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

XFEL Utilization Division, JASRI

Kensuke Tono

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

XFEL properties and sciences

- Short pulse (<10 fs)
- High peak power (>30 GW)
- Coherent

Ultrafast observation beyond the speed of atomic motion

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

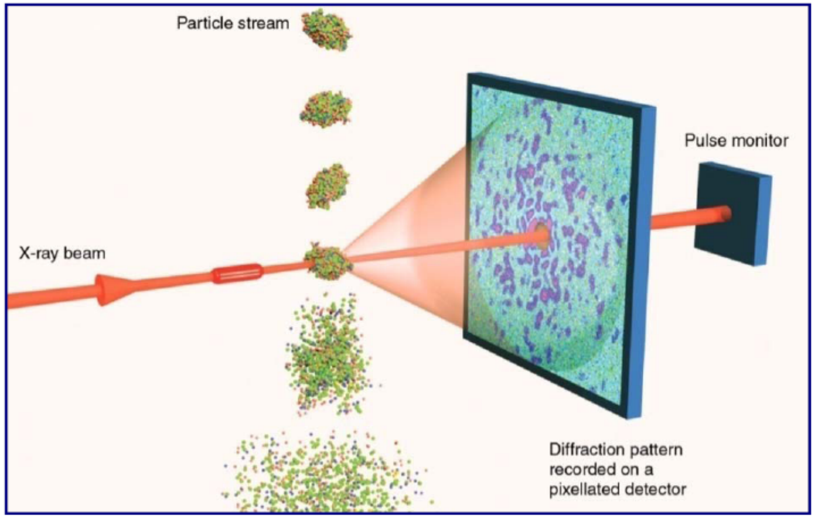
Ultrahigh intensity opens new regime of X-ray-matter interactions

- Beyond linear response

(Image from Wikipedia)

Ultrafast observation
"See before destruction"

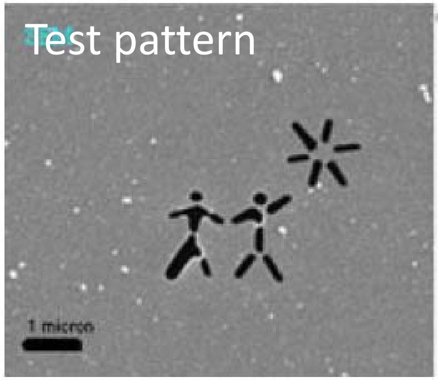
- **Single-shot imaging**

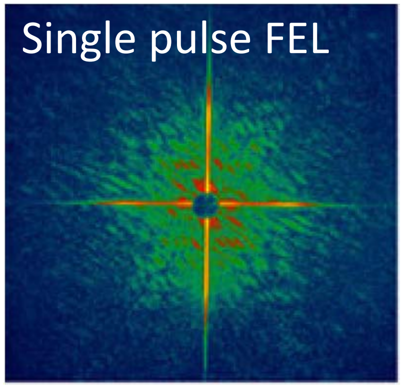


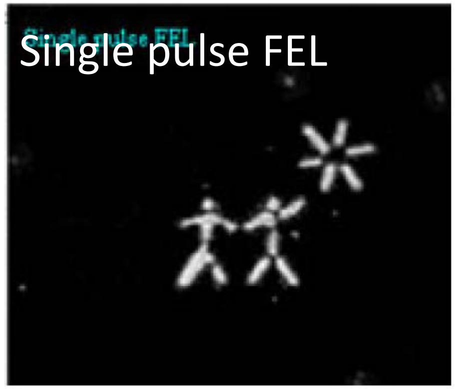
1. See before damaged
2. New shots on new samples
3. Ensemble of 3D orientations
4. Time resolved (~10fs):
   pump-probe imaging
   (conformational change,
   reaction process?)

Images from
H.Chapman, *Science* ('07)

First demonstration at FLASH







Chapman et al., *Nature Physics* **2**, 839 (2006)

Femtosecond snapshot of a *live* cell

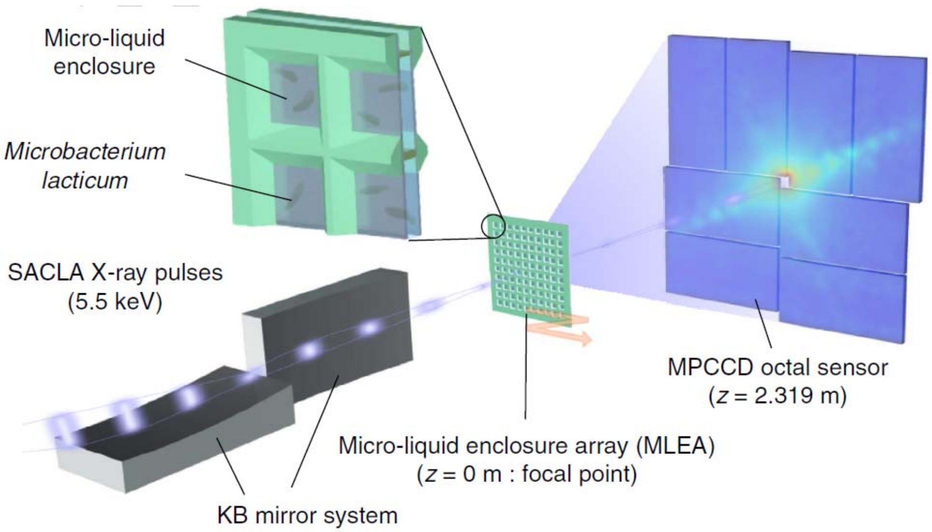
nature
COMMUNICATIONS

ARTICLE

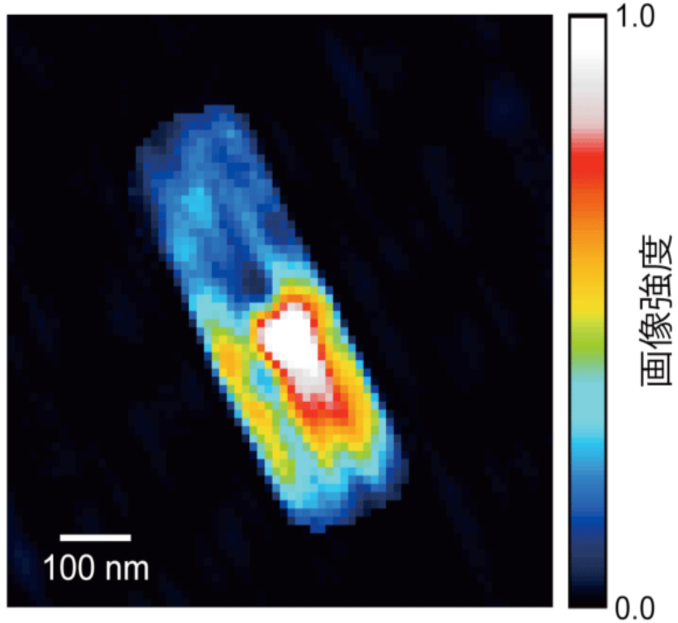
Received 17 Jul 2013 | Accepted 2 Dec 2013 | Published xx xxx 2013 DOI: 10.1038/ncomms4052 OPEN

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

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



XFELで観察した生きている細胞の画像



High intensity application

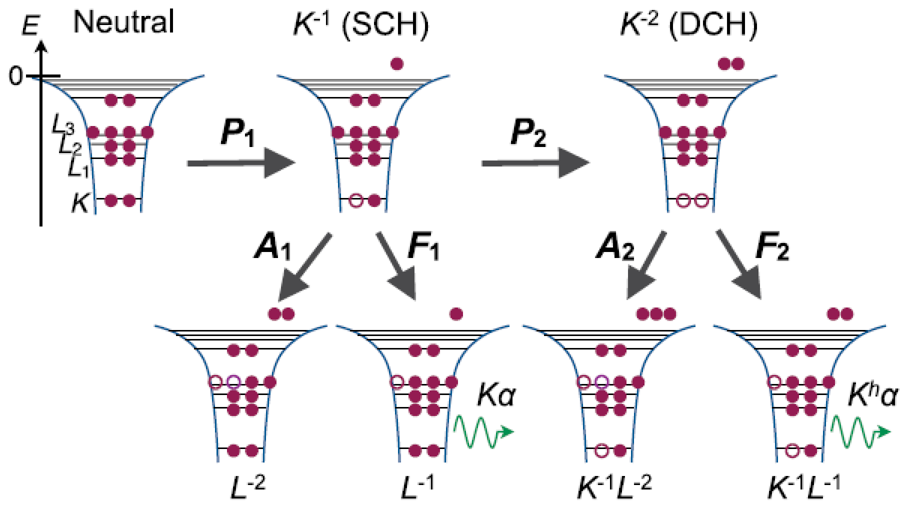
K. Tamasaku et al, PRL Vol.111 (2013)

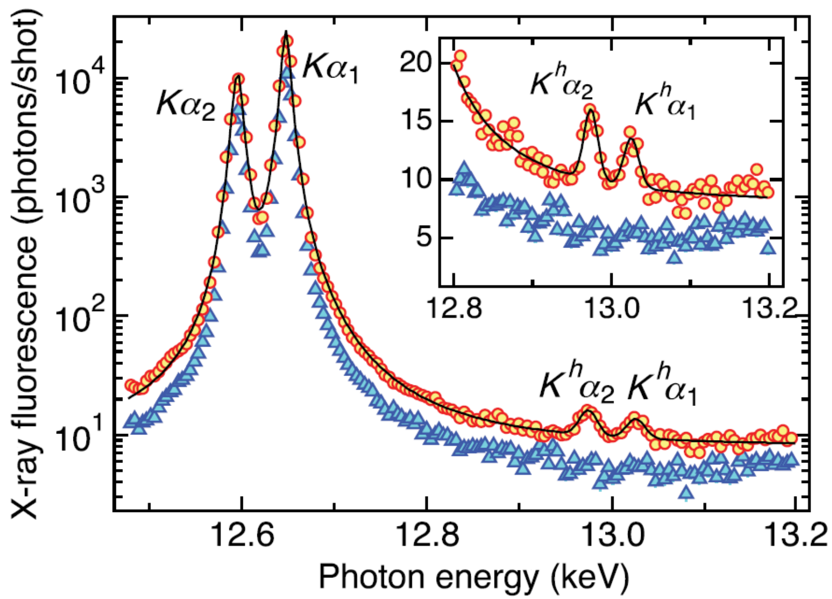
Emission from double core hole atoms

1 µm focusing

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

Double core hole of Kr





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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

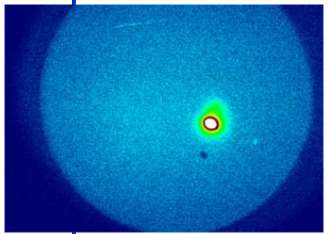
Coherent

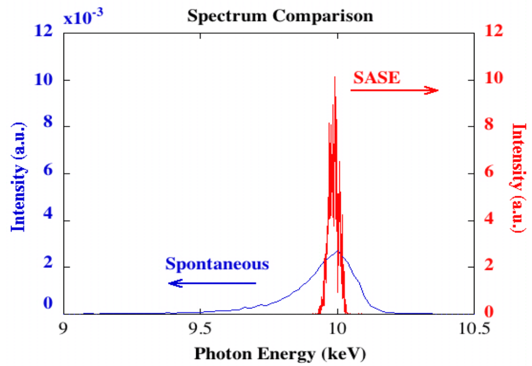
- Transverse only
- Multimode in longitudinal

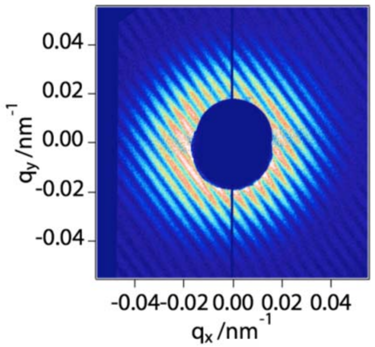
High intensity

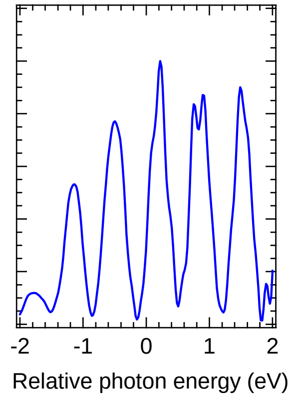
- Pulse energy ~0.5 mJ @10 keV (~3×10^{11} photons)
- Peak power >50 GW@10 keV

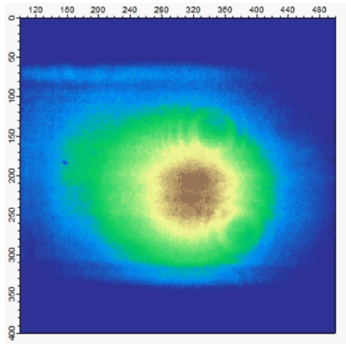
Shot-by-shot fluctuation







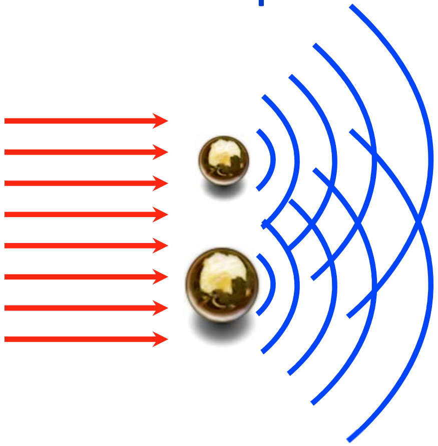


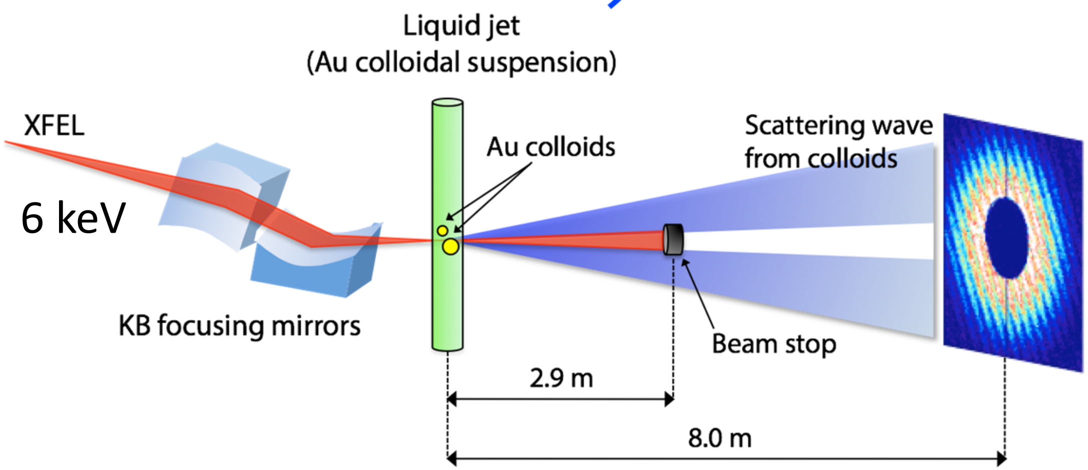


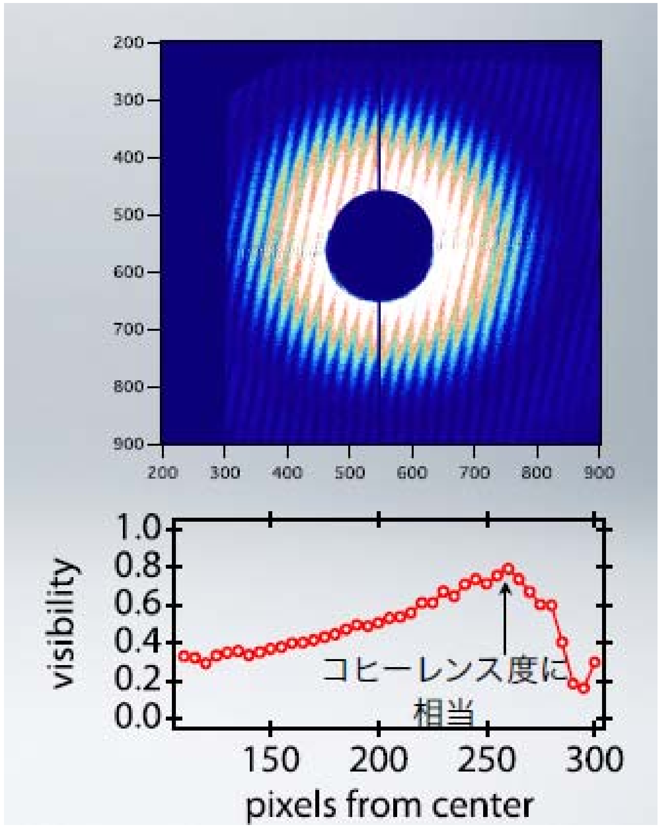
Coherent (transverse only)

Interference between scattering waves from two particles

Inoue (U. Tokyo) et al., in preparation





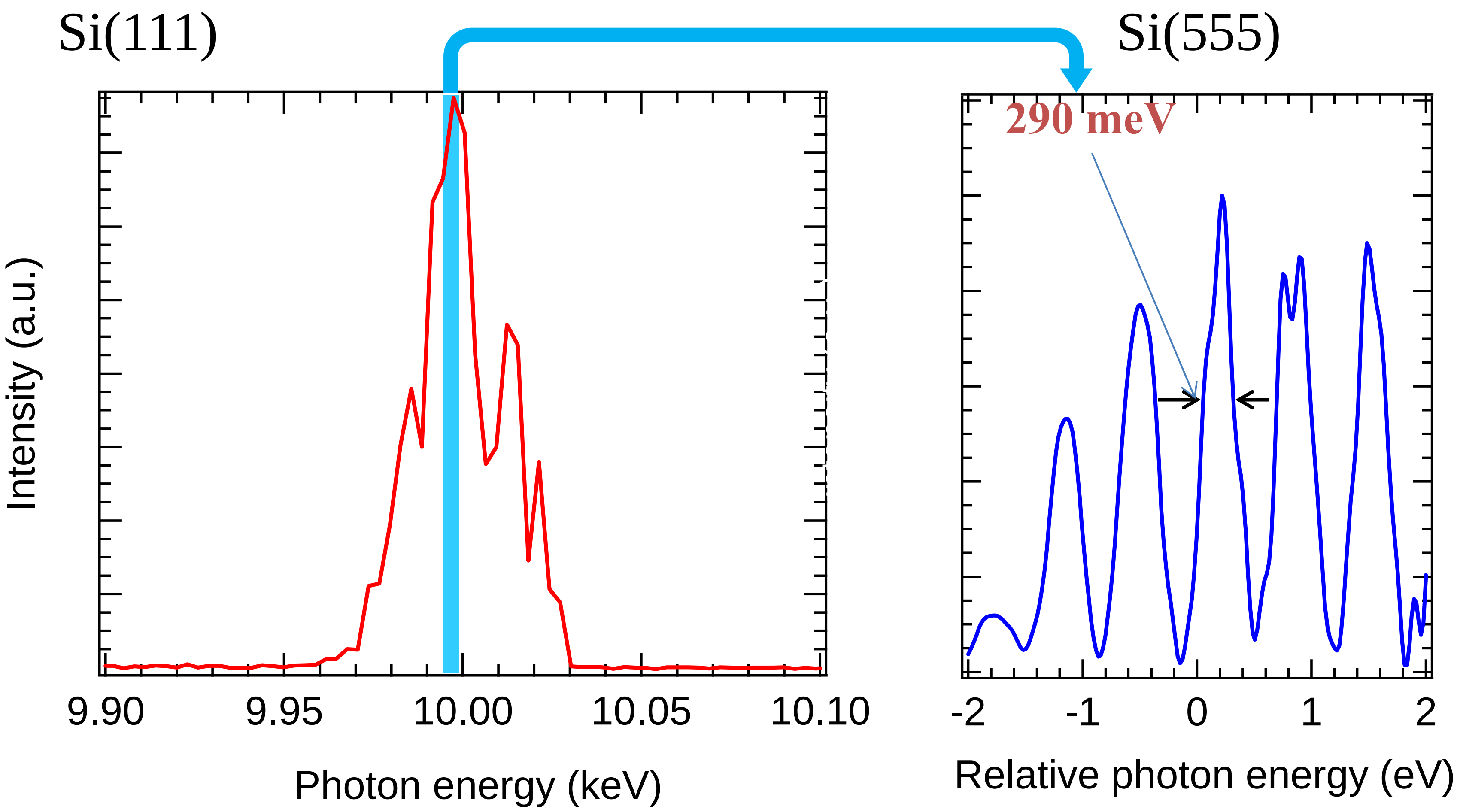


Total degree of coherence: ~0.6

~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.



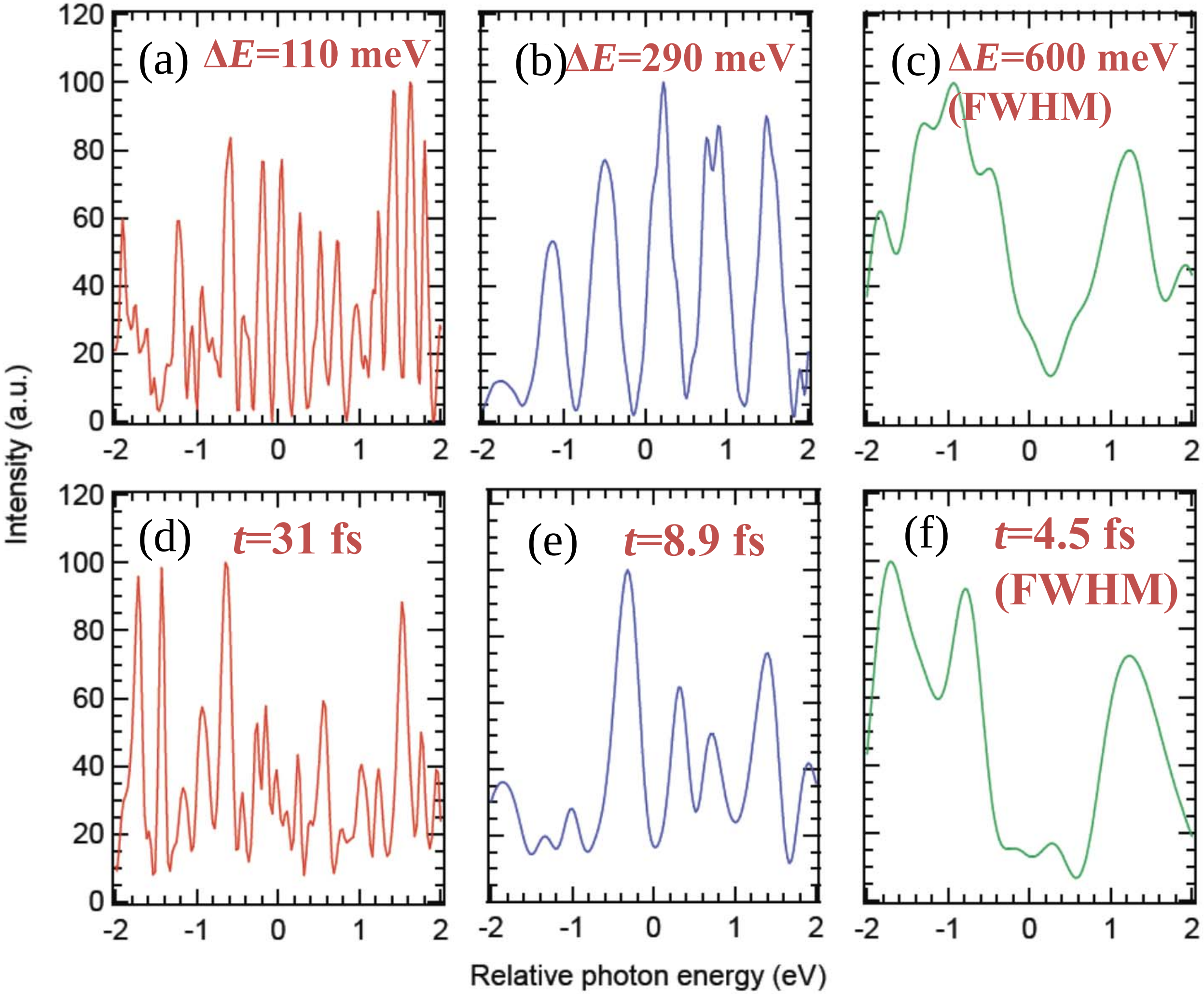
Y. Inubushi *et al.*, *Phys. Rev. Lett*. **109**, 144801 (2012)

Spectra at different pulse durations

Inubushi (JASRI) et al.
PRL 109, 144801 (2012)

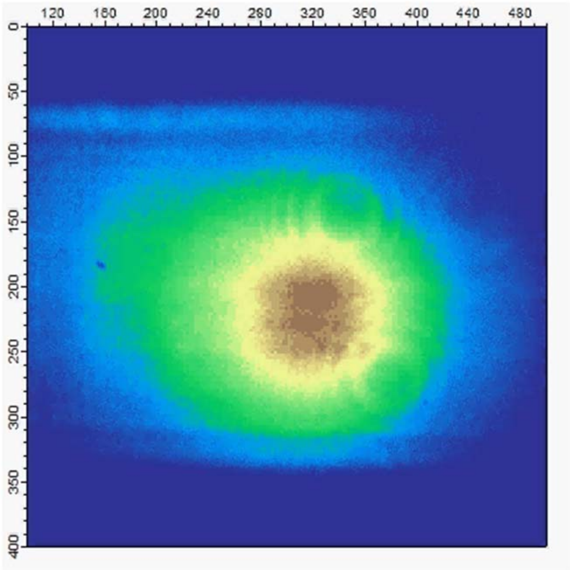
Measured Spectra

Simulated spectra

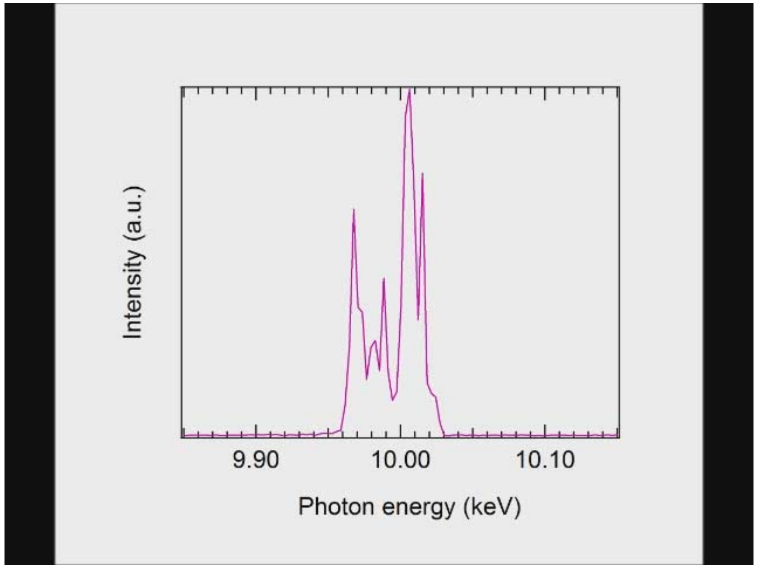


Shot-by-shot fluctuation

Intensity/position



Spectrum



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

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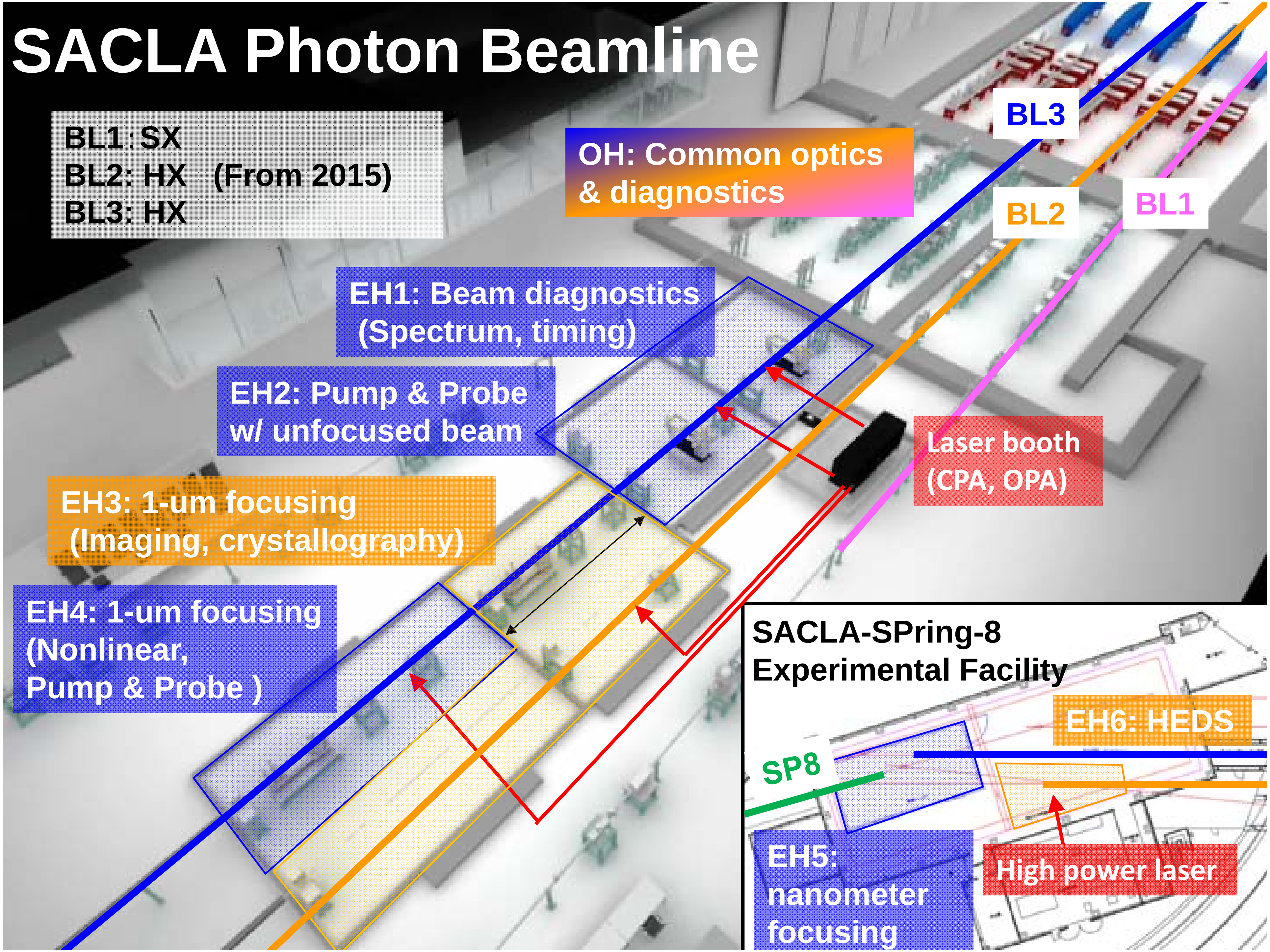
Design concept

- **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

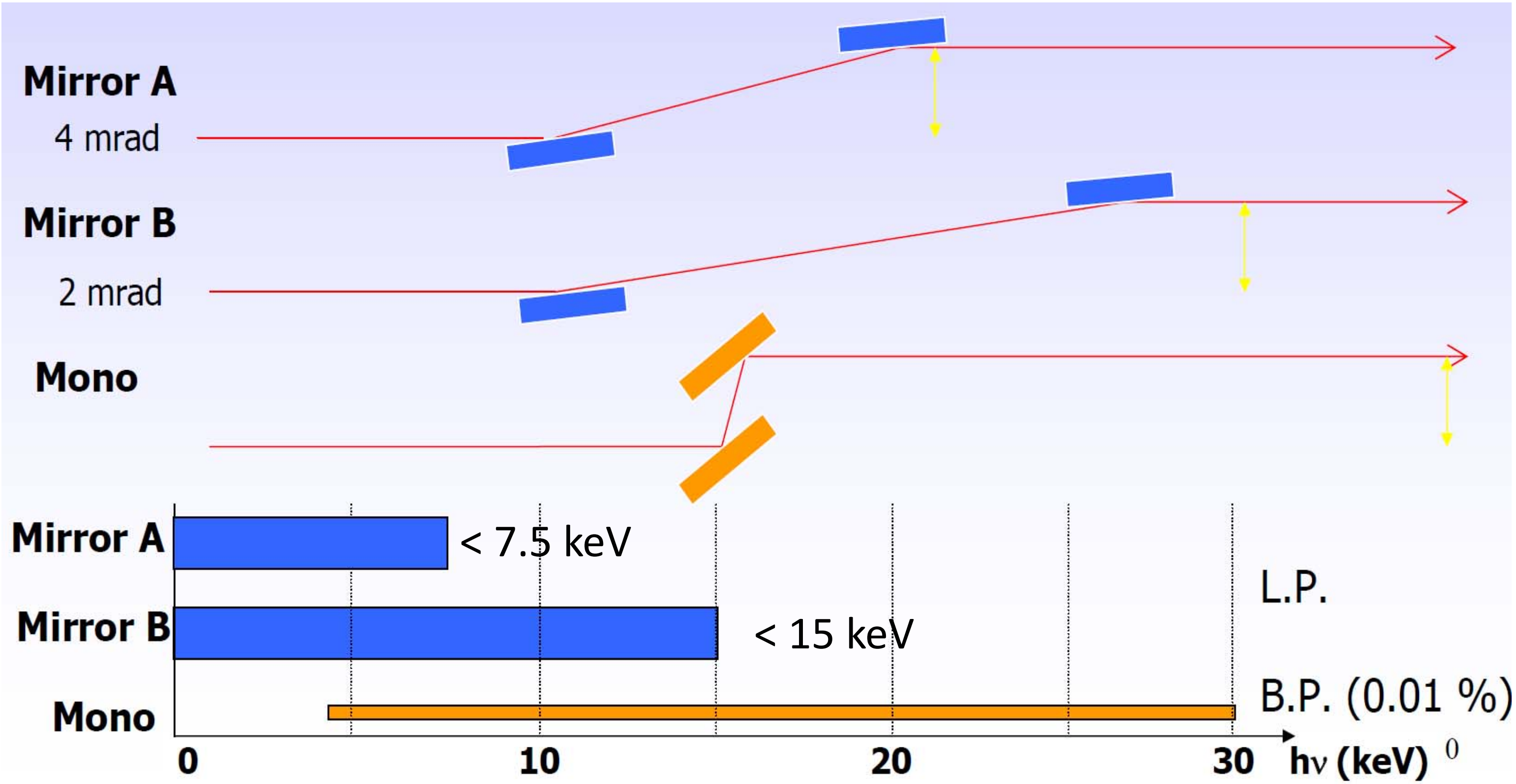


SACLA Photon Beamline
BL1 : SX
BL2: HX (From 2015)
BL3: HX
OH: Common optics & diagnostics
BL3
BL2
BL1
EH1: Beam diagnostics (Spectrum, timing)
EH2: Pump & Probe w/ unfocused beam
Laser booth (CPA, OPA)
EH3: 1-um focusing (Imaging, crystallography)
EH4: 1-um focusing (Nonlinear, Pump & Probe)
SACLA-SPring-8 Experimental Facility
EH6: HEDS
SP8
EH5: nanometer focusing
High power laser

Beamline optics

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})



Optical elements for XFEL

XFEL features	Demands
Short pulse (<10 fs)	Damage free
High peak power (>30 GW)	
Coherent	Speckle free

Damage on a mirror material

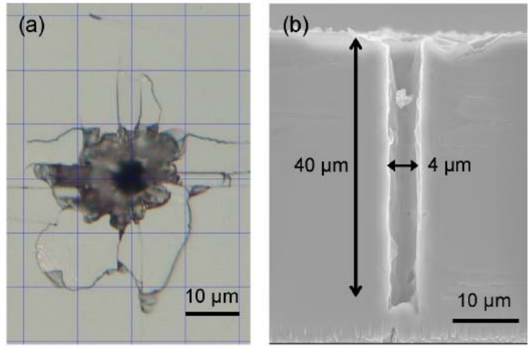
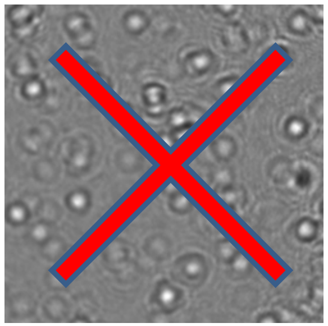


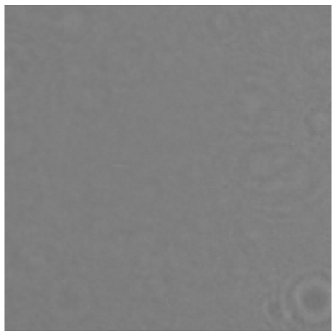
Fig. 2. (a) Optical microscope image of irradiated silicon viewed from surface at fluence of 57 μJ/μm². (b) Cross sectional SEM image of (a) prepared by focused ion beam sampling.

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

Typical Be window



Speckle-free Be window



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

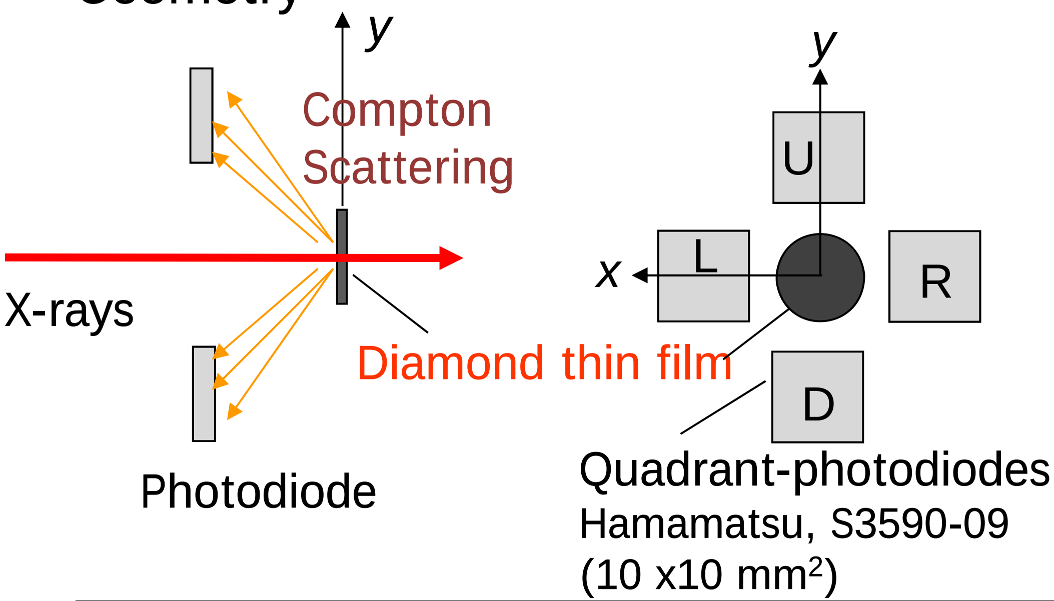
Ultraprecise mirror finished by Elastic Emission Machining

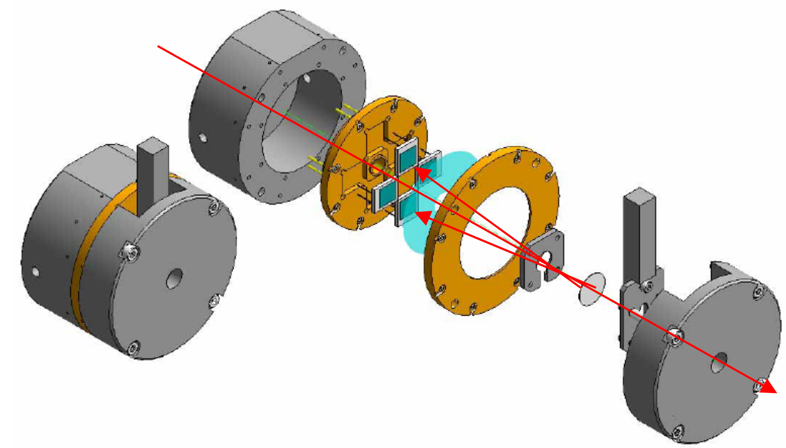
Mimura et al., Rev. Sci. Instrum. **79**, (2008)

On-line photon diagnostics:
Beam monitor (intensity/position)

Geometry

K. Tono et al. *RSI* 82, 023108 (2011)

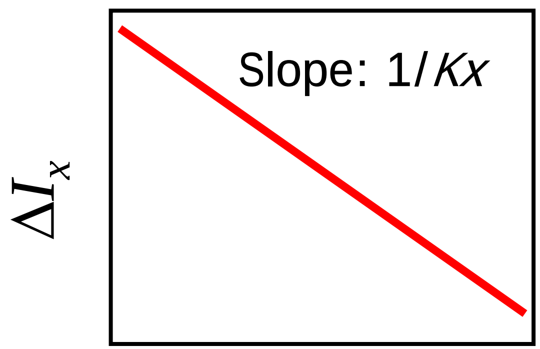




Intensity $I \propto \left(I_L + I_R + I_U + I_D\right)$

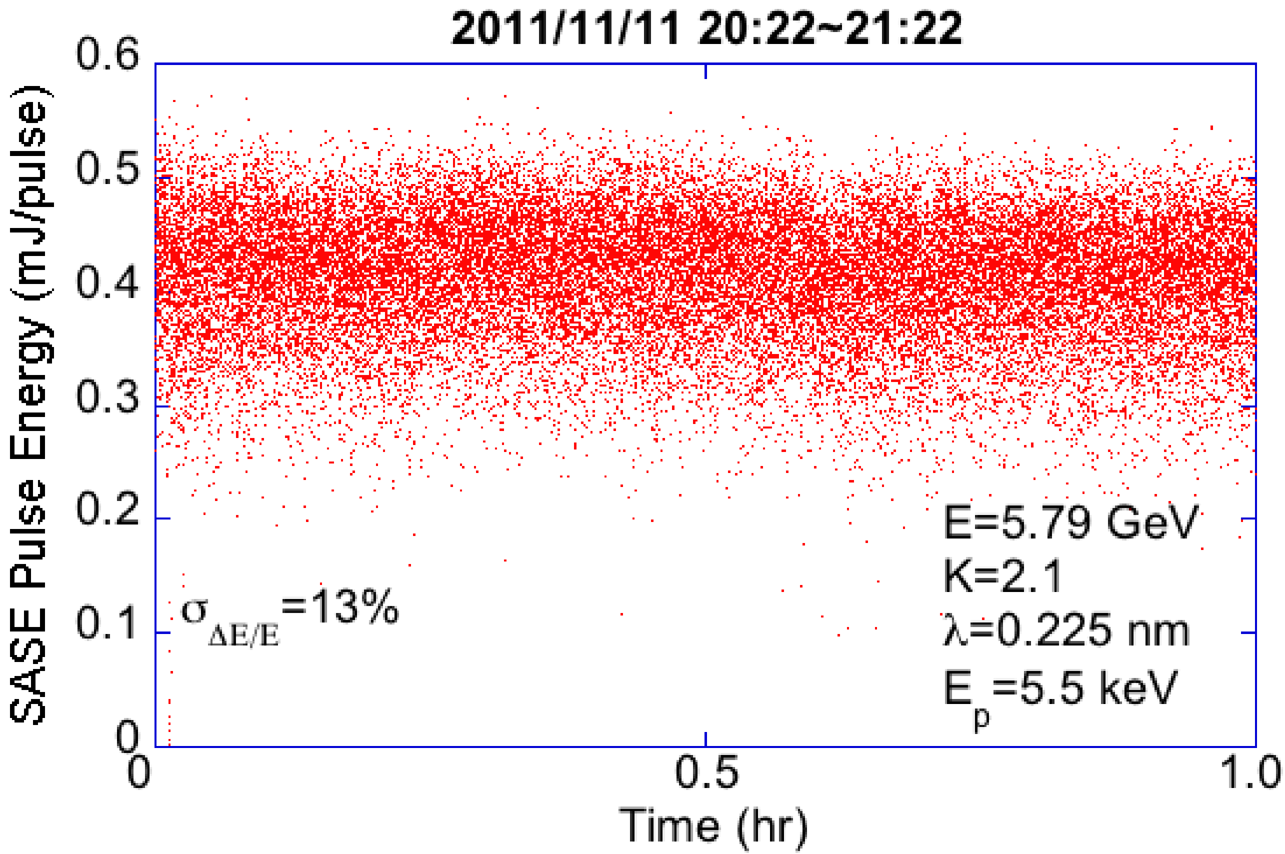
Position $x = K_x \dfrac{I_\mathrm{L} - I_\mathrm{R}}{I_\mathrm{L} + I_\mathrm{R}} = K_x \Delta I_x$

$$y = K_y \frac{I_\mathrm{U} - I_\mathrm{D}}{I_\mathrm{U} + I_\mathrm{D}} = K_y \Delta I_y$$

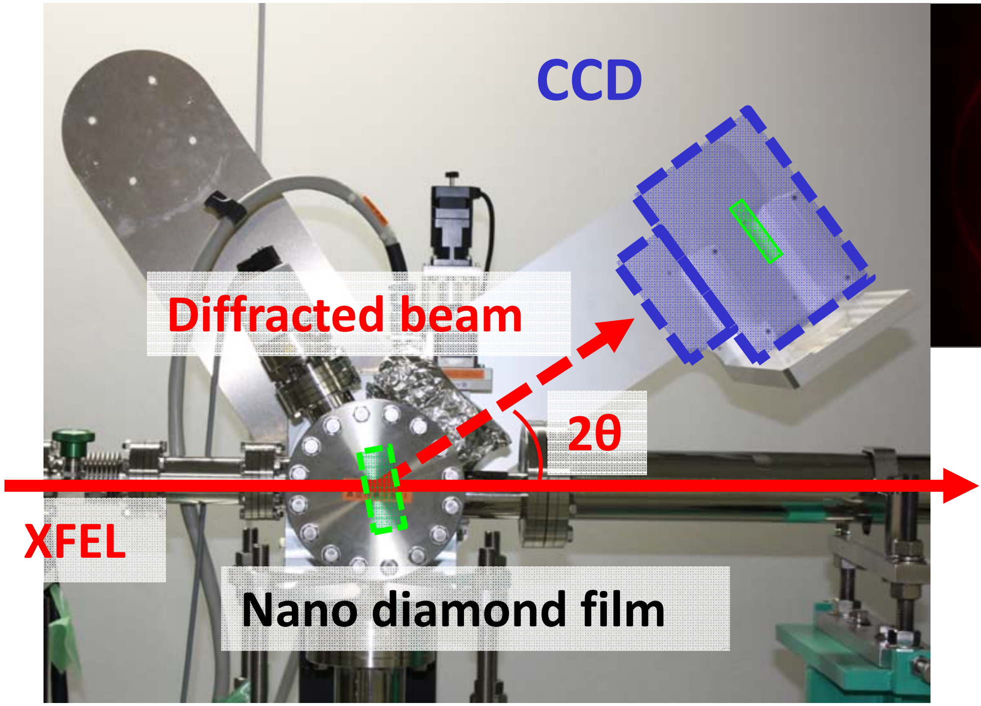


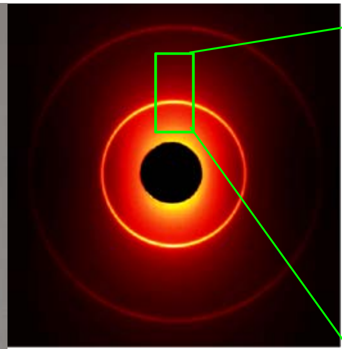
Alkire et al., *J. Syn. Rad.* 7, 61 (2000).

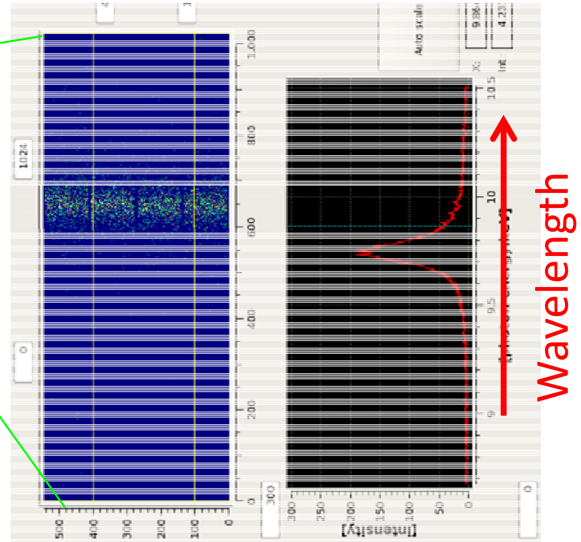
Shot-by-shot measurement of pulse energy



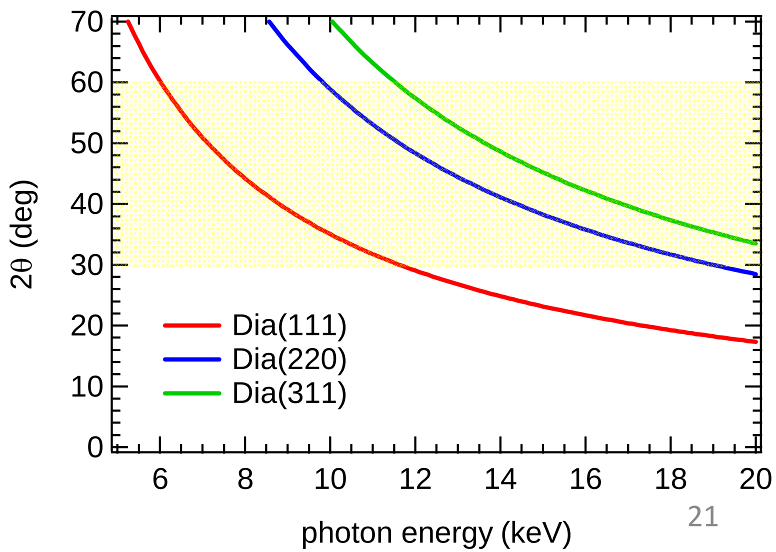
Wavelength (photon-energy) monitor







Measurable range: 6 - 20 keV

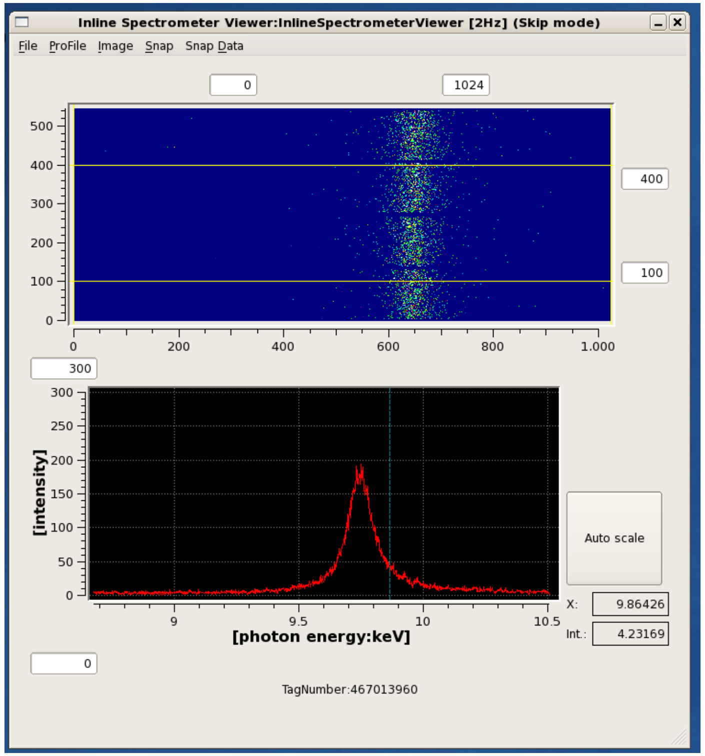


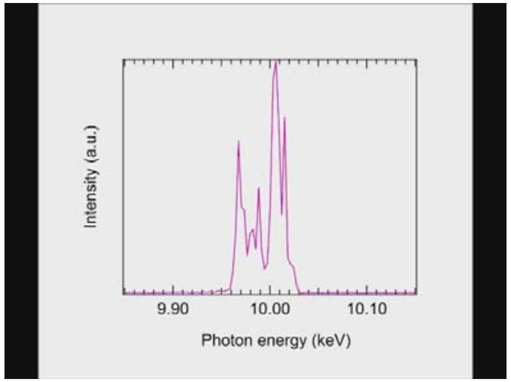
Wavelengths (λ) are calculated from positions of Debye-Scherrer rings on MPCCD.

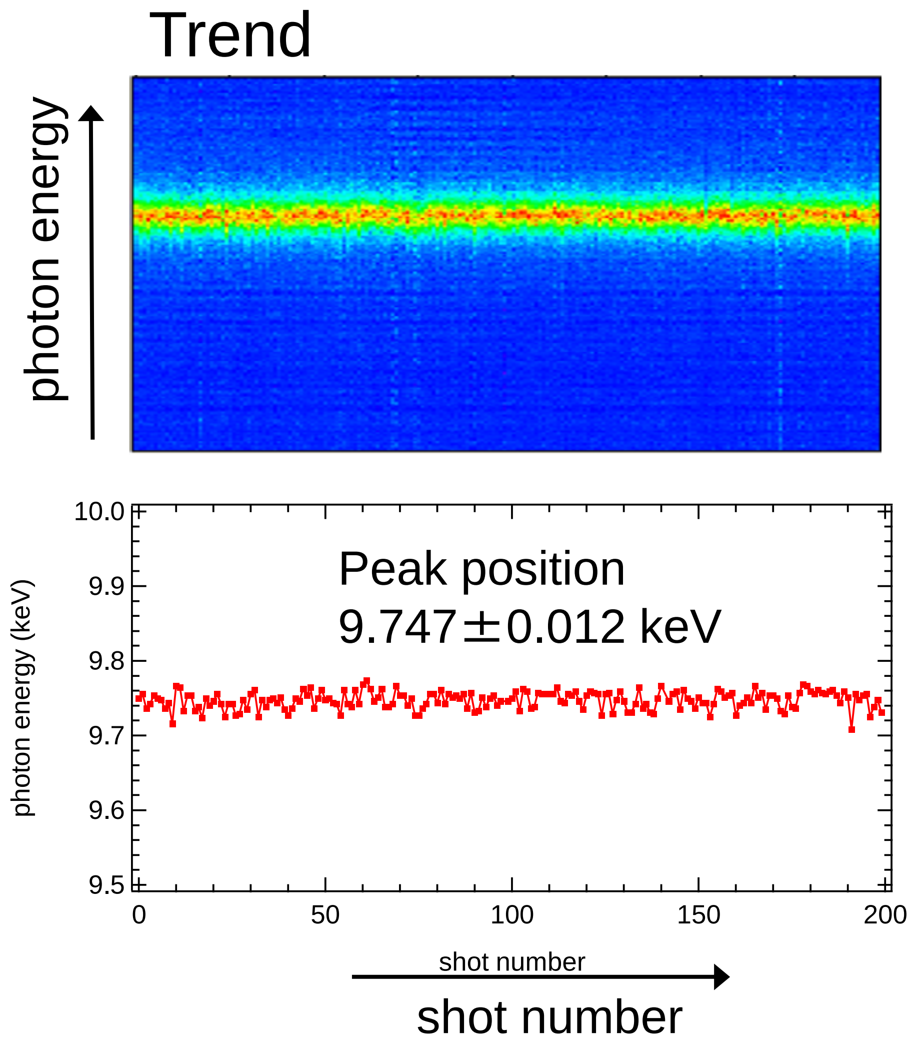
$2d\sin\theta = n\lambda$

Inubushi -san

Shot-by-shot measurement



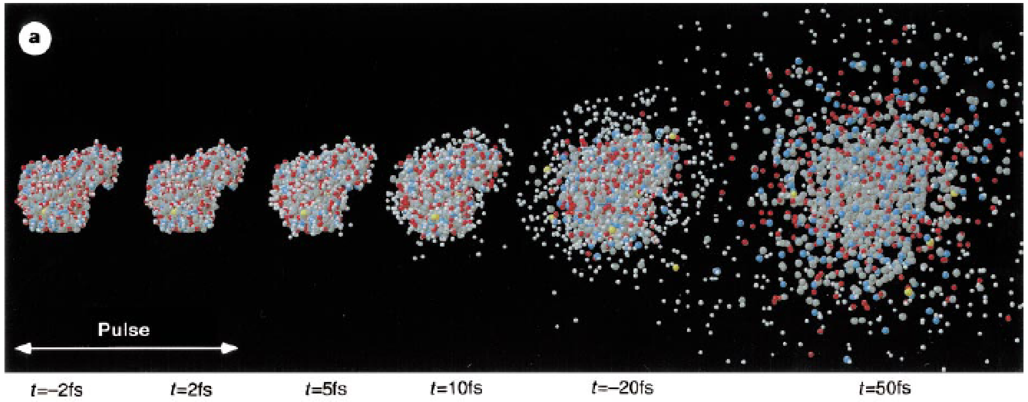




Contents

Single-shot measurement is mandatory.

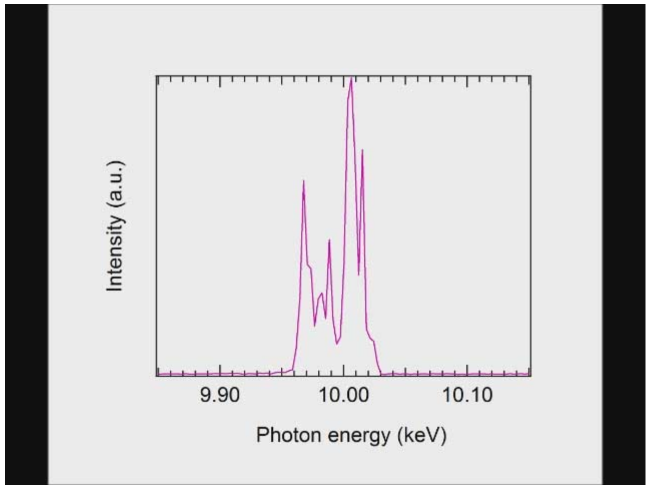
- Even a single pulse destroys a sample.



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

- Pulse-by-pulse fluctuation of XFEL pulses.

Difficult to repeat measurement in the same condition.

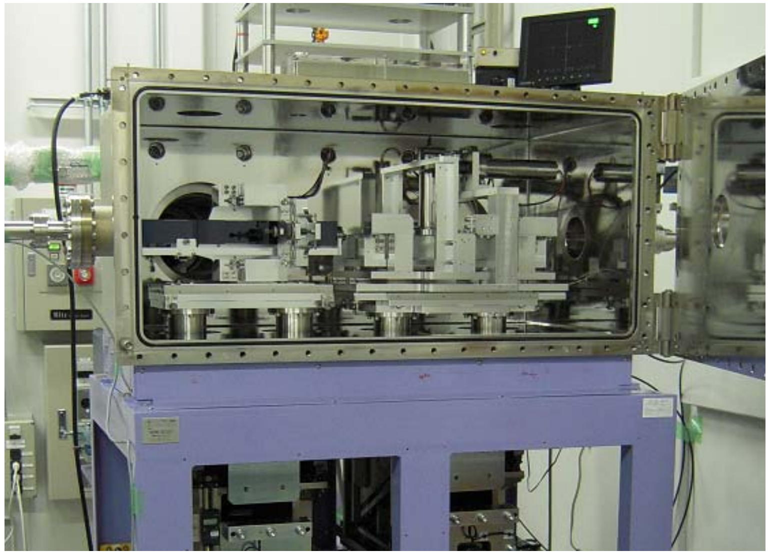


Instrumentation for single-shot measurement

- High photon flux
  - Focusing
- Fast sample exchange
  - Injectors
  - Fixed targets with a fast scanning system
- Fast & sensitive X-ray detection
  - High performance detectors
    - High sensitivity, high frame rate, high dynamic range, large area, …
- Fast & reliable data acquisition (DAQ)
  - High performance computers
  - High speed network
  - Large storage system

Focusing

1-um focusing mirrors



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

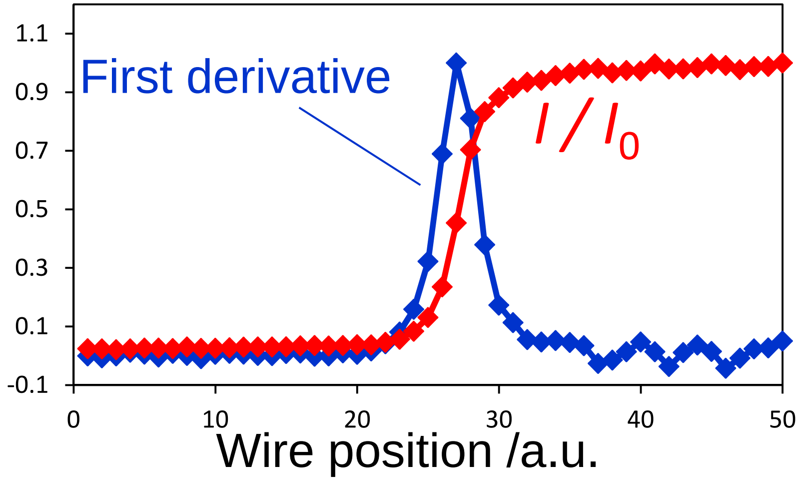

nature photonics | LETTERS
PUBLISHED ONLINE: 16 DECEMBER 2012 | DOI: 10.1038/NPHOTON.2012.306

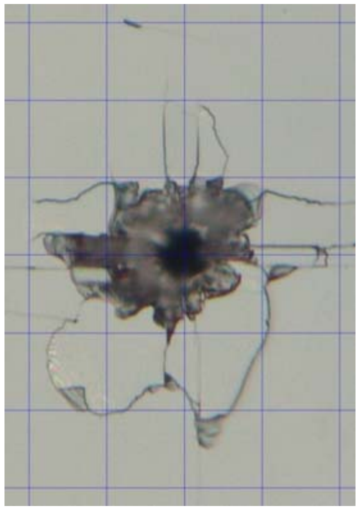
Focusing of X-ray free-electron laser pulses with reflective optics

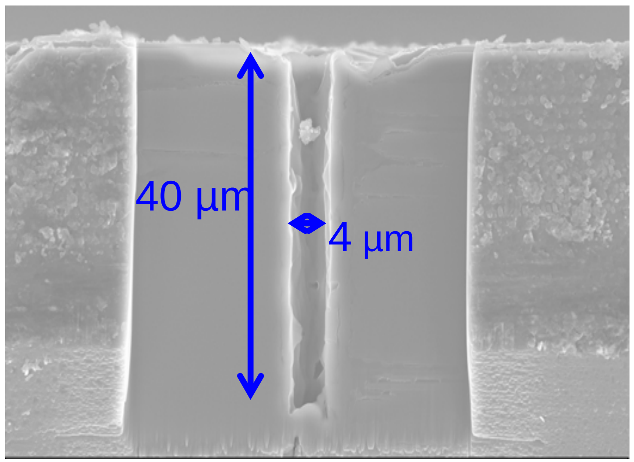
Hirokatsu Yumoto[1]*, Hidekazu Mimura[2], Takahisa Koyama[1], Satoshi Matsuyama[3,4], Kensuke Tono[1], Tadashi Togashi[1], Yuichi Inubushi[5], Takahiro Sato[5], Takashi Tanaka[5], Takashi Kimura[6], Hikaru Yokoyama[3], Jangwoo Kim[3], Yasuhisa Sano[3], Yousuke Hachisu[7], Makina Yabashi[5], Haruhiko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

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





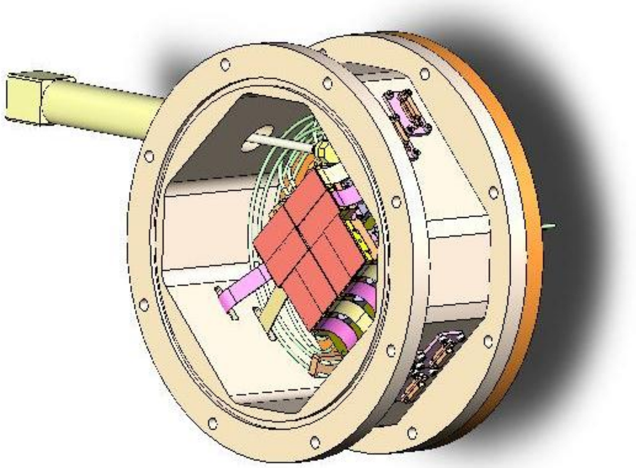


Koyama et al, OE **21**, 15382 (2013)

Detector


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

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

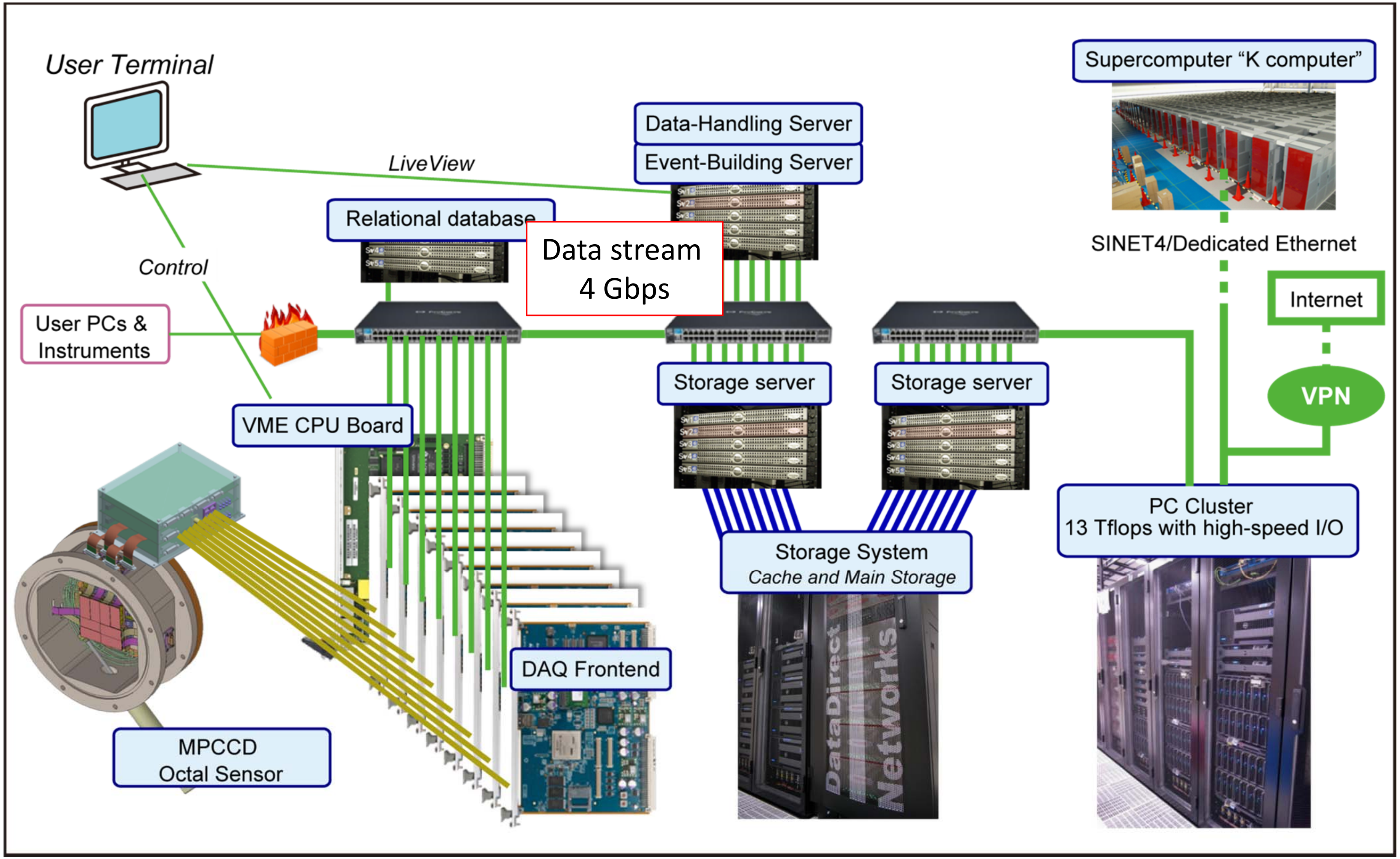


Octal Sensor Detector (100 x 100 mm)
2048 x 2048 pixels

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)

Joti, Kameshima (JASRI)
Hatsui (RIKEN) et al.



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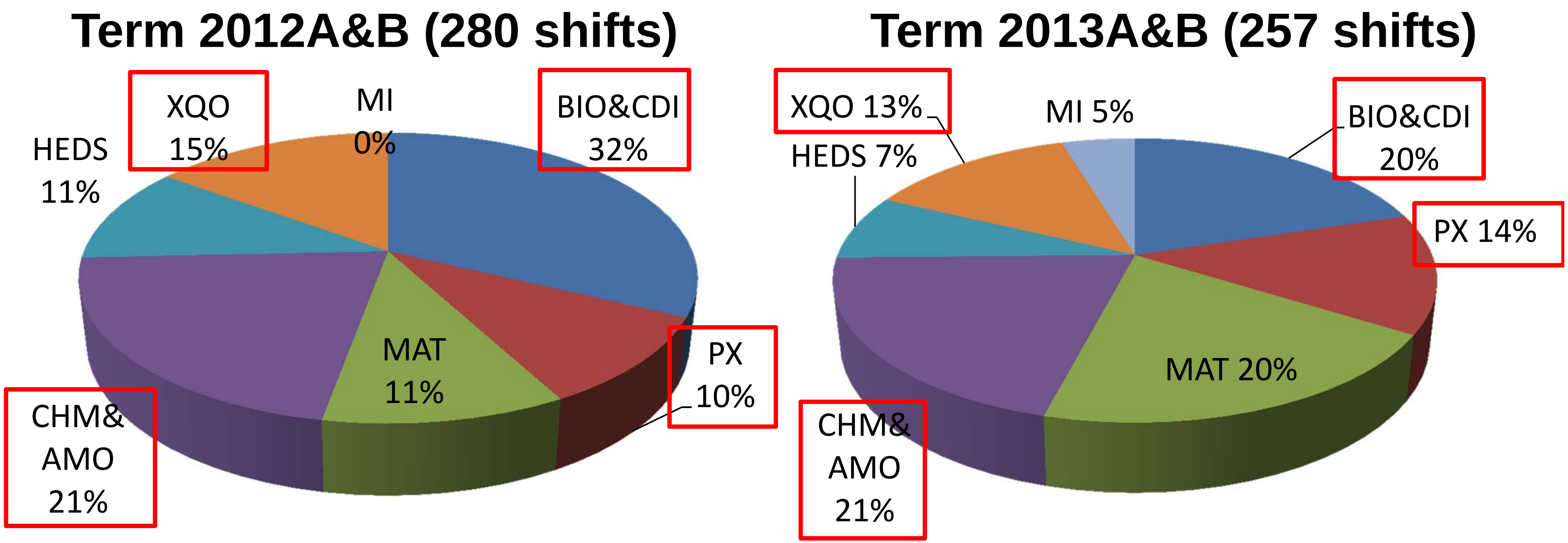
   - Coherent diffraction imaging (CDI)
   - Femtosecond protein crystallography
   - Time-resolved X-ray absorption spectroscopy (XAS)
   - Nonlinear X-ray optics

User experiments in FY2012&2013



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

XFEL as a probe, as a trigger

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

"See before destruction" (1)
CDI for ***single-particle*** structure analysis

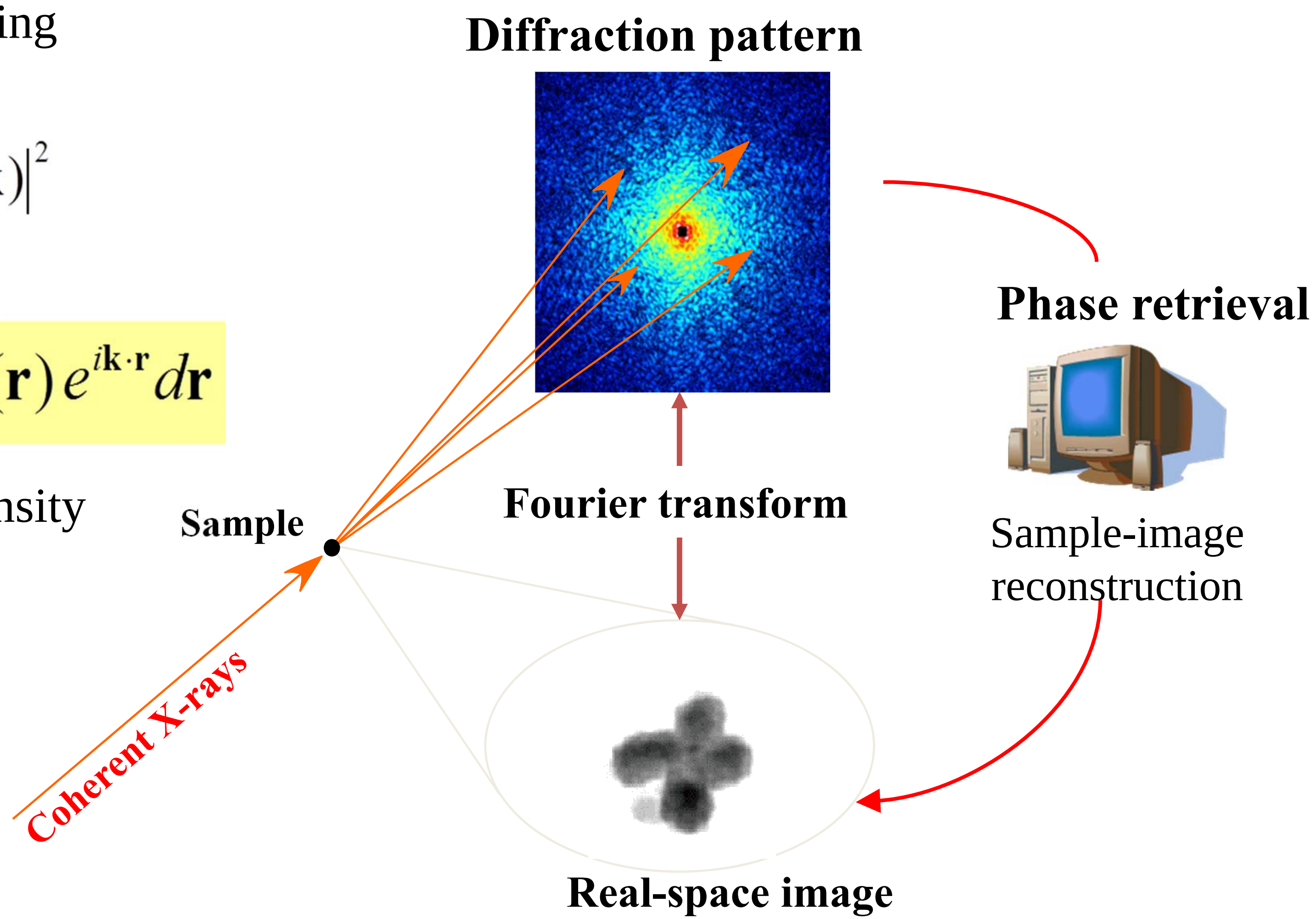
Differential scattering cross section

$$\frac{d\sigma}{d\Omega} = Pr_e^2 |F(\mathbf{k})|^2$$

Structure factor

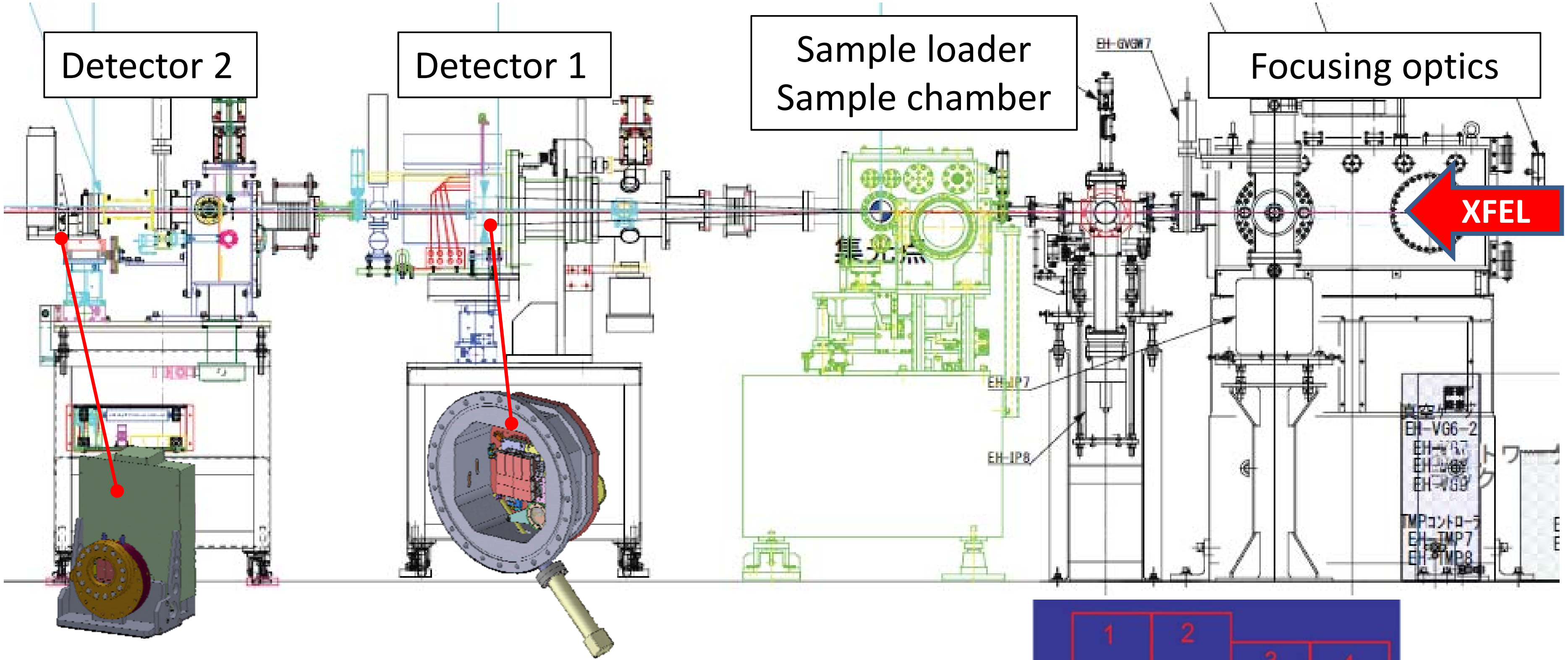
$$F(\mathbf{k}) = \int \rho(\mathbf{r}) e^{i\mathbf{k}\cdot\mathbf{r}} d\mathbf{r}$$

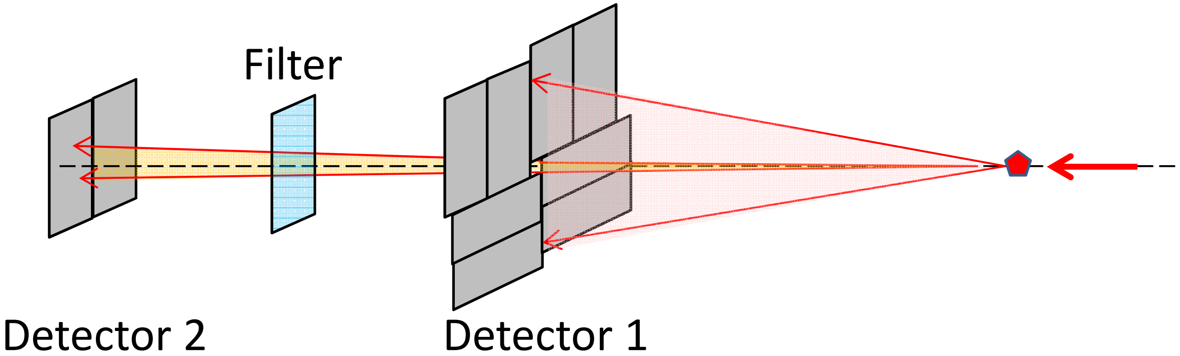
$\rho(\mathbf{r})$: Electron density

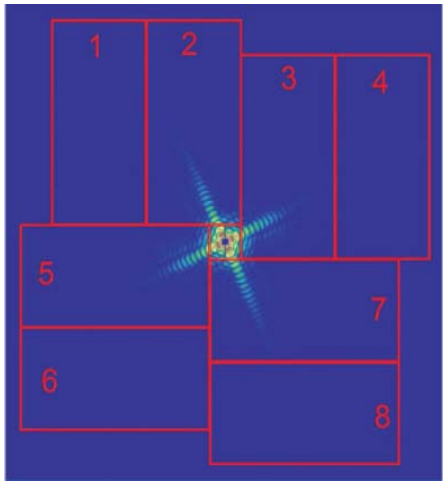


Y. Nishino *et al*., Phys. Rev. Lett **102**, 018101 (2009).

Typical setup for CDI



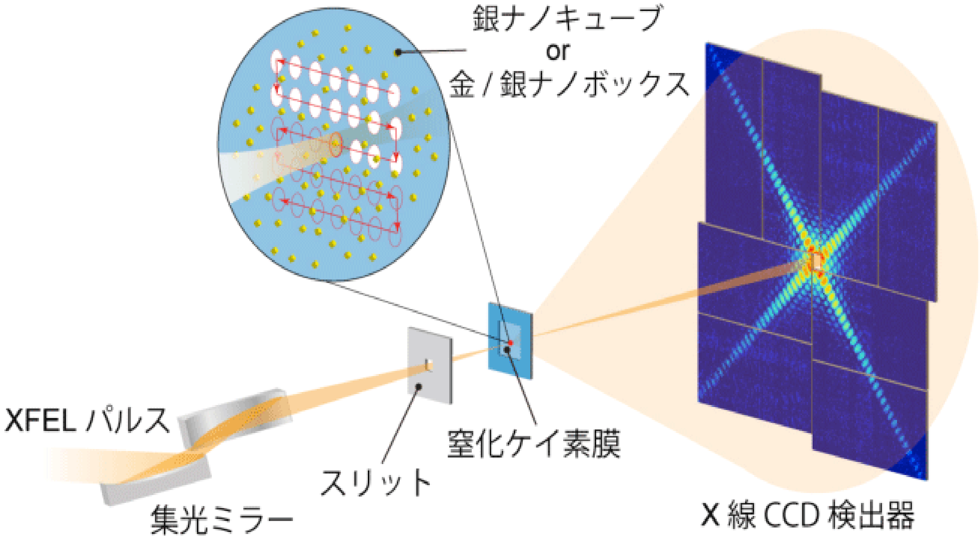


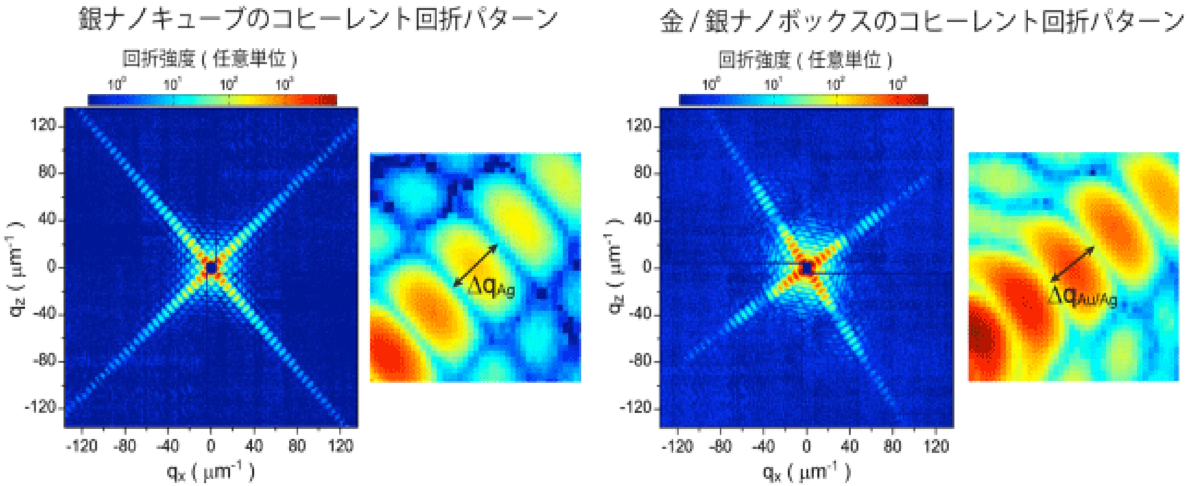


Kameshima et al., Rev. Sci. Instrum **85**, 033110 (2014)

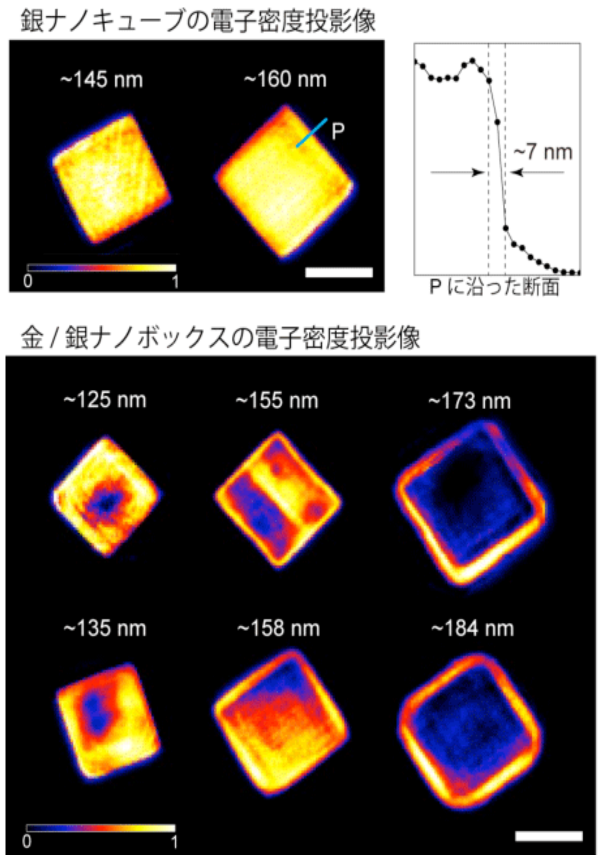
CDI of nanomaterials

Takahashi et al., *Nano Lett.* **13**, 6028 (2013)



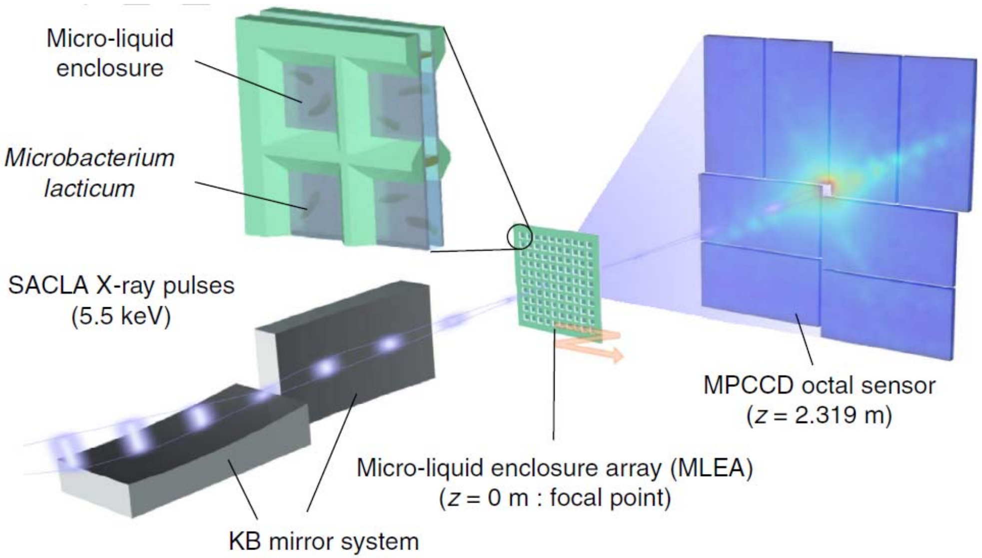


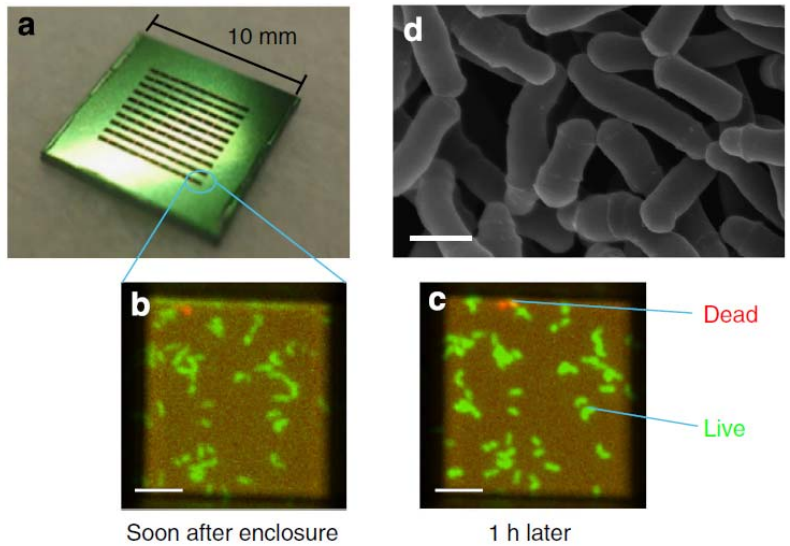
Resolution ～7 nm

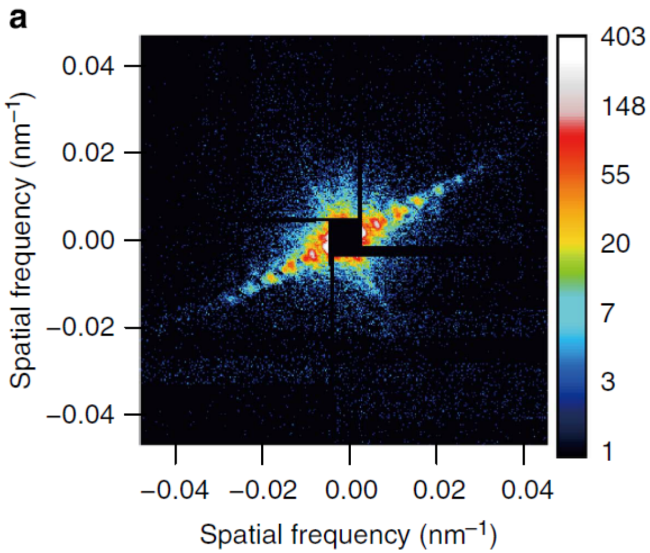


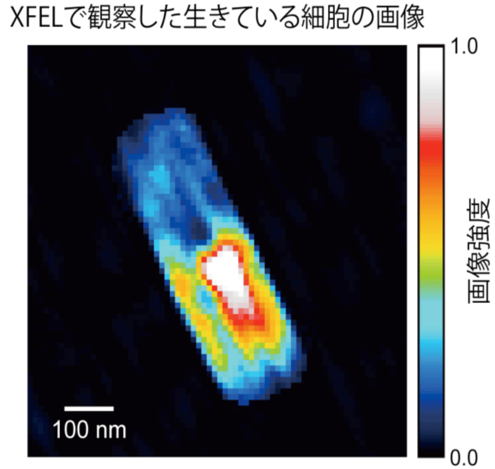
CDI of live cell

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

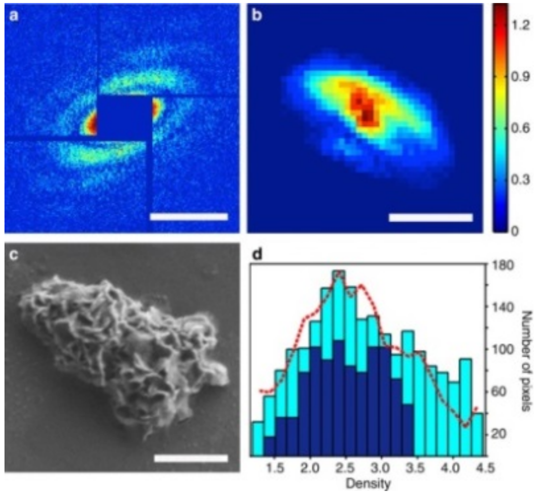




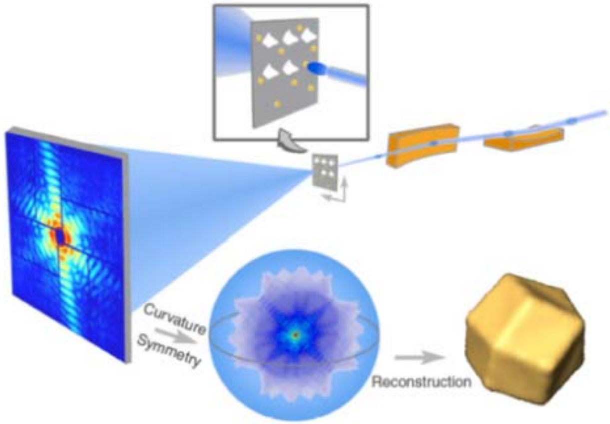




And more



"Macromolecular structures probed by combining single-shot free-electron laser diffraction with synchrotron coherent X-ray imaging,"
M. Gallagher-Jones et al., *Nature Commun.* (2014)



"Single-shot three-dimensional structure determination of nanocrystals with femtosecond
X-ray free-electron laser pulses,"
R. Xu et al., *Nature Commun.* (2014)

“See 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

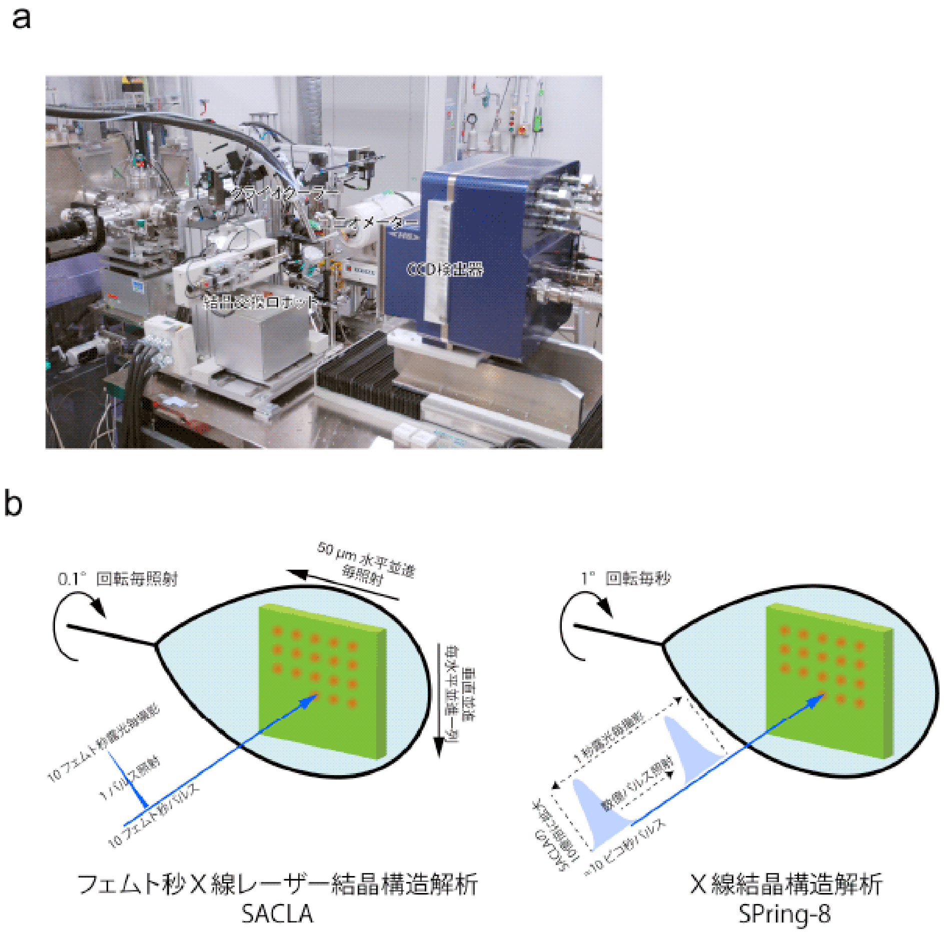
Femtosecond crystallography

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

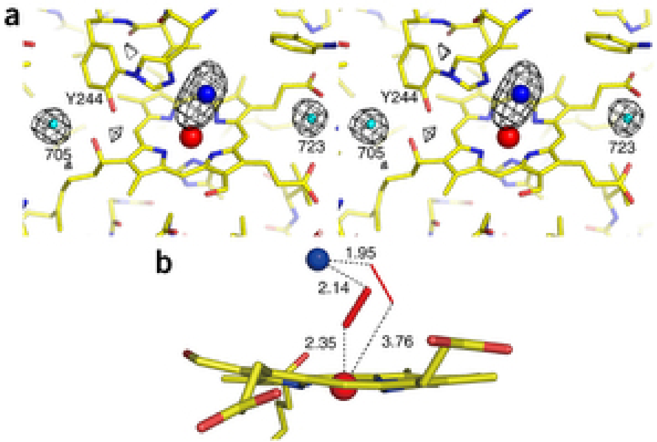
Determination of damage-free crystal structure of an X-ray–sensitive protein using an XFEL

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]

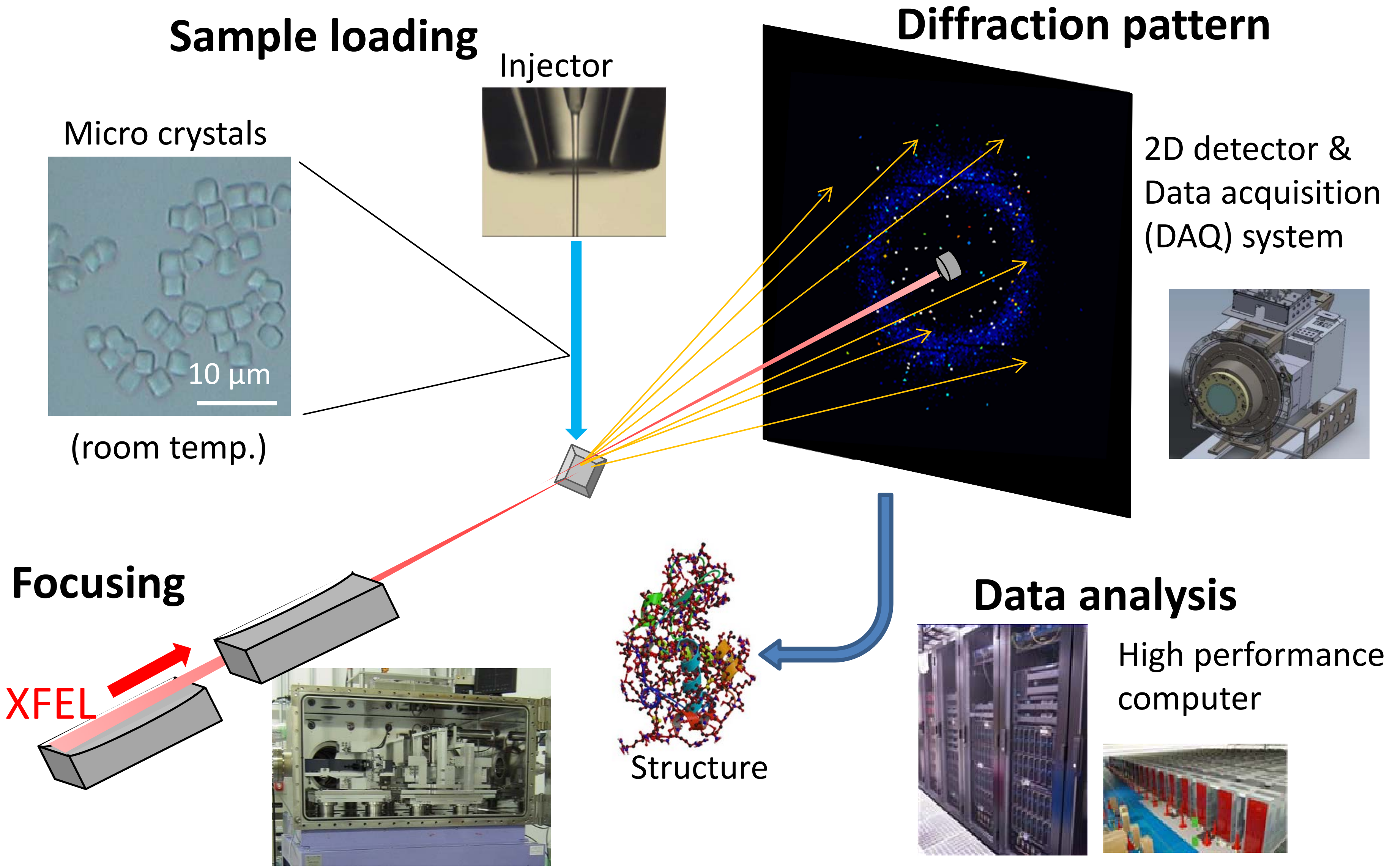
Hirata et al., *Nature Methods* **7**, 735 (2014).



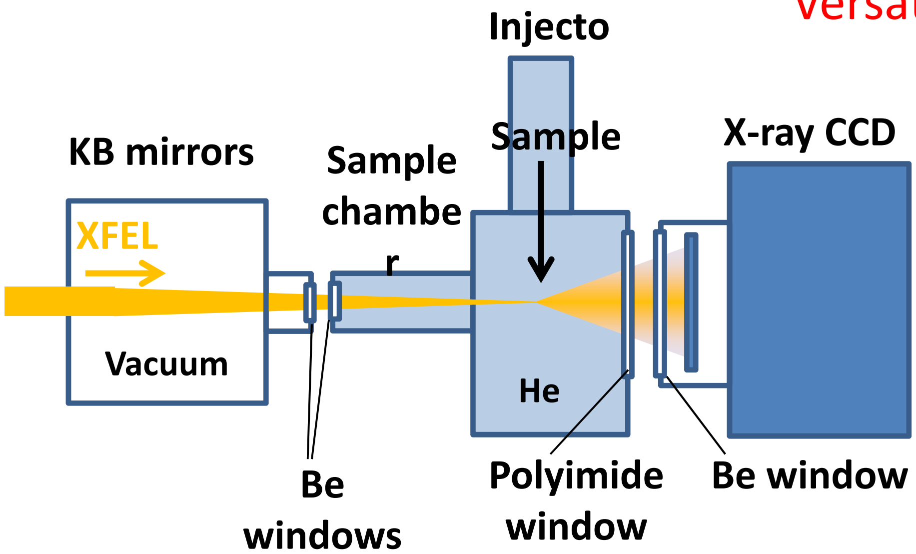
Damage-free structure



Serial femtosecond crystallography (SFX)

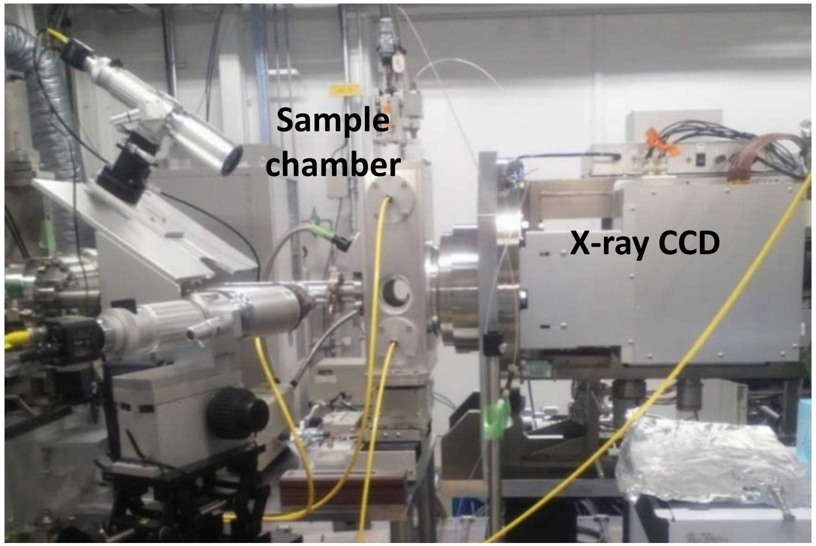


DAPHNIS (Diverse Application Platform for Hard x-ray diffractioN In SACLA)

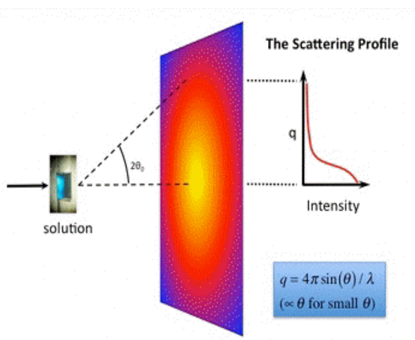


Versatile platform for SFX (diffraction/scattering)

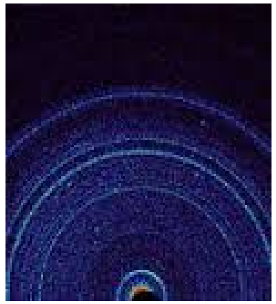
- Components are separated to be handled easily.
- Operated under atmospheric pressure (He atmosphere)
- Flexible system adaptable to various sample injectors
- Extensible to pump-probe measurement



Also applicable to diffraction & scattering experiments for variety of solution and solid samples with P&P capability.

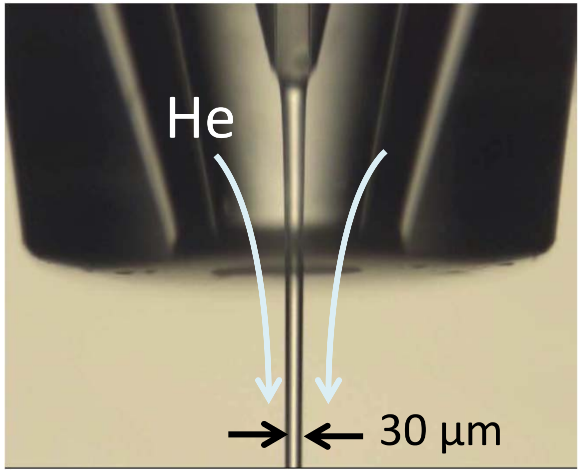
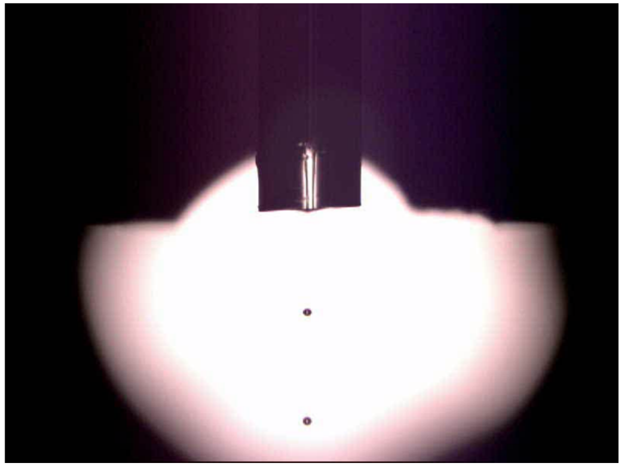


solution scattering

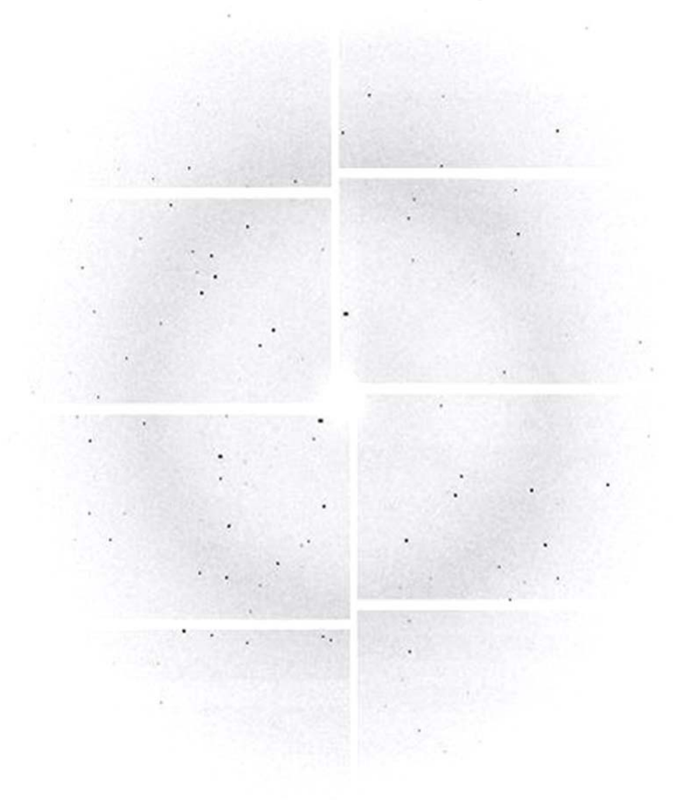


powder diffraction

Fluid injectors

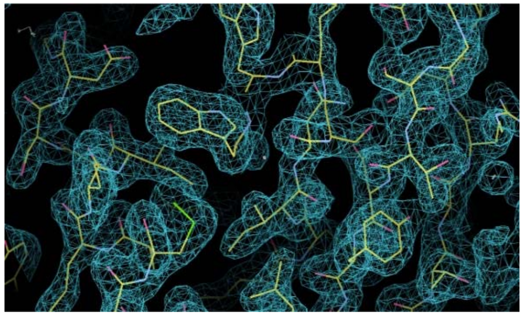
Continuous beam	High-viscosity sample	Droplets
		
Flow rate = ～ 0.4 mL/min	～0.5 µL/min	～0.1 µL/min
Proteins : ～100 mg	Proteins : ～0. 1 mg	Proteins : <0.1 mg

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%)

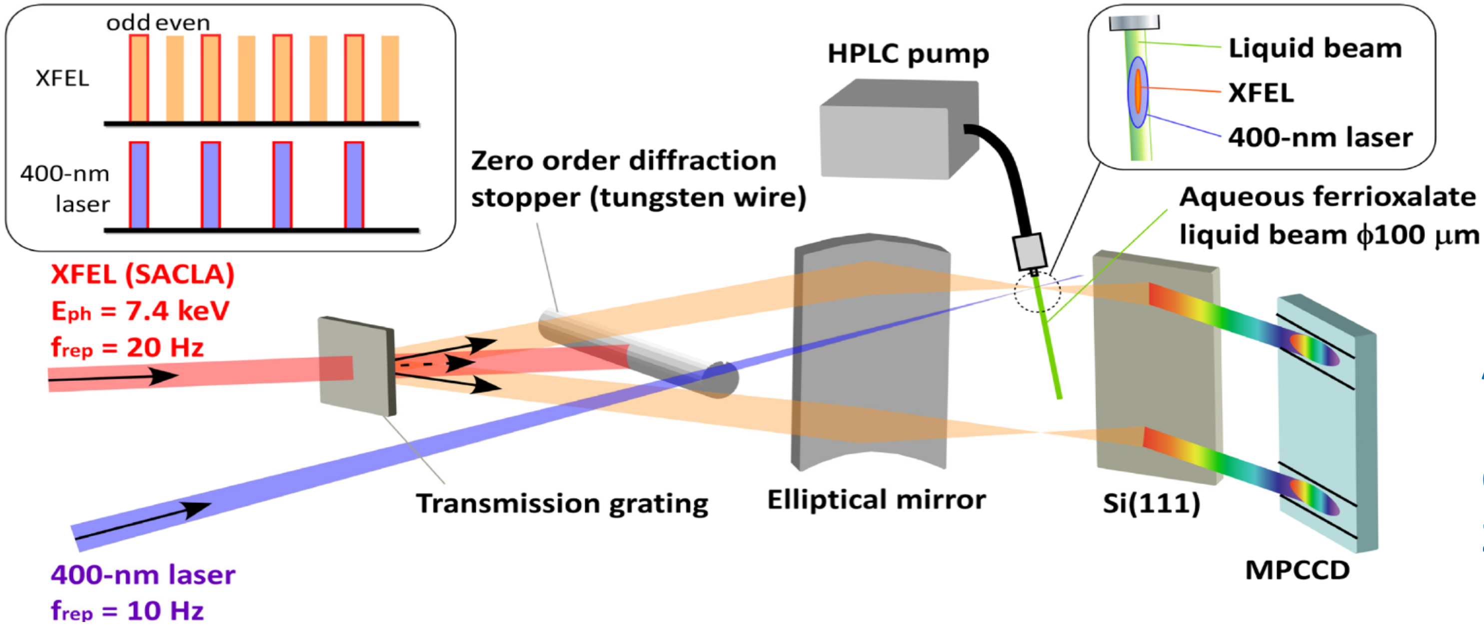
Indexable images: 13,912 (20%)

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

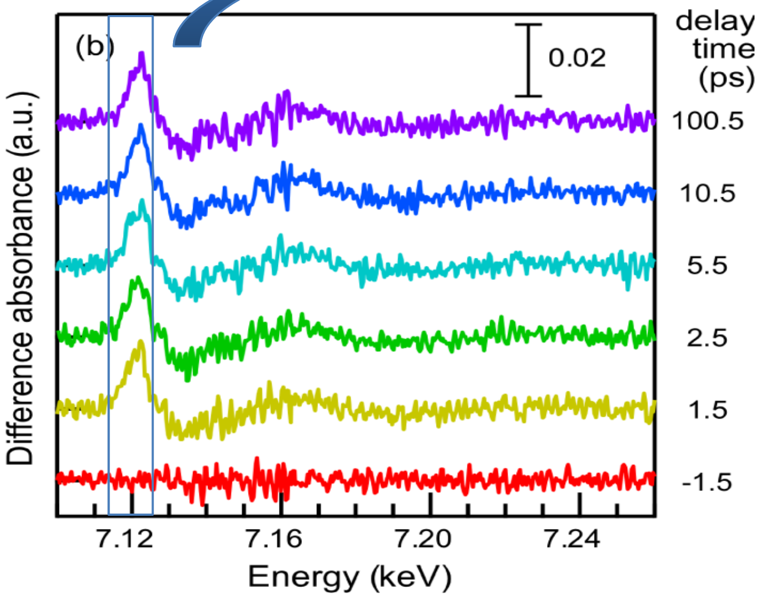
Time-resolved XAS for ultrafast chemistry



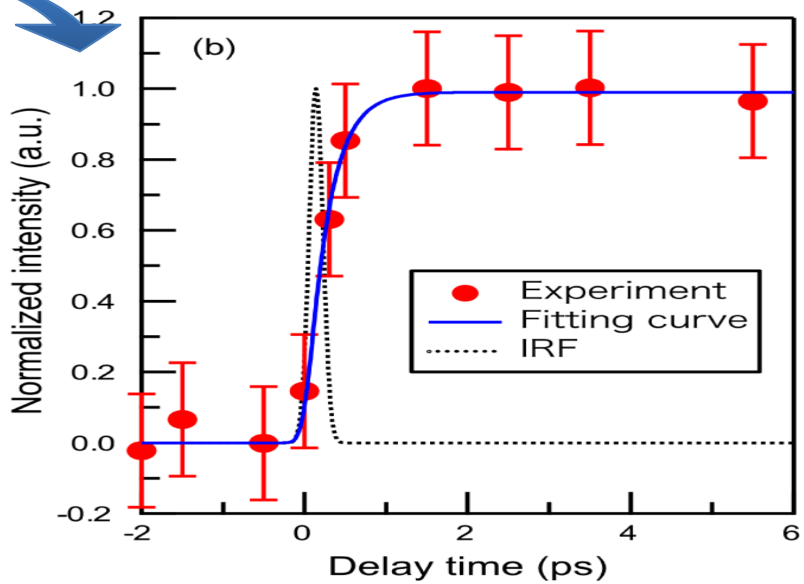
Katayama et al APL **103**, 131105

Obara et al, OE **22**, 1105 (2014

Differential spectrum



Temporal evolution



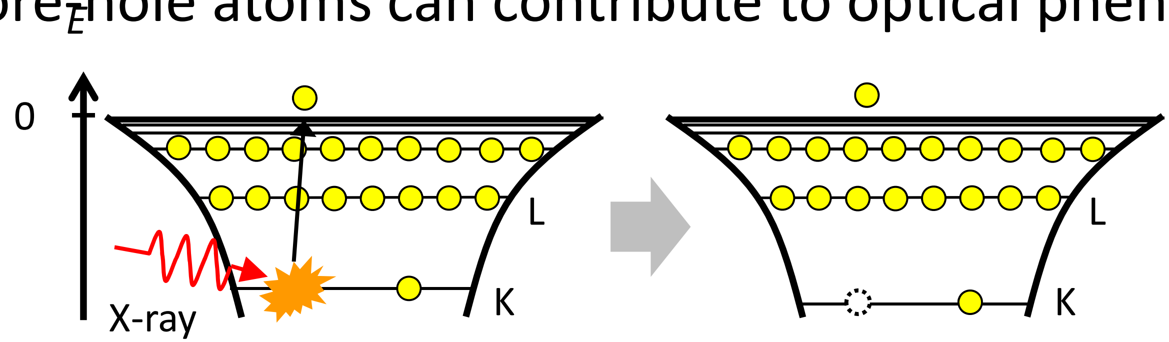
Light-matter interaction under intense X-ray irradiation

- Nonlinear X-ray optics
  - Multi-photon processes
  - Novel optical responses in the X-ray region
  - X-ray amplification

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

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

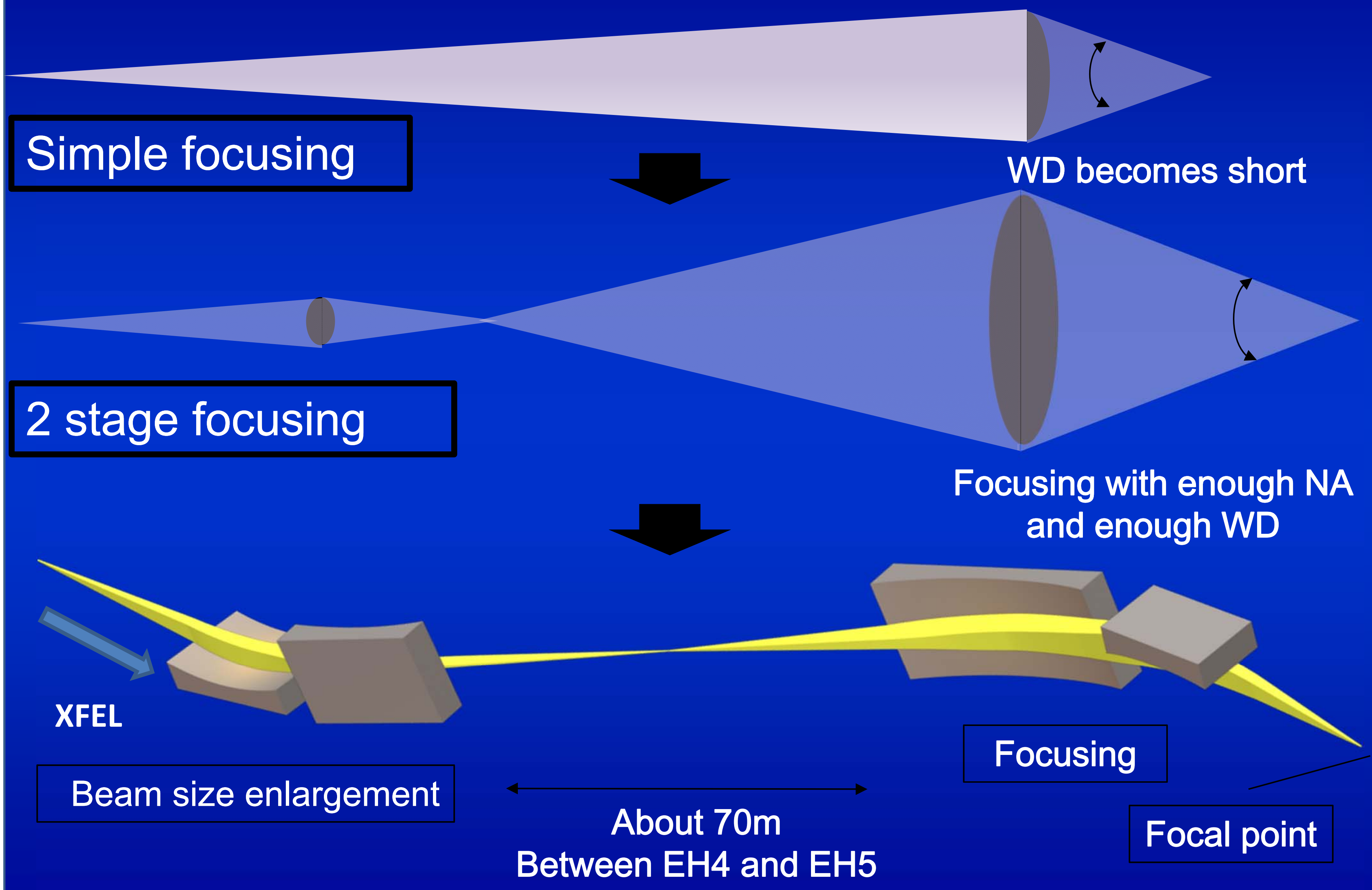
Pulse energy ~10^{-4} J

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

Pulse duration ~10^{-14} s

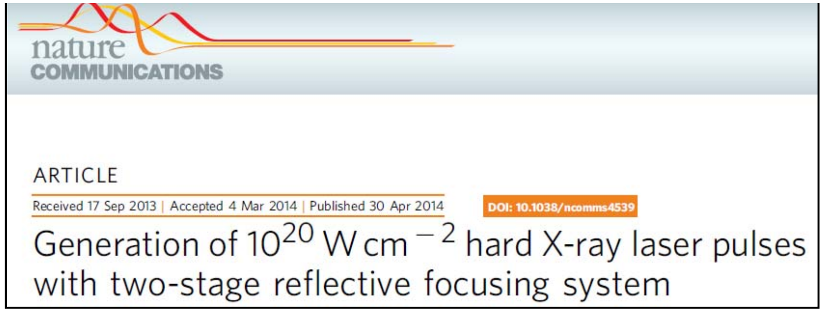
Focusing XFEL down to < 100 nm, an intensity reaches 10^{20} W/cm^2

2-stage focusing for creating nanometer spot



Results

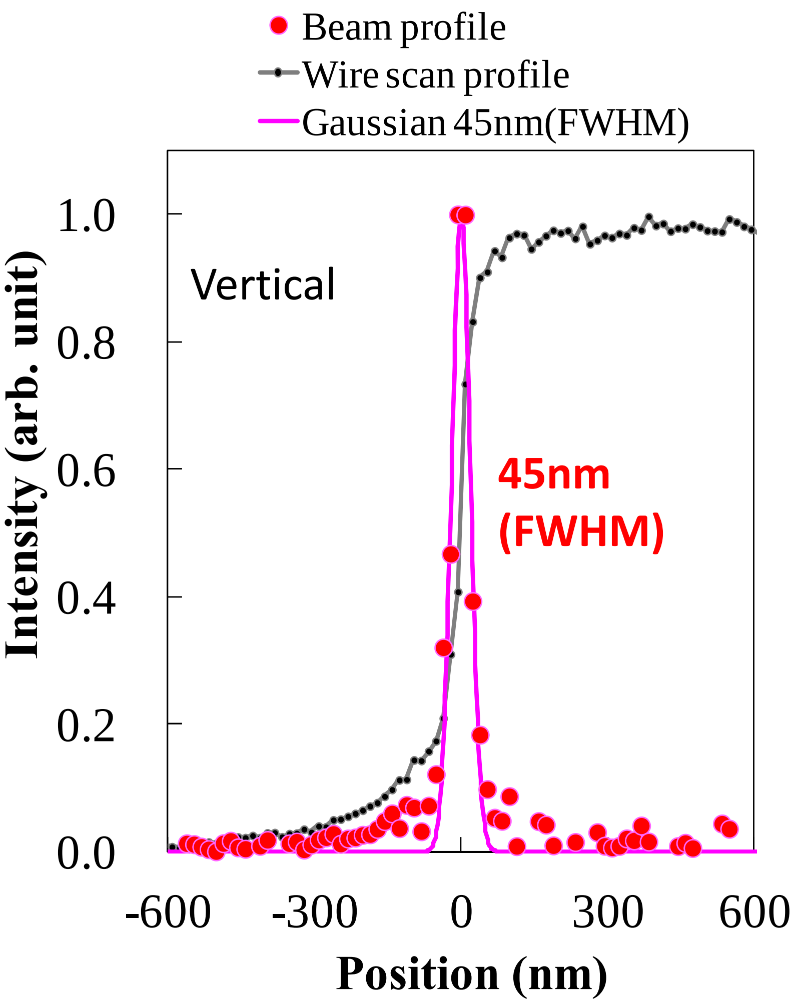
Mimura et al,
Nature Comm., DOI: 10.1038/ncomms 4539 (2014)

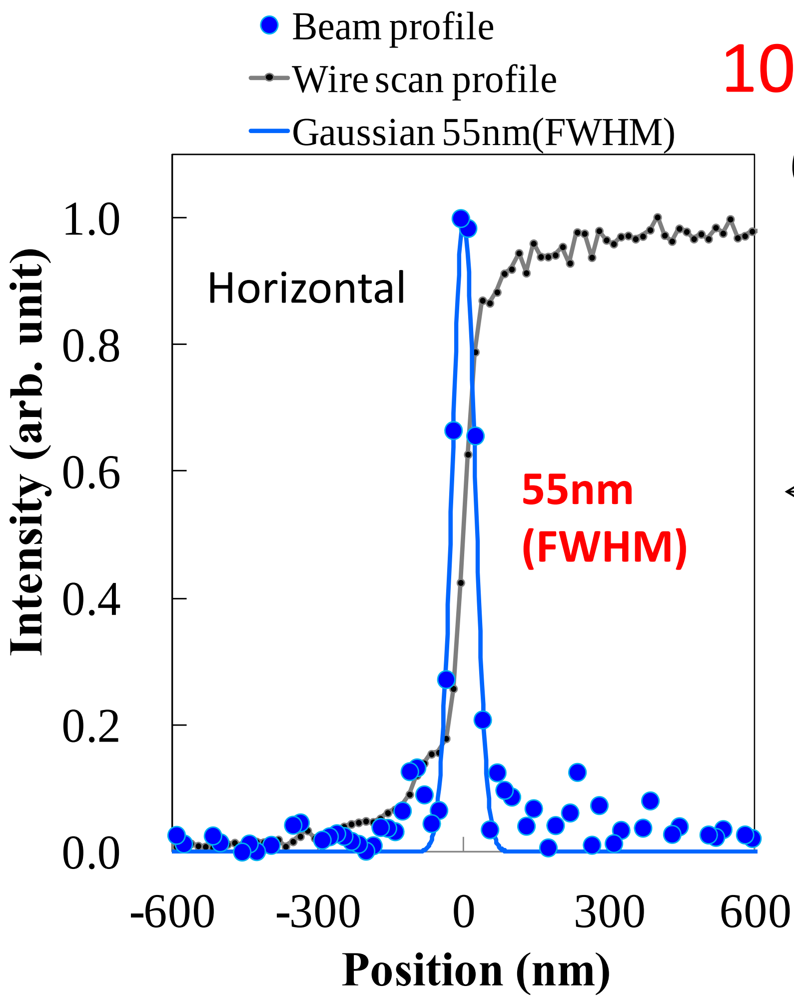

nature
COMMUNICATIONS

ARTICLE

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

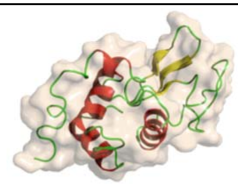
Generation of 10^{20} W cm^{-2} hard X-ray laser pulses with two-stage reflective focusing system





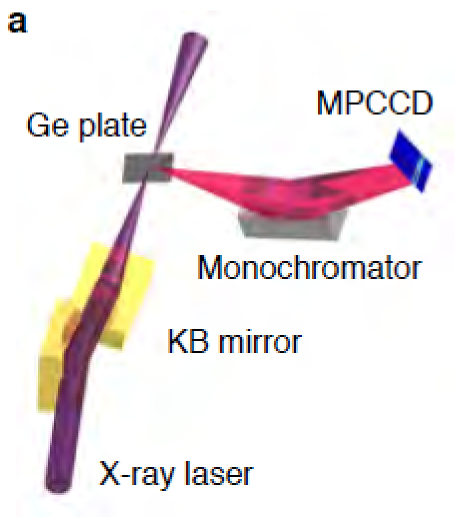
10^{20} W/cm^2
@ 10 keV

~10 nm



Protein

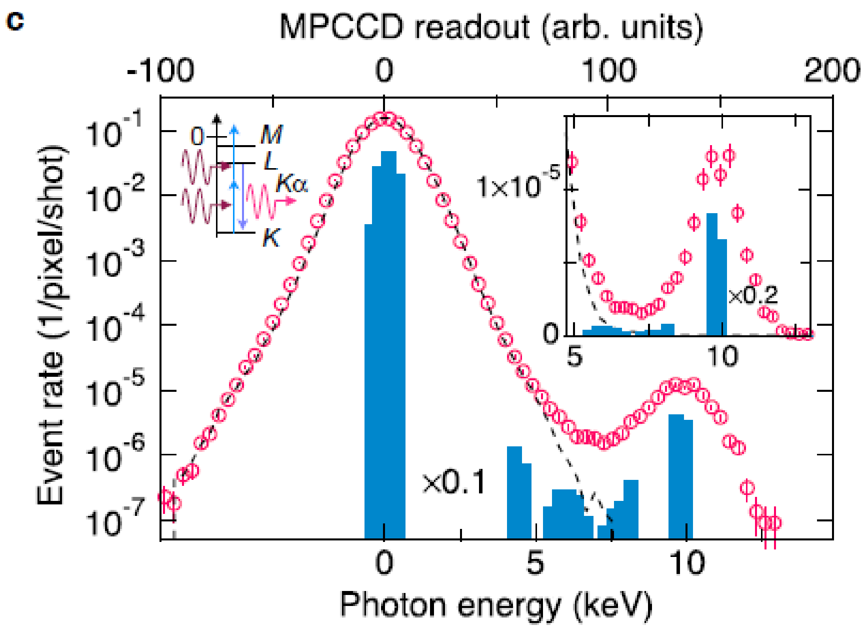
Application: Two-photon absorption



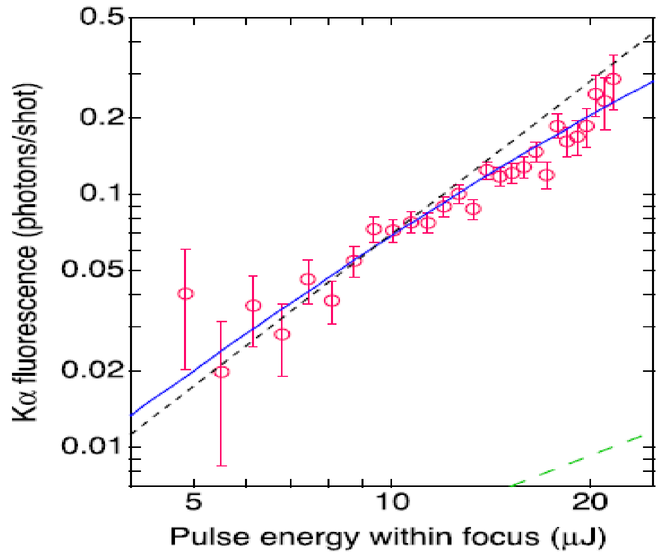
50 nm focusing ⇒ ~10^{20} W/cm^2

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

Tamasaku-san
Nature Photon (2014)



Intensity dependence: close to quadratic



Summary

- Novel properties and sciences of XFEL
  - Ultra-brilliant, ultra-short, and coherent
  - Beyond static, statistical, perturbative 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 snapshots of samples
  - Damage-free crystallography
  - X-ray-matter interaction under ultra-high photon flux
  - Pump-probe measurement

Outlook

- Upgrade of SACLA
  - Self seeding
  - Multi-beamline operation (BL1, BL2, BL3)

- New instruments
  - Ultimate focusing
  - High power lasers
  - Detector upgrade