High Energy X-ray Diffraction (BL04B2)

1. Introduction

The BL04B2 beamline is designed for diffraction and scattering experiments involving an energy, more than 38 keV, for various kinds of materials. The construction on the major part of the beamline is scheduled to finish in June 1999 and test operations are expected to start after that.

2. Beamline

A schematic view of the beamline optical components is shown in Fig. 1. The light source is the SPring-8 bending magnet, which has an angular divergence is 0.73 mrad. A bent crystal monochromator utilizing X-ray focusing at the sample position is placed at 45 m position from the beam source. The Bragg angle of the monochromator crystal is fixed at θ = 3 degrees in the horizontal plane. Using Si(111), Si(220), Si(311), Si(511) reflections, it is possible to tune X-ray beam higher than 38 keV. At the focusing point in the experimental hutch, the expected energy resolution ΔE/E is about 10^{-4} and photon flux is about 10^{12} photons/sec.

3. Experimental Facilities

Four kinds of experimental equipment are located in tandem in the experimental hutch as shown in Fig. 2. The descriptions of equipment are as follows.

3.1 Two-axis Diffractometer for High Energy X-rays

High energy X-ray diffraction at the synchrotron source can provide users the ability to study the structures of encapsulated liquids and glasses in their transmission geometries with great advantages: a higher resolution in real space due to the wide range of momentum transfer Q, smaller correction terms, especially for absorption correction, a reduction of truncation errors, the possibility of operation in extreme environments (high and low temperatures, high pressure), and a direct comparison between X-rays and neutron diffraction data.

For amorphous material diffraction experiments, a high energy X-ray diffractometer with a horizontal scattering plane has been installed at BL04B2. Heavy sample environment can be mounted at the sample position. On the sample rotation (ω), a horizontal and vertical translation table allows a precise positioning of the sample in the beam. Two 2θ arms exist for scattered photon counting: 2θ step scans using a NaI scintillation counter or a Ge solid state detector for normal diffraction experiments and area detection using a position sensitive detector for high-Q diffraction experiments. A special setup is available for the sample stage. In this case, a large vacuum chamber can be mounted on the table in order to reduce the background due to air scattering.

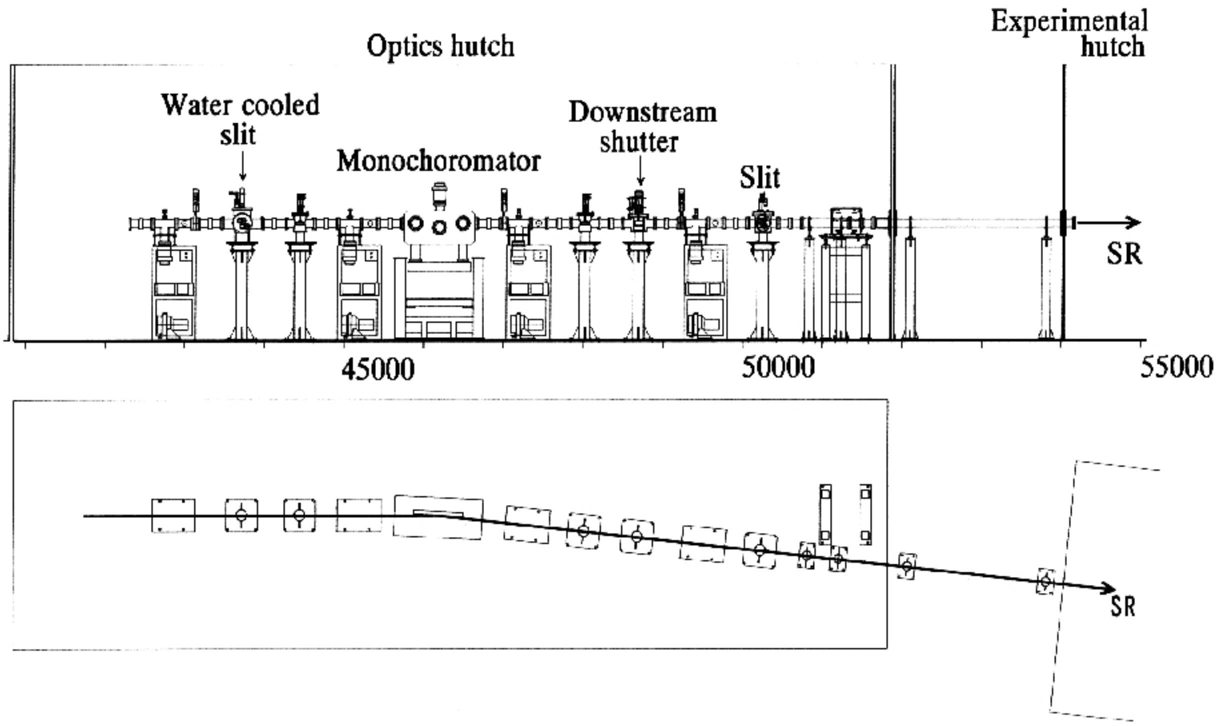

Fig.1. A Schematic view of the beamline components.

3.2 Single Crystal Diffractometer with a Cylindrical Imaging Plate

This diffractometer is designed for the structure analyses of microcrystals of small molecules using high energy monochromatic X-rays. The highly brilliant X-rays in the high energy region of the BL04B2 beamline assures us of the highly accurate structure analyses of microcrystalline materials. The diffractometer, manufactured by MAC Science, is equipped with a three circle goniometer and a cylindrical imaging plate. The three circle goniometer, which has ω, κ, and ϕ axes, enables versatile orientation of the crystals, giving us chances to collect data with high completeness and high redundancy. The cylindrical imaging plate has a sliding mechanism, enabling the Weissenberg motion. The data collection, including the successive integration of each frame, and the scaling, will be performed automatically.

3.3 High Pressure Vessel

Small angle X-ray scattering spectra for supercritical metallic fluids are measured using a monochromatized X-ray beam with an energy of 38 keV. The scattered X-rays are detected using imaging plates with a camera length of about 4,000 mm. The observable momentum region is from 0.05 Å to 0.67 Å. The supercritical conditions of metallic fluids at high temperatures and pressures are achieved using an internally heated high pressure vessel and a sapphire cell of our own design. Figure 3 shows a side view of the high pressure vessel. The vessel is made of superhigh tension steel and it permits measurements up to 1,700 ˚C and 2,000 bar. The incident and scattered X-ray beams pass through diamond and Be windows, respectively. The sapphire cell and heating assemblies are set up at the center of the vessel as shown in the figure. The vessel is pressurized by a high purity grade He gas. Permission had been obtained from the government to install the high pressure gas apparatus including the vessel and a compressor.

3.4 Imaging Plate System for High Energy X-ray

This system is a standard type diffractometer that uses an imaging plate detector (RAXIS IV++, Rigaku); a diamond anvil cell (DAC) for high pressure experiments will be mounted mainly. This diffractometer loads four sheets of imaging plate, two of which are of the normal type of 100 × 100 μm resolution (pixel size), and two of which are of the high resolution type with 50 × 50 μm resolution (called blue IP). Users can select the IP type for their experiments.

The X-ray optics components consist of 4-blade xy slits made from a 5 mm thick Ta plate, an ion chamber as a direct beam monitor, a fast X-ray shutter with Pt attenuators, a guide block of a pinhole collimator, and a θ-goniometer with a microscope for alignment. The Silicon Graphics computer controls automatic IP reading procedure, sample oscillation, shutter/attenuator motion, camera distance changes from 0.2 m to 1.5 m, and the total X-ray diffraction measurements procedure including data collections.

This IP detector will work in the small angle experiments using the above-mentioned high pressure vessel.

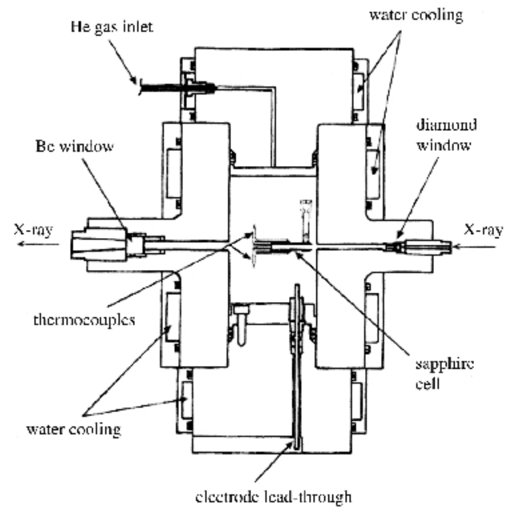

Fig. 3. A side view of the high pressure vessel.

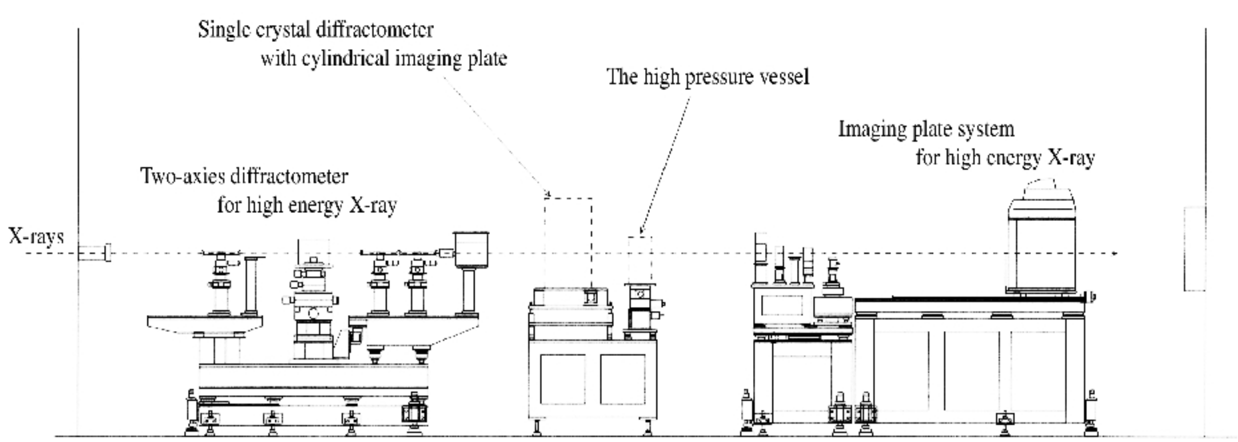

Fig. 2. Experimental facilities in the experimental hutch.