BL02B1 Crystal Structure Analysis

BL02B1 is designed for studies of single crystal structure analysis and phase transition phenomena for crystalline material. A HUBER multi-circle diffdractometer and vacuum oscillation camera are utilized for user experiment with the X-ray energy range of 5 ~ 115 keV. These equipments are used for university and company researchers who works in the field of physics, chemistry and industrial application.

The X-ray source of this beamline is the standard SPring-8 bending magnet. The photon flux at the sample position is estimated to be 10^{12} photons/sec.

The multiaxial diffractometer and the low temperature vacuum IP camera are available at the experimental hutch. The diffractometer has two sets of 2θ axis. These two 2θ arms are designed for two different measurement mode, one is for a precise higher angular resolution measurement and the other is for the high speed and lower angular resolution measurement.

Area of research

Single crystal structure analysis in X-ray wide energy range

Precise X-ray diffraction analysis of the lattice or charge modulation originated from the phase transition at low temperatures

Keywords

Scientific field

Single Crystal Structure analysis, Phase transition in solid state material, Orbital ordering, Charge ordering, Charge density wave, Inorganic material, Organic material, High energy X-ray diffraction.

Equipment

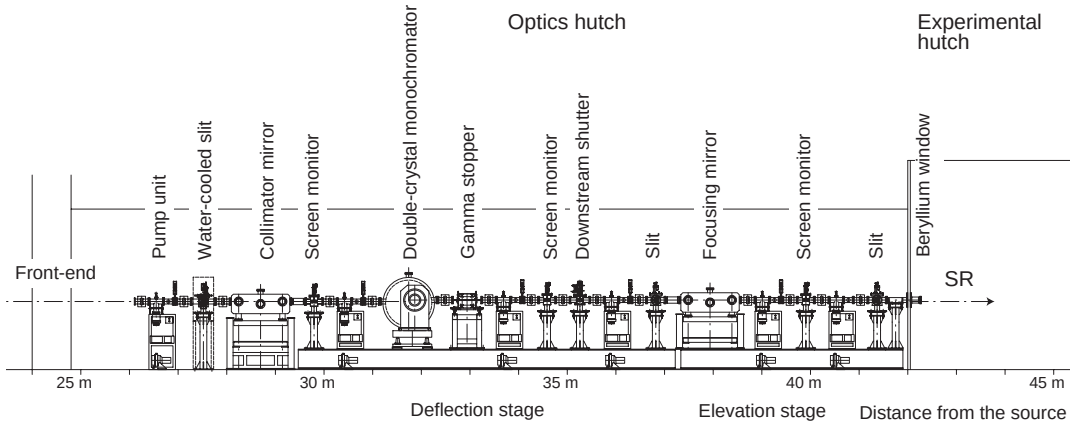
Four circle diffractometer, Vacuum oscillation camera, Refrigerator, Furnace, Scintillation counter, Imaging plate, High pressure (DAC) Experiment at low temperature

Souce and optics

We have two mirror system at an upstream and a downstream position of the monochromator. The mirrors are coated with platinum. The X-ray beam is vertically collimated by the first mirror to obtain a high-energy resolution ($\Delta E/E \sim 10^{-4}$) in the energy range of 5~60 keV. The second monochromator crystal and the second mirror will focus the beam at the sample position in the horizontal and vertical direction, respectively.

The beam shape at the sample position is 0.1 mm in vertical and 3 mm in horizontal in the smallest case. At this state the photon flux at the sample position is estimated to be 10^{12} photons/sec. The higher harmonics contamination in the incident X-ray is reduced to less than 10^{-9} with use of the double X-ray mirror.

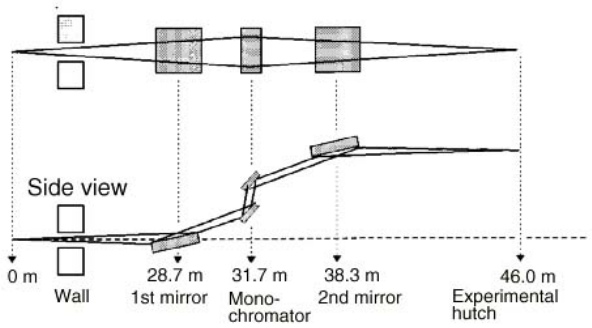
In the energy range above 60 keV, these mirrors will be removed from the optical axis. In this case, the higher harmonics elimination will be accomplished by detuning the double crystal monochromator and the beam can be focused only in the horizontal plane.



Schematic view of beamline

X-rays at sample

Energy range	5 ~ 115 keV
Energy resolution	$\Delta E/E = 10^{-4}$
Photon flux	10^{12} phonons/s
Beam size	3×0.1 mm^2



Optical layout of the Crystal Structure Analysis Beamline for low energy case (less than 20 keV)

Experimental stations

We have only one experimental hutch. In the hutch two experimental set-up is equipped. One is the 7-axis diffractometer and another is the vacuum oscillation camera. The diffractometer is positioned at the upstream side of the oscillation camera. Followings are the description of them.

7-circle diffractometer

BL02B1 has HUBER 5020 6+1-circle diffractometer system. It has a long 2θ arm with an additional short 2θ arm.

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.

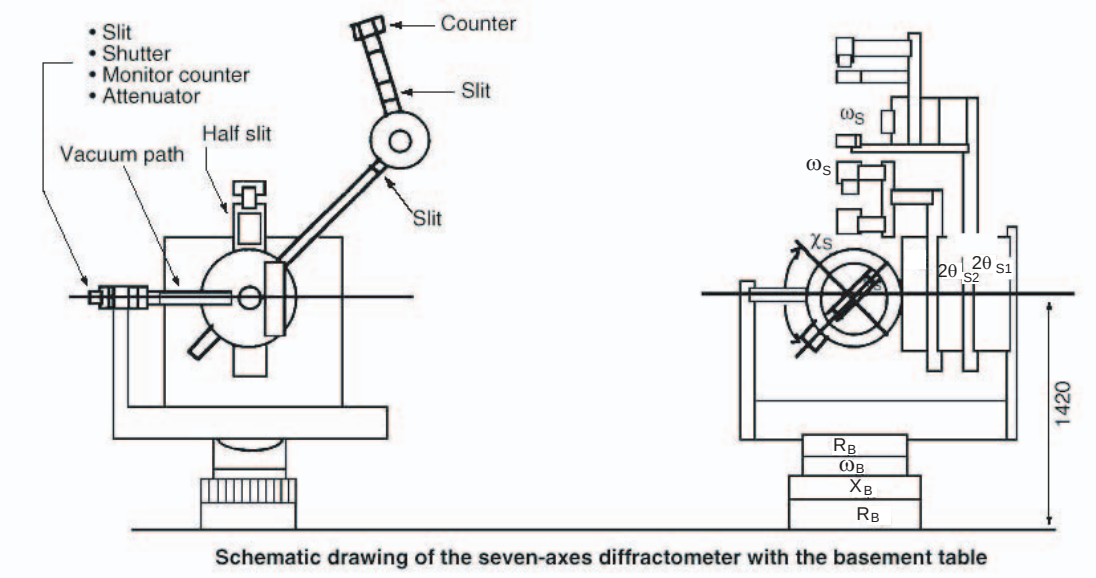
The short 2θ arm are designed for high speed Bragg points collection like a conventional 4-circle diffractometer with an automatic software. The precise automatic UB-maxtix definition is performed with vertical and horizontal half slits as well as automatic receiving slits system.

Sample condition

The sample condition is controlled by a refrigerator or a 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 1000 K.

The Weissenberg camera system is available for scanning in a reciprocal space. The camera operation is realized by the combination motion of the translation of the IP sheet and the ϕ rotation of the sample.

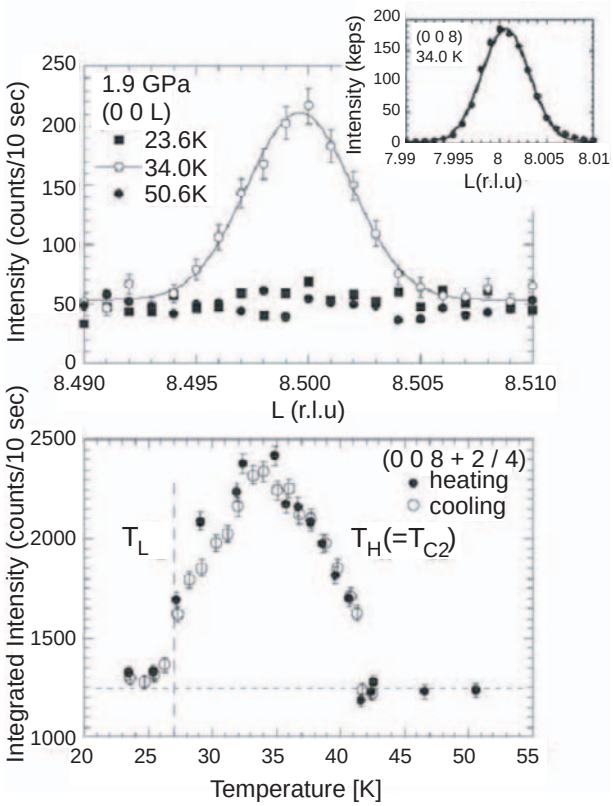
Experimantal conditions	Low-temperature experiment with large refrigerator (T > 8 K)	Low-temperature experiment with small refrigerator (T > 10 K)	Room temperature	High-temperature experiment with vacuum furnace (T < 1000 K)
Bragg points collection	6+1-circle diffractometer with long 2θ arm and MXC software (DAC available)	6+1-circle diffractometer with short 2θ arm and MXC software	6+1-circle diffractometer with short 2θ arm and MXC software	6+1-circle diffractometer with short 2θ arm and MXC software
Reciprocal space 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	---	6+1-circle diffractometer with long 2θ arm and analyzer	6+1-circle diffractometer with long 2θ arm and analyzer



Schematic drawing of the seven-axes diffractometer with the basement table

An example result

The temperature variation of (0 0 8+2/4) super lattice reflection for CeP at 1.9 GPa. Upper figure shows the profile functions at some temperatures. Lower figure shows the temperature variation ofthe super lattice reflection, where we can see clearly two successive phase transitions.



References

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2) Y. Noda, T. Shobu, K. Iwasa and M. Kohgi: SPring-8 user report #1999A-0398.
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Low temperature vacuum camera

A low temperature vacuum camera was developed for the purpose of measuring the crystal structure change accompanied by photo irradiation. The sample was set on top of a refrigerator. The diffraction signal from the oscillating micro crystal is recorded on the cylindrical Imaging Plate. To obtain the high S/N recording in low temperature the whole system including the IP sheet and refrigerator is set in a vacuum chamber.

As a result, S/N ratio of 10^5 was attained for typical experiment. The low temperature experiment down to 20 K is also available. The photo excited state of a crystal has successfully analyzed.

Specifications of the camera

• Goniometer

Rotation	1 axis oscillation (ϕ-axis) within horizontal plane
Distance from goniobase to sample	50 mm ± 2 mm

• X-ray detector

Cylindrical imaging plate and photo-multiplier

IP size	200.0 × 334.5 mm
IP shape	Cylinder form
Pixel size	100 μm × 100 μm
Reading out region	2000 × 3345 pixels
data size	16,001,024 byte/frame
Camera radius	79.5 mm
Direct beam position	(IPX, IPY) = (1014 1247)

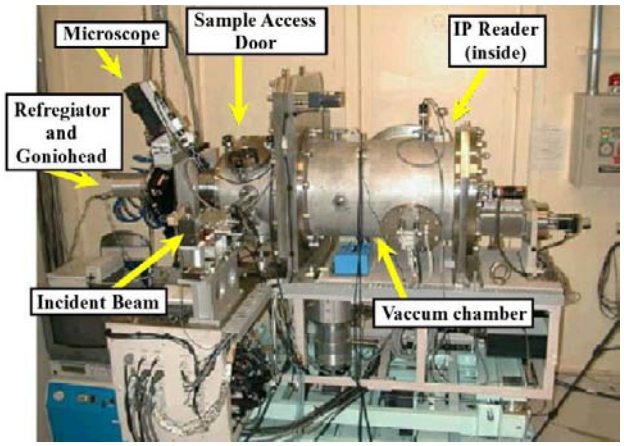
2θ region	- 93 ~ +148 degree
Horizontal region	± 52 degree
Exposure time/frame	Any

• Temperature control

Closed cycle refrigerator (APD 201)	20 K ~ 300 K
Sensor	Diode (LS#10, CryoCalD3)

• Vacuum condition

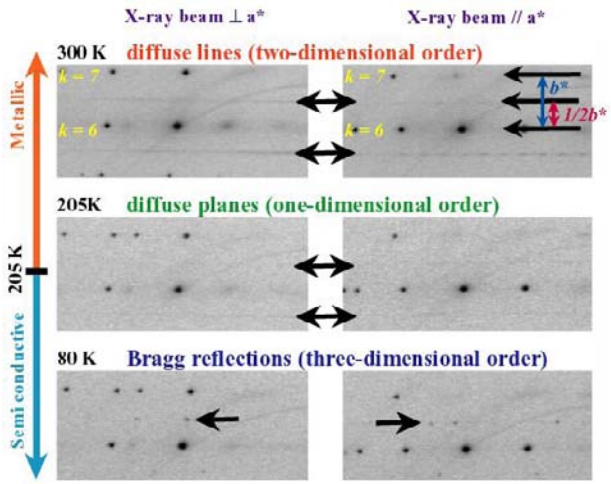
From air to 10^{-4} Pa (RP and TMP)



Schematic drawing of the low temperature vacuum camera

An example result

The temperature variation of the diffuse streak for an iodo-bridged mixed-valence diplatinum complex with a linear chain structure $Pt_2(EtCS_2)_4I$. The phase transition was characterized by the successive change of dimensionality of the Pt valence ordering. The two dimensional ordering was observed at room temperature. With decreasing temperature the ordering modifies to one dimennsional at 205 K. With further cooling, three dimensional ordering appears as observed at 80 K.



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