Crystal Structure Analysis (BL02B1)

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1. Introduction

BL02B1 was amongst the first ten public beamlines to be built at SPring-8. It was the first beamline to reach the end of its construction phase and commissioning started in May 1997. Start-up experiments to check the beamline performance were carried out before the summer shutdown and the beamline opened for public use in October 1997.

2. Outline of BL02B1

BL02B1 is a standard bending-magnet beamline for research involving diffraction and scattering in the energy range of 5-90keV. Its design is similar to the XAFS BL01B1 beamline. The schematic configuration has been described elsewhere [1]. BL02B1's main optics contain two bent-plane mirrors and a double crystal monochromator with sagittal focusing system. The mirrors are coated with platinum.

In the low energy region (E<30keV), the X-ray beam is vertically collimated by the first mirror before the monochromator, thereby producing high-energy resolution with high-photon flux. The second monochromator crystal and the second mirror after the monochromator respectively focus the X-ray beam in the horizontal and vertical planes at the sample position. Double mirror reflection should eliminate higher harmonics.

In the high-energy region (E>30keV), these mirrors will be removed from the optical axis. Higher harmonics elimination can then be accomplished by detuning the double crystal monochromator and focusing the beam in the horizontal plane using only the second crystal.

The monochromator's adjustable inclined geometry allows the vacuum in-situ interchange of the diffraction planes produced by a pair of monochromator crystals. A user can select a Si(111), (311) or (511) diffraction plane, depending on the most suitable energy and resolution.

An outline of the experimental station, together with some apparatus designed by the SPring-8 users group, has been reported in [2]. The main station in the experimental hutch incorporates a specially designed diffractometer. There is also equipment to meet a range of user requirements.

3. Present Status of Optics and Experimental Station

3.1. Optical elements

The beamline mirrors have been available since the test operation in February 1998. These mirrors produce a focused beam size of about 0.1 mm in vertical plane at the sample position. They intensify photon flux in accordance with expectations and results of systematic examination of their performance will be reported soon.

The monochromator is operational but requires precise tuning using a piezo translator. Precise tuning studies started recently.

The monochromator's present horizontal focusing ability fell short of expectations. This was a result of crystal distortion associated with the monochromator crystal

mounting. Efforts are now being made to improve the horizontal focusing.

3.2. Experimental station and apparatus

(1) Diffractometer

As has been described elsewhere, the mechanical performance of the diffractometer is satisfactory [2,3]. But the operational software is incomplete, restricting the diffractometer's scanning method capabilities. Consequently it will be particularly difficult to carry out conventional structure analysis for some time.

(2) High pressure experiments

Research subjects at BL02B1 include structure analysis under high pressures using a diamond anvil high-pressure cell (DAC). A telescope with a CCD camera can be attached to the diffractometer in order to put a sample in DAC at the center of the experimental optics. It is a very powerful tool when a sample is very small like 10 μm size.

We plan to install a pressure measurement system soon. This will use a ruby-fluorescence technique to measure pressure in the DAC sample chamber.

(3) Temperature control

Two cryostats were installed at BL02B1. One is for structure analysis experiments, using a half-sphere Be window, to simplify absorption correction. The other is for large samples, such as a powder pellets. This will use a cylindrical Be window and include provisions for the installation of DAC. Off-line tests have already confirmed that it possible to achieve a temperature of about 8K. On-line tests are now being planned, but it will be some time after summer shutdown in 1998 that they are available to users. An electrical furnace with a half-sphere Be window is also prepared for the high temperature region, 300-1000K. Off-line tests are being planned.

(4) Imaging plate cameras

The vacuum camera comprises a cylindrical vacuum chamber in which an imaging plate is attached to the inner side for applying oscillation photographic methods. It can be mounted on the diffractometer's ϕ-table. Many experiments have been already performed.

A Weissenberg photograph system using an imaging plate can be also be attached to the diffractometer. The results of test running were not satisfactory as we could not reproduce the reciprocal lattice from a photograph. Further adjustments, improvements and studies will be necessary for practical application of this system.

References

[1] For example, SPring-8 Ann. Report, 84-85(1996).

[2] Y. Noda *et al.*, J. Synchrotron Rad. **5** (1998) in press.

[3] Y. Noda *et al.*, SPring-8 Ann. Report, 86-87(1996).