PAL-XFEL beamline status and plans

Intae Eom

November 14, 2023
**PAL-XFEL beamline instruments**

### XSS (X-ray Scattering and Spectroscopy)

- **Instrumentations**
  - Femtosecond X-ray Scattering (FXS)
  - Femtosecond X-ray Liquidography (FXL)
  - X-ray emission spectrometries (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 (XANES)
  - 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 (XAS/XES)
  - 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

**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

**Ultrafast X-ray imaging of the light-induced phase transition in VO₂**


- Real space SX imaging of photo-induced phase transition in VO₂ nanodomains

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


- Visualization of photo-induced spin waves
Research highlights in 2023

- The ultrafast energy transfer process in the confined nanoparticle system


- Tracking liquid domains during water-ice phase transition

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


PAL-XFEL user operation statistics

- User operation days
- Beamtime shifts (1 shift = 12 hours)

- # of User experiments:
  - 2017: 19
  - 2018: 36
  - 2019: 54
  - 2020: 63
  - 2021: 62
  - 2022: 89
  - 2023: 77

- # of User publications:
  - 2017: 1
  - 2018: 3
  - 2019: 5
  - 2020: 11
  - 2021: 14
  - 2022: 15
  - 2023: 14
## 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)

### Provided beamtime shifts

<table>
<thead>
<tr>
<th>Beamtime category</th>
<th>2023-1st</th>
<th>2023-2nd</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Regular beamtime</strong></td>
<td>HX 82 shifts (24h support 12 days)</td>
<td>HX 104 shifts (24h support 34 days)</td>
</tr>
<tr>
<td>(70 days per half year)</td>
<td>SX 36 shifts</td>
<td>SX 36 shifts</td>
</tr>
<tr>
<td><strong>R&amp;D beamtime</strong></td>
<td>R&amp;D: 12 days (12 shifts)</td>
<td>Director’s beamtime: 9 days (18 shifts)</td>
</tr>
<tr>
<td>(1st half 23 days, 2nd half 27 days)</td>
<td>Beamline M/S: 11 days (17 shifts)</td>
<td>Potential users: 6 days (12 shifts)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>New instrumentation: 3 days (6 shifts)</td>
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<tr>
<td></td>
<td></td>
<td>Screening beamtime: 2 days (4 shifts)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Tutorial beamtime: 0.5 days (1 shift)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Beamline M/S: 6.5 days (13 shifts)</td>
</tr>
</tbody>
</table>
Science opportunities at PAL-XFEL

**Research topics**

**Condensed matter physics and Material sciences**
- Thermal / Non-thermal melting dynamics
- Phase transitions
- Phonon / Lattice dynamics
- Spin/Charge/Magnetic properties

**Chemical sciences**
- Bond dissociation / Formation / Isomerization
- Charge transfer / Recombination / Localization
- Chemical reaction / Catalytic reaction

**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

**Instrumentations**

- **HX**
  - XSS
  - NCI
  - SSS

- **SX**
  - FXS
  - CXI
  - RSXS

- **FXL**
  - RIXS (RXES)
  - Bragg-CDI

- **WAXS**
  - CXI

- **FTH**
  - RSXS

- **XANES**
  - XAS/XES

- **XES**
  - XAS/XES

- **SFX**
  - SFX

**Science opportunities at PAL-XFEL**

22nd International Advisory Committee Meeting (November 13-14, 2023)
1. Time-resolved XFEL experiments for condensed matter physics and material sciences

- **Quantum material research (FXS, RSXS)**
  : Finding and controlling hidden physics under specific sample environments

- **Multiplexing measurement (SAXS + WAXS + XES)**
  : Simultaneous measurements from single particle

- **Development of XFEL imaging techniques**
  : Dark / Bright field XFEL microscopy
  - Time-resolved CXI with nanofocusing
  - Fourier transform holography (SX)

Dr. Chun’s presentation in PX6

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

Dr. Nam’s presentation in PX7
Sample environments for quantum material research at FXS, RSXS

- High T sample holder
- Cryostream with a goniometer (40 – 300 K)
- Low temperature/High Magnetic field (9 T, 4 K) in collaboration with PSC

RSXS sample environments
- Low-temperature diffraction chamber for Tender X-rays in collaboration with Sogang Univ.
- 6-axis cryostat manipulator
  - Base $T_{\text{sample}}$ ~20 K (liquid helium)
- Avalanche photodiode detector 0-D point detector
- Horizontal linear polarized X-ray
- Horizontal sample ($\theta$) and detector (2$\theta$) rotation (r-polarization configuration)
- Plan: Controllable magnetic field
  - 2D detector (AXIS)
  - Pump option (THz, Mid-IR)
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

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

Setup in XSS hutch (side view)

<table>
<thead>
<tr>
<th>Strong</th>
<th>Weak</th>
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</thead>
</table>
| **XAS** | Sensitivity to redox state  
Easy to measure | Hard to identify spin state |
| **XES** | Sensitive to spin state  
Hard to measure transient signal |

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

![Image of setup in XSS hutch](image_url)

![Graph showing Fe^{3+}, Kβ emission](graph_url)
Dr. Sanghan Park

**Fourier transform holography (SX)**

- **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.

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

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

**Focused beam profiles @ 9.5keV**

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

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Nano-focused CRL (portable)  
Dr. Sangsoo Kim

- 25-CRL stack
- Wave propagation (H)
- Wave propagation (V)

<table>
<thead>
<tr>
<th>Energy</th>
<th>Focal length</th>
<th>Spot Size</th>
<th>Effective Aperture</th>
<th>Transmission</th>
</tr>
</thead>
<tbody>
<tr>
<td>10.0 keV</td>
<td>300 mm</td>
<td>160~180 nm</td>
<td>258 μm</td>
<td>0.5</td>
</tr>
</tbody>
</table>
Sample deliveries for Macromolecular dynamics

Dr. Jaehyun Park

- **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

- 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

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

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

Spin-crossover complex: photo-activated spin state conversion

Experimental condition

- Sample: 50 mM Fe(phen)$_3$Cl$_2$ in water (150 µm 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

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

Spin-crossover complex: photo-activated spin state conversion
Hard X-ray RIXS, RXES using Self-seeded FEL

1) RIXS: Incident energy fixed, Bragg angle scanned

2) RXES: Incident energy scanned, Bragg angle fixed

Courtesy of Dr. S.H. Chun & T.K. Choi at PAL-XFEL
“Resonant X-ray emission spectroscopy using self-seeded hard X-ray pulses at PAL-XFEL”, JSR 2023
X-ray microscopy with self-seeding beam

- 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

- pink beam shows much better signal-to-noise ratio
- seeded beam shows no background signal

- CRLs objective lens : 37 EA (with radius of curvature 50um)
  - magnification: x21
  - seed-beam shows much better signal-to-noise ratio

- No background signal from CRLs itself
Observing ultrafast structural change in metals by two-color XFELs

- No noticeable changes in time
- Requires a longer time delay in the range of ps ~ ns scale
2\textsuperscript{nd} Hard X-ray undulator line of PAL-XFEL
Construction plan for 2nd Hard X-ray undulator line of PAL-XFEL

<table>
<thead>
<tr>
<th></th>
<th>HX1</th>
<th>HX2</th>
<th>SX1</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Undulator period, mm</strong></td>
<td>26</td>
<td>35</td>
<td>35</td>
</tr>
<tr>
<td><strong>Undulator K (max)</strong></td>
<td>1.87</td>
<td>3.5</td>
<td>3.5</td>
</tr>
<tr>
<td><strong>FEL photon energy, keV</strong></td>
<td>6.5 ~ 20</td>
<td>2.0 ~ 10.0</td>
<td>0.3 ~ 1.2</td>
</tr>
<tr>
<td><strong>Specialized range, keV</strong></td>
<td>9~15 keV ( &gt; 1 mJ )</td>
<td>2 ~ 8 keV ( &gt; 3 mJ )</td>
<td></td>
</tr>
</tbody>
</table>

- 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

- 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 hutch 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