

## BL39XU Magnetic Materials

BL39XU is an undulator beamline that is dedicated to hard X-ray spectroscopy and diffractometry requiring control of the X-ray polarization state. The major applications of the beamline are X-ray magnetic circular dichroism (XMCD) spectroscopy and resonant/non-resonant X-ray magnetic scattering in 3d transition metals and compounds, rare-earth elements, and 5d metals. The most important feature of BL39XU is the tunability in X-ray polarization states; horizontal/vertical linear, right/left circular, or arbitrary elliptical polarizations are available using diamond X-ray phase plates. The experimental station is equipped with the two-axis diffractometer with a polarization analyzer, and the XMCD spectrometer. Available sample environments are 20 ~ 300 K and 2 T using the electromagnet with the closed-cycle helium refrigerator. For further high-field and low-temperature environments, the 10 T superconducting magnet system is ready to use. A helicity-modulation technique at 40 Hz for precise XMCD measurements is routinely used. This technique allows extremely high quality XMCD spectra obtained in a short acquisition time.

### Area of research

X-ray magnetic circular dichroism (MCD) spectroscopy  
Element-specific magnetometry  
X-ray emission spectroscopy and its magnetic circular dichroism  
Resonant or non-resonant magnetic scattering

### Keywords

#### Scientific field

X-ray magnetic circular dichroism, Element-specific magnetometry, Magnetic EXAFS, Magnetic diffraction, Magnetic scattering, Polarization XAFS

#### Equipment

X-ray phase plate, Diffractometer, Electromagnet, Superconducting magnet, Cryostat, Polarization analysis

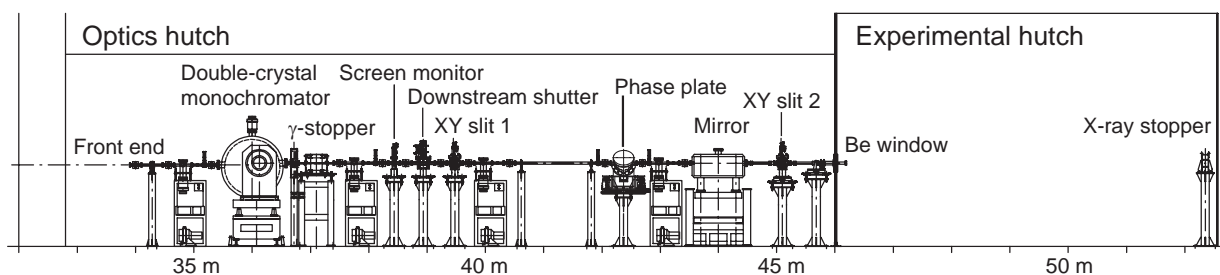
### Source and optics

This beamline is equipped with an in-vacuum type undulator and a rotated-inclined double-crystal monochromator. The combination of fundamental / third harmonics of undulator radiation with the Si 111 reflection of the monochromator enables an energy range from 5 to 37 keV. The photon flux onto a sample is maximized at every X-ray energy by synchronous tuning between the undulator gap and the

monochromator angle.

A platinum-coated mirror of horizontal deflection is used to reduce the amount of higher harmonics to less than  $10^{-4}$ . The cutoff energy is adjustable from 8 to 20 keV with an appropriate glancing angle between 2 and 9 mrad. The mirror is mechanically bendable for providing a horizontally focused beam (0.2 mm in FWHM).

### Beamline layout



A diamond X-ray phase retarder (XPR) is installed between the monochromator and the mirror. It functions as a quarterwaveplate with high efficiency. Crystals of various thicknesses (0.34, 0.45, 0.73, and 2.7 mm) are available; selection of an appropriate crystal allows the use of high rate (> 90%) circular polarization of both helicities at 6~16 keV. Vertical linear polarization of a reasonable polarization rate (40 ~ 90%) is also available with an XPR used under the halfwaveplate condition.

### X-ray beam parameters

Energy range	5 ~ 37 keV
Resolution $\Delta E/E$	$2 \times 10^{-4}$
Flux at sample	$4 \times 10^{13}$ photons/s
Beam size at sample (FWHM)	$0.6 (V) \times 2 (H) \text{ mm}^2$
Higher harmonic content	$< 1 \times 10^{-4}$
Linear polarization rate	99.9 %
Circular polarization rate	> 90 %

### Experimental stations

#### Facilities

- Diffractometer  
one rotation axis for sample orientation, two axes for counter, and four-circle goniometer for polarization analysis
- Electromagnet  
 $H = 0.6, 1.1, 2.0 \text{ T}$  with a 45, 20, 10 mm-poles gap, respectively
- Superconducting magnet with variable temperature insert  
 $H = 0 \sim 10 \text{ T}$   
temperature range: 1.7 ~ 300 K
- Cryostat A  
temperature range : 15 ~ 300 K, fitted with the electromagnet
- Cryostat B  
temperature range : 15 ~ 300 K, fitted with the four-circle goniometer
- Ionization chambers
- NaI scintillation counter
- Si (Li) detector
- Silicon drift chamber detector
- Fluorescence ionization chamber (Lytle detector)
- Lock-in amplifier for magnetic circular dichroism measurements in helicity-modulation mode.

#### Two-axis diffractometer with polarization analyzer

The diffractometer is designed for resonant/non-resonant magnetic scattering in ferro/antiferro magnets. The component two-axis goniometer is for the orientation of

samples and is capable of supporting either the electromagnet or the 10 T superconducting magnet. The combination of this two-axis and the four-circle goniometer on the robust  $2\Theta$ -table allows diffraction experiments with linear polarization analysis of the scattered beam. Perfect Si channel-cut crystals and plane crystals of good mosaicity (LiF and graphite) are available for mounting on the four-circle goniometer. The angular resolution of the two-axis goniometer is 0.36 sec/step, while that of the four-circle one is 0.72 sec/step. Furthermore, the  $2\Theta$ -table is used as an optical bench for X-ray magnetic circular dichroism (XMCD) experiments.

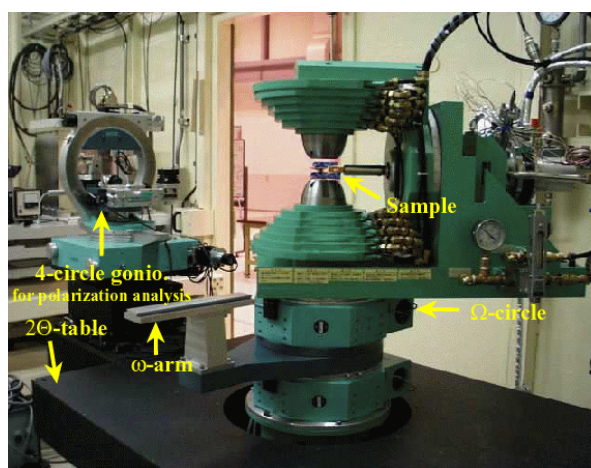


Fig.1. The two-axis diffractometer with a polarization analyzer. The electromagnet and the closed-cycle helium refrigerator are mounted on the  $\Omega$ -circle.

#### Electromagnet and closed-cycle helium refrigerator

The normal conducting electromagnet is used for XMCD and magnetic diffraction experiments. It generates a maximum magnetic field of 2 T at a 45 A feeding current with a set of poles placed 10 mm apart. Three sets of poles with different gaps are replaceable, and the available magnetic field changes correspondingly. The maximum field available with the use of each set of poles is shown in Table 1. The field polarity is reversed in 1.7 s by changing the polarity of the feeding current. The magnet is mountable on the  $\Omega$ -circle of the diffractometer and is rotatable about the vertical axis. Additionally, the poles are rotatable in the vertical plane; field directions can be changed in parallel and perpendicular orientations, or at any desired angle with respect to the incident X-ray beam. This compatibility to the diffractometer and the flexibility in the field direction allows experiments of resonant exchange scattering using the electromagnet.

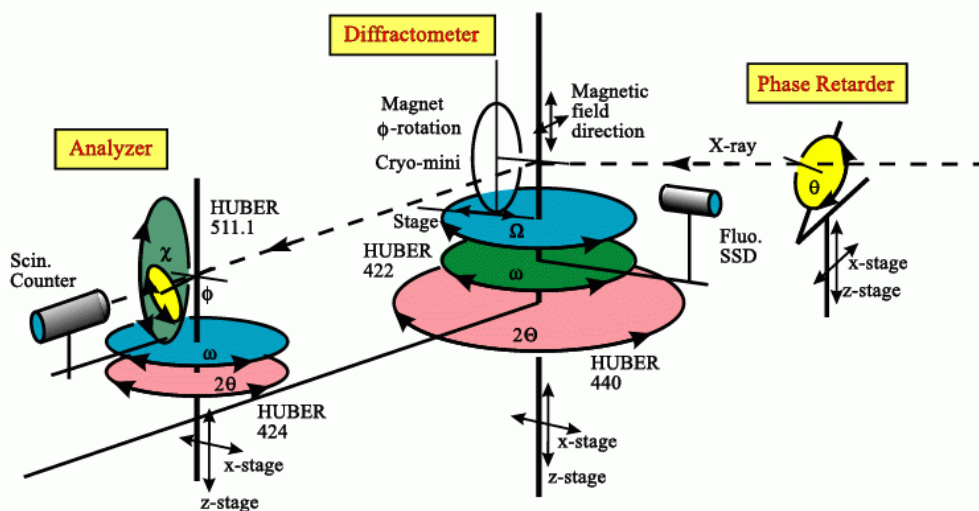


Fig. 2. The configuration of the axes of the diffractometer

The closed-cycle helium refrigerator can cool a sample to 20 K within 90 min. The controllable temperature range spans between 20 and 300 K. The cold head is fit to the electromagnet. The refrigerator generates very little vibration making possible diffraction measurements of single crystalline samples at low temperatures. A number of sample rods are available; users can choose one appropriate for their experiment (absorption or diffraction) as well as for fitting to the gap between the poles. The lowest temperatures available with the sample rods are shown in Table 1.

Table 1. The maximum magnetic field and the lowest temperatures available from the use of a set of poles and a compatible sample rod

Pole gaps (mm)	45	20	10
Maximum magnetic field (T)	0.61	1.09	1.98
Lowest temp. (absorption) (K)	20	26	24
Lowest temp. (diffraction) (K)	48	---	---

### 10-T superconducting magnet

The superconducting magnet (SCM) is designed for XMCD experiments under a magnetic field up to 10 T. The assembly of a variable temperature insert (VTI) allows measurements between 1.7 and 300 K. The SCM is mountable on the  $\Omega$ -circle of the diffractometer.

Figure 3 shows a schematic drawing of the SCM system. The split-type superconducting coils are equipped to generate a field in the horizontal direction. The diameter of the magnet clear bore is 54 mm. A sample is placed inside a 25 mm-diameter cylinder in the VTI. The SCM has

X-ray transparent Be windows at both front and back (on the field axis) and on both sides (perpendicular to the field). The opening size of the front and back windows is  $\phi 10$  mm, while the sides are  $\phi 20$  mm. This design of the coils and the windows allows XMCD measurements in either transmission or fluorescence mode as well as non-resonant magnetic diffraction experiments with a  $90^\circ$  scattering angle. The SCM is equipped with a liquid helium recondensing cooler which enables continuous operation of the SCM for more than 7 days with no additional coolant needed.

At the moment (December 2003), the SCM is usable for XMCD experiments but not for diffraction measurements because of a vibration problem. Sample vibrations are found to have the amplitude of approximately  $\pm 30 \mu\text{m}$ , caused by the vibration of the recondensing cooler. Vibration of this magnitude results in a small degradation in the quality of XMCD measurements; the effect is negligible in most cases. However, this vibration will seriously influence data taken during diffraction measurements with a single crystalline sample. To obtain correct diffraction data, an R&D for suppressing the vibration is in progress.

Table 2. Available sample environments using the superconducting magnet

Magnetic field (T)	0 ~ 10
Temperature range (K)	1.7 ~ 300

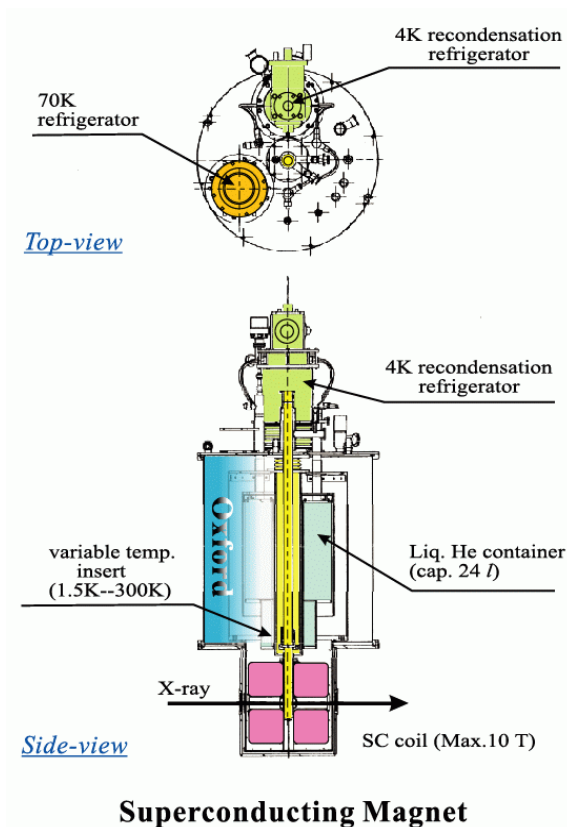


Fig. 3. A schematic illustration of the superconducting magnet installed in the experimental hutch

### Helicity-modulation XMCD

At BL39XU, the helicity-modulation technique for precise XMCD measurements has been developed by combining an XPR with a phase-sensitive (lock-in) detection system. This technique provides us with extremely high quality XMCD spectra in short measurement times. A dichroism signal in the order of  $10^{-4}$  is obtainable with a good signal-to-noise ratio for 10 s-integration times at each energy point with a properly prepared sample. Additionally, XMCD measurements during a magnetic saturation process and under high magnetic fields generated with a superconducting magnet are feasible.

### Accessories

- Four-jaw slit
- Replaceable attenuator
- Vacuum pipe for X-ray path
- Oil-free scroll pump
- Light chopper (chopping frequency : 5 ~ 20000 Hz)
- Digital oscilloscope
- Multi-channel analyzer
- Digital multimeter
- Pen recorder

- Monitor camera system
- Dewar vessel for liquid nitrogen
- Magnetometer
- Desiccator and vacuum pump
- Ultrasonic cleaner

### Examples of experimental data

Figure 4 shows an example of XMCD data measured at BL39XU. An XMCD spectrum of  $Mn_3ZnC$  powder sample was recorded at the Mn K-edge (6540 eV), using the helicity-modulation technique in the transmission mode. The XMCD signal is as small as 0.1% of the XANES step height. Good statistic accuracy of 0.01% was obtained in a measuring time of about one hour.

An element-specific magnetization curve is obtained by monitoring XMCD amplitude at the absorption edge of a particular element. Figure 5 shows element-specific magnetization curves of Gd (a) and Fe (b) of a  $Gd(20 \text{ \AA})/Fe(20 \text{ \AA})$  multilayer film. This results clearly shows that Gd and Fe magnetic moments couple antiferromagnetically. The magnetization curves of Gd and Fe are quite different from the total magnetization curve, shown by a solid line in Fig. 5(a).

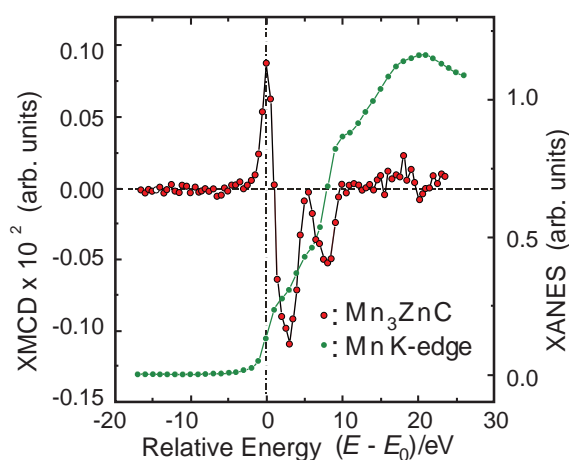


Fig. 4. An X-ray magnetic circular dichroism (XMCD) spectrum of  $Mn_3ZnC$  in the ferromagnetic phase at 300 K, measured at the Mn K-edge ( $E_0 = 6540 \text{ eV}$ ). The XANES spectrum is compared.

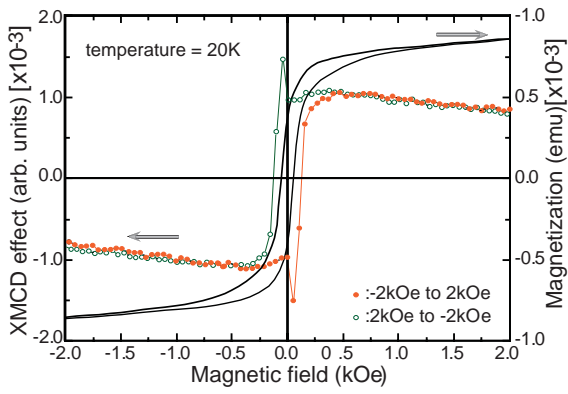


Fig. 5 (a).

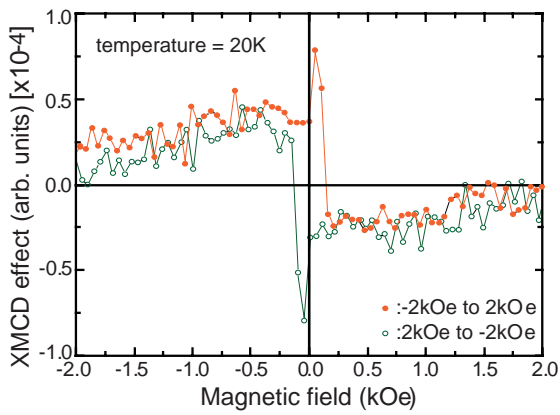


Fig. 5 (b).

Fig. 5. Element-specific magnetization curves of a Gd(20 Å)/Fe(20 Å) multilayer film: (a) Gd magnetization curve (dots), compared with the total magnetization curve (solid line), and (b) Fe magnetization curve (dots).

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### Contact information

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