Macromolecular Assemblies (BL44XU)

1. Introduction

There exist various biological macromolecular assemblies consisting of proteins, nucleic acids, sugars, lipids, and other substances in living cells. These molecular assemblies play key roles in many biological reaction systems, like protein synthesis, chromosomes, RNA synthesis, DNA synthesis, photosynthesis, respiration, membrane transport, cell adhesion, and cell signaling. More than 10,000 protein structures are now known [1] since the first crystal structure of hemoglobin [2] and myoglobin [3] were determined. On the other hand, only a few macromolecule assembly structures have been determined by X-ray crystallography. This is because of the difficulties faced in the preparation, crystallization, X-ray diffraction measurement, and crystal structure determination of large molecular assemblies. A beamline for biological macromolecular assemblies at BL44XU, which is specially designed to collect high resolution and high quality diffraction data of macromolecule assembly crystals with large unit cells, has been operating since May 1999.

2. Beamline and Optics

A schematic side view of the BL44XU is shown in Fig. 1. X-rays from an in-vacuum type undulator are monochromatized by a rotating-inclining double crystal monochromator in the optics hutch. The monochromatized X-ray beam introduced into the experimental hutch is reflected by a rhodium-coated mirror. The mirror can be used for focusing or collimating purposes as well as for cutting off higher-order harmonics. The total photon flux at the sample position is on the order of 10^{13} photons/sec.

The approximate beam size of the full width at half maximum (FWHM) at the sample position is about 1.0(W) × 0.7(H) mm^2 without focusing. The photon flux after the 70-micron collimator is on the order of 10^{11} photons/sec after focused by a mirror.

3. High Precision Goniometers

Two individual single rotation axis (horizontal and vertical) goniometers are mounted on a goniometer stage, and either of them can be used according to users' requirements.

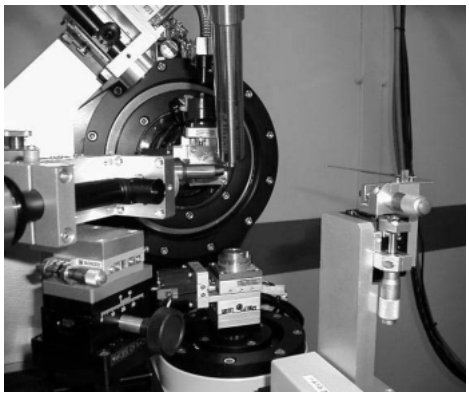

Fig. 2. High precision goniometers.

4. Detectors

Two different type of area detectors can be used at this beamline. One is a 3 × 3 array-type CCD detector, a PX210 (Oxford Instruments, UK), which was originally designed and developed by the detector-design group of Structural Biology Center of the Argonne National Laboratory [3], and the other is an imaging plate system DIP2040 (MAC Science, Japan). The sample to detector distance can be changed from 150mm to 1000mm. The CCD detector unit can be lifted up or brought down between +15mm and -5mm and it can be tilted 0 - 45 degrees to collect higher resolution data.

These detectors and goniometers are controlled by a PC running on Linux as well as the camera and the goniometer stage.

Fig. 3. CCD detector installed in the experimental hutch.

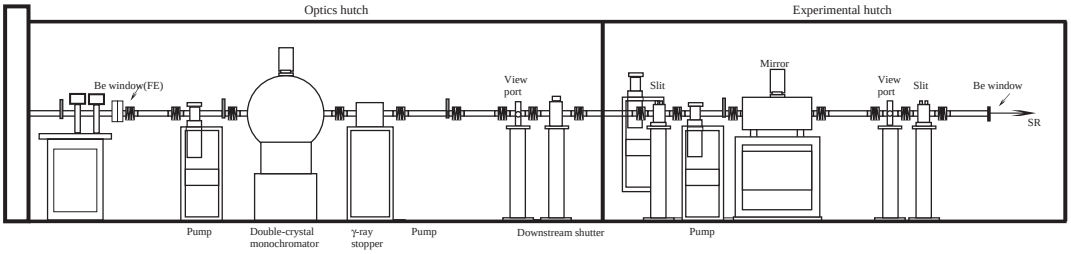

Fig.1. Schematic view of Optics Hutch of BL44XU.

5. Peripheral Devices

A nitrogen gas stream cooler (Niki Glass, Japan) can be used for data collection at cryo temperatures (~100K). This system produces liquid nitrogen for cooling purposes from the air, and it does not require liquid nitrogen supplements.

Several workstations with a large disk storage capacity, which are connected to control computers of detectors with a 10/100BaseT Ethernet, are used for data processing (Fig. 4).

6. Data Collection and Processing

Raw image data are processed on the Linux boxes at the beamline. These workstations are clustered and connected with a 200GB file server by Ethernet. The users can process their diffraction data on these workstations or download raw image files (or processed data) on their PCs via the Ethernet. All data can be saved on the DDS3 tapes.

At present, MOSFLM [5] is used to process DIP2040 data, and d*TREK (MSC, USA) is used to process PX210 data. DPS [6] and/or HKL [7] are possible other choices for data processing.

References

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[4] E. M. Westbrook and I. Naday, in *Methods in Enzymology*, edited by C. W. Carter, Jr. and R. M. Sweet **276** (Academic Press, New York, 1997) 244.

[5] A. G. W. Leslie, in *Crystallographic Computing 5*, edited by D. Moras, A. D. Padjarny and J. C. Thierry (Oxford University Press, Oxford, 1991) 50.

[6] M. G. Rossmann and C. G. Beek, in *Data Collection and Processing, Proceedings of the Study Weekend at the University of Sheffield, 8 - 9 January, 1999).*

[7] Z. Otwinowski and W. Minor, in *Methods in Enzymology*, edited by C. W. Carter, Jr. and R. M. Sweet, **276** (Academic Press, New York, 1997) 307.

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Fig. 4. Data processing workstations at the beamline.

Light Source	
In-vacuum-type undulator	
λ_u	32 mm
N_{period}	140
Wavelength range	7 - 16 keV
X-rays at Samples	
$\Delta E / E$	$< 2\times10^{-4}$
Photon Flux	12^{12} photons/s
Beam size (focused)	130 mm (H)×70 mm (V) (FWHM)
Facilities in Experimental Station	
Goniometer	Two independent axes
Sample translation	±30 mm
Collimator	Double pinhole (100μm, 70μm etc.)
Sample-to-detector distance	150 - 1000mm
Microscope	CCD camera (×250, ×125)

Facilities in Experimental Station	
Cryocooler	Auto N_2 supply ~ 95K
Detectors	
PX-210	3×3 array CCD
Readout	2 sec.
Area size	210×210 mm^2
Pixels	3072×3072
DIP-2040	Imaging Plate
Readout	240 sec.
Area size	400mmϕ
Pixels	4000×4000
Data Processing WS	
OS	RedHat Linux 6.1J/6.2J
CPU	Pentium III 700MHz
Memory	1GB
File server	200GB
Software	MOSFLM (ver.6.01) d*TREK (ver.5.5) CCP4 (ver.4.0.1)