BL04B2
High Energy X-ray Diffraction

1. Introduction

Beamline BL04B2 is dedicated to structural studies of disordered materials by pair distribution function (PDF) analysis using high-energy synchrotron X-rays. Reliable PDF measurements require wide wavenumber vector Q ($Q = 4\pi\sin\theta/\lambda$) ranges and high statistical accuracy, making BL04B2 an important facility for investigating glasses, liquids, nanomaterials, and functional compounds.

Until FY2021, total scattering experiments at BL04B2 were carried out using a diffractometer equipped with seven series of point-type semiconductor detectors (PD system). Although this system enabled stable PDF measurements, each experiment required approximately three hours, which severely limited high-throughput studies. To partially address this issue, a new sample changer with a high-temperature furnace and automated alignment system was developed in FY2021, enabling sequential measurements of up to 21 samples at different temperatures [1]. However, the outdated detector system remained a bottleneck.

To overcome this limitation, the introduction of two-dimensional detectors was pursued. As a preparatory step, the Si(111) monochromator was replaced with a Si(511) monochromator in FY2022 [2]. This modification allowed stable operation at 113 keV while effectively suppressing high harmonic contamination, a prerequisite for the use of two-dimensional detectors.

In FY2023, a new high-throughput X-ray total scattering system was installed downstream of the hutch, consisting of two 2D CdTe photon-counting detectors (LAMBDA 750k, X-Spectrum GmbH) and a nitrogen blower (100–1100 K). One detector, placed at ~510 mm (2θ = 0.2°–9.0°), is optimized for low-angle scattering, while the second detector, typically set at ~140 mm (2θ = 8.5°–30°), offers a tunable geometry to maximize the Q range and intensity for amorphous or low-crystalline materials. With this configuration, the system covers a Q range of 0.2–27 Å^{-1}.

To enable measurements of highly crystalline samples, a motion mechanism was introduced for the second detector so that both detectors can be positioned at the same sample-to-detector distance when needed. This allows the system to perform high-angular-resolution total scattering experiments across a range of materials.

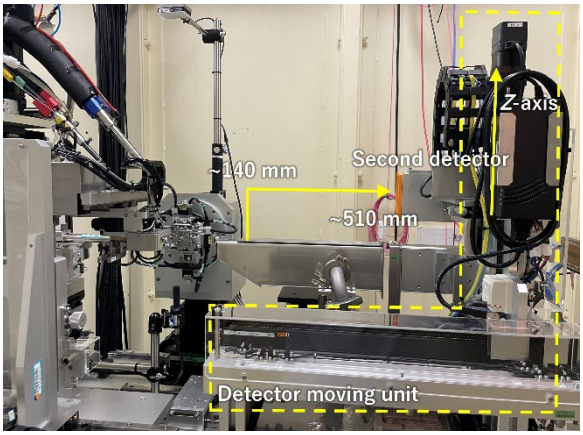

Fig. 1. High-throughput X-ray total scattering system for a highly crystalline sample.

2. Development of high-throughput X-ray total scattering diffractometer

The detector moving unit was installed for the second detector of the high-throughput X-ray total scattering system (HT system), as shown in Fig. 1. For measurements requiring higher angular

resolution, particularly for crystalline materials, a second detector was positioned at ~510 mm, the same distance as used for the low-angle detector. Adjusting its vertical (Z-axis) position allowed a maximum scattering angle of ~25°, which is sufficient for most total scattering experiments. Multiple measurements at different Z-axis positions ensured the smooth merging of scattering patterns and a comprehensive Q range of 0.2–23 Å^{-1}. The experimental geometry could be readily modified in accordance with the sample or desired Q range.

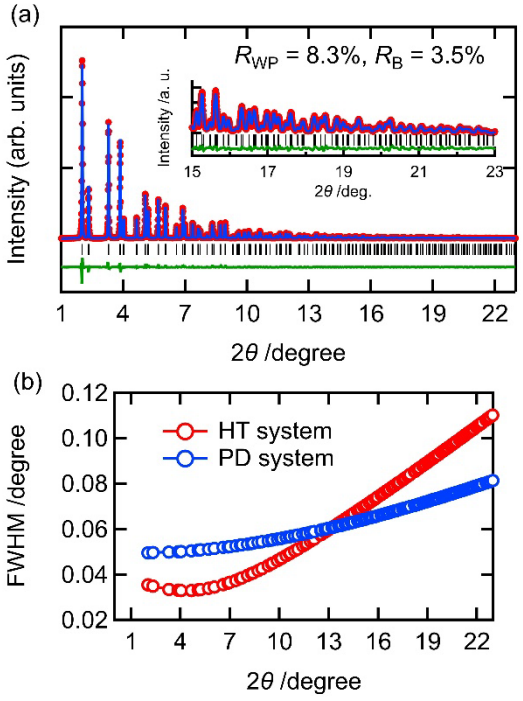

Fig. 2. (a) XRD pattern and Rietveld refinement of CeO_2. (b) Full width at half maximum of Bragg peaks obtained by the Rietveld fitting of CeO_2.

Rietveld refinement using Jana2020 software for the diffraction pattern of CeO_2 (NIST) obtained with 60 s exposure revealed good agreement between observed and calculated values [3]. Although the full width at half maximum (FWHM) of low-angle Bragg reflections (2θ < 10°) was broader than that obtained with the previous setup, the results demonstrate that the system can reliably characterize highly crystalline samples requiring high-energy X-rays.

The results of the PDF analysis of Ni powder using PDFgui [4], with exposure times of 10 and 180 s, showed excellent agreement between experimental and calculated $G(r)$ patterns over the range of 1–20 Å. These results confirm that the newly implemented system provides accurate total scattering data for both low- and high-crystallinity samples. Overall, the upgrade enables a flexible and efficient evaluation of a wide variety of materials, demonstrating the versatility of the system for comprehensive total scattering studies.

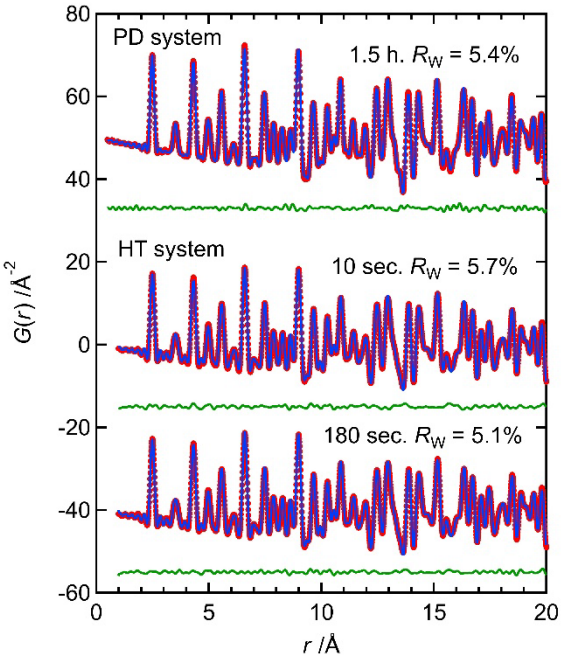

Fig. 3. $G(r)$ patterns and PDF fitting result for Ni obtained using PDFgui.

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References:

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[4] Farrow, C. L. Juhas, P. Liu, J. W. Bryndin, D. Božin, E. S. Bloch, J. Proffen, T. & Billinge, S. J. L. (2007). *J. Phys. Condens. Matter* **19**, 335219.