Small Angle X-ray Spectroscopy

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Abstract

The small angle X-ray spectroscopy analyzes size and shape of the nanoscale matter by scattering of synchrotron raditation X-ray. SAXS is usually used in researches about energy materials, display, and semiconductors. In this report, the principle and the experimental setup of SAXS was presented. Also, the SAXS experiment of the bovium serum albumin(BSA) protein solution in static and flow mode was conducted in PLS-II.

INTRODUCTION

Synchrotron radiation is the radiation emitted by charged particles whose trajectories are curved by the magnetic field. For the third generation storage rings, such as PLS-II, wigglers and undulators are used as sources of synchrotron radiation. The undulator radiation has higher brightness than wiggler radiation. However, the wiggler radiation has broader bandwidth.

The beamline of the storage ring is divided by two sections. First, in the front-end, monitoring the position of photon beam, defining the angular acceptance, and filtering the unwanted spectrum are conducted. In the photon transfer line(PTL), photons with single wavelength are extracted by monochromator, and transmitted to the sample after passing through optics in the beamline.

In this report, experiment of small angle X-ray spectroscopy(SAXS), one of the spectroscopy for nanoscale materials, was conducted in 4C beamline of PLS-II.

SMALL ANGLE X-RAY SPECTROSCOPY

The small angle X-ray spectroscopy is the type of spectroscopy that analyzes size and shape of the nanoscale matter such as polymer, metal, and liquid crystals by synchrotron raditation X-ray. It was a static spectroscopy at first, but time-resolved experiments became possible. SAXS is usually used in studies that are concerned with energy materials, display, and semiconductors.

The spatial resolution of SAXS is 1μm ~ 1nm, better than light microscopy but worse than electron diffraction and X-ray crystallography. The control of sample-to-detector distance shifts the range of scattering angle, changing measurable q-range and the q-resolution. In PLS-II, the sample-to-detector distance is controlled by moving the sample. On the other hand, PETRA-III beamline changes the sample-to-distance by changing the position of the detector.

In the SAXS beamline of PLS-II(see Fig. 1), undulator is used as a source of synchrotron radiation due to its high brightness and coherence. Double-crystal monochromator(DCM) is used to select the certain wavelength used for the experiment. The focusing mirror is employed behind the monochromator for focusing of X-ray beam.

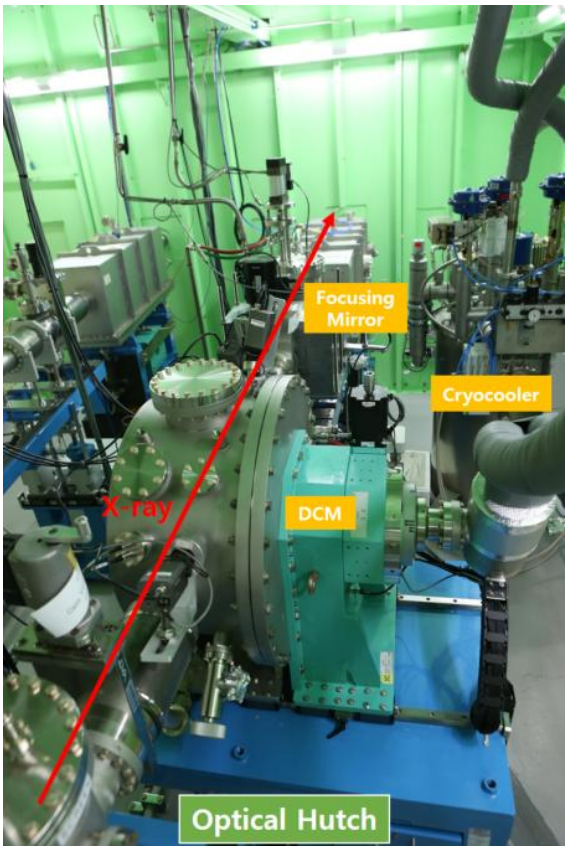

Figure 1: (Excerpt from Ref. [1]) The photograph of optical hutch of SAXS beamline in PLS-II.

There are two methods of SAXS. First, transmission-SAXS(T-SAXS) gains image by scattering X-ray with the sample directly. This method is easy to measure and analyze. However, any possible scattering and transparency of the sample can affect the quality of the image. T-SAXS requires high-energy and high-flux X-ray and bulk/solution sample. Grazing-incidence SAXS(GI-SAXS) gains the image by directing the thin film sample at a low angle. The advantage of GI-SAXS is its high sensibility and easy preparation of the sample. However, the scattering from the surface internal structure can occur. GI-SAXS also requires a special setup for the experiment.

The intensity of the scattering image of the solution can be written as (1):

$$I(q) = Mc(\rho_1 - \rho_2)^2 |F(q)|^2 S(q) \quad (1)$$

where M is the molecular weight, c is the concentration, ρ_1, ρ_2 are the scattering density of the particle and the solvent, respectively, F(q) is the form factor which is determined

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by the shape of the molecule, and S(q) is a structural factor, which is determined as 1 if the solution is dilute. The scattering image converted to a 1D plot through azimuthal averaging for analysis of the data.

EXPERIMENTS

In this section, the SAXS experiment of the bovine serum albumin(BSA) protein solution in static and flow mode was conducted in PLS-II(see Fig. 2). The experiment was conducted in both static mode and flow mode. The static mode is simple to implement. The flow mode can mitigate radiation-induced damage by constantly flowing the sample exposed to the X-ray beam, but the time available for the experiment is limited. Figure 3 shows the instrument for sample delivery for the SAXS measurement. Before the measurement of the SAXS image of BSA protein, a scattering image of air without solution sample and water without BSA have to be shot for background data.

Figure 2: The photograph of SAXE beamline of PLS-II.

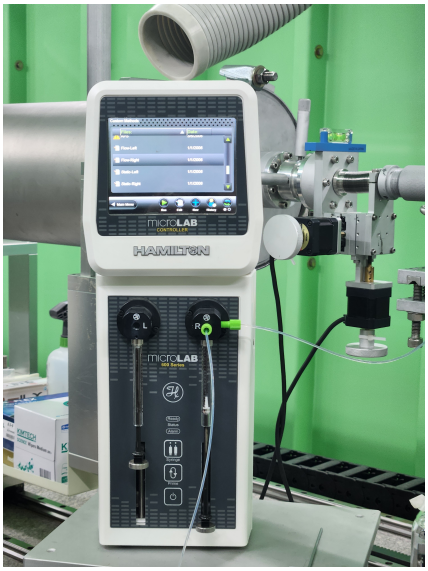

Figure 3: The photograph of the instrument for sample delivery for the SAXS measurement.

In static mode, the exposure time was set to 10 seconds. Ten images were shot in total, and the average radial profile of the scattering images was used for analysis. Fig. 4 shows the scattering image(upper) and the average radial profile of ten images(lower) of BSA protein.

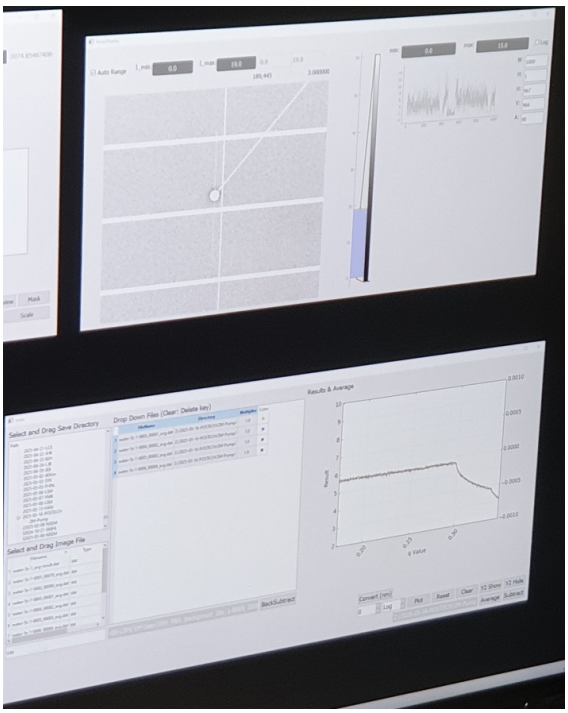

Figure 4: The scattering image(upper) and the average of radial profile of the images(lower) of BSA protein gained in static mode.

In flow mode, the exposure time was set to 5 seconds and 8 images were shot. Fig. 5 is the average radial profile of ten scattering images(lower) of BSA protein.

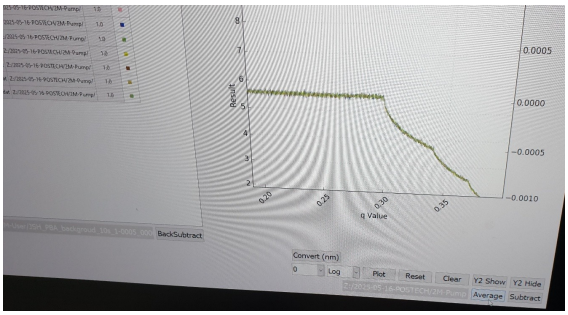

Figure 5: The average radial profile of ten scattering images(lower) of BSA protein gained in flow mode.

Fig. 6 shows the logarithmic plot of the intensity of the scattering image. The black line indicates the static mode, the blue line indicates the flow mode, and the red line represents the background signal respectively. The intensity profile gained by flow mode contained less noise than that gained by static mode. This suggests that mitigating radiation damage to the sample is important.

CONCLUSION

The small angle X-ray spectroscopy is the type of spectroscopy that measures size and shape of the nanoscale mat-

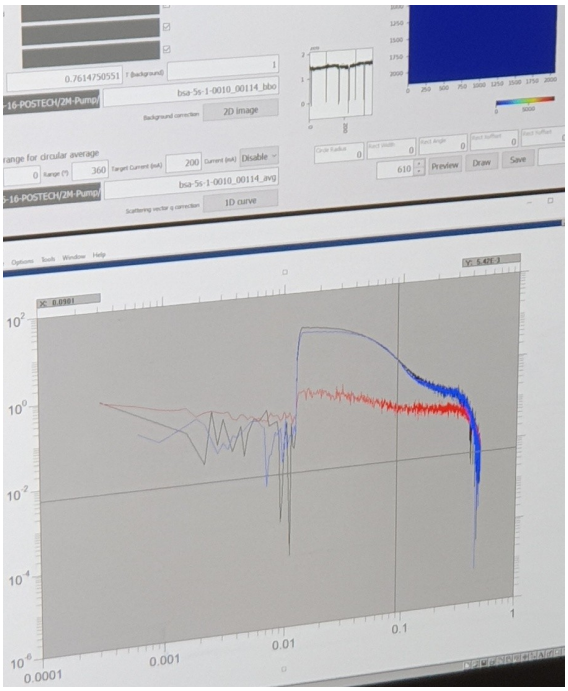

Figure 6: The logarithmic plot of the intensity profile of the scattering image.

ters, such as energy materials, display, and semiconductors. In this report, the principle and setup of the SAXS beamline was introduced. Also, the experiment of SAXS with BSA protein solution and acquisition of the data of the protein by analyzing the radial profile of the scattering image were demonstrated.

REFERENCES

[1] K. S. Jin. *Lecture note on Small-angle X-ray scattering (SAXS)*, Division of Advanced Nuclear Engineering (DANE), POSTECH, Pohang, Korea, May. 2025