BL44B2
RIKEN Materials Science I

1. Introduction

BL44B2 has become recognized as one of the best total scattering beamlines, maintaining a balance among *Q* (scattering vector) range (30 $Å^{-1}$), *Q* resolution (10^{-3} $Å^{-1}$), and statistics (0.3 %) [1]. Such triple-3 data have made it possible to reveal various structural aspects, such as valence electron densities [2], and short- and long-range orders [3]. The outcomes can be attributed to the development of hardware (OHGI) [4] and software (ReLiEf) [5] on the angle-dispersive detector system.

2. Recent Activities

The angle-dispersive system has functioned well for inorganic materials so far. On the other hand, it turned out that the system was not practical for organic materials. The great influence of Compton scattering has made it quite challenging to see Bragg peaks and diffuse scattering from organic materials composed of light elements. For example, the Compton scattering intensity from carbon, which is a typical light element, is twenty times as high as the total scattering intensity even at 20 $Å^{-1}$ in *Q*.

Compton scattering is commonly subtracted from the whole scattering intensity with an empirical formula after measurement. The subtraction can cause a change in appearance; however, the subtracted data do not improve in quality at all. Therefore, Compton scattering needs to be separated in the process of measurement, especially for organic materials.

From the Compton shift, an energy resolution of 1% was found to be necessary for the separation of Compton scattering from total scattering. Silicon drift detectors (SDDs), which have been used mainly for spectroscopy, were chosen as energy-dispersive detectors for Compton-free total scattering measurements. An energy of 30 keV has been typically used as incident X-rays to balance the *Q* range and *Q* resolution, in which case the quantum efficiency of the silicon sensor 1 mm thick is only about 30%. To improve the efficiency, a high-*Z* (CdTe) sensor was employed instead of a Si sensor for backscattering. Although the energy resolution of the CdTe sensor is just half as high as that of the Si sensor, it is possible to separate Compton scattering from total scattering at backscattering, where the Compton shift is larger than that at forward scattering.

In FY2024, nine SDDs and six CdTe detectors were installed on the ω stage of the two-circle diffractometer every 10 degrees for forward and backscattering, respectively. Moreover, experimental settings have been optimized so that the angle-dispersive system, installed on the 2θ stage, can be used along with the energy-dispersive system. The settings consist of a low-temperature apparatus, a high-temperature apparatus, and a CCD camera for sample alignment. Before optimization, all the components had been fixed on the diffractometer. However, they interfered with the use of the energy-dispersive system. Therefore, the three components needed to be placed off the diffractometer somehow. In the end, the components were rearranged as shown in Fig. 1. In this setting, low-temperature gas goes down to a sample, while high-temperature gas goes up to a sample. The CCD camera in a horizontal position allows one to look at the sample image in a mirror under the sample. The three components can move out of the sample automatically when they are not being used. Optimizing the experimental settings has enabled the simultaneous combination of the angle-dispersive system with the energy-dispersive one, allowing for temperature changes in a sample between 100 and 1000 K.

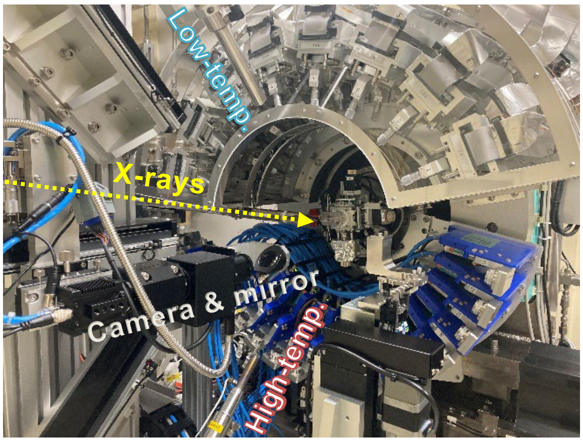

Fig. 1. Settings optimized for both the detector systems (in a state out of the sample).

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