BL04B2
High-energy X-ray Diffraction

1. Introduction

Beamline BL04B2 is used to study the structure of disordered materials by pair distribution function (PDF) analysis. To obtain PDF patterns, a diffractometer with seven series of point-type semiconductor detectors was used. In FY2021, we developed a new sample changer combined with a high-temperature furnace and a fully automated alignment system that allowed total X-ray scattering measurements of up to 21 samples at different temperatures to be performed automatically [1]. However, because the detector system was not renewed, the time required for each measurement remained at around 3 hours. This hindered high-throughput X-ray total scattering measurement at this beamline.

To overcome this limitation, we have been preparing to introduce two-dimensional detectors in this beamline. For example, in FY2022, we substituted the Si(111) monochromator with the Si(511) one [2]. This change allowed us to effectively maintain the X-ray energy at 113 keV while eliminating diffractions caused by high-harmonic X-ray contamination in experiments. This modification is essential for utilizing two-dimensional detectors.

During FY2023, the new system, named the high-throughput X-ray total scattering diffractometer, was installed. Prior to installing this new system, the activity conducted downstream of the experimental hutch in BL04B2 was combined with that in BL10XU, which is dedicated to high-pressure XRD experiments [3]. Then this new system was installed downstream of the hutch. In this report, we describe the installation, design, and performance of the new system in detail.

Fig. 1. Photographs of the (a) old and (b) new experimental setups downstream of BL04B2.

2. Installing new setup in BL04B2

A diffractometer for high-pressure XRD with an imaging-plate detector was placed downstream of the experimental hutch [Fig. 1(a)]. Currently, the experimental setup in BL10XU is superior to the measurement system above so that the activity using the system in BL04B2 was combined with that in BL10XU. This allowed us to install the new system alongside the old system until its removal in

March 2023. After removing old instruments, the new system was prepared in April 2023. Then all the instruments were assembled [Fig. 1(b)]. This relocation allowed us to replace the old instrument with the high-throughput X-ray total scattering diffractometer. The construction and commissioning of the new system started in April 2023, after the removal of the old system in March 2023.

3. Brief explanation of the new setup

The new measurement system (high-throughput X-ray total scattering diffractometer) consists of a sample changer, a nitrogen blower at high/low temperatures (100 K–1073 K), and two 2D detectors linked to the sample environment. The sample changer can hold up to 50 capillary samples and the samples are mounted on a spinner stage automatically. The two-dimensional CdTe detector (LAMBDA CdTe 750k, X-Spectrum GmbH, Germany) implemented in this system has a smaller pixel size than those of the widely used flat-panel detectors and enables the acquisition of high statistical data in a shorter time. Currently, by using X-rays at a photon energy of 113 keV, the measurable Q range is from 0.2 to 23.5 Å^{-1} and each measurement can be accomplished in several minutes with a high signal-to-noise ratio.

4. Typical results and conclusion

Typical *S*(*Q*), and *G*(*r*) results for silicate glass with an exposure time of 100 s are shown in Fig. 2. From this figure, it is confirmed that an exposure time of several minutes is sufficient for the measurement of silicate glass.

This system facilitates X-ray total scattering measurements of temperature changes, and the required measurement time becomes up to 100 times faster than that with the existing measurement system in BL04B2. This improvement enables us to obtain comprehensive structural information of materials with different compositions and synthesis conditions, which had been difficult in the past.

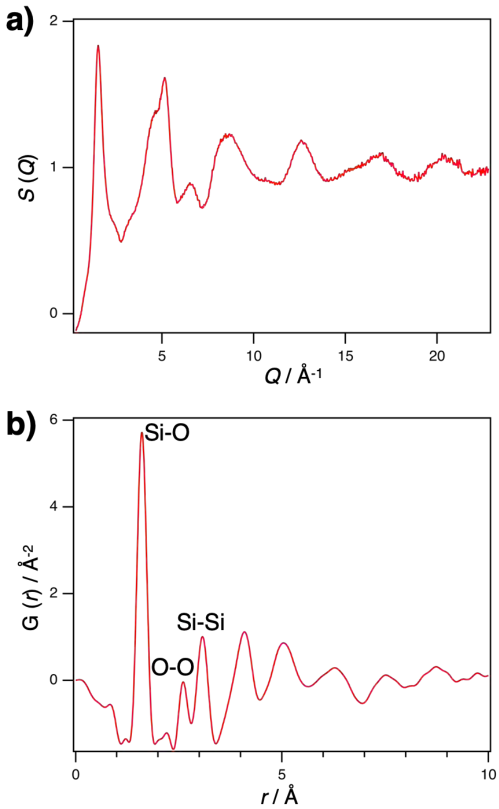

Fig. 2. Typical results for silicate glass with exposure time of 100 s. a) *S*(*Q*). b) *G*(*r*).

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