Cheiron School, SPring-8, Japan
19th September, 2015

Atomic and Molecular Physics at SACLA

Kiyoshi Ueda

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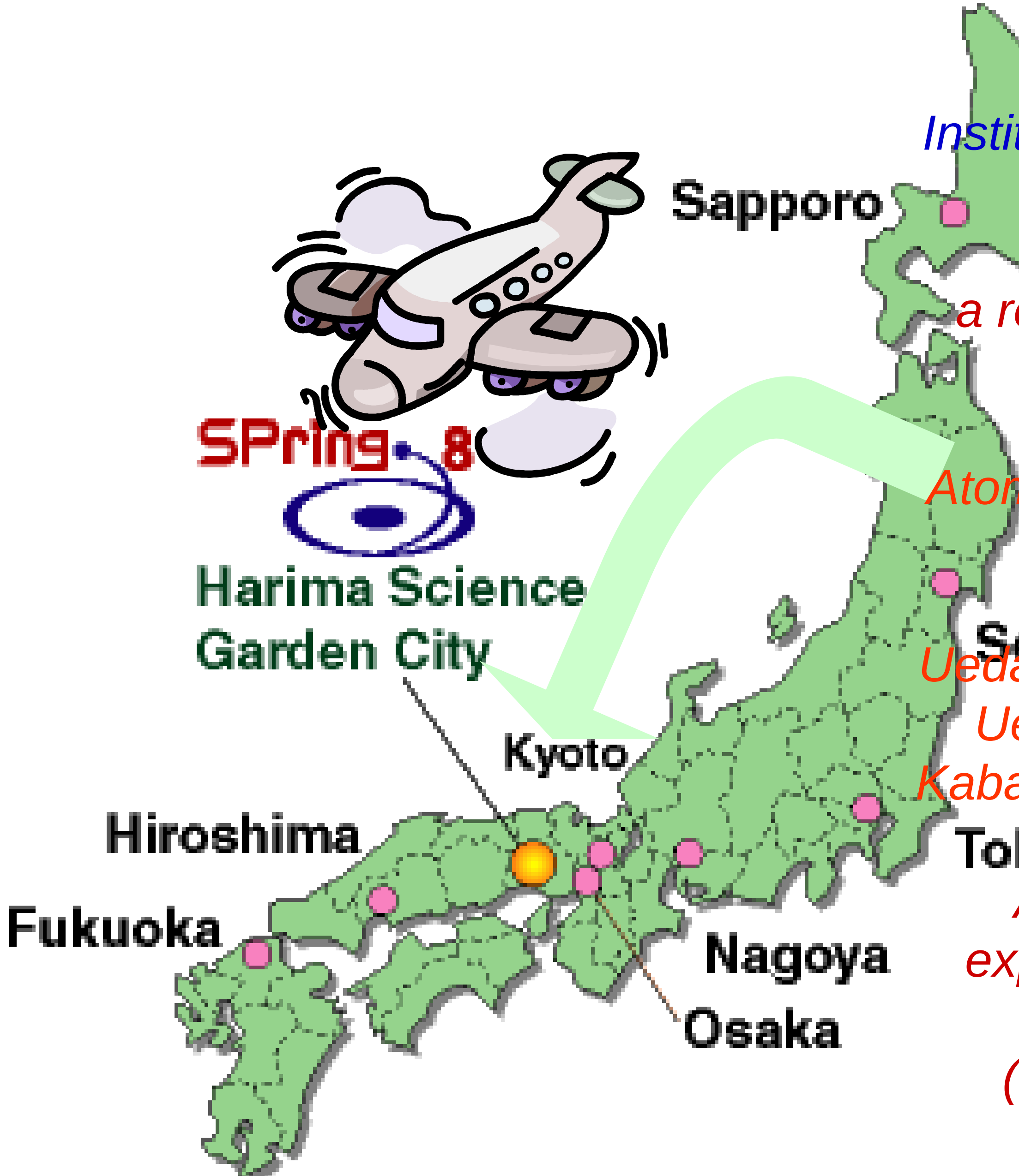
Introduction of myself

Prof. Kiyoshi Ueda
at
Tohoku University （Sendai）
Institute of multidisciplinary research
for advanced materials

Leading
a research group for electron and molecular dynamics



Atomic and molecular science using Synchrotron Radiation
Ueda, JPB 36, R1 (2003);
Ueda & Eland, JPB 38, S839 (2005);
Ueda, JPSJ 75, 032001 (2006);
Kabachnik et al. PR 451, 155 (2007).

Application of the SR-based experimental techniques to laser experiments
(femtosecond laser and FEL)

Outline

Introduction to XFEL science

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– Deep inner-shell multi-photon ionization of Ar and Xe atoms
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– IR-probe experiment of XFEL-ignited nanoplasma dynamics

Summary and outlook

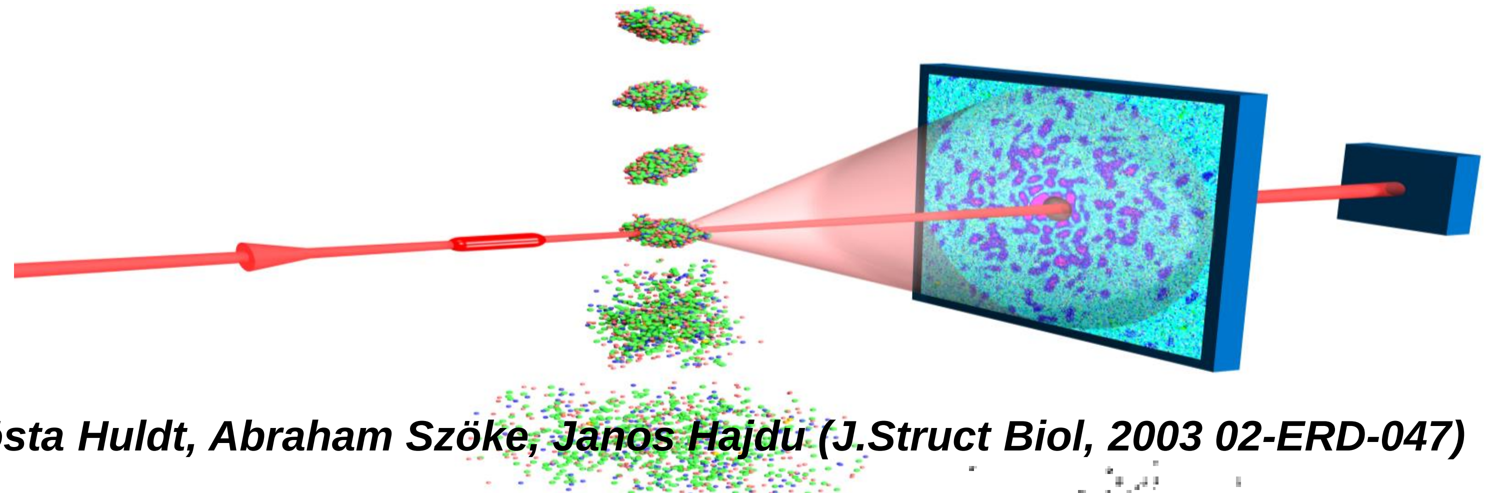
EUV-X FELs in the world

Swiss FEL (2017), Korean FEL, Shanghai FEL, etc., are coming!

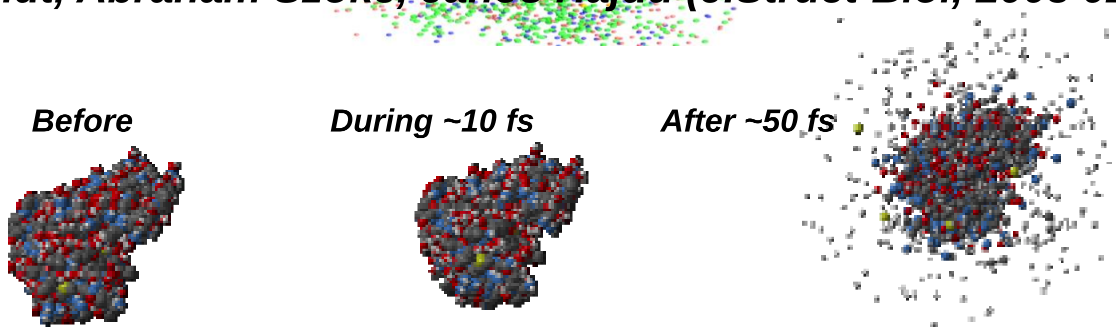
Characteristic properties of FEL pulses

Coherent, intense, and ultra-short pulses at short wavelengths (EUV to X –rays)

Coherent X-ray imaging of non-crystalized samples



Gösta Huldt, Abraham Szöke, Janos Hajdu (J.Struct Biol, 2003 02-ERD-047)

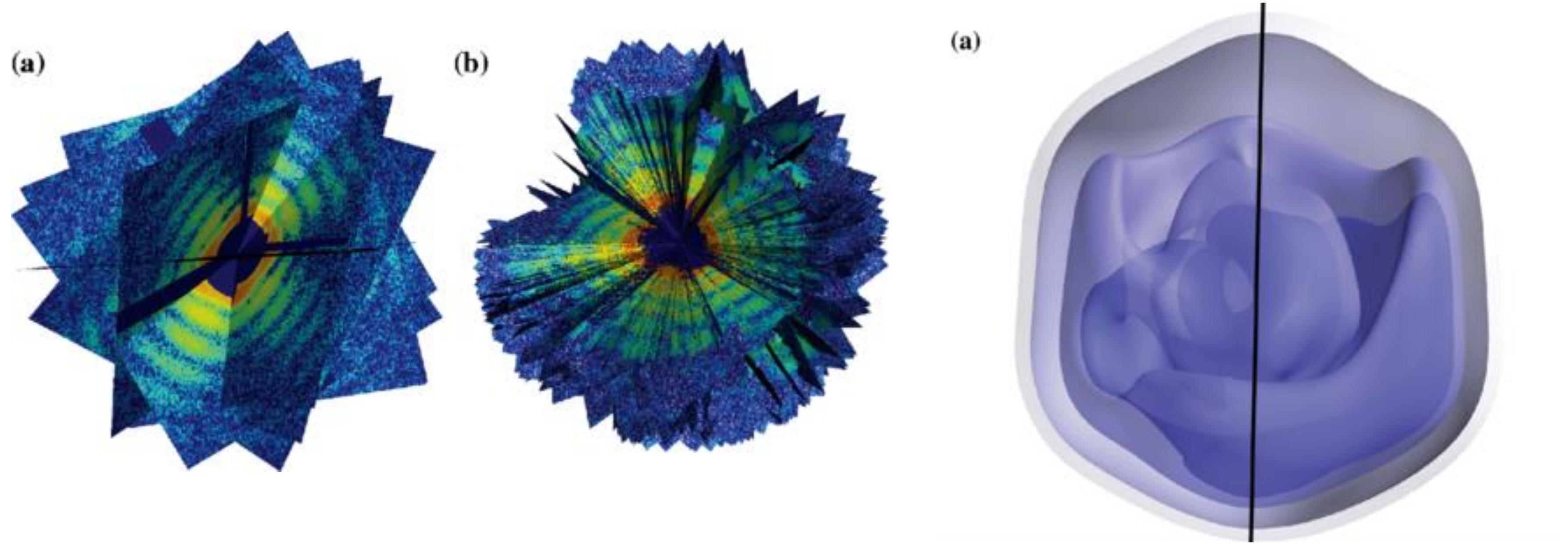


Neutze, Wouts, van der Spoel, Weckert, Hajdu   Nature 406, 752 (2000)

Single Mimivirus Particles Intercepted and Imaged with an X-ray laser

Seibert et al.   Nature **470**, 78–81 (2011) *only 2D…..*

Coherent X-ray imaging of non-crystalized samples



Three-Dimensional Reconstruction of the Giant Mimivirus Particle with an X-Ray Free-Electron Laser

Ekeberg *et al.* Phys. Rev. Lett. **114**, 098102 (2015)

Can we get 3D image from a single shot data?

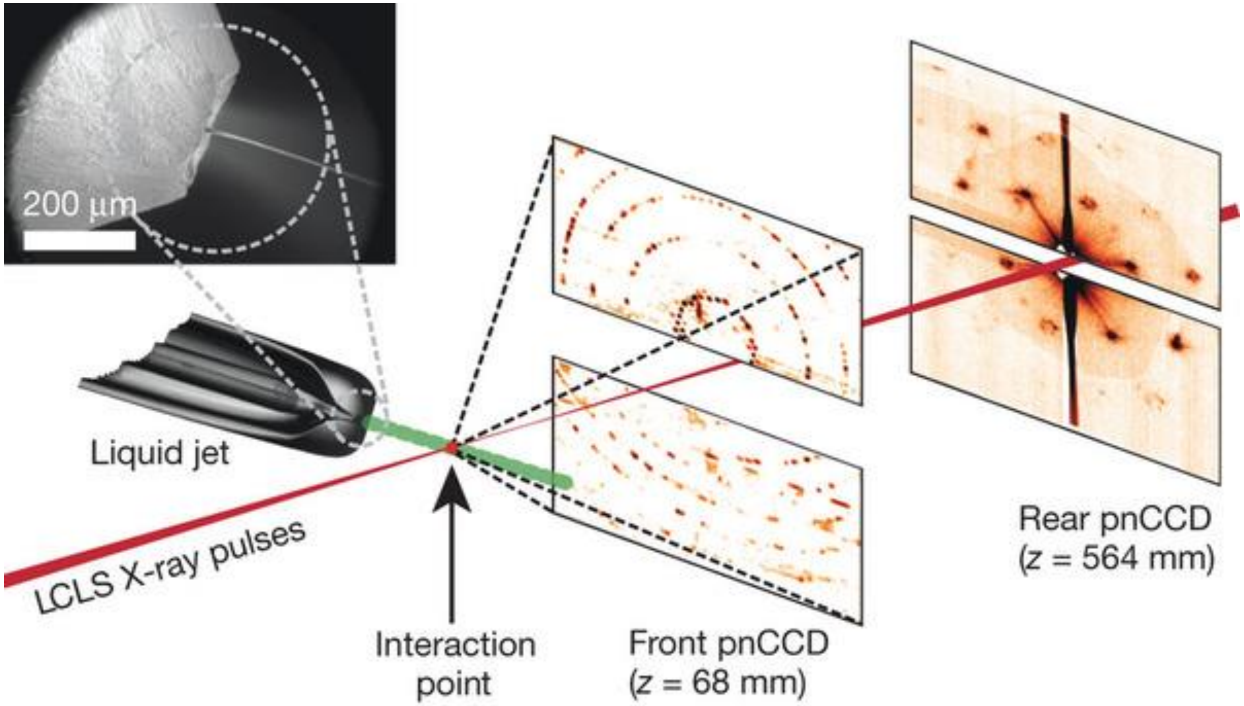
In principle, “Yes” but in practice….

Characteristic properties of FEL pulses

Intense and ultra-short pulses at **X –rays**

Why X-rays? structure determination at atomic resolution

*Femtosecond X-ray Protein Nano-crystallography, Chapman et al., Nature **470,** 73–77 (2011).*



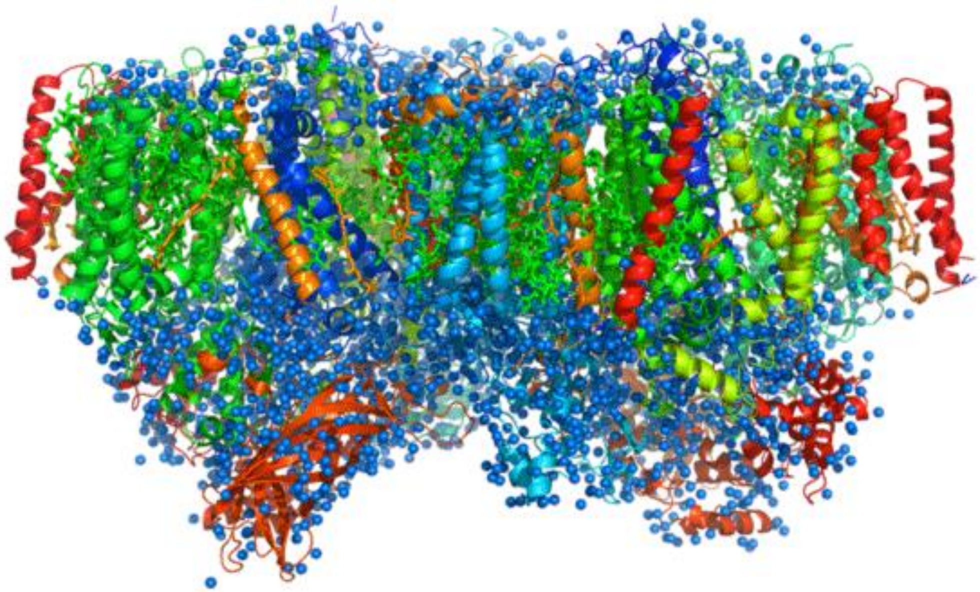
*High-Resolution Protein Structure Determination by Serial Femtosecond Crystallography, Boutet et al. Science **337,** 362 (2012).*

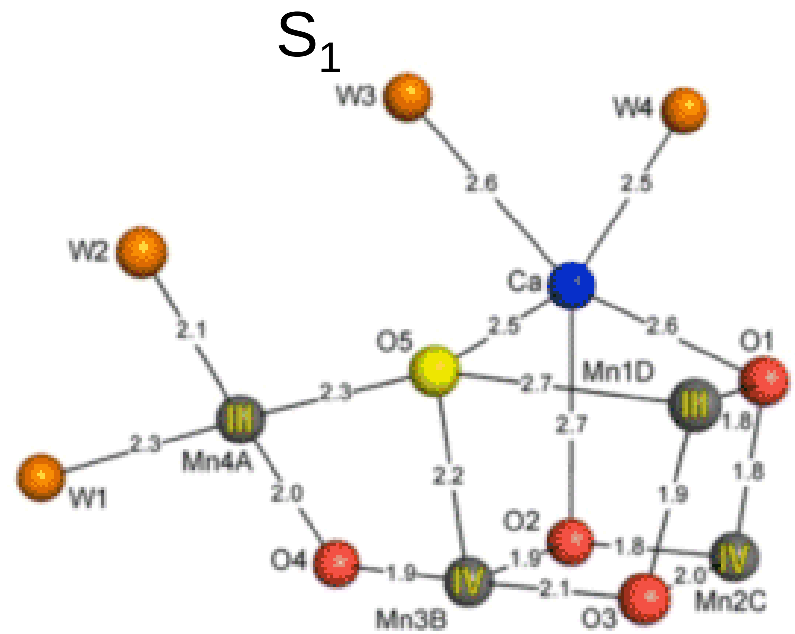
*Natively Inhibited Trypanosoma brucei Cathepsin B Structure Determined by Using an X-ray Laser, Redecke et al. Science **339**, 227 (2013).*

Photo-system II

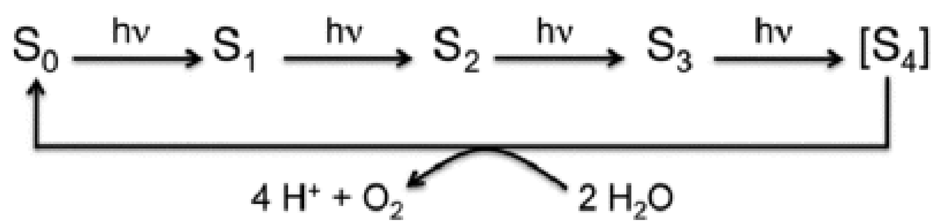
Native structure of photosystem II at 1.95 Å resolution revealed by a femtosecond X-ray laser (SACLA)

Suga et al, Nature (2014), doi:10.1038/nature13991





"Determination of damage-free crystal structure of an X-ray sensitive protein using an XFEL"
Nature Methods (2014), doi:10.1038/NMETH.2962



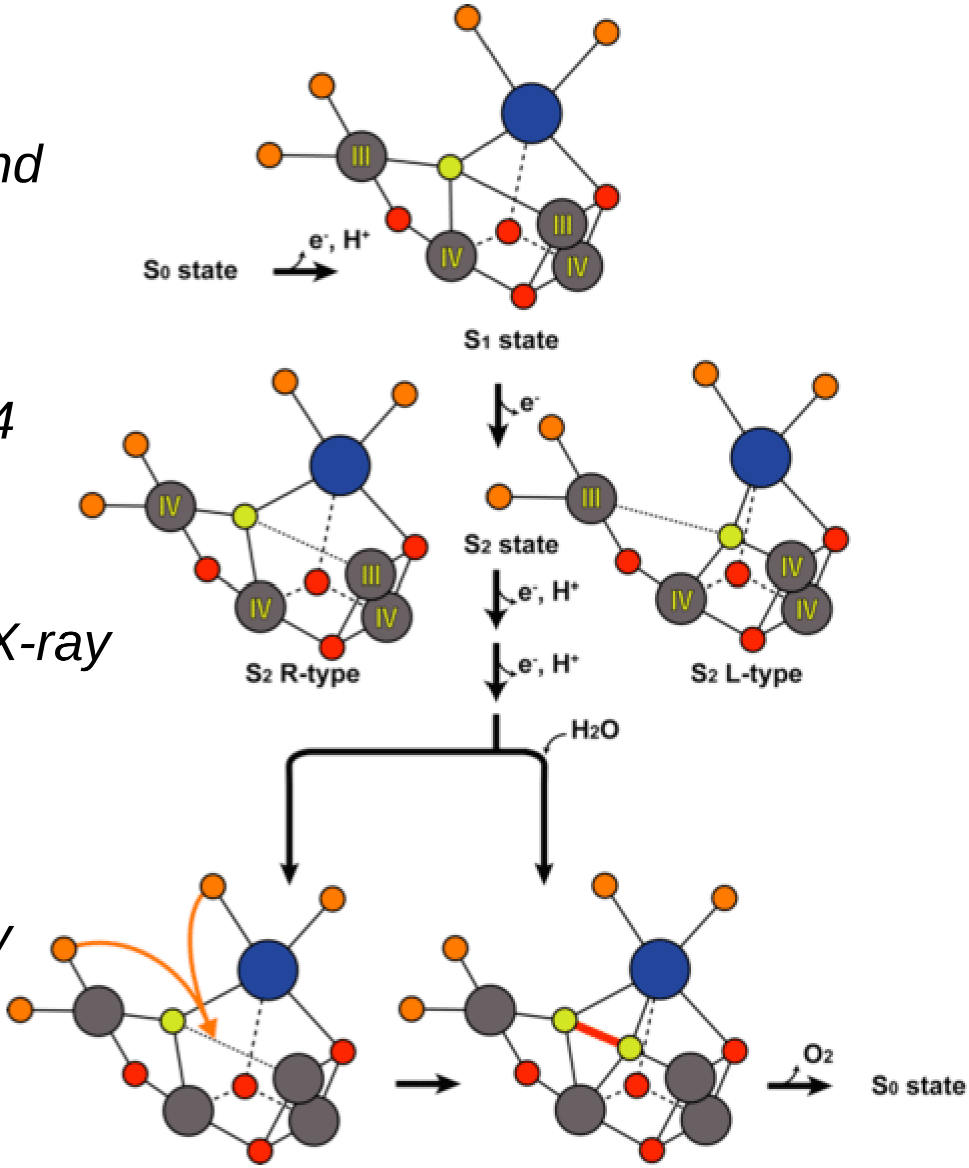
SR results (Nature 2011) had radiation damage….

Dynamic behavior of photo-system II

"The Mn_4Ca photosynthetic water-oxidation catalyst studied by simultaneous X-ray spectroscopy and crystallography using an X-ray free-electron laser"
Rosalie Tran et al
Phil. Trans. R. Soc. B 369 20130324 (2014) doi: 10.1098/rstb.2013.0324

"Taking snapshots of photosynthetic water oxidation using femtosecond X-ray diffraction and spectroscopy"
Jan Kern et al
Nature Comm. 5. 4371 (2014)

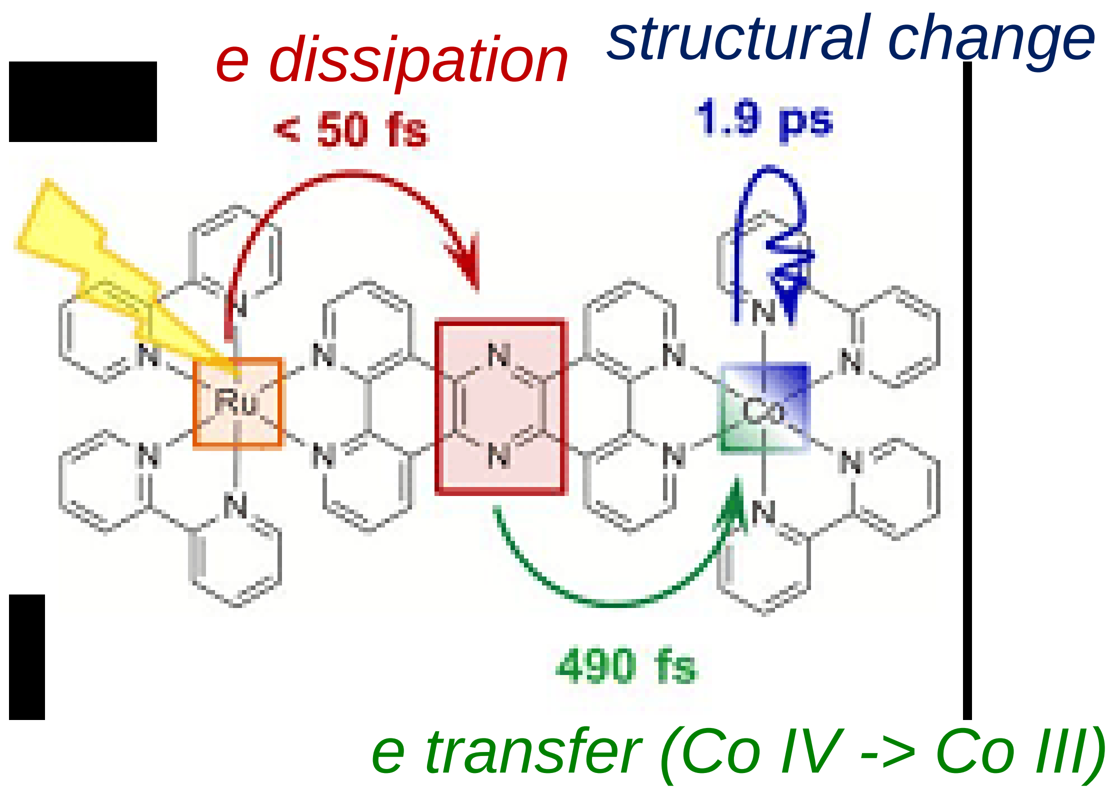
"Serial time-resolved crystallography of photosystem II using a femtosecond X-ray laser"
C. Kupitz et al
Nature (2014)
doi:10.1038/nature13453



Suga et al, Nature (2014), doi:10.1038/nature13991

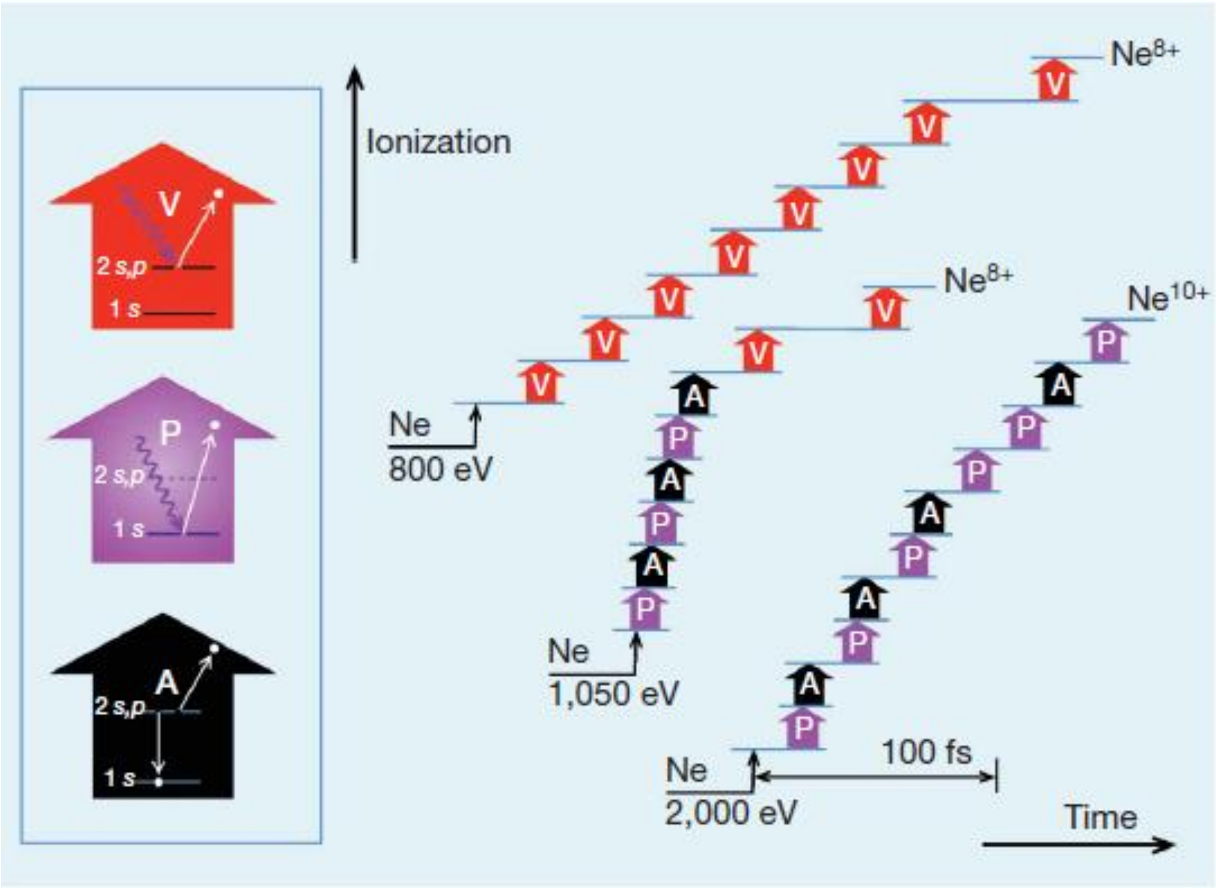
Towards artificial photosynthesis

Visualizing the non-equilibrium dynamics of photo-induced intramolecular electron transfer with femtosecond X-ray Pulses (SACLA) Canton et al. *Nature Comm. 6, 6359 (2015)*



Characteristic properties of FEL pulses

Intense 10^{14} W/cm^2 (EUV) - 10^{20} W/cm^2 (X)



Femtosecond electronic response of atoms to ultra-intense x-rays
*L. Young et al., Nature **466**, 56 (2010).*

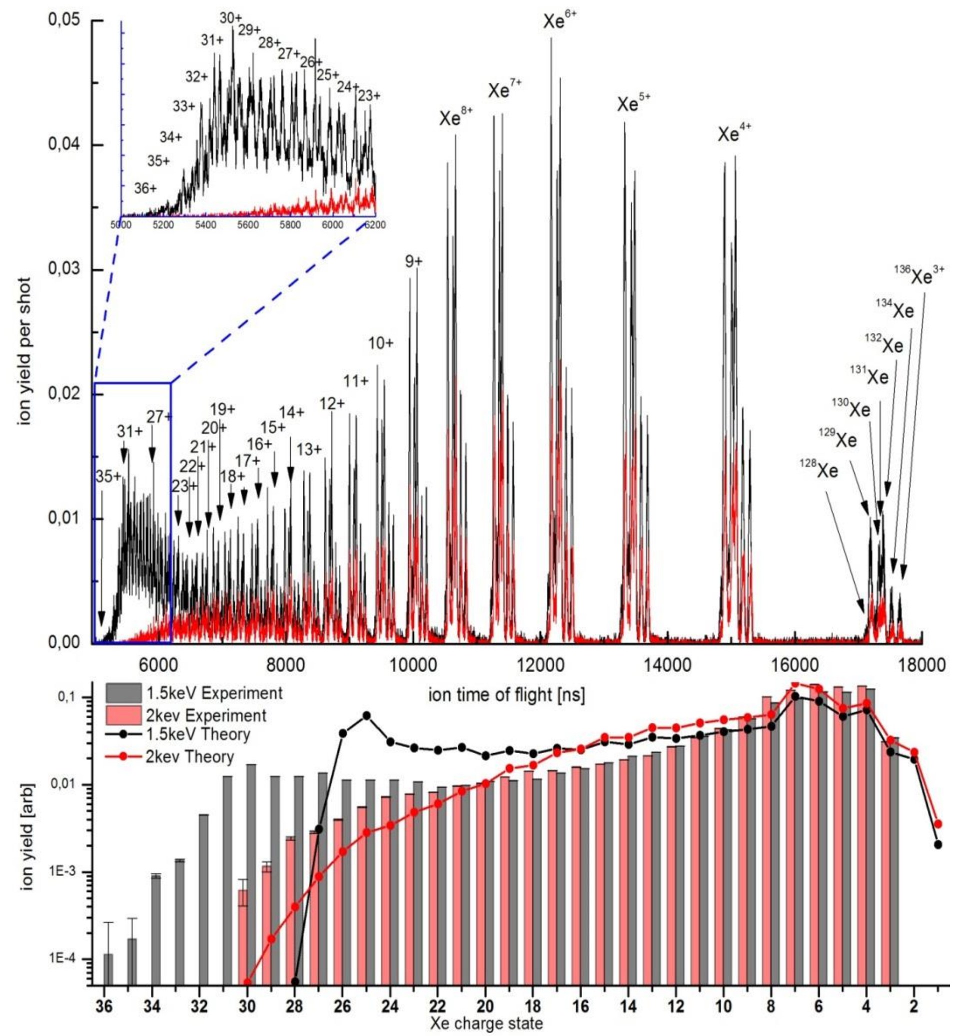
One LCLS pulse at 2 keV can remove all ten electrons from the neon atom.

The pulse is so intense that it causes electronic damage to the sample.

Non-linear X-ray atomic Physics

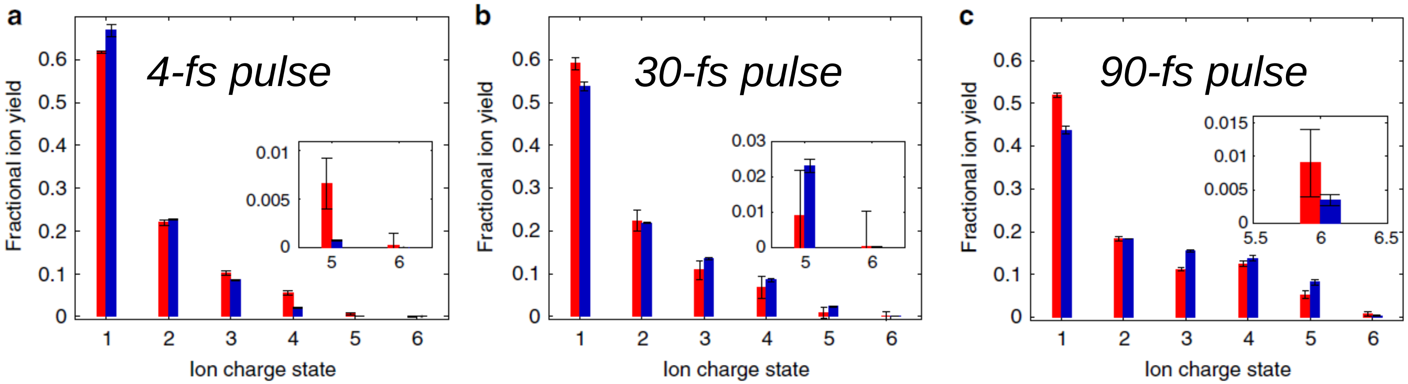
Ultra-Efficient Ionization of Heavy Atoms by Intense X-Rays

B. Rudek, S-K. Son, L. M. Foucar, S. W. Epp, B. Erk, R. Hartmann, M. Adolph, R. Andritschke, A. Aquila, N. Berrah, C. Bostedt, J. Bozek, N. Coppola, F. Filsinger, H. Gorke, T. Gorkhover, H. Graafsma, L. Gumprecht, A. Hartmann, G. Hauser, S. Herrmann, H. Hirsemann, P. Holl, A. Hömke, L. Journel, C.Kaiser, N. Kimmel, F. Krasniqi, K-U. Kühnel, M. Matysek, M. Messerschmidt, D. Miesner, T. Möller, R. Moshammer, K. Nagaya, B.Nielsson, G. Potdevin, D. Pietschner, C. Reich, D. Rupp, R.Santra, G. Schaller, I. Schlichting, C. Schmidt, F. Schopper, S. Schorb, C-D. Schröter, J. Schulz, M. Simon, H. Soltau, L. Strüder, ***K. Ueda****, G. Weidenspointner, J. Ullrich, A. Rudenko, and D. Rolles*
Nature Photonics 6, 858 (2012).

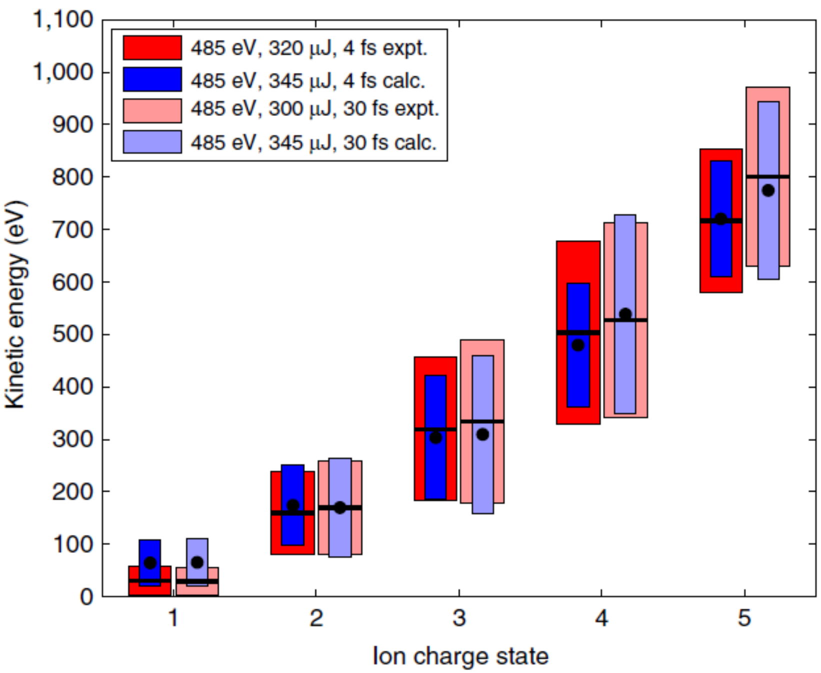


X-ray induced Coulomb explosion

Femtosecond X-ray-induced explosion of C_{60} at extreme intensity (hν=485 eV)

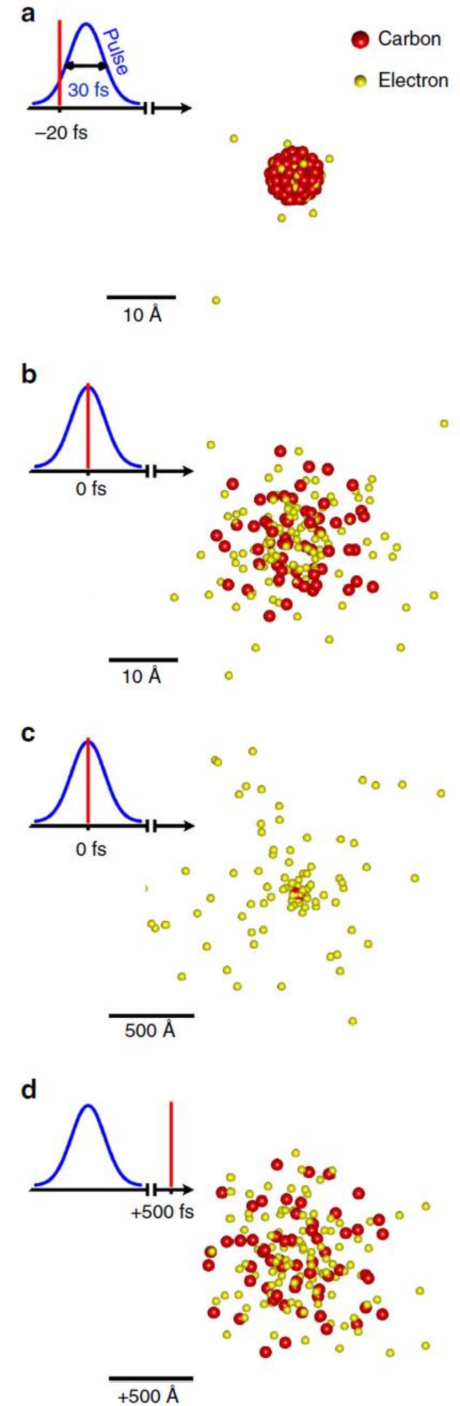


Charge state distributions



Kinetic energy distributions

B.F. Murphy, T. Osipov, Z. Jurek, L. Fang, S.-K. Son, M. Mucke, J.H.D. Eland, V. Zhaunerchyk, R. Feifel, L. Avaldi, P. Bolognesi, C. Bostedt, J.D. Bozek, J. Grilj, M. Guehr, L.J. Frasinski, J. Glownia, D.T. Ha, K. Hoffmann, E. Kukk, B.K. McFarland, C. Miron, E. Sistrunk, R.J. Squibb, K. Ueda, R. Santra & N. Berrah, NATURE COMMUNICATIONS | 5:4281 | DOI: 10.1038/ncomms5281.



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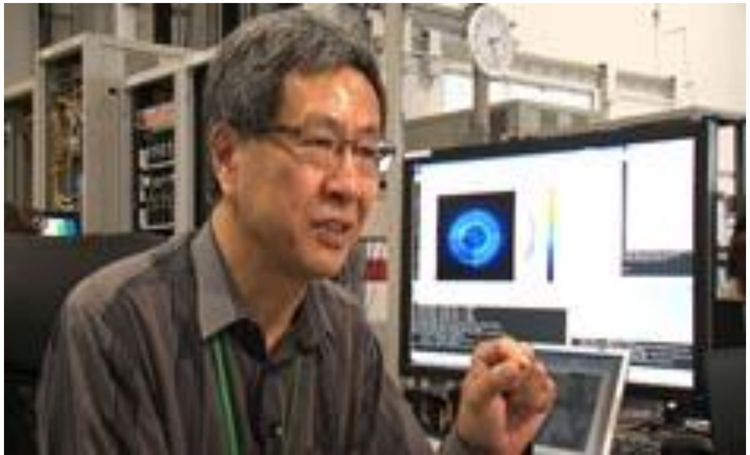
Summary and outlook

SACLA XFEL (lased on 7 June 2011)



on air!

SACLA XFEL

Commissioning beam time: Nov. 2011-Feb. 2012

7-11 Nov. 2011: Detector test (no real FEL beam…)

……

20-24 Feb. 2012: Serial femtosecond crystallography

User beam time started in March 2012

Serial femtosecond x-ray crystallography: phasing

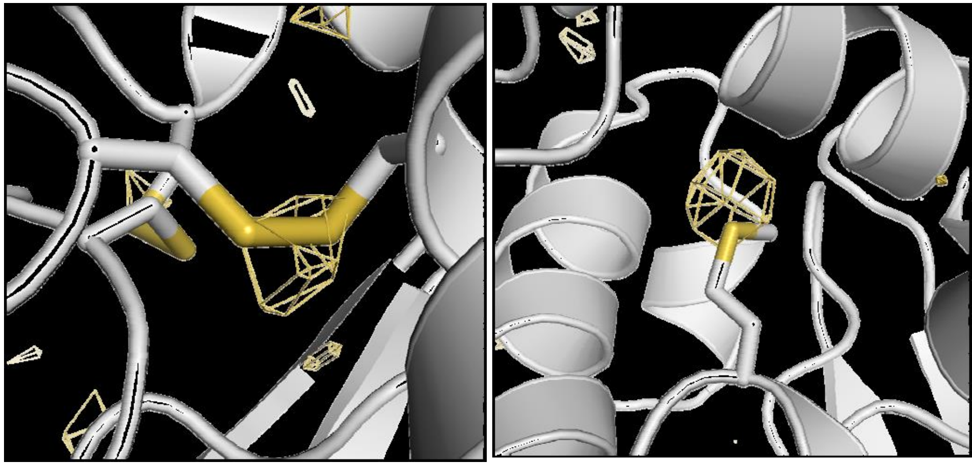
Phasing of the serial femtosecond x-ray crystallography had been relying on molecular replacements…

If the structure is completely unknown, phasing approaches make use of anomalous dispersion in the scattering signals from specific atoms.

Anomalous signal from S atoms in protein crystallographic data from an X-ray free-electron laser

T.R.M. Barends, L. Foucar, R.L. Shoeman, …. K. Ueda and I. Schlichting

*Acta Cryst. D **69**, 838-842 (2013).*



One of the first experiments at SACLA

For phasing with a heavy atom, one has to take account of high x-ray intensity

Multi-wavelength anomalous diffraction at high X-ray intensity

*S.-K. Son, H. N. Chapman, and R. Santra, Phys. Rev. Lett. **107**, 218102 (2011).*

However…..

De novo protein crystal structure determination from X-ray free-electron laser data

*T.R.M. Barends et al., Nature **505,** 244 (2014).*

Conventional phasing method based on anomalous dispersion worked….

What should we do with SACLA?

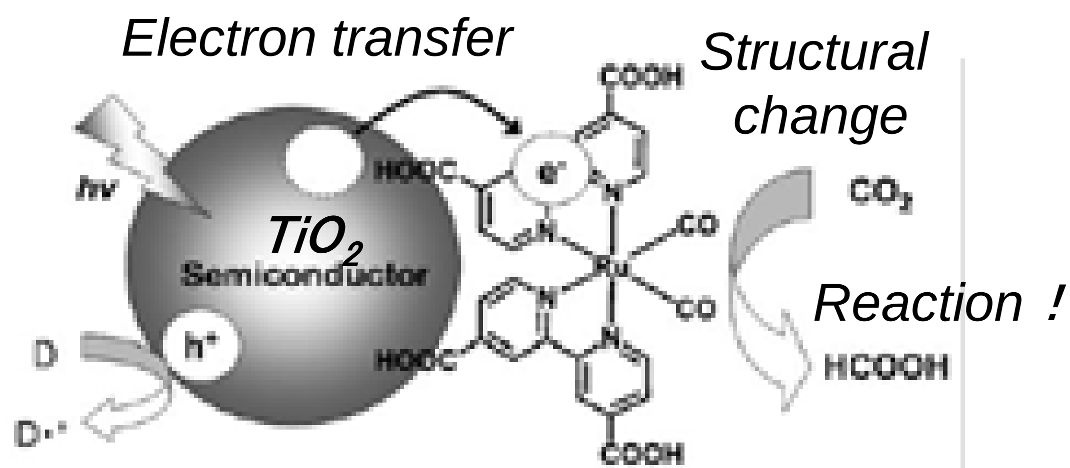
Novel structure determination

- phasing
- radiation damage

understanding dynamic behavior of heavy atoms!

Dynamic structure and light-induced reaction

- Femtosecond electronic and structure changes



Dynamic behavior of metal atoms in strong XFEL fields

Sato et al. Angew. Chem. ***49****, 5101 (2010)* （Toyota）

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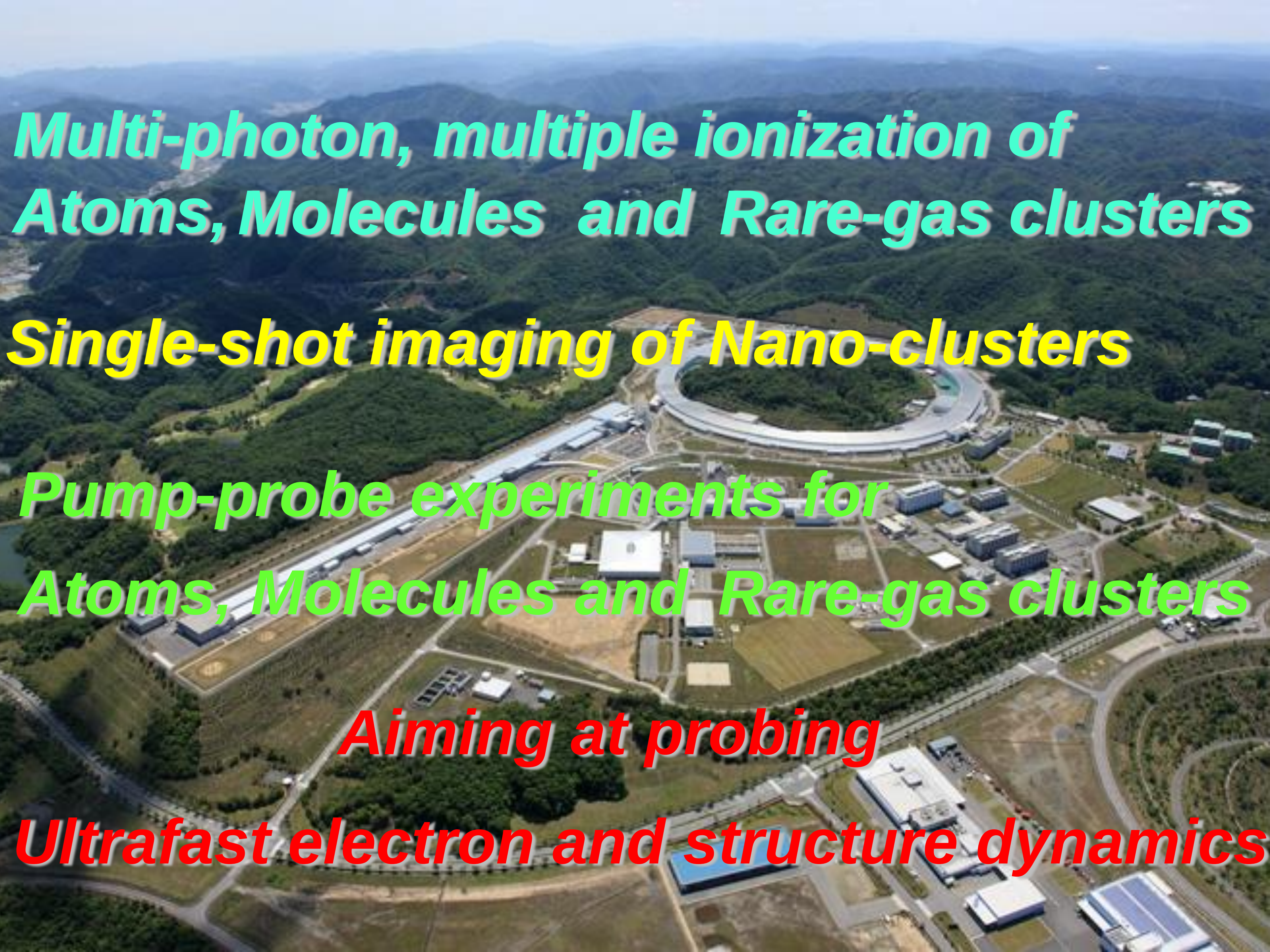


Multi-photon, multiple ionization of Atoms, Molecules and Rare-gas clusters

Single-shot imaging of Nano-clusters

Pump-probe experiments for Atoms, Molecules and Rare-gas clusters

Aiming at probing

Ultrafast electron and structure dynamics

SACLA atomic & molecular beam times

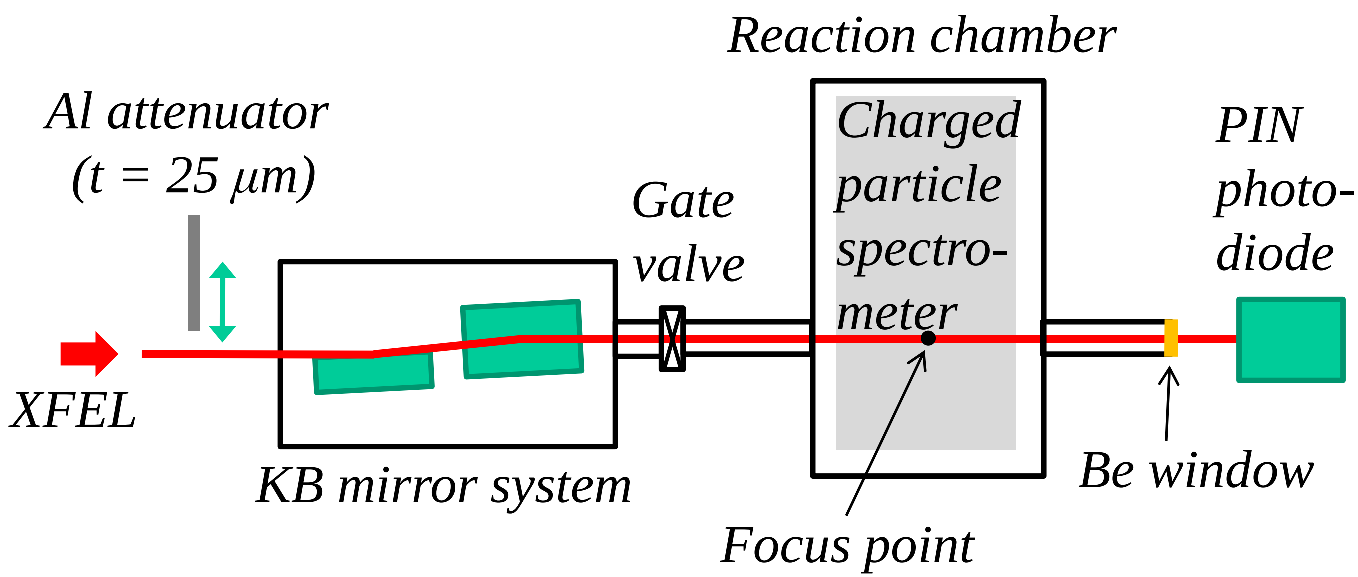
First two beam times in 2012:
Atoms, molecules and atomic clusters

Third beam time in 2013: pump-probe, failed

Fourth beam time in 2014:
Single-short imaging of giant xenon clusters

Fifth and sixth beam times in 2014:
Pump-probe experiment on clusters
Seventh beam time in 2015: pump-probe, failed

Experimental configuration @ SACLA BL3 EH3



XFEL pulses

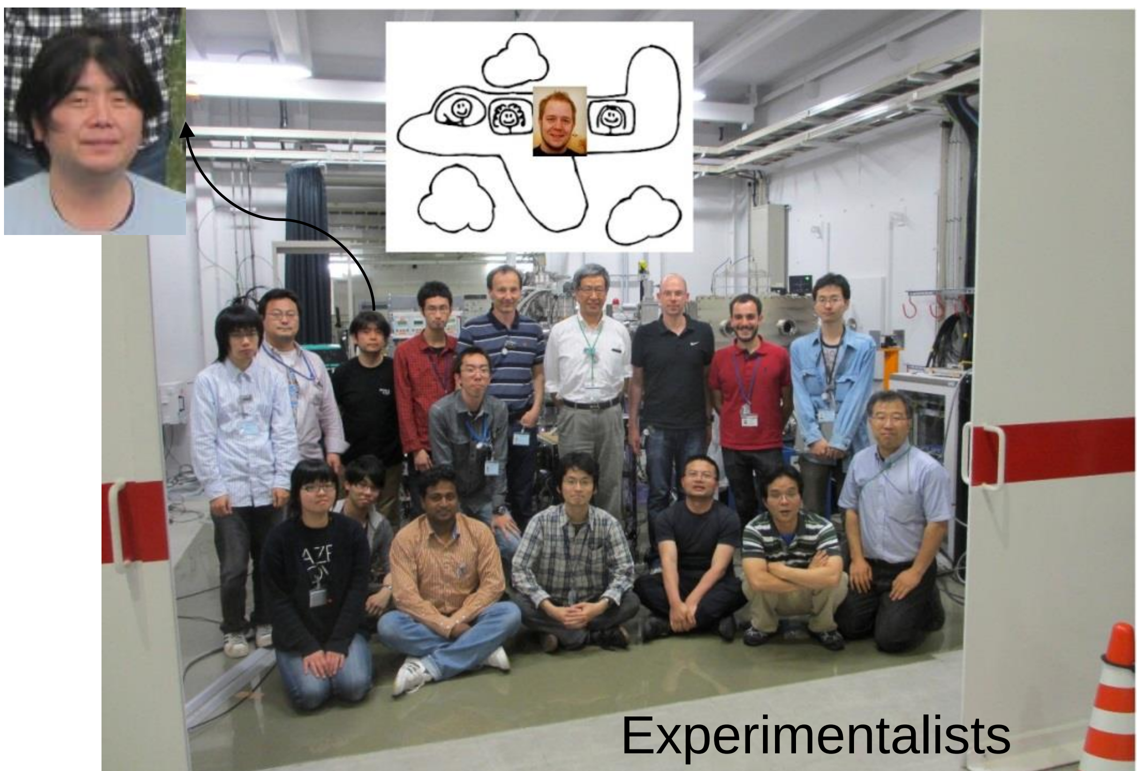
Photon energy: 5 and 5.5 keV
(Wavelength: 0.25 and 0.22 nm)
Band width: ~60 eV (FWHM)
Repetition: 10-30 Hz
Pulse energy before KB mirror:
~240 μJ (~3 ×10^{11} photons) @5.5keV
Fluctuation of pulse energy:
±25% (50% FWHM)

@Focus point
Focus size: ~1.5 μm (FWHM)
Peak fluence:
~47 μJ/μm^2 (atoms, clusters),
~26 μJ/μm^2 (molecules)

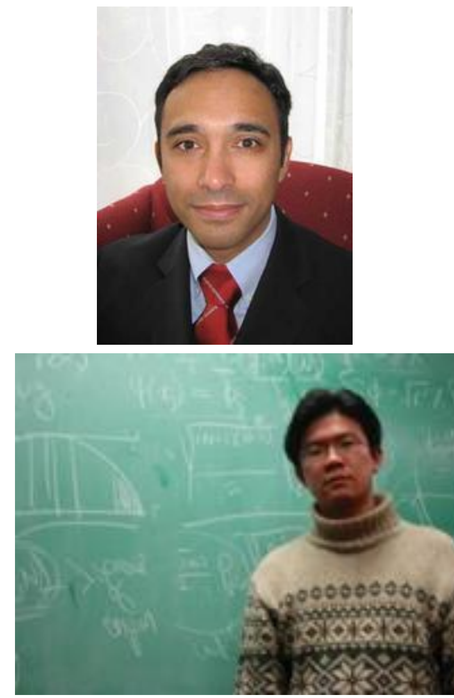
Sample gas was introduced as a pulsed super sonic gas jet to the focus point.

I. Deep inner-shell multiphoton absorption by intense x-ray free-electron laser pulses

H. Fukuzawa, S.-K. Son, K. Motomura, S. Mondal, K. Nagaya, S. Wada, X.-J. Liu, R. Feifel, T. Tachibana, Y. Ito, M. Kimura, T. Sakai, K. Matsunami, H. Hayashita, J. Kajikawa, P. Johnsson, M. Siano, E. Kukk, B. Rudek, B. Erk, L. Foucar, E. Robert, C. Miron, K. Tono, T. Togashi, Y. Inubushi, T. Sato, T. Katayama,T. Hatsui, T. Kameshima, M. Yabashi, M. Yao, R. Santra, and K. Ueda [PRL **110**, 173005 (2013) & JPB **46**, 164024 (2013)]

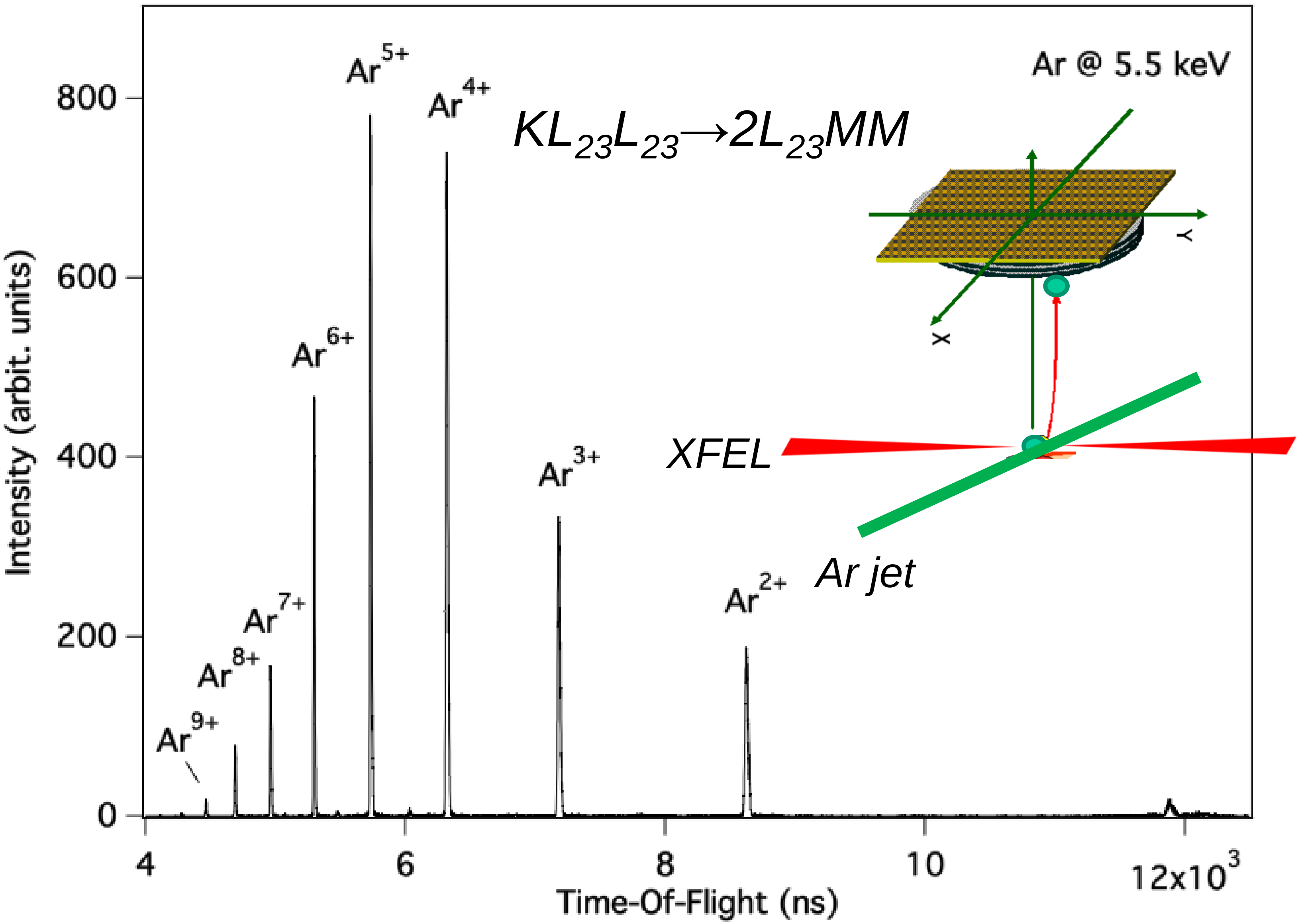


Experimentalists

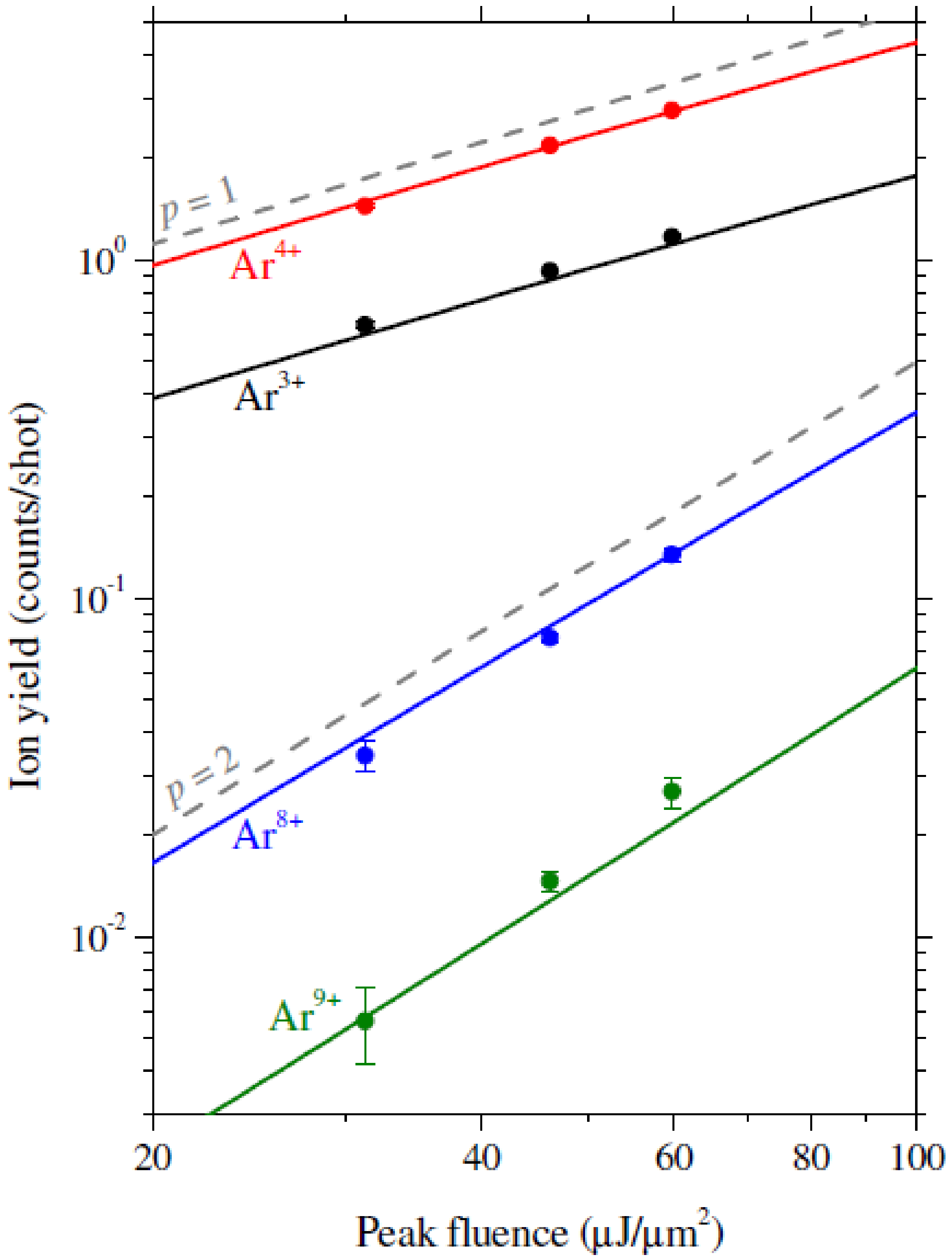


Theorists

Time of Flight spectrum of argon ions



XFEL fluence dependence for Ar^{n+} yields



Ar^{4+}: single photon K-shell ionization → $KL_{23}L_{23}$ Auger → $2L_{23}MM$ Auger

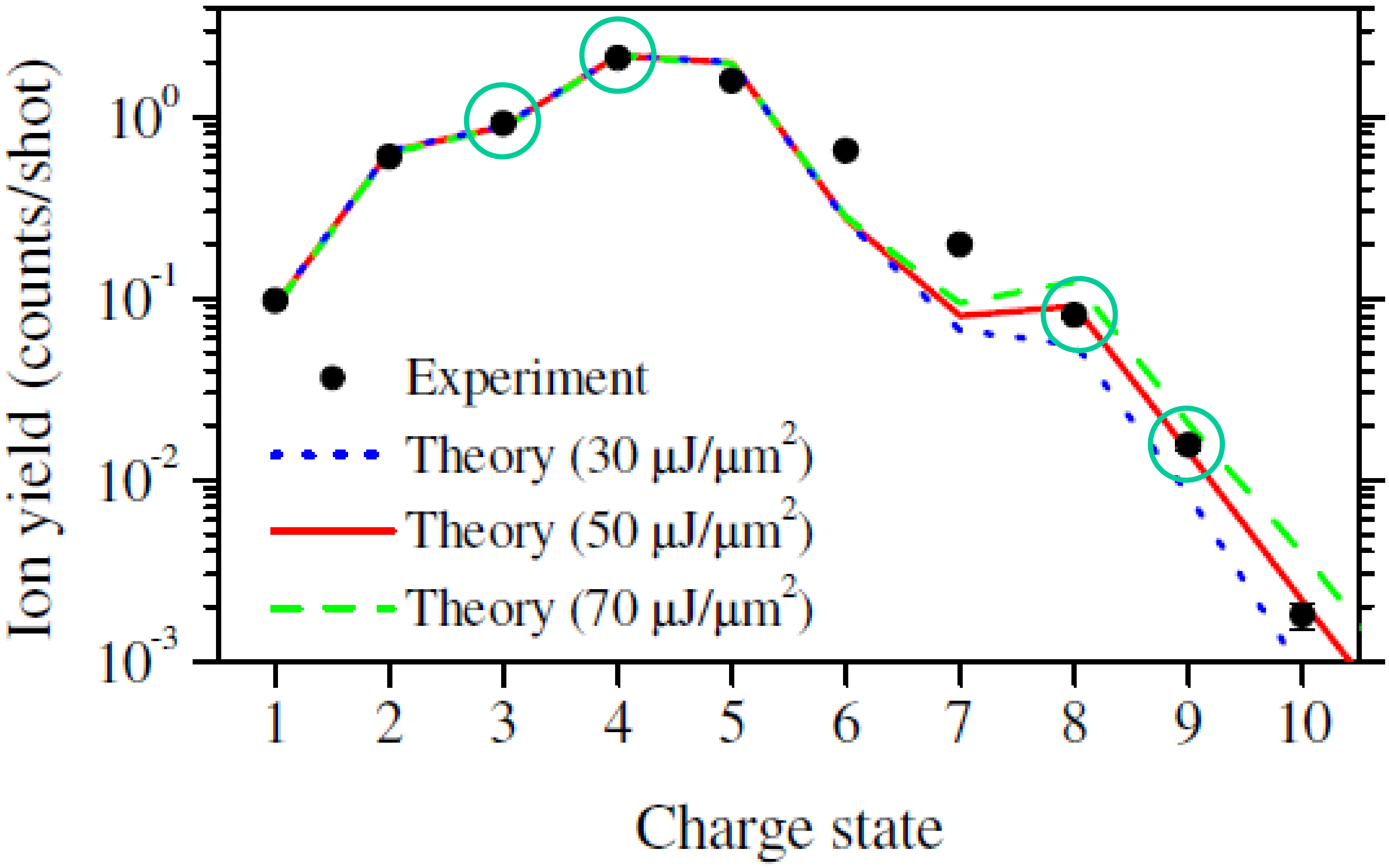
Ar^{3+}: single photon K shell absorption → $KL_{23}M$ Auger → $L_{23}MM$ Auger

Ar^{8+}, Ar^{9+}: sequential two photon K shell ionization

Bench mark ab initio calculation reproduces fluence dependence and relative ratios.

In the theory, the pulse shape of Gaussian of 30 fs (FWHM), and Gaussian focal shape of 1 μm (FWHM) ×1 μm (FWHM) are assumed.

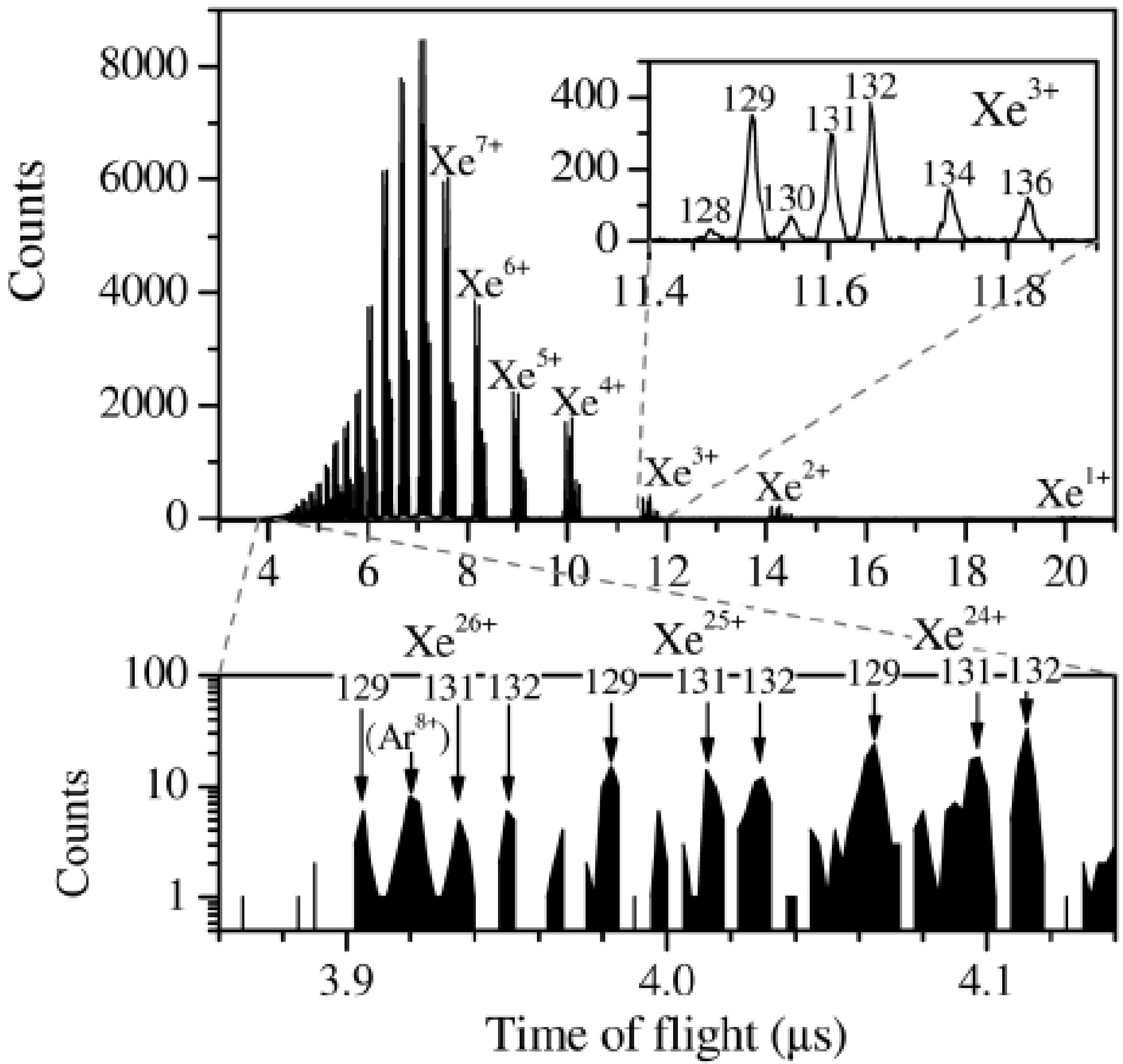
Charge state distribution of Ar: experiment and theory

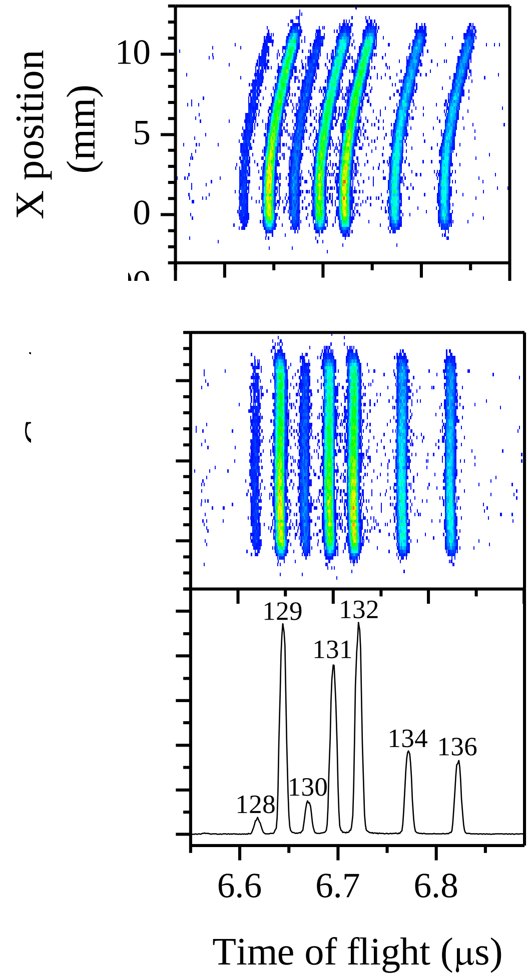


By comparison with theory, we obtained peak fluence of 50 μJ/μm^2 in the experiment!

Time of Flight spectrum of xenon ions

5.5 keV, 50 μJ/μm² at SACLA

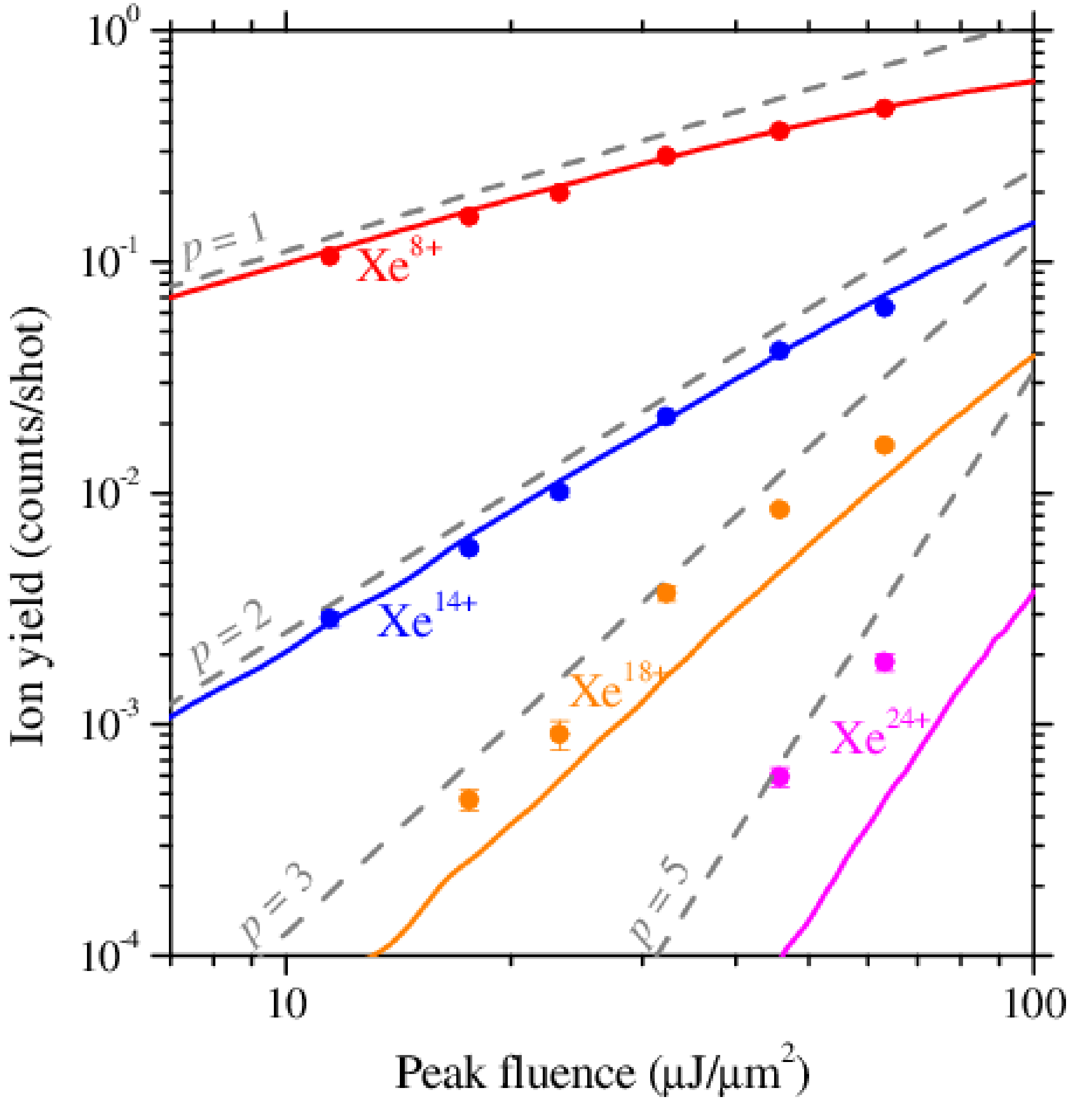




2D position resolved TOF improves the resolution!

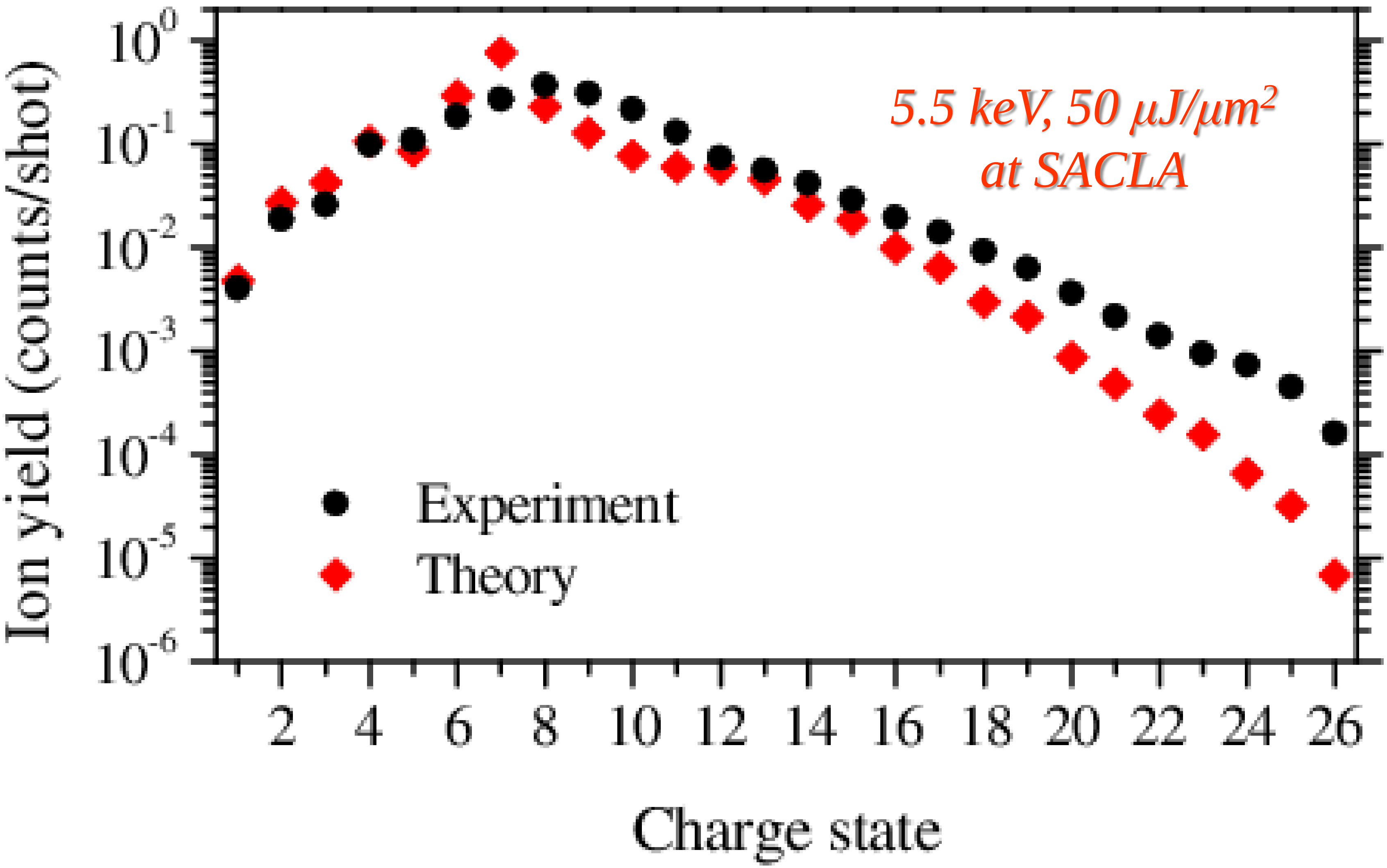
High charge states Xe^{n+} with n up to 26 are produced!

XFEL fluence dependence for Xe^{n+} yields



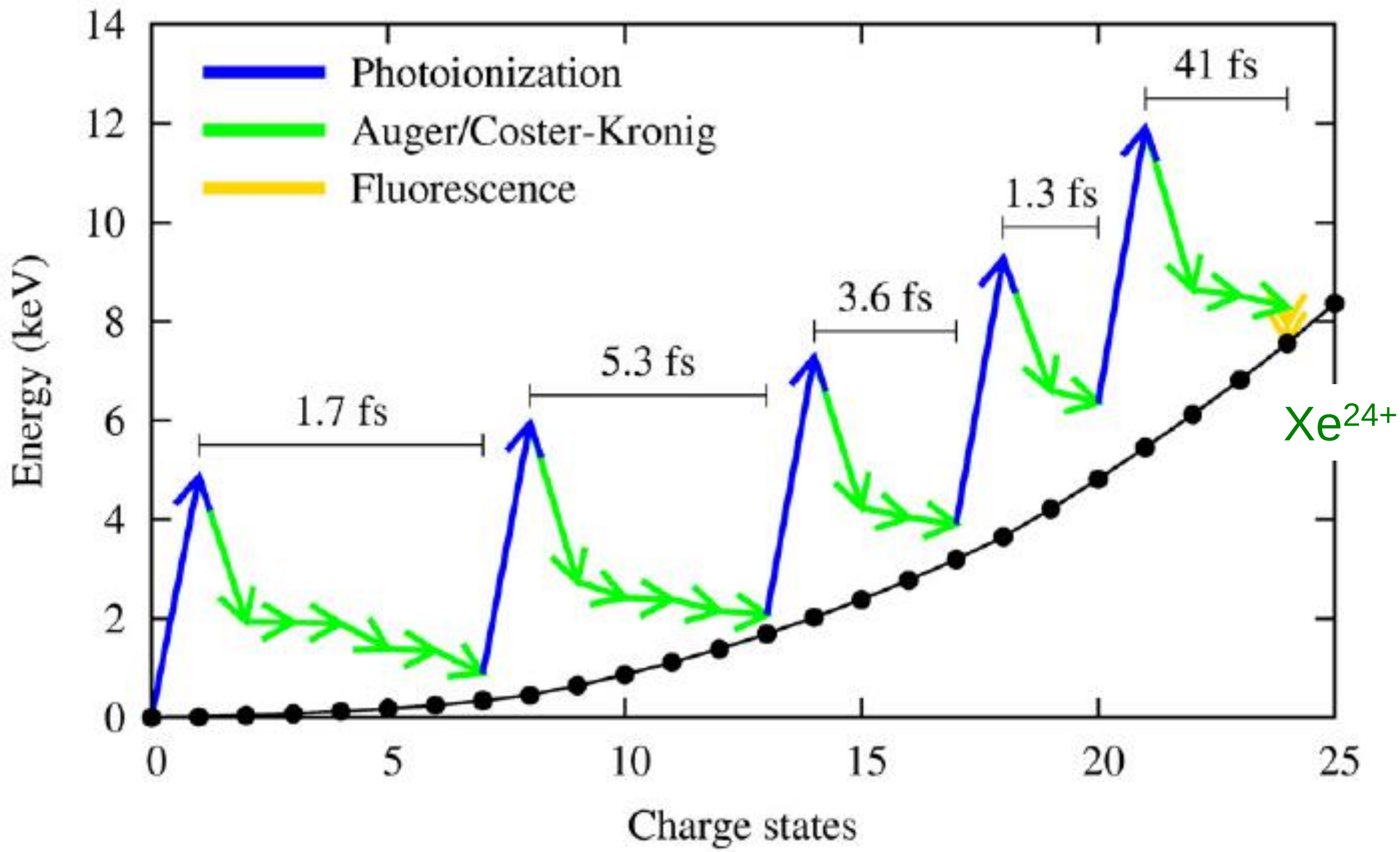
With help of ab initio calculations, we find that the observed high charge states ($n \geq 24$) are produced via five-photon absorption, evidencing the occurrence of multiphoton absorption involving deep inner shells.

Xenon ion charge distributions (exper. vs theory)



A newly developed theoretical model shows good agreement with the experiment!

An exemplary pathway of multiphoton multiple ionization

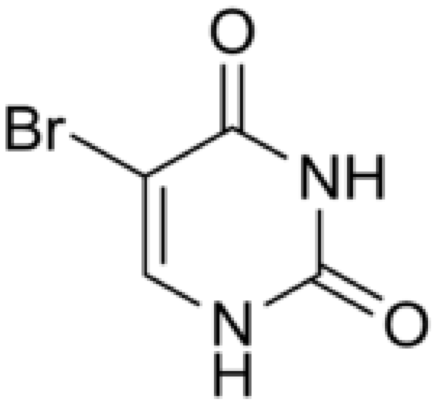


A newly developed theoretical model elucidates the complex pathways of sequential electronic decay cascades accessible in heavy atoms, *revealing that L shell ionization and sequential electronic decay cycles are repeated multiple times within the XFEL pulse duration of ~10 fs.*

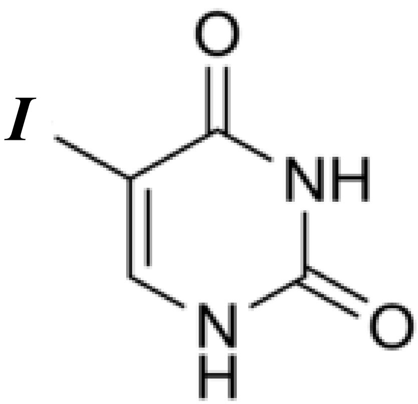
*Fukuzawa, Son et al. PRL **110**, 173005 (2013)*

Relevance to other fields: Radiation damage

Radio-sensitizer

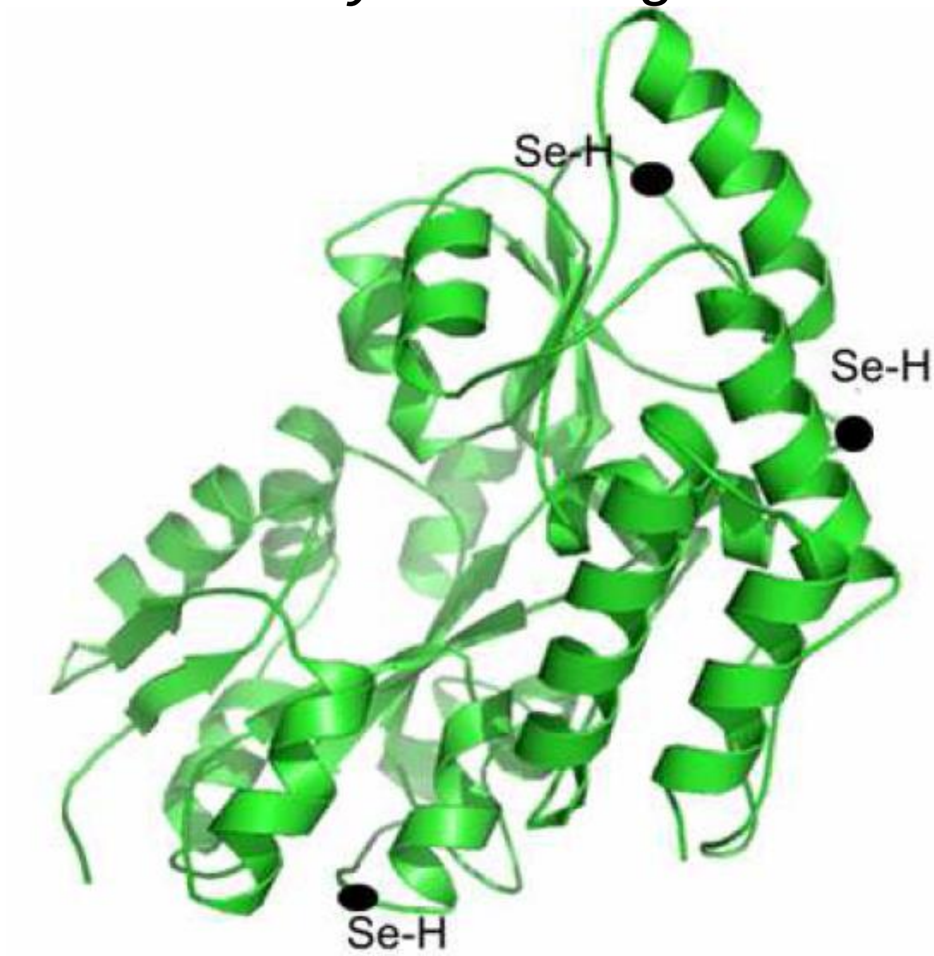


5Br-Uracil



5I-Uracil

Anomalous X-ray scattering



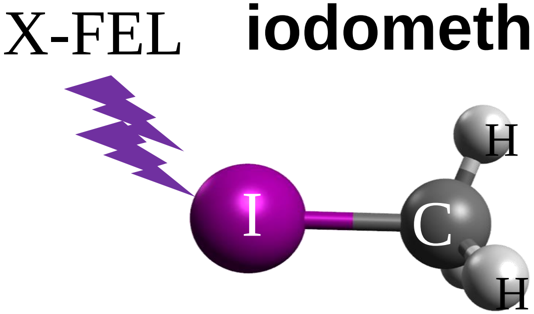
Multiwavelength anomalous diffraction at high X-ray intensity

S.-K. Son, H. N. Chapman, and R. Santra, Phys. Rev. Lett. 107, 218102 (2011).

II. Charge transfer and molecular dissociation following deep inner-shell multi-photon multiple ionization of CH_3I and 5-Uracil molecules by intense x-ray free-electron laser pulses from SACLA

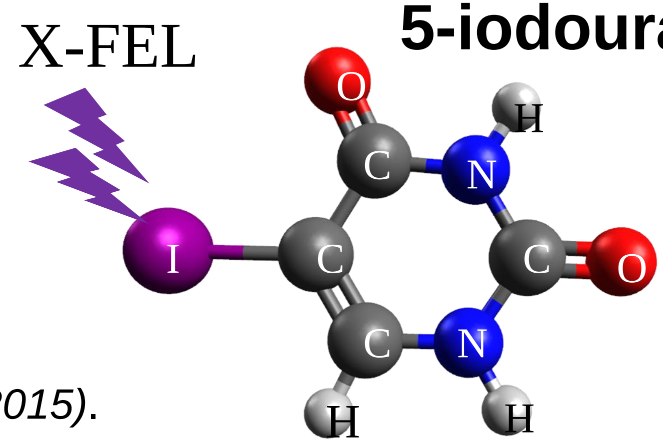
K. Motomura, E. Kukk, H. Fukuzawa, K. Nagaya, S. Omura, S. Wada**,**
S. Mondal, T. Tachibana, Y. Ito, T. Sakai, K. Matsunami, A. Rudenko,
C. Nicolas, X.-J. Liu, C. Miron, Y. Zhang, Y.H. Jiang, J. Chen, A. Milam, D. Kim,
K. Tono, T. Hatsui, Y. Inubushi, M. Yabashi, H. Kono, M. Yao and K. Ueda

iodomethane



K. Motomura et al., J. Phys. Chem. Lett. ***6****, 2944 (2015)*.

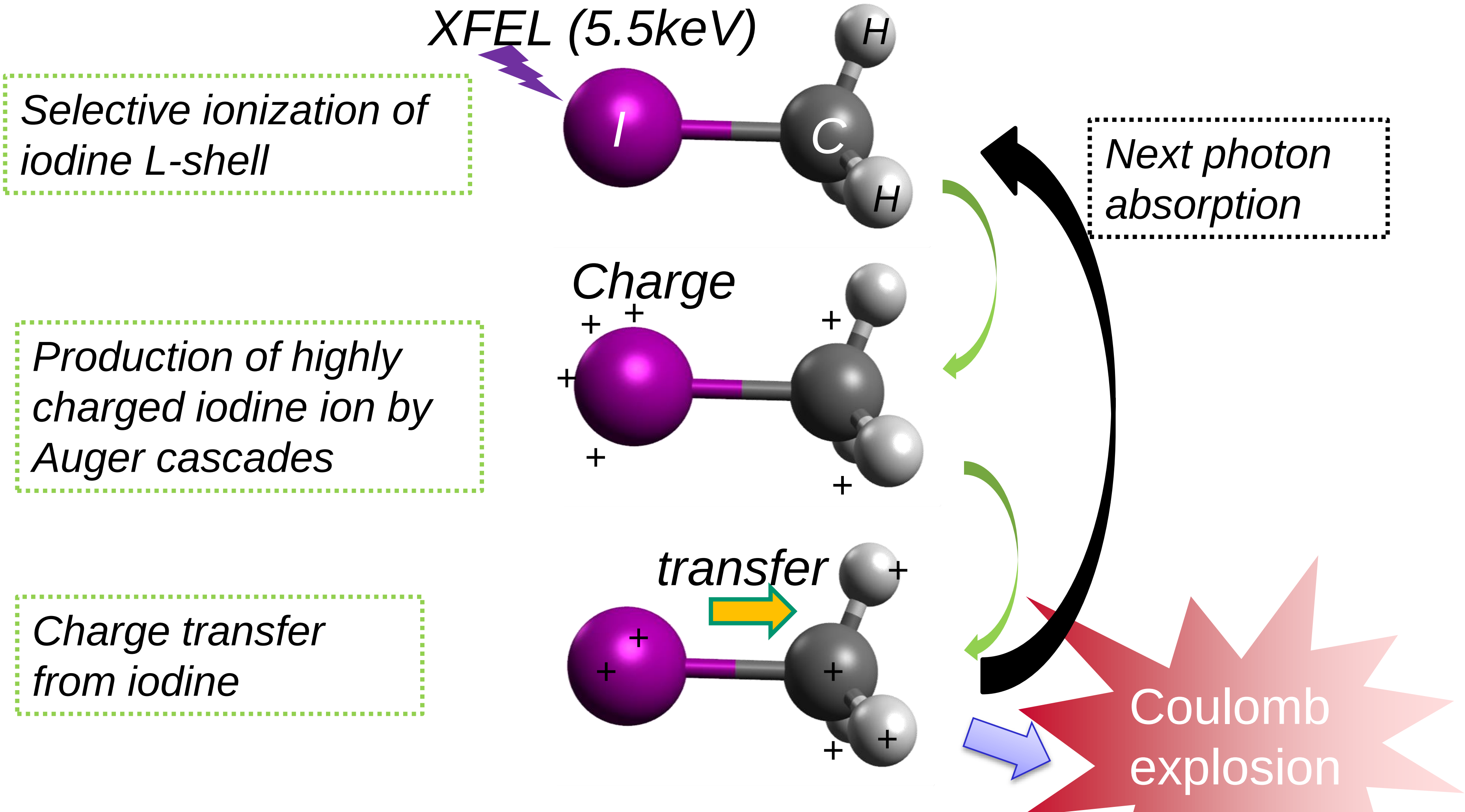
5-iodouracil



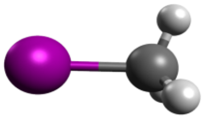
in preparation

Ionization of iodomethane

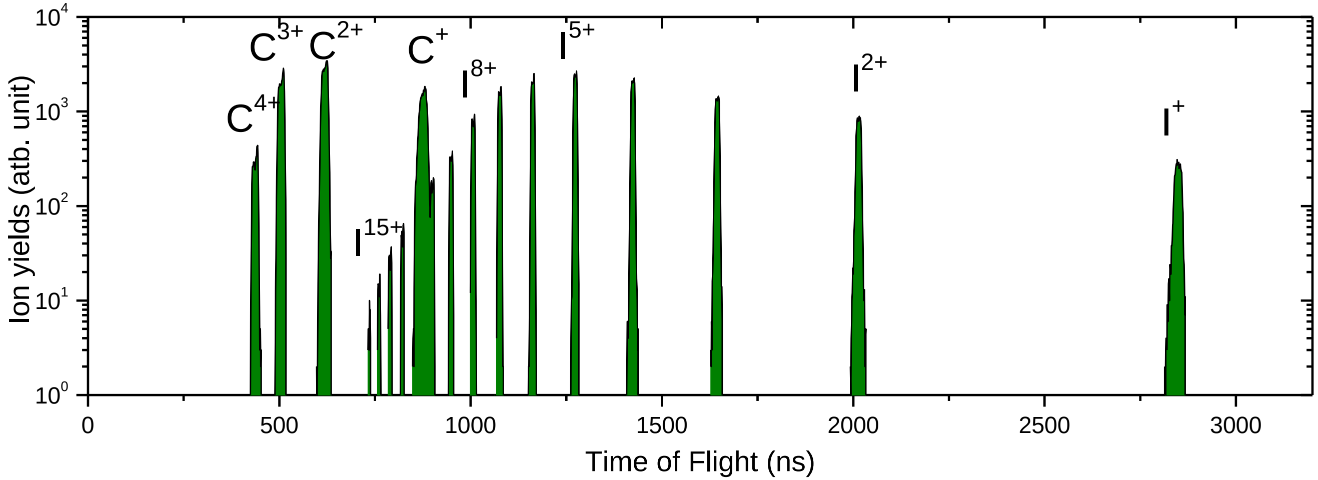
We expect that the ionization proceeds with this sequence.



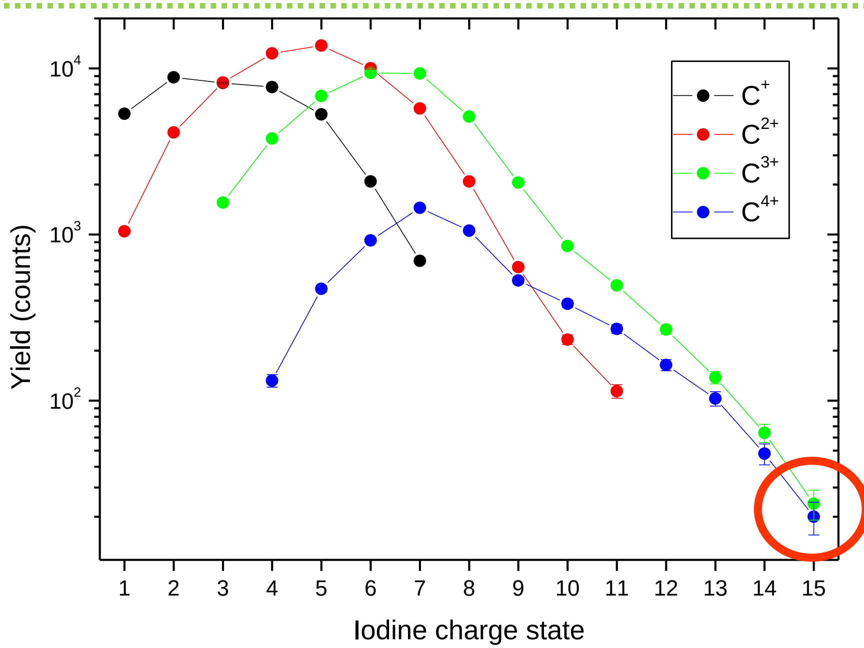
We tried to understand these processes by measuring momentum of ion fragments.

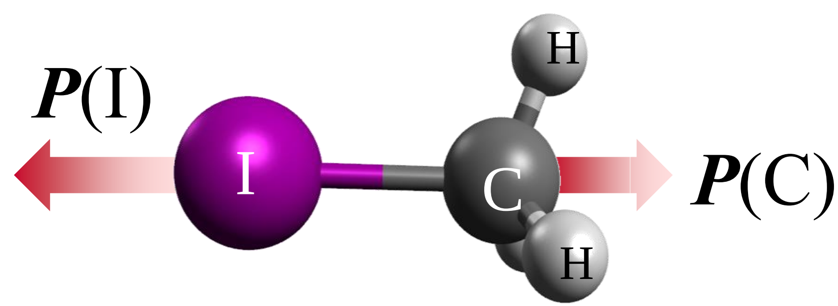


Iodomethane: Charge distribution



Momentum conservation: $\boldsymbol{P}(\mathrm{H1})+\boldsymbol{P}(\mathrm{H2})+\boldsymbol{P}(\mathrm{H3})+\boldsymbol{P}(\mathrm{C})+\boldsymbol{P}(\mathrm{I}) = \mathbf{0}$

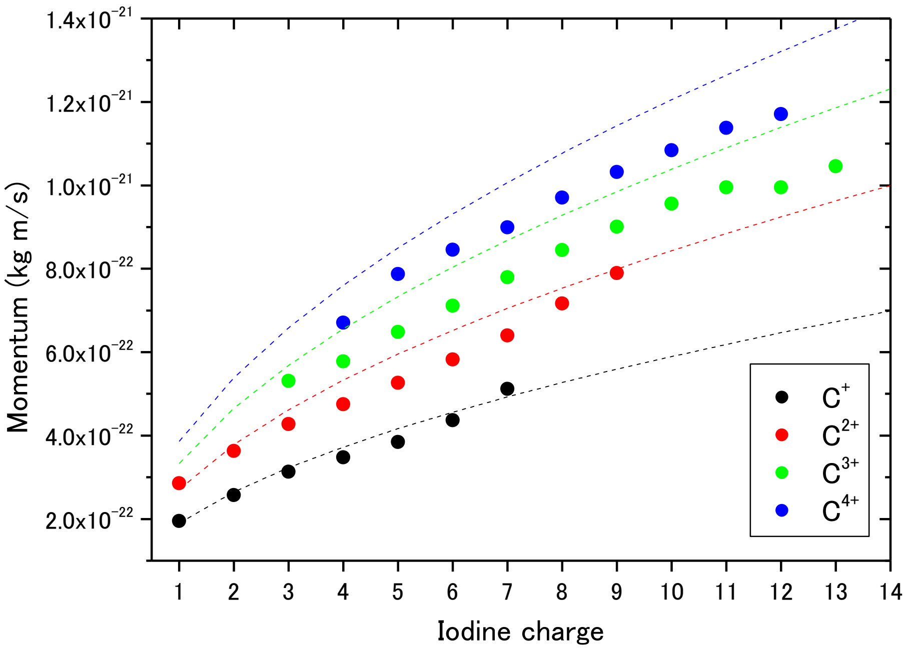




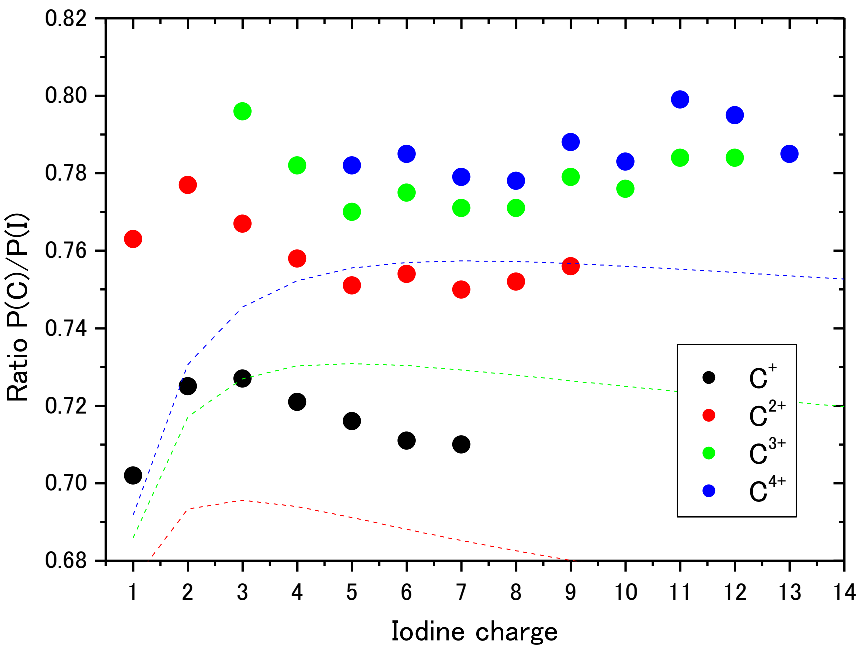
$I^{15+} + C^{4+} + 3H^{+} \rightarrow (CH_3I)^{22+}$

Charge state dependence of ion momentum

Charge state dependence for the momentum of carbon ions



Ratio of the momentum of carbon ions and iodine ions



Dashed line: *Simulation with instant charge build up and transfer within the intact molecule; the charges are arranged before the Coulomb explosion starts.*

The results of the simulation do not agree with the experimental results. Dissociation may compete with the charge buildup and transfer.

Charge build up and transfer model

*We introduced two parameters "**τ**" and "**R**".*

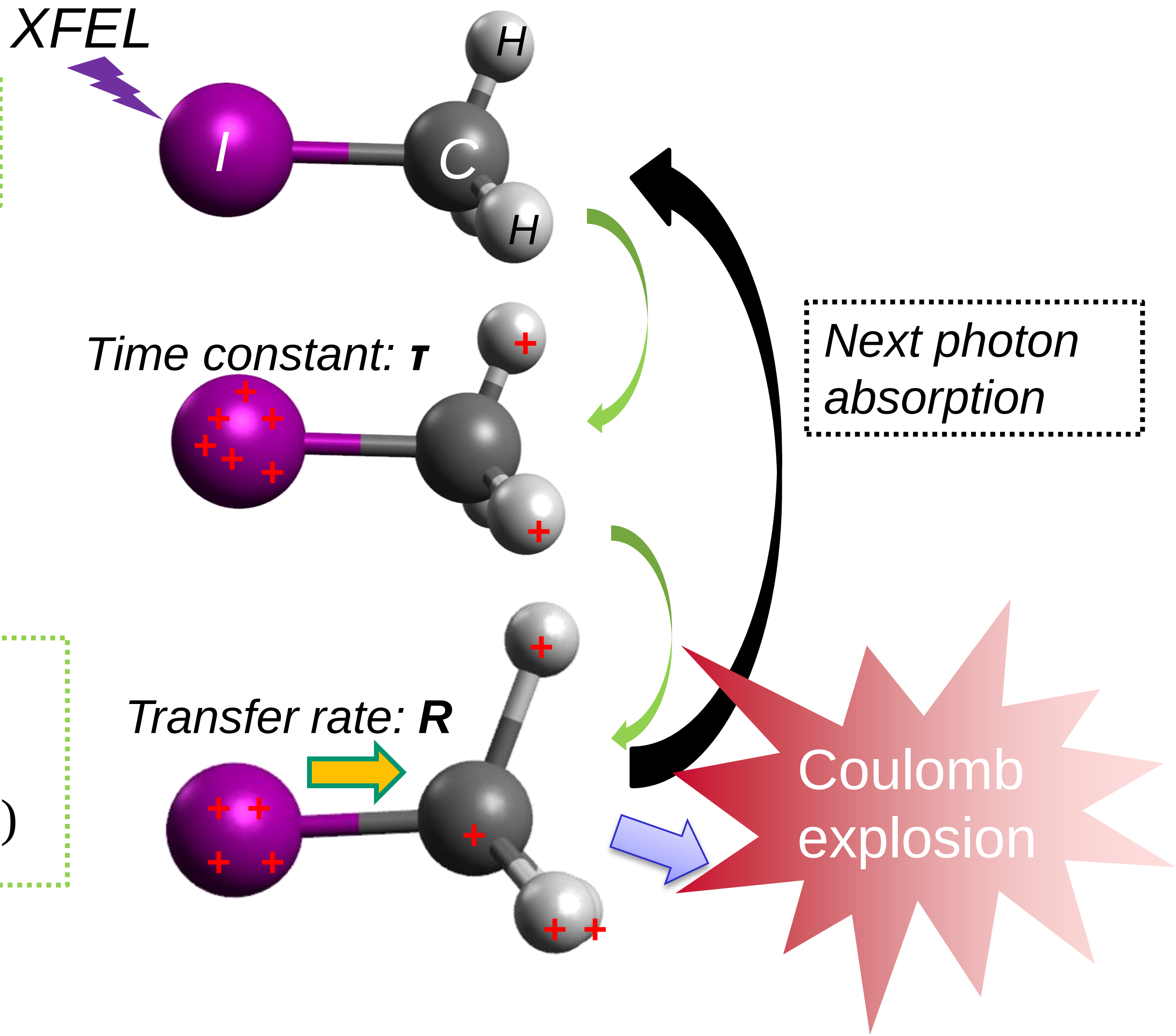
XFEL ionizes iodine L-shell

Charge build up in a molecule.

$$Q_{total}(t) = Q\left(1 - e^{-\frac{t}{\tau}}\right)$$

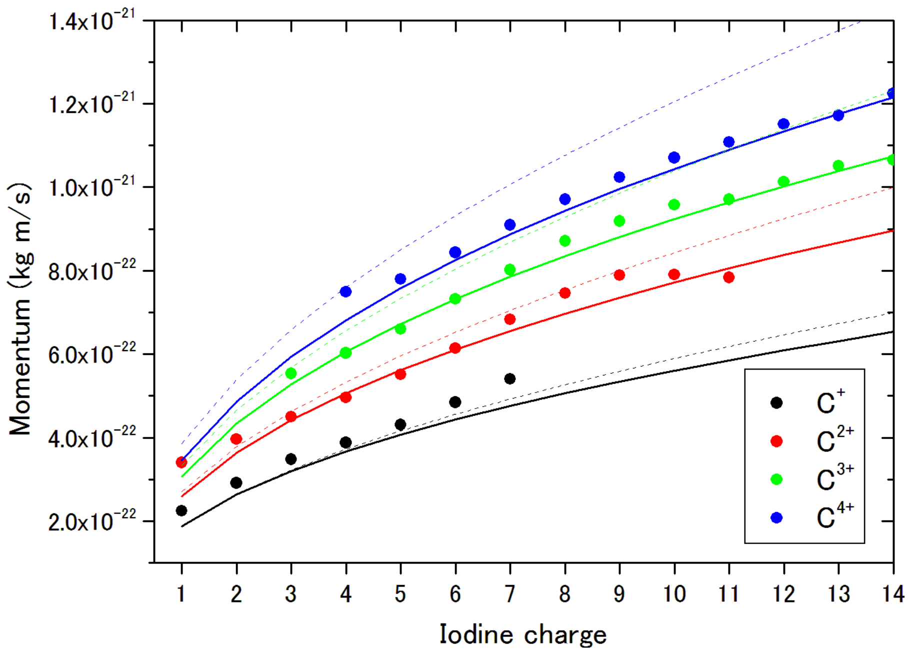
Charge transfer from iodine

$$\frac{dQ_{CH3}(t)}{dt} = R \times Q_I(t)$$

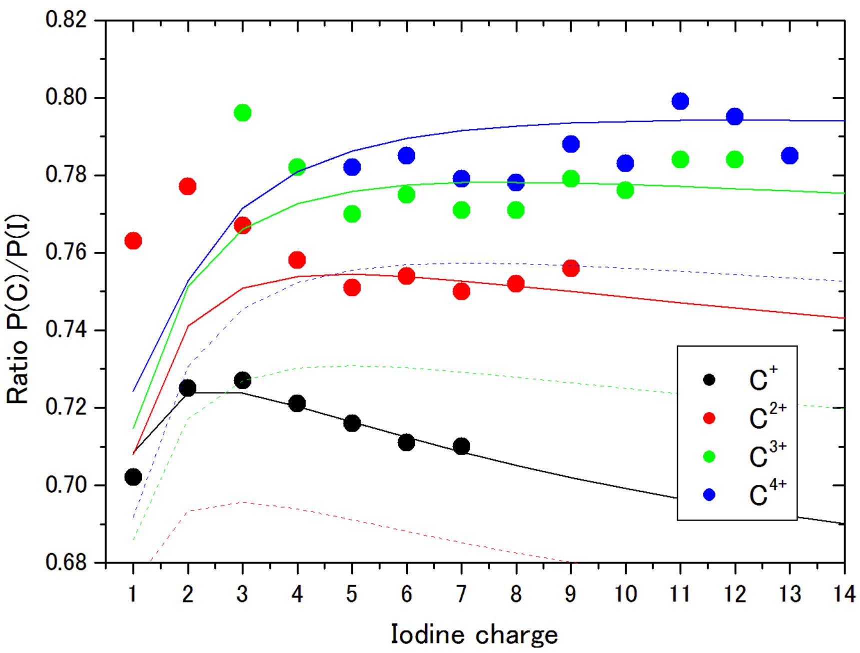


Comparison with charge build up and transfer model

Charge state dependence for the momentum of carbon ions



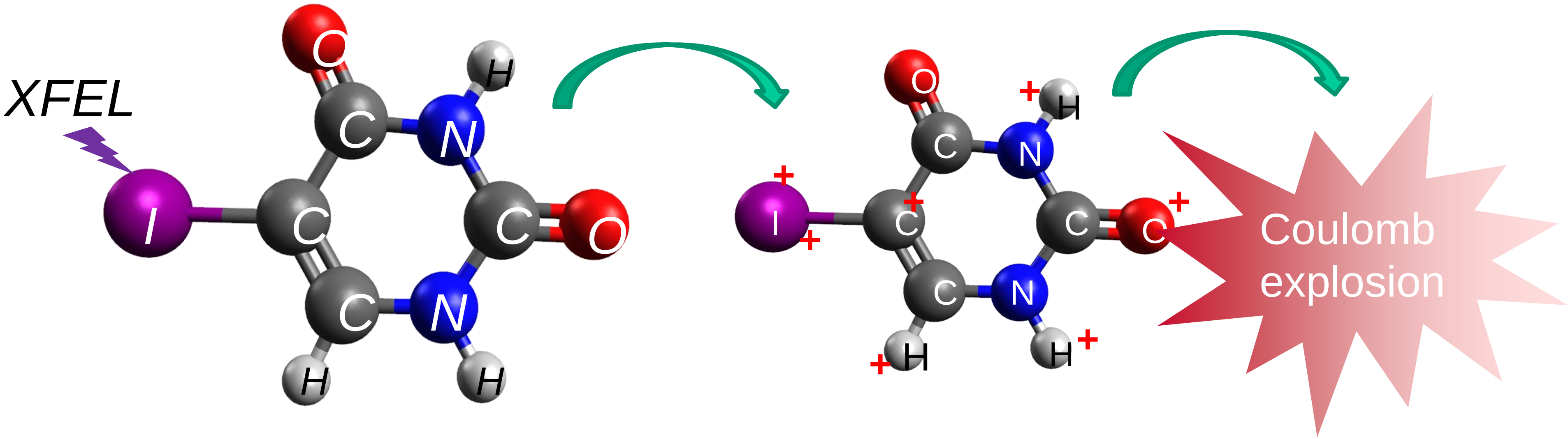
Ratio of the momentum of carbon ions and iodine ions



Solid line: *Simulation with charge build up and transfer*
Dashed line: *Simulation with Instant charge buildup and transfer*

Using the parameters ***τ = 9 fs*** *and* ***R = 0.37 fs^{-1}****, the simulations agree with experimental results.*
τ of 9 fs is consistent with the results of atomic xenon results and roughly the same as the XFEL pulse width (~10 fs).

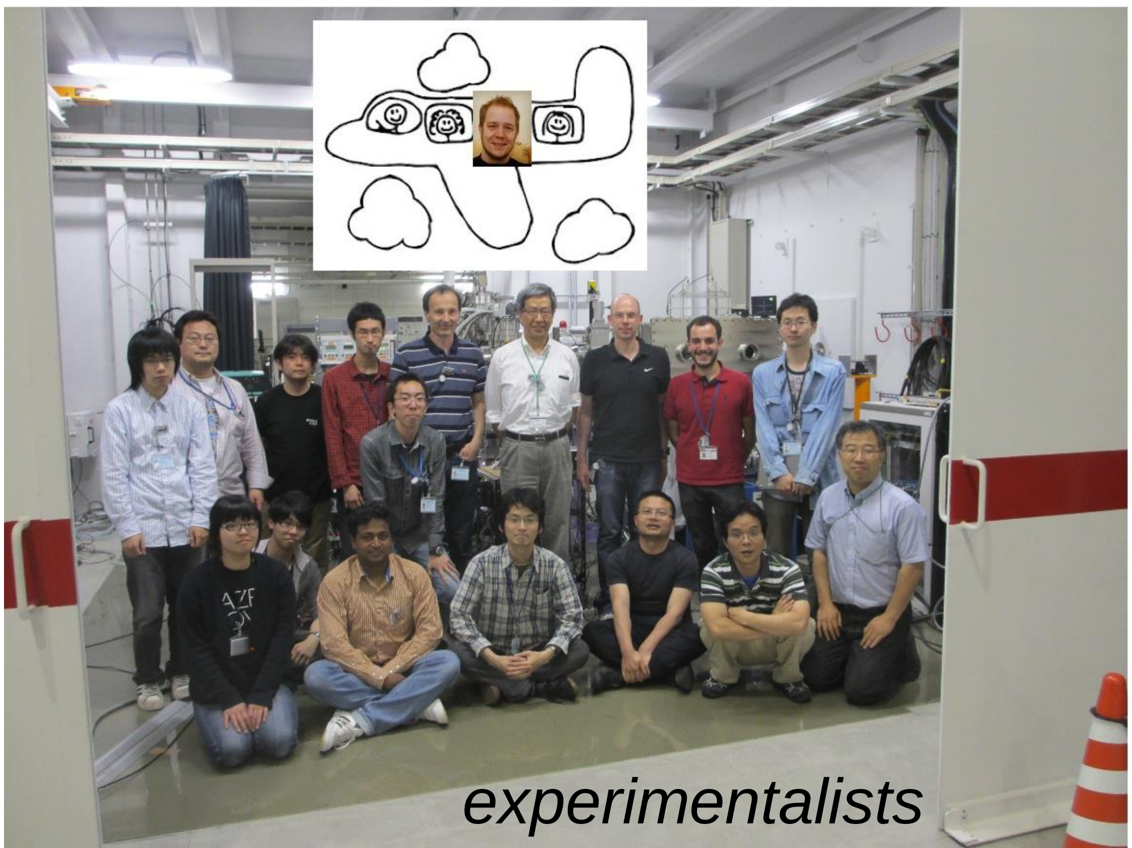
5-iodouracil ($C_4H_3IN_2O_2$)



We obtained the experimental data similar to CH_3I and analyzed them employing a charge-build up and transfer model.
*Using the parameters **τ ~ 10 fs** and **R ~ 0.5 fs^{-1}**, the simulations agree with experimental results.*
These numbers are almost the same as those for CH_3I.

III. Efficient Nanoplasma Formation from Argon Clusters Irradiated by the Hard X-ray Free Electron Laser

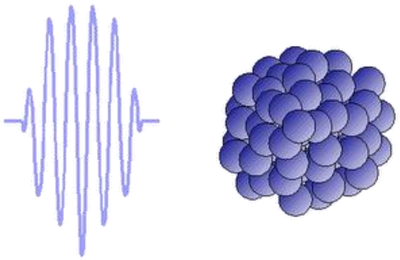
T. Tachibana, Z. Jurek, H. Fukuzawa, K. Motomura, K. Nagaya, S. Wada, P. Johnsson, M. Siano, S. Mondal, Y. Ito, M. Kimura, T. Sakai, K. Matsunami, H. Hayashita, J. Kajikawa, , X.-J. Liu, E. Robert, C. Miron, R. Feifel, , J. Marangos, K. Tono, T. Togashi, Y. Inubushi, T. Hatsui, M. Yabashi, B. Ziata, S. Son, M. Yao, R. Santra, and K. Ueda (*Sci. Rep.*, ***5,*** *10977 (2015).).*



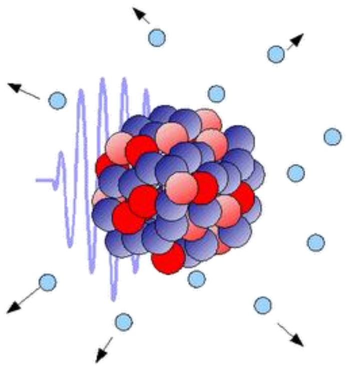
experimentalists

theorists

Nanoplasma formation by intense laser irradiation

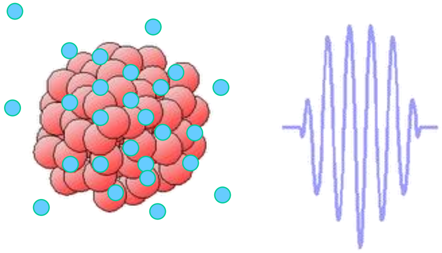


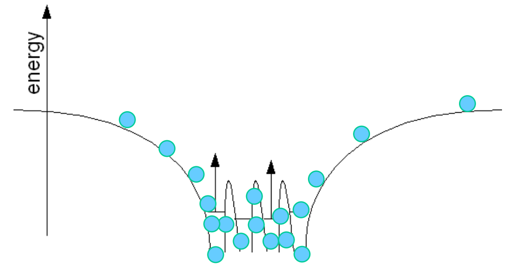
Laser irradiation into cluster



Many atoms in the cluster are ionized

Nanoplasma is formed when the electrons ejected from atoms trapped by the Coulomb potential of the multiply charged cluster ion.





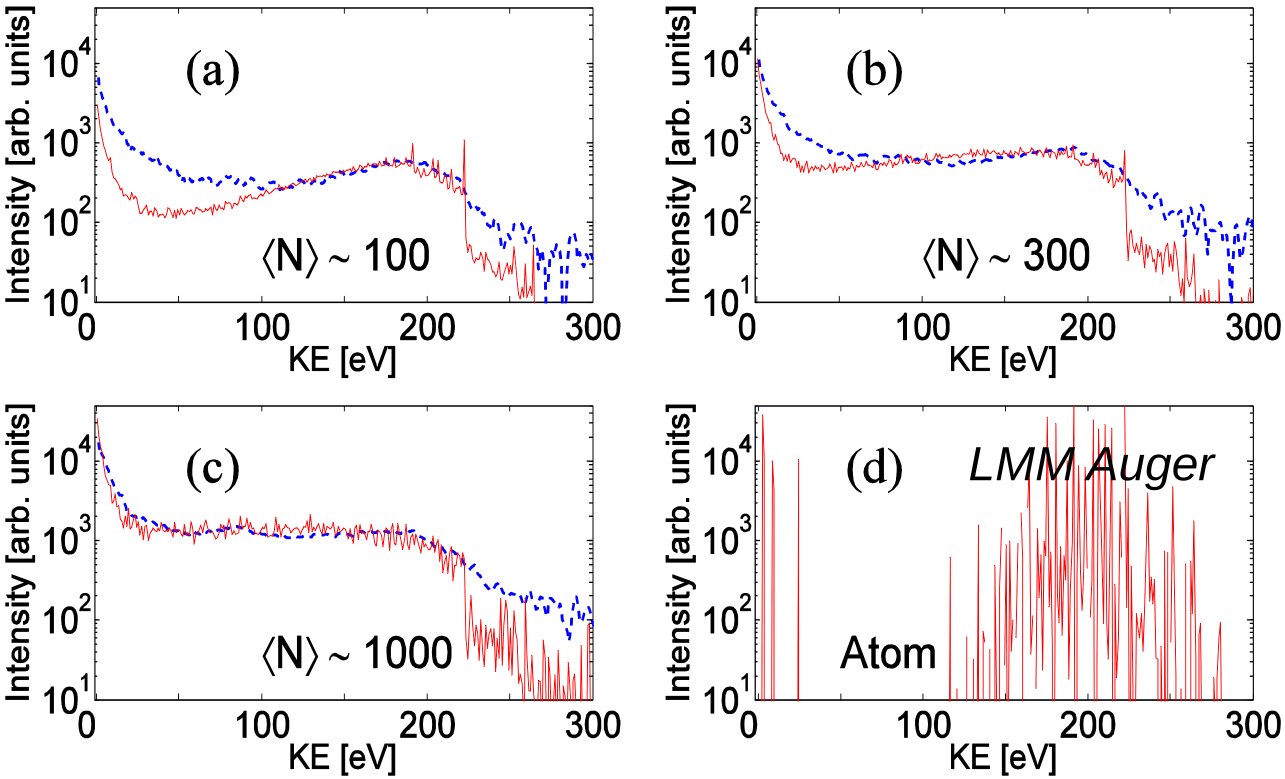
Is nanoplasma also formed by intense hard x-ray pulse irradiation?

How is nanoplasma formed?

Experimental & theoretical electron spectra of Ar clusters

Photon energy: 5 keV
Ar K edge: 3.2 keV

Dashed blue: Experiment,
Solid red: Theory

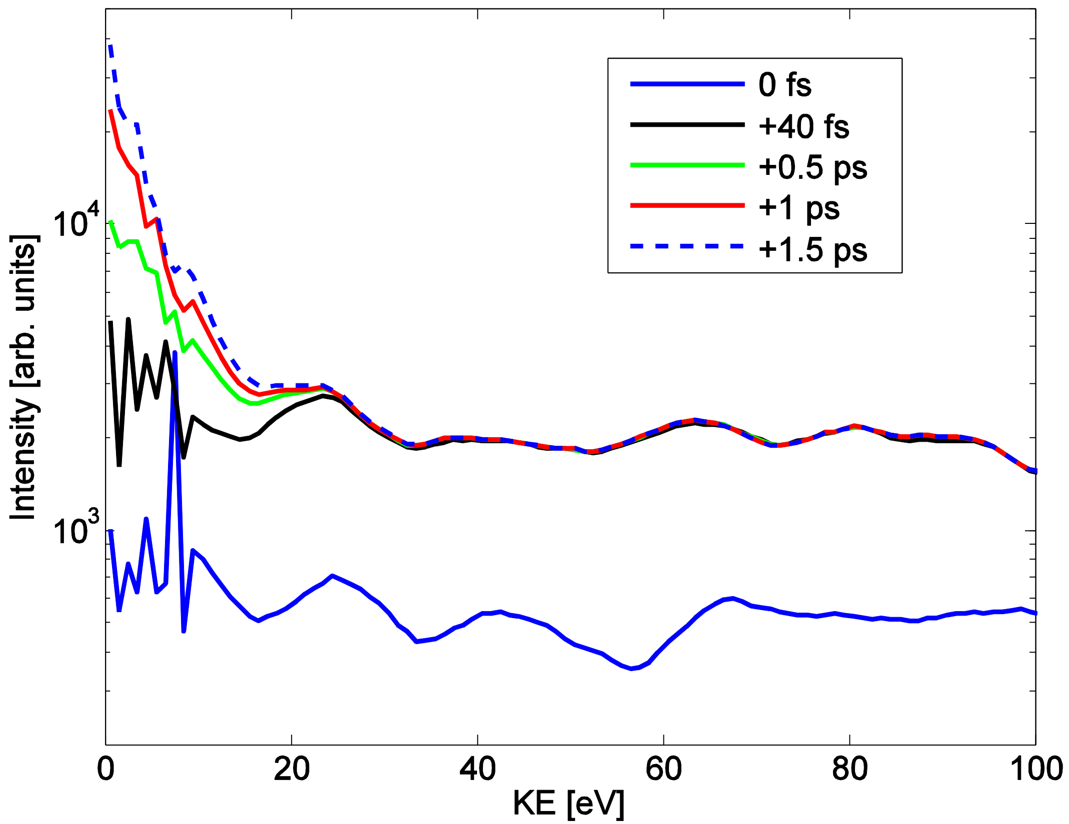


A plateau in the spectra is produced by the deceleration of the electrons. With the increase of the cluster size, a stronger potential builds up, decelerating the emitted electrons more.

The strong peak at zero kinetic energy is due to the thermal emission from nanoplasma.

*T. Tachibana, Z. Jurek, H. Fukuzawa et al., Sci. Rep., **5**, 10977 (2015).*

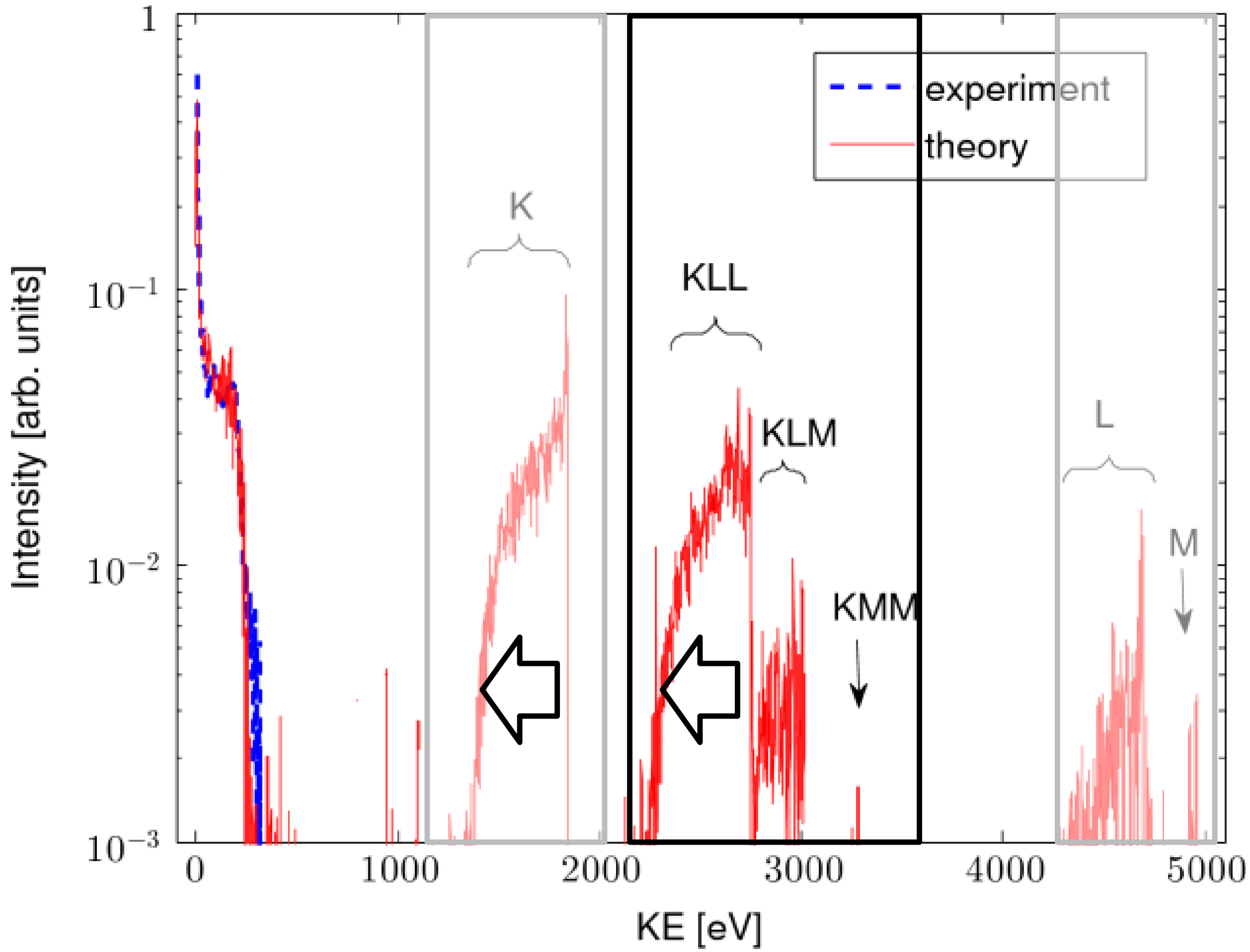
Time evolution for the theoretical electron spectrum



During the XFEL pulse, only the plateau is formed. The main peak at 0 eV develops after the XFEL pulse when the ionic system has started to expand and let some of trapped electrons escape from nanoplasma.

T. Tachibana, Z. Jurek, H. Fukuzawa et al., Sci. Rep., ***5****, 10977 (2015).*

Electron spectra of Ar_{1000} in the whole region



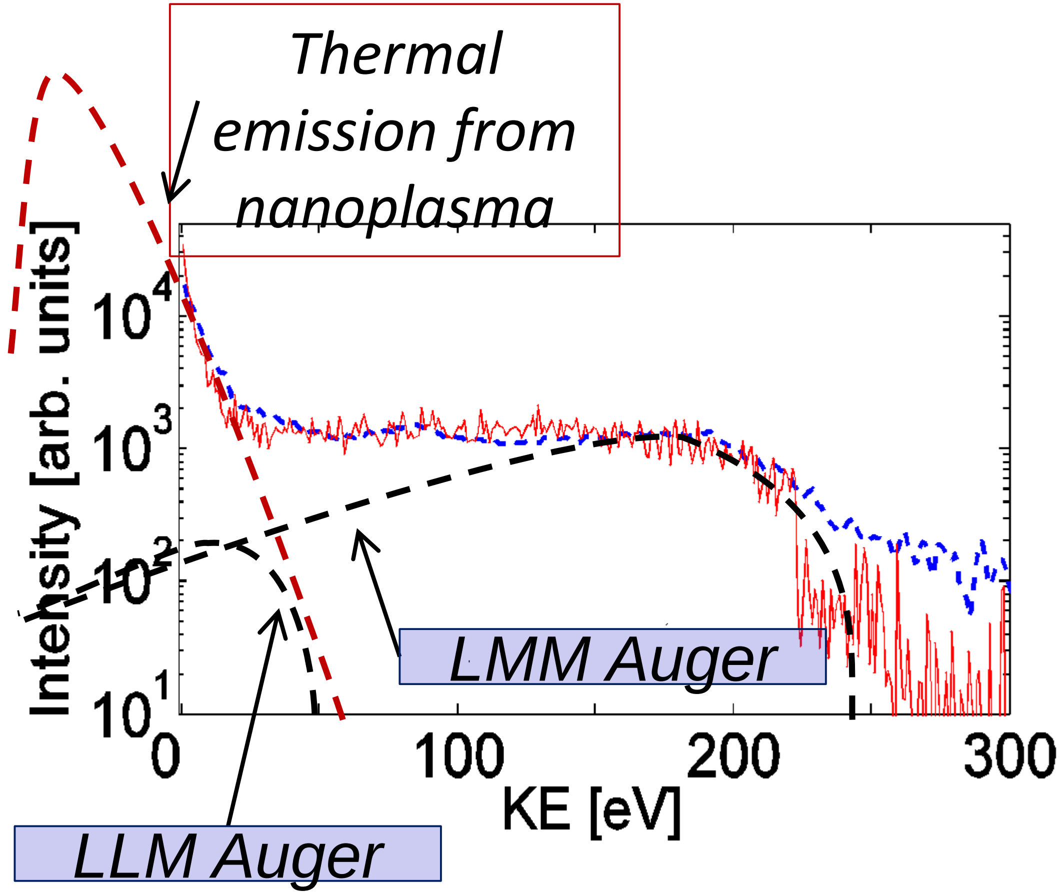
Photon energy : 5 keV
Ar K edge : 3.2 keV

Photo electron
Auger electron

Emitted electrons are decelerated ~500 eV

*T. Tachibana, Z. Jurek, H. Fukuzawa et al., Sci. Rep., **5**, 10977 (2015).*

Origin of the slow electrons that can be trapped

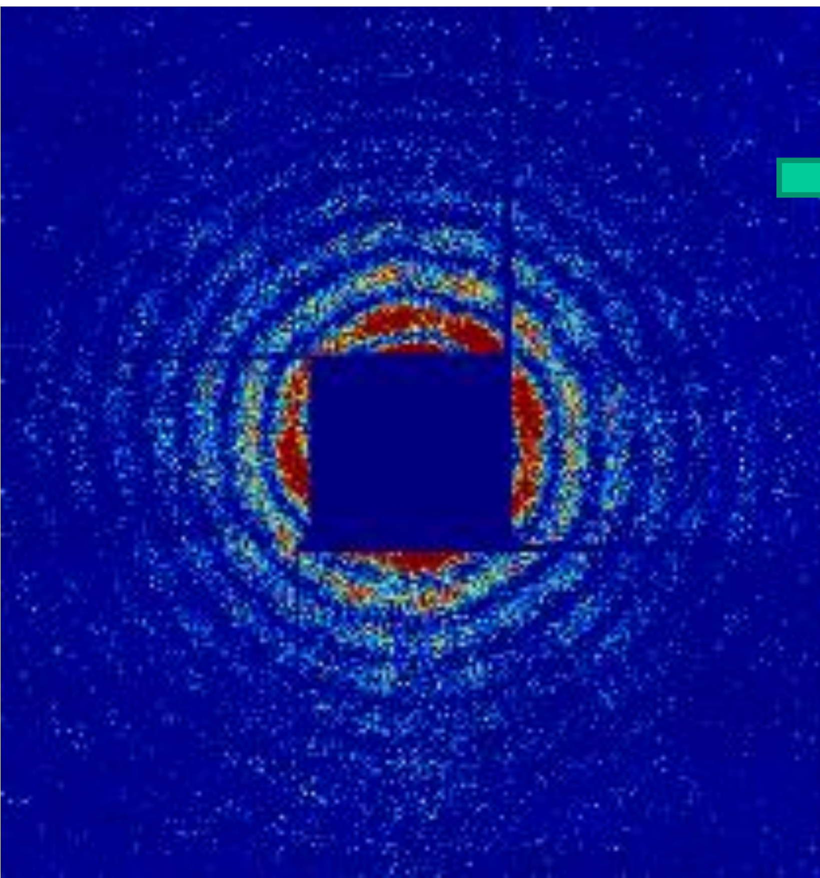


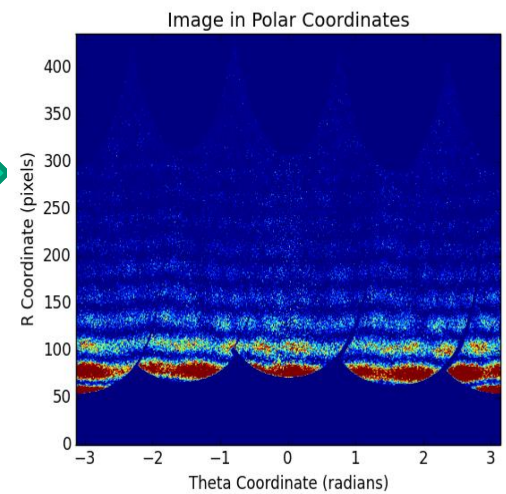
The majority of trapped electrons are created by impact ionizations caused by low-energy Auger and secondary electrons.

T. Tachibana, Z. Jurek, H. Fukuzawa et al., Sci. Rep., ***5****, 10977 (2015).*

IV. Single-shot imaging of giant Xe clusters with X-ray free-electron laser (5.5 keV)

T. Nishiyama, ***C. Bostedt***, ***K. Nagaya***, *K. R. Ferguson*, *C. Hutchison*, *H. Fukuzawa*, *K. Motomura*, *S. Wada*, *T. Sakai*, *K. Matsuami*, *T. Tachibana*, *Y. Ito*, *W. Q. Xu*, *S. Mondal*, *T. Umemoto*, *C. Nicolas*, *C. Miron*, *K. Kameshima*, *Y. Jochi*, *K. Tono*, *H. Hatsui*, *M. Yabashi*, *M. Yao*, *and K. Ueda* (in preparation).



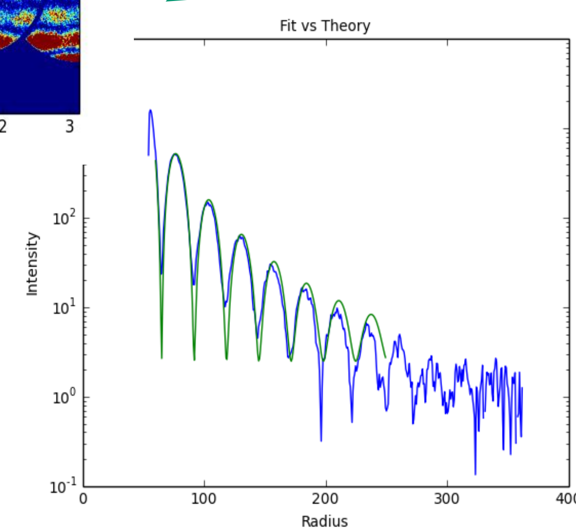


Diameter: 250nm

~1.1×10^8 atoms

FEL fluence:

~10 μJ/μm^2



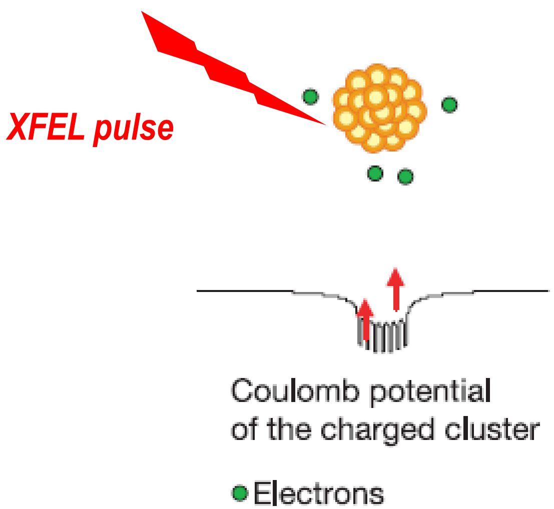
V. Real-time study on the ultrafast plasmon resonance heating of nanoplasma produced by the XFEL irradiation to rare gas clusters at SACLA

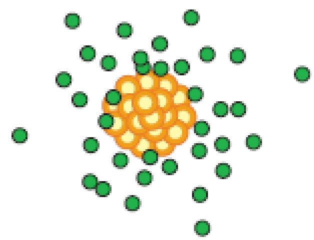
Y. Kumagai, W. Xu, Z. Jurik, H. Fukuzawa, K. Motomura, S. Mondal, T. Tachibana, Y. Ito, K. Nagaya, T. Sakai, K. Matsunami, T. Nishiyama, M. Yao, S. Wada, T. Umemoto,C. Nicolas, C. Miron, T. Togashi, K. Tono, Ogawa, S. Owada, M. Yabashi, B. Ziata, S. Son, R. Santra, and K. Ueda
(in preparation)

Dynamics of nanoplasma produced by XFEL

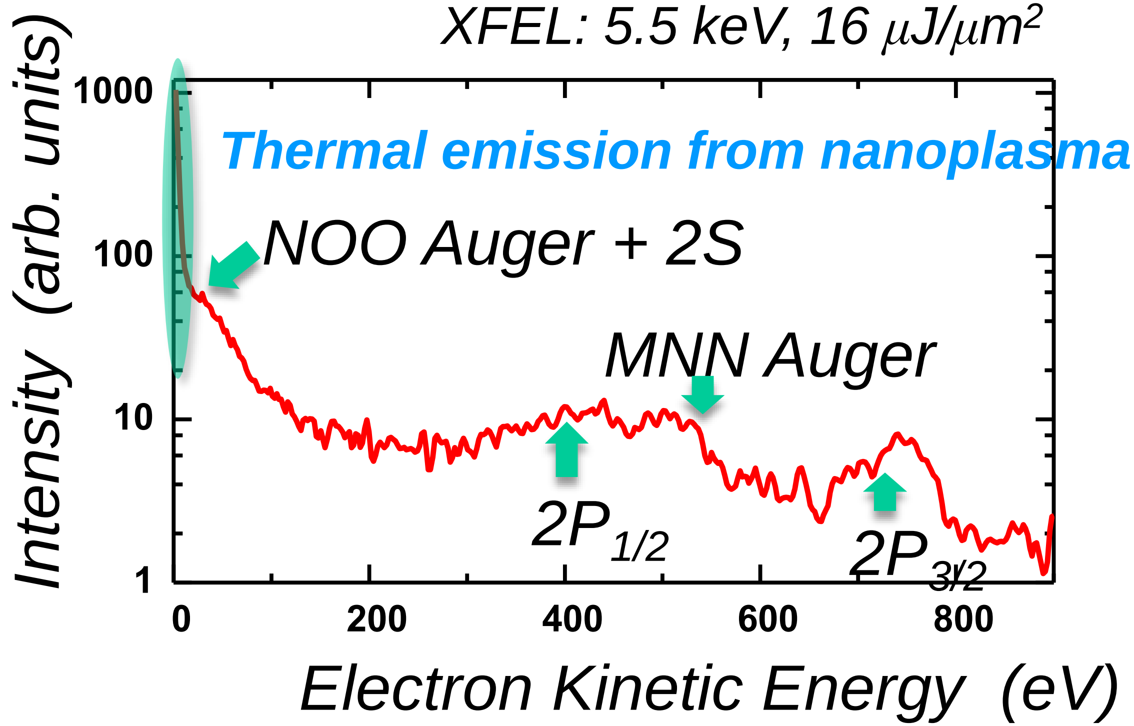
- Photoionization
  Auger cascades



- Nanoplasma formation



Xe clusters
<N> ~ 5000 @ SACLA
XFEL: 5.5 keV, 16 μJ/μm²

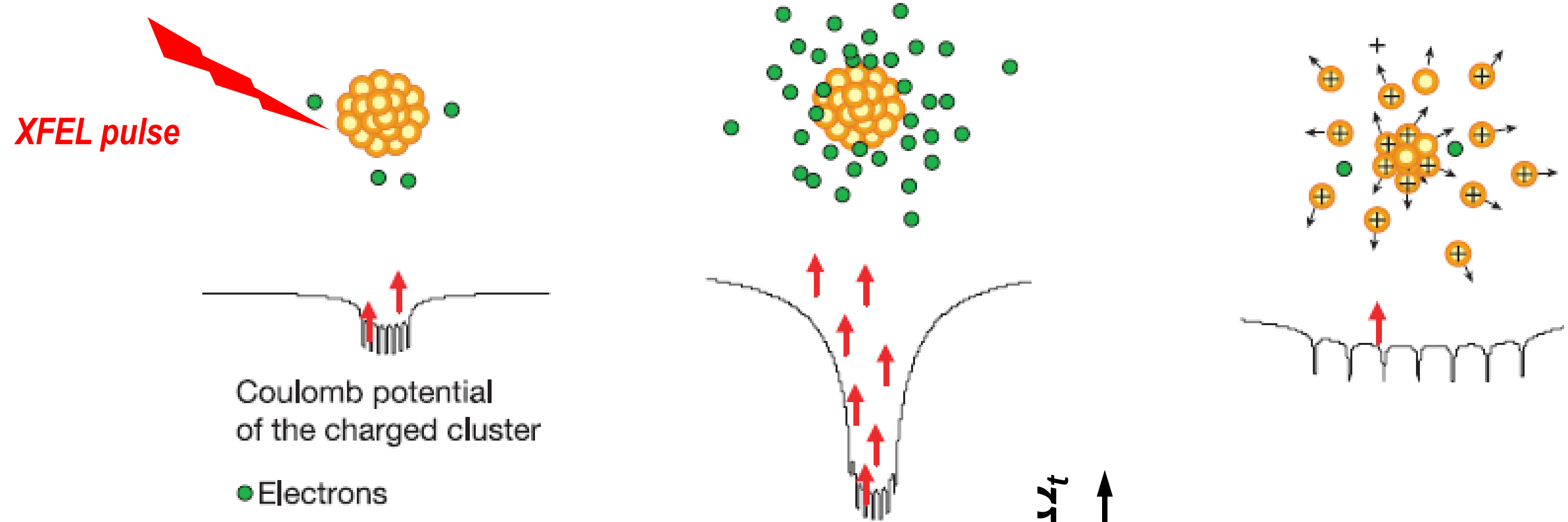


To search the plasmon resonance heating

Photoionization
Auger cascades

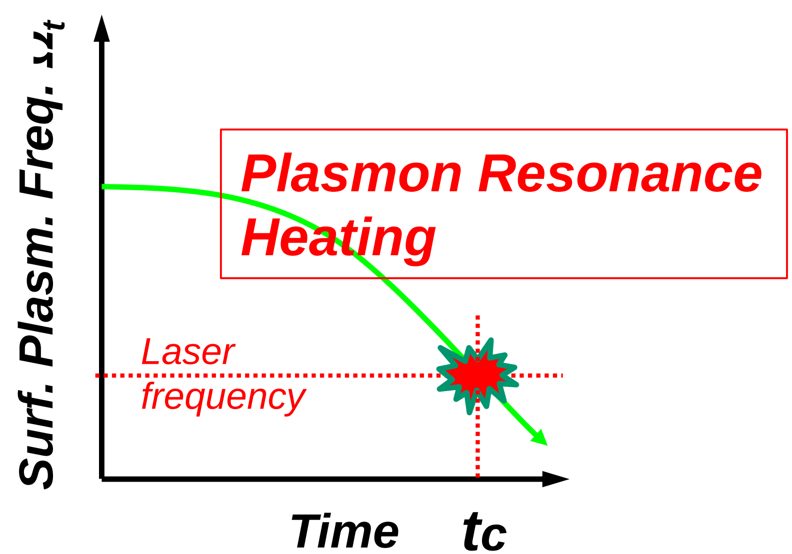
Nanoplasma formation

Coulomb explosion



Surface-plasma frequency

$$\Omega_t (\propto \sqrt{\frac{N_t Z_t}{R_t^3}}) = \frac{\omega_{pl}}{\sqrt{3}}$$



Experimental setups

■ XFEL @ SACLA

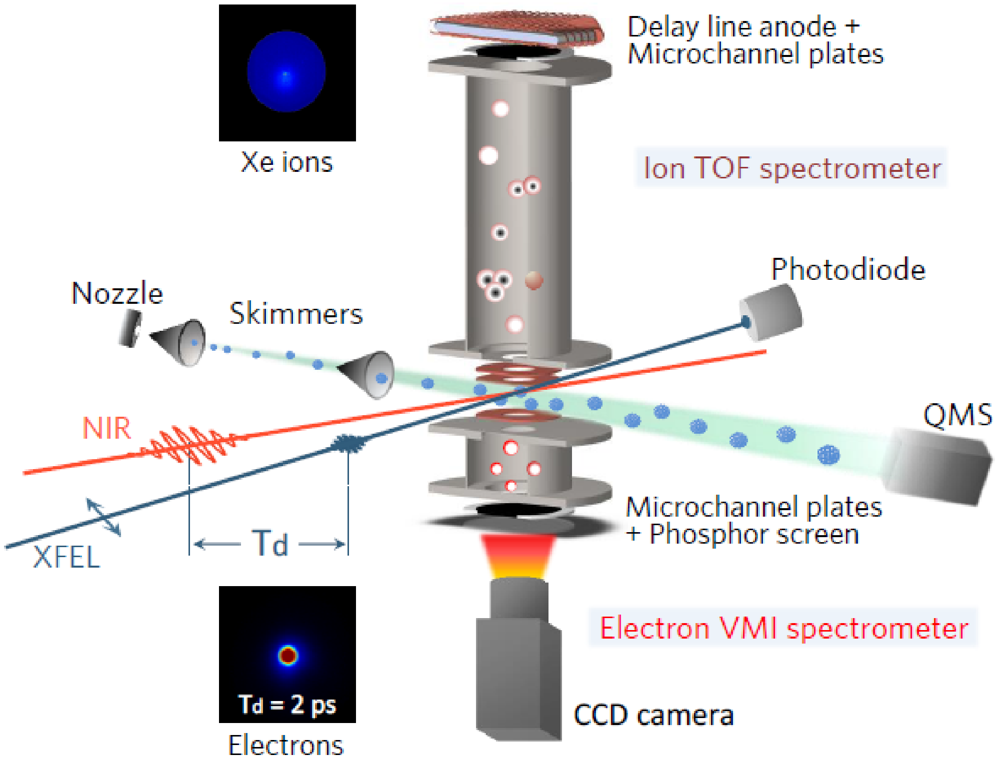
Photon energy: 5.5 keV
Spectral width: ~33 eV (FWHM)
Repetition rate: 30 Hz
Pulse duration: <10 fs
Focused beam size: ~1 μm
Peak intensity: ~3.28 / 3.38 x 10^{16} W/cm^2

■ NIR laser

Wavelength: 800 nm
Repetition rate : 30 Hz
Pulse duration : 80 fs
Focused beam size: ~200 μm
Intensity: ~2.7 / 5.1 x 10^{12} W/cm^2

■ Clusters

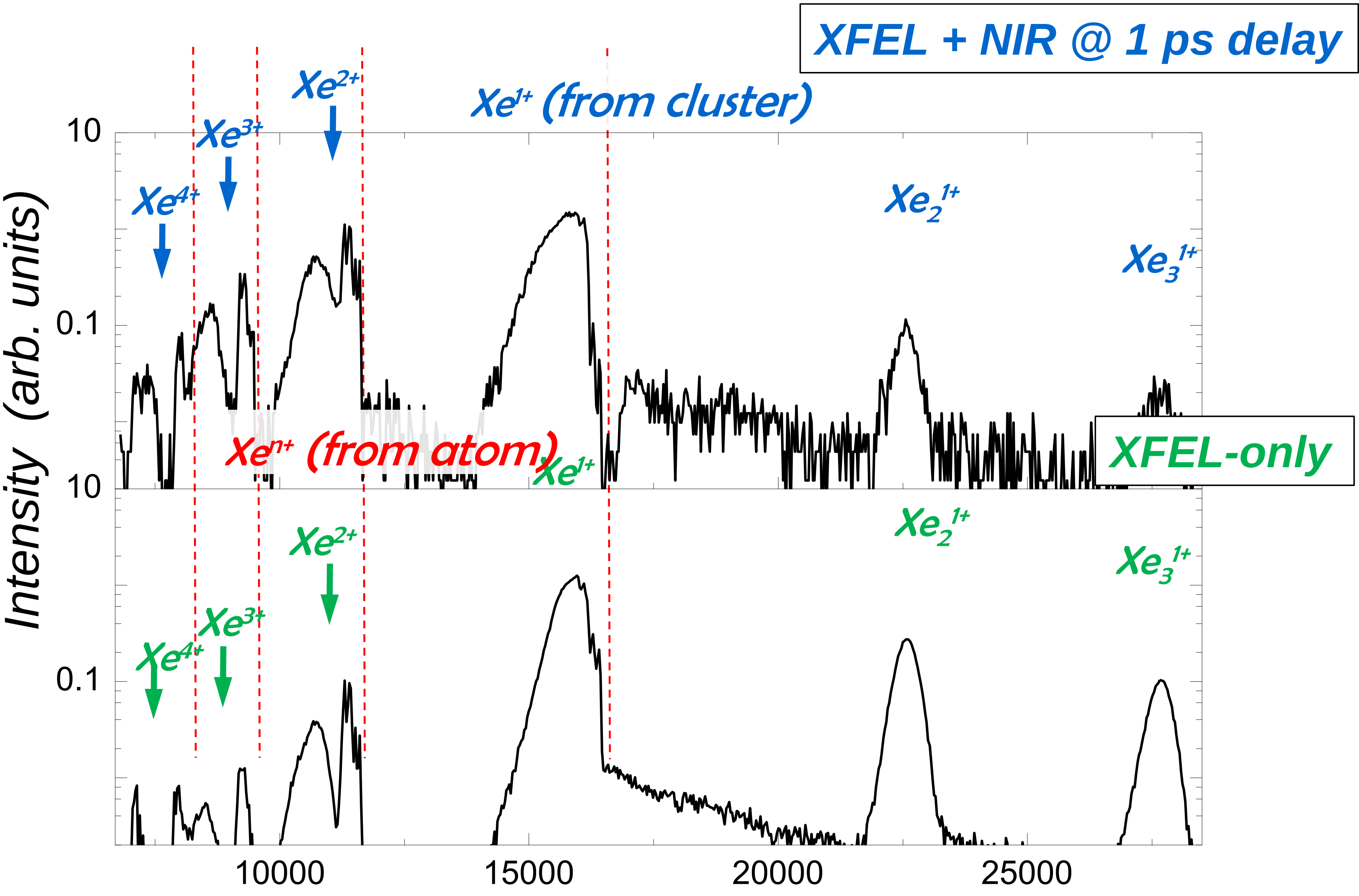
Size of Xe clusters: ~5000 atoms
Nozzle diameter: ~250 μm



K. Tono *et al.*, New J. Phys. **15**, 083035 (2013);
A. T. J. B. Eppink and D. H. Parker, Rev. Sci. Instrum. **68**, 3477 (1997); K. Motomura *et al.*, J. Phys. B. **46**, 164024 (2003)

Time-resolved TOF spectra of Xe clusters

XFEL: 16 μJ/μm²; NIR: 4.1 nJ/μm²



Outline

Introduction to XFEL science

Introduction to SACLA

Atomic and Molecular Physics at SACLA

– Deep inner-shell multi-photon ionization of Ar and Xe atoms
– Photoion-photoion coincidence imaging following deep inner-shell multi-photon ionization of CH3I and 5I-uracil
– Electron spectroscopy on cold nanoplasma formation from argon, krypton and xenon clusters
– Single-shot imaging of xenon nano-clusters
– IR-probe experiment of XFEL-ignited nanoplasma dynamics

Summary and outlook

Summary

- Deep inner-shell multi-photon absorption of Ar and Xe atoms by **SACLA** XFEL pulses – Electronic damage

- Photoion-photoion coincidence imaging following deep inner-shell multi-photon absorption by **SACLA** XFEL pulses (CH_3I, 5I-uracil) – Radiation damage in the atomic level

- Electron spectroscopy of argon and xenon clusters heated by **SACLA** XFEL pulses – Nanoplasma formation

- Single-shot imaging of xenon nano-clusters – Influence of the electronic damage to the imaging

- IR-probe experiment of XFEL induced nanoplasma formation – Nanoplasma dynamics

What's next…

- IR/XFEL pump – XFEL probe for I-contained molecules
  Intra-molecular charge transfer via ionic fragmentation
- X-ray imaging for UV/XFEL induced nanoplasma from giant clusters
  Influence of nanoplasma formation to X-ray imaging….
- UV pump – XFEL probe for photocatalytic molecules
  Intra-molecular charge transfer via TR X-ray spectroscopy
- Serial femtosecond X-ray crystallography
  Phasing vs radiation damage


The end

Thank you very much for your attention!