Crystal Structure Analysis (BL02B1)

1. Introduction

BL02B1 is designed for studies of single crystal structure analysis and phase transition phenomena for solid state physics. We have installed a 6+1-circle diffractometer, vacuum oscillation IP camera, Weissenberg camera and powder diffraction system. They are utilized in combination with the bending magnet synchrotron light source. This report describes the available X-ray source and apparatus.

2. X-ray Source

The X-ray source of BL02B1 is the SPring-8 standard bending magnet. The spec of the magnet is described on a webpage (http://www.spring8.or.jp/ENGLISH/facility/bl/insertion/Bending/index.html).
The white spectrum X-ray beam is monochromatized by the SPring-8 standard double crystal monochromator. We use two Pt coated X-ray mirrors: the first one is set at the upstream side of the monochromator and the second one is set at the downstream side ofthe monochromator. A schematic drawing of these optical components is shown in Fig. 1.

Depending on the experimental condition, a user can tune the bending of the 2nd mirror along the vertical direction. Then the user can obtain a parallel or focused X-ray beam. In the focused state, the full width of the half maximum (FWHM) of the beam along the vertical direction becomes 100 μm at the sample position. On the other hand, the focusing along the horizontal direction is not available at the moment.

The X-ray energy of the beam is controlled through a computer by users. We have tested the energy range from 5 keV to 70 keV. The actual photon number at the sample position in the square region with a height of 0.1 mm and a width of 1 mm is estimated as 10^{11} photons/sec at 30 keV with focused X-ray beam.

3. Experimental System

3.1 Diffractometer

BL02B1 has HUBER 5020 6+1-circle diffractometer system. It has a long 2θ arm with an additional short 2θ arm. The short 2θ arm has a characteristics that the motor speed is very high and vertical and horizontal half slits as well as automatic receiving slits are equipped. The short 2θ arm is mainly utilized for Bragg points collection like a conventional 4-circle diffractometer with the automatic software, MXC produced by MAC Science Co. Ltd. The long 2θ arm, which has an angle resolution of 0.0001 deg, is utilized for the mapping scan in a reciprocal space, diffuse scattering observation or high resolution powder pattern observation with a scintillation counter.

The long 2θ arm can be equipped with two kinds of analyzer crystal: one is a noise reduction and high resolution analyzer, and another is used for polarization analysis. The motion of the long arm is controlled with original software, which is called 4-circle commands made by a user group. A Solid State Detector is available with this long arm.

3.2 Oscillation IP Camera

A vacuum IP camera is equipped on the ϕ-circle for small crystal structure analyses. The temperature is limited at room temperature, and the IP reader is available on an off-line system. The target crystal size is less than 20 μm in diameter. A new automatic oscillation imaging plate (IP) camera is developed in the summer of 1999. The public use of this camera is planned to start from the beginning of 2000. The processes of X-ray exposure and reading out are managed automatically by a computer. The entire process is performed in vacuum to obtain a high S/N ratio measurement. The sample is set on a rotating refrigerator to utilize the oscillation camera motion. The available temperature range of this camera is expected from room temperature to 25 K.

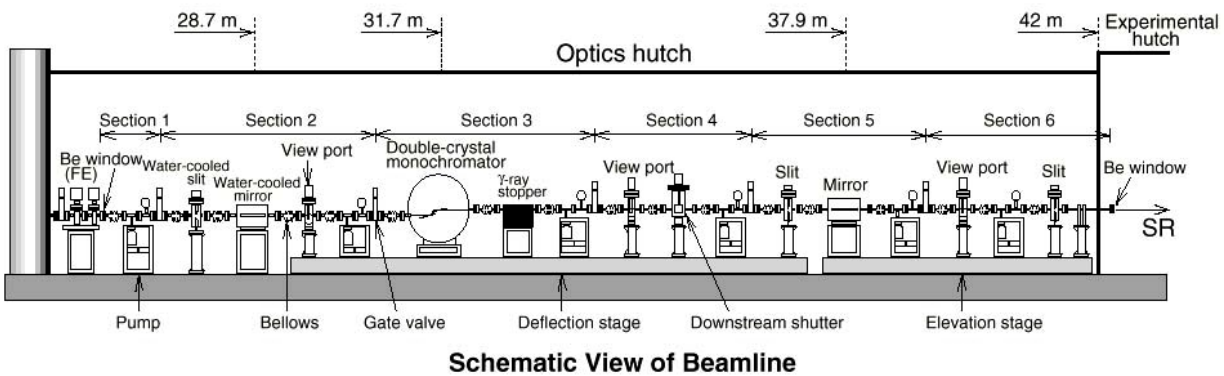

Fig. 1. Schematic drawing of optical system in BL02B1.

3.3 Powder Diffraction

A powder diffraction experiment can be performed in two ways. One uses a long 2θ arm with an analyzer crystal and scintillation counter. The FWHM of the powder pattern profile with this method achieved 0.008 degree [1]. The other method is a recording the Debye ring from the sample on an IP sheet. This method can finish the measuring one sample in 1 hour. From the autumn of 1999, BL02B2 will start to offer the IP sheet powder diffraction experiment for public use.

3.4 Weissenberg Camera

The Weissenberg camera system is designed for scanning in a reciprocal space. The operation of this camera is based on the combination motion of the translation of the IP sheet and the ω rotation of the sample.

3.5 *Temperature Control System*

BL02B1 is equipped with two refrigerators and one vacuum furnace system. The smaller refrigerator, with a half-spherical Be dome shroud, is designed for Bragg intensity collection. The available temperature is down to 15 K. The larger refrigerator is equipped with a cylindrical Be dome shroud and sample can, and it is reachable down to 8 K. The larger one is utilized for diamond anvil cell (DAC) or EXAFS measurement. When the see-through vacuum shroud made with plastic material is used, the available temperature is about 25 K. The vacuum furnace is operated with a water coolant system and can obtain temperatures above 1,000 K.

3.6 Summary of Experimental System

The user can choose some combination of the diffraction and temperature control systems. In Table 1 the available combinations of them are summarized.

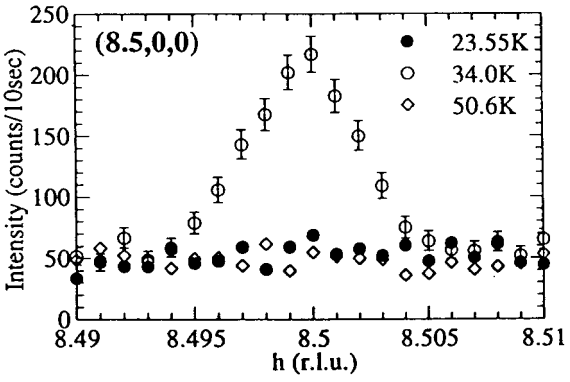

Fig. 2. Result of single crystal DAC experiment at 1.8 GPa and below 40 K.

4. Examples

Figure 2, as an example, shows results of the DAC experiment at low temperature. The development of the super lattice spot caused by the lattice modulation associated with the antiferromagnetic ordering in CeP was observed at 1.8 GPa and below 40 K [2]. Figure 3 also shows an example of the diffuse pattern observed in $NdNbO_4$ at 980 K, just above the structural transition temperature [3].

5. Machine Time and Preparation

The machine time for user experiments in BL02B1 is terribly crowded. Even though users made high quality scientific proposal, their machine time was always reduced to a short schedule. It is strongly suggested that users should learn about experimental operation and make thorough preparations before their experiments.

This beamline is managed by four sub-groups. Their group names and representative members are as follows. Structure Phase Transition: Yukio Noda (Tohoku Univ.), Diffuse Scattering Analysis: Kennichi Ohshima (Tsukuba Univ.), Chemical Reaction: Koshiro Toriumi (Himeji Inst. Tech.) and Powder Diffraction: Hideo Toraya (Nagoya Inst. Tech.). Those proposing new experiments should be consulted with one of them or a beamline scientist to get precise information on the beamline's condition and to schedule training on its operation.

References

[1] Y. Noda, *et al.*, SPring-8 User Experiment Report #1997A-0027.

[2] Y. Noda, *et al.*, SPring-8 User Experiment Report #1999A-0398.

[3] Y. Kuroiwa, *et al.*, SRMS-2 (1998) in press.

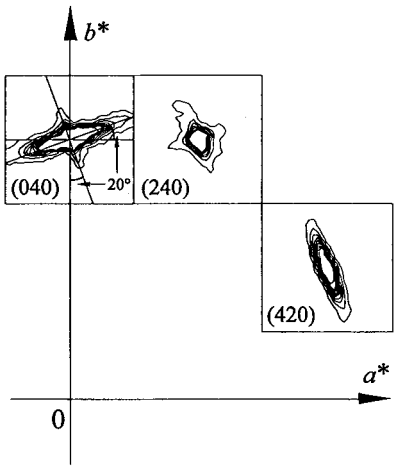

Fig. 3. High temperature experimental result at 980 K.

Table 1. Experimental Setup Summary

	Low Temperature Experiment with Large Refrigerator (T > 8K)	Low Temperature Experiment with Small Refrigerator (T > 10K)	Room Temperature	High Temperature Experiment with Vacuum Furnace (T < 1000K)
Bragg Points Collection	6+1-circle diffractometer with long 2θ arm and MXC software (DAC available)	6+1-circle diffractometer with short 2θ arm and MXC software Automatic Vacuum Oscillation Camera (T > 25K)	6+1-circle diffractometer with short 2θ arm and MXC software Automatic Vacuum Oscillation Camera	6+1-circle diffractometer with short 2θ arm and MXC software
Bragg Points Observation	Weissenberg Camera	Weissenberg Camera	Weissenberg Camera	Weissenberg Camera
Diffuse Scattering and Mapping Measurement	6+1-circle diffractometer with long 2θ arm and 4-circle commands software (DAC available)	6+1-circle diffractometer with long 2θ arm and 4-circle commands software	6+1-circle diffractometer with long 2θ arm and 4-circle commands software (DAC available)	6+1-circle diffractometer with long 2θ arm and 4-circle commands software
Powder Diffraction	6+1-circle diffractometer with long 2θ arm and analyzer Curved IP Sheet		6+1-circle diffractometer with long 2θ arm and analyzer Curved IP Sheet	6+1-circle diffractometer with long 2θ arm and analyzer Curved IP Sheet