X-ray Free Electron Laser Part-2 Photon Beamline and Experiments

XFEL Utilization Division, JASRI

Kensuke Tono

Contents

1. XFEL sciences
2. Photon beam properties
3. Photon beamline: Optics and diagnostics
4. Experimental stations
5. Experiments at SACLA

Contents

1. XFEL sciences
2. Photon beam properties
3. Photon beamline: Optics and diagnostics
4. Experimental stations
5. Experiments at SACLA

XFEL properties and sciences

- Short pulse (<10 fs)
- High peak power (>60 GW)
- Coherent

Ultrafast observation beyond the speed of atomic motion (Femtosecond snapshot)

- Beyond static image
  - Imaging functions (motion pictures of chemical reaction, phase transition, etc.)
- Beyond statistical image
  - Imaging fluctuations, rare events

Ultrahigh intensity opens new regime of X-ray-matter interactions

- Beyond linear response

Femtosecond snapshot

Imaging of a *live* cell

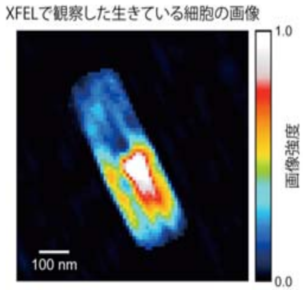
nature COMMUNICATIONS

ARTICLE

Received 17 Jul 2013 | Accepted 2 Dec 2013 | Published xx xxx 2013 DOI: 10.1038/ncomms4052 OPEN

Imaging live cell in micro-liquid enclosure by X-ray laser diffraction

Kimura, Nishino et al., ***Nat. Comm.*** (2013).



Damage-free protein crystallography

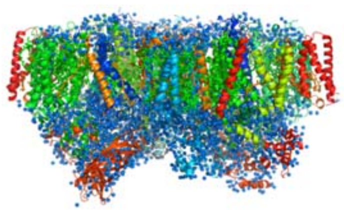
LETTER

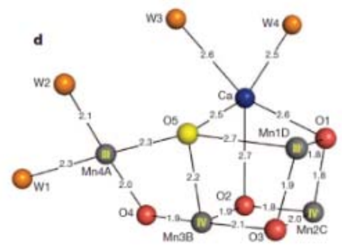
doi:10.1038/nature1399

Native structure of photosystem II at 1.95 Å resolution viewed by femtosecond X-ray pulses

Suga, Shen *et al.*, ***Nature*** (2015).

Photosystem II (PSII) Reaction center





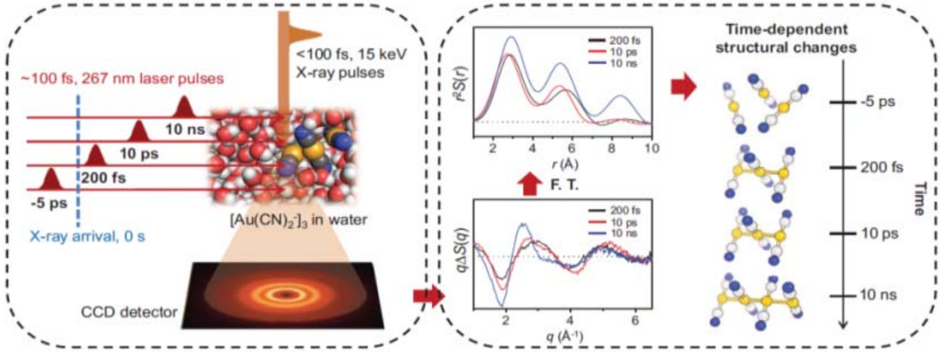
Ultrafast dynamics in chemical reaction

LETTER

doi:10.1038/nature14163

Direct observation of bond formation in solution with femtosecond X-ray scattering

Kim, Ihee, Adachi, et al., ***Nature*** (2015).



Ultrahigh intensity application: X-ray nonlinear optics

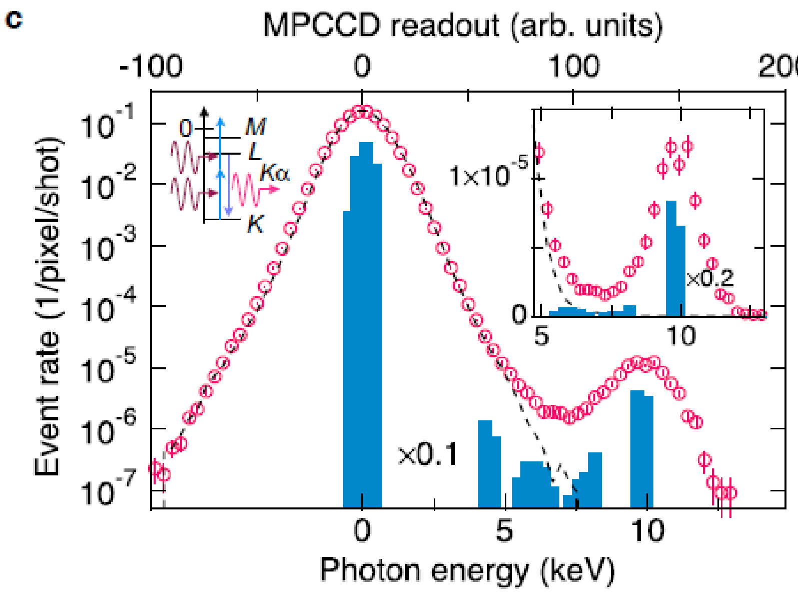
50 nm focusing ⇒ ~10^{20} W/cm^2

Multiphoton process

nature photonics | LETTERS
PUBLISHED ONLINE: 16 FEBRUARY 2014 | DOI: 10.1038/NPHOTON.2014.10

X-ray two-photon absorption competing against single and sequential multiphoton processes

Tamasaku et al., ***Nat. Photon*** (2014)

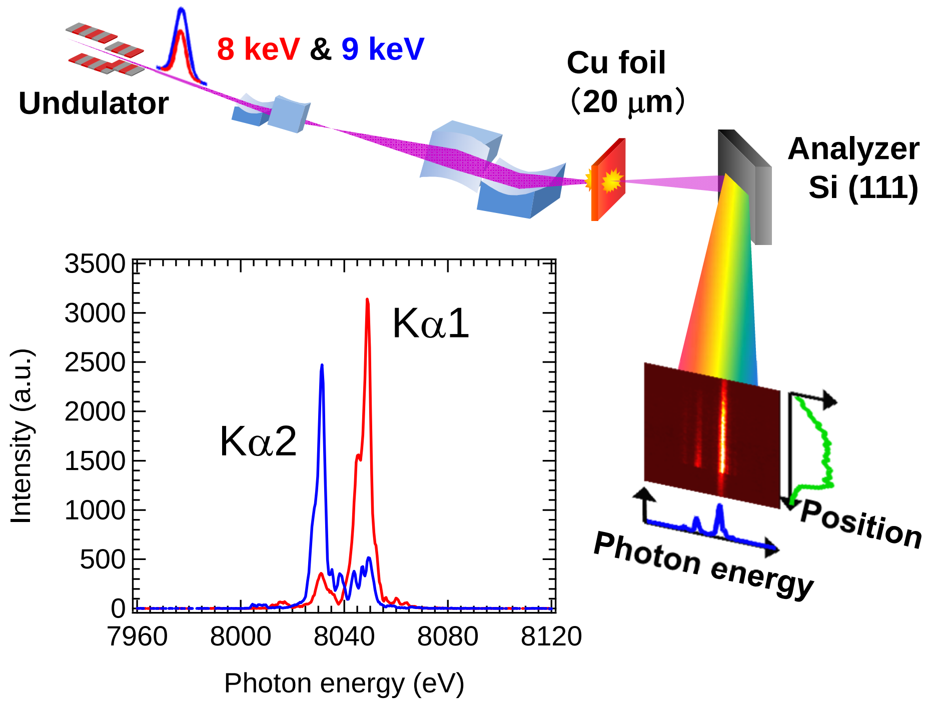


Amplification of x-ray pulse using 2-pulse XFEL

LETTER
doi:10.1038/nature14894

Atomic inner-shell laser at 1.5-ångström wavelength pumped by an X-ray free-electron laser

Yoneda et al., ***Nature*** (2015)



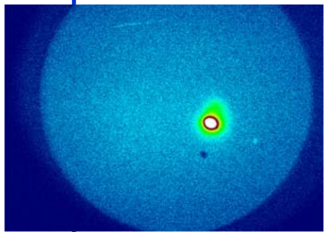
Contents

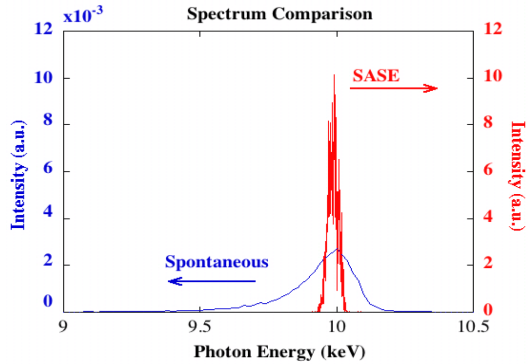
1. XFEL sciences
2. Photon beam properties
3. Photon beamline: Optics and diagnostics
4. Experimental stations
5. Experiments at SACLA

Properties of SASE XFEL beam

Low emittance & short pulse

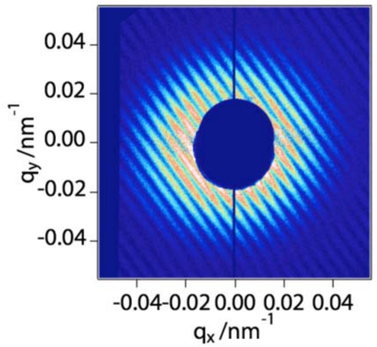
- Source size ~30 μm@10 keV
- Divergence ~2 μrad@10 keV
- Bandwidth ~5×10^{-3}
- Pulse duration <10 fs

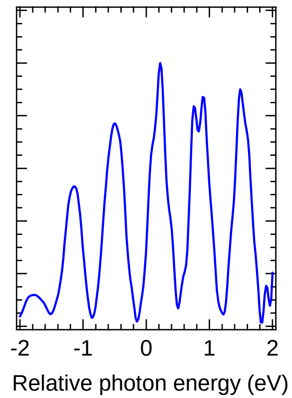




Coherent

- Transverse only
- Multimode in longitudinal

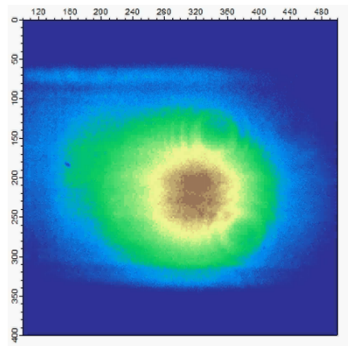




High intensity

- Pulse energy ~0.6 mJ @10 keV (~4×10^{11} photons)
- Peak power >60 GW@10 keV

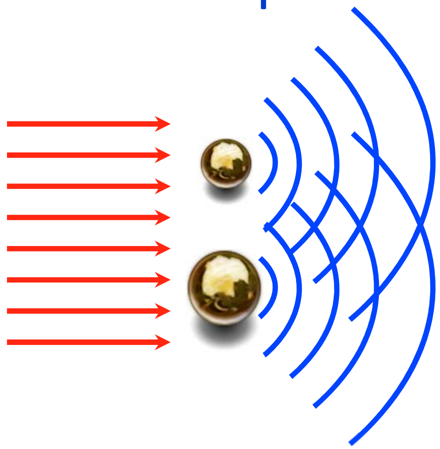
Shot-by-shot fluctuation

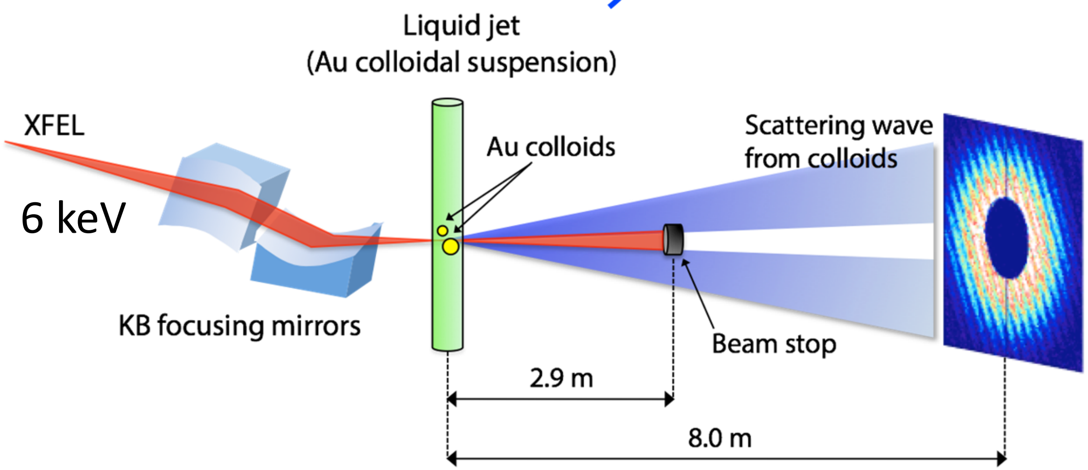


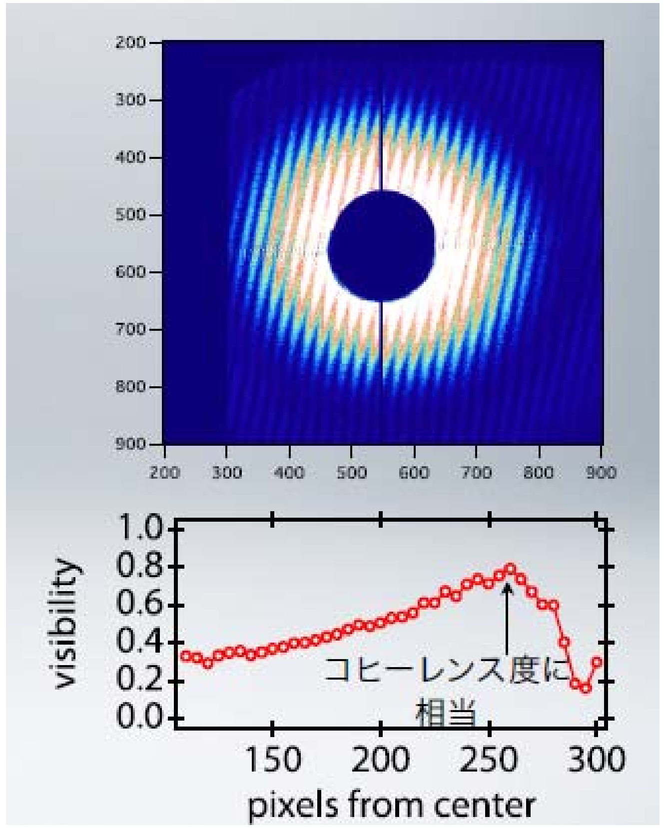
Coherent (transverse only)

Interference between scattering waves from two particles

Inoue (U. Tokyo) et al., IUCrJ, in press (2015)





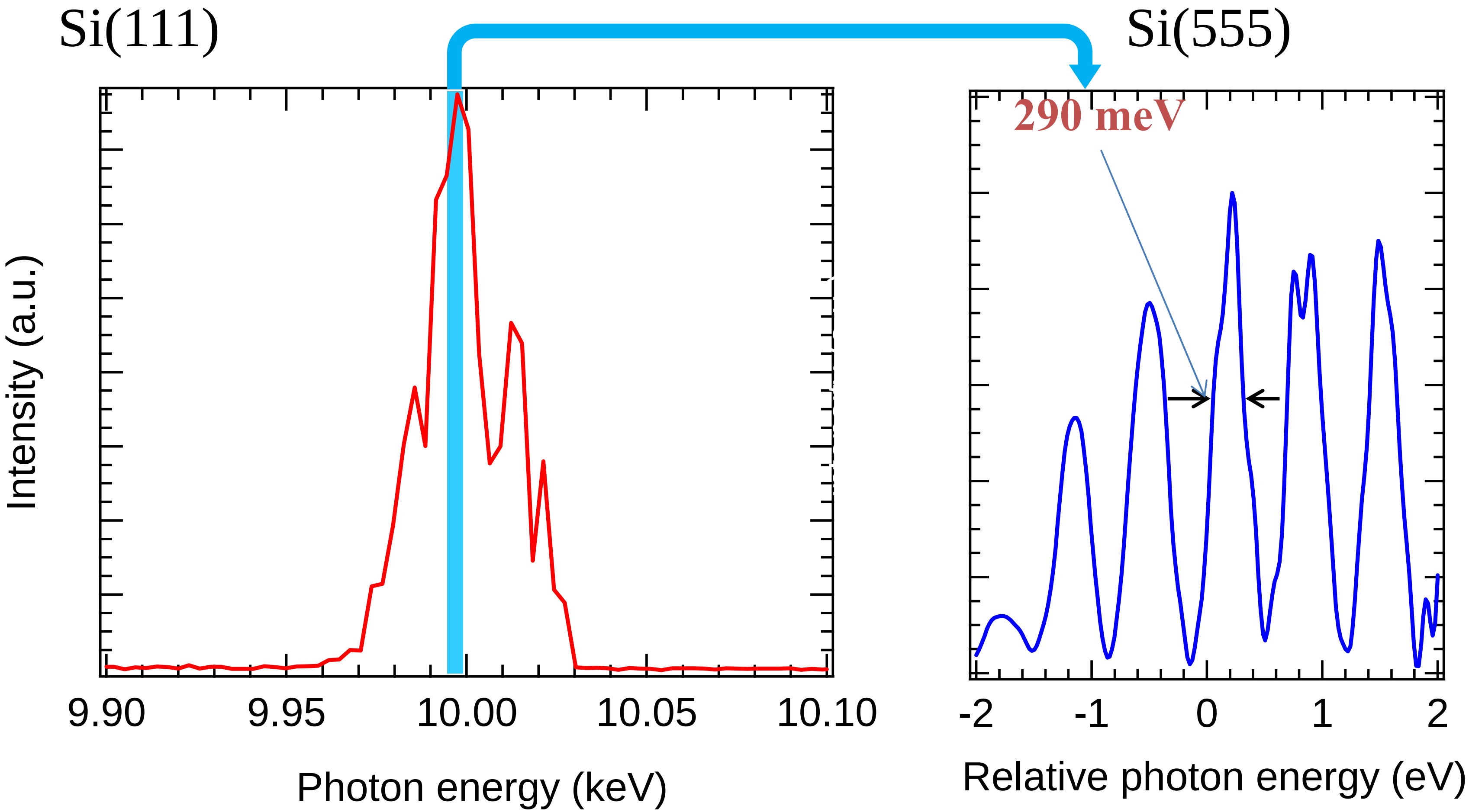


Total degree of coherence: ~0.6

~80% of the total power is in the dominant mode (TEM_{00})

Multimode

Spectrum of single XFEL pulse consists of thousands of spikes due to multi optical modes.



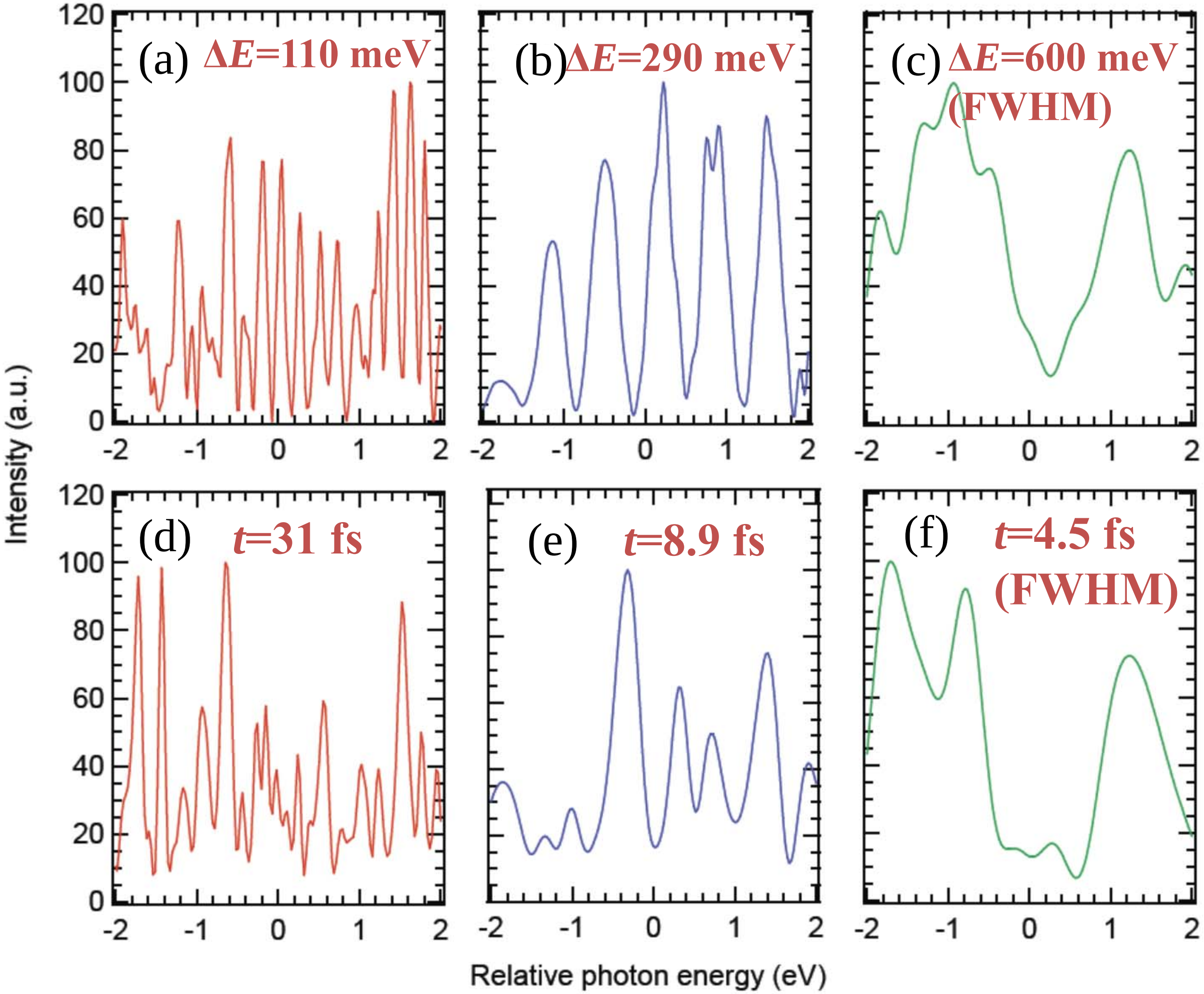
Y. Inubushi *et al.*, *Phys. Rev. Lett*. **109**, 144801 (2012).

Spectra at different pulse durations

Inubushi et al., Phys. Rev. Lett. 109, 144801 (2012)

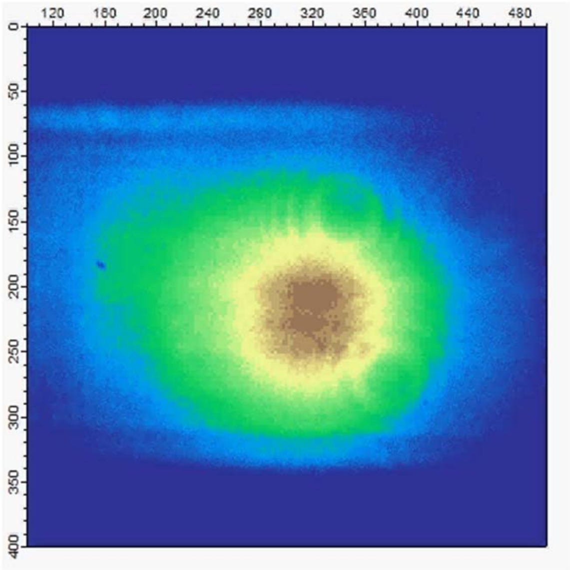
Measured Spectra

Simulated spectra

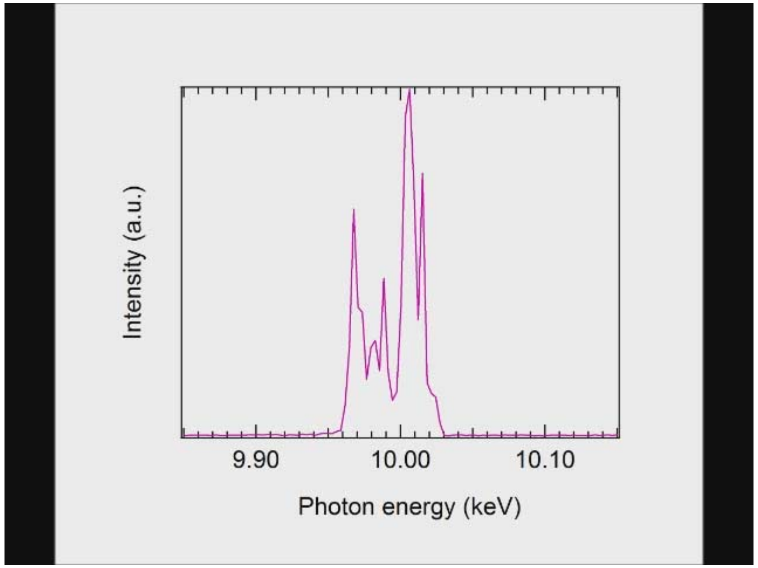


Shot-by-shot fluctuation

Intensity/position



Spectrum



Photon-beam parameters and experimental data should be collected in a shot-by-shot manner.

Contents

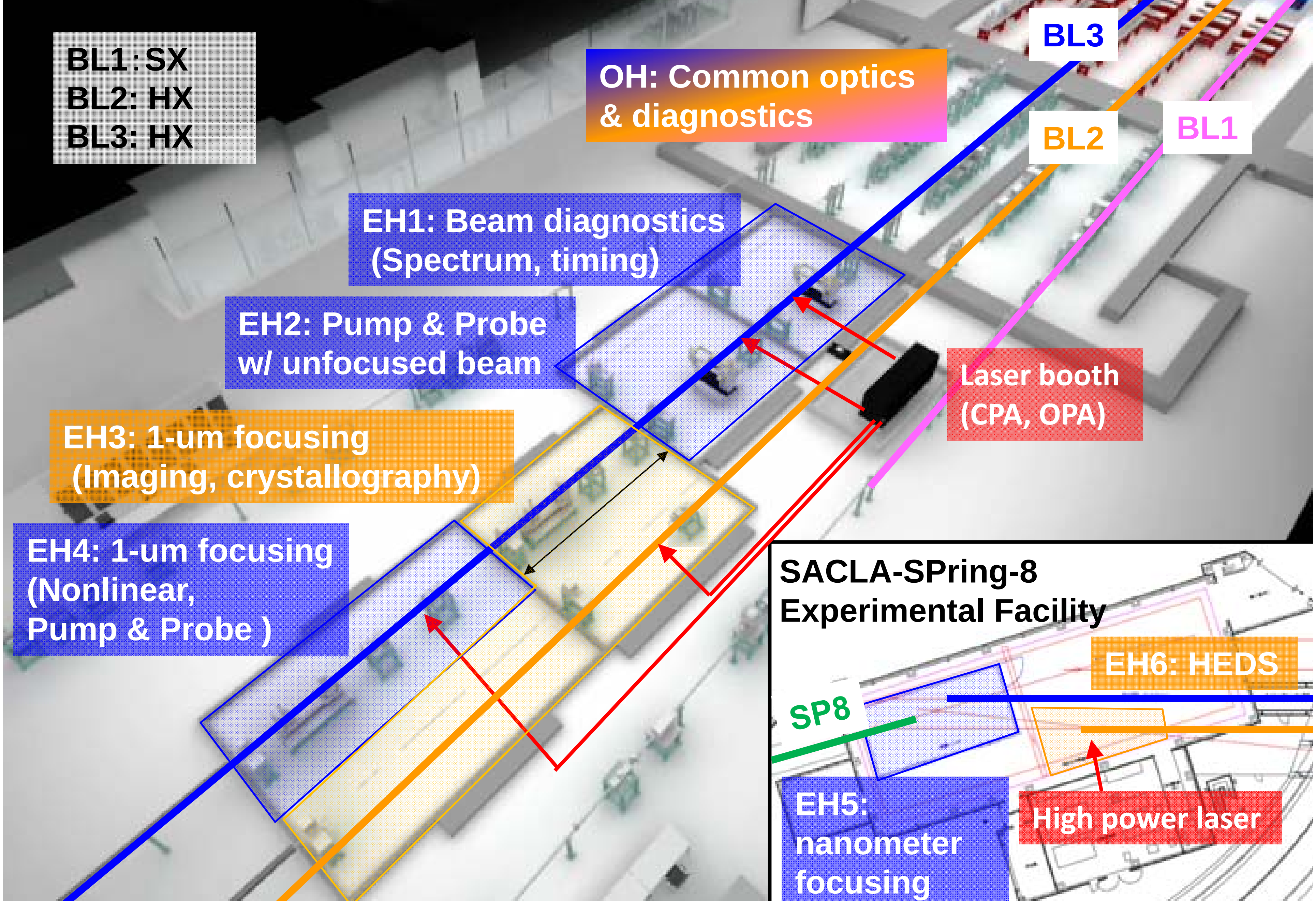
1. XFEL sciences
2. Photon beam properties
3. Photon beamline: Optics and diagnostics
4. Experimental stations
5. Experiments at SACLA

Design concept

- **Main optics & diagnostics** are centralized in Optics Hutch
  -> Transport & online diagnostics of a photon beam with low emittance, short pulse, and high coherence.
  -> Fine electron-beam tuning with X-ray optics & diagnostics.

- Experimental stations provide only basic infrastructure (e.g., optical laser, focusing system)
  -> Enough space for various experimental instruments

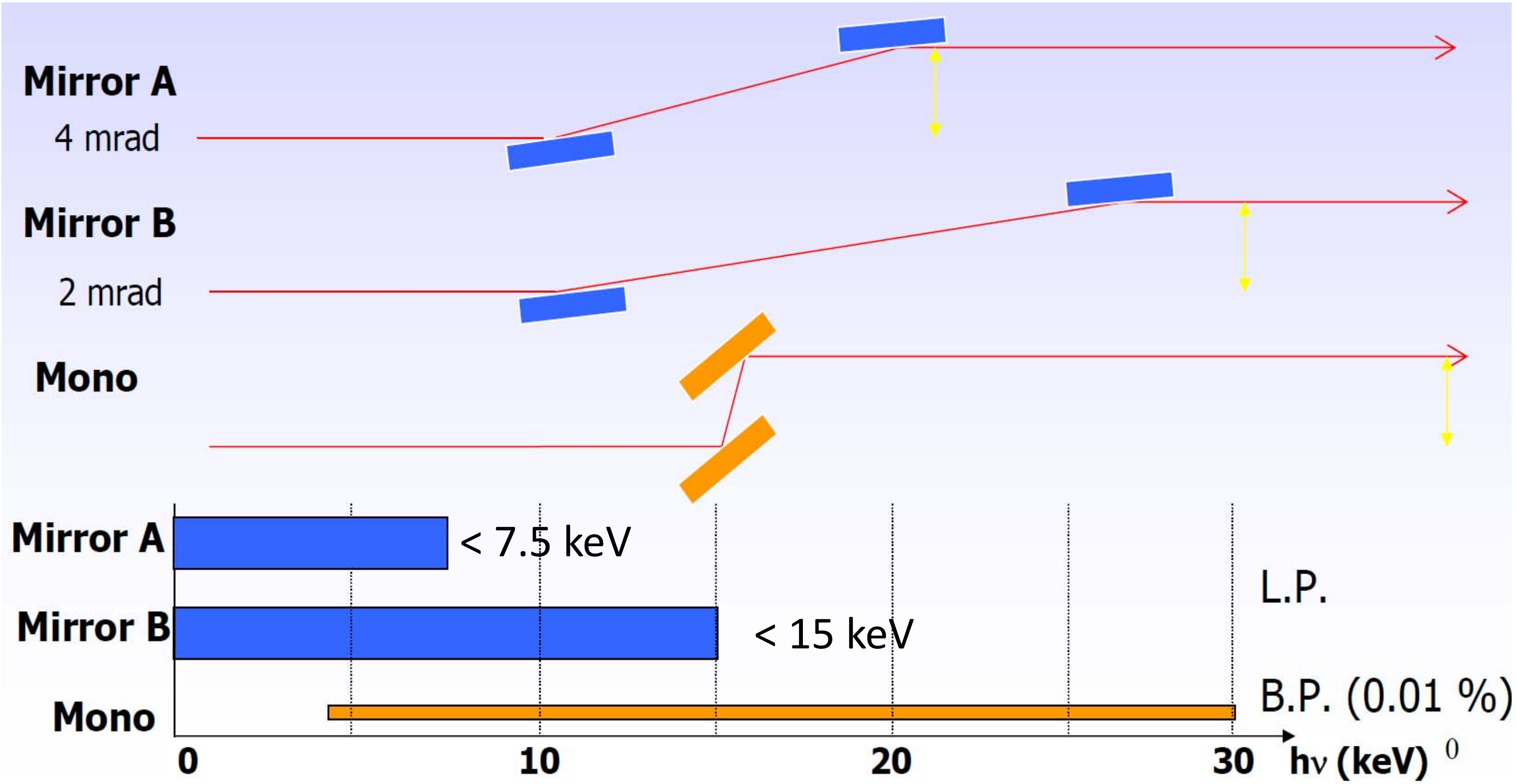
SACLA Photon Beamline



Common optics in optics hutch

Transport XFEL beam & filter out unnecessary lights

- Double plane mirrors (2 sets): Low-pass filter (Bandwidth of output beam ~5×10^{-3})
- Double crystal monochromator (DCM, Si 111): Band-pas filter (~1×10^{-4})



Damage/speckle free optical elements

XFEL features	Need to avoid…
Short pulse (<10 fs)	⇒ Damage
High peak power (>60 GW)	
Coherent	⇒ Wavefront distortion

Damage on a mirror material

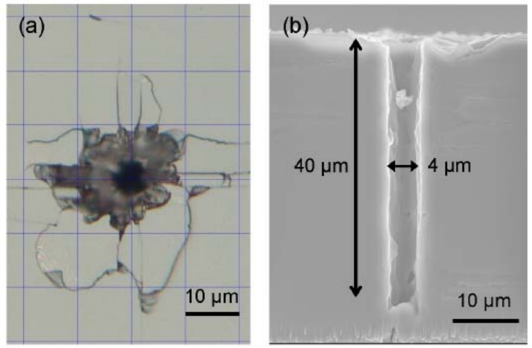
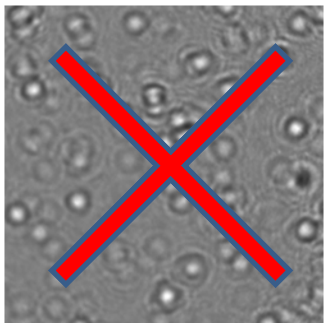


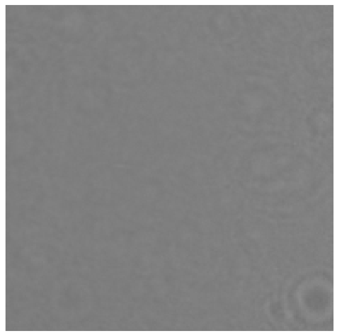
Fig. 2. (a) Optical microscope image of irradiated silicon viewed from surface at fluence of 57 $\mu J/\mu m^2$. (b) Cross sectional SEM image of (a) prepared by focused ion beam sampling.

Koyama et al., Opt. Exp. 21 (2013)

Typical Be window



Speckle-free Be window

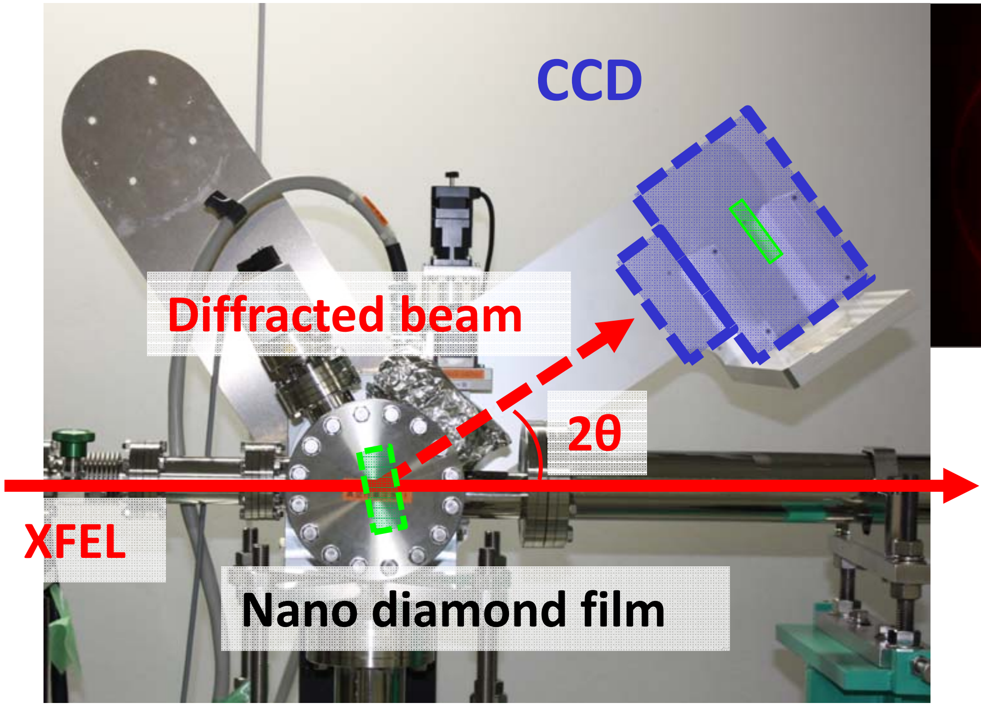


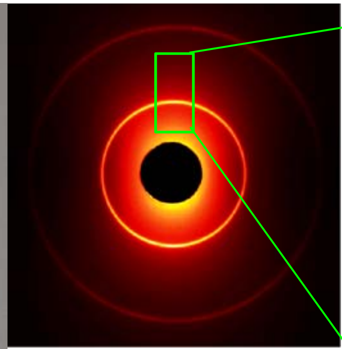
Goto et al., Proc. of SPIE 6705 (2007)

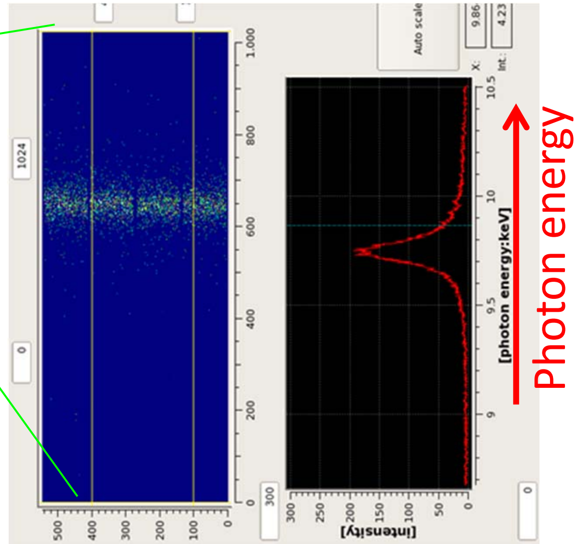
Ultraprecise mirror finished by Elastic Emission Machining

Mimura et al., Rev. Sci. Instrum. **79**, (2008)

On-line photon diagnostics: Wavelength (photon-energy) monitor



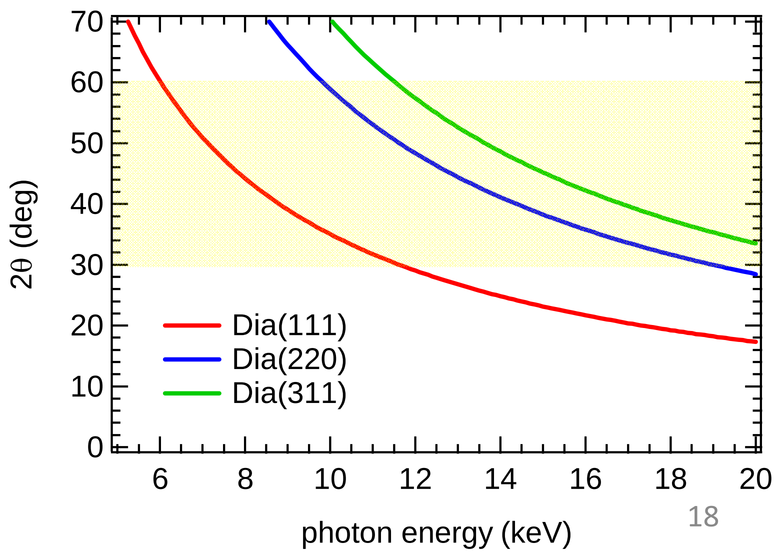




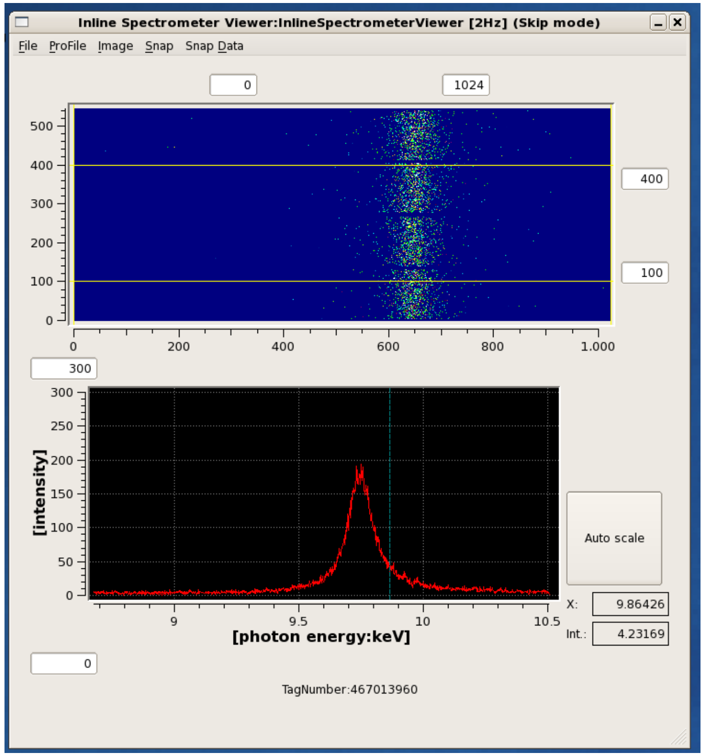
Wavelengths (λ) are calculated from positions of Debye-Scherrer rings on MPCCD.

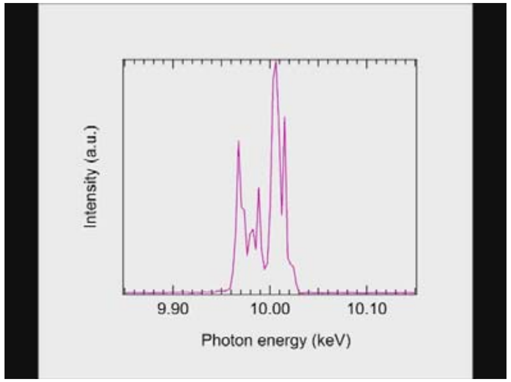
$2d\sin\theta = n\lambda$

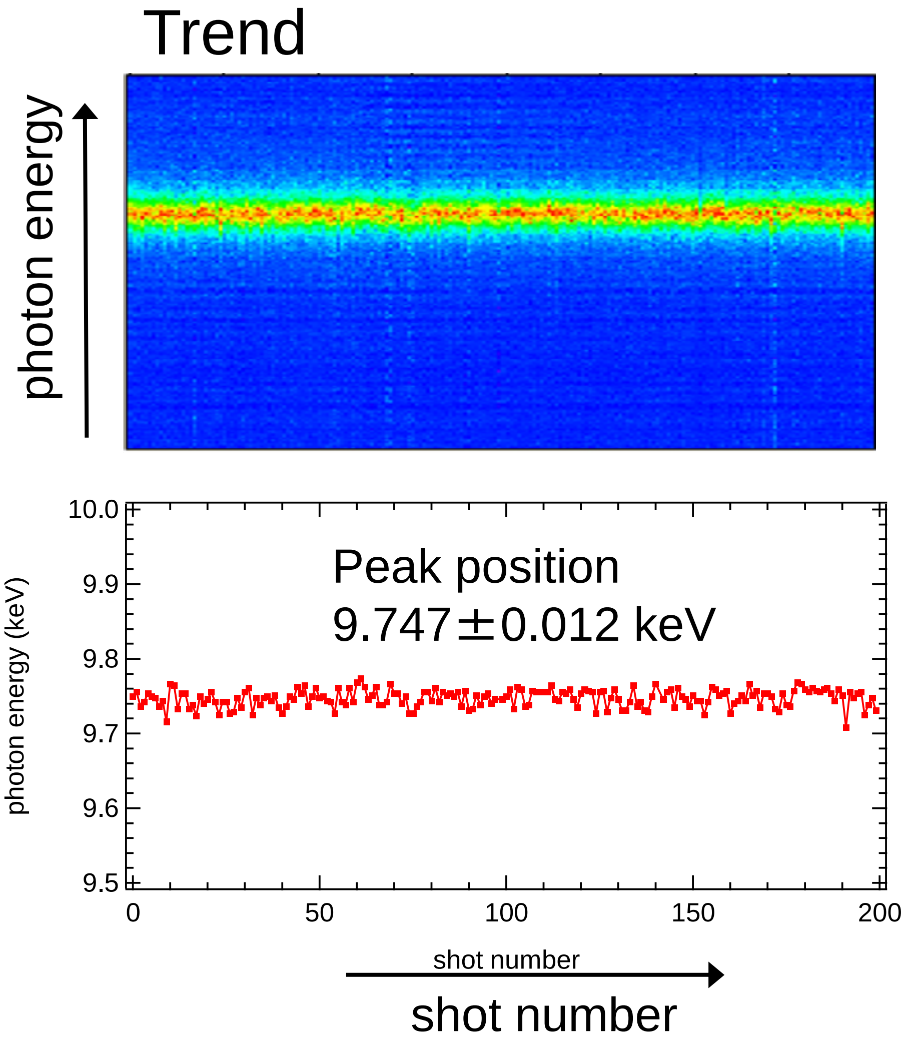
Measurable range: 6 - 20 keV



Shot-by-shot measurement





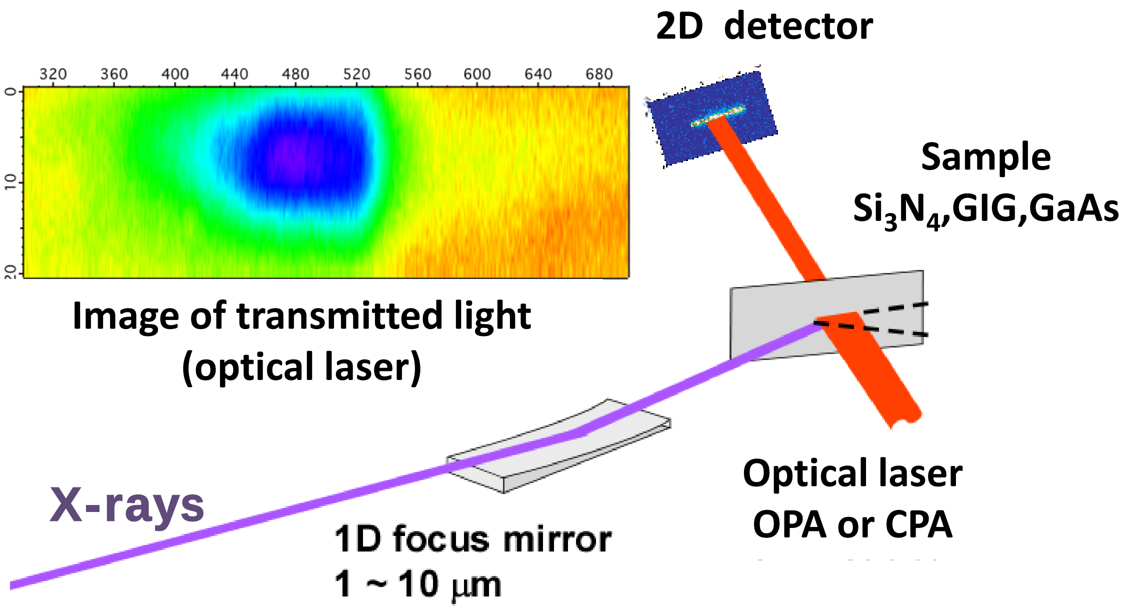


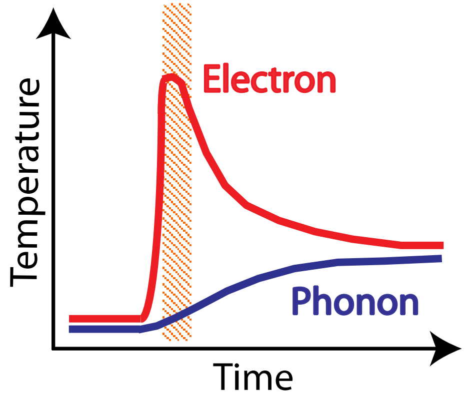
Arrival timing monitor

Katayama –san
Sato-san (U. Tokyo)

Intense X-ray irradiation induces optical-transmittance change of a semiconductor (Spatial decoding technique)

T. Sato et al
APEX 8, 012702 (2015)





two temperature model

Electron-electron scattering of photoelectrons
-> cascade hot-carrier generation

Optical transmittance decreases

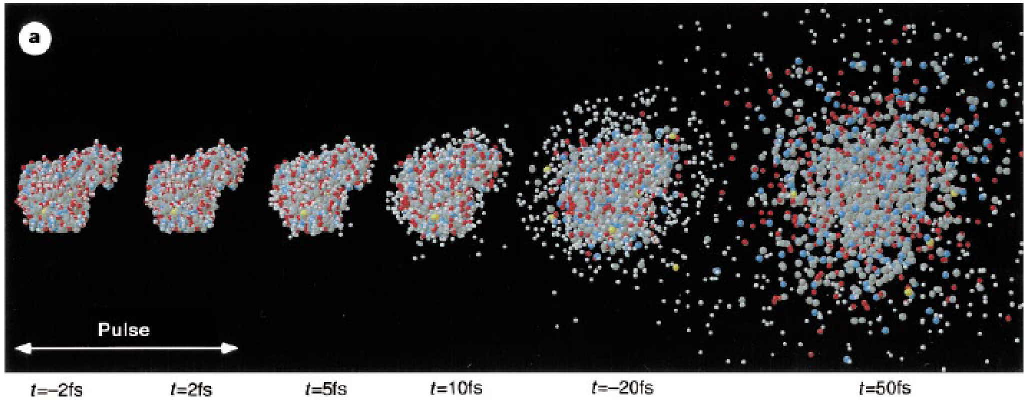
S. M. Durbin, et al., X-ray pump optical probe cross correlation study of GaAs *Nature Photonics* **6**, 111 (2012)
M. Harmand, et al., Achieving few-femtosecond time-sorting at hard X-ray free-electron lasers *Nature Photonics* **7**, 215 (2013)
N. Hartmann, et al., Sub-femtosecond precision measurement of relative X-ray arrival time for free electron lasers *Nature Photonics* **8**, 706 (2014)
M. R. Bionta, et al., Spectral encoding method for measuring the relative arrival time between x-ray/optical pulses *Rev. Sci. Instrum.* **85**, 083116 (2014)

Contents

1. XFEL sciences
2. Photon beam properties
3. Photon beamline: Optics and diagnostics
4. Experimental stations
5. Experiments at SACLA

Single-shot measurement is mandatory.

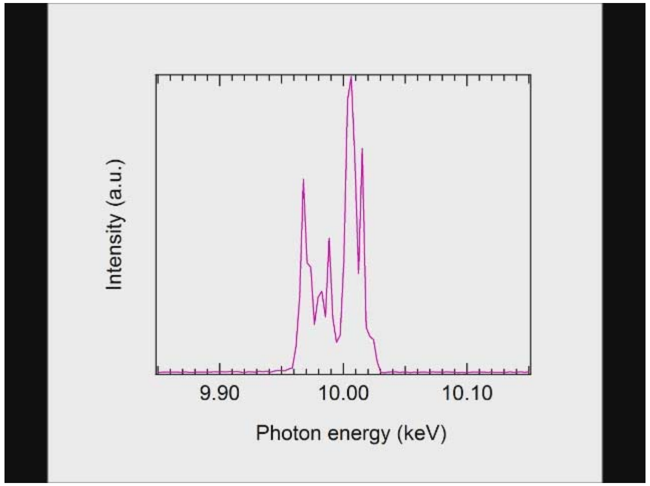
- Even a single pulse destroys a sample.



Neutze et al., Nature 406, 752 (2000)

- Pulse-by-pulse fluctuation of XFEL pulses.

Difficult to repeat measurement in the same condition.

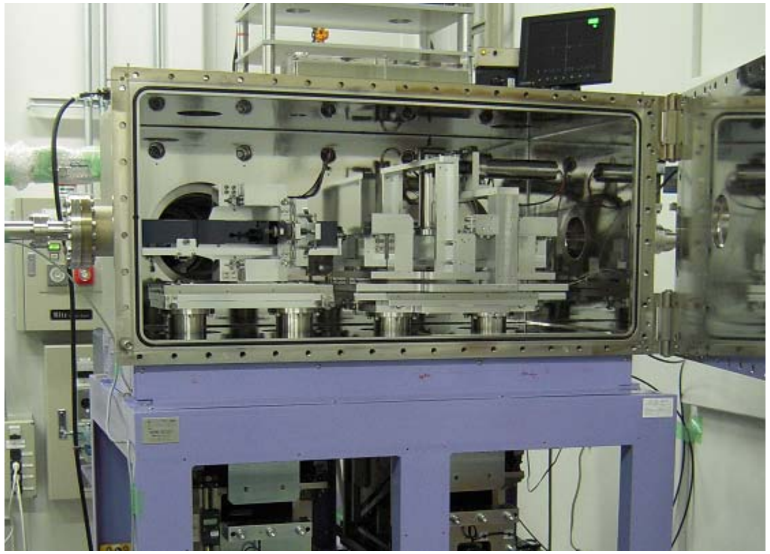


Instrumentation for single-shot measurement

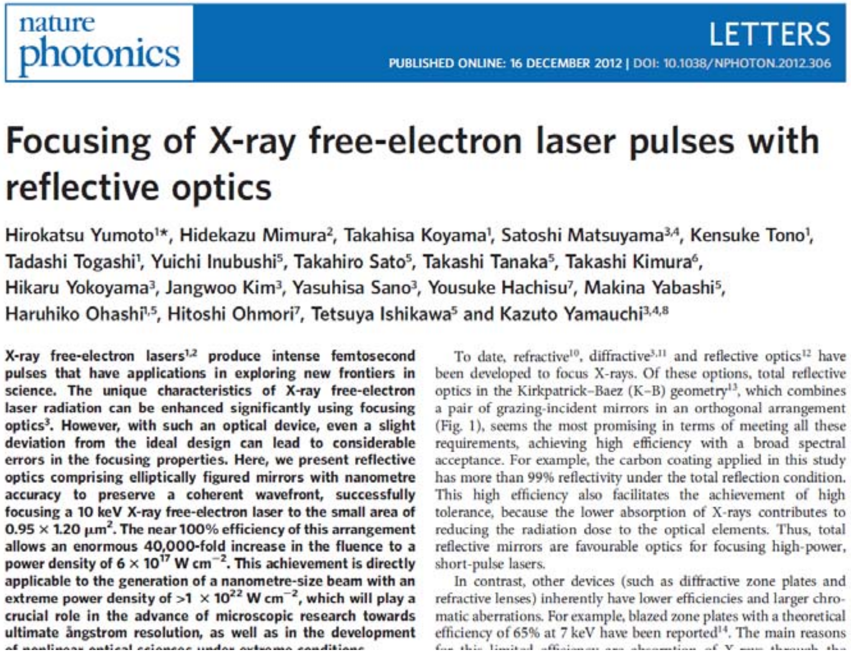
- High photon flux
  - Focusing
- Fast sample exchange
  - Particle injectors
  - Fixed targets with a fast scanning system
- Fast & sensitive X-ray detection
  - High performance detector
- Fast & reliable data acquisition (DAQ)
  - High speed DAQ system with high performance computers & high speed network
  - Large storage system

Focusing

1-um focusing mirrors



Yumoto et al Nature Photon. **7**, 43 (2013)



nature photonics

LETTERS

PUBLISHED ONLINE: 16 DECEMBER 2012 | DOI: 10.1038/NPHOTON.2012.306

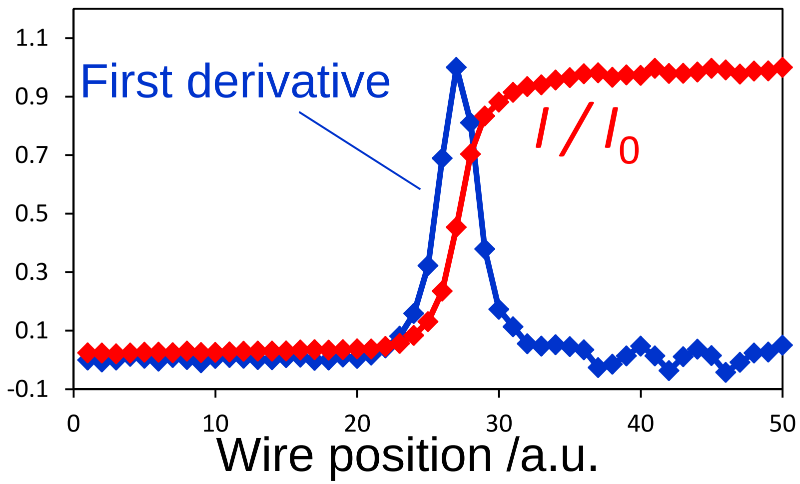
Focusing of X-ray free-electron laser pulses with reflective optics

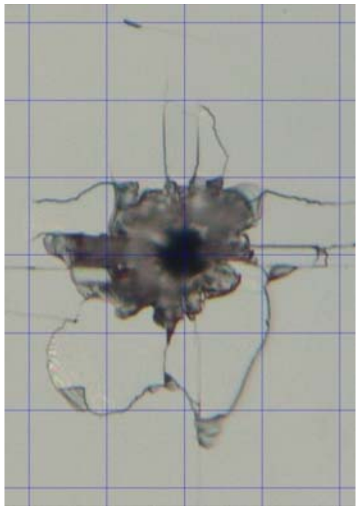
Hirokatsu Yumoto[1*], Hidekazu Mimura[2], Takahisa Koyama[1], Satoshi Matsuyama[3,4], Kensuke Tono[1], Tadashi Togashi[1], Yuichi Inubushi[5], Takahiro Sato[5], Takashi Tanaka[5], Takashi Kimura[6], Hikaru Yokoyama[3], Jangwoo Kim[3], Yasuhisa Sano[3], Yousuke Hachisu[7], Makina Yabashi[5], Haruhiko Ohashi[1,5], Hitoshi Ohmori[7], Tetsuya Ishikawa[5] and Kazuto Yamauchi[3,4,8]

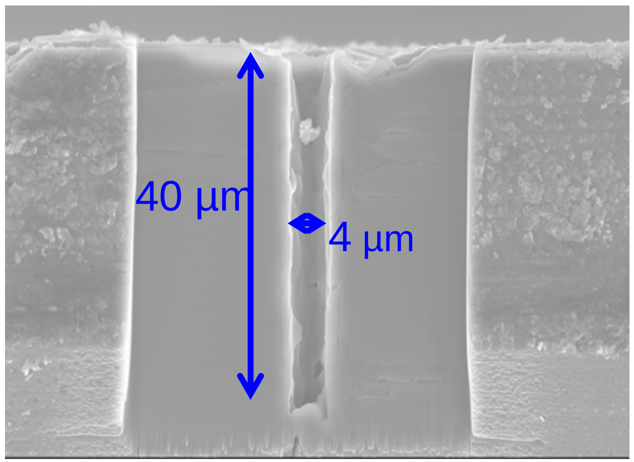
X-ray free-electron lasers[1,2] produce intense femtosecond pulses that have applications in exploring new frontiers in science. The unique characteristics of X-ray free-electron laser radiation can be enhanced significantly using focusing optics[3]. However, with such an optical device, even a slight deviation from the ideal design can lead to considerable errors in the focusing properties. Here, we present reflective optics comprising elliptically figured mirrors with nanometre accuracy to preserve a coherent wavefront, successfully focusing a 10 keV X-ray free-electron laser to the small area of $0.95 \times 1.20\ \mu m^2$. The near 100% efficiency of this arrangement allows an enormous 40,000-fold increase in the fluence to a power density of 6×10^{17} W cm^{-2}. This achievement is directly applicable to the generation of a nanometre-size beam with an extreme power density of $>1 \times 10^{22}$ W cm^{-2}, which will play a crucial role in the advance of microscopic research towards ultimate ångstrom resolution, as well as in the development

To date, refractive[10], diffractive[3,11] and reflective optics[12] have been developed to focus X-rays. Of these options, total reflective optics in the Kirkpatrick–Baez (K–B) geometry[13], which combines a pair of grazing-incident mirrors in an orthogonal arrangement (Fig. 1), seems the most promising in terms of meeting all these requirements, achieving high efficiency with a broad spectral acceptance. For example, the carbon coating applied in this study has more than 99% reflectivity under the total reflection condition. This high efficiency also facilitates the achievement of high tolerance, because the lower absorption of X-rays contributes to reducing the radiation dose to the optical elements. Thus, total reflective mirrors are favourable optics for focusing high-power, short-pulse lasers.

In contrast, other devices (such as diffractive zone plates and refractive lenses) inherently have lower efficiencies and larger chromatic aberrations. For example, blazed zone plates with a theoretical efficiency of 65% at 7 keV have been reported[14]. The main reasons

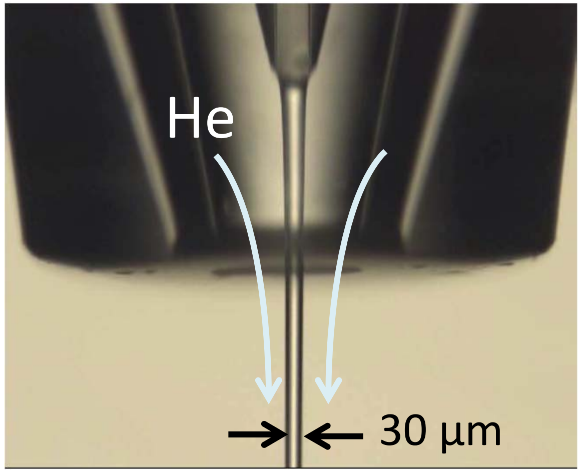
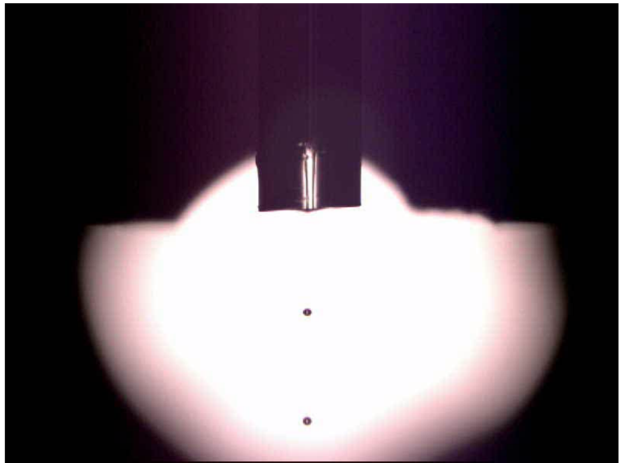






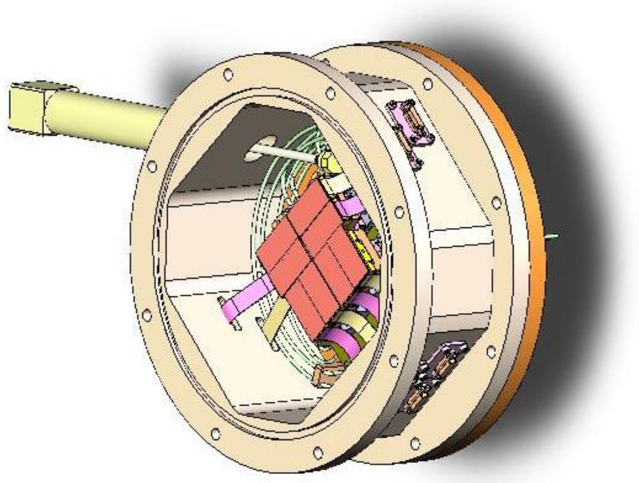
Koyama et al, Opt. Exp. **21**, 15382 (2013)

Particle injectors

Continuous beam	High-viscosity sample	Droplets
		
Flow rate = ~ 0.4 mL/min	~0.5 µL/min	~0.1 µL/min
Proteins : ~100 mg	Proteins : ~0. 1 mg	Proteins : <0.1 mg

High-performance detector

- Multi-port CCD (MPCCD)
  - High sensitivity
  - Low noise
    - (single-photon detection capability)
  - Fast (60 fps)
  - Large area (□100 mm)



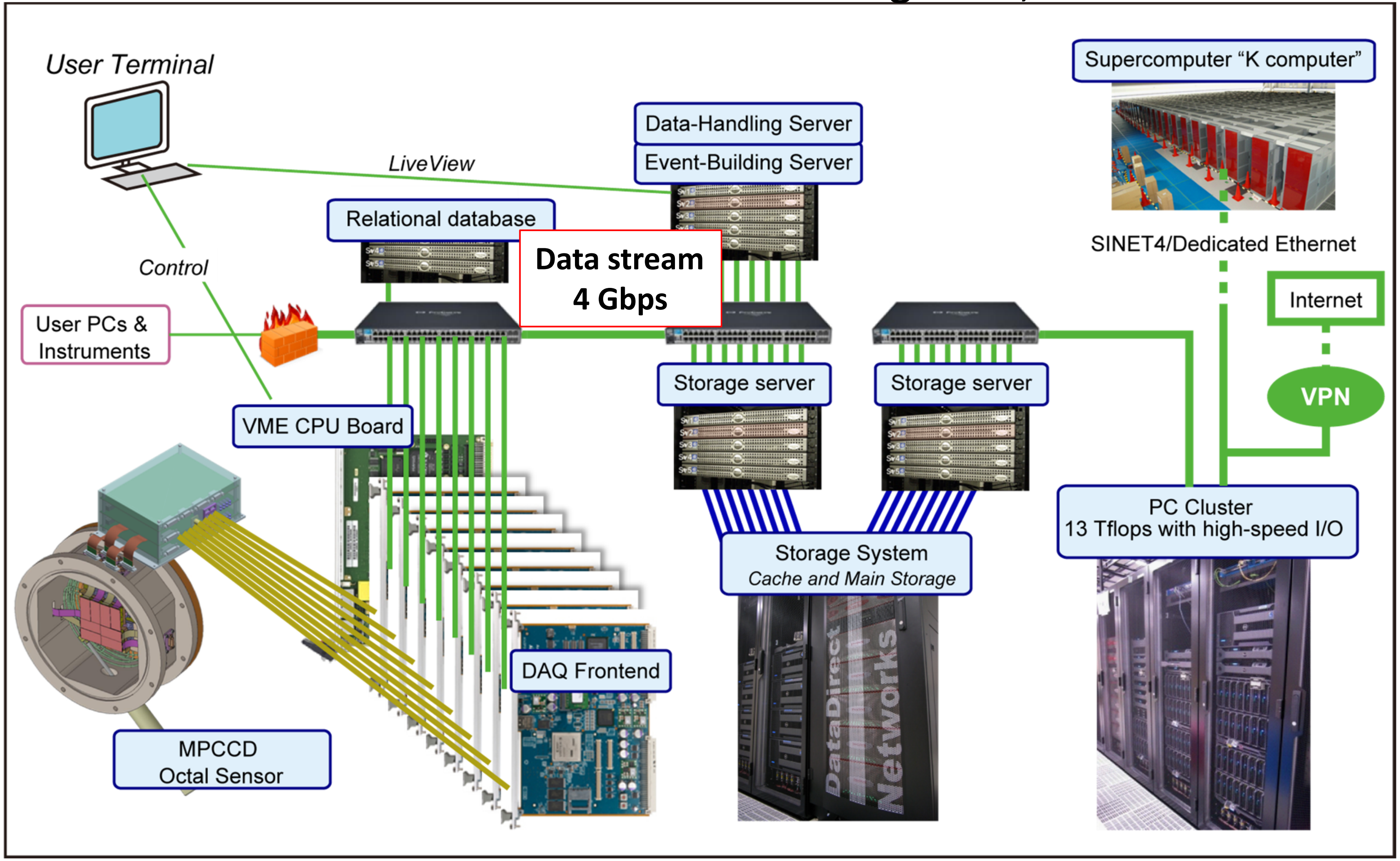
Octal Sensor Detector (100 x 100 mm)
2048 x 2048 pixels

Kameshima , Hatsui et al., Rev. Sci. Instrum. 85 (2014)

Specification	
Frame rate	≥60 fps
Pixel size	50 μm
Noise	300e$^-$
Q.E.	~70 % @ 6 keV ~20 % @ 12 keV
Dynamic range	14 bits
System noise	< 0.2 ph.@ 6 keV
Full well	~ 3000 ph. @6keV ~ 1500 ph. @12keV

Data acquisition (DAQ) system

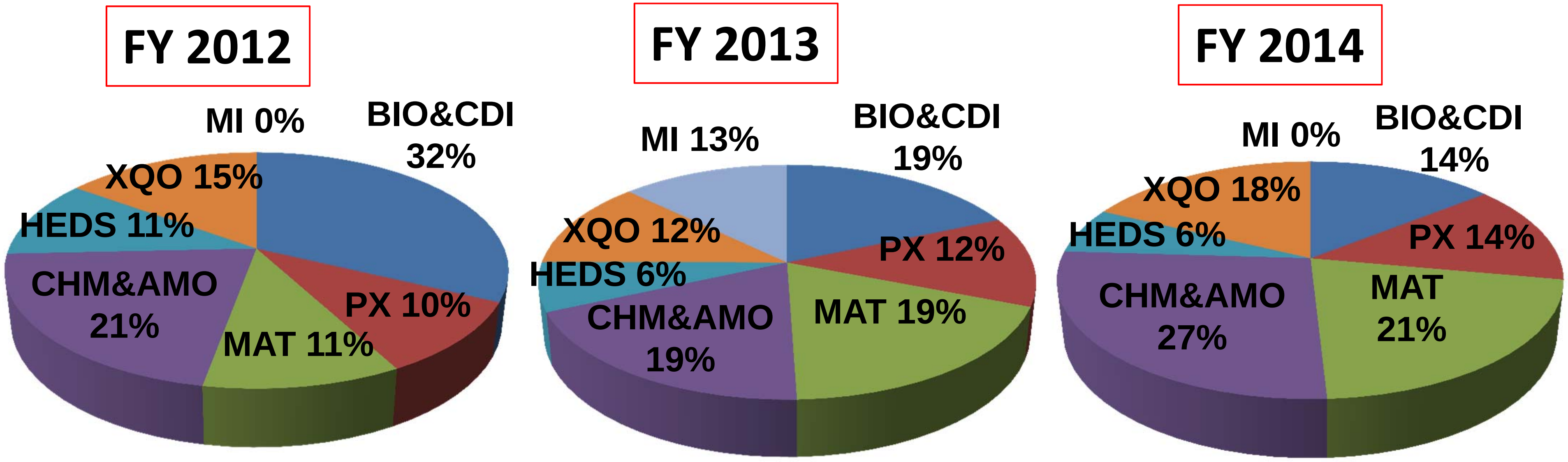
Joti-san, Kameshima-san,
Yamaga-san, Hatsui-san et al.



Contents

1. XFEL sciences
2. Photon beam properties
3. Photon beamline: Optics and diagnostics
4. Experimental stations
5. Experiments at SACLA
   – Coherent diffraction imaging (CDI)
   – Femtosecond protein crystallography
   – Time-resolved X-ray absorption spectroscopy (XAS)
   – Nonlinear X-ray optics

Variety of research fields at SACLA



BIO:	Imaging biology
CDI:	Coherent diffraction imaging
PX:	Protein crystallography
MAT:	Ultrafast materials science
CHM:	Ultrafast chemistry
AMO:	AMO science
HEDS:	High energy density science
XQO:	X-ray quantum optics
MI:	Methods and instrumentation

~50 proposals accepted in a year (acceptance ratio ~50%)

XFEL as a probe, as a trigger

- Observation in a “diffraction-before-destruction” scheme.
  - Coherent diffraction imaging (CDI)
  - Femtosecond protein crystallography (PX)
- Observation of ultrafast phenomena
  - Time-resolved X-ray spectroscopy
- Light-matter interaction under intense X-ray irradiation: XFEL as a trigger of novel optical phenomena
  - Nonlinear X-ray optics, X-ray amplification

“Diffraction before destruction” (1)
CDI for ***single-particle*** structure analysis

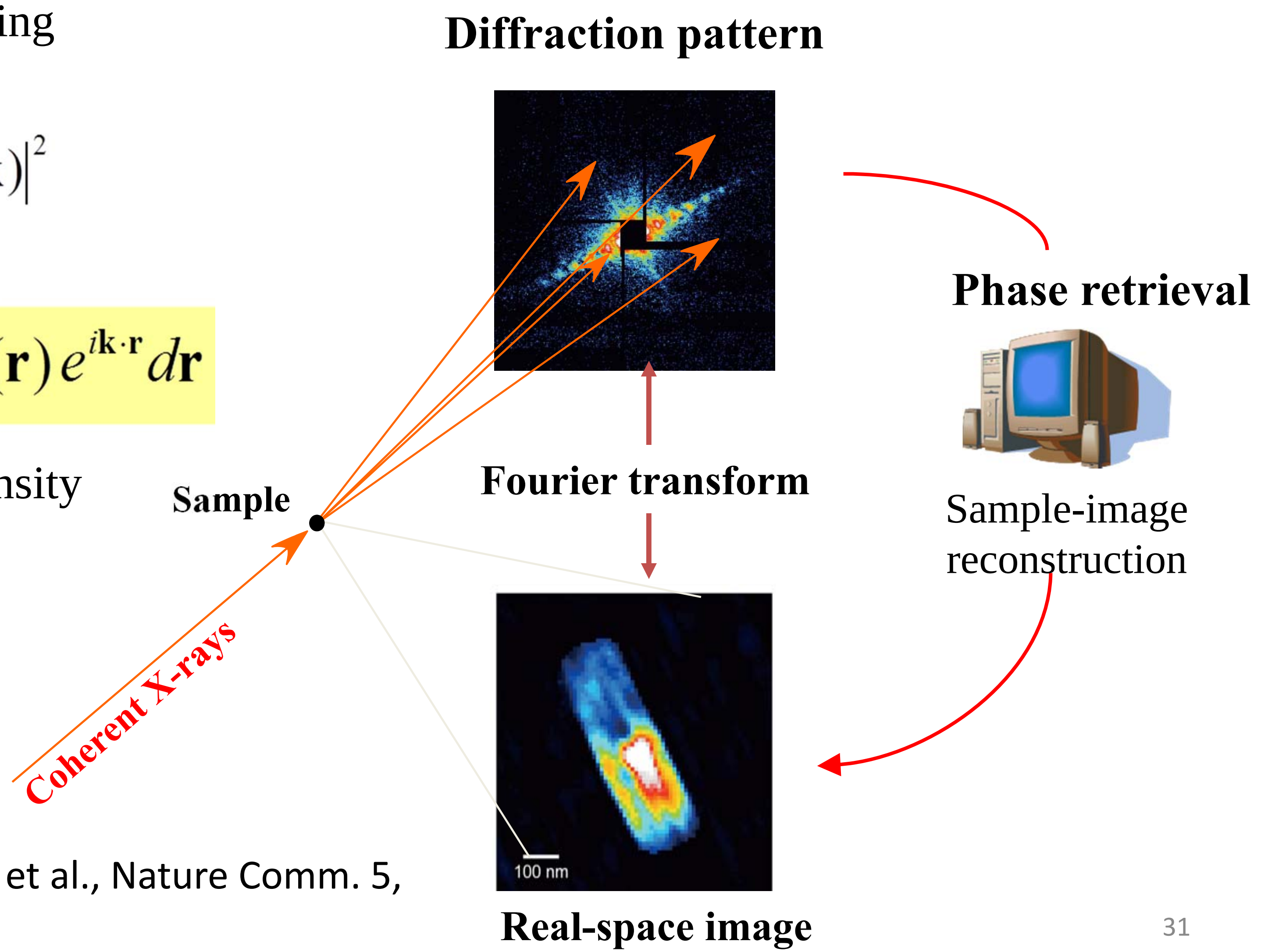
Differential scattering cross section

$$\frac{d\sigma}{d\Omega} = P r_e^2 \left| F(\mathbf{k}) \right|^2$$

Structure factor

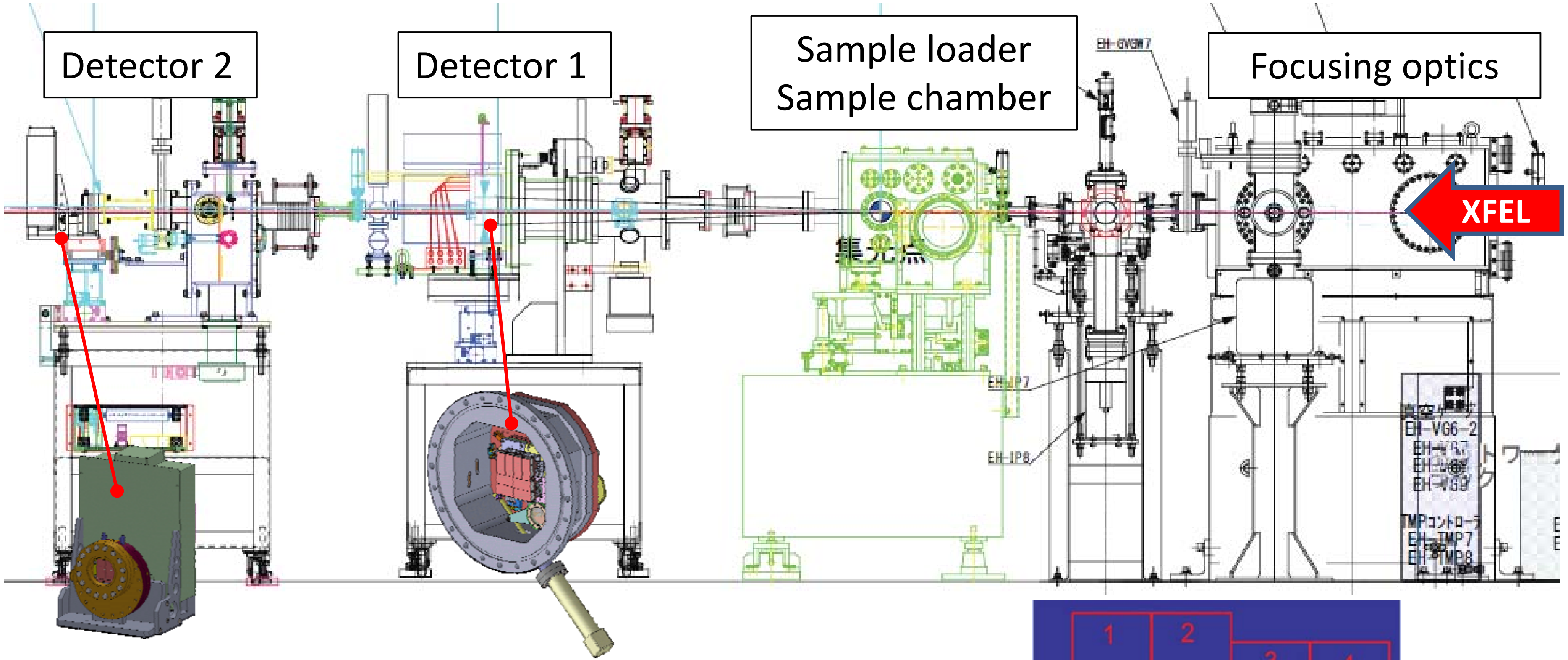
$$F(\mathbf{k}) = \int \rho(\mathbf{r}) e^{i\mathbf{k}\cdot\mathbf{r}} d\mathbf{r}$$

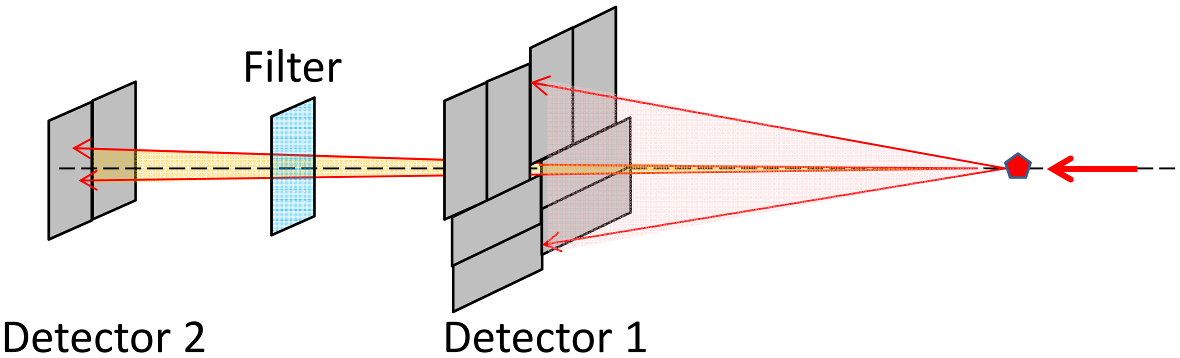
$\rho(\mathbf{r})$: Electron density

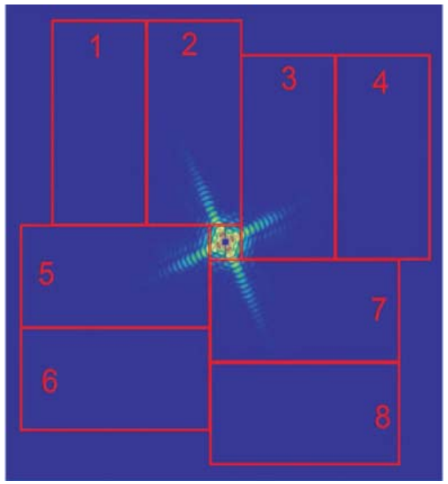


Images from Kimura et al., Nature Comm. 5, 3052 (2013).

Typical setup for CDI



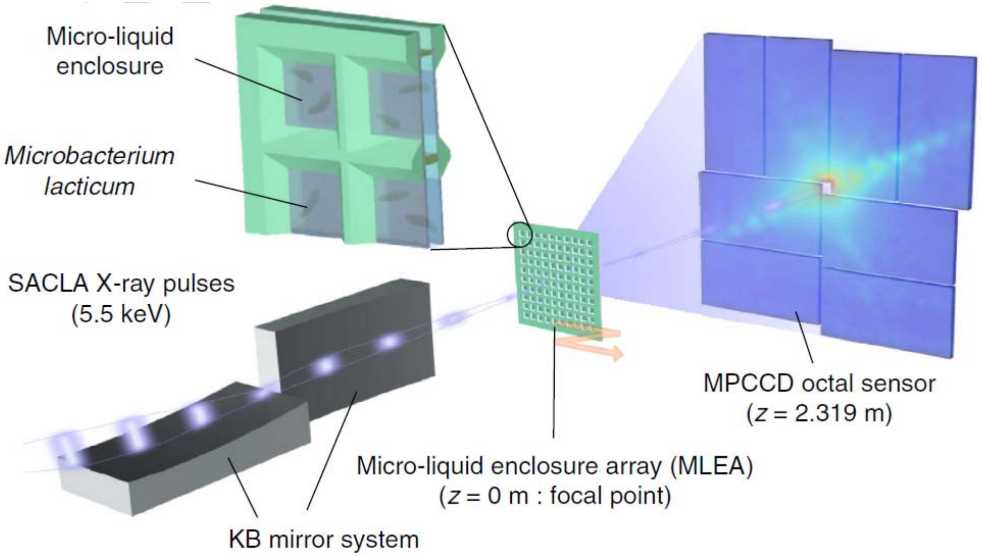


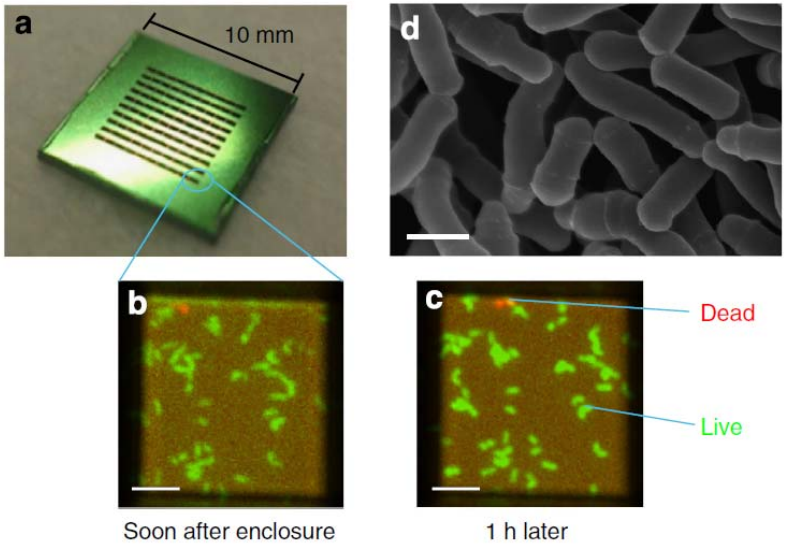


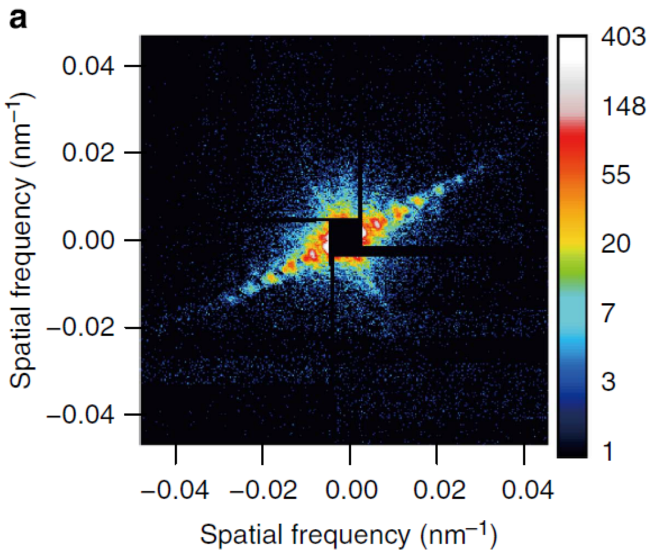
Kameshima et al., Rev. Sci. Instrum **85**, 033110 (2014)

CDI of live cell

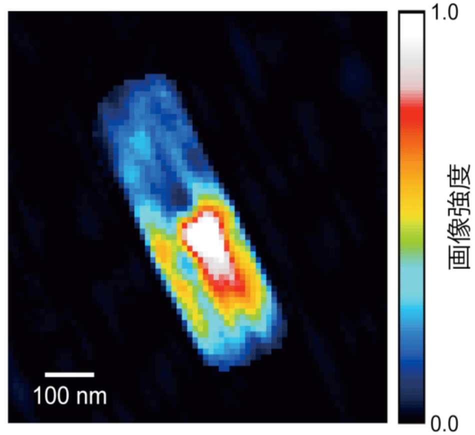
Kimura et al., *Nature Communications* **5**, 3052 (2013).





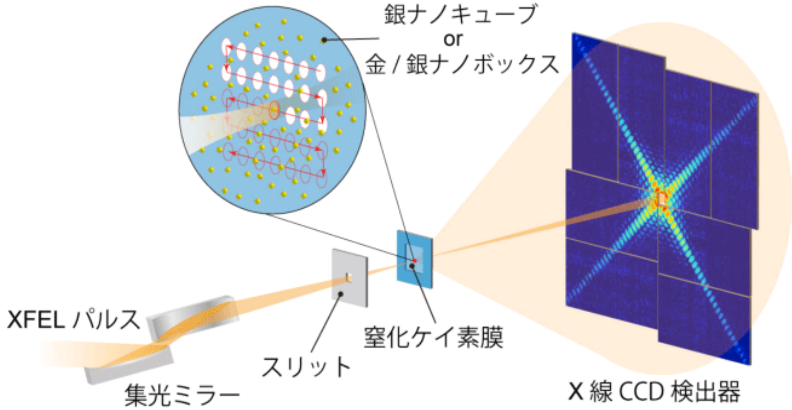


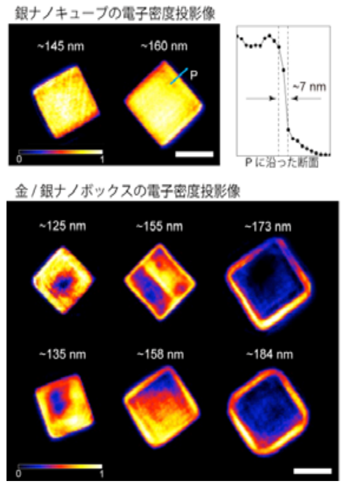
XFELで観察した生きている細胞の画像



CDI of nanomaterials

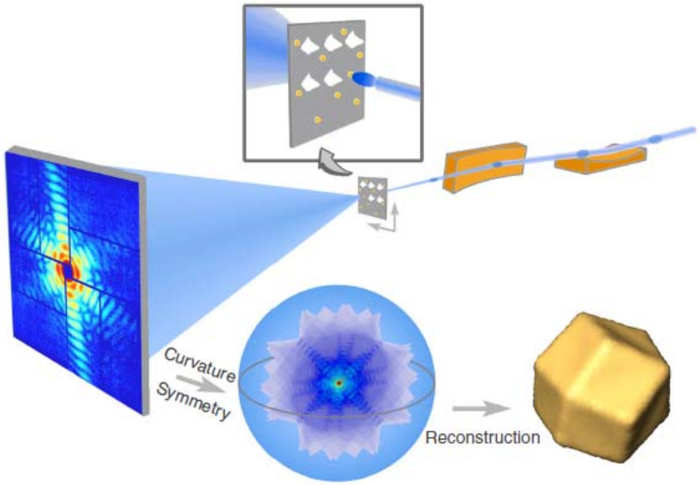
Metal nano-cubes [Takahashi et al., *Nano Lett.* (2013)]





Resolution ~7 nm

3D structure of gold nanocrystals [R. Xu et al., *Nat. Comm.* (2014)]



Resolution ~5.5 nm

“Diffraction before destruction” (2)
Femtosecond protein crystallography

- Damage free
  - Room temperature measurement
- Dynamics
  - Pump-probe capability
- Two major methods
  - For large, high-quality crystals
  - For small crystals

Femtosecond crystallography

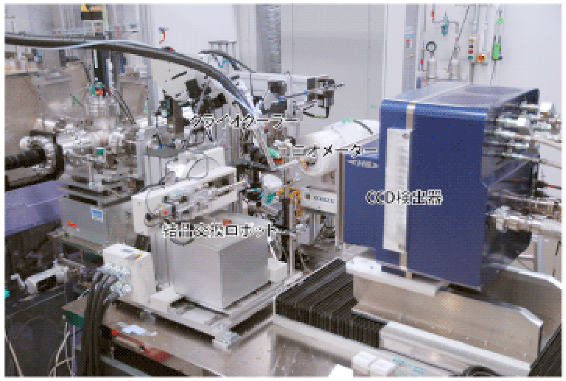
NATURE METHODS | VOL.11 NO.7 | JULY 2014 | 735

Determination of damage-free crystal structure of an X-ray–sensitive protein using an XFEL

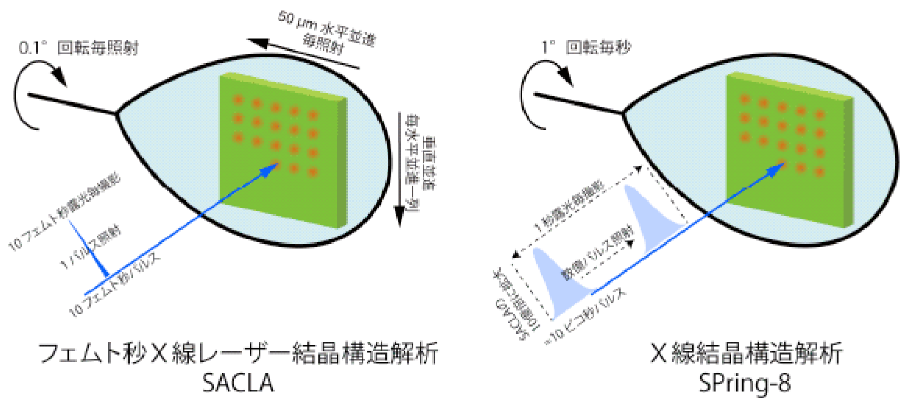
Kunio Hirata[1,2,9], Kyoko Shinzawa-Itoh[3,9], Naomine Yano[2,3], Shuhei Takemura[3], Koji Kato[3,8], Miki Hatanaka[3], Kazumasa Muramoto[3], Takako Kawahara[3], Tomitake Tsukihara[2–4], Eiki Yamashita[4], Kensuke Tono[5], Go Ueno[1], Takaaki Hikima[1], Hironori Murakami[1], Yuichi Inubushi[1], Makina Yabashi[1], Tetsuya Ishikawa[1], Masaki Yamamoto[1], Takashi Ogura[6], Hiroshi Sugimoto[1], Jian-Ren Shen[7], Shinya Yoshikawa[3] & Hideo Ago[1]

Hirata et al., *Nature Methods* **7**, 735 (2014).

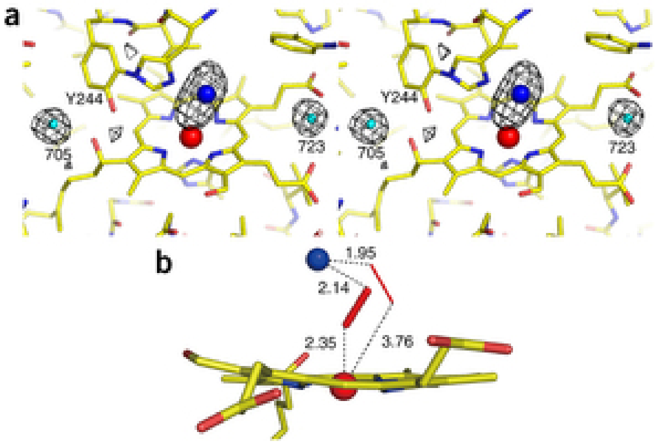
a



b

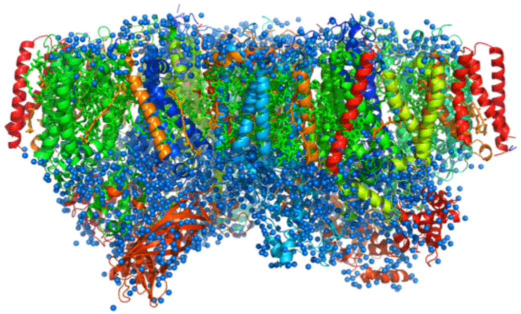


Damage-free structure

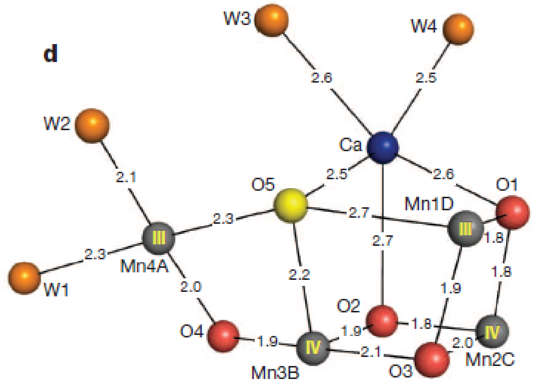


Damage-free structure of photosystem II

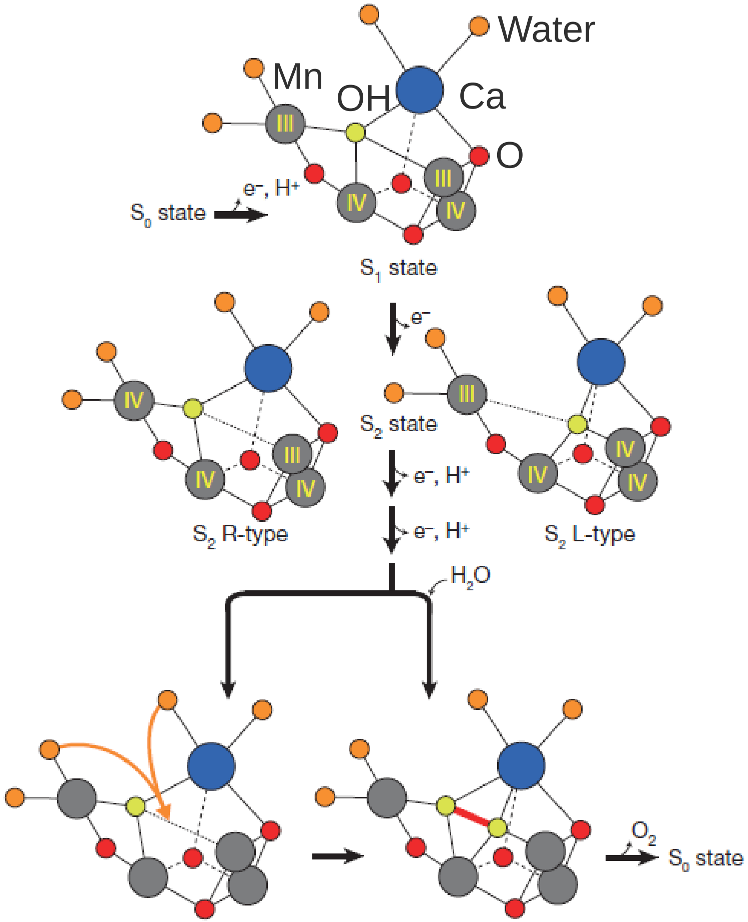
Structure of photosystem II



oxygen evolving complex where oxidation of water into dioxygen occurs.

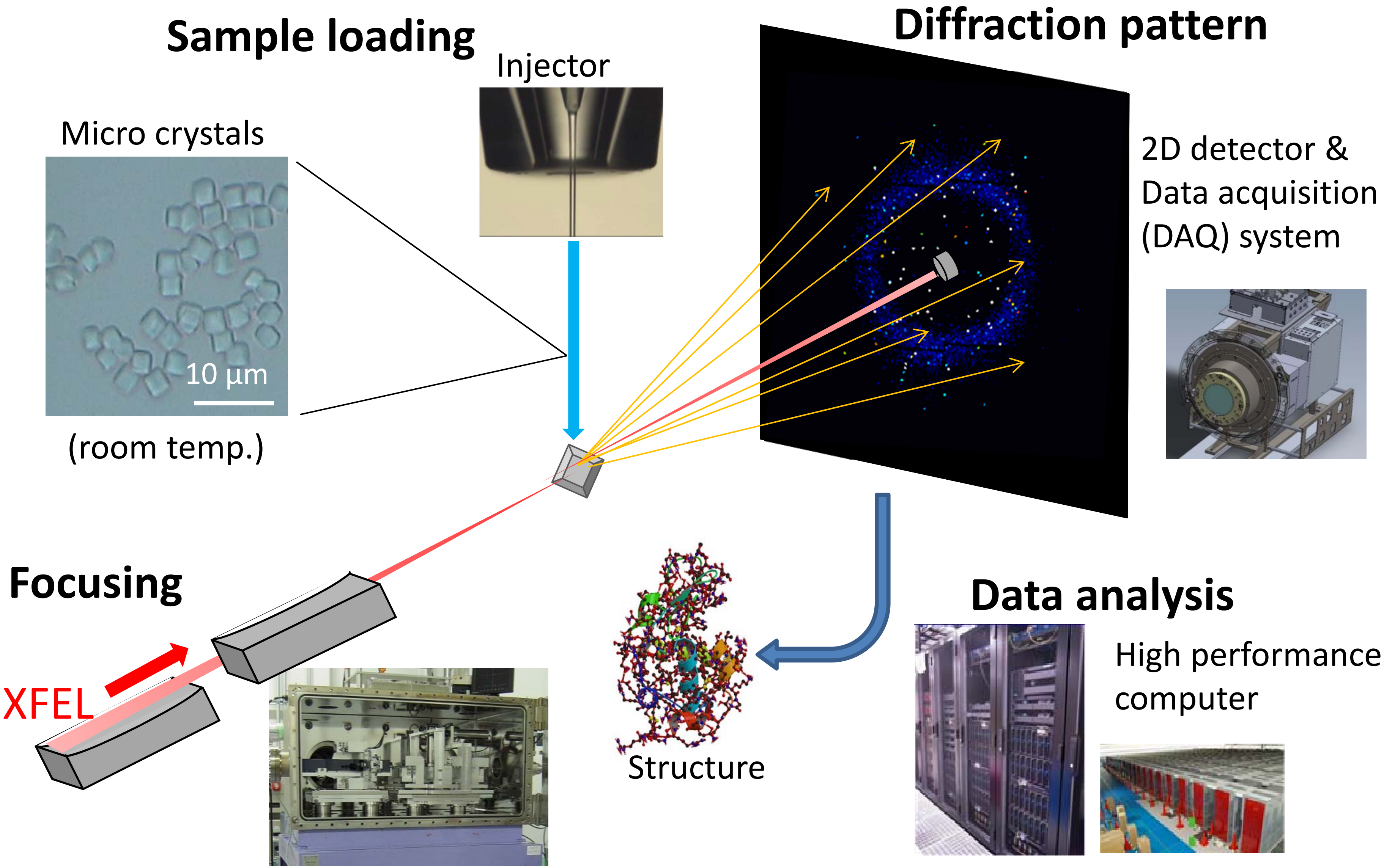


Possible mechanism for the oxygen evolving reaction.

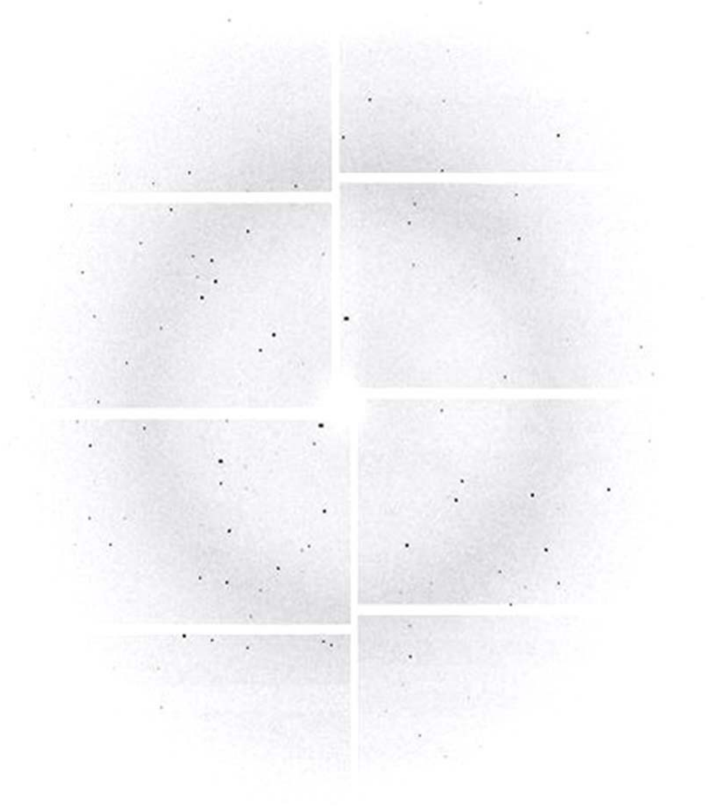


Suga, Shen *et al.*, ***Nature*** (2015)

Serial femtosecond crystallography (SFX)

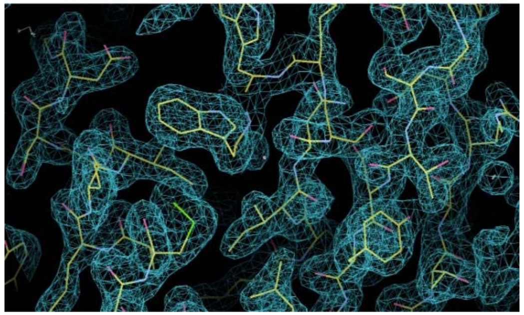


Single-shot diffraction patterns of Lysozyme



Lysozyme
（Average crystal size: ~5 μm）

Electron density map



Resolution <2 Å

Statistics

Shot number: 70,000

Number of Images with diffraction spots : 21723 (Hit rate： 31%)

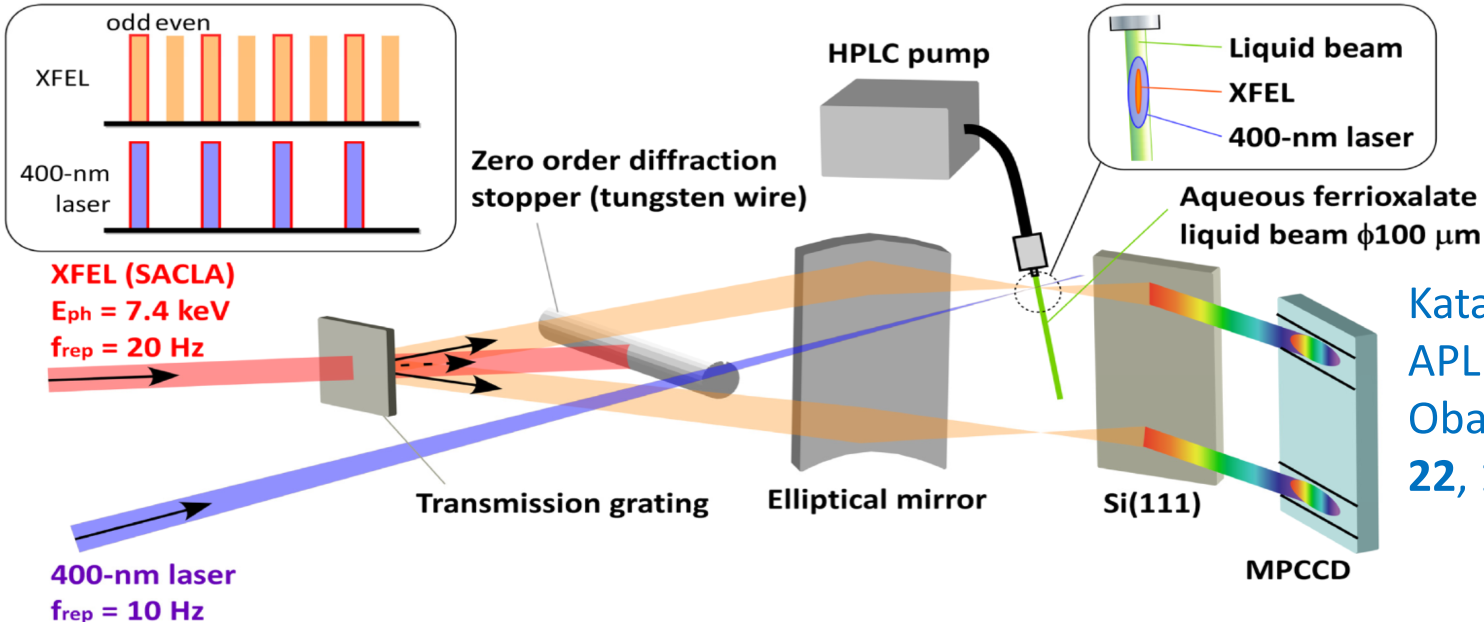
Indexable images: 13,912 (20%)

Measurement time: 1 hour (20 Hz)

Time-resolved measurements for probing ultrafast phenomena

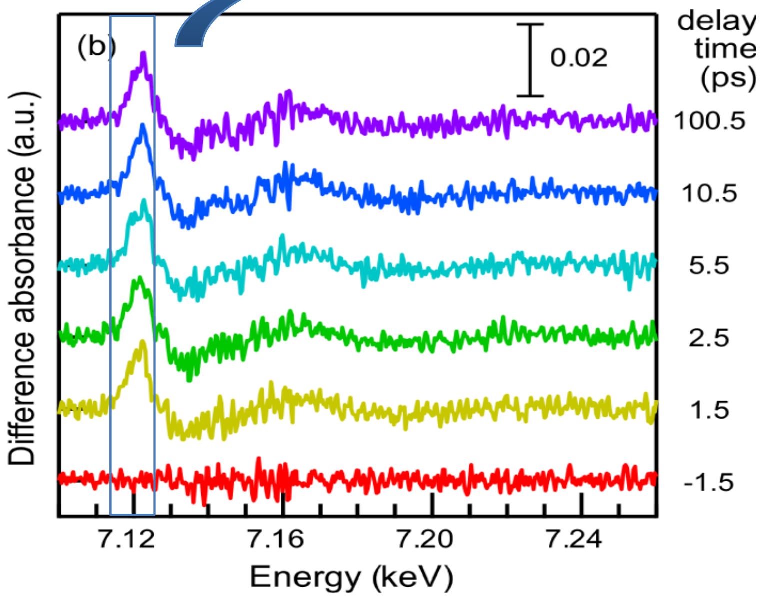
- Time-resolved X-ray absorption/emission spectroscopy (XAS/XES)
- Time-resolved X-ray diffraction/scattering
- Time-resolved photoelectron spectroscopy
- Ultrafast probe for high energy density sciences
  - Laser shock compression of materials
  - Ultrafast probe of plasma

Time-resolved XAS for ultrafast chemistry

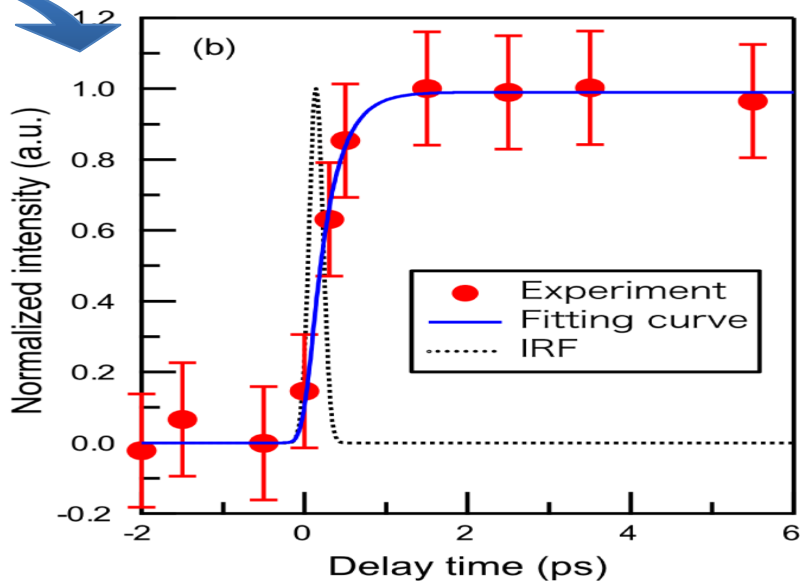


Katayama et al, APL **103**, 131105
Obara et al, OE **22**, 1105 (2014)

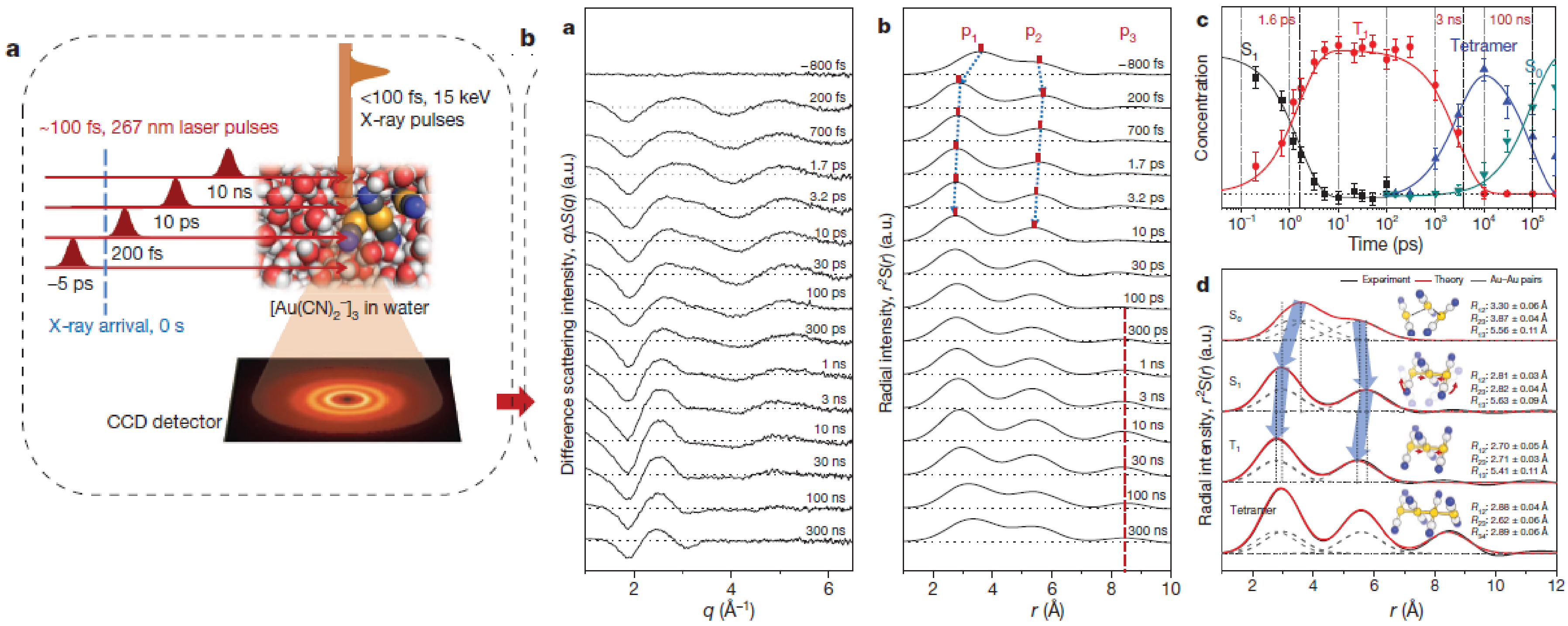
Differential spectrum



Temporal evolution



Time-resolved wide-angle X-ray scattering (WAXS) for tracing ultrafast structural change



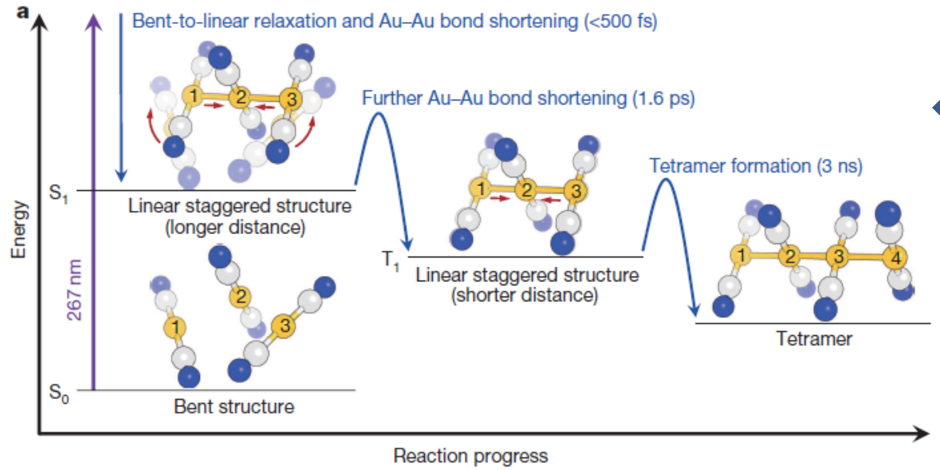
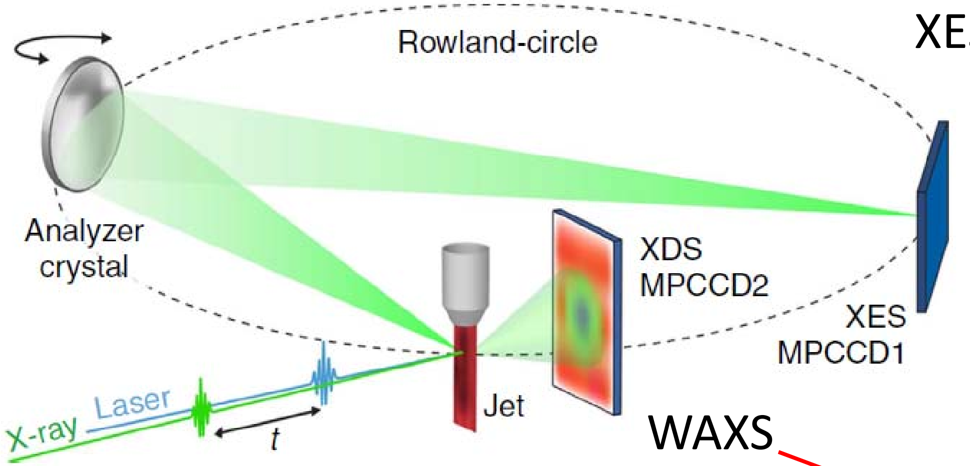


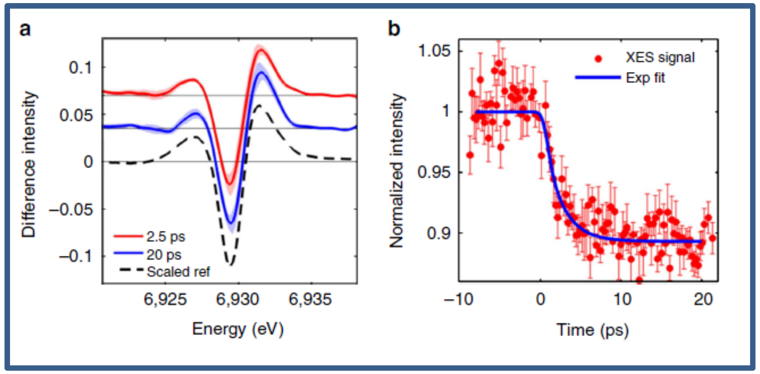
Photo-excited species and timescales of structural changes.

Kim, Ihee, Adachi, et al., ***Nature*** (2015).

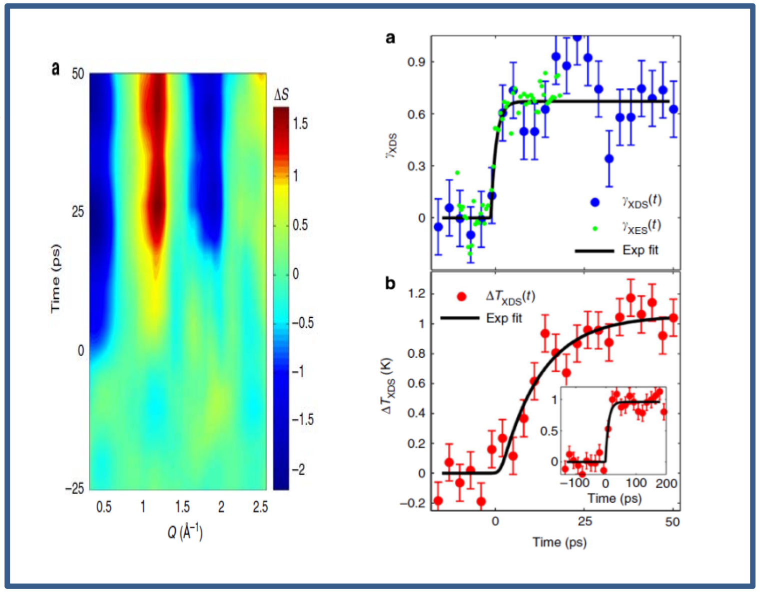
Simultaneous measurement of time-resolved X-ray emission spectroscopy (XES) and WAXS



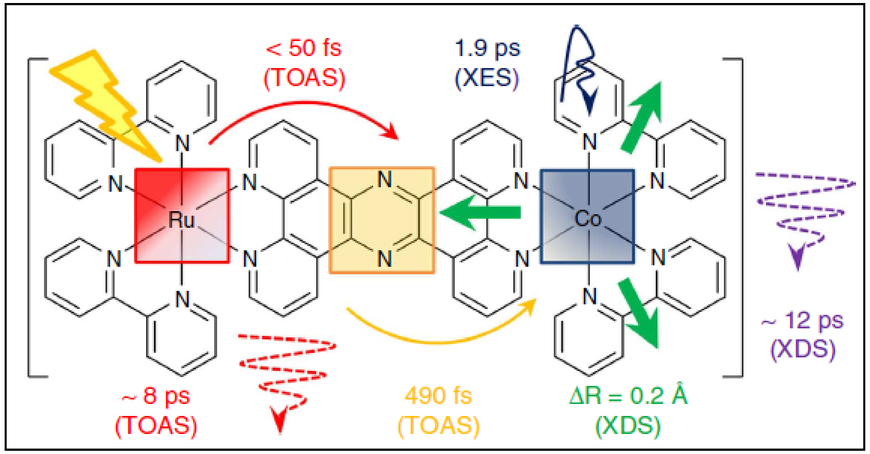
XES



WAXS



Bimetallic donor–acceptor complex: Model system for artificial photosynthesis.



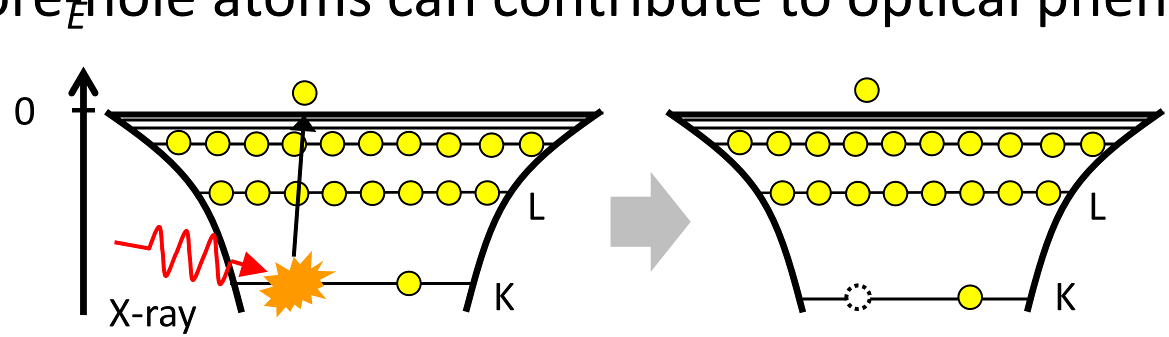
Light-matter interaction under intense X-ray irradiation

- Nonlinear phenomena via interaction with intense XFEL
  - Double core-hole generation
  - Two-photon absorption
  - Saturable absorption
  - Amplification of x-ray pulse

Nonlinear phenomena via interaction with intense XFEL

Intense XFEL pulse interacts with atoms within a time scale comparable to a core-hole lifetime.

- *Multi-photons* can be involved.
- Core-hole atoms can contribute to optical phenomena.



Lifetime: fs ~ as

<u>Nonlinear phenomena associated with core-hole atoms</u>

- Double core-hole generation
- Two-photon absorption
- Saturable absorption
- Amplification of x-ray pulse

To obtain enough XFEL intensity for nonlinear phenomena

XFEL intensity (irradiance)

$$I = \frac{E}{S \cdot \tau} \quad (\mathrm{W/cm^2})$$

Pulse energy

$\sim 10^{-4}$ J

Focal spot

$\sim 10^{-10}$ $\mathrm{cm^2}$ (= 100 nm x 100 nm)

Pulse duration

$\sim 10^{-14}$ s

Focusing XFEL down to < 100 nm, an intensity reaches 10^{20} $\mathrm{W/cm^2}$

High intensity application

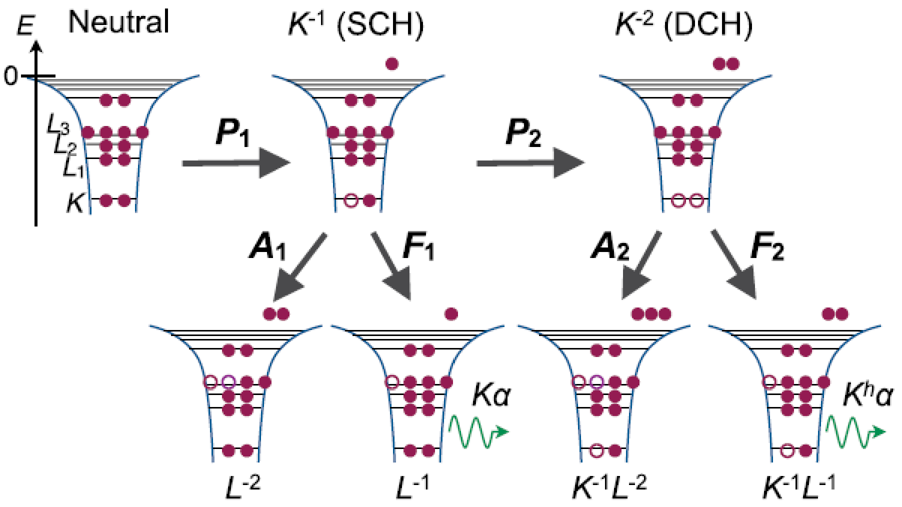
K. Tamasaku et al, PRL Vol.111 (2013)

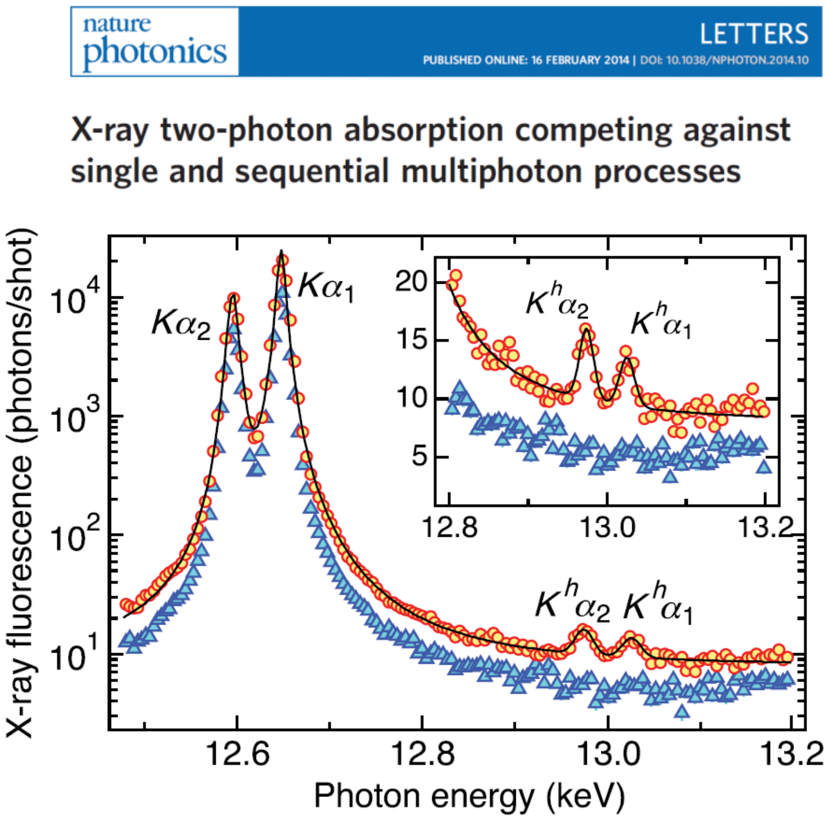
Emission from double core hole atoms

1 μm focusing

- 100 uJ/10 fs = 10 GW (after 1-μm KB)
- Focusing size: ~1x1 μm^2
- 10 GW/$(1\ \mu m)^2$~ 10^{18} W/cm^2

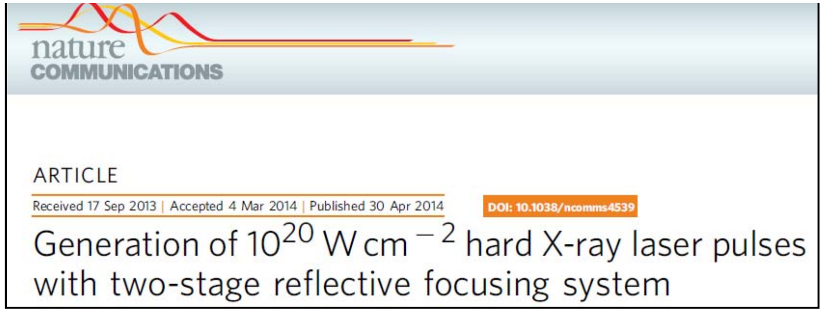
Double core hole of Kr





2-stage focusing for creating nanometer spot

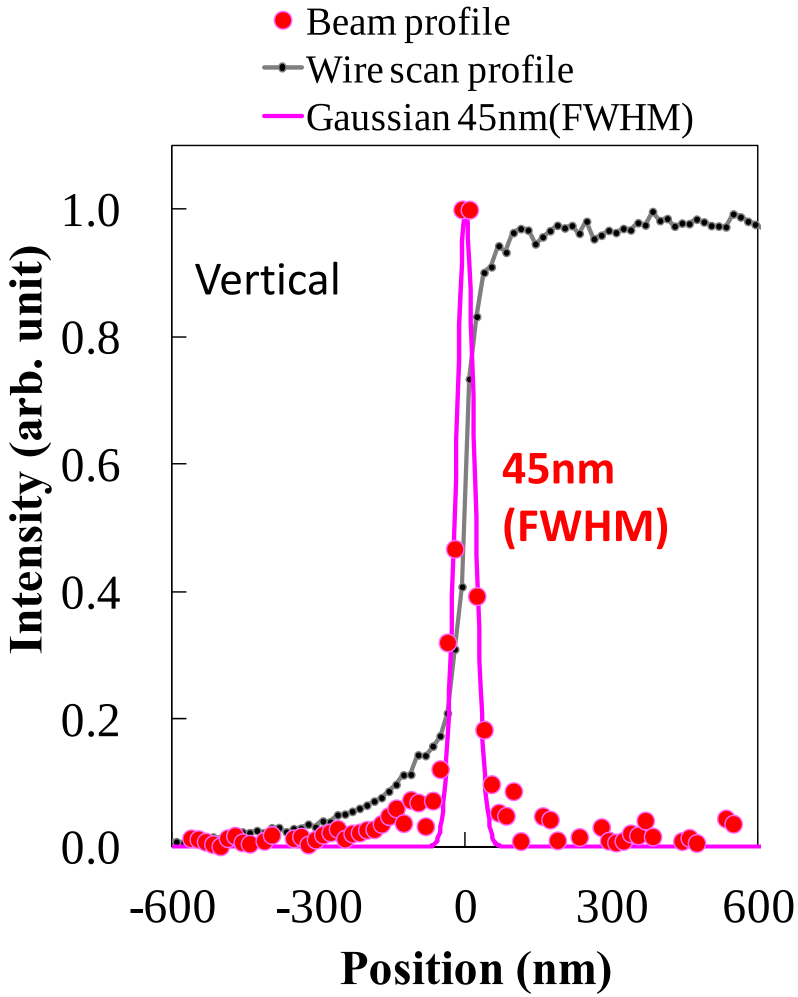
Mimura et al,
Nature Comm., DOI: 10.1038/ncomms 4539 (2014)

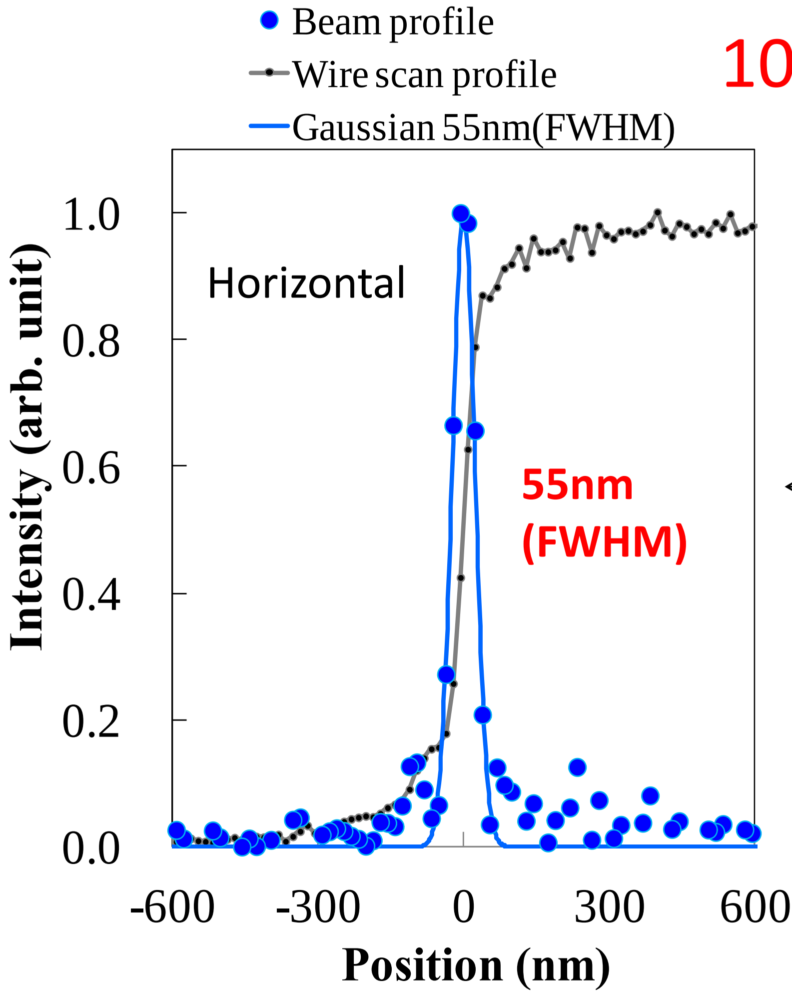

nature COMMUNICATIONS

ARTICLE

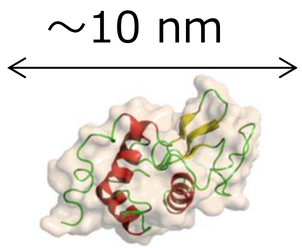
Received 17 Sep 2013 | Accepted 4 Mar 2014 | Published 30 Apr 2014 DOI: 10.1038/ncomms4539

Generation of 10^{20} W cm $^{-2}$ hard X-ray laser pulses with two-stage reflective focusing system





10^{20} W/cm^2 @ 10 keV



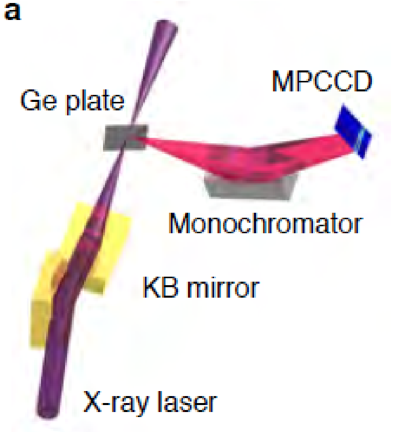
Protein

X-ray nonlinear optics: two-photon absorption

50 nm focusing ⇒ ~10^{20} W/cm^2

Tamasaku et al., *Nature Photon.* (2014)

a



nature photonics

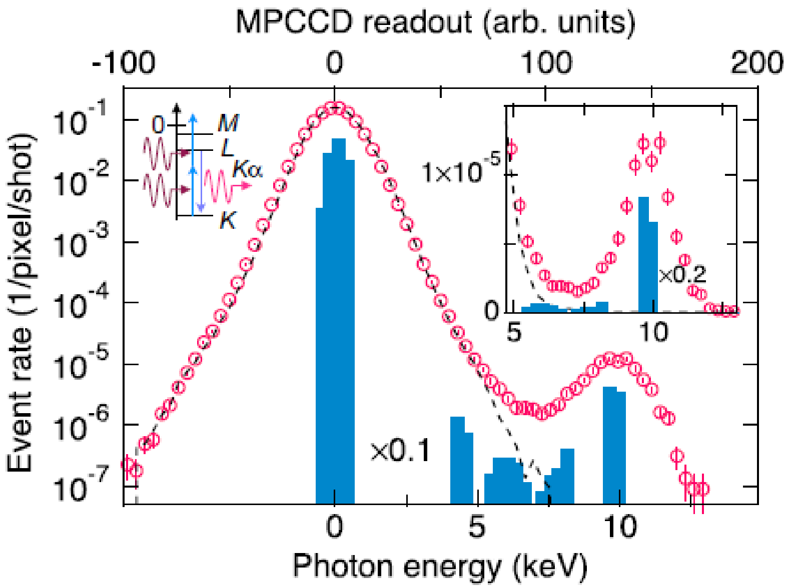
LETTERS

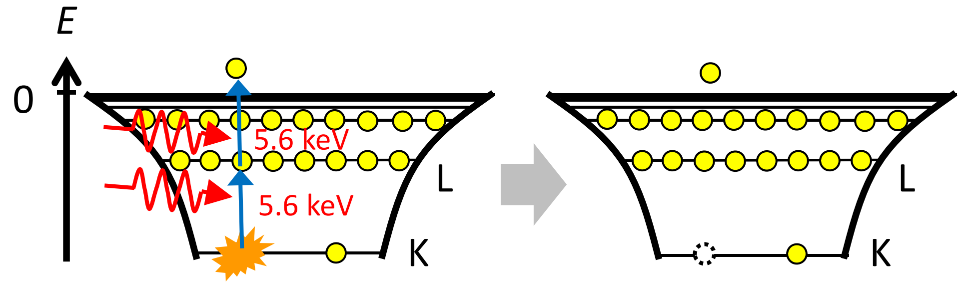
PUBLISHED ONLINE: 16 FEBRUARY 2014 | DOI: 10.1038/NPHOTON.2014.10

X-ray two-photon absorption competing against single and sequential multiphoton processes

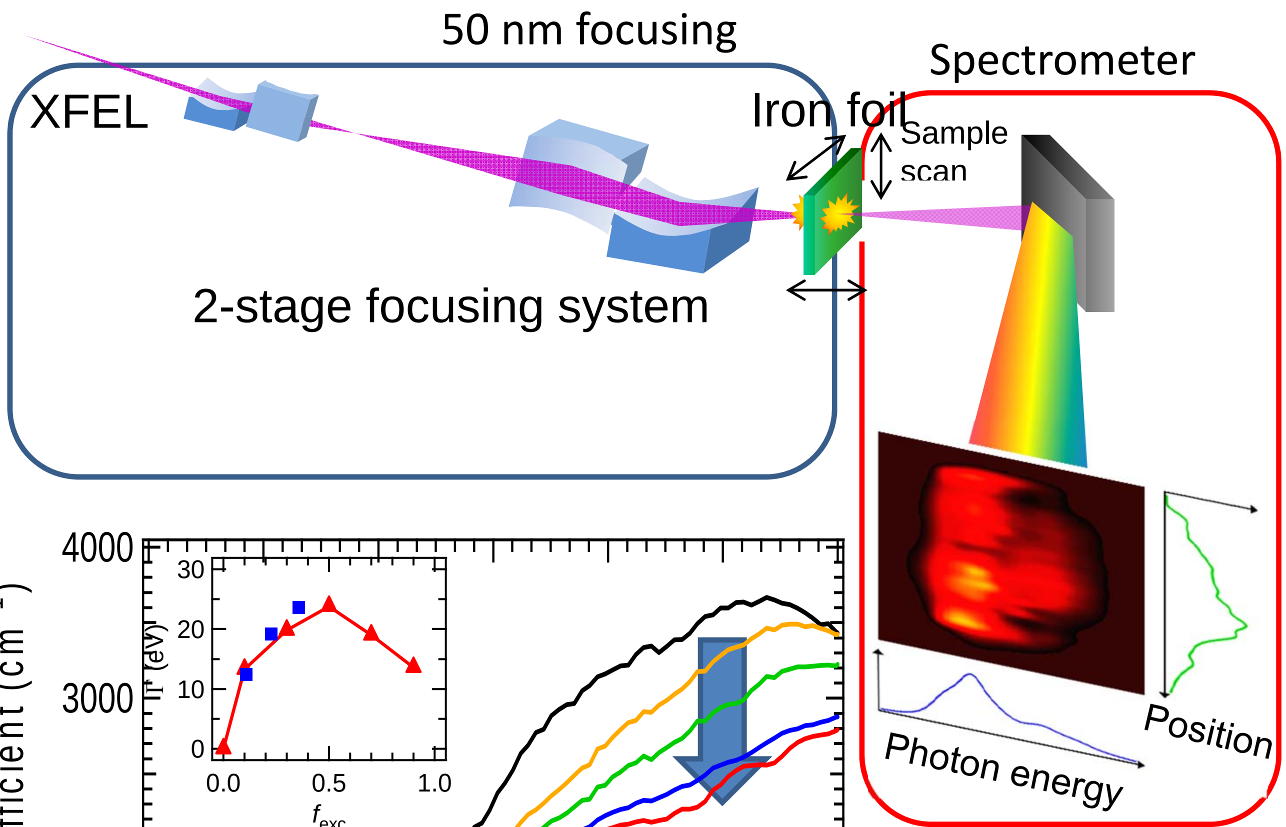
K-shell core-hole of Ge (absorption edge: 11. 1 keV) is created by absorption of two 5.6-keV photons

c

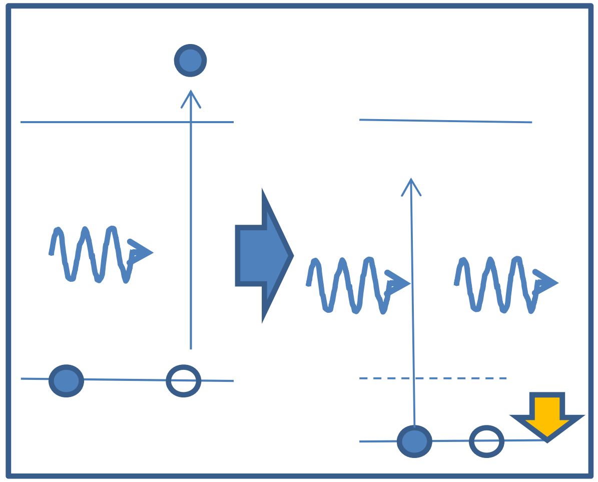


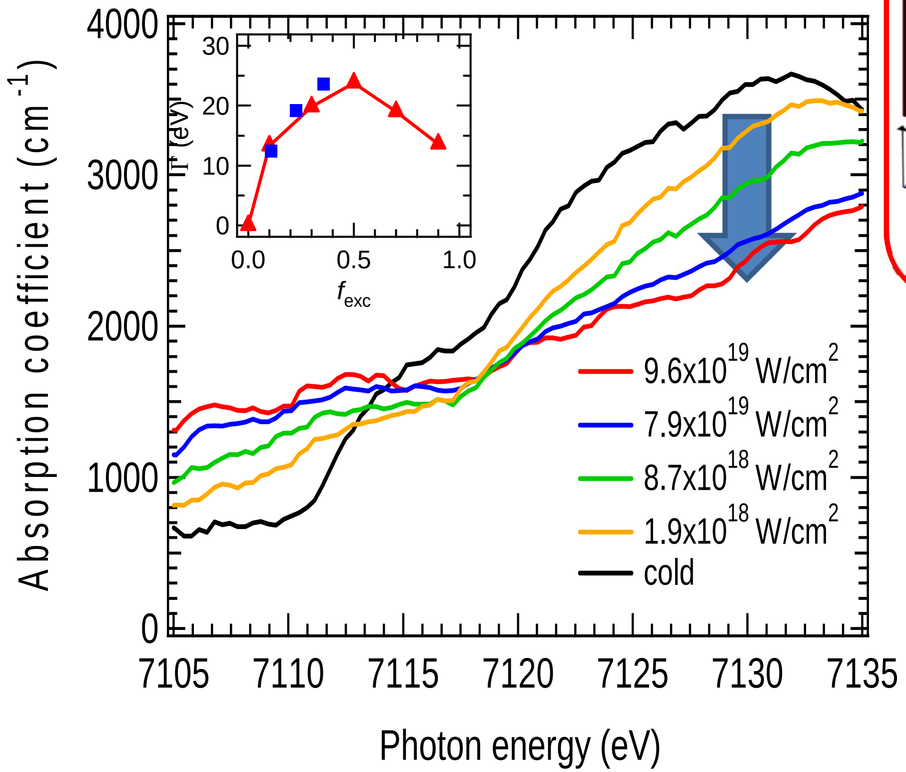


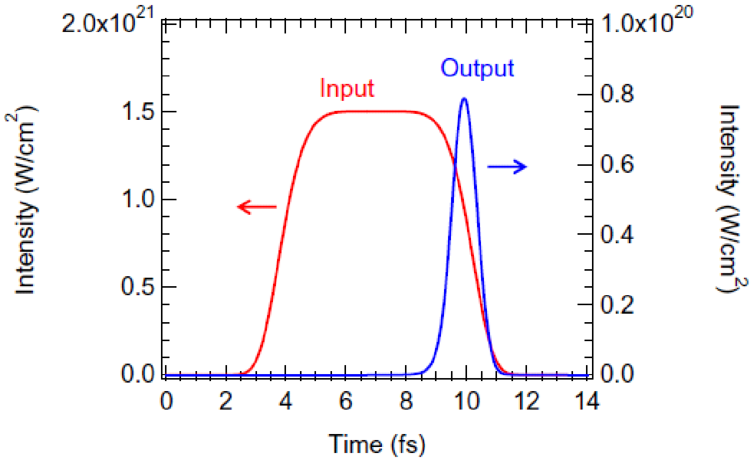
Saturable absorption at Iron K-edge



Yoneda, Inubushi, et al.,
Nat. Comm. (2014).





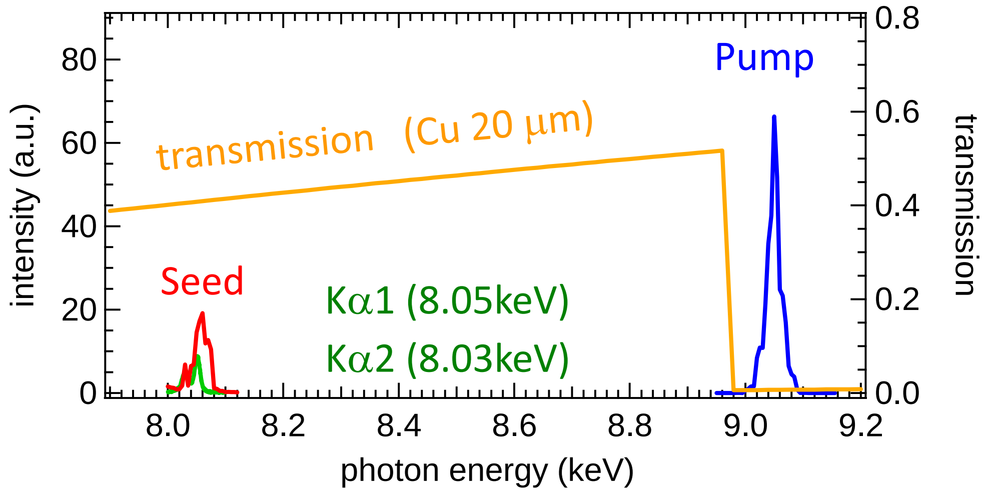


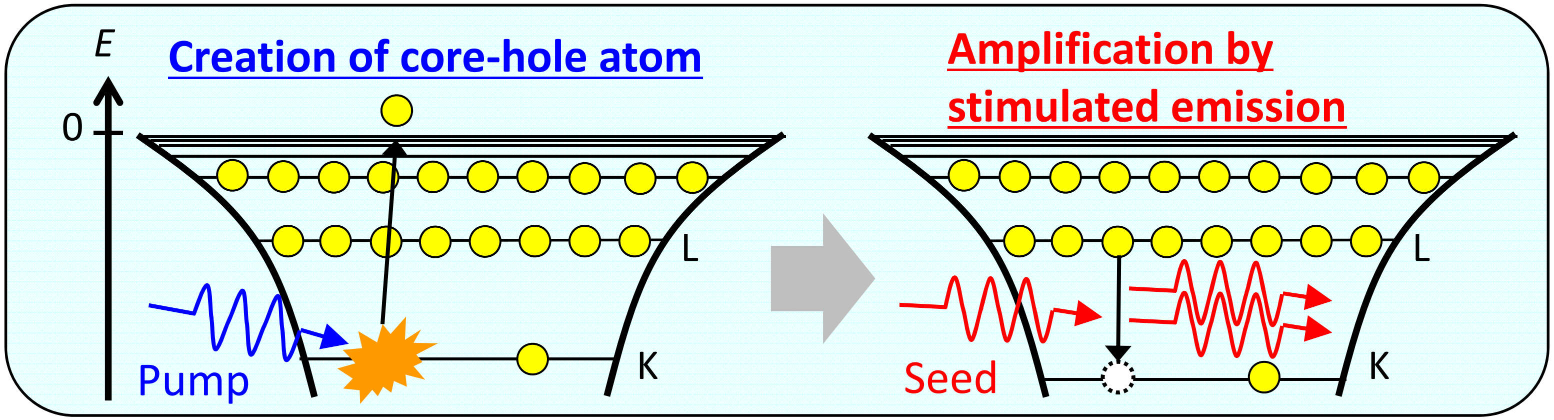
Amplification of x-ray pulse using 2-color XFEL

Prof. Yoneda
(Univ. EC)

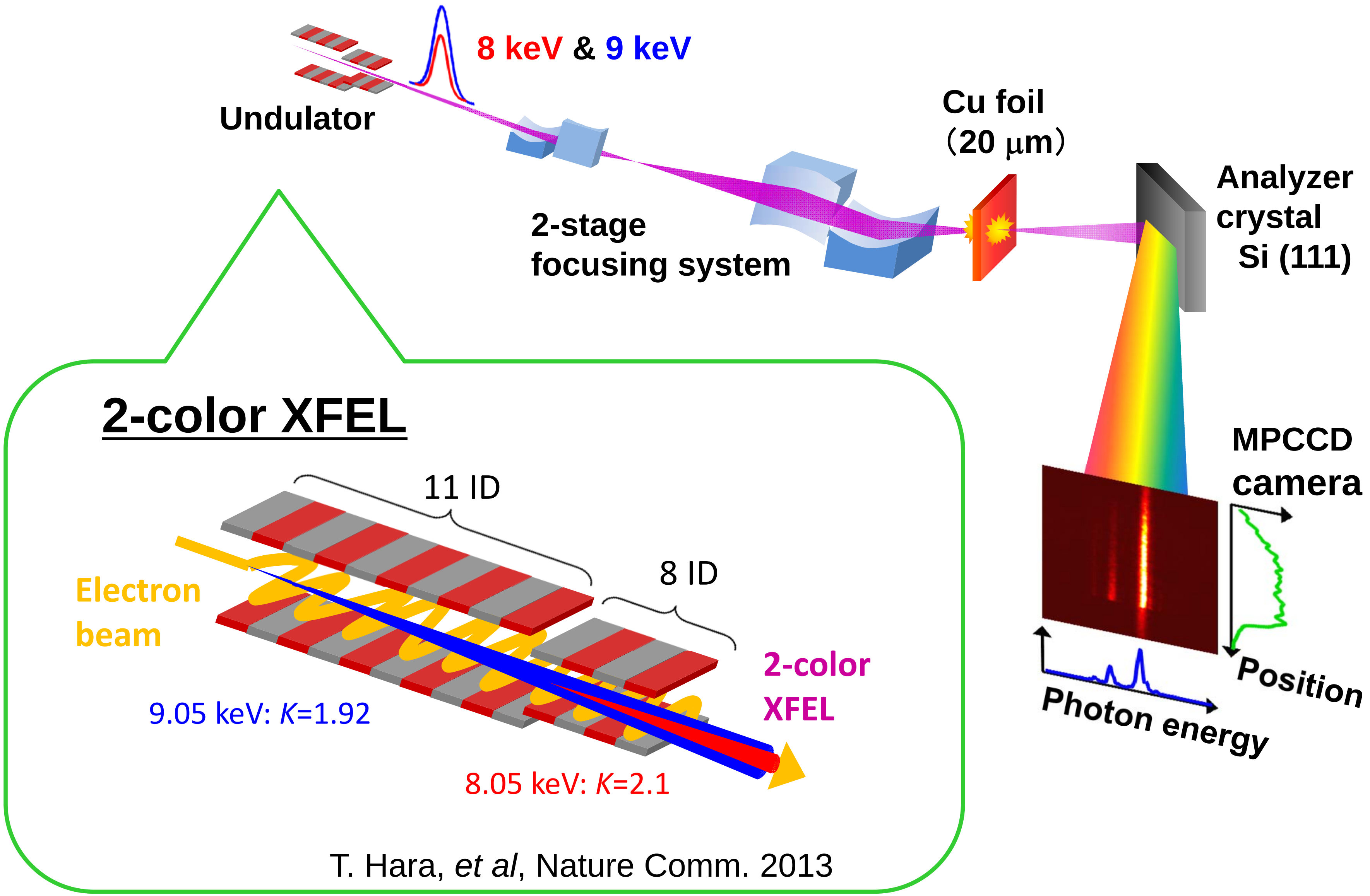
Pump pulse (9keV) creates core-hole atoms.

Seed pulse (8keV) interacted with core-hole atoms is amplified by stimulated emission.



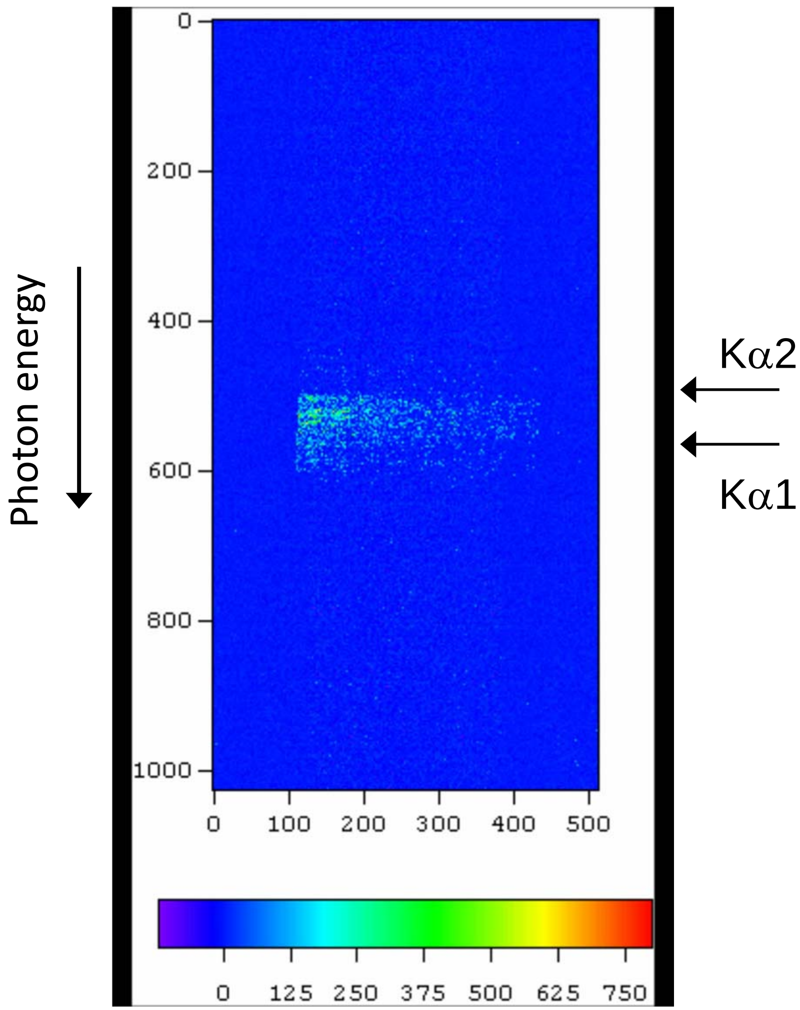


Experimental setup

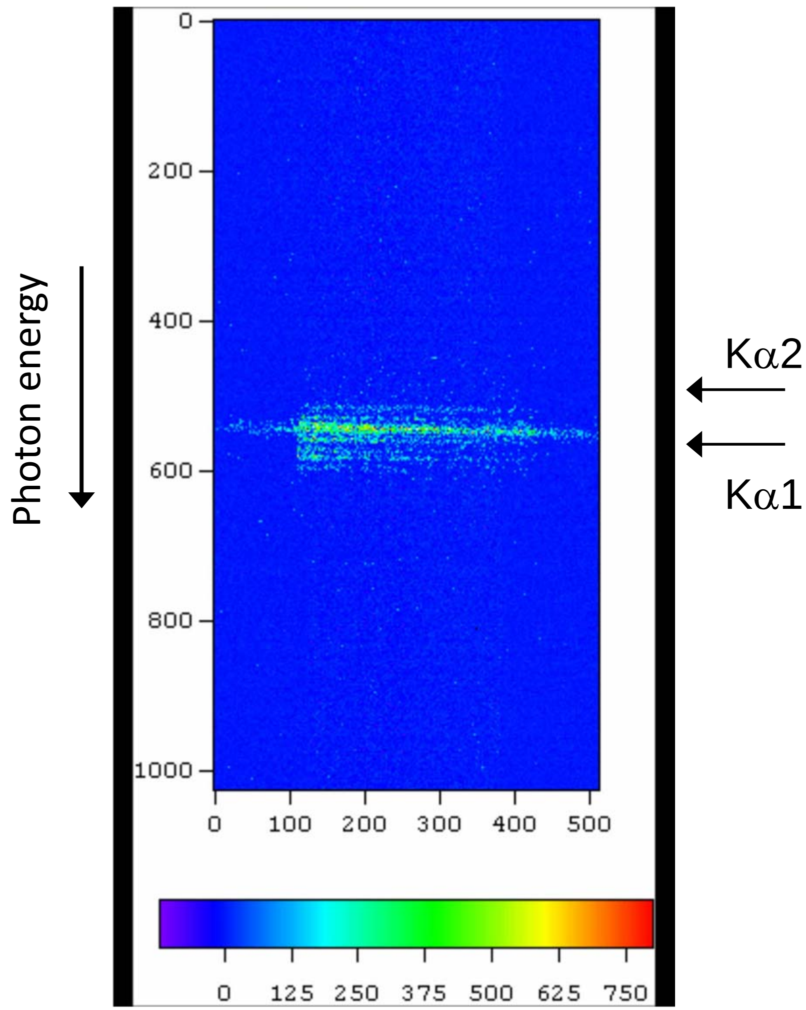


Amplification of x-ray pulses

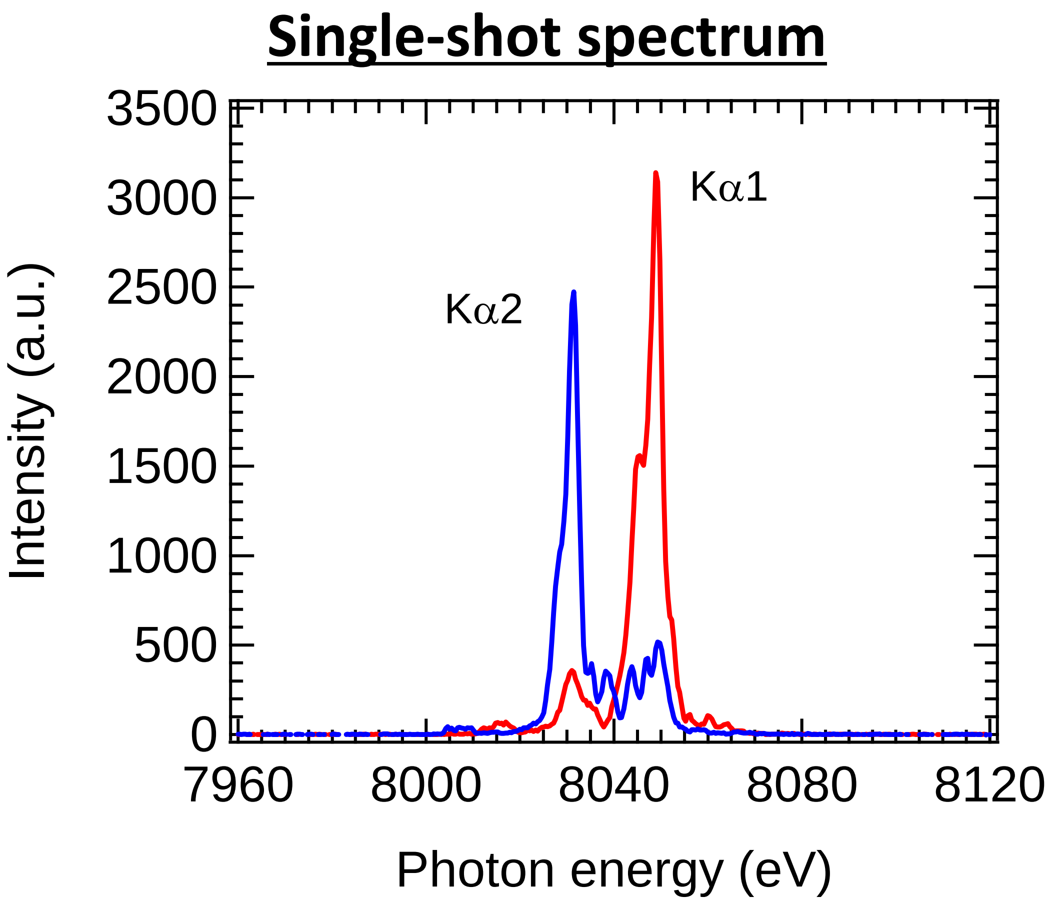
Intensity : 1x10^{18} W/cm^{2}



Intensity : 5x10^{19} W/cm^{2}



Spectrum of amplified x-ray pulse



We achieved amplification of x-ray pulse.

- **Creation of population inversion by intense XFEL pulses**
- **Confirmation that XFEL pulses are applicable as seed pulses**

Summary

- Novel properties and sciences of XFEL
  - Ultra-brilliant, ultra-short, and coherent X-ray pulses
  - Beyond static, statistical pictures
- Beamline for XFEL
  - Damage-free & speckle-free optics
  - Single-shot, nondestructive diagnostics
- Experimental instrumentation for single-shot measurement
  - Focusing optics, sample injectors, detectors, DAQ system, femtosecond laser
- Experiments at SACLA
  - Femtosecond snapshot of sample; “diffraction before destruction”
  - Ultrafast science by pump-probe measurement
  - X-ray-matter interaction under ultra-high intensity

Outlook

- Upgrade of SACLA
  - Double-pulse operation
  - Multi-beamline operation (BL1, BL2, BL3)
  - Self seeding (under development)

- New instruments
  - Experimental platforms for time-resolved measurement
    - SFX
    - X-ray spectroscopy
  - Ultimate focusing
  - High power lasers (500 WT x 2)
  - Detector upgrade
    - Vacuum-compatible MPCCD module
    - MPCCD phase III for higher sensitivity
    - SOPHIAS for wider dynamic range, small pixel