

## BL04B2

### High Energy X-ray Diffraction

#### 1. Introduction

Beamline BL04B2 is used in structural studies for disordered materials by pair distribution function (PDF) analysis. The PDF analysis using high-energy X-ray diffraction is useful for quantitatively determining 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, which provides 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. Recently, we have developed a new sample changer combined with a high-temperature furnace and a fully automated alignment system on beamline BL04B2. The previous sample changer could only operate with 10 samples at most and was also limited to room temperature (RT) operation with manual alignment. The new automated sample changer system (manufactured by Rigaku Aihara Seiki, Japan) can load up to 21 samples. Since automated high-energy XRD and total X-ray scattering measurements with the analysis of temperature dependence (from RT to 1200°C) are also available in the same system, the system allows total X-ray scattering measurements of up to 21 samples with different temperatures to be performed automatically<sup>[1]</sup>.

In FY1999, a dedicated PDF analysis system using a Ge semiconductor point detector was developed<sup>[2]</sup>, and in FY2013, a system with three CdTe point detectors installed at 16° intervals was adopted, reducing the measurement time to less than half of the previous measurement time. Furthermore,

in FY2017, the system was upgraded to a system with four CdTe semiconductor detectors and three Ge semiconductor detectors installed at 8° intervals, further reducing the measurement time<sup>[3]</sup>. However, the oldest of the seven semiconductor detectors, the CdTe detector, began to malfunction frequently. Therefore, it was replaced. Here, we report its details.

#### 2. Replacement of old CdTe semiconductor point detector on the dedicated X-ray total scattering measurement system

The CdTe detector currently in use, X-123CdTe, is manufactured by AMPTEK and has an area of 25 mm<sup>2</sup> and a thickness of 1 mm. The advantage of semiconductor detectors is that they have sufficient energy resolution to discriminate fluorescence from the sample and the signal of the higher harmonics reflection of the monochromator crystal; the energy resolution (FWHM) of the CdTe detector is less than 3.1 keV, whereas that of the Ge detector is less than 1.0 keV. The disadvantage of the Ge detector is the refilling of liquid nitrogen every day, which interrupts the measurement. On the other hand, the advantage of the CdTe detector is its smallness, which is suitable to cover low diffraction angle regions where space is limited. Another advantage of the CdTe detector is that it adopts the Peltier device cooling system due to a small detector element.

For the replacement of the old CdTe detector, a CdTe detector (XCT551-SP8) that uses a DSP (APU101X-CT-SP8) with a built-in power supply for the detector is available from TechnoAP, which

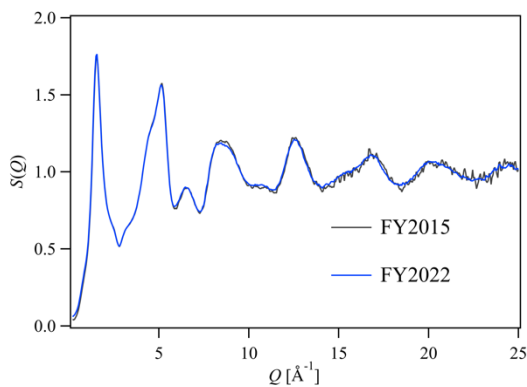


Fig. 1. Total structure factor  $S(Q)$  for glassy  $\text{SiO}_2$  up to  $25 \text{ \AA}^{-1}$ .

can directly operate the high-speed digital signal processing of the preamplifier signal. This DSP has already been used in the digital signal processing of the multichannel analyzer (MCA) of the Ge semiconductor detectors of BL04B2, so we

introduced the new XCT551-SP8 CdTe detector. The area and thickness of the detector are  $25 \text{ mm}^2$  and  $1 \text{ mm}$ , respectively, as in X-123CdTe.

Figure 1 shows the  $S(Q)$  of the silicate glass obtained using the new CdTe semiconductor detector. Figure 2 shows the energy spectrum acquired with the new DSP. In the energy spectrum, we can see both X-rays of  $37.7 \text{ keV}$  from  $\text{Si}(111)$  and those of  $113.1 \text{ keV}$  from  $\text{Si}(333)$ . The PDF data of a comparable quality to previous data have been obtained and direct access to the DSP is possible. We will also aim to realize X-ray total scattering measurements with a consistently low background by automatically separating elastic scattering, Compton scattering, and fluorescence scattering components for each measurement sample.

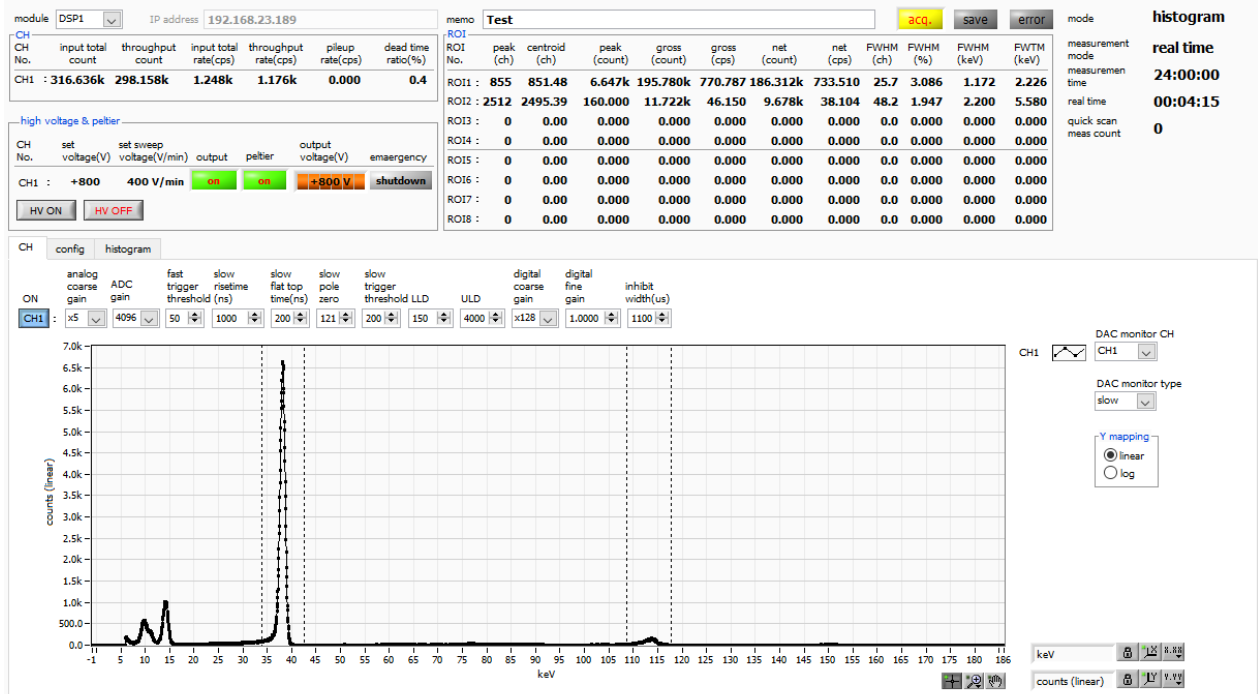


Fig. 2. Energy spectrum acquired with a new CdTe detector at  $\text{Si}(111)$  in BL04B2.

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