest mechanical vibration is observed in the 700 pulse s^{-1} case, indicating the worst resonant vibration feature. 169 Hz is a dominant frequency component in both mechanical vibration and DCM photon fluctuation. 169 Hz and 253.5 Hz are, respectively, the second and the third harmonics of 84.5 Hz. It seems that the dominant component, 169 Hz, is magnified on its way to the crystal-carrier assembly due to the structural resonance effect. However, at 1400 pulse s^{-1} (harmonic motion of 700 pulse s^{-1}), there is no significant harmonic resonance contribution. It is shown that mechanical vibration is induced at 280 Hz, but there is no coupling with DCM photon fluctuation because the 280 Hz vibration does not coincide with a structural resonance. At 450 and 700 pulse s^{-1}, the resonant vibration frequency of 224.5 Hz is significantly induced, but is not transferable to the DCM photon fluctuation. In practice, the low-frequency component of the DCM photon fluctuation is induced and produces a noisy EXAFS spectrum. However, at higher slew rates (1400 and 2600 pulse s^{-1}), the influence of mechanical vibration becomes weaker, and the resultant beam stability enhances the EXAFS data quality, as shown in Fig. 3.

5. Conclusions

From our preliminary study, we have demonstrated the feasibility of quick EXAFS scanning using the ‘broomstick’ double-crystal monochromator. Our results indicate that the constant non-resonant velocity of linear travel leads to scan motion stability for the ‘broomstick’ sliding mechanism. The total scan time for the 4766–5966 eV range (titanium *K*-edge absorption spectrum) is 30–60 s, and the resolved energy interval is in the 0.35–0.65 eV range for a fixed elapsed time (20 ms) per data point. We estimate that the upper limit of scan speed is roughly 100 eV s^{-1}, which is our QEXAFS operational target.

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