BL04B2
High Energy X-ray Diffraction

1. Introduction

BL04B2 is dedicated to structural studies for disordered materials by pair distribution function (PDF) analysis. PDF analysis using high-energy X-ray diffraction can quantitatively determine the local structure of disordered materials at low scattering angles with a wide Q range. BL04B2 is equipped with two Si crystals as a monochromator, providing fixed-energy X-rays of 37.7 keV from Si(111), 61.4 keV from Si(220), and 113.1 keV from Si(333) (third-harmonic generation). The energy at 61.4 keV is mainly used in the PDF analysis. The diffractometer dedicated to PDF analysis was developed in FY1999[1] and has been operational for 20 years. Recently, we developed a rapid time-resolved diffraction measurement system, which uses a large two-dimensional flat-panel detector. This system successfully extracted crucial information about the crystallization process of amorphous solid electrolytes for lithium batteries [2].

Here, we report the upgrade status of the dedicated diffractometer for disordered materials in FY2019. Seven semiconductor detectors were installed for accurate diffraction measurements [3]. This setup suppresses background noise from the instrument and provides a sufficient energy resolution to discriminate the signals from fluorescence X-rays from those of the higher harmonic reflection of the monochromator crystal.

2. Improvement of the dedicated diffractometer system with seven point-type detectors

The dedicated diffractometer for disordered materials has operated for 20 years at the BL04B2 beamline of SPring-8. The first-generation detector was intrinsic germanium (Ge), whereas the second-generation one was a triple-cadmium telluride (CdTe) detector.

The first advantage of the Ge detector is its supersensitivity, which is important for the high diffraction angle region because the diffraction intensity is very weak at the high scattering vector Q (high diffraction angle) region due to the decay of the Q-dependent atomic form factor. However, the efficiency of the 15-mm-thick Ge detector (MIRION TECHNOLOGIES GL0515) is almost the same as that of the 1-mm-thick CdTe detector (AMPTEK X-123CdTe) at 61 keV, but it is 1.8 times higher at 113 keV. In addition, the thickness of the detector element of Ge is much larger than that of CdTe. It is confirmed that approximately a two-times higher gain is obtained with a Ge detector compared with the CdTe detector at 61 keV, suggesting that an almost four times higher gain is obtained in the case of 113 keV.

The second advantage of a semiconductor detector is that its energy resolution is sufficient to discriminate the fluorescence X-ray signals from those of the higher harmonic reflection of the monochromator crystal. The energy resolution (FWHM) of the CdTe detectors is better than 3.1 keV at 61 keV, whereas that of Ge detectors is better than 1.0 keV. On the other hand, the advantage of the CdTe detector is its small size, which is suitable to cover low diffraction angle regions where the space is limited.

The third advantage of the CdTe detector is that it adopts the Peltier device cooling system due to a small detector element. The present upgrade was installed in four CdTe detectors for low diffraction angle regions and triple Ge detectors with an automated liquid nitrogen filling system for high diffraction angle regions.

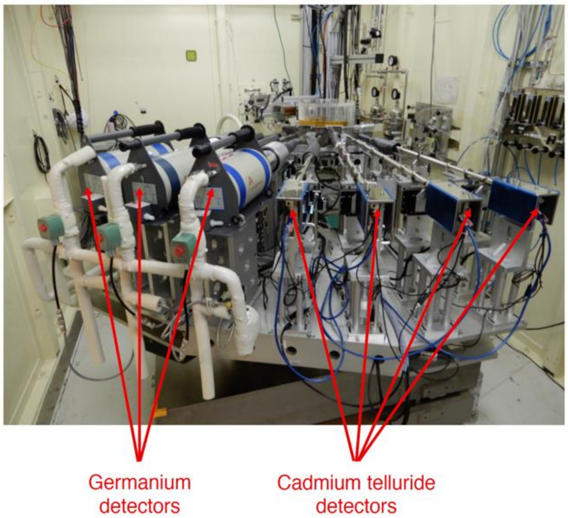

Fig. 1. Hard X-ray diffractometer installed at the high-energy X-ray diffraction beamline BL04B2 of SPring-8. Diffractometer has a horizontal scattering plane.

Figure 1 shows the typical setup of the upgraded diffractometer. This seven-detector system allows diffraction data up to Q = 30 Å^{-1} to be measured with a scan of diffraction angle (2θ) for the first detector from 0.3° to 9.5° ($Q = (4\pi/\lambda)\sin\theta$, where 2θ is the scattering angle and λ is the photon (X-ray) wavelength). Hence, diffraction data can be provided up to 57.5° when the incident energy of X-rays is 61.6 keV. At an incident energy of 113 keV, it is possible to use triple Ge detectors, which provide diffraction data up to Q=25 Å^{-1} by a scan of the diffraction angle for the first detector up to 9° to give diffraction data up to 25.3°. Figure 2 compares the new (quad-CdTe and triple-Ge) setup with the old (triple-CdTe) one on the structure factors $S(Q)$ of glassy SiO_2. The measurement was approximately three times faster than the previous setup. In particular, the statistical accuracy at high Q was significantly improved, which is critical for performing accurate PDF analysis with a high real-space resolution.

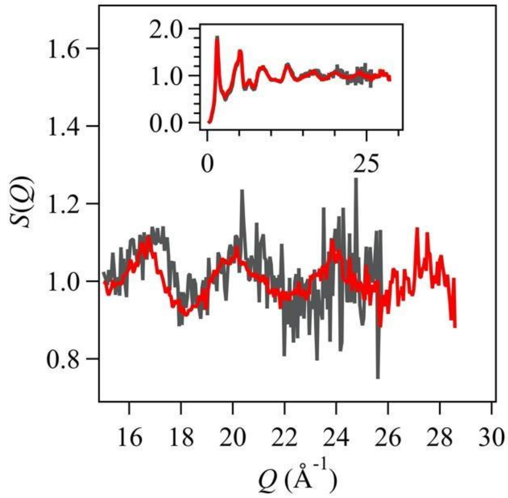

Fig. 2. Total structure factors, $S(Q)$ for glassy SiO_2 from 15 Å^{-1} to 30 Å^{-1}. Inset shows the $S(Q)$ from 0.2 Å^{-1} to 30 Å^{-1}. Old (black) and new (red) setup. It took approximately 30 min to collect each data point.

Koji Ohara[*1], Hiroki Yamada[*1], Saori Kawaguchi[*1], Bagautdinov Bagautdin[*1], and Michitaka Takemoto[*2]

[*1] Center for Synchrotron Radiation Research Diffraction and Scattering Division

[*2] Engineering support Group

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