Bio-Crystallography I (MIR-OAS) Beamline -- Fundamental Design --

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Introduction

The SPring-8 project team approved our proposal as one of two pilot beamlines [1]. The pilot beamlines are to be constructed for standardizing many elements of beamlines. The process of the standardizations are reported in other papers in this volume. We report, in this paper, the research subjects of our beamline in macromolecular crystallography and the comprehensive design concept.

Research subjects

Multiple isomorphous replacement (MIR) is a widely used technique to resolve the phase problem in macromolecular crystallography, but it involves trial and error in the isomorphous derivative search. The derivative search has been a time consuming process and the routine structure determination by the MIR method has been difficult.

The in-vacuum type undulator installed in the SPring-8 storage ring gives the sufficiently brilliant X-rays over a wide energy range [2]. Higher-energy X-rays over 20 keV are useful for measuring reflection data from native crystals with higher resolution. The X-rays are also useful for conducting intensity measurements from the heavy atom derivatives without any anomalous dispersion effects. On the other hand, lower-energy X-rays between 9 and 18 keV are useful for measuring the reflection intensities near L3 absorption edges of heavy atoms from ytterbium to uranium including anomalous dispersion effects. By combining these two types of intensity measurements, it will be possible to determine the phase angles of the native crystal with a smaller number of derivatives than that usually used. This is the MIR method with optimized anomalous scattering (MIR-OAS), recommended by Helliwell [3].

The purpose of our beamline is the routine analysis of macromolecular crystallography by the MIR-OAS phasing. Users will be able to soak the macromolecular crystals in typical reagents of heavy atoms, and conduct preliminarily checks using X-ray equipment in a preparation room. About 20 candidates for the heavy atom derivatives can be prepared in this

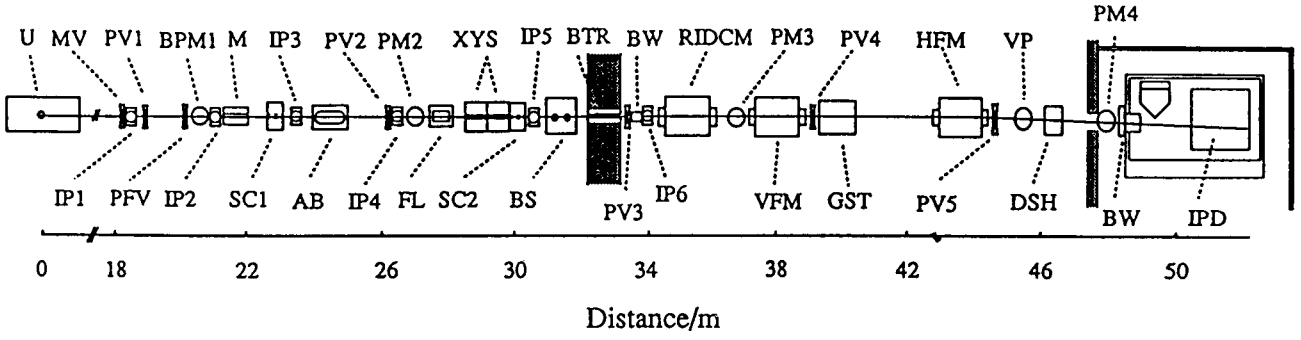

Fig.1 Schematic over view of the beamline. U: undulator, MV: manual valve, IPn: ion pump, PVn: pneumatic valve, PFV: pneumatic fast valve, BPMn: beam position monitor, M: mask, SCn: shield collimator, AB: absorber, FL: graphite filter, XYS: X-Y slit, BS: beam shutter, BTR: beam transport, BW: Be window, RIDCM: rotated-inclined double crystal monochromator, VFM: vertically focusing mirror, GST: γ-ray stopper, HFM: horizontally focusing mirror, VP: view port, DSH: downstream shutter, IPD: imaging plate detector. Suffix: n is component number.

way, and measured continuously including the native crystal with high-energy X-rays of 27 or 38 keV. If one or two heavy atom derivatives are included in the 20 candidates, an electron density map can be obtained by the MIR-OAS phasing after some supplemental data collections with the heavy atom derivatives near the L3 absorption edges.

We consider that the ultimate goal of macromolecular crystallography is to resolve the three-dimensional structures of most of the macromolecules in living cells. To reach the ultimate goal as fast as possible, we must eliminate other limitations in macromolecular crystallography. The remaining limitations are the size of sample crystals and the molecular weight of macromolecules. The high-energy and high-brilliance characteristics of our beamline are also useful for structure determinations with the sample crystals less than 50 mm size and with those of supra-complex such as ribosome particles with the molecular weights over several millions.

Design concept of the beamline

Figure 1 shows a schematic over view of the beamline. An in-vacuum type undulator with a magnetic periodicity of 3.2 cm will be installed in the storage ring. Fundamental and third-harmonic emissions will be obtained at brilliance over 10^{19} photons/s/mm^2/mrad^2/0.1%b.w. at the storage ring current of 100 mA. The energy range for the fundamentals is 9 to 18 keV, and the corresponding range for the third-harmonics is 27 to 54 keV.

For constructing undulator beamlines at the SPring-8, one of the major problems is to cope with the high power load from the light sources. The power and power density are up to 5 kW and 300 kW/mrad^2, respectively. Front end elements which could potentially be irradiated directly with the undulator light are all water-cooled and a sufficient number of measures will be taken to avoid accidental melt down or disruption [4].

To handle the tremendous power density of the undulator light, a rotated-inclined double crystal monochromator [5] will be installed at a distance of 35 m from the light source. The grazing incidence diffraction with a variable glancing angle will be used for the first crystal. When the glancing angle is set to one degree, the power density on the monochromator surface is reduced to 1/60 of that for normal incidence. The power density on the crystal surface is estimated to be less than 5 W/mm^2. The pin-post water cooling [6] of classical silicon (111) crystal may overcome this value.

Since the integrated intensity of X-rays from mosaic crystals is well known to be inversely proportional to the second power of X-ray energy, a focusing system for the high-energy X-rays is indispensable to obtain a high flux at the sample position. To meet the requirement upon the reflectivity and the focusing of the X-rays of up to 38 keV, two super mirrors (Ovonic Synthetic Materials Co.) will be introduced.

In the experimental station, as shown in Fig.2, data collection will be performed by using a screen-less Weissenberg chamber originally developed by Sakabe [7]. The Weissenberg chamber is composed of a goniostat on a three-dimensional stage and an imaging plate (IP) detector [8]. Because the high-energy X-rays cause the Weissenberg patterns to shrink, we need a long distance up to 1.5 m between the sample and the detector to expand the intervals between reflections. Large IPs over 400 x 600 mm^2, which result from the long camera distance, require a long readout time stretching over several minutes. For continuous and on-line data collections required in the MIR-OAS routine analysis, however, the readout time should be less than one minute, which is the longest exposure time expected on our beamline. In order to overcome this problem, an R&D program is in progress to develop a fast readout mechanism of the large IP by using charge-coupled device [9].

The experimental station further includes a XAFS apparatus and a cooling system for specimen. The XAFS apparatus itself determines suitable values of X-ray energy for the data collections with the heavy atom derivatives near its L3 absorption edges. The cooling system is useful to preserve the lifetime of the sample crystals [10].

When our beamline is constructed as stated above, rapid and on-line data collections should become possible. To make this beamline more effective, a software system dedicated to the

MIR-OAS routine analysis should be developed [11].

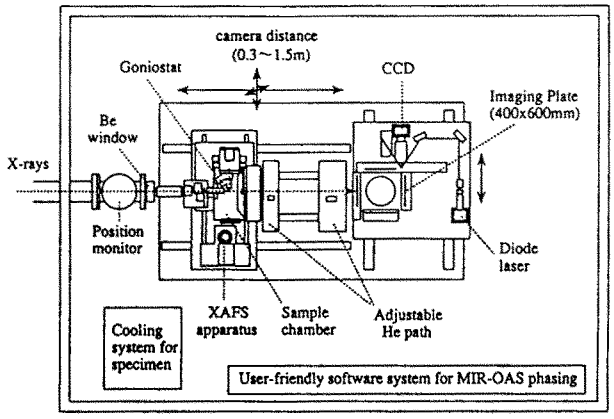

Fig.2 Schematic drawing of the experimental station.

Reference

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