BL28B2 White Beam X-ray Diffraction

BL28B2 is a multi-purpose beamline. Experiments using white X-rays are carried out. The beamline has two experimental hutches: optics hutch (OH) 2 and OH 3. The equipment in OH 2 is optimized for state-of-the-art X-ray diffraction imaging including white X-ray topography. OH 3 is for the structural study of expanded fluid metals over the critical point at high pressure and high temperature and for time-resolved energy-dispersive XAFS (DXAFS).

Area of research

White X-ray diffraction

Time-resolved energy-dispersive XAFS (DXAFS) for studies of chemical and/or physical reaction process.

Keywords

Scientific field

Topography, Crystal defects, Dispersive XAFS, High pressure and high temperature

Equipment

General-purpose diffractometer, Cryostat, Dispersive XAFS measurement system, Horizontal energy-dispersive X-ray diffractometer

Source and optics

X-rays at sample

Energy range	White (> 5 keV owing to the Be window)
Beam size	30 mm (H) × 10 mm (V)
	(at 44 m from a light source)

Experimental stations

Optics hutch 2

In the experimental hutch (Optics hutch 2), there is the attenuator unit to moderate the intensity of X-ray and filter out the low energy part of incident beam, the water filter to reduce the damage of organic crystals, the fast operating shutter which can operate with minimum exposure time of 1 msec, the diffractometer for a monochromator, and the diffractometer for samples.

Samples can be cooled down to 3.8 K by He cryostat and also heated up to 1770 K by infrared heater. X-ray films, nuclear emulsion plates, Imaging Plate (IP), and X-ray TV system are used as imaging detectors.

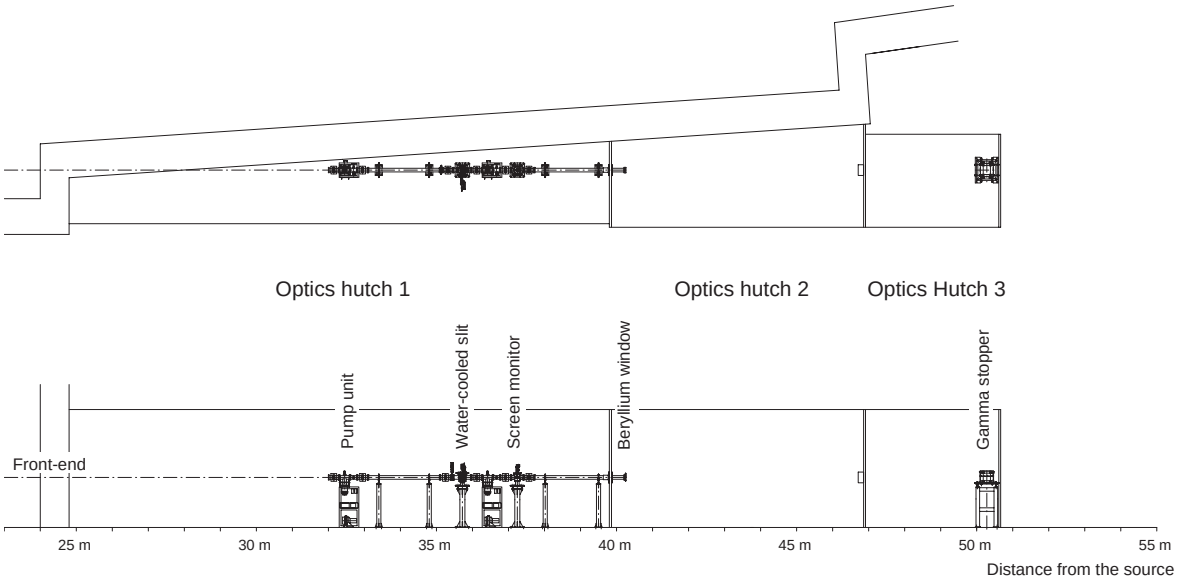
• Attenuator unit

It consists of six water cooled metal plates.

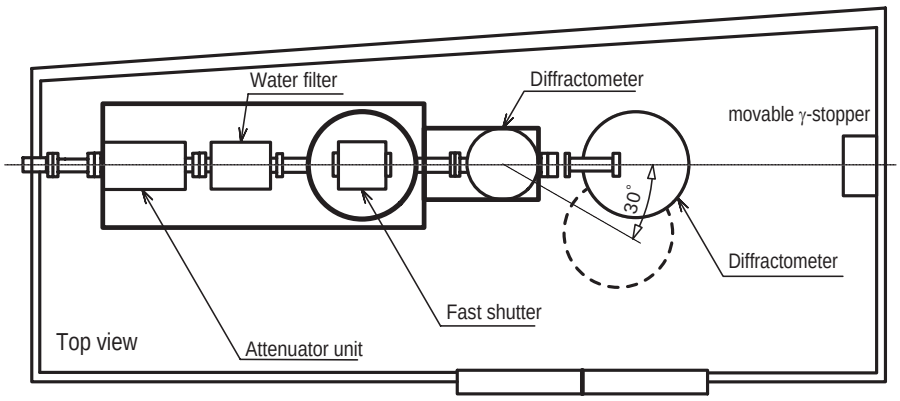
Pb 15 mm, Al 2 mm, Al 5 mm, Cu 1 mm, Cu 2 mm, Fe 1 mm.

Pb 15 mm is used as a slow shutter.

All plates can be controlled by PC.



Schematic view of beamline



Schematic view of BL28B2 optics hutch 2

• Water filter

The filter has variable length from 5 mm to 50 mm.

• Fast shutter

Two tantalum plates of 5 mm thick are operated with rotary solenoid.

It has minimum exposure time with 1 msec and resolution of 1 msec.

The aperture size is 5 mm × 5 mm.

• Additional diffractometer

Vertical-axis ω-2θ diffractometer is equipped for a single-bounce crystal monochromator.

• Main diffractometer

Vertical-axis ω-2θ diffractometer is equipped for samples.

The stage for detectors is set on 2θ arm.

Detector translation stage on the χ goniometer.

X-Y-Z-R_x-R_y goniometer head on the ω-axis.

• Intensity measuring devices

Ionization chamber for I_0 monitoring

NaI scintillation detectors

Ge solid state detector (SSD)

Multi-channel Analyzer with ADC&HV module for SSD

NIM-based bins, HV's, amplifiers, single-channel analyzers, voltage-to-frequency converters (VFC) for ionization chambers.

Current amplifiers and pico-ammeters

Digital multi-meter

XT-chart recorder

• Image recording devices

Imaging plate, Beam monitor with CCD

X-ray TV

Cassettes for X-ray films and photographic plates

Nuclear emulsion plates (Ilford L4) and high-resolution X-ray films (minimum stock for emergency)

• Sample environment

Infrared heater (up to 1770 K)

Cryostat (3.8 K)

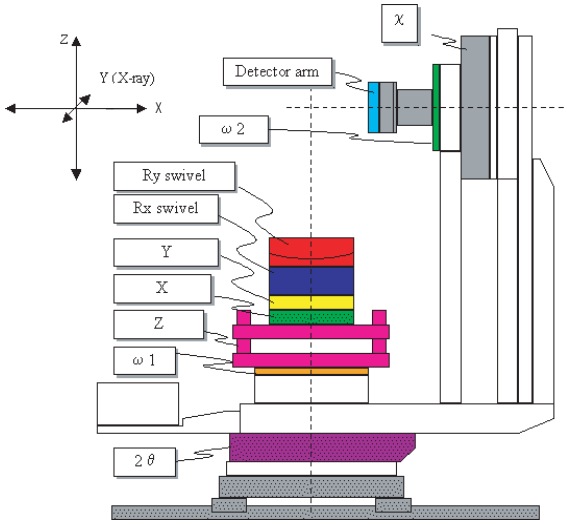
• Accessories

Optical microscope with CCD camera connected to a PC based data analysis system

Darkroom facility for photographic processing

Constant low temperature (5°C) cabinet for keeping films, plates and photo-chemicals

name	axis	resolution	Range
ω 1	Z	1/1000 deg.	360 deg.
2 θ	Z	1/1000 deg.	360 deg.
X	X	2/1000 mm	± 20 mm
Y	Y	2/1000 mm	± 20 mm
Rx swivel	X	1/1000 deg.	± 7.5 deg.
Ry swivel	Y	1/1000 deg.	± 7.5 deg.
Z	Z	2/1000 mm	± 50 mm
χ	horizontal	1/1000 deg.	360 deg.
detector arm	-	2/1000 mm	± 200 mm
ω 2	horizontal	1/1000 deg.	360 deg.



Optics hutch 3

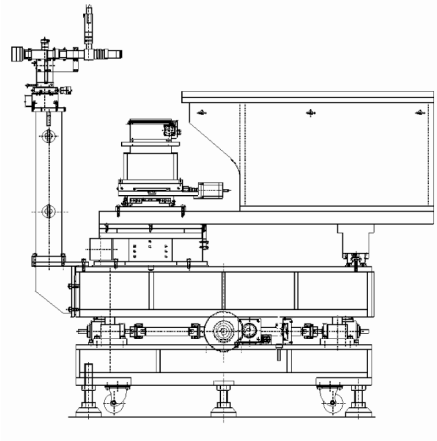
OH 3 is designed for a structural study of expanded fluid metals over the critical point at high pressure and high temperature. The main facility is a high pressure vessel and equipped with a horizontal energy-dispersive X-ray

diffractometer. The vessel has Be windows for the incident and scattered X-ray beam (2θ = 4, 7, 11.5, 15, 20, 25, 33°). High-purity He gas is used for a pressure medium, and a fluid sample contained in a single crystal sapphire cell is surrounded by compressed He gas in the vessel. The pressure is measured by a Heise gauge at an accuracy of ±0.3 MPa. The system is suitable for high pressure and high temperature X-ray diffraction measurement up to 200 MPa and 2,000°C and has been applied to investigate structural changes with volume expansion for various fluid materials.

Horizontal energy-dispersive X-ray diffractometer

The diffractometer consists of a 2θ goniometer for a detector, translation XYZ stages for sample alignment and a vertical-axis θ goniometer. The whole system is set on a height adjustable surface plate.

name	resolution	range
Sample alignment		
θ goniometer	4/1000 deg.	360 deg.
Z stage	2/10 μm	± 10 mm
XY stage	2 μm	± 50 mm
Detector alignment		
2 θ goniometer	4/100000 deg.	-40 ~ 120 deg.
Surface plate		
Z stage	0.186 μm	-5 ~ 35 mm

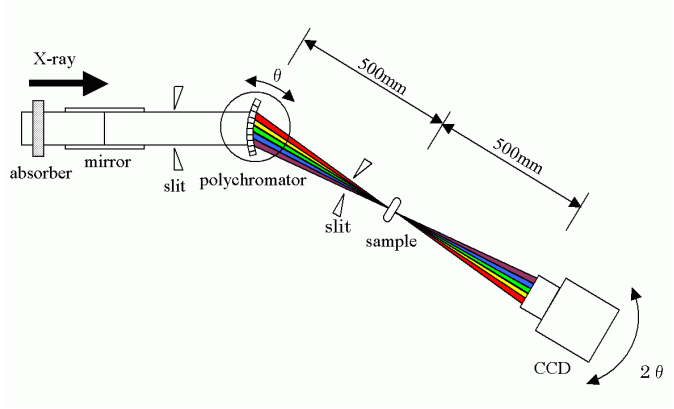


Side view of the diffractometer in OH 3

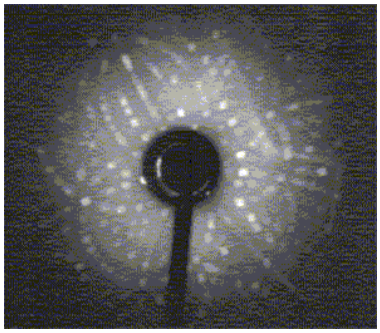
- High pressure gas vessel (helium 200 MPa, 2,000 °C)
- Pure Ge solid-state detector
- Ionization chamber

DXAFS

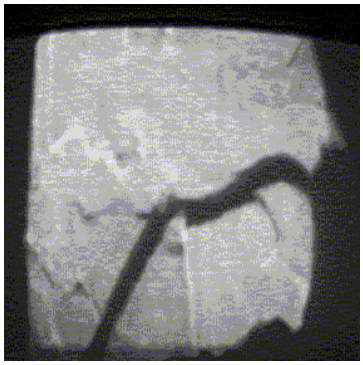
The main equipment of the DXAFS system consists of a polychromator with a bent silicon crystal, a higher-harmonic-rejecting mirror, a CCD coupled with a scintillating screen and a lens system.



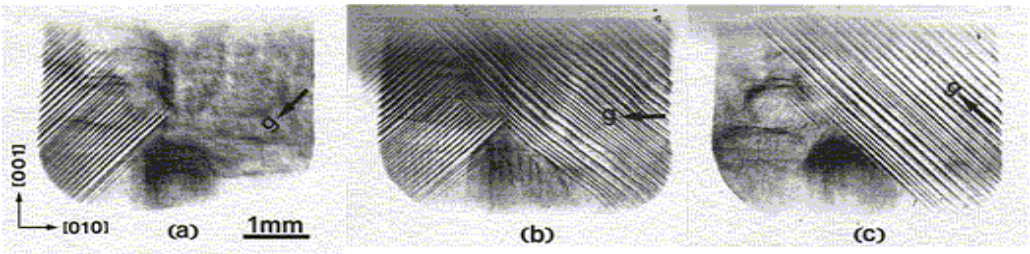
Typical results in OH 2



Laue pattern of tetragonal lysozyme crystal

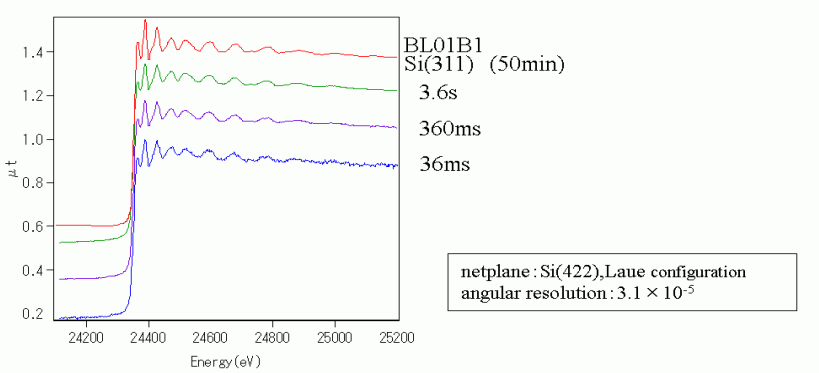


High temperature Bragg topograph of silicon steel at 965 K



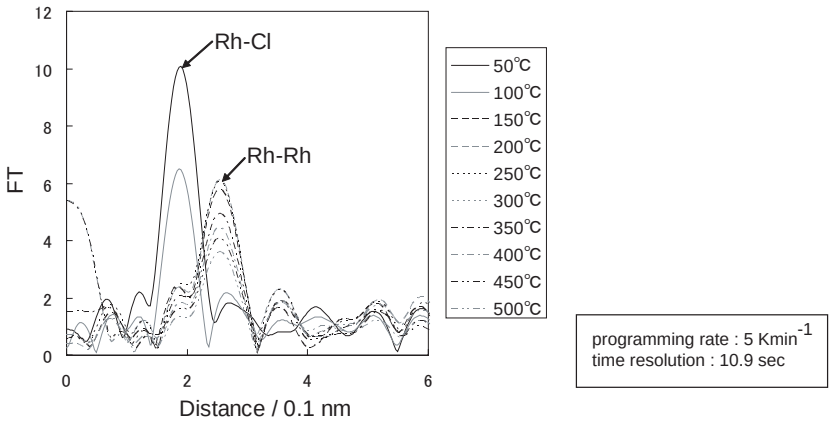
Low temperature Laue topographs of $SrTiO_3$ at 6 K

Typical results in OH 3

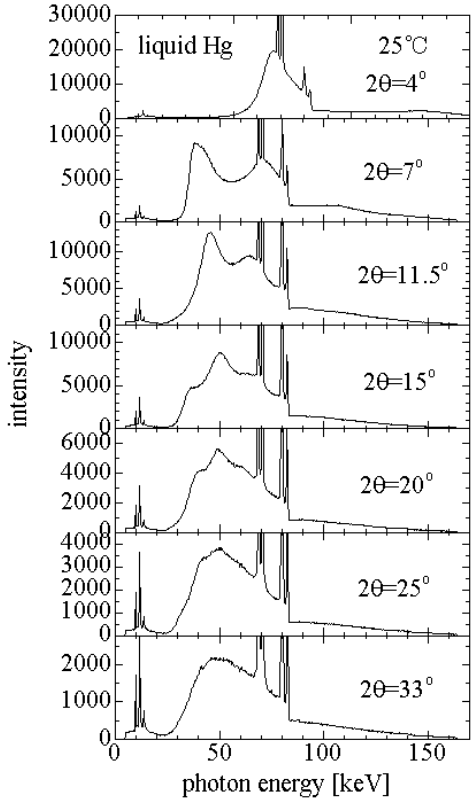


Pd K-edge XAFS spectrum of Pd foil measured at BL28B2 and BL01B1

Measurement of reduction process of Rh/SiO_2 catalyst



The sample was treated at different temparatures in H2 gas.



Spectrum of liquid Hg in standard condition

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