

BL22XU JAERI Actinide Science II

BL22XU is one of four JAERI (Japan Atomic Energy Research Institute) beamlines in SPring-8. This X-ray undulator beamline has been designed for high-pressure material studies and researches on correlated electron systems using resonant X-ray diffraction. A part of the beamline is introduced into RI laboratory in order to handle samples including uranium for the latter objective.

A high energy X-ray is useful for diffraction experiments under high pressure using a multi-anvil press or a diamond anvil cell, while a low energy X-ray is needful to perform resonant diffraction experiments for some kinds of elements. To decide the lower limit of the available X-ray energy, we considered the M-edge of uranium (~3.5 keV) at which the intensity of magnetic scattering is enhanced. In order to cover a wide energy range 3~70 keV, two sets of double crystal monochromator with different tunable energy range are installed in an optics hutch. In addition, movable beryllium windows are adopted to avoid the large absorption of low energy X-rays of around 3~4 keV.

Area of research

Materials science at high pressure

Resonant X-ray scattering (activity at RI laboratory)

Keywords

Scientific field

High-pressure single-crystal X-ray diffraction, Powder X-ray diffraction, High temperature, X-ray absorption, Strongly correlated electron systems, Resonant X-ray diffraction

Equipment

High-pressure and high-temperature apparatus, Diffractometer for extreme conditions (temperature, pressure, and magnetic field)

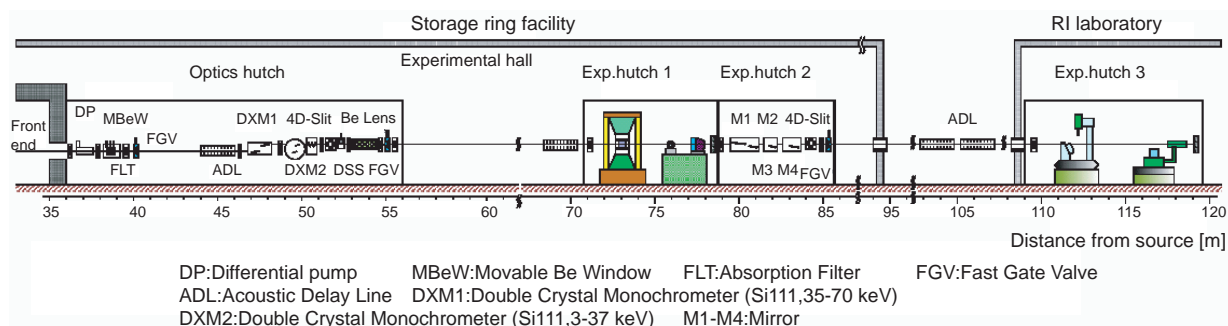
Source and optics

In the optics hutch, there are two double-crystal monochromators. One is the high-energy monochromator DXM1, which has two independent θ -axes and is used in an energy range from 35 keV to 70 keV. The other is the low-energy monochromator DXM2, which is a SPring-8 standard type monochromator and is used in an energy range from 3 keV to 37 keV. Si (111) crystals in the monochromators are cryogenically cooled by liquid nitrogen. As a focusing device, a set of beryllium refraction lenses is installed in the optics hutch. In addition, to focus X-rays and to cut higher harmonics, a first mirror and three second mirrors are installed in the experimental hutch 2. The first mirror M1 is a mechanically-bent plane mirror for vertical

focusing. The second mirrors M2, M3, and M4 are cylindrical mirrors for horizontal focusing, and are used in energy ranges 3~7 keV, 7~16 keV and 16~30 keV, respectively.

X-rays at sample

Energy range	35 ~ 70 keV by DXM 3 ~ 37 keV by DXM2
Energy resolution	$\Delta E/E \sim 10^{-4}$
Photon flux	2×10^{13}
Beam size	At 14.4 keV
(at exp. hutch 3)	normal : 2.0 (V) \times 3.2 (H) mm ² focusing : 0.5 (V) \times 0.4 (H) mm ²



Schematic view of beamline

Experimental stations

Experimental hutch 1 (at storage ring building)

SMAP-180

To study structure and properties of matter under high-temperature and high-pressure conditions, a cubic-type multi-anvil apparatus, SMAP-180 (SPring-8 Multi-Anvil Press with maximum load of 180 ton), is installed. This press is capable to generate pressures up to 12 GPa and temperatures up to 1500°C. With this press, angle-dispersive X-ray diffraction (ADX) measurements and density measurements by means of X-ray absorption are possible. The ADX method has the advantage of accuracy and resolution over energy-dispersive X-ray diffraction method, which is commonly used for multi-anvil presses. In the ADX, diffraction pattern is collected using an imaging plate (IP). A radial slit system reduces background X-rays from materials that surround the sample without increasing the data acquisition time too much. For adjustment and real-time observation, a CCD camera with an X-ray image intensifier can be used. Thanks to the intense, high-energy monochromatic X-rays up to 70 keV, precise X-ray diffraction measurements in wide Q range are possible. The high-energy X-rays will also allow us to study heavy elements. For absorption measurements, two ionization chambers are used.



SMAP-180

Diffractometer for diamond anvil cell (DAC)

In order to perform high pressure experiments, a diffractometer for diamond anvil cell (DAC) is installed. This system is designed for both single crystal and powder X-ray diffraction measurements. The system mainly consists of three parts, sample positioning stages, detectors, and a microscope. On the basic setup of sample stages including an ω -axis, translations and an arc-stage, various components can be mounted; a χ - ϕ circle goniometer, a 4K He closed cycle cryostat, a simple DAC holder. Two types of detectors, an imaging plate (IP) and a charge coupled device (CCD)

detector, are equipped. The IP is the size of $400 \times 400 \text{ mm}^2$, and the sample-detector distance can be changed from 200 mm to 730 mm. The CCD detector is mounted on the 2θ arm. The area size is $70 \times 70 \text{ mm}^2$, and the sample-detector distance can be changed from 100 mm to 200 mm. The sample mounted on the diffractometer can be monitored by the microscope. With use of the microscope, the sample pressure can be also measured by the ruby luminescence method.



Diffractometer for diamond anvil cell

Experimental hutch 3 (at RI laboratory)

Resonant X-ray diffraction

The experimental hutch 3 is located in radioisotope (RI) laboratory. Resonant X-ray diffraction studies, mainly at K-edge of 3d transition metals, L-edge of lanthanides, and M-edge of actinides (3.5 ~ 9 keV), are planned. The system is designed to utilize low energy X-rays. Two diffractometers are installed in the hutch. One is a 4-circle diffractometer with vertical 2θ arm, which is used for usual diffraction studies of single crystal. The other is a 2-circle diffractometer with horizontal 2θ arm, on which a bulky apparatus, such as a superconducting magnet, can be mounted. Both diffractometers are equipped with a polarization analyzer. In addition, there is a phase retarder to control the polarization of incident X-rays, and magnetic circular dichroism experiment can be performed. Sample conditions can be controlled by refrigerators (low temperature), diamond anvil cells (high pressure), and magnets (magnetic field).



2-circle diffractometer with superconducting magnet

Facilities for sample condition

- He closed cycle refrigerator 1
 $T > 5 \text{ K}$
 Diamond anvil cell can be attached.
- He closed cycle refrigerator 2
 $T > 5 \text{ K}$
 Azimuthal angle dependence of high angle scattering can be measured.
- He flow refrigerator
 $T > 2 \text{ K}$
- Superconducting magnet (only available on 2-circle diffractometer)
 $B < 6 \text{ T}$ (horizontal magnetic field)
 $T > 4 \text{ K}$

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