RIKEN Beamlines

1. Introduction

In the previous fiscal year, RIKEN finished commissioning of two beamlines (BL45XU and BL44B2) for structural biology.

RIKEN also started in FY 1997 new construction of an undulator beamline at BL29IN for synchrotron radiation physics, in particular for the instrumentation needed for the extensive use of highly coherent X-rays. The location of BL29IN provides the possibility of extension to 1 km. However, the first phase construction started as a standard length beamline inside the experimental hall of the storage ring building. The beamline was completed as BL29XU in November 1998, followed by a series of commissioning tasks between January and March 1999.

In the first supplemental budget of FY 1998, construction of a 30 m undulator and its accompanying beamline were approved as a three year project. There are only four long straight sections in the storage ring to accommodate the 30 m undulator, so the beamline is a very valuable one for the entire SPring-8 facility. After some discussion, it was decided that the location of the beamline would be BL19IS, which can be extended to a 300 m medium length beamline. It was also decided that the energy region to be covered by the undulator would be X-rays because of the technical status to manage the coherent radiation at that time.

Extension of BL29XU was approved for construction as a two-year project in the third supplemental budget of FY 1998. The project includes construction of a transport channel, an experimental station at the 1 km site, and a building to accommodate the experimental station.

2. RIKEN Beamline I (BL45XU)

2.1 Trichromatic MAD Station of BL45XU

In structural biology, three-dimensional knowledge of biological macromolecules is indispensable for understanding biological functions. The compilation of three-dimensional structures, at atomic resolution, would play an important role in protein engineering and drug design.

The introduction of synchrotron radiation has accelerated the accumulation of three-dimensional structures. Furthermore, tunability over a wide energy range of synchrotron radiation has led to the development of the multi-wavelength anomalous diffraction (MAD) method [1], which gives phases from a single anomalous scatterer [2]. Utilizing the third generation SR with the maximized advantage of the MAD method, BL45XU is designed to contribute to the structural biology of RIKEN.

Anomalous scattering contributes minimally, so that the accuracy of the intensity data collection is technically the most important issue. To realize such an experimental environment for MAD, the “trichromatic concept” has been introduced by developing high quality diamond crystals [3,4]. This concept is based on the fact that MAD data sets at three different wavelengths are easily taken for the same protein crystal without changing the setting by a tandem vertical undulator and trichromator.

We finished the initial construction of the beamline at the beginning of 1998, and the trichromatic undulator beams have been utilized for MAD data collection. Initially, an imaging plate detector (RIGAKU R-AXIS IV) was installed and used for MAD data collection [5]. We have collected MAD data sets including several anomalous compounds (zinc, mercury, selenium, etc.).

The crystal of blasticidin S deaminase (BSD) was analyzed as the first sample. BSD is an enzyme that includes one Zn per one molecules (M_w = 13 k Dalton), and a homo tetramer of BSD is included in an asymmetric unit. The XANES spectrum of BSD clearly showed the absorption edge of a Zn atom. Four Zn positions of an asymmetric unit were clearly shown in the anomalous and dispersive difference Patterson maps, and the MAD phased initial electron density map, which clearly showed the secondly structure and side chains, was smoothly calculated. The model building and the refinement of BSD were progressed satisfactorily [6].

On the other hand, a dichromatic diffraction experiment by using two wavelengths at the same time was carried out as a more efficient use of the trichromator. Two different wavelength diffractions at the remote and the edge of an anomalous scatterer were exposed on the same imaging plate, and one more wavelength diffraction was exposed on the other imaging plate. The dichromatic diffraction images were processed by using the ordinal indexing software. Moreover, the anomalous and dispersive difference Patterson maps clearly showed the position of the Zn atoms, and the structural analysis was sufficiently progressed. This result shows the advantage of coupling of trichromator and the imaging plate detector.

By the end of 1998, we had already determined seven new protein structures, which included two more Zn protein crystals, two Hg derivative crystals and one Se-Met crystal.

2.2 The Small Angle X-ray Scattering Station of BL45XU

The small angle X-ray scattering (SAXS) station at BL45XU is designed to perform solution X-ray scattering and fiber diffraction of biological systems.

In 1998, we have replaced two components: the bending block of the mirror and the first crystal holder of the monochromator. The goniometer for the mirror previously placed above the mirror chamber was changed to a position under the mirror chamber. The vibration of the mirror was reduced, and the exchange of mirror material was greatly facilitated. In March we finally achieved good focusing in both vertical and horizontal directions. The beam size at the focal position was measured to be 0.35 mm × 0.15 mm (2.35 σ) by a beam profile monitor (Hamamatsu Photonics). As for the monochromator, we initially used diamond 111 in the Bragg geometry of (111) faced synthetic diamond (Sumitomo Electric Industry) with a dimension of $7 \times 3.5 \times 0.5$t mm^3 as the first crystal. In September, the Bragg geometry was replaced with Laue geometry of (100) faced synthetic diamond with $8 \times 7 \times 0.3$t mm^3. The advantages of using Laue geometry rather than Bragg geometry are: (a) a reduction in the absorption of the transmitted beam, (b) availability of large crystals compared to that of (111) faced crystals, and (c) a reduction in the footprint size of the first crystal, which enables the full acceptance of incident beams. We were concerned that the high heat density from reduction of the footprint size might introduce instability in the beam, however, no differences in stability before or after Laue geometry replacement was observed [7,8]. In the latter part of 1998, we focused on exploring the detector characteristics (X-ray image intensifier with CCD (XR-II+CCD)), especially for use in solution X-ray scattering. Two samples, polystyrene latex and apoferritin, were used. The profiles were submitted to various analyses, including Guinier analysis, power law analysis, size distribution analysis, and the calculation of radial electron density distribution. The practical protocol for usage of XR-II+CCD has been established [9,10].

The SAXS station accepted public users within 20 % of total beamtime. In 1998A, there were seven proposals, which included two proposals in polymer science. Most proposals dealt with diffraction systems rather than solution. The number of public users increased to 13 in 1999A. The beamtime for each proposal ranged from three to six shifts. Since the beamtime is rather tight, public users who want to be this station are requested to contact Dr. T. Fujisawa before submitting a proposal.

3. RIKEN Beamlines II (BL44B2)

3.1 Optics

RIKEN Beamline II (BL44B2) is dedicated to macromolecular crystallography in white (Laue) and monochromatic X-ray modes and to XAFS experiments in diluted biological materials. White x-rays from a bending magnet are focused with a 1 m long platinum-coated bent cylinder mirror (Laue mode) or monochromatized with a fixed-exit double crystal (Si(111)) monochromator that is then focused with the mirror (monochromatic mode). The glancing angle (2-5 mrad), the radius of curvature (3 - 7 km), and the vertical position (± 15 mm) of the mirror are adjusted for the optimum focusing at the sample position. The photon flux at the sample position is estimated to be 10^{15} photons/sec (7 - 27 keV, Laue mode), and 10^{12} photons/sec in 0.1 % b.w. (at 20 keV). In the monochromatic mode, diffraction or XAFS experiment is feasible in the energy range of 6 - 30 keV and energy resolution of $\Delta E/E \sim 2 \times 10^{-4}$ (20 keV).

3.2 Diffraction Station

Either an on-line IP (R-AxisIV, Rigaku) or CCD (marccd165, Marresearch) system is available as the two-dimensional X-ray detector. The CCD detector has a circular shape and a diameter of 165 mm; the crystal-to-detector distance can be set from 80 mm to 400 mm. The readout time of the CCD system (3.4 sec) is much shorter than that of the R-Axis IV (4.5 min), and this is a real advantage for effective data collection. For example, typical data collection can be done within 30 minutes with the CCD, while 7 hours are required with the R-Axis IV.

A cryostat system using cooled nitrogen gas is equipped at the beamline, and samples can be cooled down to 80 K with the system. We are planning to install another cryostat system with cooled helium gas which can cool samples down to 40 K.

Micro-spectrophotometers, a pulse Nd: YAG laser, a YAG-pumped dye laser, and some optical components (mirrors, lenses, *etc*) are equipped at the beamline and these can be used for optical monitoring and initiation of reactions in the crystals.

We collected more than 50 datasets so far in the monochromatic mode, including high resolution datasets (up to 0.85 Å resolution), MAD datasets (Fe K, Pt L, and Ir L edges), and reaction-intermediate datasets. Data analyses are in progress and some brand-new structures will be published in the near future. Time-resolved Laue experiments were performed as well, and optimization of the reaction conditions in the crystals is in progress.

3.3 XAFS Station

A 19-element Ge SSD (EG&G Ortec) was installed for fluorescence XAFS measurement of diluted biological samples. Signals from the SSD are processed with digital X-ray processor boards (DXP, XIA), which can perform pulse height analysis at a high counting rate (up to 100 kcps), and the XAFS measurement system is controlled by PC-based software (LabVIEW5.0).

Tests of the XAFS data collection system for optimized energy resolution with the SSD and DXP boards are almost finished, and data collection and analysis of diluted biological samples are in progress.

4. RIKEN Beamline III (BL29XU)

4.1 Light Source, Transport Channel and Optics

The light source of this beamline is a standard in-vacuum undulator with 32 mm magnetic period [11]. The front end and the transport channel are also of standardized undulator type of SPring-8 [12,13]. A double crystal monochromator is installed in the beamline [14]. This beamline has two connected radiation shielding hutches for optics and an experimental station. Safety interlock and beamline control systems are the same as those in other standard X-ray undulator beamlines in SPring-8.

4.2 Experimental Station

BL29XU has one experimental station that is covered by a radiation shield hutch with a volume of 5(D) x 3(W) × 3.3(H) m^3. The upstream surface of the hutch lies at 51 m from the source. The hutch has eight cable ducts on the ceiling and the wall, and one of them is used for a laser introduction path from the laser booth outside. An air conditioner stabilizes the room temperature within 0.1 K. An additional insulating cover over the instruments can achieve one order better temperature stability.

We have two movable optical benches, one for X-ray goniometers and the other for the visible optical elements. The X-ray bench is 2(D) × 1(W) m^2 and has six goniometer mounts that translate along and perpendicular to the optical axis. The bench for the laser has a size of 2×1.5 m^2 and can be set with many mirrors and lenses.

Various stepping motor driven stages, goniometers, and diffractometers are available, such as rotational-, swivel-, and translation-stages, θ-, coaxial-θ-, 2-axis-θ-goniometers, and χ–ϕ–ω–θ-diffractometers. The finest θ-goniometer has a resolution of 1/400 arcsec/pulse with good stability. Prepared detectors are NaI scintillation counter, ionization chamber, PIN photo diode, APD, SSD, CCD camera, and streak camera.

The goniometers, detectors and other instruments are controlled by the computers. Data acquisition is based on the commercial NIM module system. The basic counting and timing modules are installed. To get the timing of X-ray pulses, the RF signal is delivered from the RF station via a small jitter cable. A VME system will be added in the near future to obtain better control for a 1 km beamline.

4.3 Laser Synchronization System

A synchronization system of an intense short pulse laser to the hard X-ray synchrotron radiation (SR) pulses has been developed for research on nonlinear optical processes, a pump-probe experiment, and coherent harmonic generation using electron beams. A regeneratively amplified mode-locked Ti: sapphire laser in class IV is installed in a booth constructed near the experimental hutch, to which the output laser beam is guided through a duct with steering mirrors. The 2 ps laser pulses are synchronized to the SR pulses by locking the laser to the RF frequency of the ring. By applying both the laser and SR beams simultaneously on the same gold photocathode of a streak camera, the synchronization was monitored and found to be achieved with a precision of a few ps.

5. RIKEN Beamline IV (BL19IS)

5.1 Light Source, Transport Channel and Optics

The light source of this beamline will be a 30 m undulator, the details of which are in the section entitled “Insertion Devices” in this volume. The installation of the undulator requires rearrangement of ring magnets in the straight section. Extensive studies were made by the Accelerator Group to find solutions.

Since the expected minimum gap of the magnetic arrays of the undulator is larger than that for a conventional 4.5 m undulator, heat load on the first optical element is comparable to that in a conventional X-ray undulator beamline. Therefore, the transport channel and optics of this beamline were designed by combining standard components for conventional X-ray undulator beamlines. A new cryogenic cooling system, described in the section entitled “Transport Channel and Optics” in this volume, will be used for the crystal monochromator. Special attention was given to the preparation of the mirror-polished Be window to preserve spatial coherence of the X-ray beam.

5.2 Experimental Stations

This beamline will have three tandem experimental stations. The sizes of the first and the second hutches will be 5 m (along the beamline) × 3.3 m × 3.3 m (height), and that of the third one will be 5 m × 3.3 m × 4.5 m. The first station will be equipped with a versatile precision diffractometer, which is designed to be nearly the same as that at BL29XU in order to maintain compatibility. For precise experiments, the temperature in the hutch will be stabilized with an air conditioner. The second station will be an open hutch for general and public use. In the third one, a superconducting magnet and a multi-axis diffractometer will be installed to study diffractions under a high magnetic field. The detecting and data acquisition systems, as well as the control system, will be identical to those at BL29XU.

6. 1 km Extension of BL29XU

Design study of the extended transport channel of

BL29XU, as well as a building to accommodate experimental stations at the 1 km site, was carried out. Two-storied vacuum ducts will connect the existing experimental hutch in the storage ring building and a new experimental hutch to be constructed in the 1 km building. Between the two buildings, the beam ducts are placed in the open air except when they are inside 64 distributed covers for the pumping units. The height difference of the two-storied beamlines is designed to be 1 m.

The 1 km building has a 300 m^2 experimental hall, in which a 24 m^2 shielding hutch will be constructed. A part of the hall floor, where the shielding hutch is placed, is cut away from other parts of the floor to prevent vibration. Two preparation rooms (each 55 m^2) are located next to the experimental hall.

All hardware, including the building, will be completed within FY 1999. The commissioning of the beamline is planned to start from the beginning of FY 2000.

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