Public Beamline

BL01B1 XAFS

This beamline is designed for the transmission mode XAFS measurements using ionization chambers. The various applications of XAFS can be done in the energy range of 3.8 ~ 113 keV.
Using the cryostat and furnaces, the sample temperature can be controlled in the range of 10 ~ 1870 K. A Lytle type detector and 19-element Ge solid-state detector are available for the measurement of the excited spectra. A conversion electron yield detector is available for layered synthetic materials and bulk sample. A θ-2θ stage is used for glancing-angle XAFS measurements.

Area of research

XAFS in wide energy region (3.8 to 113 keV)
XAFS of dilute systems and thin films

Keywords

Scientific field

XAFS, EXAFS, XANES, High energy X-ray (XAFS), Dilute sample, Thin film

Equipment

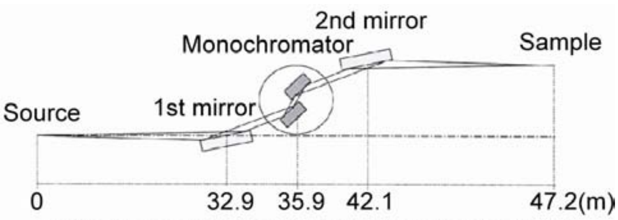
Cryostat, Furnace, 19-element SSD, CEY detector, θ-2θ stage

Source and optics

The main optics consists of a first collimating mirror, a fixed-exit double crystal monochromator and a second focusing mirror. The mirrors are rhodium coated.

X-rays at sample

Energy range	3.8 ~ 113 keV
Energy resolution	$\Delta E/E = 3 \times 10^{-5} \sim 2 \times 10^{-4}$
Photon flux	$10^9 \sim 10^{11}$ photons/s

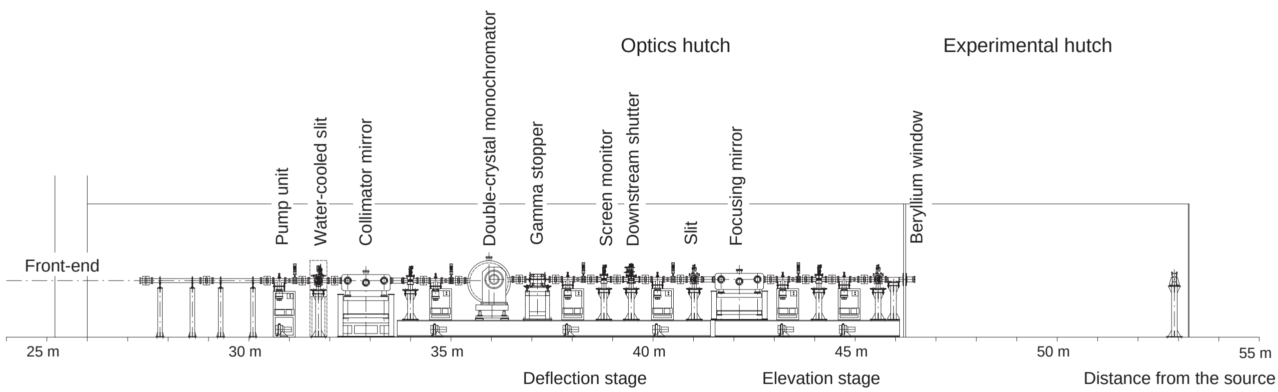


Optical layout for energy range less than 60 keV

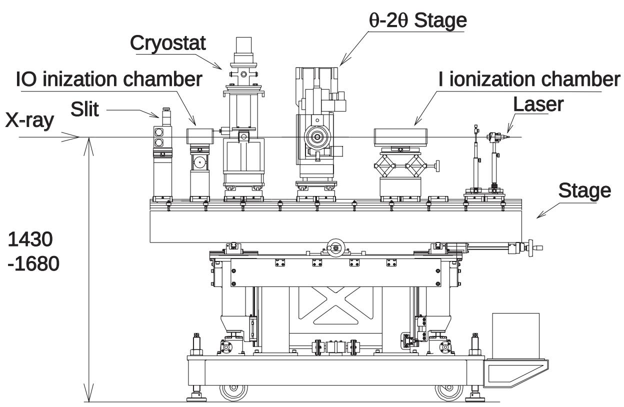
Experimental stations

Overview of XAFS measurement system on optical bench

In the experimental hutch, measurement equipments are prepared for XAFS measurements, including detectors, a cryostat, electric furnaces, stages and a slit [1]. The beam height changes from 0 to 250 mm together with the glancing angle of the mirror. To follow the beam height, the measurement equipments are mounted on the Al honeycomb base, which can be adjusted by a pulse motor-driven vertical translation stage. Available measurement modes of XAFS are described in the following sections.



Schematic view of beamline



Schematic view of optical bench for XAFS measurement system

i) Detectors

Described in following sections

ii) Sample temperature controllers

- Water-cooled electric furnace with Kapton window
  Control range of temperature : 295 ~ 1070 K
- Muffle furnace with Kapton window
  Control range of temperature : 295 ~ 1870 K
- Cryostat with Kapton window
  Control range of temperature : 10 ~ 300 K
  Stability : ~ ± 0.1 K
  Cooling power : 8 W (2nd stage @20 K)

iii) Optics

- θ-2θ stage with Z stage
  Step θ : 0.005 arcsec/pulse, 2θ : 0.002 deg/pulse
- Vertical translation stage for optical bench
  Size : 1.2 × 2 m^2, Stroke : 250 mm, Step : 0.5 μm/pulse
- Slits
  Stepping motor driven blade : 3 mm thick tantalum,
  Stroke :±10 mm, Step ; 2 μm/pulse

iv) Measurement devices

Power supply for NIM bin, High-voltage power supplies for ion chamber and Ge-SSD, Current amplifier, 2ch filter, VF converter, Quad counter, Spectroscopy amplifier, SCA, MCA etc.

Measurement system in transmission mode

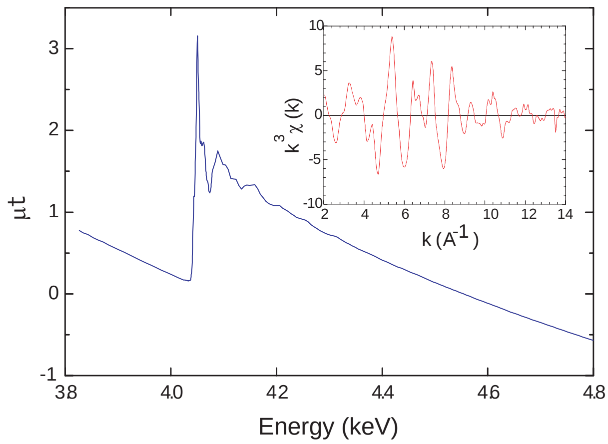
The transmission mode is standard for XAFS measurement and is performed by two ionization chambers. The covered energy range is from 3.8 to 113 keV. The adequate combination of ionization chambers and filling gases should be selected depend on the energy range of the experiment as followings.

i) Gas-flow type ionization chamber

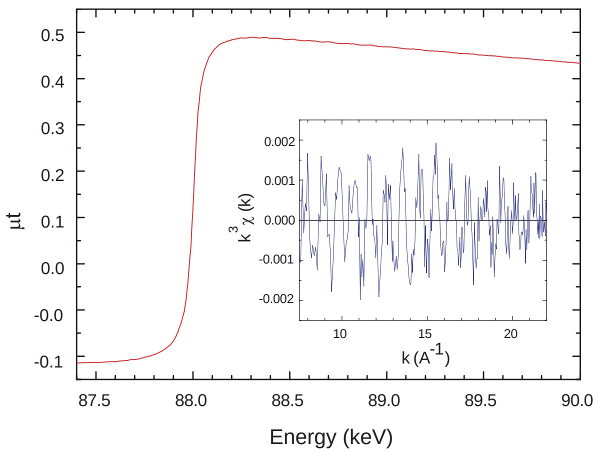
Gas	Helium, Nitrogen, Argon, Krypton, and its mixture
Length/number	6.5 cm/1, 17 cm/2, 31 cm/1
Maximum voltage	2,000 volts

ii) Gas-closed type ionization chamber

Gas	Xenon (1 atm.)
Length/number	17 cm/1, 31 cm/1
Maximum voltage	2,000 volts



Ca K-edge XAFS spectra of $Ca(OH)_2$ measured in transmission mode at room temperature [2]

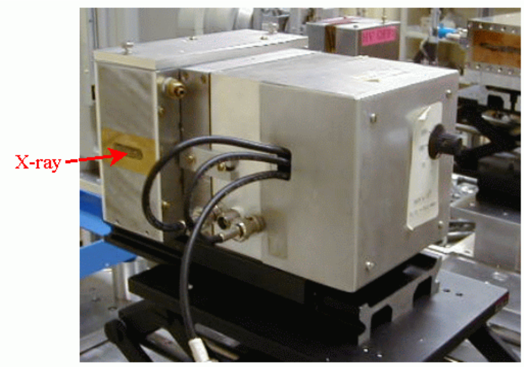


Pb K-edge XAFS spectra of Pb foil in transmission mode at 12 K [3]

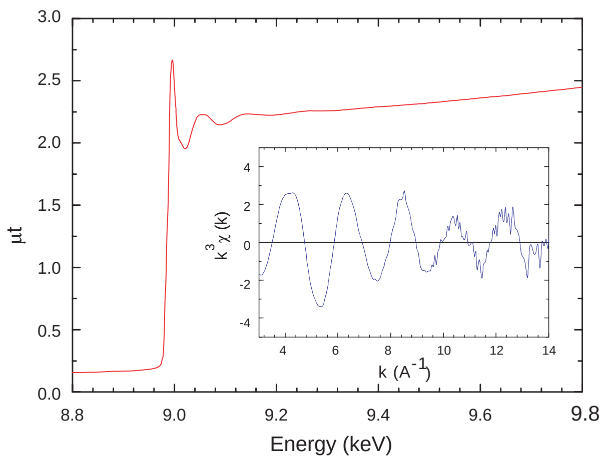
Lytle type detector for fluorescence mode XAFS

The Lytle type detector, which is an ionization chamber with large detection area, is available for fluorescence mode XAFS.

Energy range	3.8 ~ 50 keV
Sample state	dilute sample concentration > 0.1 %
	thin film sample thickness > 100 nm



View of Lytle type detector

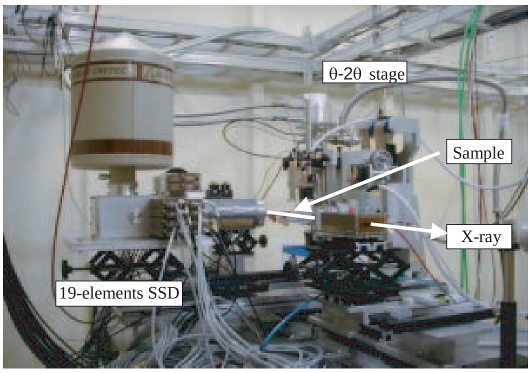


Cu K-edge XAFS spectra of 0.5 wt% Cu/Al_2O_3 catalyst at room temperature [4]

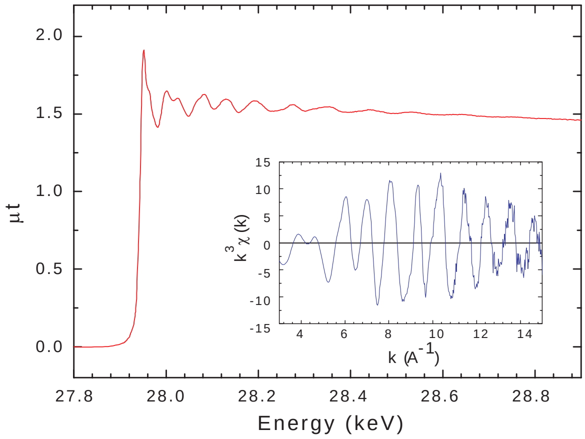
19-element Ge solid-state detector for fluorescence mode XAFS

The 19-element Ge solid-state detector is used for fluorescence mode XAFS for dilute or thin film samples which require the higher energy resolution to measure the isolated fluorescence signal from target materials.

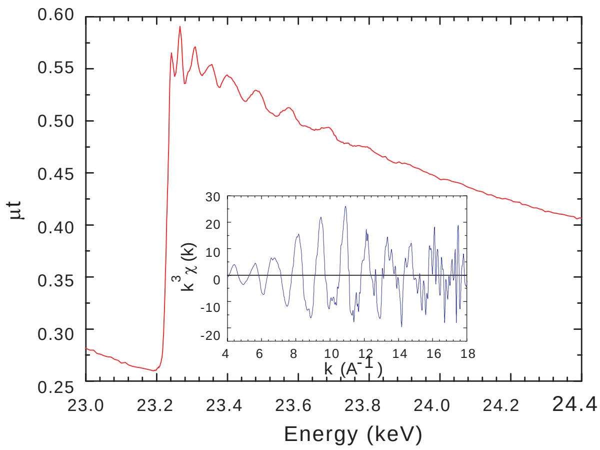
Detectable size	100 mm^2 × 10 mmt × 19 elements
Energy range	3.8 ~ 113 keV
Sample condition	dilute sample concentration > 1 ppm
	thin film sample thickness > 0.1 nm



View of glazing-incidence fluorescence XAFS for thin film sample with 19-element SSD



In K-edge XAFS spectra of 8 wt% In in 3 nm $In_xGa_{1-x}N$ film at room temperature [5]

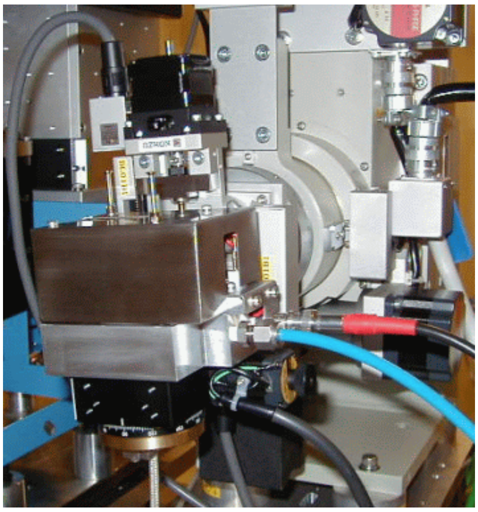


Rh K-edge XAFS spectra of 80 ppm Rh-colloid / EtOH-H_2O at room temperature [6]

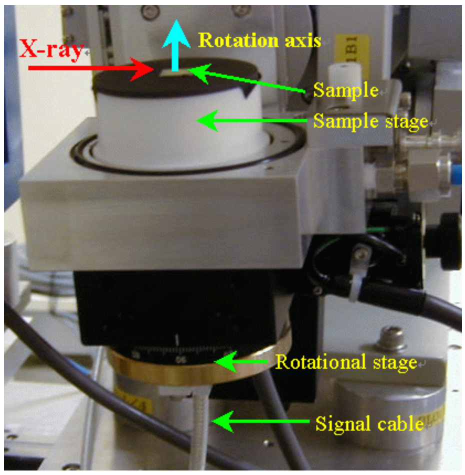
Measurement system in conversion electron yield (CEY) mode

The CEY detector will be mainly used for the thin film samples which has high concentration of core-hole atom. CEY mode has the advantage of detecting signals with high efficiency in comparison with fluorescence mode. Sample stage can be continuously rotated by stepping motor to remove diffraction spikes in XAFS spectrum by setting the diffraction condition off.

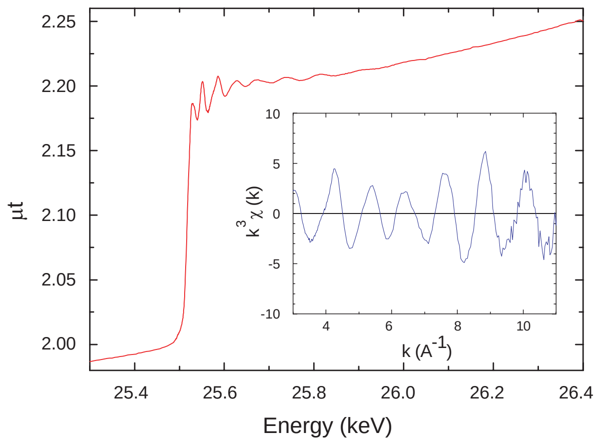
Sample stage size	50 mmϕ
Energy range	3.8 ~ 50 keV
Sample condition	thin film sample thickness > 0.5 nm
	and others



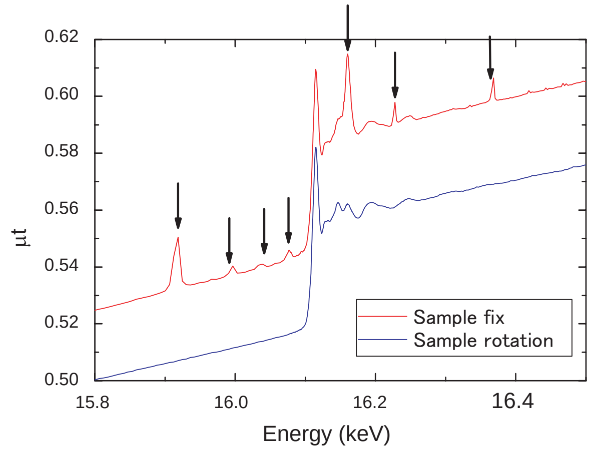
CEY detector mounted on θ-2θ stage



Sample mounted on sample stage



Ag K-edge XAFS spectra of 1 nm Ag thin film on Si substrate at room temperature [7]



Sr K XAFS spectra of (Ba,Sr)TiO_3 thin film on sapphire substrate measured by CEY detector with and without sample rotation [8]. Arrows, diffraction spikes from sapphire substrate.

References

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