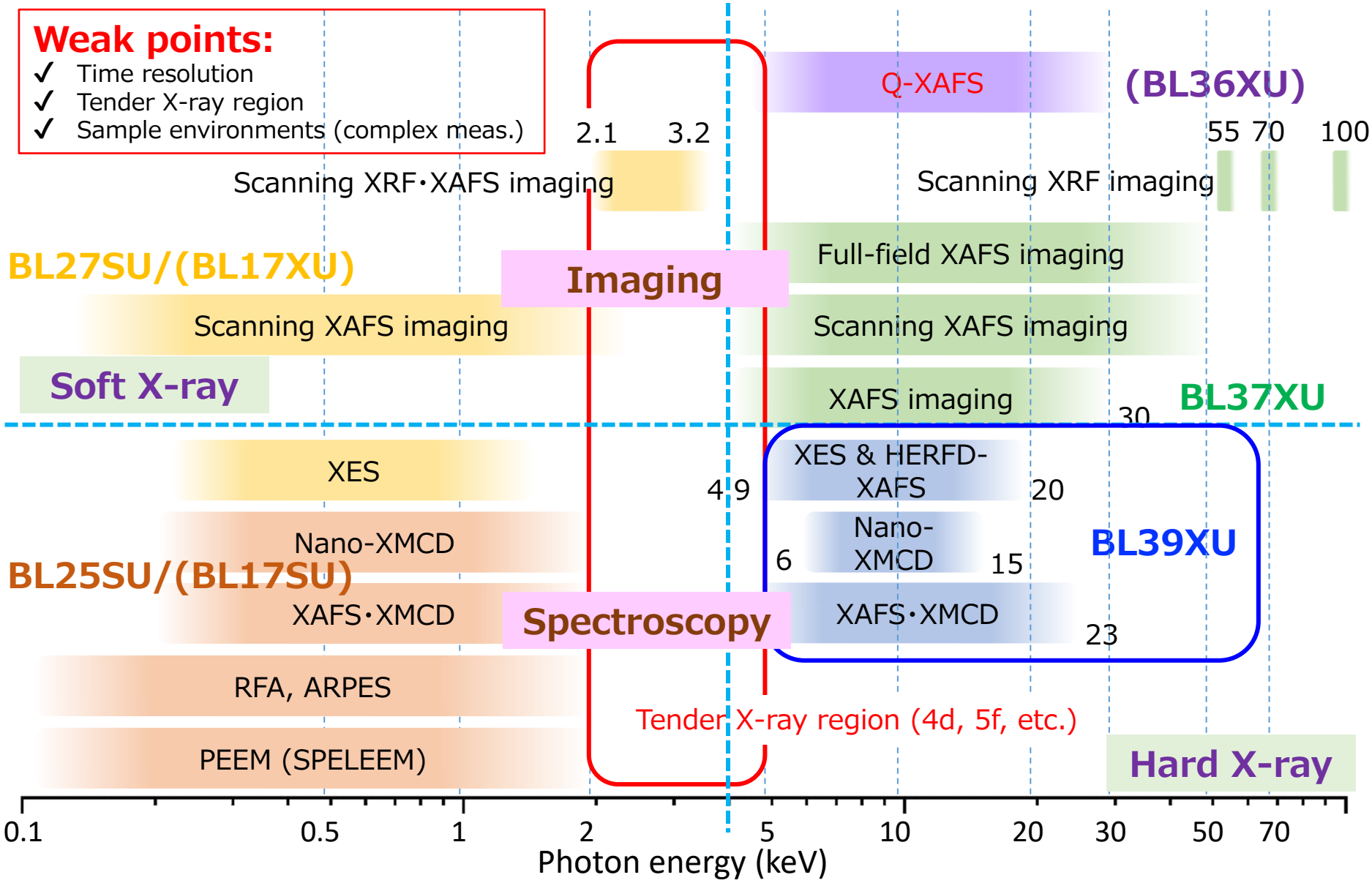
BL39XU のアップグレードの現状について

公益財団法人 高輝度光科学研究センター (JASRI)
放射光利用研究基盤センター
分光推進室

河村　直己
東　晃太朗, 登野　健介
三浦　芳樹, 稲田　達彰

Past & present status: Spectroscopy BLs

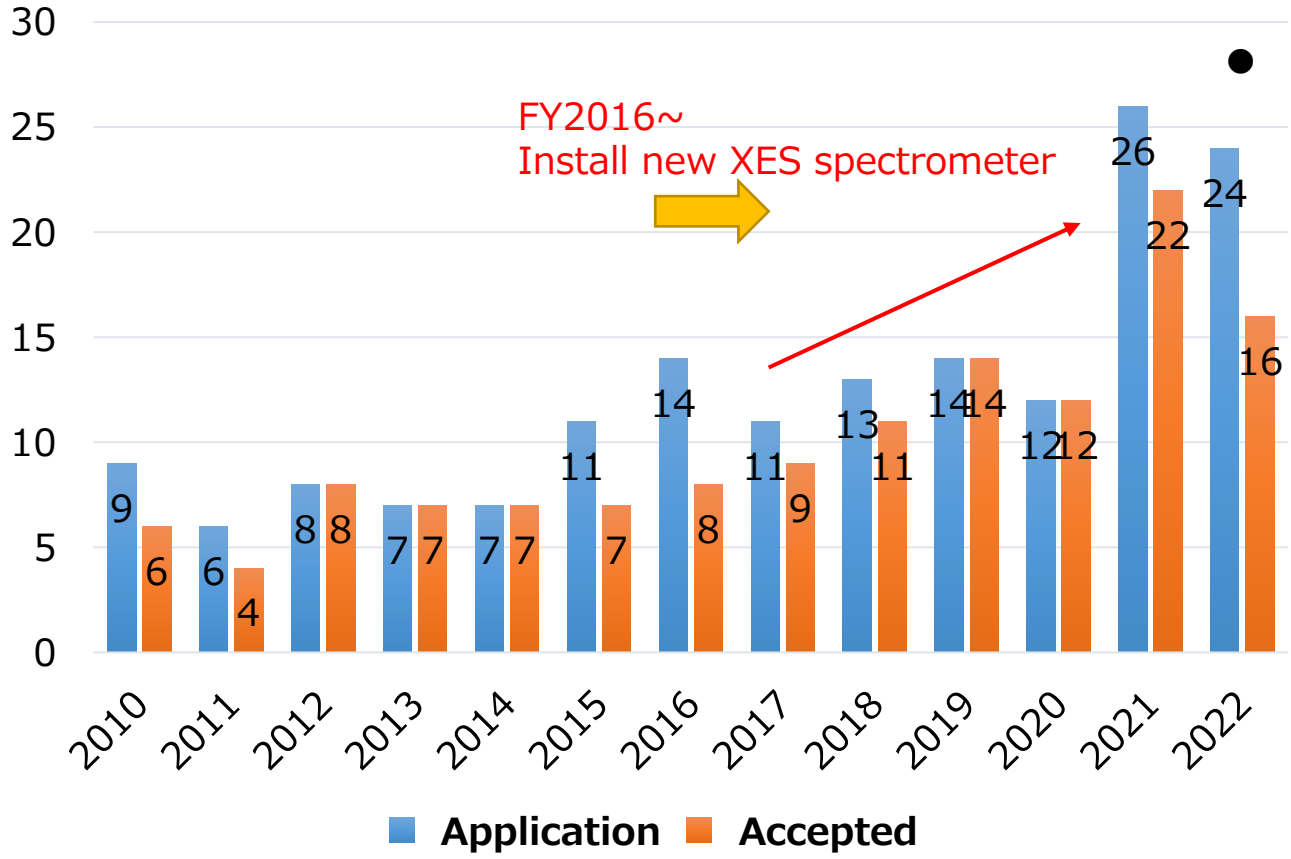


BL39XU: Experimental methods

Method & Field	Abbreviation	Instruments
EH1: X-ray spectroscopy under extreme conditions		
Extreme XMCD High-pressure EXAFS	E-XMCD HP-EXAFS	Circular pol., Mag. field, High press., Low/high temp.
EXAFS/XAFS Polarization XAFS **EH1-①**	EXAFS-XANES P-EXAFS	Microbeam, Multi-element SDD Vertical (Circular) pol.
X-ray emission spectroscopy Inelastic scatter. X-ray Raman scatter. **EH1-②**	XES RIXS/NIXS XRS	XES spectrometer Vertical/Circular pol., Mag. Field, High press., Low/High temp.
Resonant X-ray mag. Scatter.	XRMS	Circular pol., Mag. field
EH2: X-ray nanospectroscopy		
Nano-XMCD Nano-XAFS	N-XMCD N-EXAFS	Circular pol., Mag. field Multi-element SDD
Nano-XRD XAFS/XMCD 2D imaging **EH2**	N-XRD Img-CT	Carry-on Mag. field, Multi-axes stage for CT
X-ray Fluo. Holography	XFH	Microbeam, Carrry-on
Others (Development)	Others	

BL39XU: Proposals for XES exp.

XES-related proposals



- Proposals and shifts: account for 30~40% of the total beamtime
- Scientific fields:
  - ✓ Physics & Chemistry
  - ✓ Environment chem.
  - ↓
  - ✓ Increase of industrial users (Catalysts・Batteries・Environment)
- Non-proprietary priority & proprietary proposals

Term	Shifts
2023A	48
2022B	48
2022A	30
2021B	51

Gradual increase of application & accepted proposals

BL39XU: Beamline Upgrade

BL39XU Upgrade (July 2023 ~ July 2024)

1. Nano-spectroscopy imaging: Replace **higher-harmonics cut mirrors (HCM)**
2. High activity: Construct **new experimental hutch** for X-ray emission spectroscopy
3. High efficiency: Install **focusing mirrors (KB, Wolter)** for each experimental hutch
4. Various polarization: Install **double X-ray phase retarder (DXPR)**
   with crossed-Nicols type

EH1: X-ray spectroscopy under multiple extreme conditions

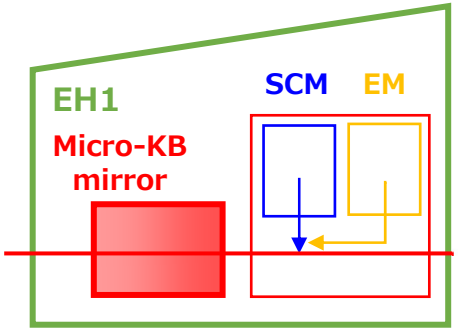
- ✓ XAFS･XMCD + XRD
- ✓ High magnetic field & high pressure

EH2: X-ray emission spectroscopy

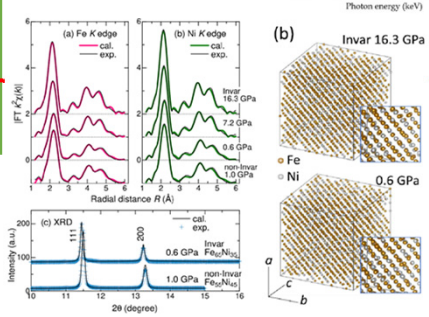
- ✓ XES･HERFD-XAFS･XRS
- ✓ Operando/in-situ meas.

EH3: X-ray nanospectroscopy

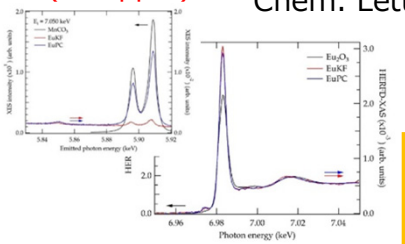
- ✓ XAFS･XMCD, XRF, imaging
- ✓ Pump-probe meas.



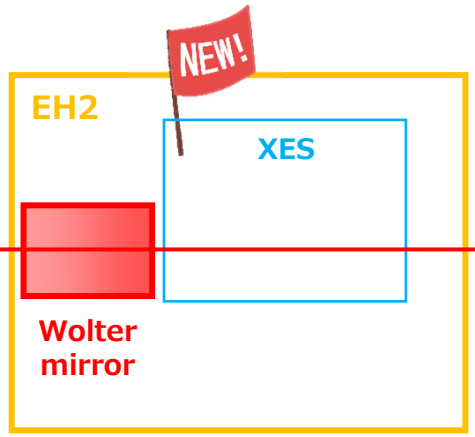
EXAFS + XRD:
PRB 103, L220102 (2021).

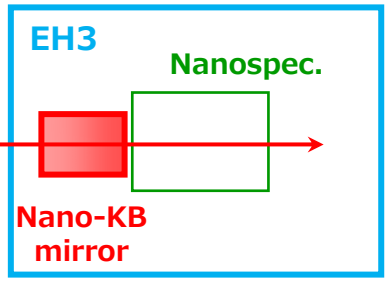


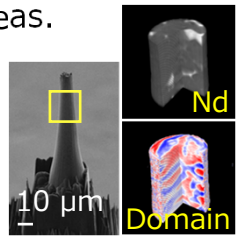
Eu (< 5 ppm)



XES/HERFD for dilute elements:
Chem. Lett. 50, 1570 (2021).







Nd-Fe-B magnet

XMCD-CT:
APEX 11, 036601 (2018).

Beamline name

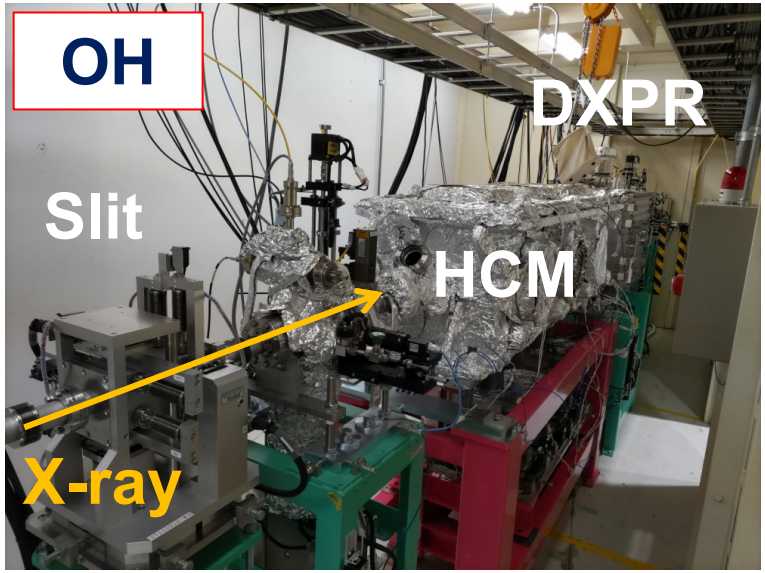
■ ビームラインの名称変更

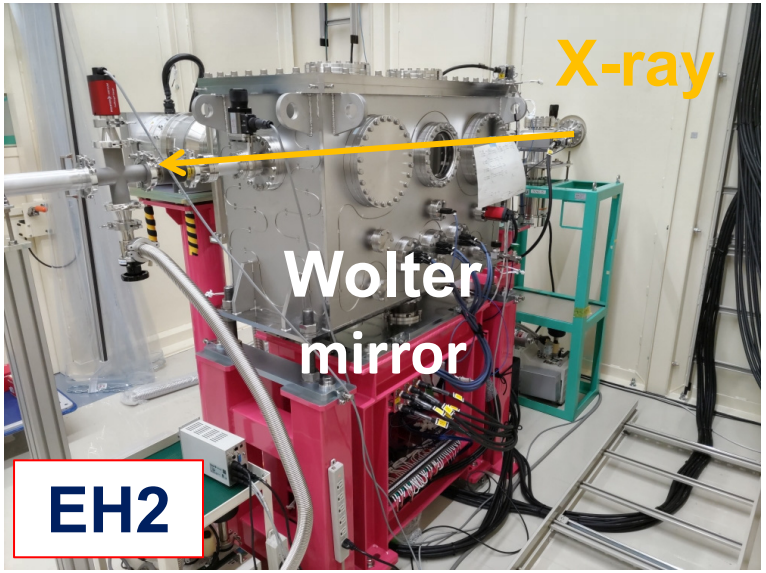
X線吸収･発光分光
X-ray absorption and emission spectroscopy

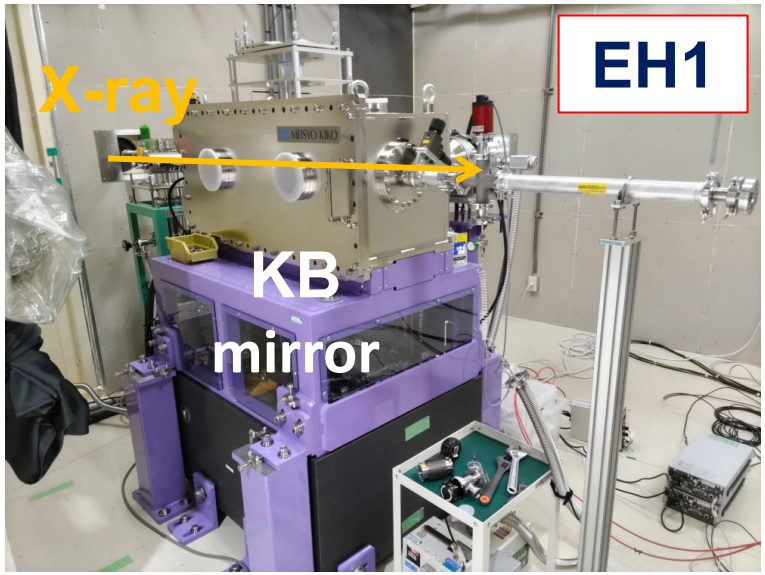
- ✓ 複合極限環境下 XAFS & XMCD @ EH1
- ✓ X線発光分光 (XES) @ EH2
- ✓ X線ナノ分光 (XAFS & XMCD) @ EH3

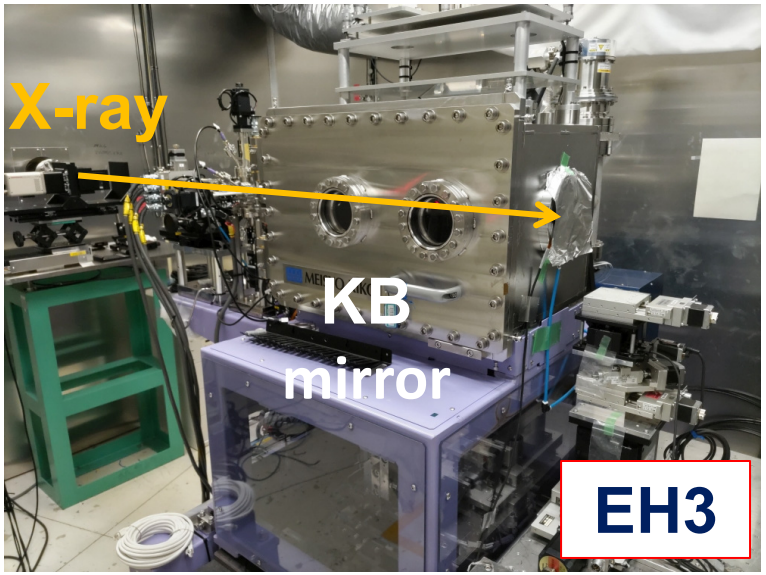
■ 磁性材料 (Magnetic materials) [2002~]
■ 生体分析 (Physicochemical) [1997~]

BL39XU: Current status @ each hutches



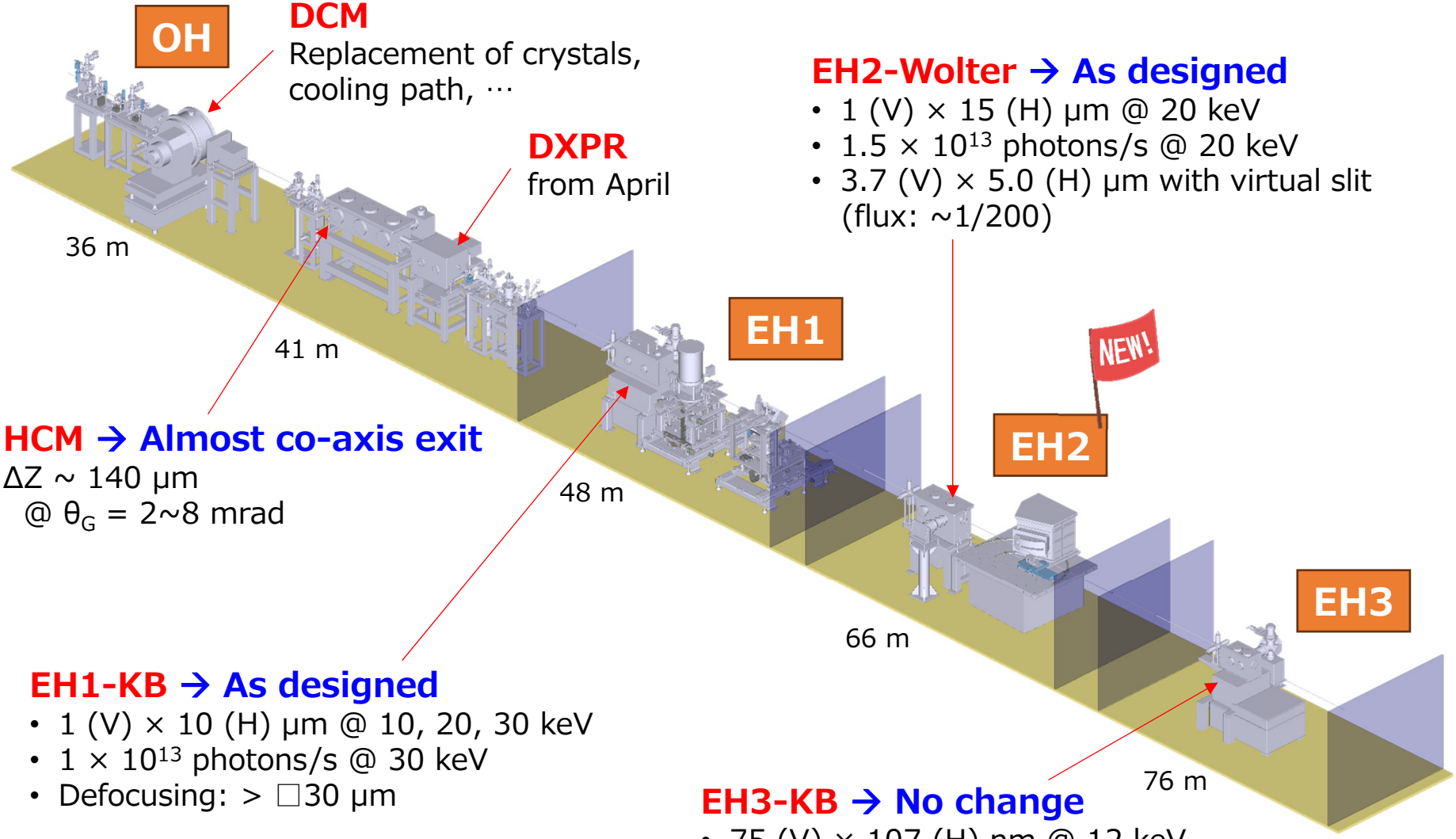






BL39XU: Commissioning status

Since January 2024



EH2-Wolter → As designed
- 1 (V) × 15 (H) μm @ 20 keV
- 1.5 × 10^{13} photons/s @ 20 keV
- 3.7 (V) × 5.0 (H) μm with virtual slit (flux: ~1/200)

HCM → Almost co-axis exit
ΔZ ~ 140 μm
@ θ_G = 2~8 mrad

EH1-KB → As designed
- 1 (V) × 10 (H) μm @ 10, 20, 30 keV
- 1 × 10^{13} photons/s @ 30 keV
- Defocusing: > □30 μm

EH3-KB → No change
- 75 (V) × 107 (H) nm @ 12 keV
- ~10^{11} photons/s

BL39XU: Before and after upgrade

	After	Before
OH	• **Triple** HCM **(co-axis)** • **Double** XPR (plan) • Attenuator in UHV (Al × 10, Si × 5)	• **Single** HCM **(deflect)** • **Single** XPR • Attenuator in vacuum (Al × 5)
EH1: **Extreme XAS·XMCD**	• **KB mirror (UHV)** ✓ 1 (V) × 10 (H) μm ✓ < **30 keV** ✓ 3 × 10^{13} photons/s @ 10 keV	• KB mirror **(He)** ✓ 1.5 (V) × 9.5 (H) μm ✓ < **9.5 keV** ✓ 3 ×10^{12} photons/s @ 7 keV
EH2: **XES·HERFD-XAS**	• **Monolithic Wolter (UHV)** ✓ **1 (V) × 15 (H) μm** ✓ < 20 keV ✓ 2 × 10^{13} photons/s @ 12 keV	• HCM bent (Vacuum) ✓ **300 (V) × 110 (H) μm** ✓ < 28 keV ✓ 6 × 10^{12} photons/s @ 12 keV
EH3: **Nano XAS·XMCD**	• KB mirror (UHV) ✓ 75 (V) × 110 (H) nm (confirm) ✓ **4.92** ~ 16 keV ✓ ~10^{11} photons/s @ 12 keV	• KB mirror (UHV) ✓ 53 (V) × 47 (H) nm (record) ✓ **6** ~ 16 keV ✓ ~10^{11} photons/s @ 12 keV

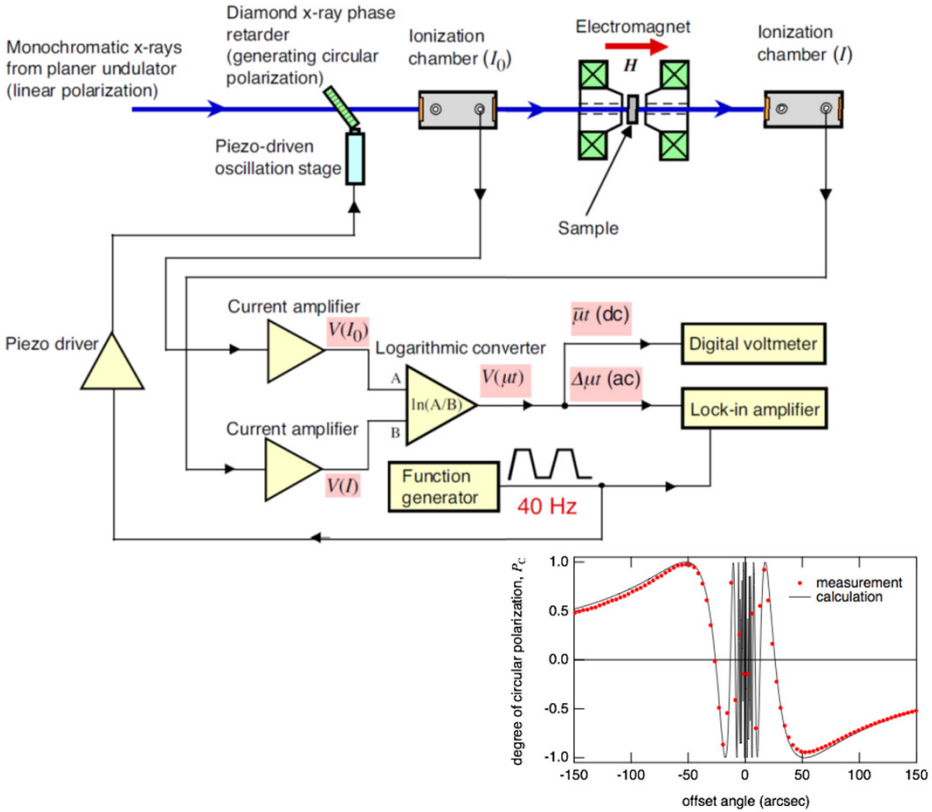
※ Not include HCM reflection

BL39XU: Variable polarization

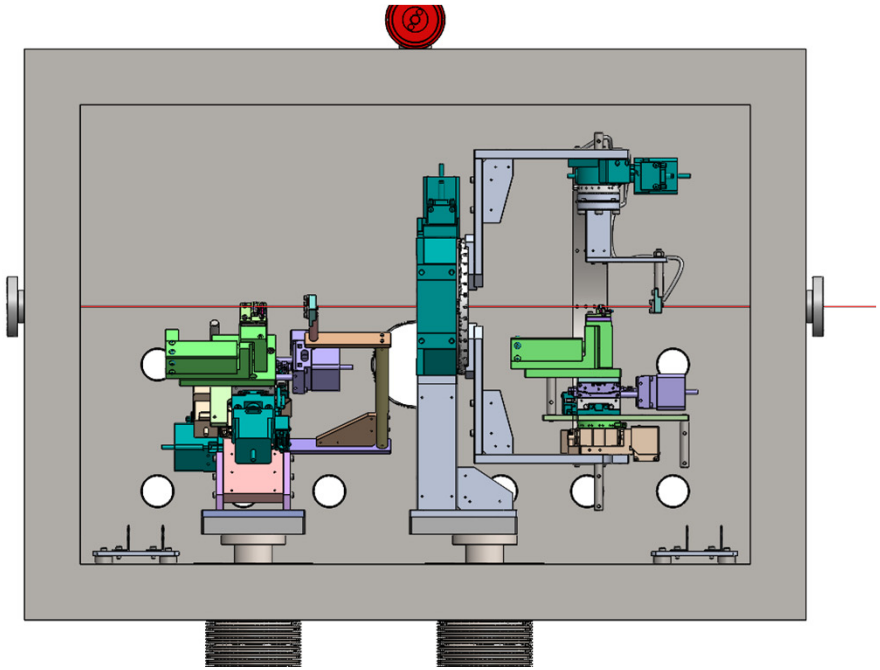
Fast polarization switching

- ✓ Left- and right-circular polarization switching
- ✓ Horizontal- and vertical-linear polarization switching
- ✓ Distribution of X-ray fluorescence signals by gate-circuit

Helicity-modulation technique → Linear-polarization modulation



Double X-ray phase retarder system



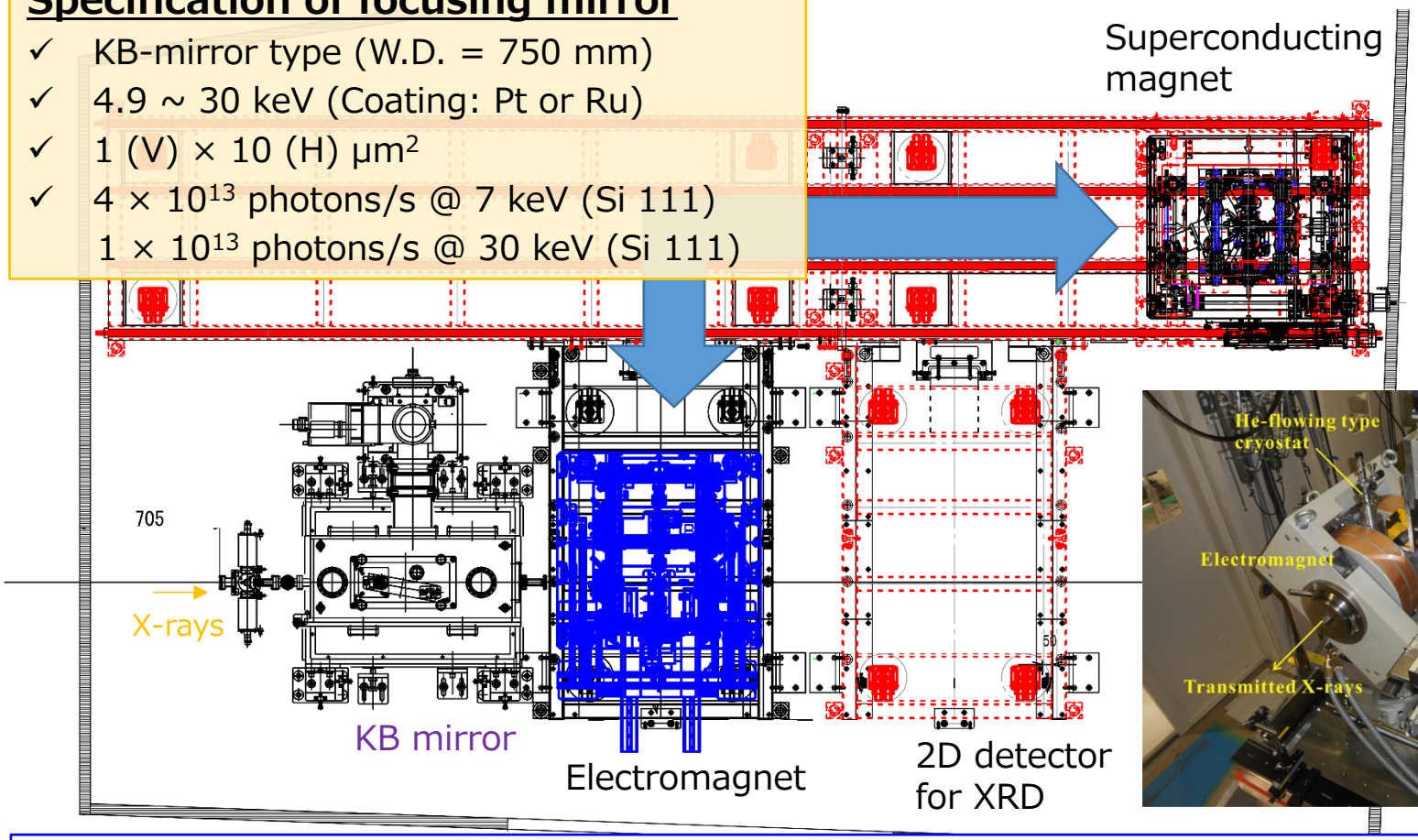
M. Suzuki, et al., JJAP **37**, L1488 (1998).

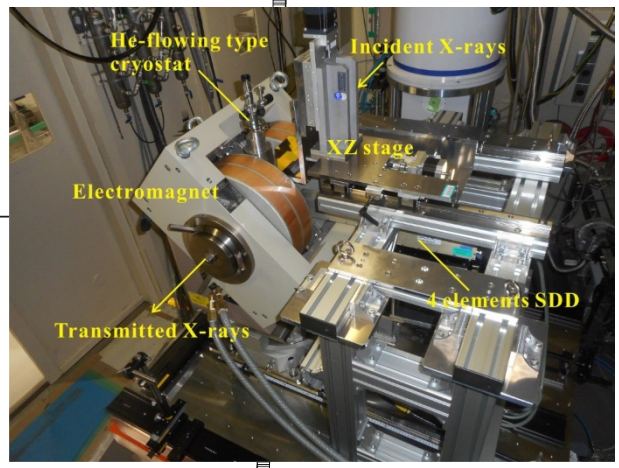
BL39XU: Exp. Hutch1 (EH1)

XAS & XMCD spectroscopy under multiple-extreme conditions

Specification of focusing mirror

- ✓ KB-mirror type (W.D. = 750 mm)
- ✓ 4.9 ~ 30 keV (Coating: Pt or Ru)
- ✓ 1 (V) × 10 (H) μm^2
- ✓ 4 × 10^{13} photons/s @ 7 keV (Si 111)
  1 × 10^{13} photons/s @ 30 keV (Si 111)





Future issues:

- Expansion to higher magnetic field, higher pressure, and its complex conditions
- 2D imaging detector with high-efficiency for XRD measurements

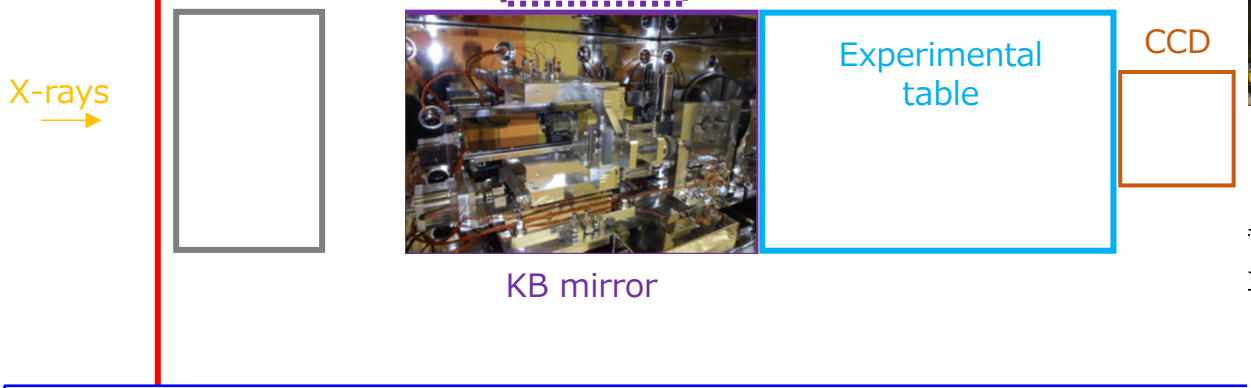
BL39XU: Exp. Hutch3 (EH3)

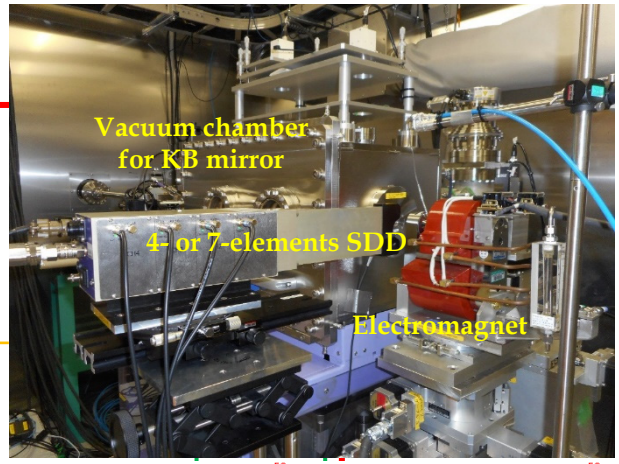
XAS & XMCD microscopy & imaging

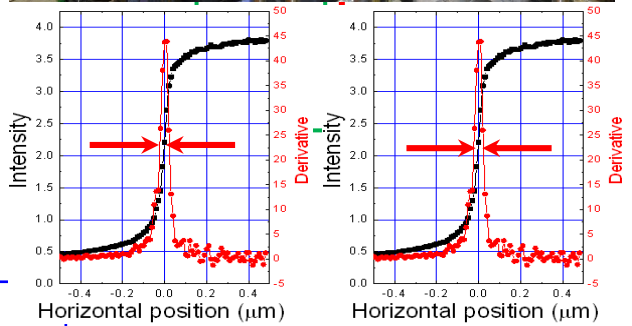
- ✓ Temperature: RT → expand LT/HT measurements
- ✓ Magnetic field: ~2.4 T (EM) / ~1 T (Projection EM)
- ✓ Fluorescence detector: 4- or 7-elements SDD + Fast DSP

Specification of focusing mirror

- ✓ 4.92 ~ 16 keV (Coating: Rh) (W.D. = 100 mm)
- ✓ 100~300 (V) × 100~300 (H) nm^2
- ✓ 3×10^{11} photons/s @ 11 keV (□100 nm)
  1×10^{12} photons/s @ 11 keV (□300 nm)







53 nm　47 nm

Minimum beam size with virtual slit

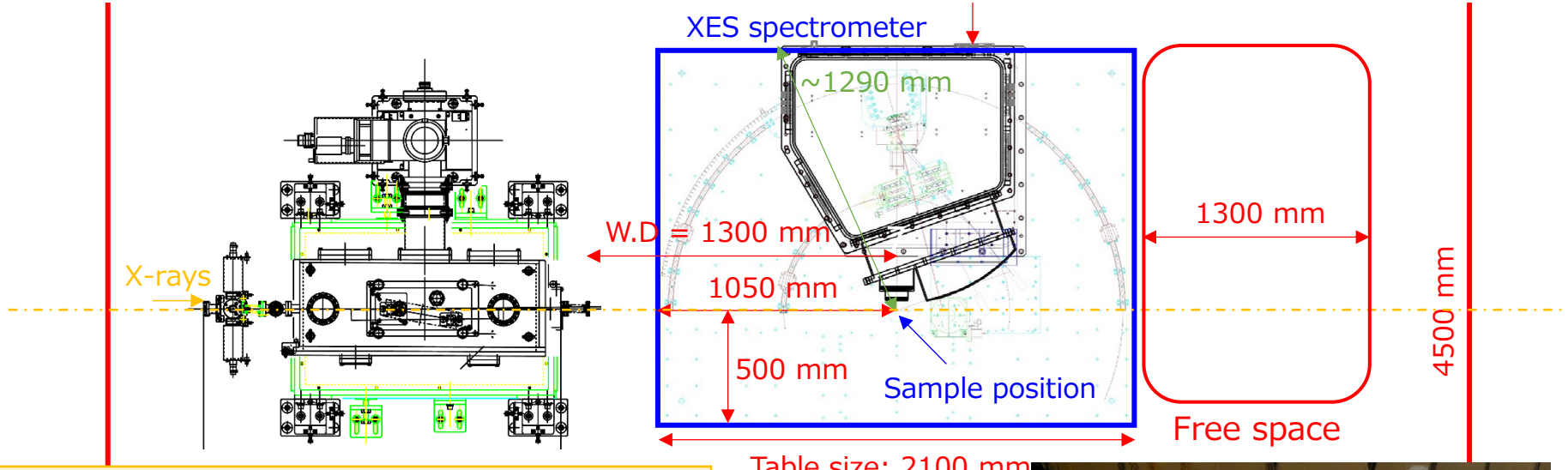
Future issues:

- Temperature control system (cryostat, heater, etc.)
- High-speed data acquisition → high-efficiency fluorescence system

BL39XU: Exp. Hutch2 (EH2)

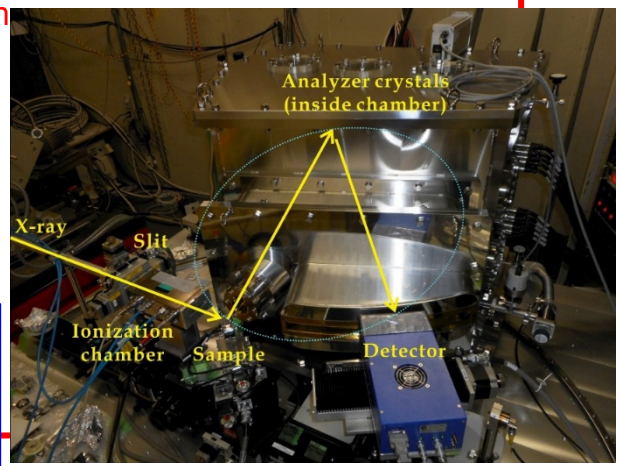
XES·HERFD-XAS spectroscopy

- ✓ Available emission energy range: 4.4 ~ 20 keV
- ✓ Detector: PILATUS 100K (Si), SOPHIAS (Si), PiXirad-2 (CdTe)



Specification of focusing mirror

- ✓ Wolter-mirror type (W.D. = 1300 mm)
- ✓ 4.9 ~ 20 keV (Coating: Ru)
- ✓ 1 (V) × 15 (H) μm^2
- ✓ 2 × 10^{13} photons/s @ 12 keV (Si 111)



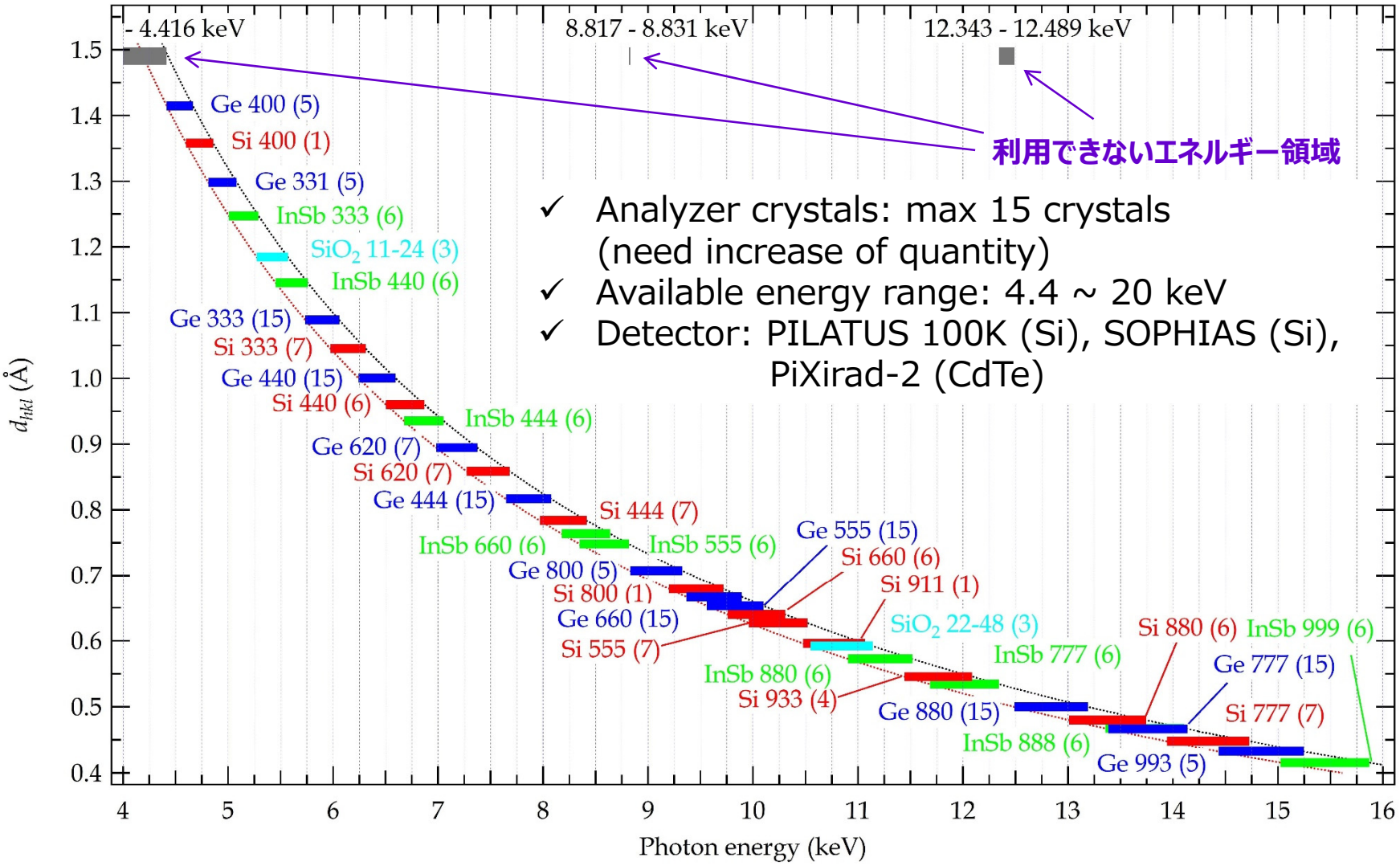
Future issues:

- Expanded with high efficiency above 20 keV and below 4.4 keV
- High efficiency, high throughput, sample environment

BL39XU: Analyzer crystals for XES

X-ray emission spectroscopy

( ) Available analyzer crystals



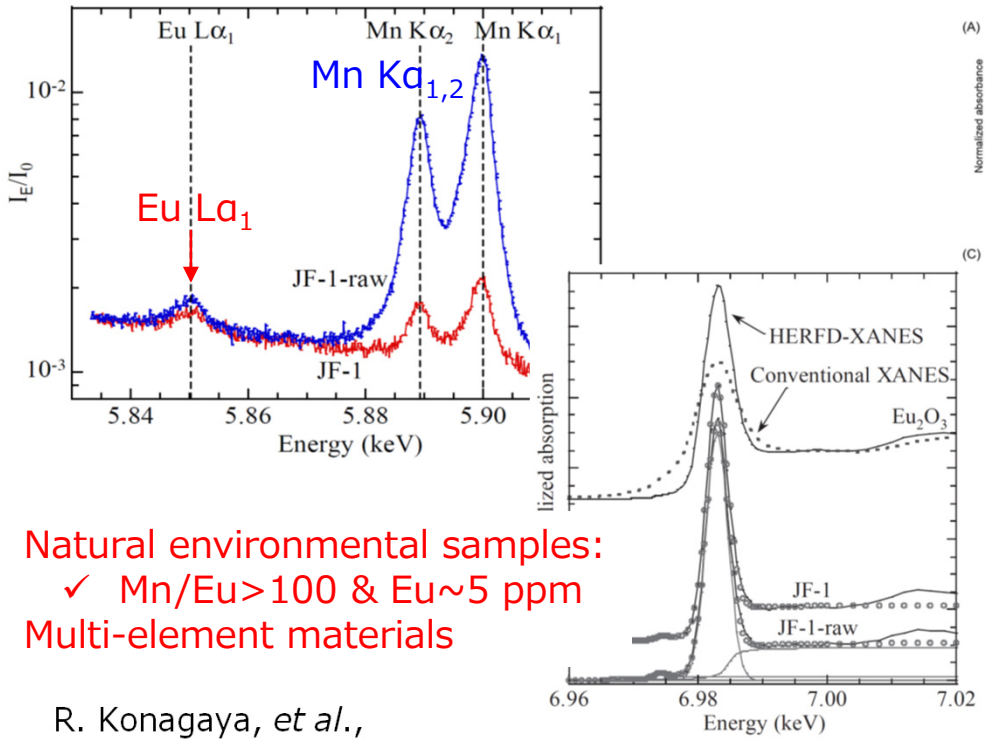
- ✓ Analyzer crystals: max 15 crystals (need increase of quantity)
- ✓ Available energy range: 4.4 ~ 20 keV
- ✓ Detector: PILATUS 100K (Si), SOPHIAS (Si), PiXirad-2 (CdTe)

BL39XU: XES & HERFD-XAS @ EH2

HERFD-XAS in catalytic and environmental samples

- ✓ High-energy resolution & precision measurements
- ✓ Extreme small variation in reaction phenomena
- ✓ Electronic state of buried elements

Valence estimation of dilute Eu in natural environmental samples



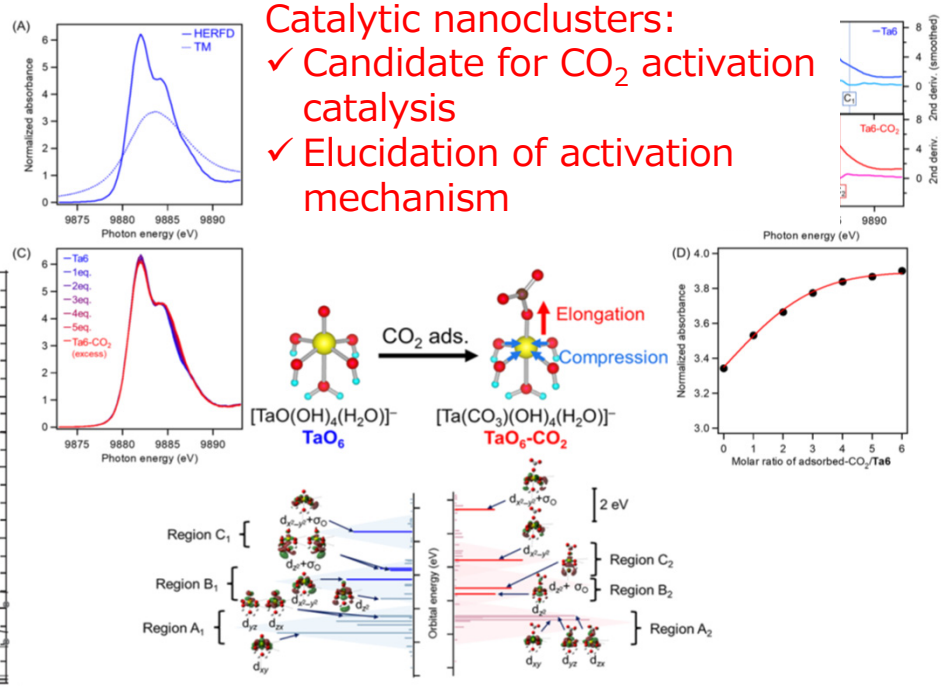
Natural environmental samples:
- ✓ Mn/Eu>100 & Eu~5 ppm

Multi-element materials

R. Konagaya, *et al.*, Chem. Lett. **50**, 1570 (2021).

CO_2 activation on polyoxotantalete nanocluster

Catalytic nanoclusters:
- ✓ Candidate for CO_2 activation catalysis
- ✓ Elucidation of activation mechanism



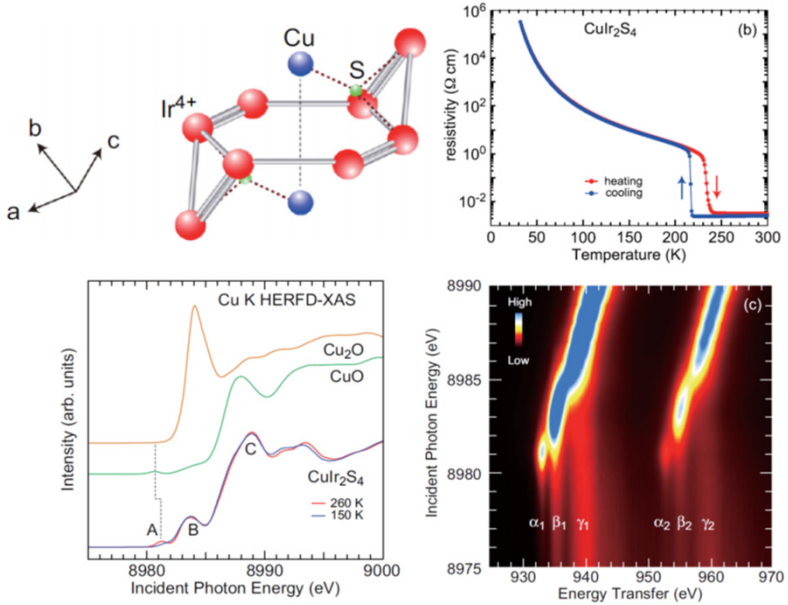
T. Matsuyama, *et al.*, J. Phys. Chem. C (2024). *to be published.*

BL39XU: XES & HERFD-XAS @ EH2

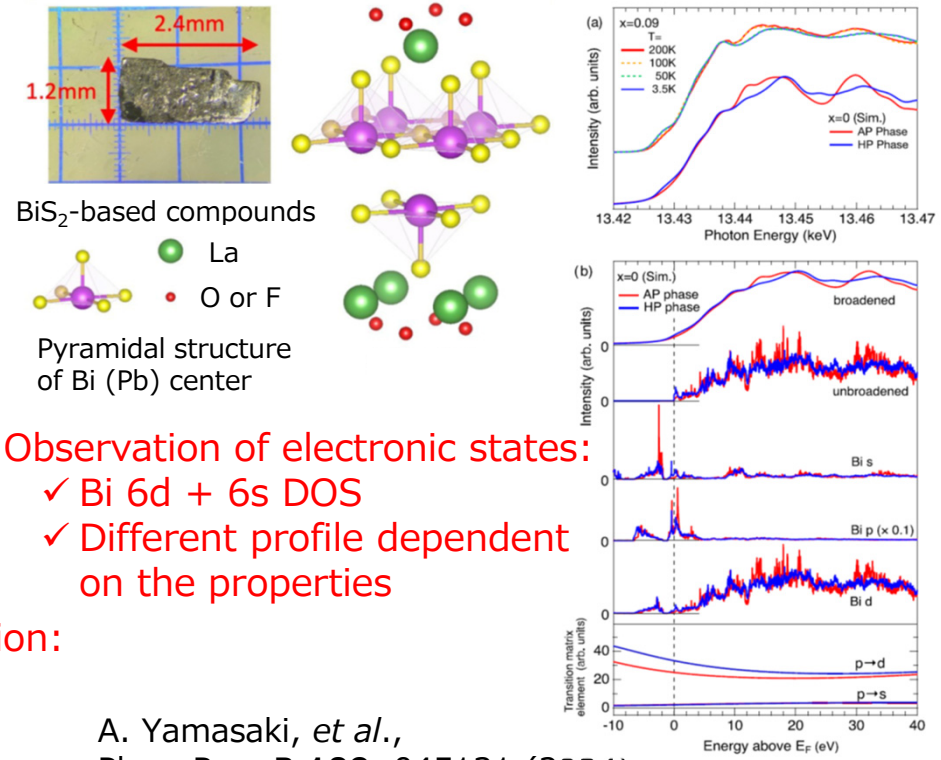
XES & HERFD-XAS for strongly correlated electron systems

- ✓ Precise valence estimation and chemical shifts
- ✓ Electronic states under extreme conditions
- ✓ Symmetry in electronic orbitals using X-ray polarization

Metal-insulator transition: origin of phase transitions



BiS_2-based superconductors: toward high performance



Observation of electronic states:
- ✓ Bi 6d + 6s DOS
- ✓ Different profile dependent on the properties

Electronic states due to phase transition:
- ✓ Metal-insulator (MI) transition
- ✓ Cu valence & bonding states

H. Sato, *et al.*, Phys. Rev. B **106**, 155151 (2022).

A. Yamasaki, *et al.*, Phys. Rev. B **109**, 045131 (2024).

Summary of BL39XU upgrade

X-ray absorption and emission spectroscopy

Experimental stations

- ✓ EH1: X-ray spectroscopy under multiple-extreme conditions
- ✓ EH2: X-ray emission spectroscopy (New hutch)
- ✓ EH3: X-ray nano-spectroscopy

Beamline optics

- ✓ Upgrade optics:
  - ✓ Coaxial higher-harmonics cut mirrors
  - ✓ Double X-ray phase retarder: various polarization
- ✓ Differential exhaust: Window-less transport channel

Focusing optics

- ✓ KB mirror @ EH1 → 1 (V) × 10 (H) μm, 10^{13} ph/s @ 30 keV
- ✓ Wolter mirror @ EH2 → 1 (V) × 15 (H) μm, 10^{13} ph/s @ 20 keV
- ✓ KB mirror @ EH3 → 75 (V) × 107 (H) nm, 10^{11} ph/s @ 12 keV

Public use

- ✓ Commissioning of beamline optics: 01-04/2024
- ✓ Commissioning of experimental station: 05-06/2024
- ✓ **Public use: 07/2024~**