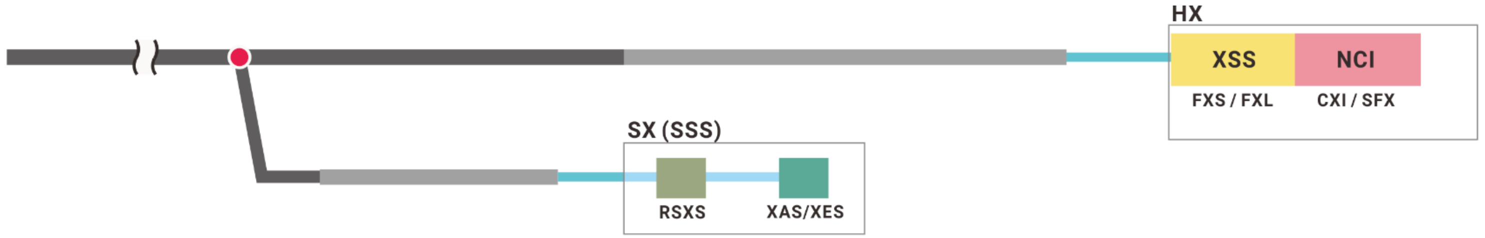
PAL-XFEL beamline status and plans

Intae Eom

November 14, 2023

POHANG ACCELERATOR LABORATORY



PAL-XFEL beamline instruments

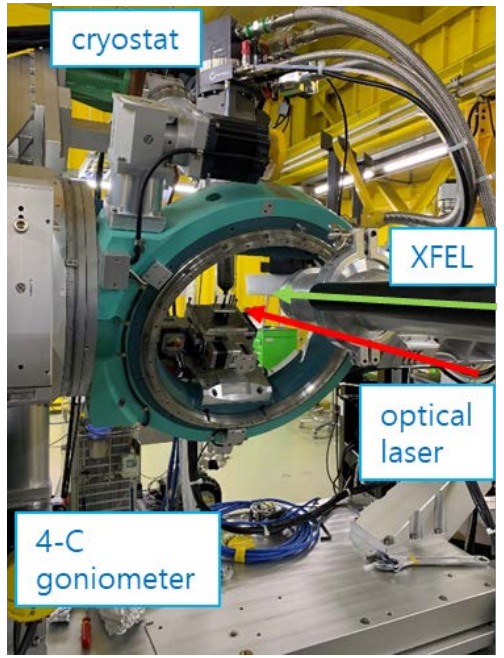
XSS (X-ray Scattering and Spectroscopy)

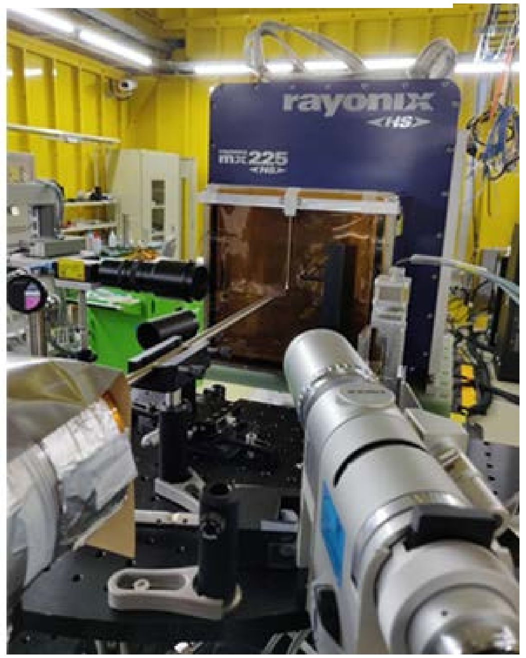
- Instrumentations

  Femtosecond X-ray Scattering (FXS)
  Femtosecond X-ray Liquidography (FXL)
  X-ray emission spectroscopies (XES)

- Specifications

  Focusing optics: Be CRL
  2-circle and 4-circle diffractometers
  Cryostream cryostat: 40 – 300 K
  Sample chamber for vacuum and gas conditions
  Liquid injector (100 um jet)





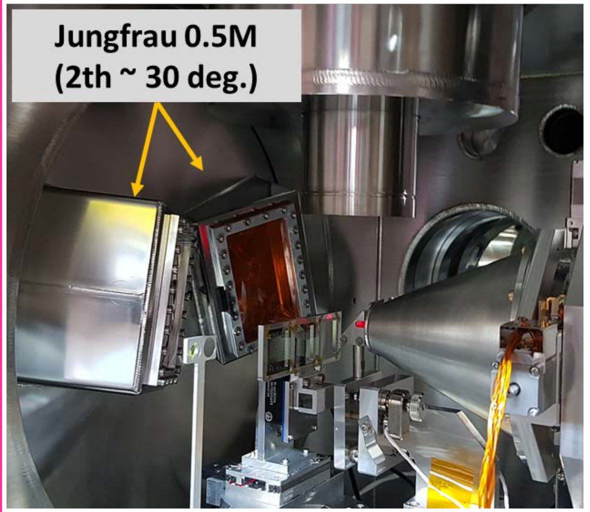
NCI (Nano Crystallography and coherent Imaging)

- Instrumentations

  Coherent X-ray Imaging / Scattering (CXI)
  X-ray Absorption Near Edge Spectroscopy (XA
  Serial Femtosecond Crystallography (SFX)
  Wide angle X-ray scattering (WAXS)

- Specifications

  Focusing optics: KB mirrors
  Dedicated sample chambers for CXI/SFX/XANES with vacuum or He environment
  tunable nanosecond laser for SFX experiments



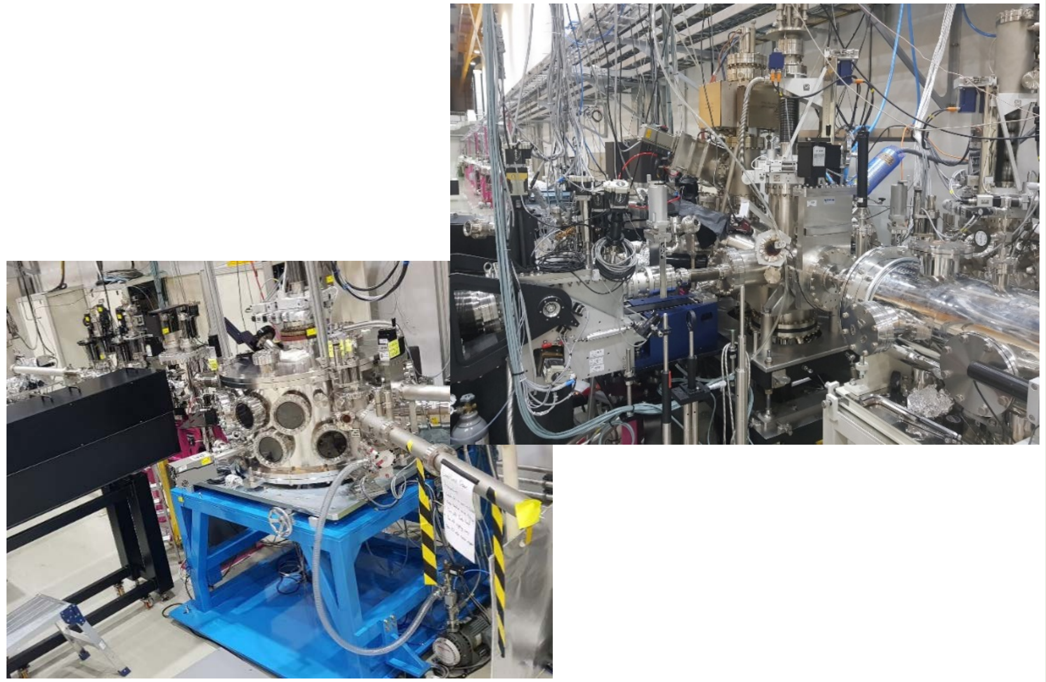
SSS (Soft X-ray Scattering and Spectroscopy)

- Instrumentations

  Resonant Soft X-ray Scattering (RSXS)
  X-ray Absorption/Emission Spectroscopy (XA
  Fourier Transform Holography (FTH)

- Specifications

  6-axis manipulator with cryostat (RSXS)
  VLS grating for 200 – 1200 eV (XAS/XES)
  Ion/electron time-of-flight (XAS/XES)



Research highlights in 2023

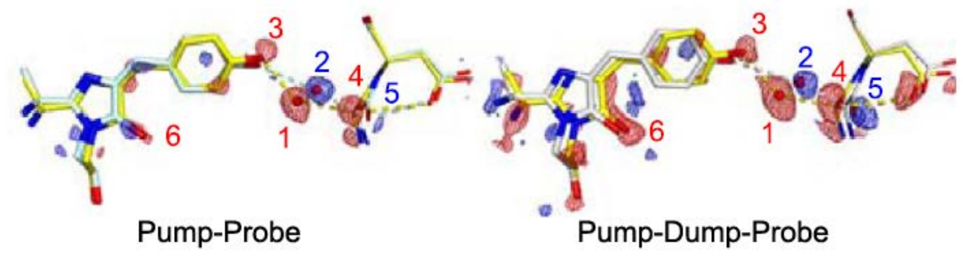
nature chemistry

HX-NCI

Article https://doi.org/10.1038/s41557-023-01275-1

Optical control of ultrafast structural dynamics in a fluorescent protein

C. D. M. Hutchison *et al.*, *Nat. Chem.* (2023)



- Coherent control of photo-isomerization of protein

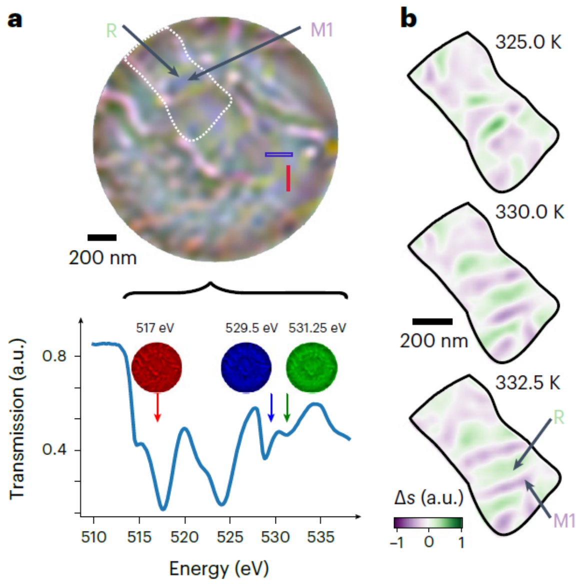
nature physics

SX-SSS

Article https://doi.org/10.1038/s41567-022-01848-w

Ultrafast X-ray imaging of the light-induced phase transition in VO_2

A. S. Johnson *et al.*, *Nat. Phys.* **19**, 215 (2023)



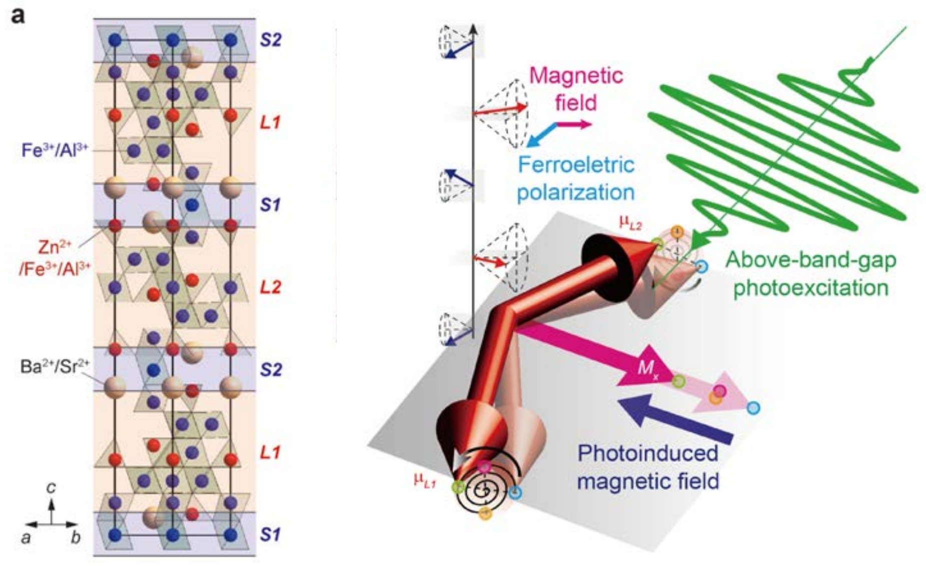
- Real space SX imaging of photo-induced phase transition in VO_2 nanodomains

RESEARCH ARTICLE ADVANCED MATERIALS www.advmat.de

SX-SSS

4D Visualization of a Nonthermal Coherent Magnon in a Laser Heated Lattice by an X-ray Free Electron Laser

H. Jang *et al.*, *Adv. Mater.* (2023)



- Visualization of photo-induced spin waves

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Research highlights in 2023

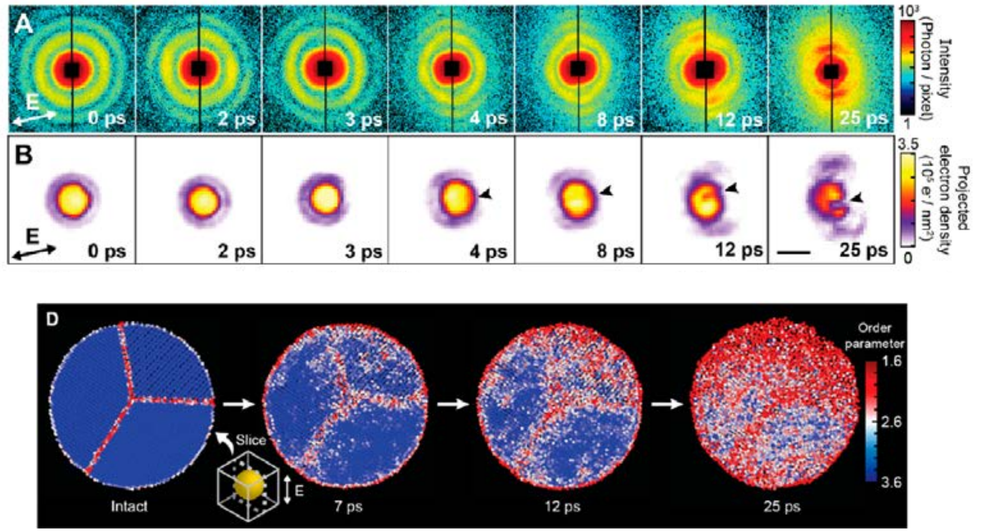
NANO LETTERS

HX-NCI

pubs.acs.org/NanoLett — Letter

Ultrafast Energy Transfer Process in Confined Gold Nanospheres Revealed by Femtosecond X-ray Imaging and Diffraction

J. Shin et al., *Nano Lett.*, **23**, 1481 (2023).



➤ The ultrafast energy transfer process in the confined nanoparticle system

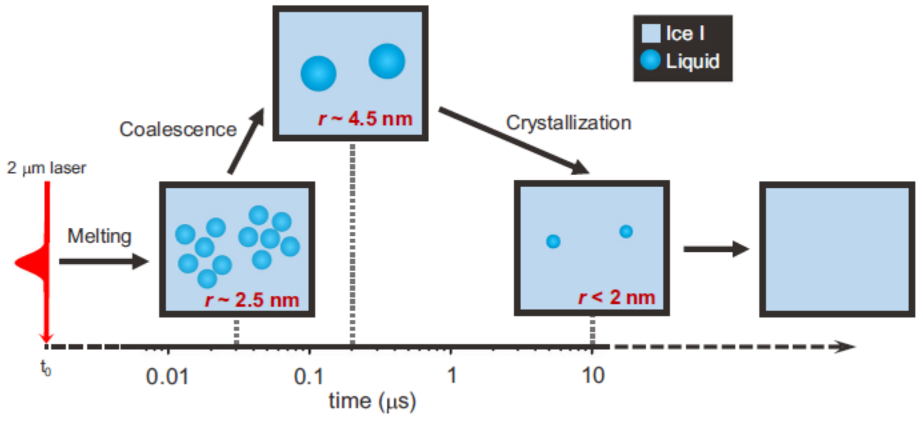
nature communications

HX-XSS

Article — https://doi.org/10.1038/s41467-023-38551-0

Melting domain size and recrystallization dynamics of ice revealed by time-resolved x-ray scattering

C. Yang et al., *Nat Comm*, **14**, 3313 (2023).



➤ Tracking liquid domains during water-ice phase transition

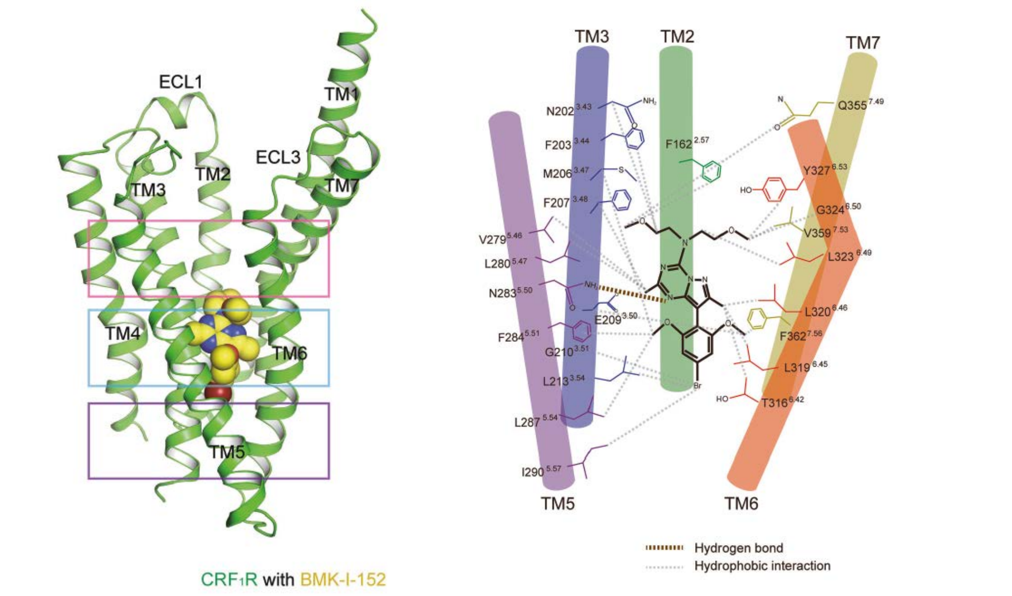
KSBMB — www.nature.com/emm

HX-NCI

ARTICLE OPEN — Check for updates

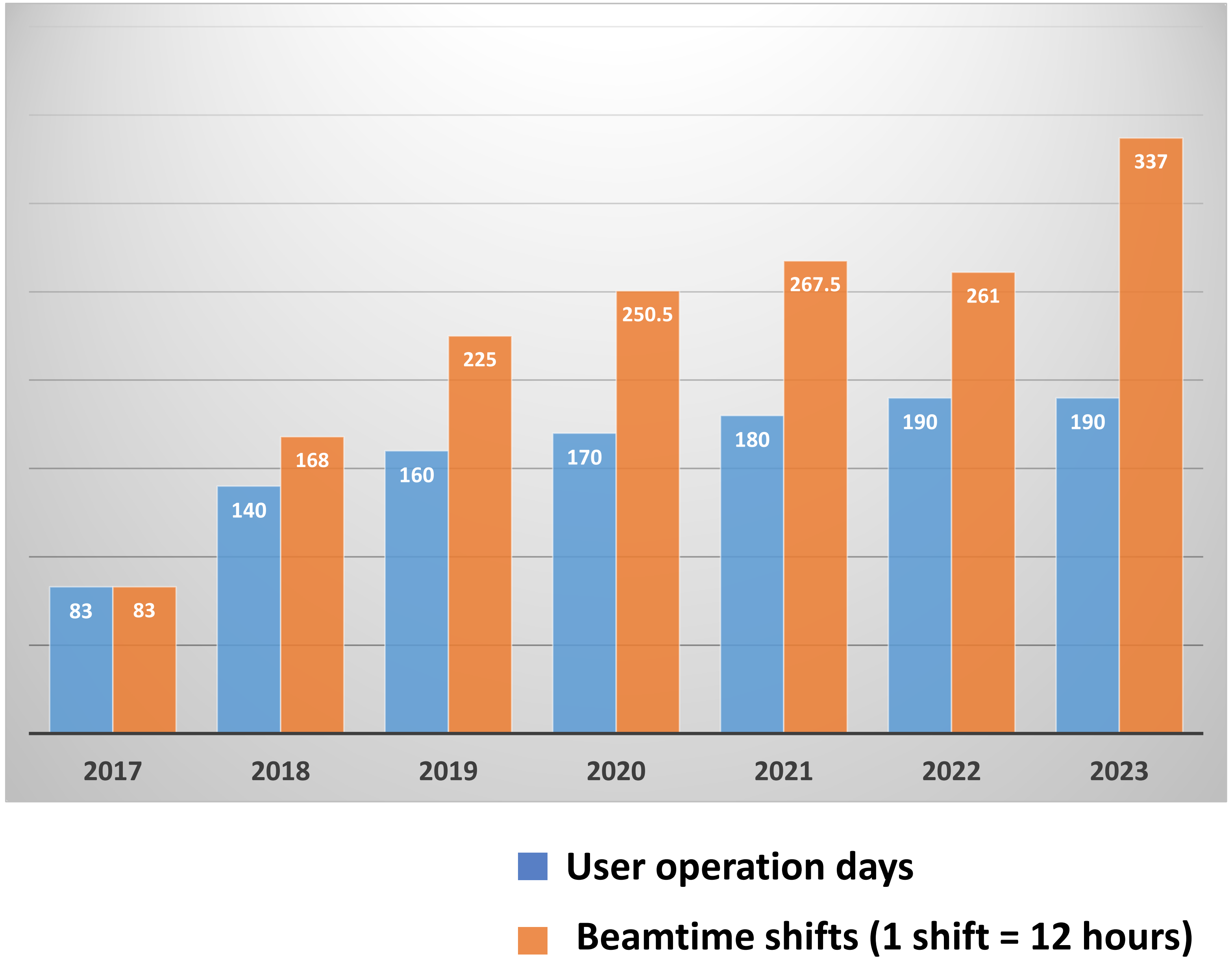
Structure-based drug discovery of a corticotropin-releasing hormone receptor 1 antagonist using an X-ray free-electron laser

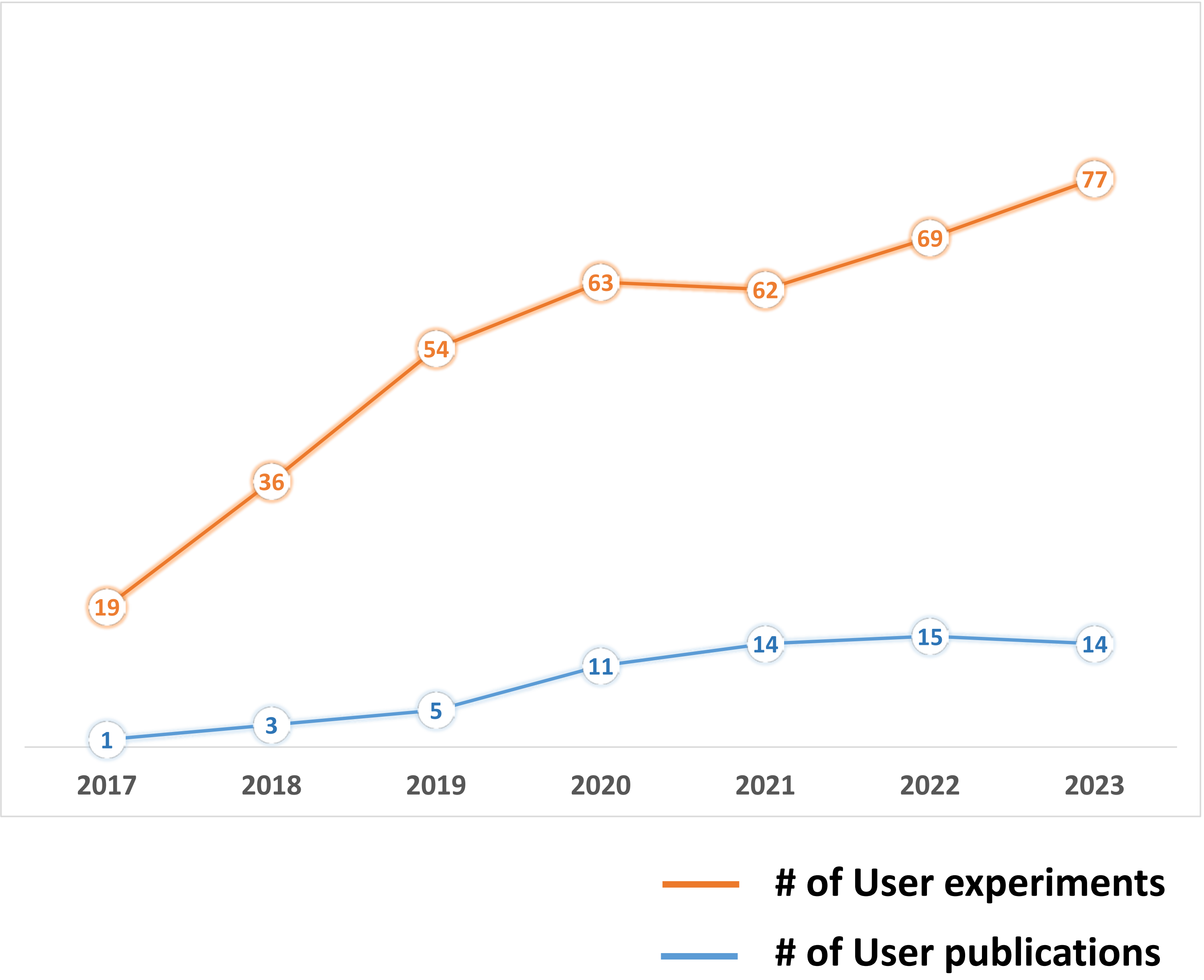
H. Kim et al., *Exp. Mol. Med.*, **55**, 2039 (2023).



➤ Role of hydrogen bond network in the active site of GPCR

PAL-XFEL user operation statistics

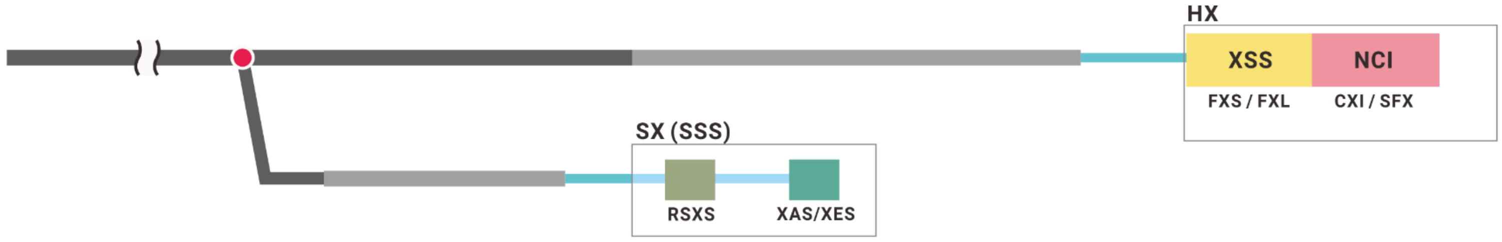




2023 PAL-XFEL beamline operation

Year	User operation	Regular beamtime	R&D beamtime
2023	190 days	140 days (Selected by KOSUA)	50 days (Selected by PAL)

Beamtime category	Provided beamtime shifts 2023-1st	2023-2nd
Regular beamtime (70 days per half year)	**HX 82 shifts** (24h support 12 days) **SX 36 shifts**	**HX 104 shifts** (24h support **34 days**) **SX 36 shifts**
R&D beamtime (1st half 23 days, 2nd half 27days)	**R&D: 12 days (12 shifts)** **Beamline M/S: 11 days (17 shifts)**	**Director's beamtime: 9 days (18 shifts)** **Potential users: 6 days (12 shifts)** **New instrumentation: 3 days (6 shifts)** **Screening beamtime: 2 days (4 shifts)** **Tutorial beamtime: 0.5 days (1 shift)** **Beamline M/S: 6.5 days (13 shifts)**



Science opportunities at PAL-XFEL

Research topics	HX: XSS	HX: NCI	SX: SSS
	Instrumentations		
Condensed matter physics and Material sciences Thermal / Non-thermal melting dynamics Phase transitions Phonon / Lattice dynamics Spin/Charge/Magnetic properties	FXS RIXS (RXES) Bragg-CDI	CXI WAXS	RSXS FTH
Chemical sciences Bond dissociation / Formation / Isomerization Charge transfer / Recombination / Localization Chemical reaction / Catalytic reaction	FXL	XANES XES	XAS/XES
Structural biology and Macromolecular dynamics Protein ternary structure / Active site of enzyme / Ligand binding site Photo-induced / Mixing-induced macromolecular dynamics Structures at a molecular level	.	SFX	.

Next science drivers for PAL-XFEL beamline sciences

1. Time-resolved XFEL experiments for condensed matter physics and material sciences

Quantum material research (FXS, RSXS) — Dr. Chun's presentation in PX6

: Finding and controlling hidden physics under specific sample environments

Multiplexing measurement (SAXS + WAXS + XES) — Dr. Nam's presentation in PX7

: Simultaneous measurements from single particle

Development of XFEL imaging techniques

: Dark / Bright field XFEL microscopy

Time-resolved CXI with nanofocusing — Dr. Nam's presentation in PX7

Fourier transform holography (SX)

2. Time-resolved XFEL experiments for chemical sciences

Implementing advanced experimental techniques (V2C, HERFD-XANES)

Multiplexing measurement (Scattering + XES)

: Precision measurements unveiling chemical dynamics

Various sample environments (Solid, Solution, Gas, Aerosol, …) — Dr. Park's presentation in PX8

3. Structural biology and Macromolecular dynamics

Initiation methods other than optical excitation

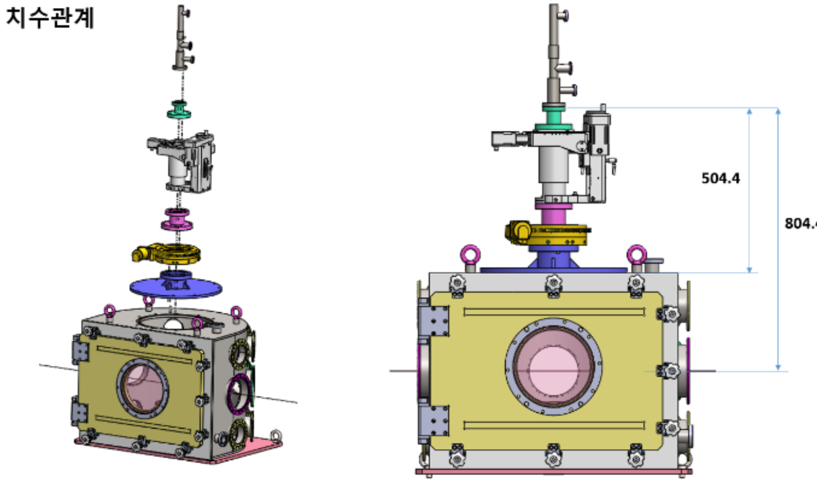
: Mixing and Injecting system

Sample environments for quantum material research at FXS, RSXS

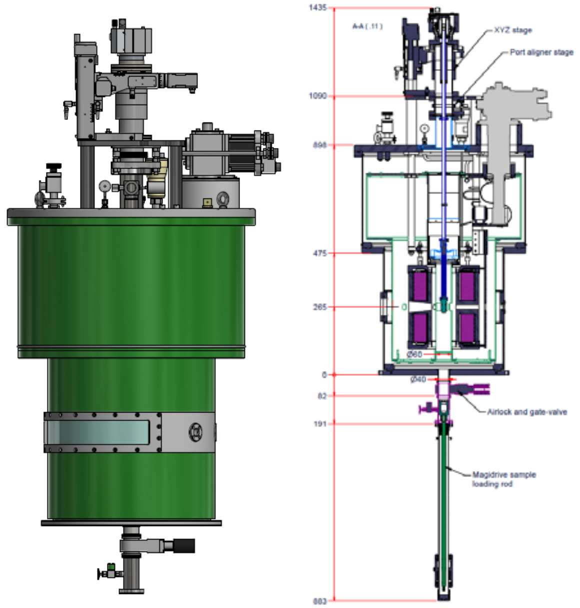
- **High T sample holder**

- **Cryostream with a goniometer (40 – 300 K)**

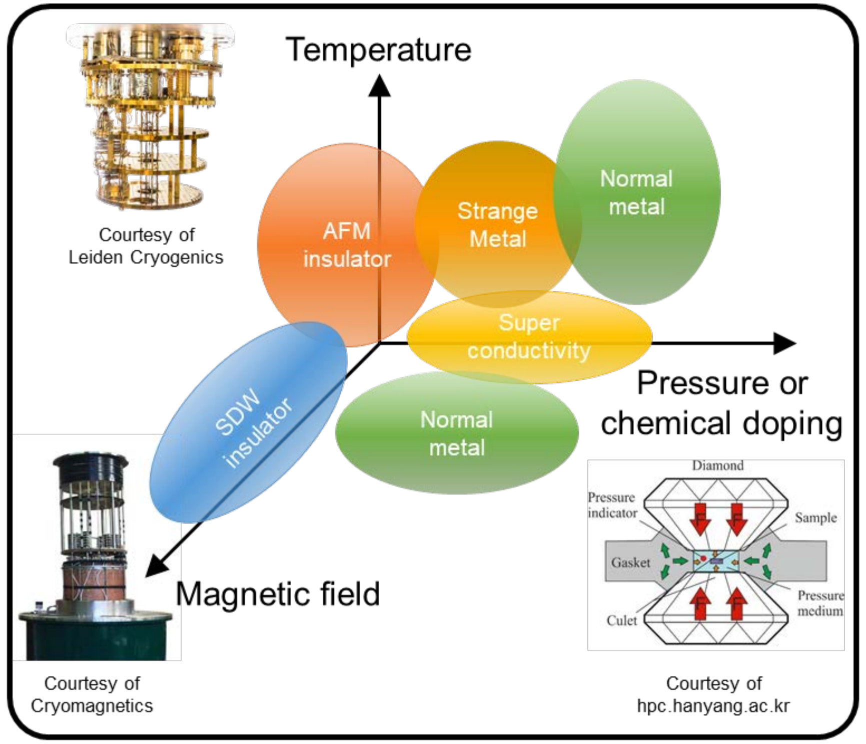
- **Low-temperature diffraction chamber for Tender X-rays in collaboration with Sogang Univ.**



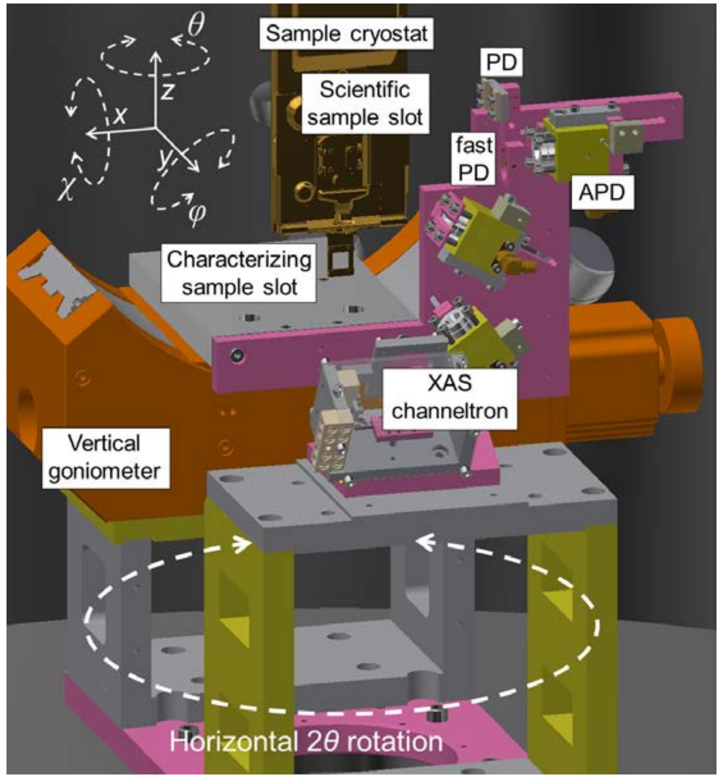
- **Low temperature/High Magnetic field (9 T, 4 K) in collaboration with PSC**



to be commissioned in 2023



- **RSXS sample environments**



- 6-axis cryostat manipulator
  - Base T_{sample} ~20 K (liquid helium)
- Avalanche photodiode detector 0-D point detector
- Horizontal linear polarized X-ray
- Horizontal sample (θ) and detector (2θ) rotation (π-polarization configuration)
- Plan: Controllable magnetic field
  2D detector (AXIS)
  Pump option (THz, Mid-IR)

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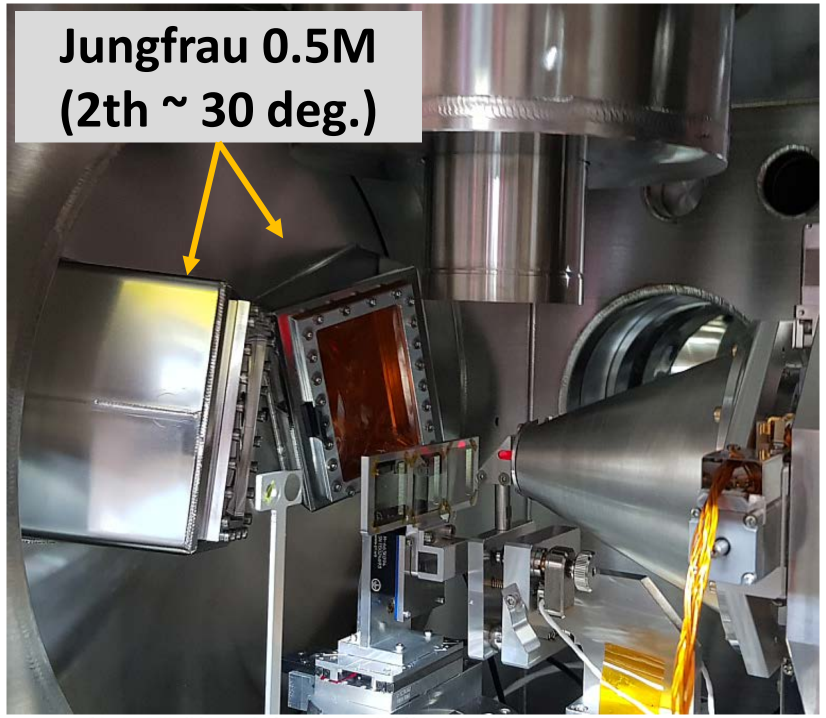
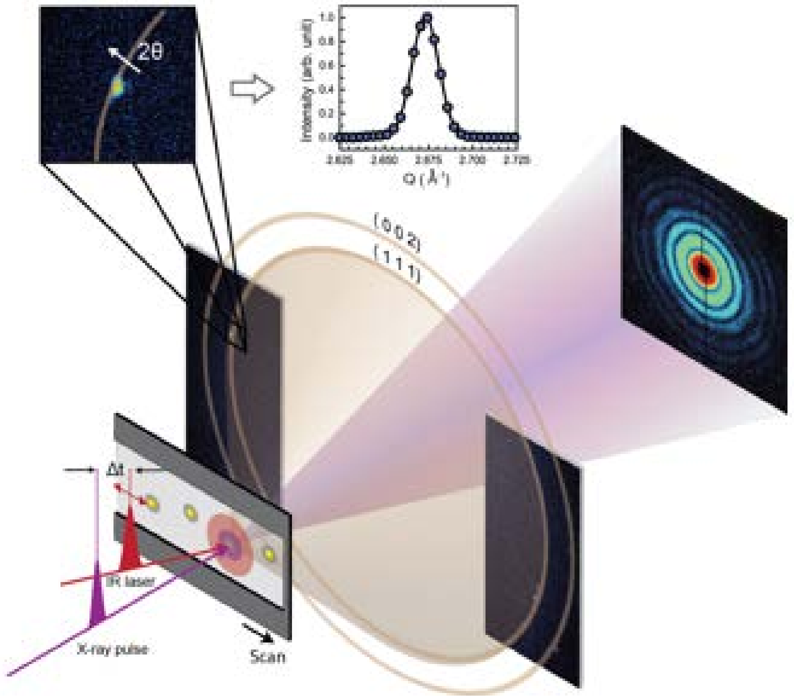
Multiplexing CXI experiment





❖ **Multi-probe by a single X-ray pulse**

· **SAXS**: **morphological information** at nanoscale resolution

- **JUNGFRAU 4M**

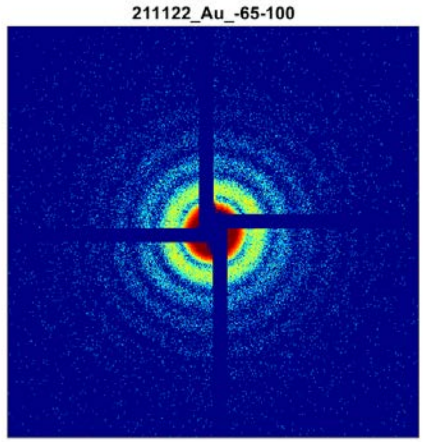
· **WAXS**: **atomic-scale structural information**

- Two 0.5M Jungfrau detectors
- Two-theta is fixed; 30±7°
- azimuth angle: ~38 % covered by two detectors
- **JUNGFRAU 5M with center hole**

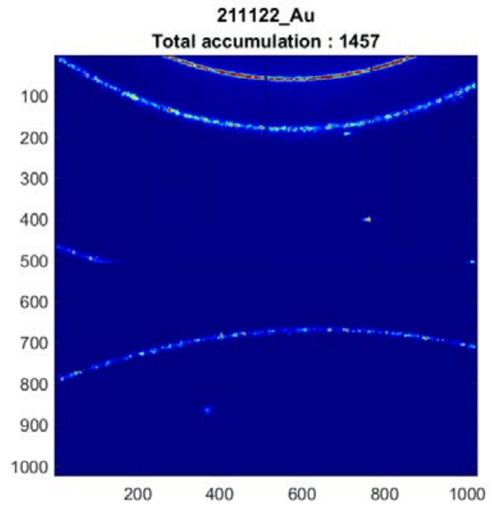
· **XES**: **electronic structure**

- Von Hamos spectrometer : Si(111) and Si(220)
- 0.5M Jungfrau detector
- acceptable 2θ range: 45 ~ 61°
- **JUNGFRAU 0.5M**

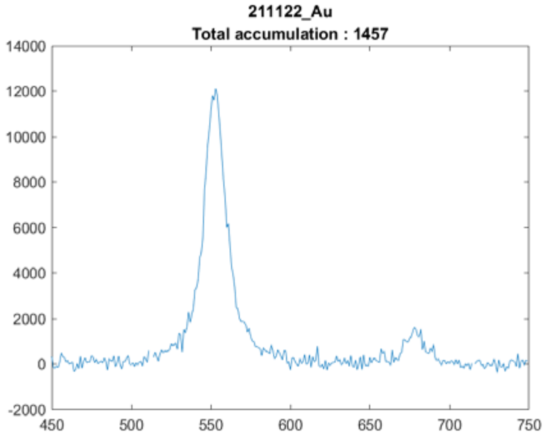
SAXS



WAXS



XES



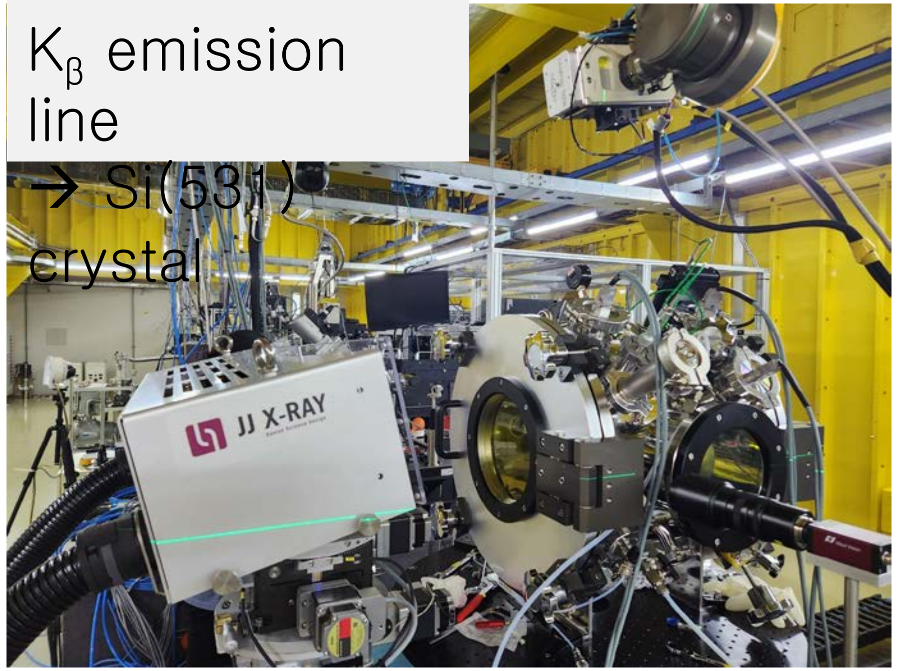
J. Shin et al., Nano Lett., **23**, 1481 (2023).

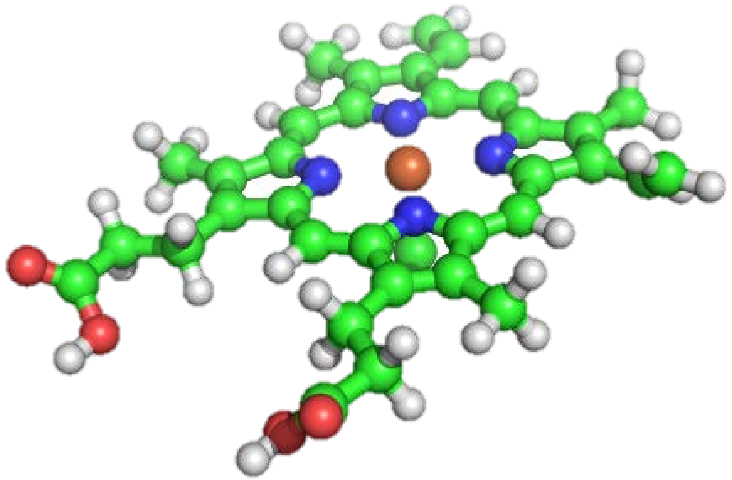
fs X-ray emission spectroscopy to reveal a hidden spin state

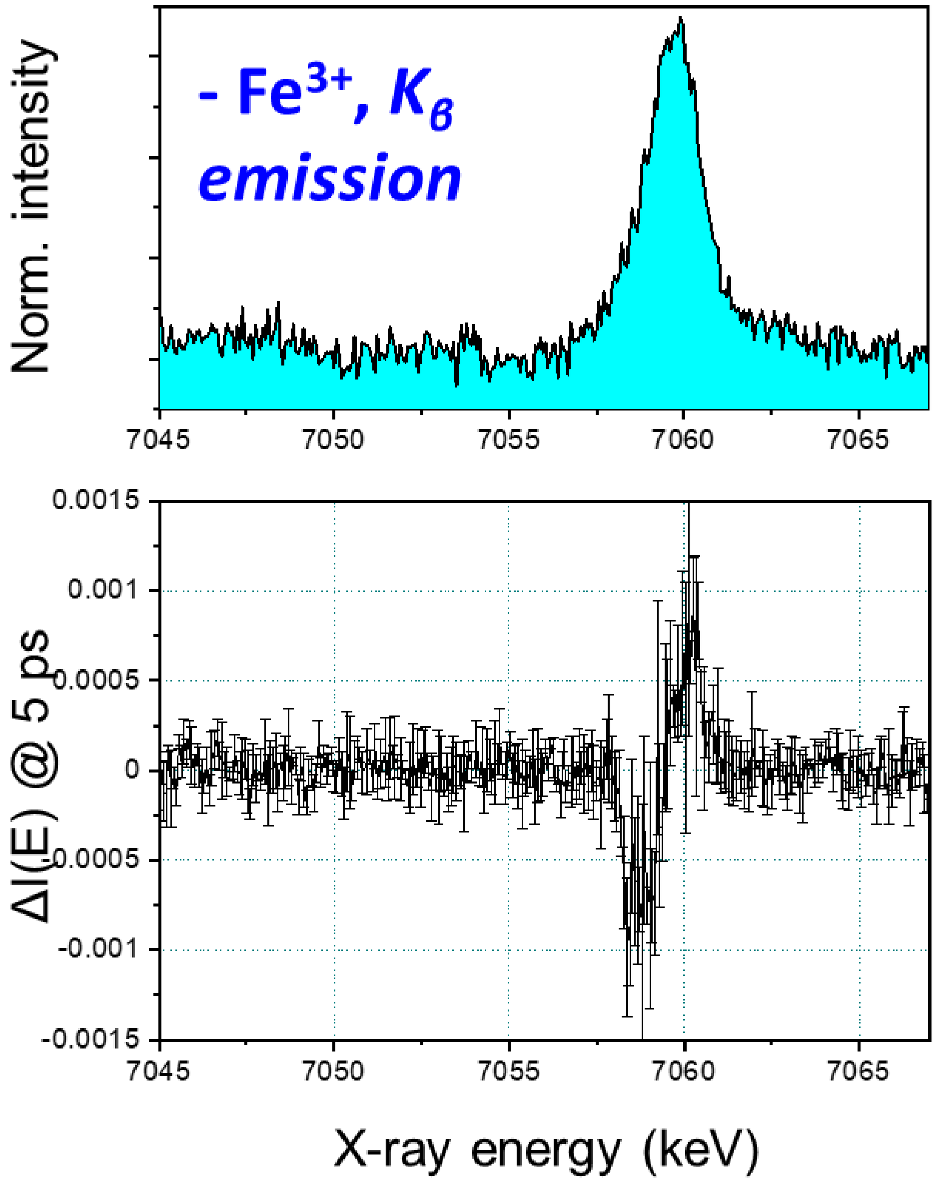
Courtesy of Prof. T. W. Kim at Mokpo univ.

Setup in XSS hutch (side view)

K_β emission line → Si(531) crystal



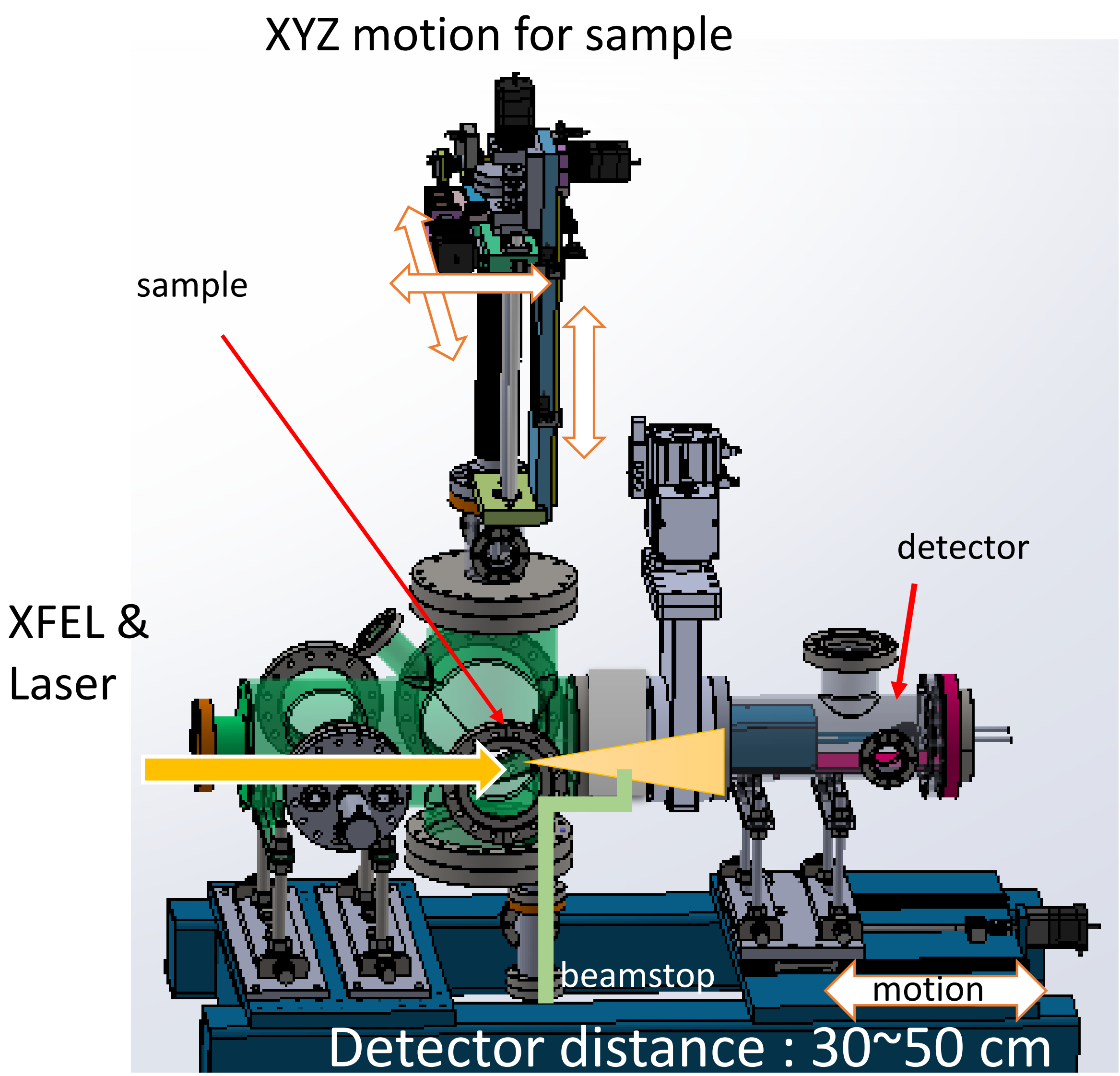


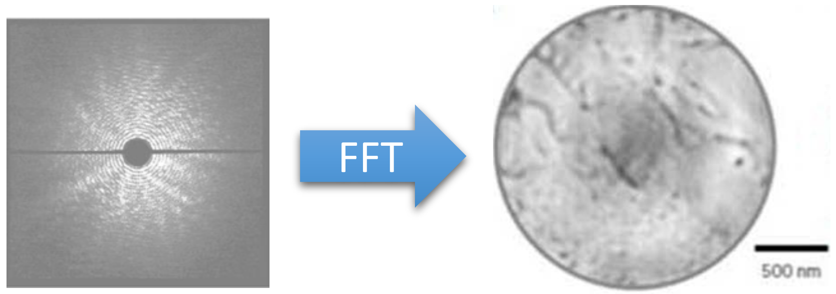


	Strong	Weak
XAS	Sensitive to **redox state** Easy to measure	Hard to identify spin state
XES	Sensitive to **spin state**	Hard to measure transient signal

Fourier transform holography (SX)

Dr. Sanghan Park



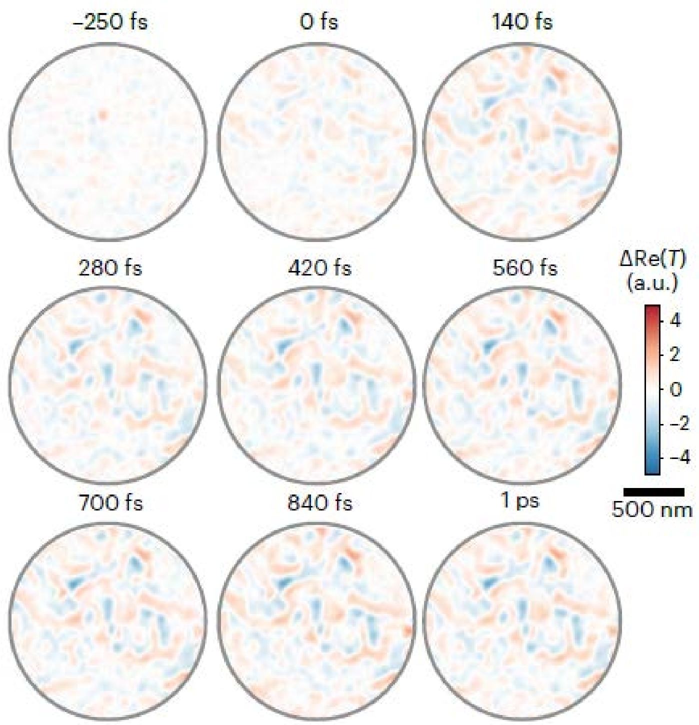


Time-resolved soft X-ray image (Spectroscopic imaging)

Sample temperature: RT~150 °C

Spatial resolution: <100 nm
Temporal resolution: ~150 fs

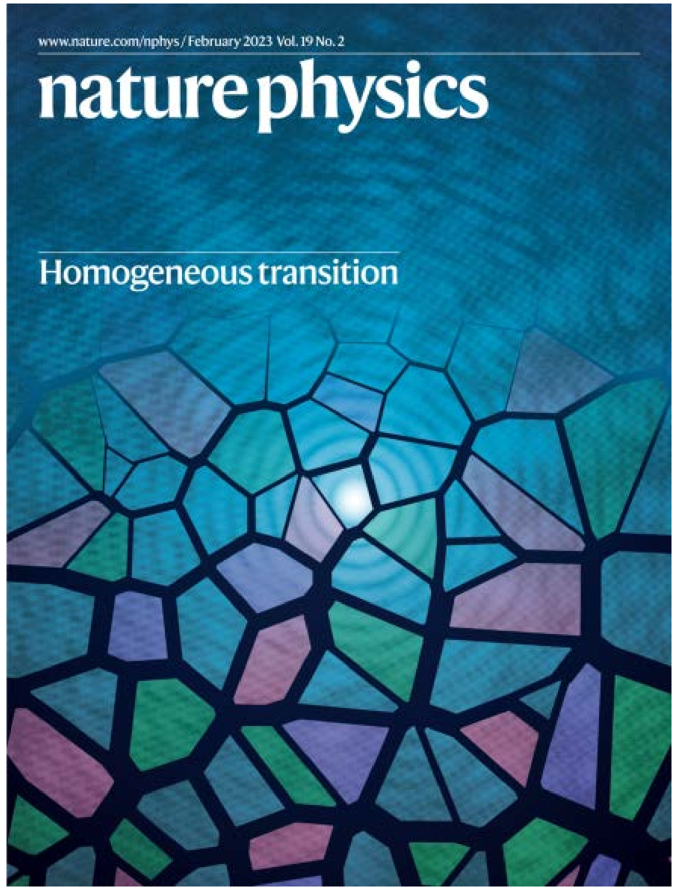
Electromagnet and low-temperature sample stage will be introduced later.



nature physics

Article https://doi.org/10.1038/s41567-022-01848-w

Ultrafast X-ray imaging of the light-induced phase transition in VO_2



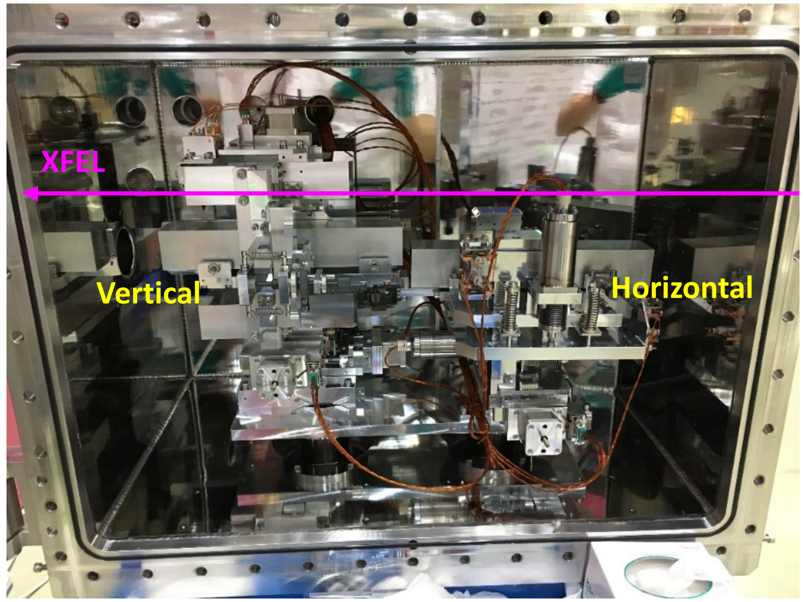
Light-induced insulator-to-metal phase transition dynamics in VO_2 by FTH

Johnson et al., Nature Physics **19**, 215 (2023)

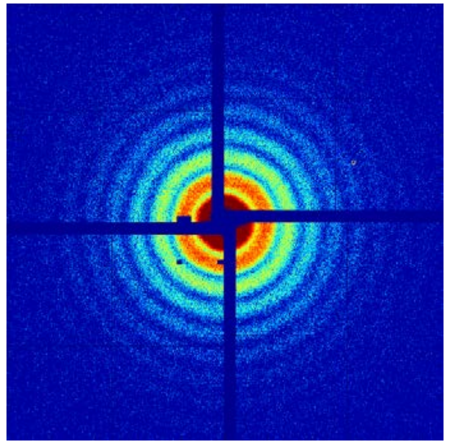
X-ray optics update

Nano-focused KB mirror (HX/SX)

Dr. Daewoong Nam



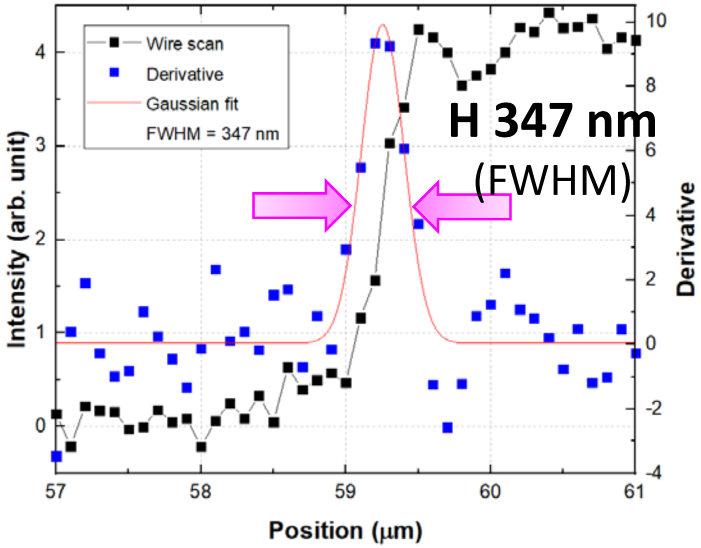
Diffraction image from 50 nm Gold nanoparticle

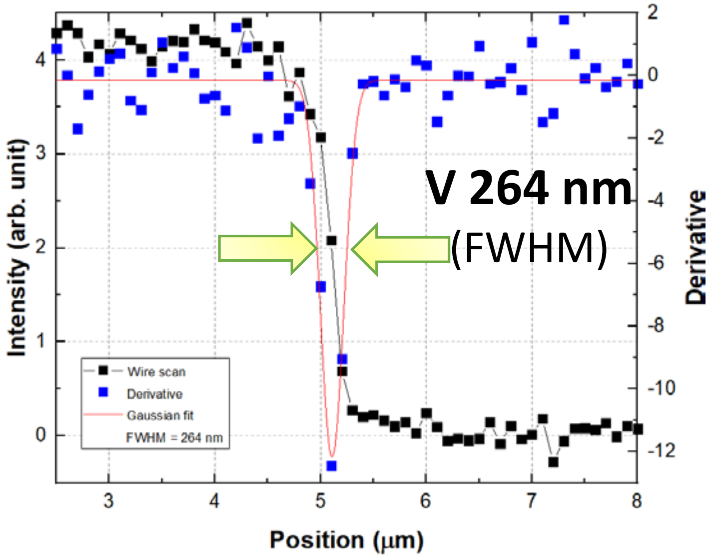


- X-ray: 5 keV
- Focus size: 500 nm (H) x 650 nm (V)

- Coating: Ir (50 nm), non-coated (Quartz)
- Focal length: 910mm (H), 600 mm (V)
- Working distance: 450 mm

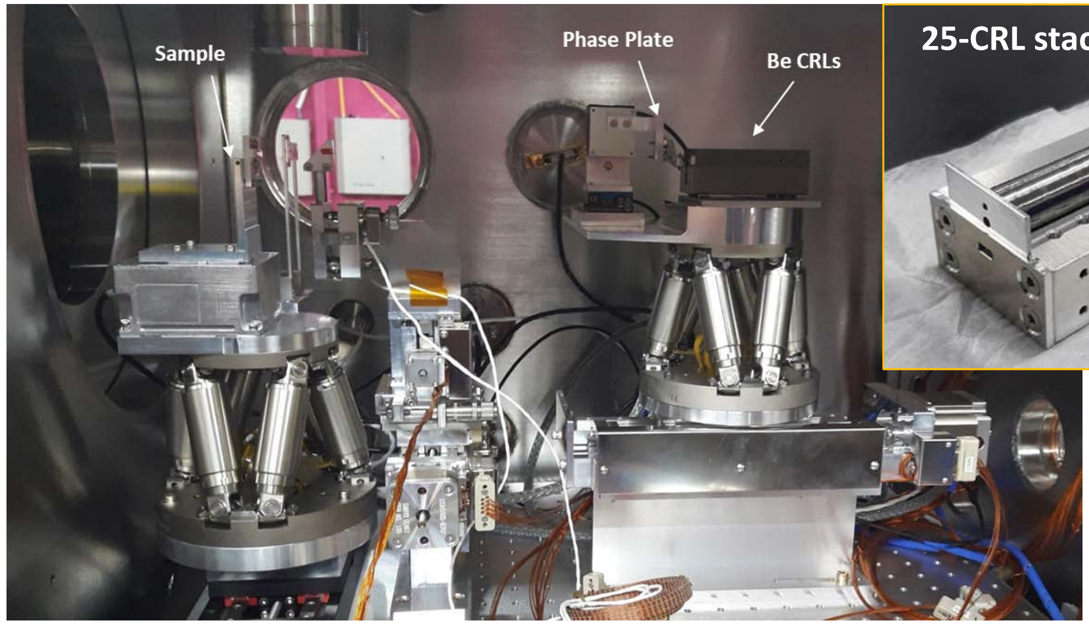
Focused beam profiles @ 9.5keV

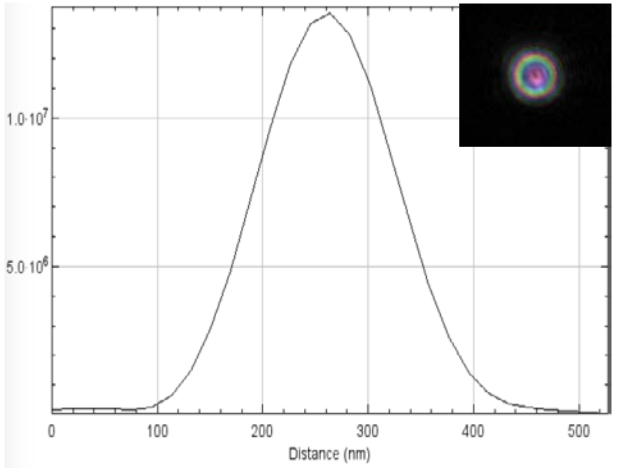


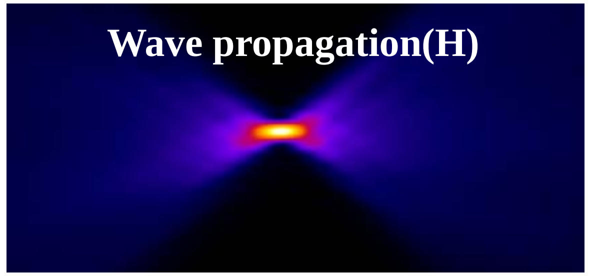


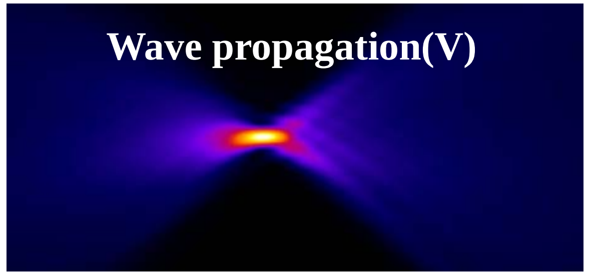
Nano-focused CRL (portable)

Dr. Sangsoo Kim





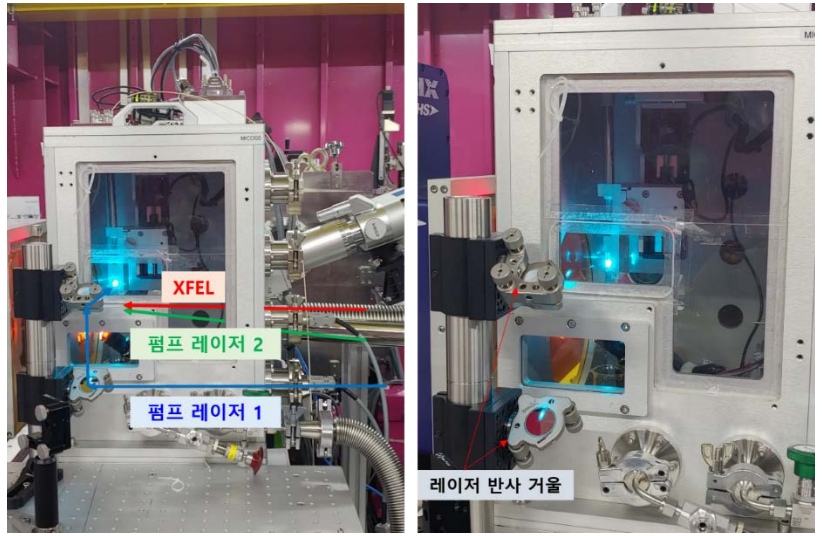


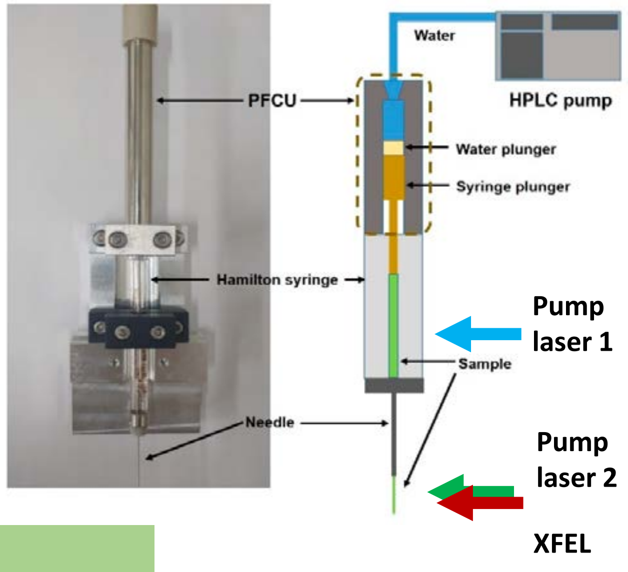


Energy	Focal length	Spot Size	Effective Aperture	Transmission
10.0 keV	300 mm	160~180 nm	258 μm	0.5

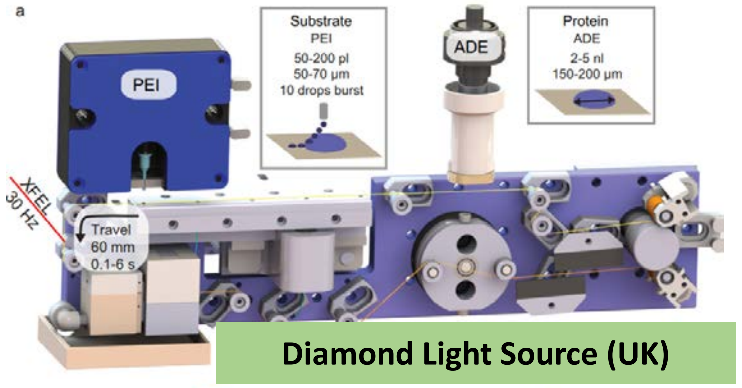
Sample deliveries for Macromolecular dynamics

Dr. Jaehyun Park

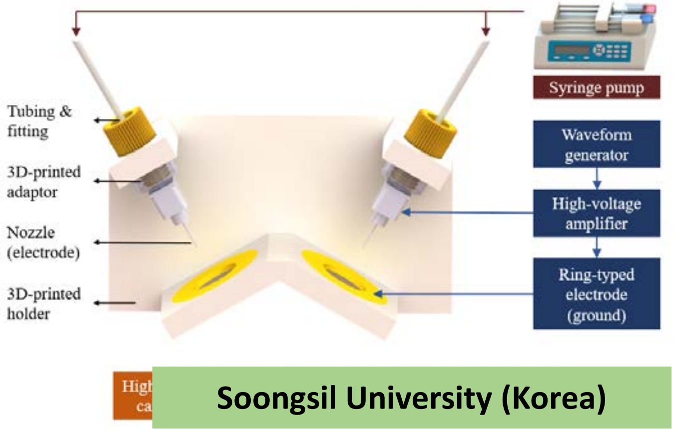




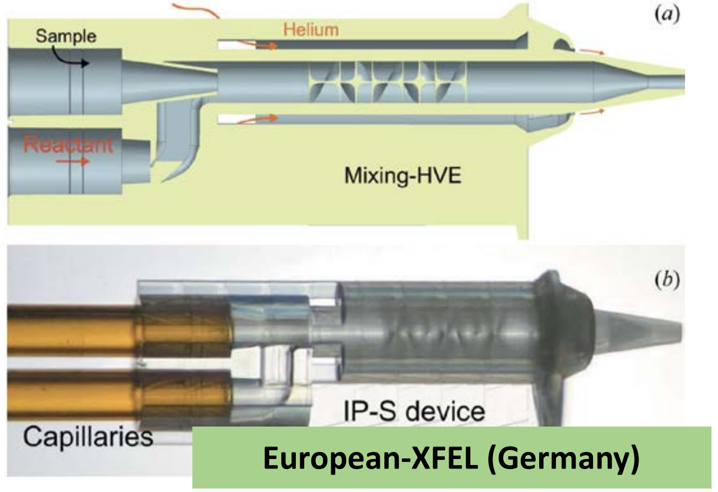
J. van Thor et al. Nature Chemistry (2023)



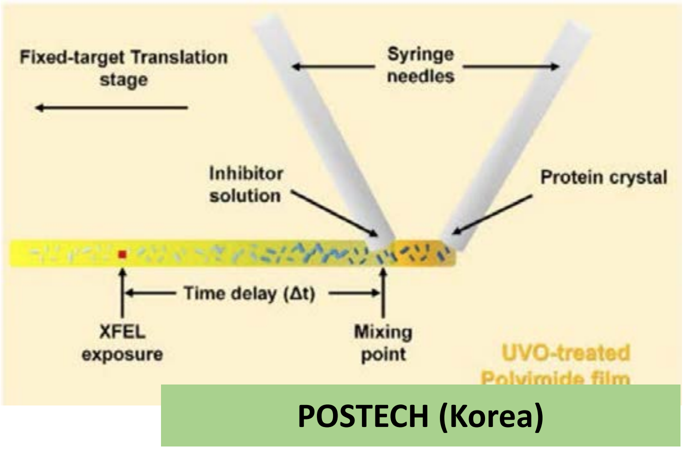
Diamond Light Source (UK)



Soongsil University (Korea)



European-XFEL (Germany)



POSTECH (Korea)

❖ Photo-induced dynamics

- Optical laser pump (& dump)
- Photo-induced structure changes
- Users from ASU, LBNL, Imperial College London, SwissFEL, etc.

❖ Molecular reaction dynamics

- Solution mixing and X-ray probe
- Ligand binding @ active sites
- Enzyme reactions
- Drug target
- Collaboration: Diamond Light Source, European-XFEL, etc.

Sample handling strategies for SFX

Liquid injection

- **Conventional techniques (phase 1)**
  - GDVN liquid jet injection (PSD)
  - LCP injection (CMD)
- **Miscellaneous**
  - Direct injection with Hamilton syringe (MLV syringe)

Fixed target

- **Stage based control (phase 2)**
  - 2D fixed target: micro-mesh
  - 1D fixed target: micro-tubing

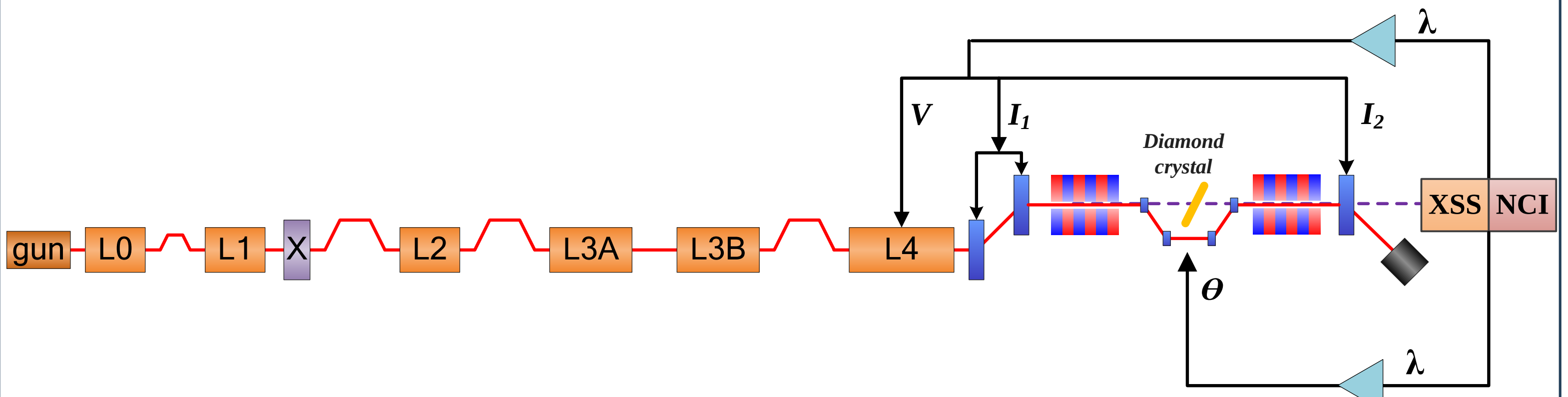
Mixing injection

- **Membrane proteins (LCP)**
  - Mixing-High viscosity extruder → Mixing-HVE injector
  - Collaboration with European-XFEL SEC group
- **Soluble proteins**
  - Aperture-based mixing device (Si-chip with multi windows)
  - Under development

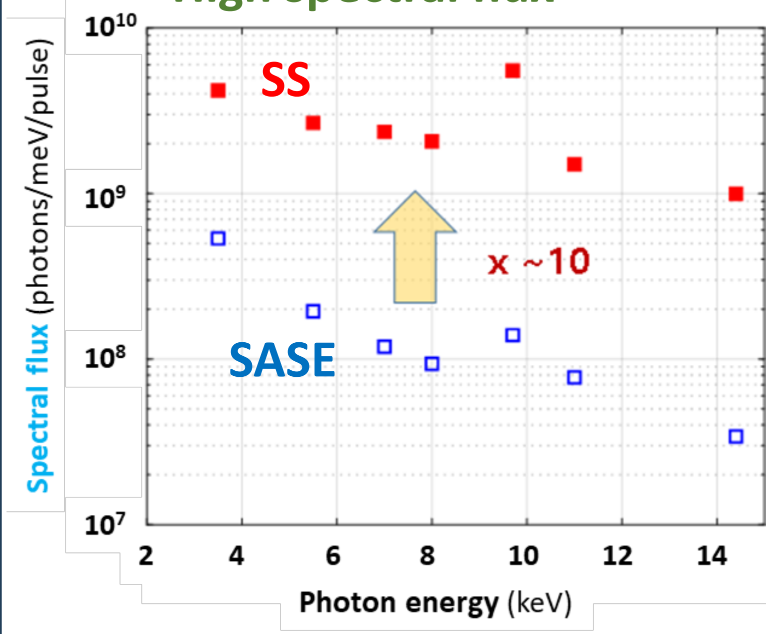
Experimental applications of advanced FEL modes at PAL-XFEL

Experimental applications of advanced FEL modes at PAL-XFEL

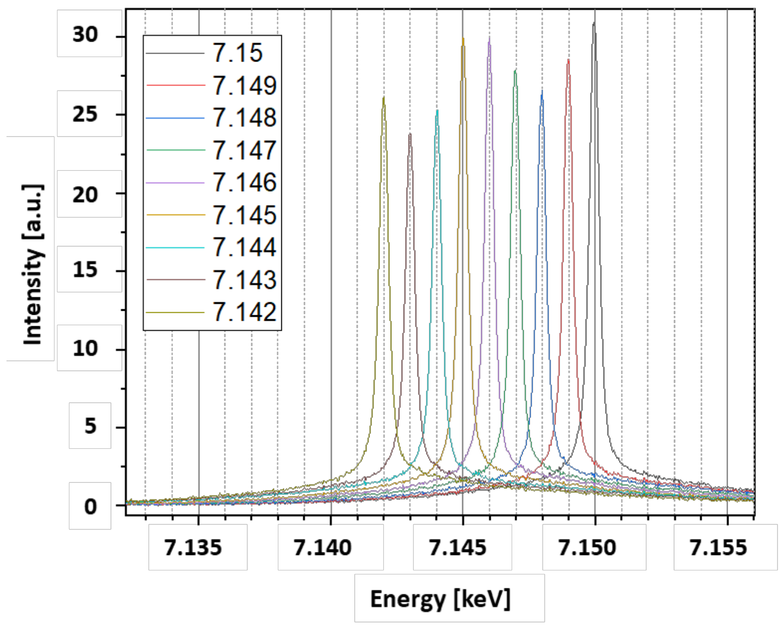
Hard X-ray self-seeding



High spectral flux

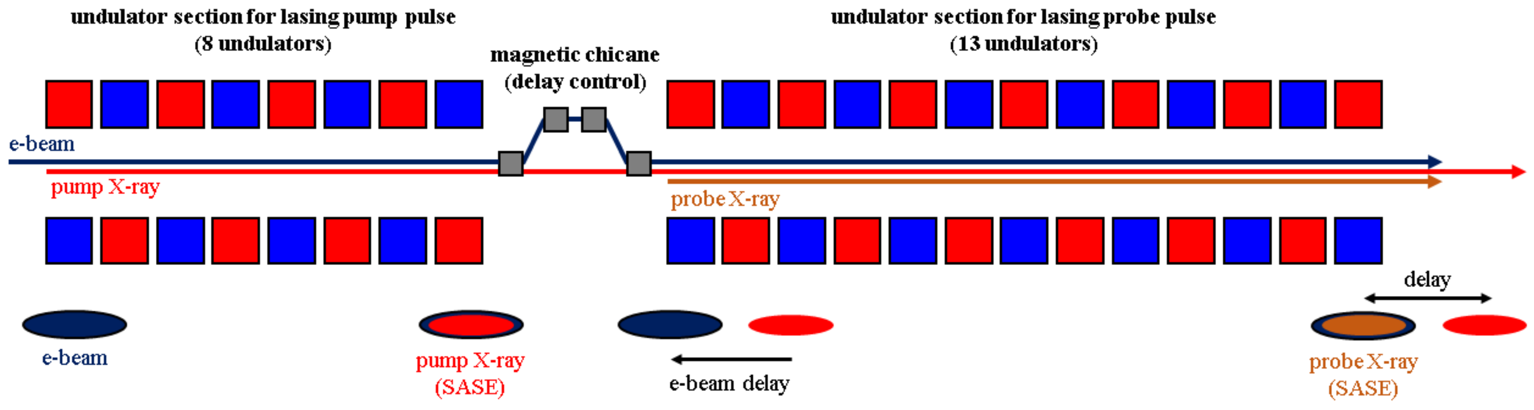


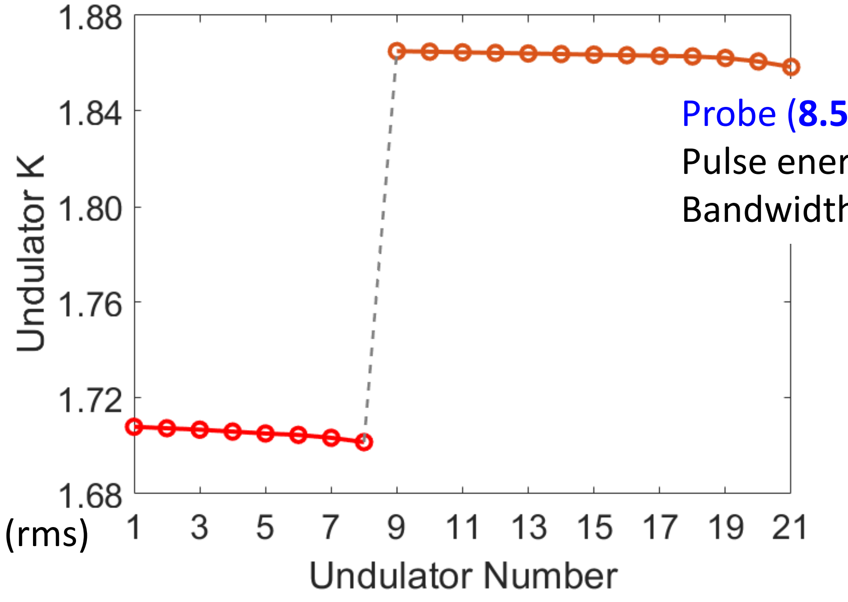
E-scan capability



- SASE bandwidth (FWHM) = 27 eV
- Self-seeding bandwidth (FWHM) = **0.22 eV**
- Averaged pulse energy: **~850 μJ**
- FEL Pulse duration = ~ 20 fs

Two-color FEL generation





Probe (8.5 keV)
Pulse energy : **~257 μJ**
Bandwidth : ~9.5 eV (rms)

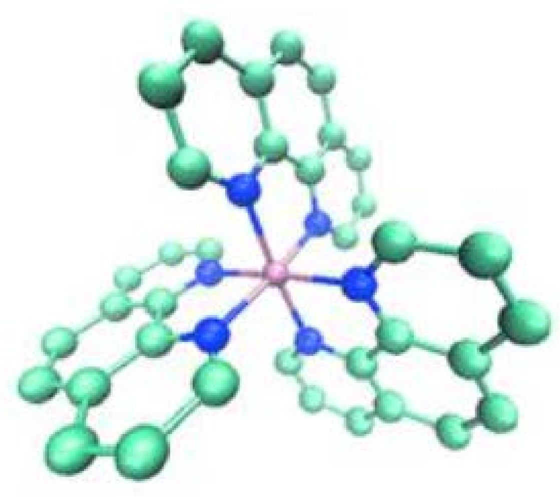
Pump (9.5 keV)
Pulse energy : **~224 μJ**
Bandwidth : ~10.9 eV (rms)

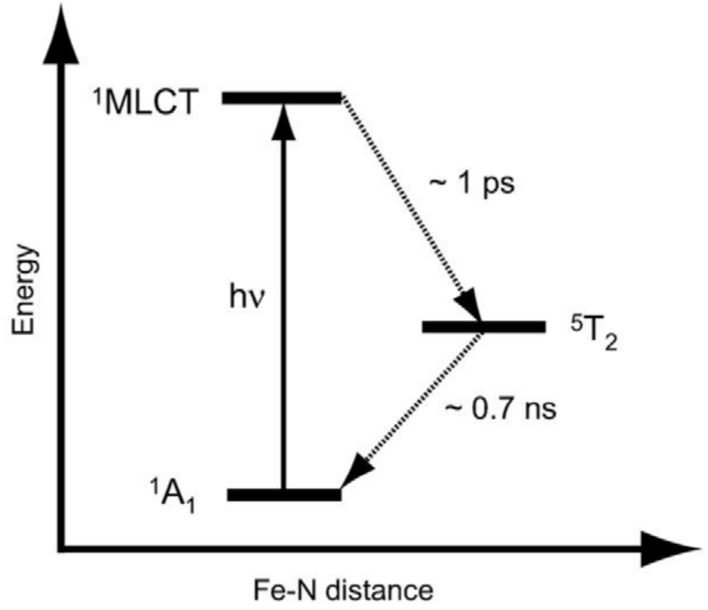
- **9.5 keV pump + 8.5 keV probe**
- **Delay between two pulses up to 120 fs**

Courtesy of Dr. J.H. Lee & R. Ma at PAL-XFEL

Time-resolved XANES using Self-seeded FEL

Spin-crossover complex: photo-activated spin state conversion





Experimental condition

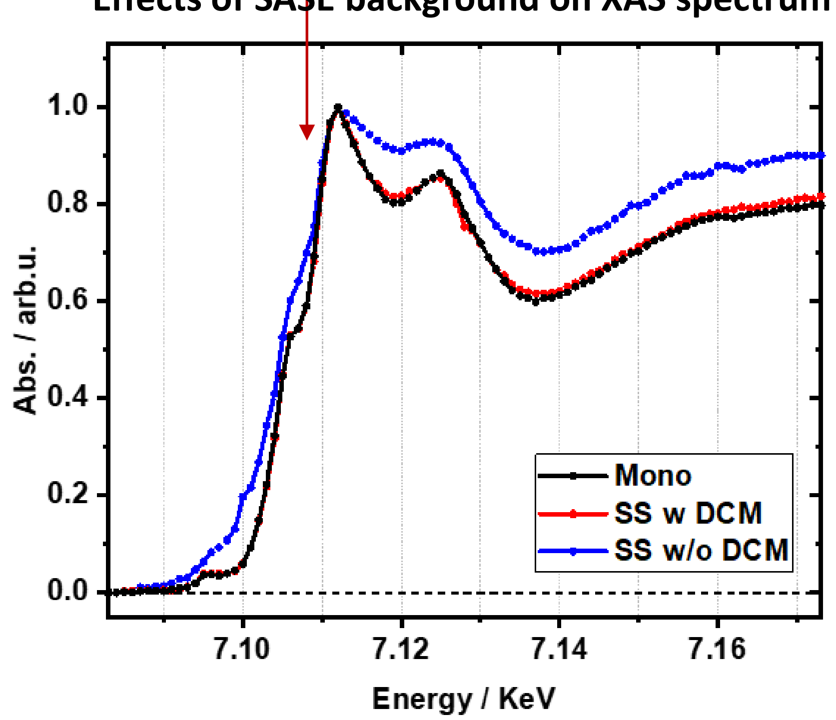
- Sample : 50 mM $Fe(phen)_3Cl_2$ in water (150 um nozzle)
- pump laser : 400 nm (100 mJ/cm^2)
- X-ray : 7.09~7.17 keV (E-scan) , ~39 μm^2 (FWHM)

SS beam (w/ DCM) shows 25 % better S/N than SASE.

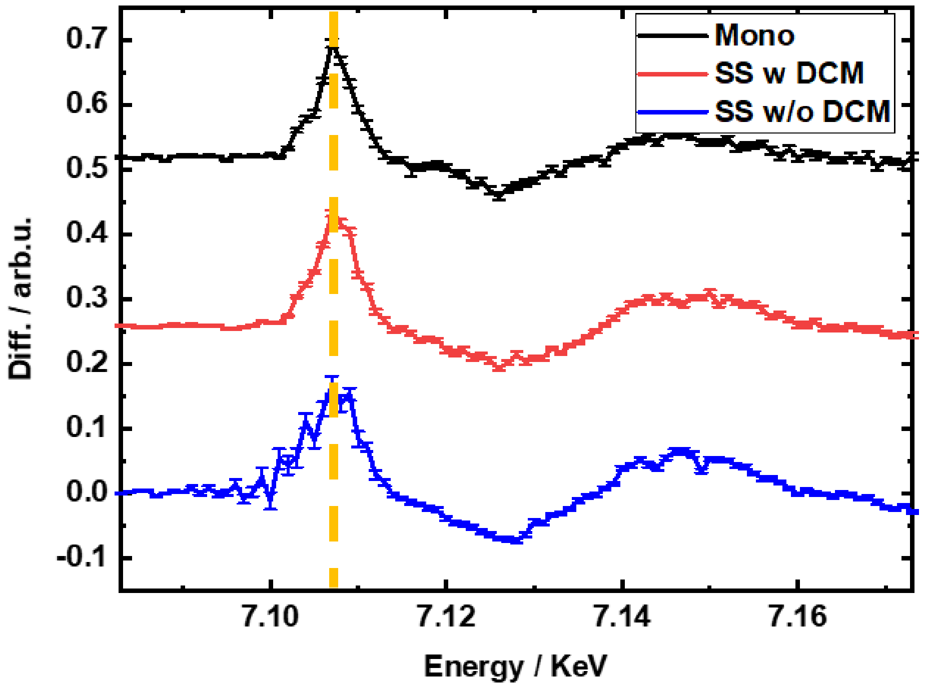
Ready to support tr-XAS experiments using SS (>5 keV)

Future plan : HERFD-XANES

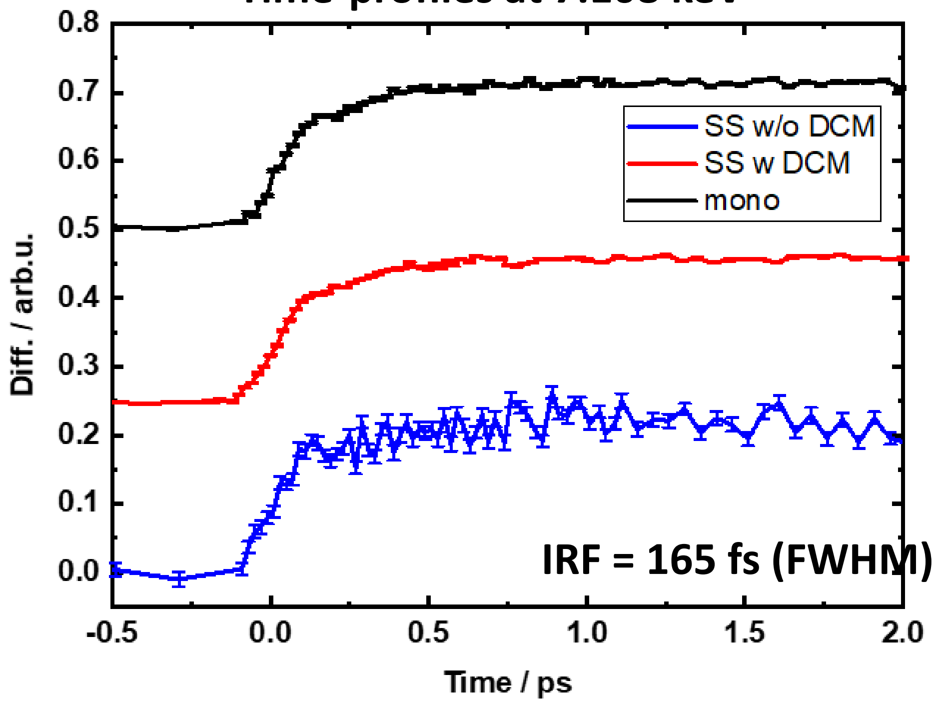
Effects of SASE background on XAS spectrum



Difference spectra at 50 ps



Time-profiles at 7.108 keV



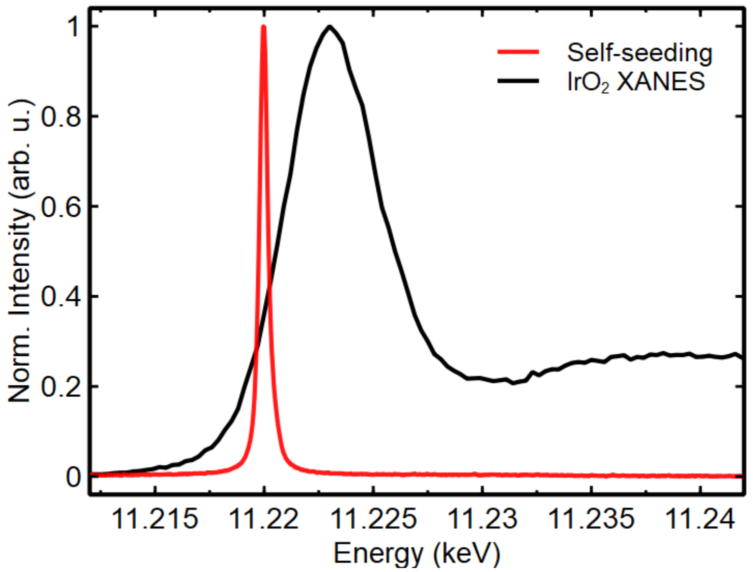
POHANG ACCELERATOR LABORATORY

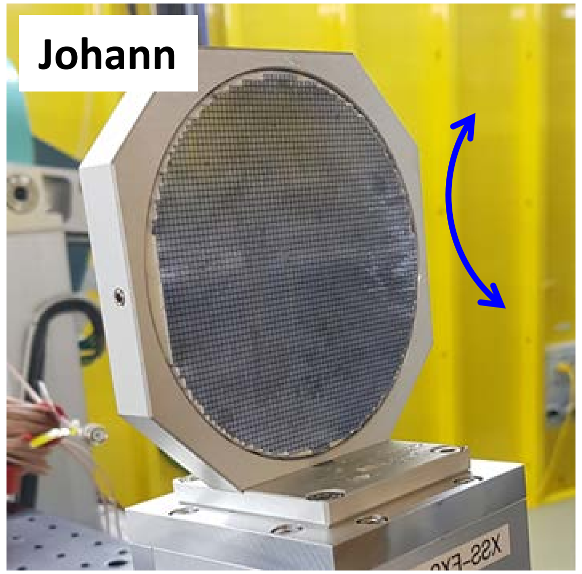
Courtesy of Dr. S.H. Chun & T.K. Choi at PAL-XFEL

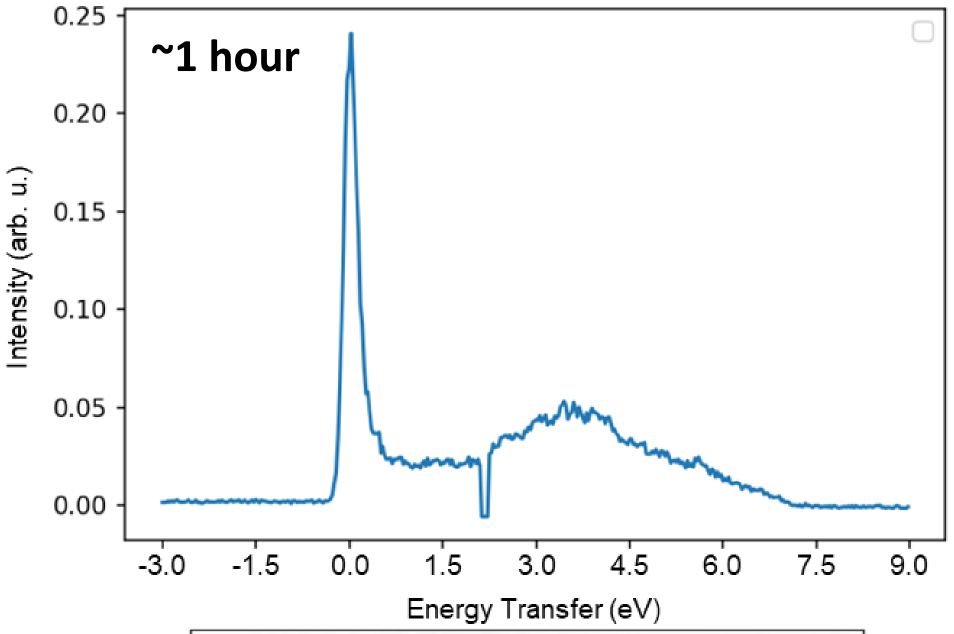
Hard X-ray RIXS, RXES using Self-seeded FEL

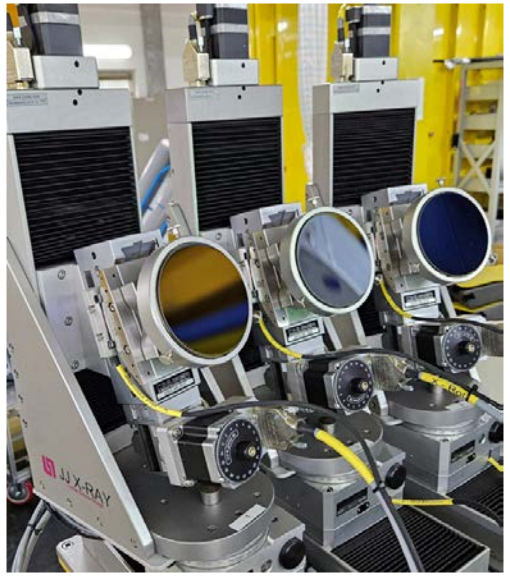
“Resonant X-ray emission spectroscopy using self-seeded hard X-ray pulses at PAL-XFEL”, *JSR 2023*

1) RIXS: Incident energy *fixed*, Bragg angle *scanned*



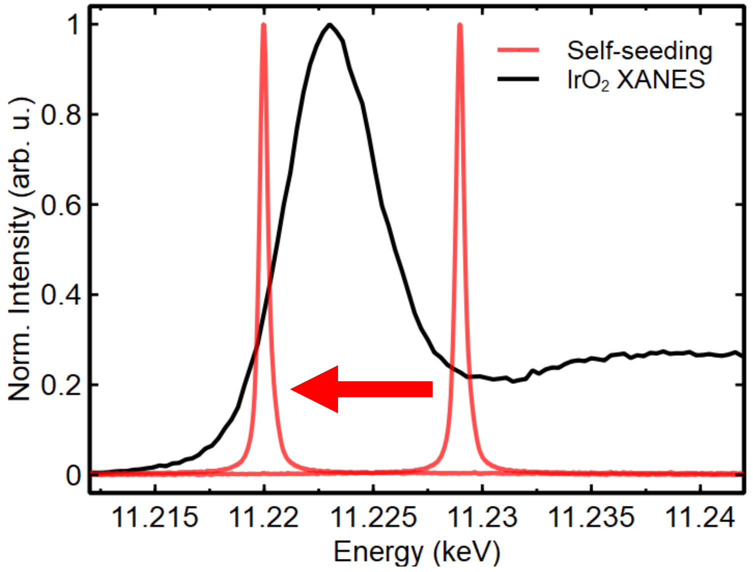


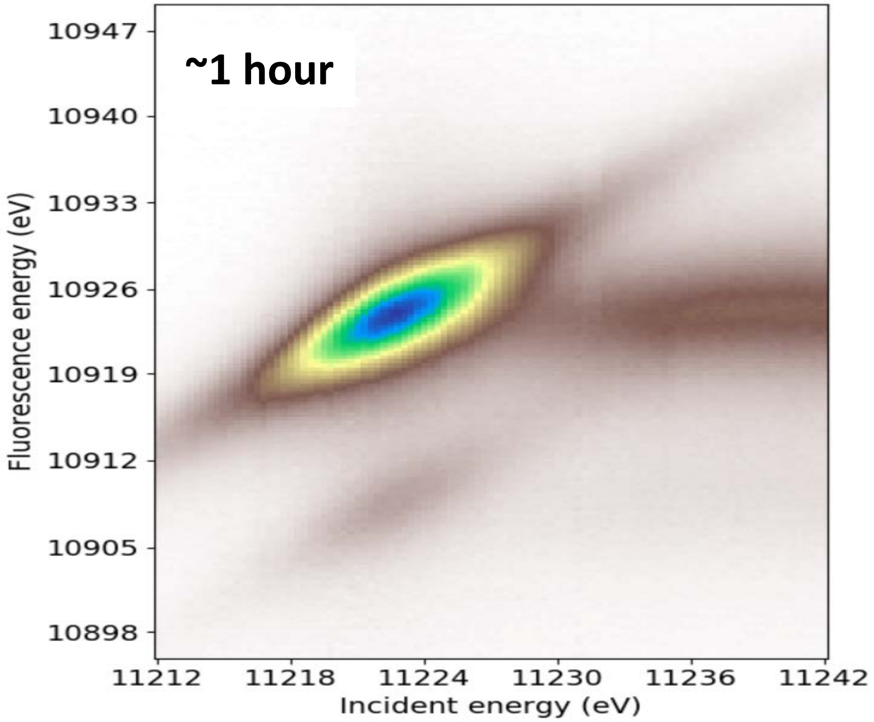


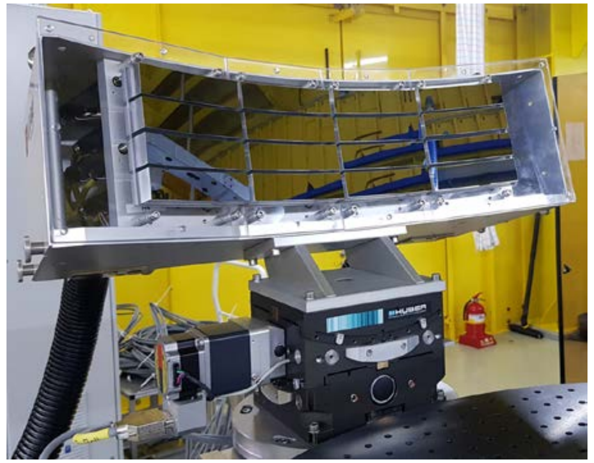


Johann-type (scanning)
- Crystal: 3 x Si(111), (220)

2) RXES: Incident energy *scanned*, Bragg angle *fixed*

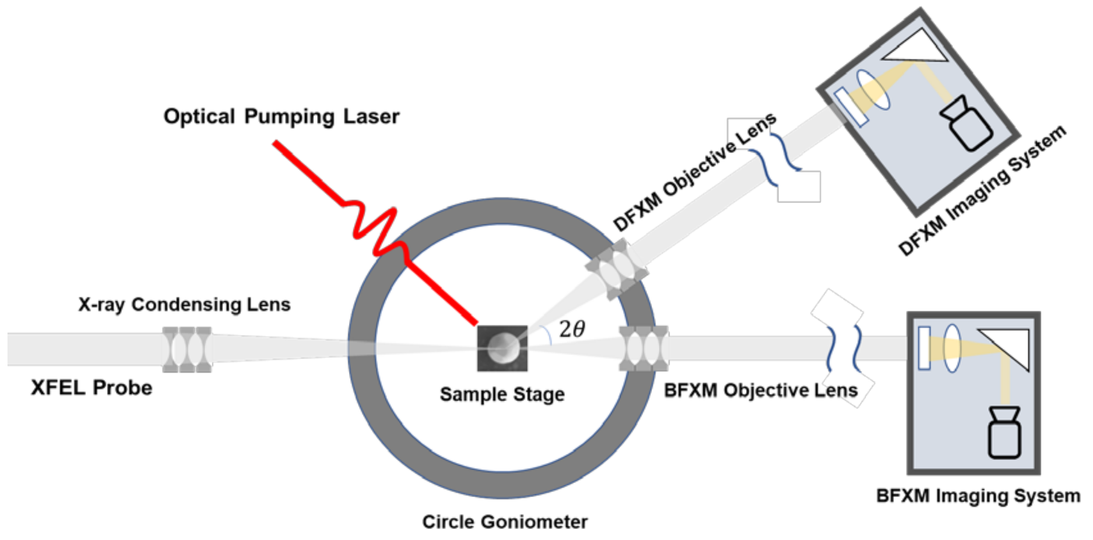




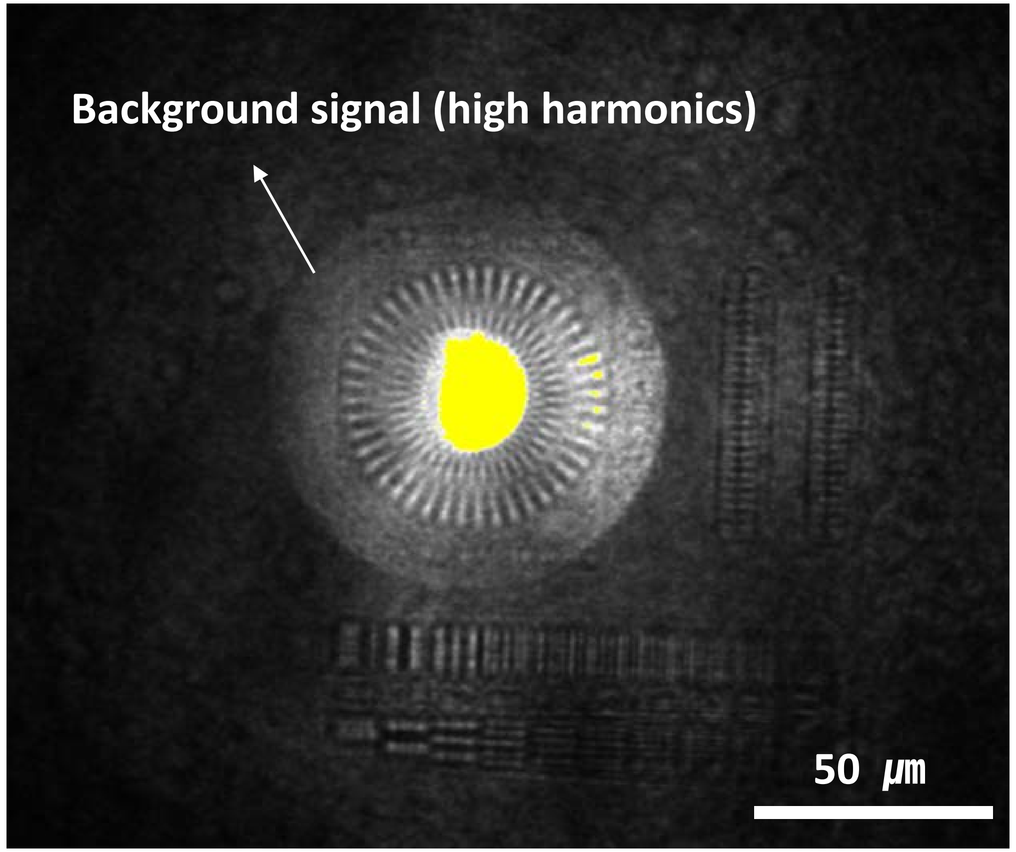


Von Hamos-type (dispersive)
- Crystal: 16 x Si (111), (220), (531)

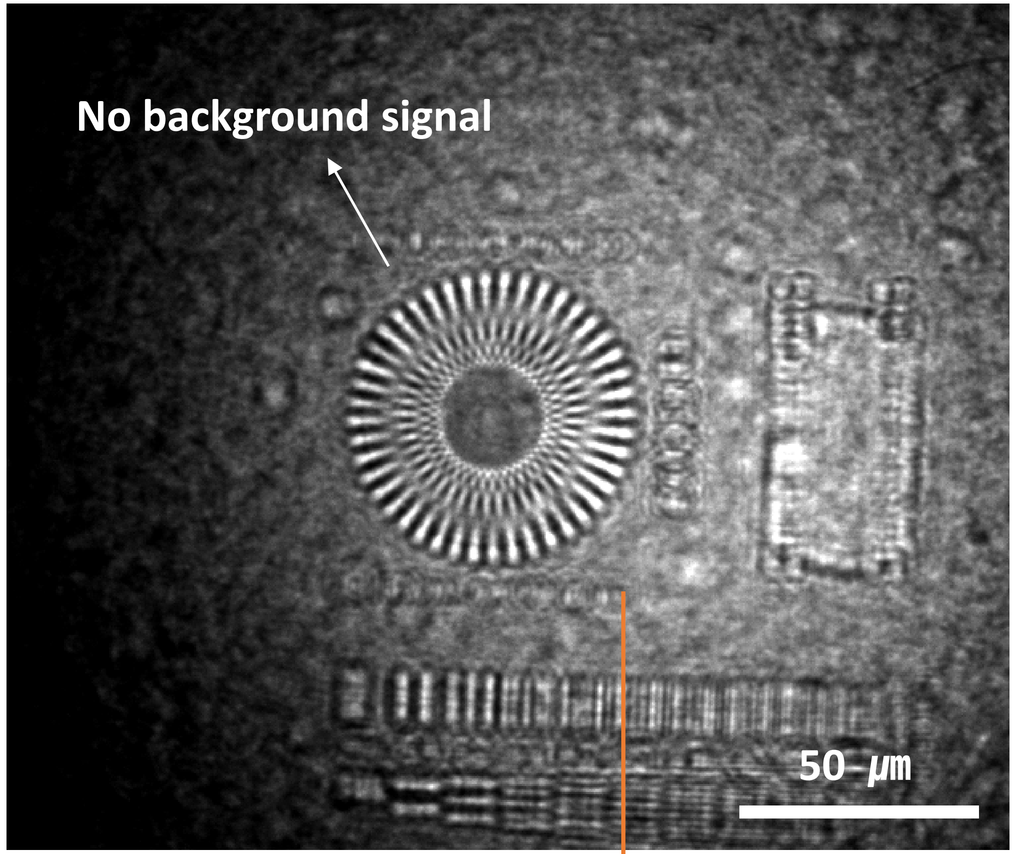
X-ray microscopy with self-seeding beam



✓ Pink beam



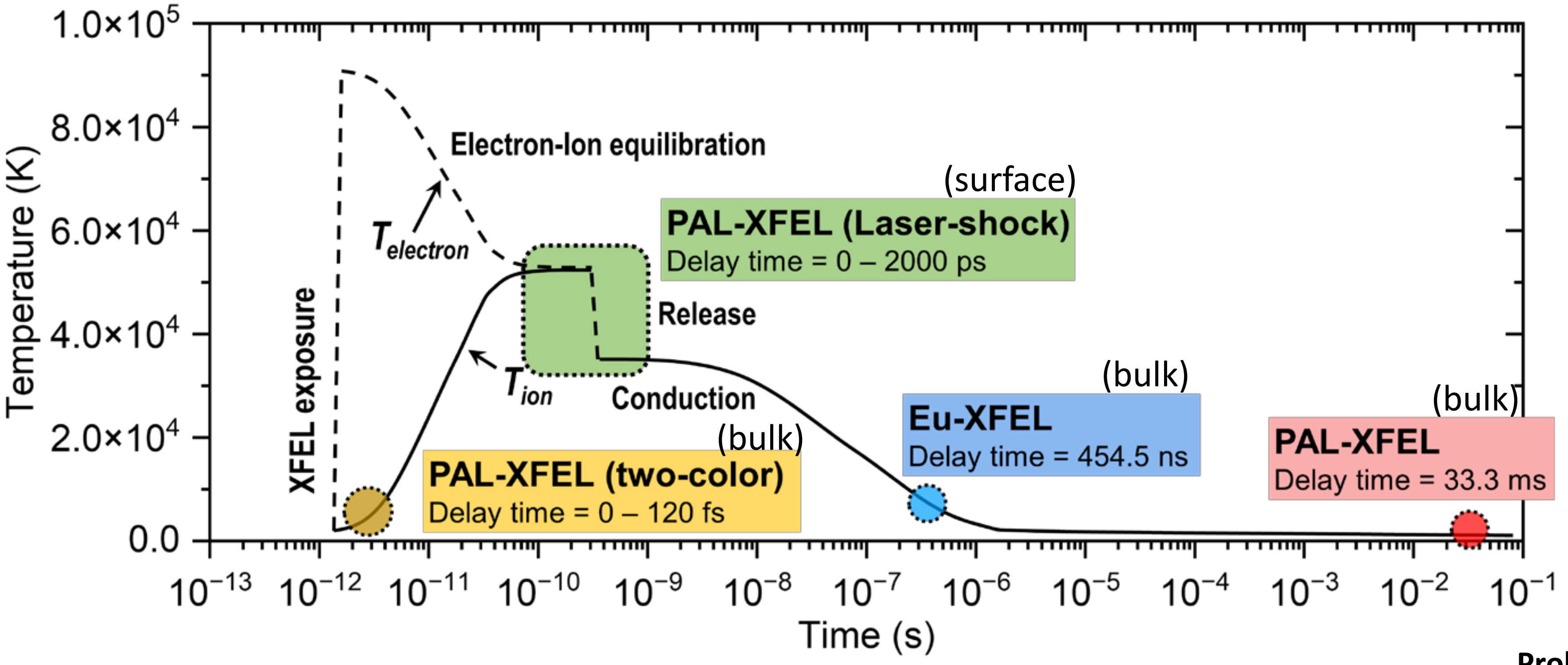
✓ Seeded beam

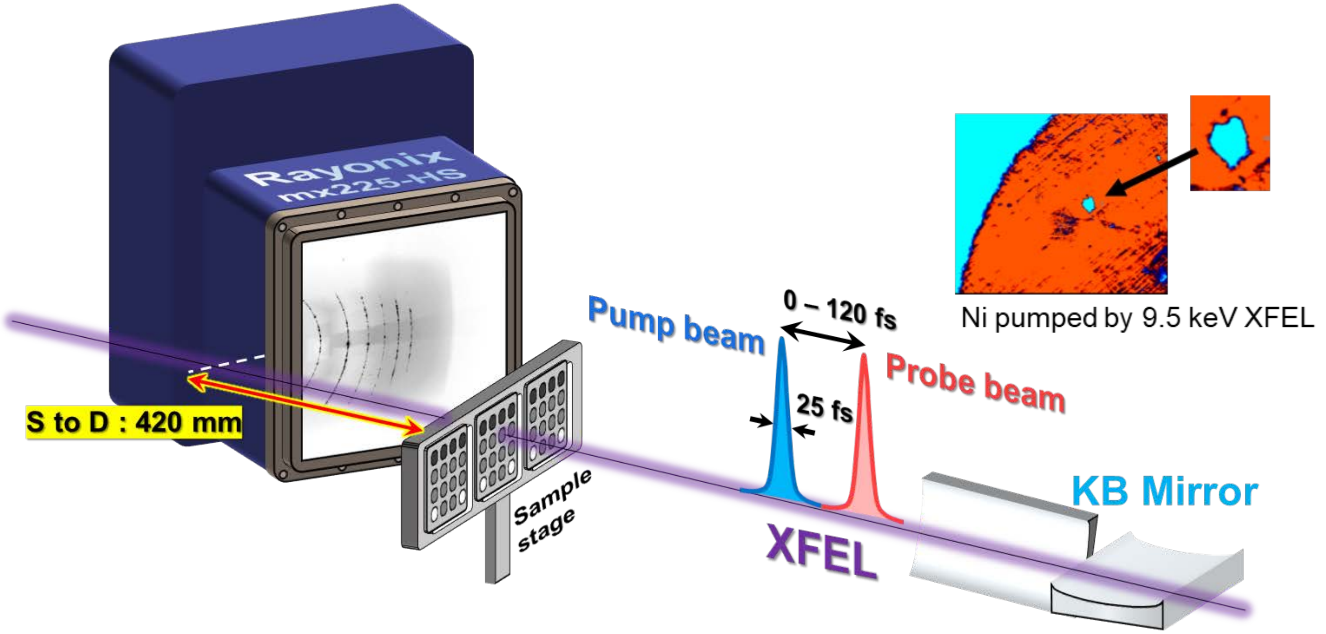


background from CRLs itself

- Bright Field X-ray Microscopy Image at E=10keV (without DCM)
  - CRLs objective lens : 37 EA (with radius of curvature 50um)
  - magnification: x21
  - seed-beam shows much better signal-to-noise ratio

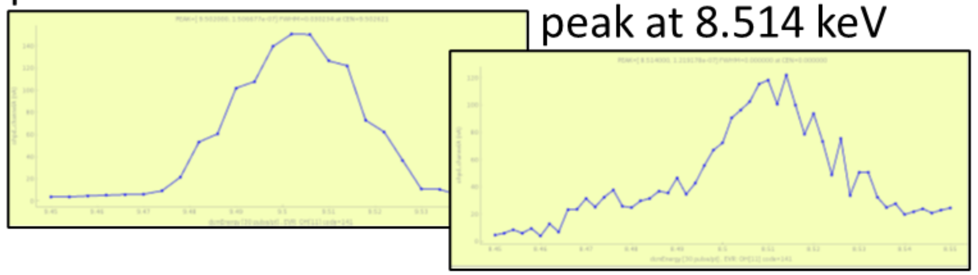
Observing ultrafast structural change in metals by two-color XFELs





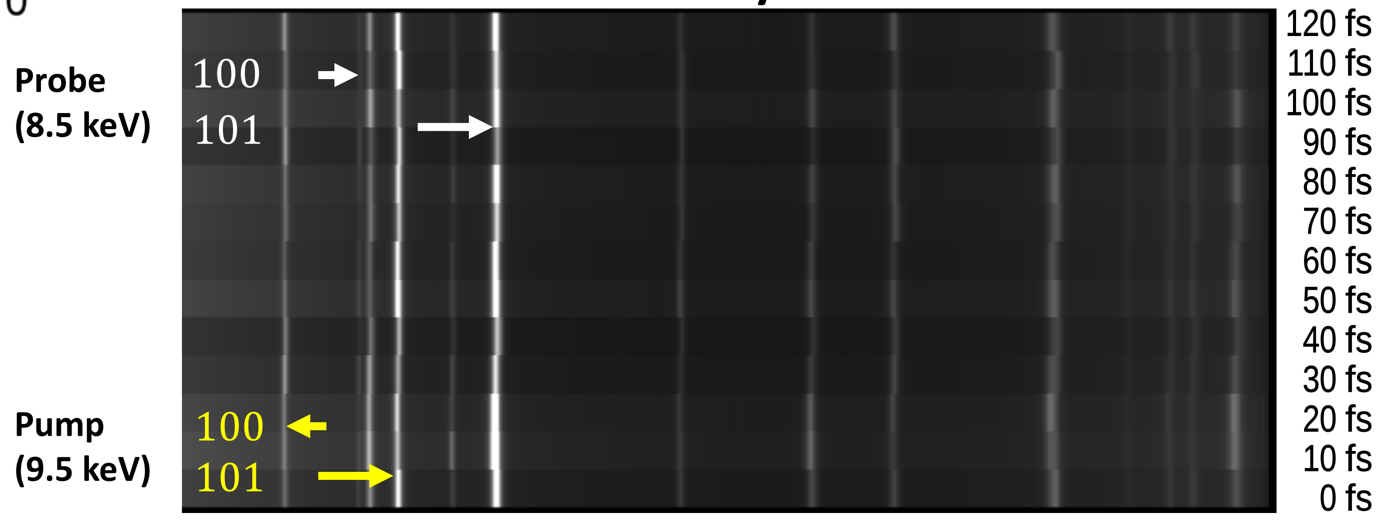
Pump:
peak at 9.502 keV

Probe:
peak at 8.514 keV



XFEL:
Photon energy: 9.5 keV pump, 8.5 keV probe
Pulse duration: 25 fs

Diffraction of Ti with **delay time**

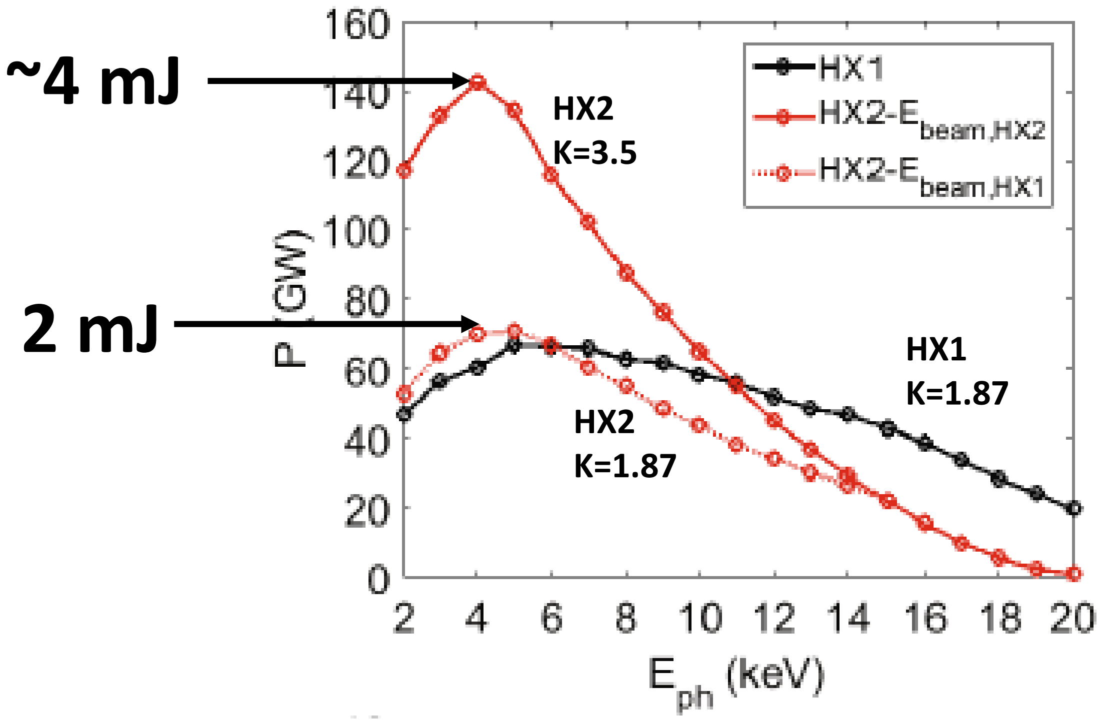


- No noticeable changes in time
- **Requires a longer time delay in the range of ps ~ ns scale**

2nd Hard X-ray undulator line of PAL-XFEL

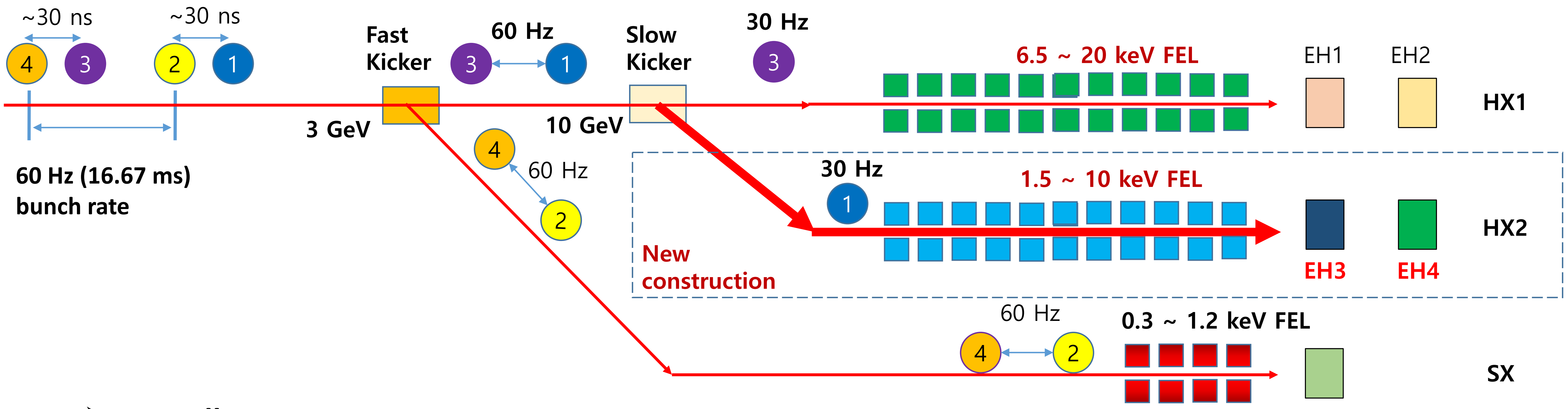
Construction plan for 2nd Hard X-ray undulator line of PAL-XFEL

	HX1	HX2	SX1
Undulator period, mm	26	35	35
Undulator K (max)	1.87	3.5	3.5
FEL photon energy, keV	6.5 ~ 20	2.0 ~ 10.0	0.3 ~ 1.2
Specialized range, keV	9~15 keV (> 1 mJ)	2 ~ 8 keV (> 3 mJ)	



- 20 undulator units & 2 experimental hutches (HX2)
- HX2 undulator parameter will be the same as SX1
- 2 – 20 keV photon energies are available both on HX1 & HX2 (Specialized range is different.)

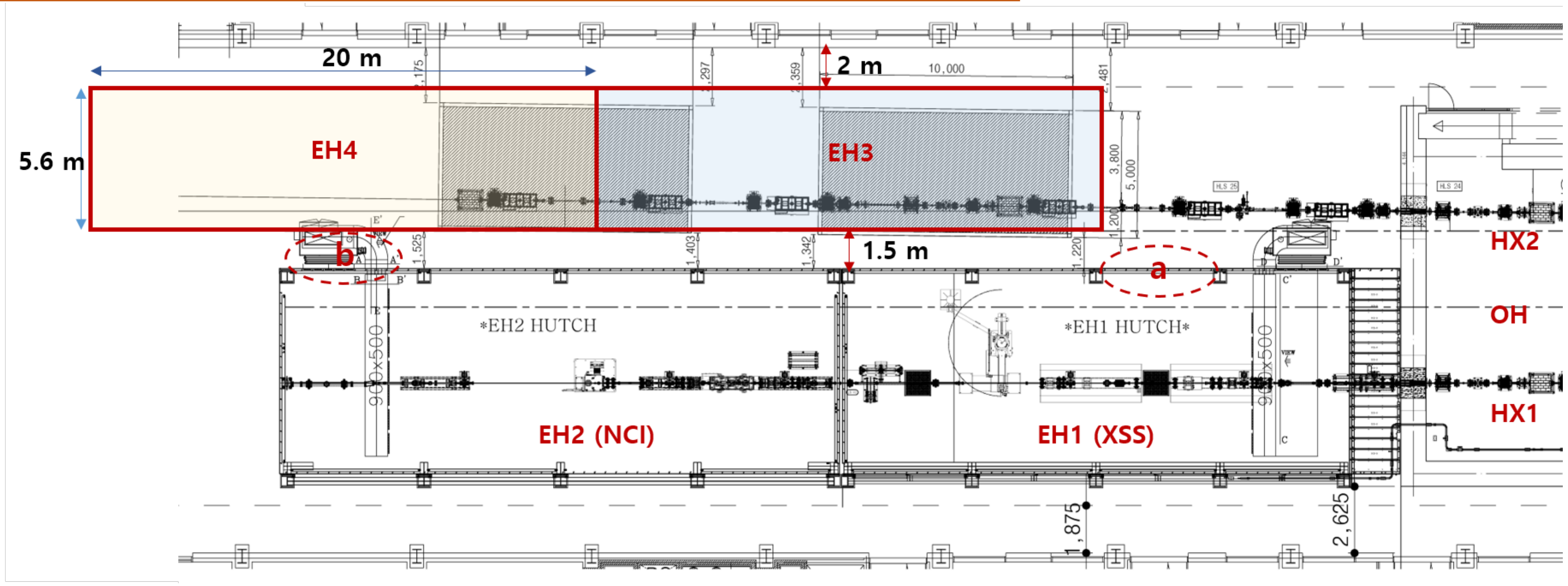
The ultimate effect of building HX2

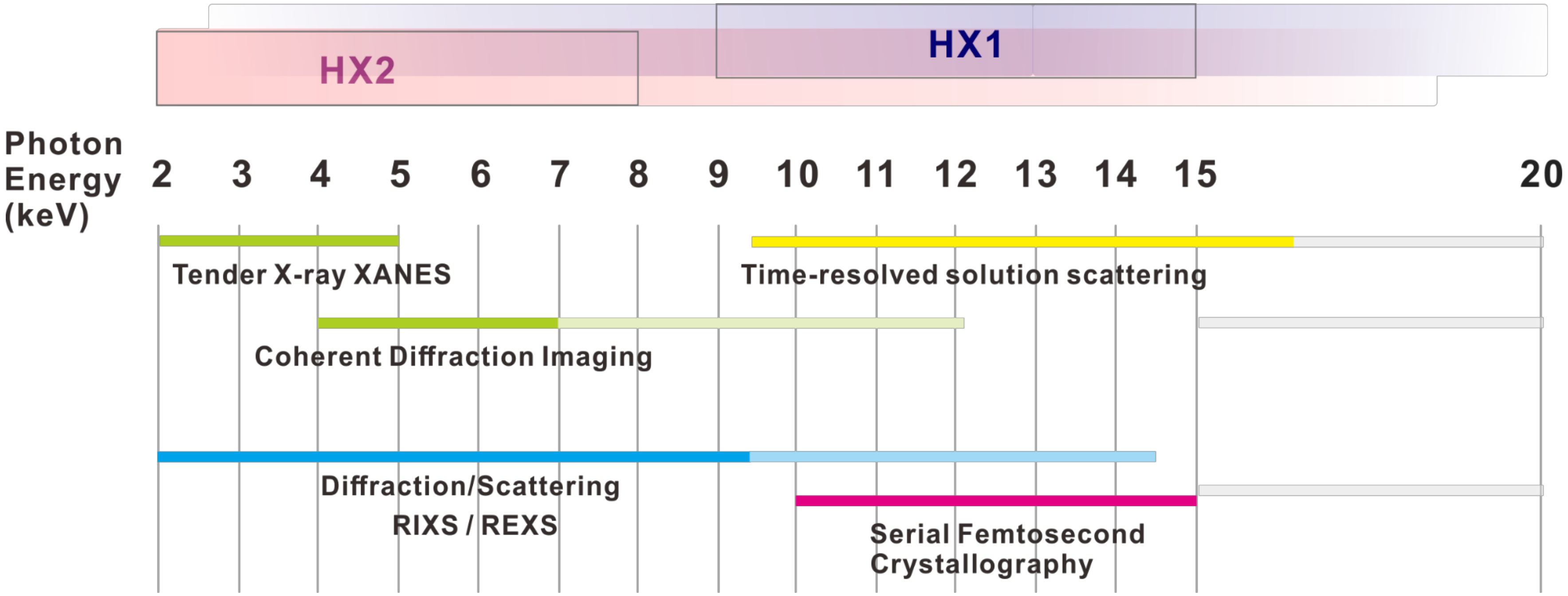


➢ **HX2 allows…**

- An increased acceptance rate of HX user experiments with higher user demand.
  (Broaden XFEL user base and Attracting new users)
- Dedicated and specialized science programs of the PAL-XFEL.
- To operate 3 independent FEL lines (HX1, HX2 & SX) through the multi-beamline operation mode.
  (Increased user beamtimes of all FEL lines as a result)

Plans for the HX hutches and instrumentations with HX2





- Distributing experimental techniques in 4 hutches based on the photon energies
- HX1/HX2 simultaneous operation
- Construction period, including commissioning (estimated) : 3 years without HX1 shutdown

Thanks to:

All PAL-XFEL Staff and Users