RIKEN Beamline

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1. Introduction

Synchrotron radiation offers a convenient and powerful source for structural studies on biological macromolecules. The brilliance and continuous spectrum of X-rays result in elucidating electron density maps routinely by way of multiple-wavelength anomalous diffraction (MAD) technique and the use of small crystals less than 10 micrometers. Thus, the RIKEN structural biology beamline **I** will be the most powerful beamline to study macromolecular structures. It also has an experimental station of small-angle X-ray scattering (SAXS) to study bio-molecules by utilizing low emittance X-ray beams with the very high small-angle resolution.

The RIKEN structural biology beamline **II** is designed to perform time-resolved crystallography on biological macromolecules by using white radiation from the bending magnet of the storage ring. This beamline consists of two experimental stations, stations for crystallography and the X-ray absorption near edge structure (XAFS) study on biological systems. They are tandemly arranged and used alternatively.

2. RIKEN structural biology beamline I

The protein crystallography (PX) station of the RIKEN structural biology bea mline I utilizes anomalous scattering from intrinsic/introduced heavy atoms in a protein crystal. Instead of using the conventional isomorphous replacement method, the electron density maps of the protein molecule are analytically calculated. In this manner this station is able to cut down its efforts to search heavy atoms, thus minimizing the time needed to determine its structure. The small-angle X-ray scattering on this beamline has a very high small-angle resolution for performing the detailed analysis of scattering data, and it also carries several specimen chambers, especially for the time-resolved studies as described below [2]. In this beamline, the synchrotron radiation from the undulators is split into two beams by a diamond crystal. This design permits the simultaneous operation of ”de facto” two experimental stations, one for protein crystallography and the other for small-angle scattering.

2-1 Insertion devices

In the beamline, two in-vacuum undulators are installed, and they are independently tuned so as to give two different energies to the beamline. The undulators have a vertically polarized magnetic circuit to prevent losing a certain amount of pho- tons by the polarization effect in the hori-zontally reflecting monochromator crystals([1]). The period of magnets is 3.7 cm and the number of periods is 37. Its peak brilliance will be 3.1×10^{18} *photons/sec/mm2/mrad2* in 0.1 % band width (ring curent 100mA is assumed).

The measurement of the magnetic field shows that two insertion devices have the tunable energy range of 4.5~16.0 keV. This covers the actual usage energies in the range of 6~14keV.(Fig.1)

Fig.1: In-vacuum vertical undulator for BL45XU

2-2 Front end and transport channel

Since the RIKEN beamline I uses two insertion devices instead of three for the standard undulator beamline, the standard front

end components are sufficient for this beamline. The baking of the front end system of the RIKEN beamline was finished. (Fig.2)

This beamline has two branches so that the two lines (PX and SAXS) of transport channel are aligned. The distance between these two lines is approximately 65 cm in the horizontal direction. The water and pressurized air supply systems and the connections for electric signal lines for standard components have been completed.

Fig.2: Transport channel of RIKEN beamline **I**

2-3 Diamond fabrication

Since this beamline is the first-branching beamline, the key to the success of branching depends upon the quality of the diamond crystal. As reported by Prof. Ishikawa, the SPring-8 Project Team and the Sumitomo Electric Industry (SEI) have carried out a joint R & D program for years. At the end of the last fiscal year we were able to produce a crystal of 7×6.5 mm and 7×3 mm for (100) and (111) respectively with an excellent crystallinity for the beamline application. The thickness of (100) and (111) crystals are 1.0 mm and 0.5 mm,respectively. The half widths of rocking curves are 2 sec and 6 sec for (100) and (111) crystals, respectively.

2-4 Non-standard components

The RIKEN beamline **I** has the following non-standard components; a diamond monochromator and a diamond trichromator. The diamond monochromator uses one pair of (111) diamond crystals. The goniometers of the monochromator employs the same mechanism as the trichromator.

The diamond trichromator has arrived from Sigma Koki Co. Ltd., and has been installed in the transport channel. (Fig. 3).

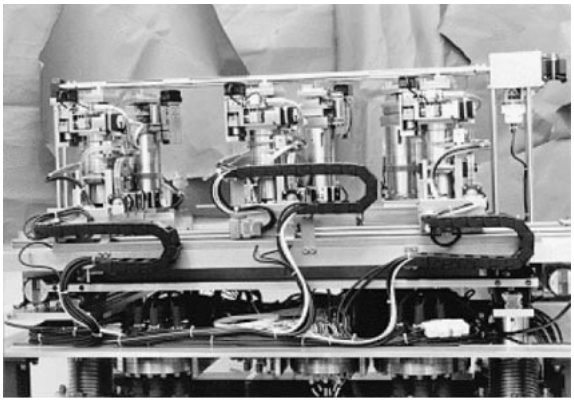

Fig.3 Side view of trichromator

Three pairs of (400) diamond crystals will be used. The movement and precision of all axes were checked by using an laser interferometer. The design of the crystal holder is still in progress.

2-5 Radiation shielding and interlock system

The construction of the radiation shield hutch has been completed. The control system such as the beamline master workstation, pulse motor driver controls, and the interlock systems are placed on the upper deck of the radiation shield hutch. Since the RIKEN beamline **I** shares one door at the optical hutch with the RIKEN beamline **II**, the safety interlock system independently monitors the opening status of the door. The PX and SAXS station are used simultaneously in such a manner that each interlock system can work independently.

2-6 Experimental stations

In the SAXS station, the sample cell is on the X-Z stage and its position is fixed facilitating the introduction of various types of sample holders. The stopped-flow, temperature jump, and high pressure stopped-flow apparatus will be prepared. The camera length can be changed by exchanging various lengthes of vacuum tubes, so that the detector stage is designed to be movable. At the initial stage, 0.5m and 1.5 m

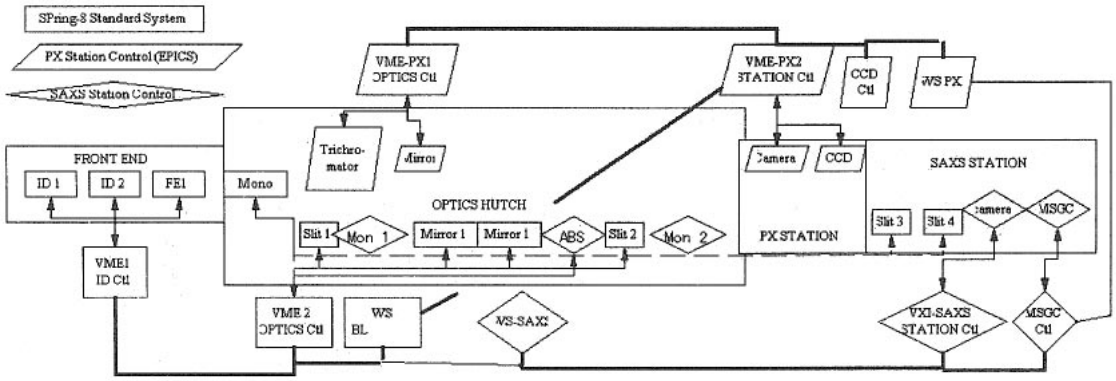

Fig. 4 Control Diagram of RIKEN beamline I

camera lengthes can be used. The fast real-time 2D detector, the microstrip gas chamber (MSGC), are used for data collection [5].

In the PX station, a five-axis horizon- tal type goniometer is employed. In order to minimize radiation damage from incident X-ray beams, the cryo-cooling system is employed . The scintillation counter will be prepared in order to measure the EXAFS spectrum for the MAD technique. A multiple array CCD X-ray detector (MCCDX) will be used [6].

2-7 Beamline control

The design of the standard SPring-8 control dictates all optical components such as the mirror, monochromator, and shutters. The SAXS control is based on the SPring-8 control system. For the SAXS station control, a VXI-based control system will be used, which permits the flexible use of various experimental modules. The control code will be written in the LabVIEW. As for the PX line, the trichromator needs 30 axes to be controlled, and a very complicated sequential control is required in order to collect MAD images. The PX line, therefore, employs a non-standard control system, EPICS program packag es [7].

A summary of the control diagram is shown in Fig.4. This station can greatly contribute to the expansion of the protein structure database, which can lead to the improved design of drugs and advances in protein engineering in the near future.

The small-angle X-ray scattering reflects the wide range of electron density fluctuation of macromolecules in solution from several nm up to submicron. The great advantage of the SAXS is its ability to observe the behaviors of structures under various environmental changes in real time [2]. The protein function in terms of its size will be delineated by the direct observation of protein macromolecules under high pressure. This should clarify the process in which the protein reaction is controlled under pressure. Although these two methods are very pop-ular, there are some other methods as well.

3. RIKEN structural biology beamline II

The use of the high flux third generation synchrotron radiation has recently made it possible to develop a science of the time-resolved protein crystallography (TRPX). The TRPX permits the display of protein molecule movements in crystals with the atomic resolution. Another important tool for biochemistry is the X-ray absorption spectroscopy at the absorption edge of heavyatoms (XAFS).

At RIKEN much research has been conducted on the photo-system **II** and heme proteins which contains the metal ion. Obtaining informations on metal ions status during function is essential to understanding the electron transfer in above system. In the XAFS station, excellent data are expected even with a dilute concentrations of heavy atoms. In order to cover these two methods, RIKEN started to construct the

RIKEN beamline **II** for XAFS and TRPX. This beamline uses a bending magnet source in order to make full use of its broad spectrum feature.

Two experimental stations are aligned in tandem and use both white and monochromatic X-rays. The detailed design of the RIKEN beamline II will be described in a separate report as part of this Annual Report.

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