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. 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. 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 while analyzing temperature dependence (from RT to 1200ºC) are also available in the same system, the system allows total X-ray scattering measurements of up to 21 samples at different temperatures to be performed automatically [1].

During FY2021, we undertook a monochromator upgrade at BL04B2 with the aim of eliminating unwanted harmonics. A detailed account of this upgrade can be found in our previous report [2]. Currently, BL04B2 features a bending magnet designed to restrict the horizontal beam divergence to 0.34 mrad by using slits for high-energy X-ray total scattering experiments. To optimize the flux, we have employed two horizontally curved Bragg monochromators: one using Si (111) and the other Si (220) crystals, both set at a fixed angle of 3°. These Si (111) and Si (220) crystals are attached to a holder with a water-cooling system [Fig. 1(a)]. The Si (220) monochromator is primarily responsible for generating the first harmonic of reflective X-rays, which allows us tomonochromatize the X-ray energy to 61.4 keV. Conversely, the Si (111) monochromator selects the third harmonic, yielding an energy of 113.4 keV. Located at a distance of 46 meters downstream of the X-ray source, the monochromators are flexible in their configuration,

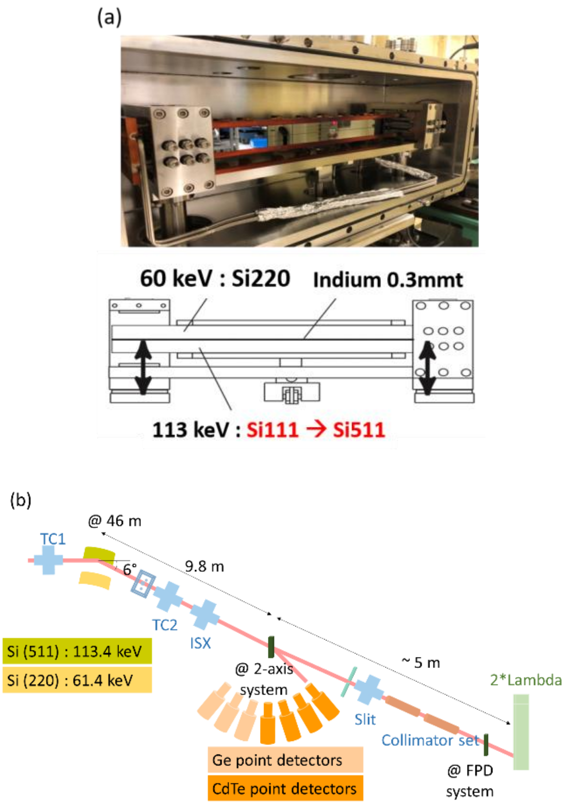

Fig. 1. (a) Current setting of two monochromators at BL04B2. (b) Layout of BL04B2 beamline. (Reproduced with permission from [2])

allowing for variable adjustments in the radius of curvature within the range from 320 to 430 m. This flexibility in modifying the radius of curvature enables us to vary the focal point within the experimental hutch between 10 and 15 m from the monochromator [Fig. 1(b)].

2. Upgrade of Monochromator

The contamination from higher harmonics of reflective X-rays, particularly Si 444 at 151.2 keV (as indicated by the dotted line in Fig. 2) was observed. To tackle this problem, we devised a resolution for BL04B2 by substituting the Si (111) monochromator with a Si (511) monochromator. This change allowed us to effectively maintain the X-ray energy at 113.4 keV while eliminating diffractions caused by high-harmonic X-ray contamination in experiments (Fig. 2). By selecting the first harmonic of reflective X-rays with the Si (511) monochromator, the resulting diffractions from higher harmonics occurred at angles that were too small to be collected, thus eliminating the interference in measurements. Meanwhile, the monochromator was bent to focus the X-ray beam at an approximately 10-m focal point and incorporated two slits along the beam path. This use of slits limits the beam size to 2 mm × 2 mm and yields a narrower peak shape in CeO_2 measurements, as demonstrated in the inset of Fig. 2. Using the formula $\Delta E/E = \cot\theta_B \cdot \Delta\theta$, we can approximate the energy resolution to be 2×10^{-3} when the focal point is set at a distance of 10 m from the monochromator [3, 4].

3. Conclusion

In conclusion, substituting the Si (111) monochromator of BL04B2 with a Si (511) monochromator yields advantages for both 2-axis point detectors and flat-panel detector systems. This upgrade eliminates X-ray peaks caused by high-harmonic X-ray contamination while preserving the X-ray energy at 113.4 keV. The energy resolution at BL04B2, achievable with a focused 2 mm × 2 mm beam in the 2-axis system, can be calculated as 2×10^{-3}. These improvements have considerably enhanced the quality of experimental data at BL04B2, reinforcing the beamline for fruitful scientific aspects.

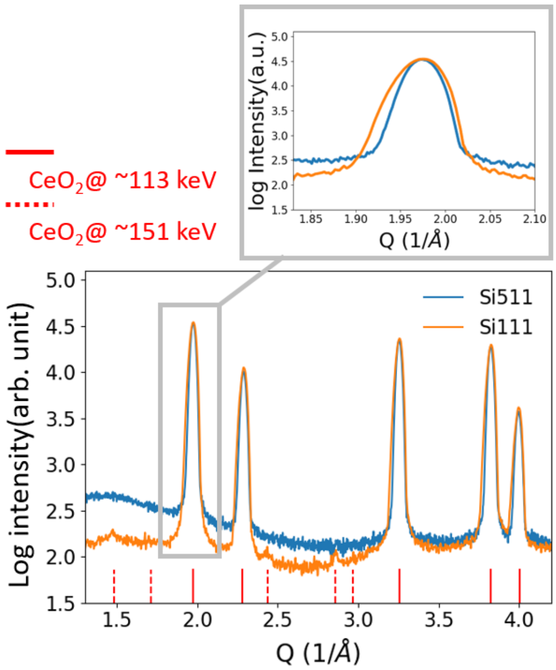

Fig. 2. CeO_2 measurements conducted with 2-axis system at BL04B2 before and after substituting the monochromator. The dotted lines show the reflections from high-harmonic Si 444 X-rays. (Reproduced with permission from [2])

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