BL40B2
Structural Biology II

1. Introduction

BL40B2 is a beamline where small-angle X-ray scattering (SAXS) is available. It is used to detect the correlation length and size of macromolecules, molecular aggregation, and so on. Since information is contained in X-rays scattered at a few degrees, the two-dimensional X-ray detector is placed a few meters away from a sample.

On the other hand, in order to detect the structure of molecular packing, the distance between the sample and the detector has to be reduced to a few hundred millimeters for wide-angle X-ray scattering (WAXS). Many users targeting soft materials often acquire structural information using both small and large camera lengths. Changing the length of the camera takes several hours. In order to reduce the time, we have developed a SAXS–WAXS switching system. In addition, to carry out user experiments efficiently, we have also introduced a system of sample exchange for X-ray solution scattering.

2. Development of mechanism for switching between SAXS and WAXS

Figure 1 shows the SAXS–WAXS switching system constructed in FY2020. This system has two X-ray two-dimensional photon-counting detectors [1,2]. When conducting SAXS measurement (see Fig. 1(a)), a vacuum path is connected from the downstream of the sample to the front of the X-ray detector Pilatus 3 S 2M (Dectris Ltd., Switzerland). The vacuum path consists of welded bellows. When switching to WAXS measurement (see Fig. 1(b)), these bellows become shorter. A WAXS detector (Eiger2 S 500K-BL40B2 (Dectris Ltd.)) and a short vacuum path are moved along the X-ray optical path. When switching to SAXS measurement, the detector and short vacuum path are out of the X-ray optical path and the bellows become longer. It takes about 30 s to switch between SAXS and WAXS.

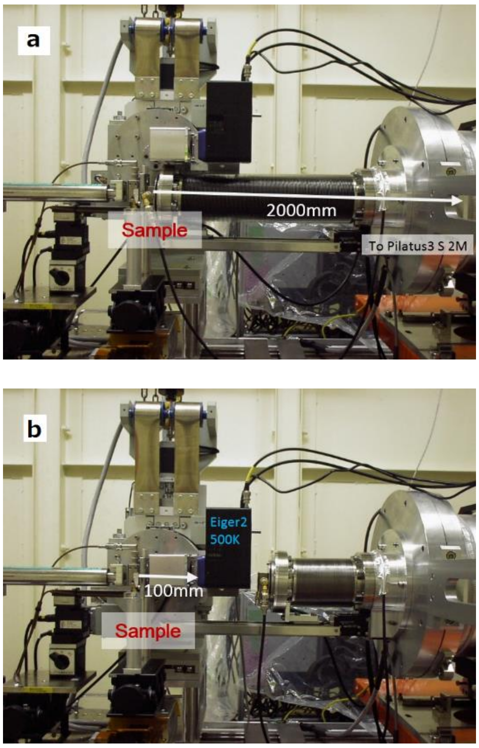

Fig. 1. Switching system between SAXS and WAXS. (a) SAXS mode with a distance of 2 m from sample to detector. (b) WAXS mode with 100 mm distance.

Figure 2(a) shows the pattern of silver behenate measured in the SAXS mode. In the Q range, 0.05 nm^{-1} < Q < 4.2 nm^{-1} was measured. In the azimuth direction, the Q range was obtained at 360 degrees. On the other hand, Fig. 2(b) shows the pattern of silver behenate measured in the WAXS mode. In the Q range, 1 $nm^{-1} < Q < 28$ nm^{-1} was measured. The Q range was acquired over 180 degrees along the azimuth direction, allowing users to measure in the vertical and horizontal directions. It will be used not only for isotropic scattering, but also for X-ray fiber diffraction and grazing incident X-ray scattering.

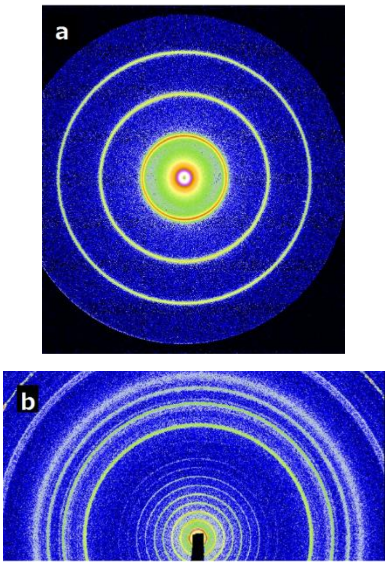

Fig. 2. (a) SAXS and (b) WAXS patterns of silver behenate obtained using switching system at wavelength of 0.1 nm.

3. Installation of liquid sample exchange system

In FY2019, we introduced an automatic cell cleaning system [3] to achieve X-ray solution scattering for a small amount of liquid sample (approximately 7 µL). This small amount of liquid sample in a glass capillary takes a liquid column shape, and this system automatically transfers this liquid column to the X-ray irradiation position by computer vision (see Fig. 3). After the scattering measurement, the capillary wall is cleaned and dried according to the specified procedure. However, the measurement sample must be manually pipetted into the funnel. To improve the efficiency of measurement, we introduced the sample exchange system (Xenocs Inc., France) for X-ray solution scattering (see Fig. 4).

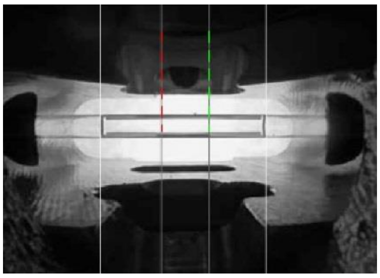

Fig. 3. A 7-µL liquid column in the glass capillary is fixed along the X-ray path. The edges of the column are captured by computer vision.

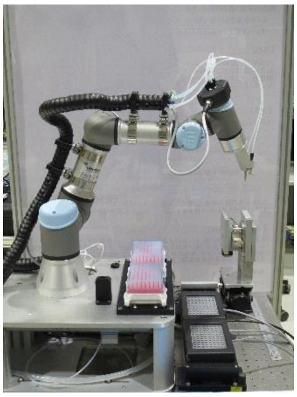

Fig. 4. Sample exchange system for X-ray solution scattering.

Figure 5 shows a schematic diagram of this sample exchange system. The five steps in this figure are explained as follows. (1) To mount a new microtip to the tip head of the robot arm, a slight pressure of the head is applied onto the opening of the tip selected in order from among the192 tips on the rack. (2) The tip is immersed in a specified sample liquid on two 96-well plates and the liquid is aspirated by the pump. (3) The tip is transferred to the funnel, which is the injection port of the sample holder, and the liquid is then released. (4) To clean and dry the capillary wall following the measurement, the cleaning nozzle of the robot arm rests on the funnel. (5) The tip is ejected from the arm with a tip ejector in preparation for installing a new tip. The used tip is dropped into the trash box. Measurements can be continuously performed by repeating these steps without opening the door of the experimental hutch. Therefore, this system is expected to operate efficiently. We are planning to conduct a connection test with beamline devices in FY2021.

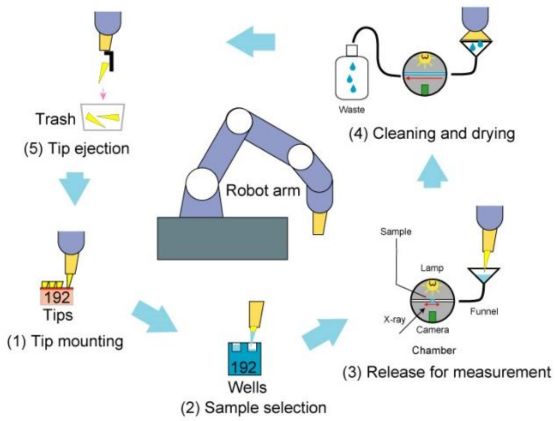

Fig. 5. Schematic diagram of the sample exchange system for X-ray solution scattering.

Noboru Ohta and Hiroshi Sekiguchi
Diffraction and Scattering Group I. Diffraction and Scattering Division, JASRI

References:

[1] N. Ohta and H. Sekiguchi, *SPring-8/SACLA Annual Report FY*2017, 78.
[2] N. Ohta and H. Sekiguchi, *SPring-8/SACLA Annual Report FY*2018, 78.
[3] N. Ohta and H. Sekiguchi, *SPring-8/SACLA Annual Report FY*2019, 72.