PAUL SCHERRER INSTITUT

Wir schaffen Wissen – heute für morgen

FEL Beamline Design and Experimental Methods
or *How to Get All Those Lovely Photons Onto My Sample and Measure Something Cool*

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Let's pretend we're starting from the experiment

In The Beginning There Was an Awesome Pulsed Source of Femtosecond X-rays

What are the priorities ?

What do we want to measure ?

How do I know anything about the X-rays ?

How do I get the X-rays onto my experiment ?

What kind of X-rays and laser do I need ?

Guaranteed: Ultrafast photochemistry and photobiology

Electronic relaxation in nanoparticles

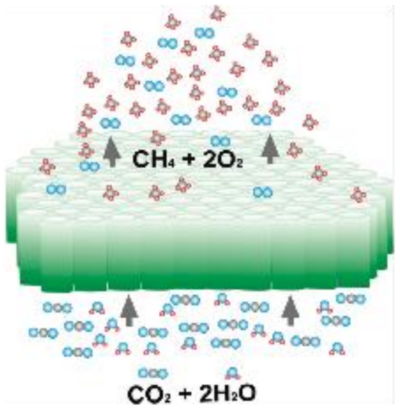
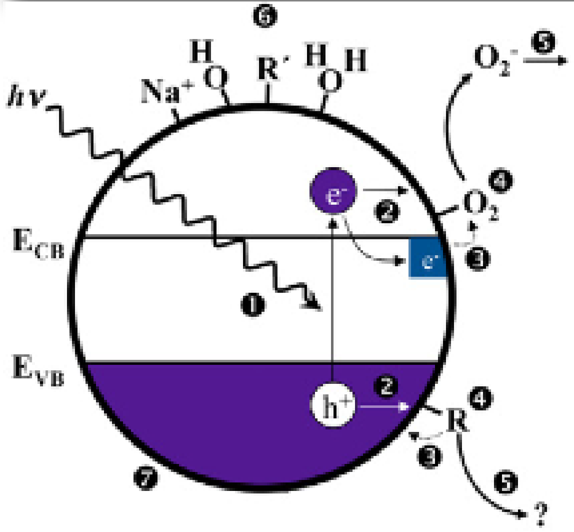


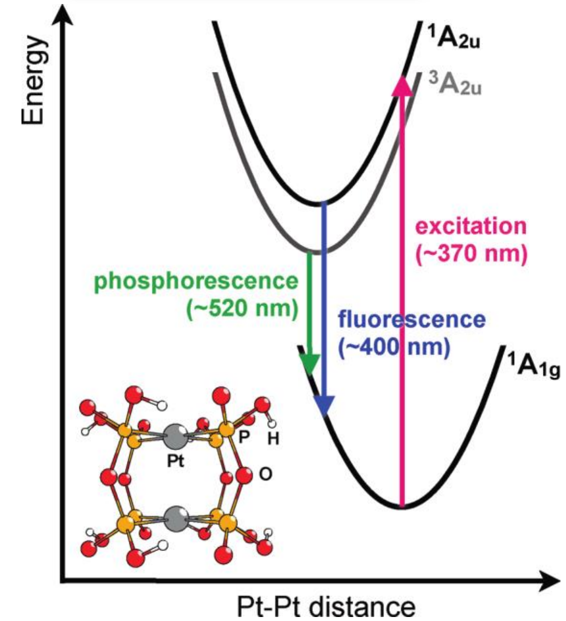
Figure 16. Depiction of flow-through photocatalytic membrane for CO_2 conversion.

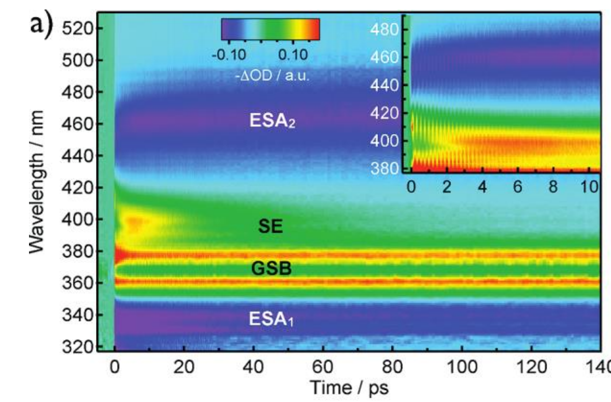


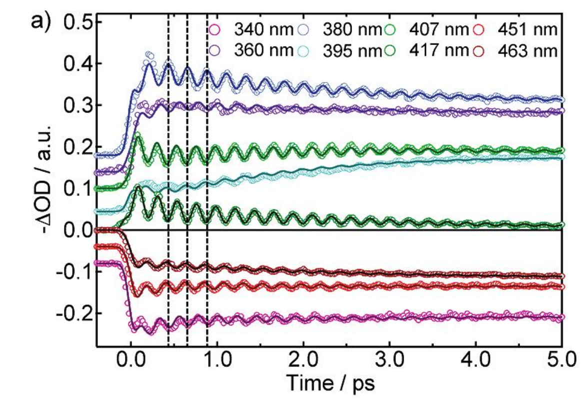
Important issues
1. excitation
2. charge transport and trapping
3. charge transfer
4. molecular adsorption
5. reaction mechanisms
6. poisons and promoters
7. surface and material structure

A. Listorti et al. *Rev. Chem. Mater.* **23**, 3381 (2011);
A. Hagfeldt et al. *Chem. Rev.* **110**, 6595 (2010);
M. Henderson *Surf. Sci. Rep.* **66**, 185 (2011);
S.C. Roy et al. *ACS Nano* **4**, 1259 (2010)

Photochemistry



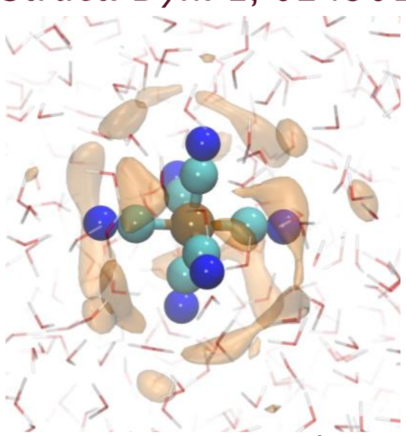




R. van der Veen et al. *JACS* **133**, 305 (2011)

Bond breaking and bond making

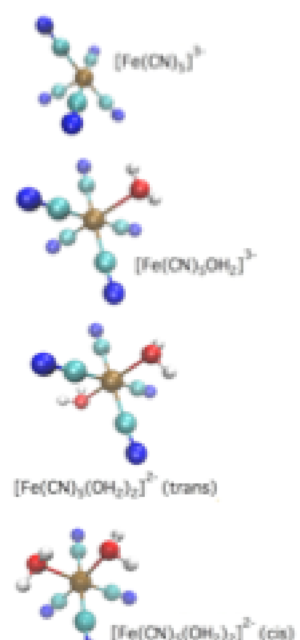
M. Reinhard, T.J. Penfold et al.
Struct. Dyn. **1**, 024901 (2014)



hν

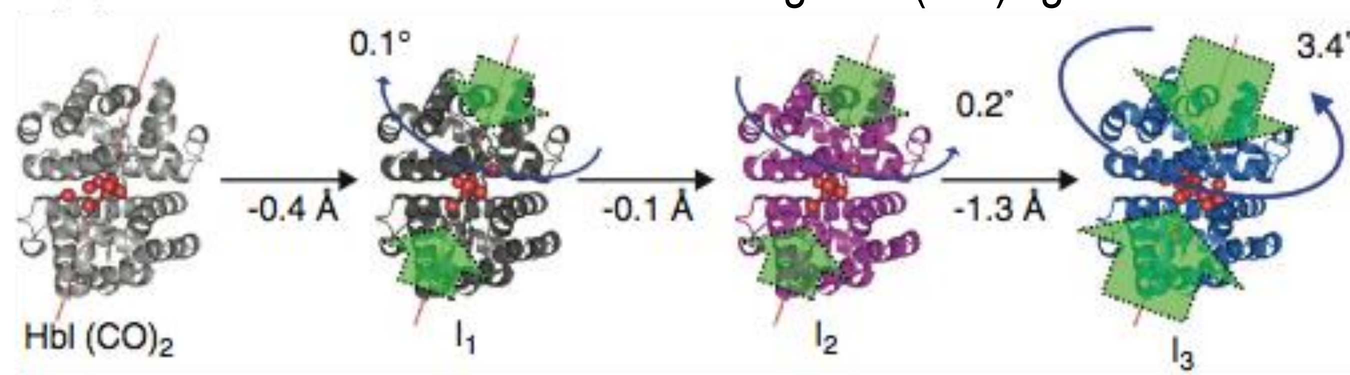
355 nm

$[Fe(CN)_6]^{4-}$



Protein structure and function

Intermediate states of homodimeric hemoglobin (HbI) ligated with CO



R. Neutze and K. Moffat *Curr. Op. Struc. Bio.* **22**, 651 (2012)

Nobel Prize for Chemistry 1999: Femtochemistry

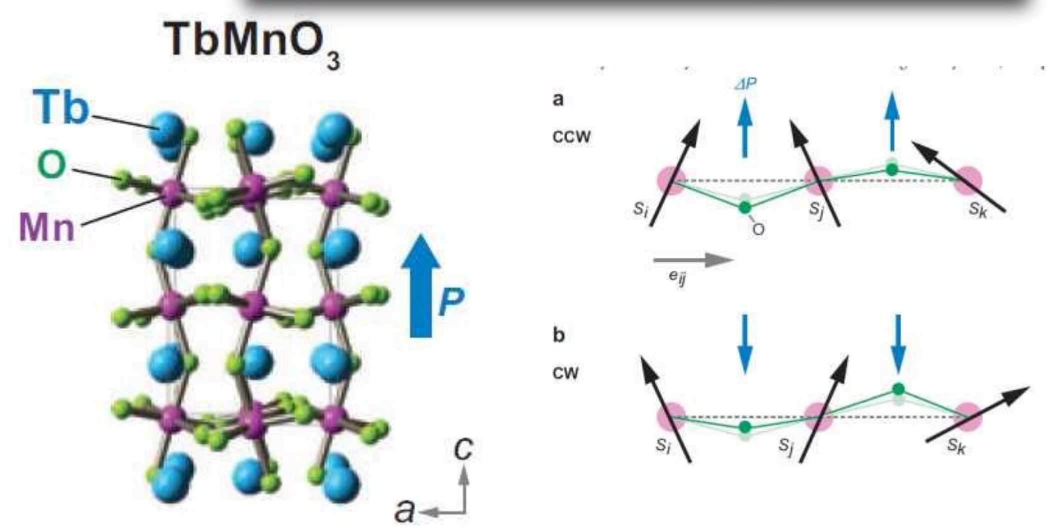
Guaranteed: Strongly correlated electron systems

1. Understanding correlated materials studying the dynamic interaction of lattice, orbital, charge & spin.

2. Use ultrashort pulses to manipulate materials on an ultrafast time scale

- magnetic structure
- electronic polarization

Electric field controls magnetic order



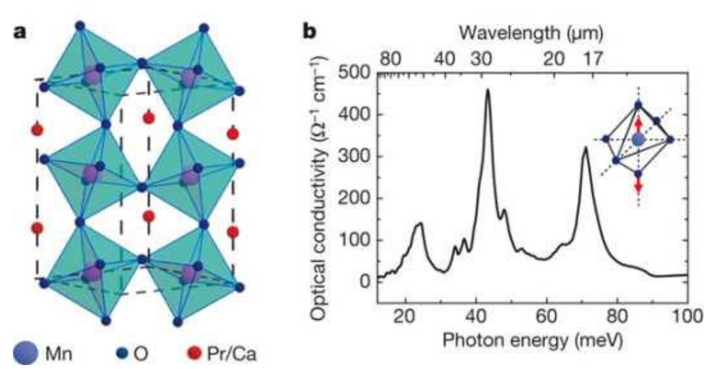
Kimura, Ann. Rev. Mater. Res. **37**, 387

- Large amplitude electro-magnon excited with THz pulse

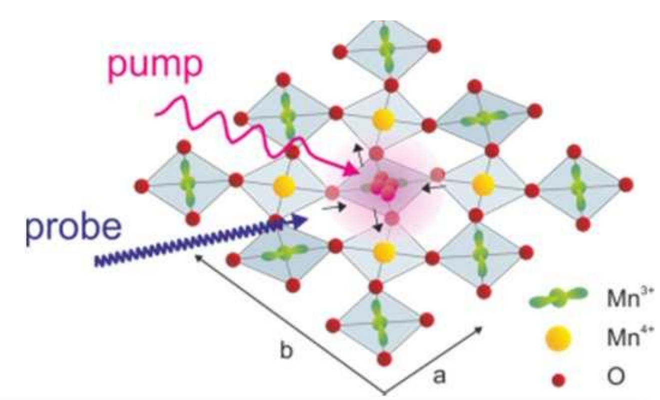
Vibrationally induced phase transitions

Insulator-metal transition
Rini *et al*. Nature (2007)

$Pr_{0.7}Ca_{0.3}MnO_3$



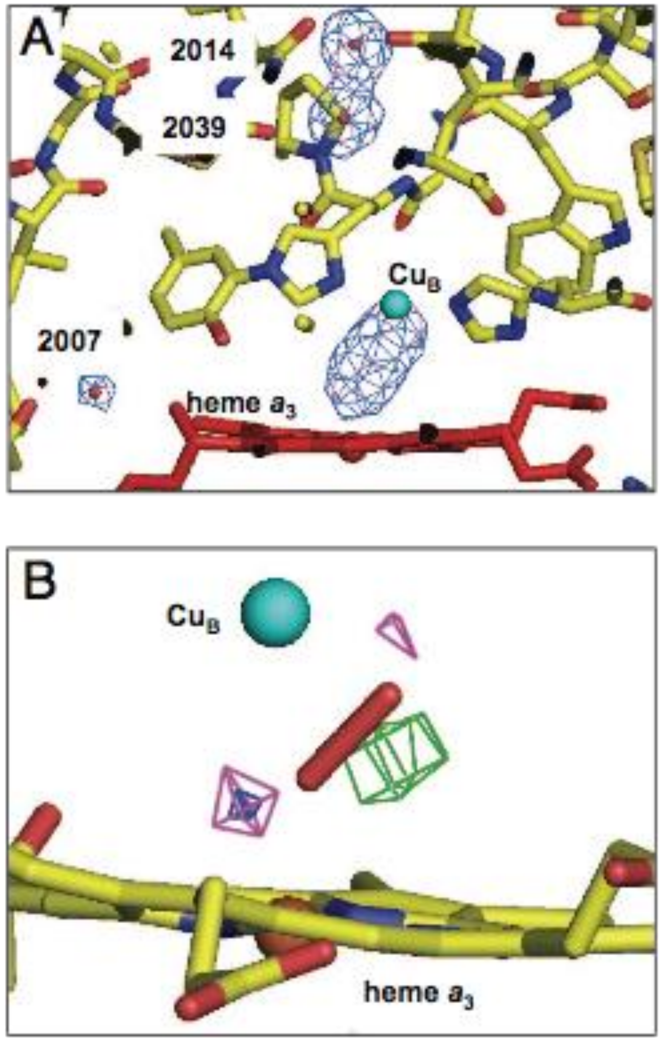
- Excitation: d-d transition in Mn^{3+}/Mn^{4+} system
- Fast removal of orbital order triggers collapse of Jahn-Teller distortion

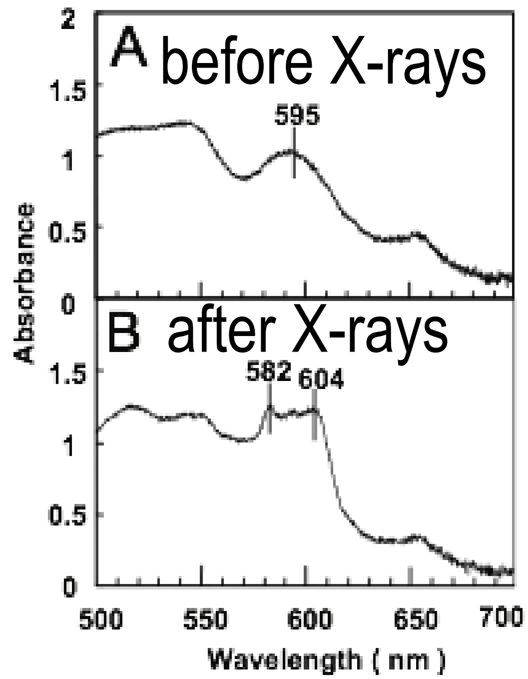


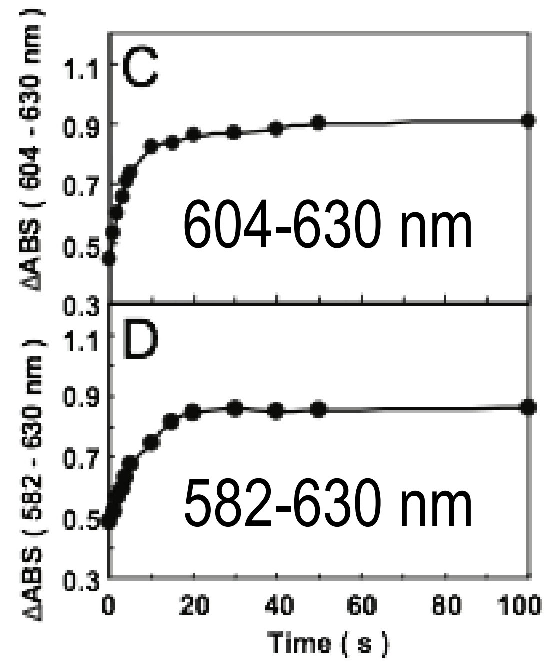
Structural and charge order dynamics

Courtesy of Paul Beaud and Gerhard Ingold

Speculative: more accurate protein structures ?



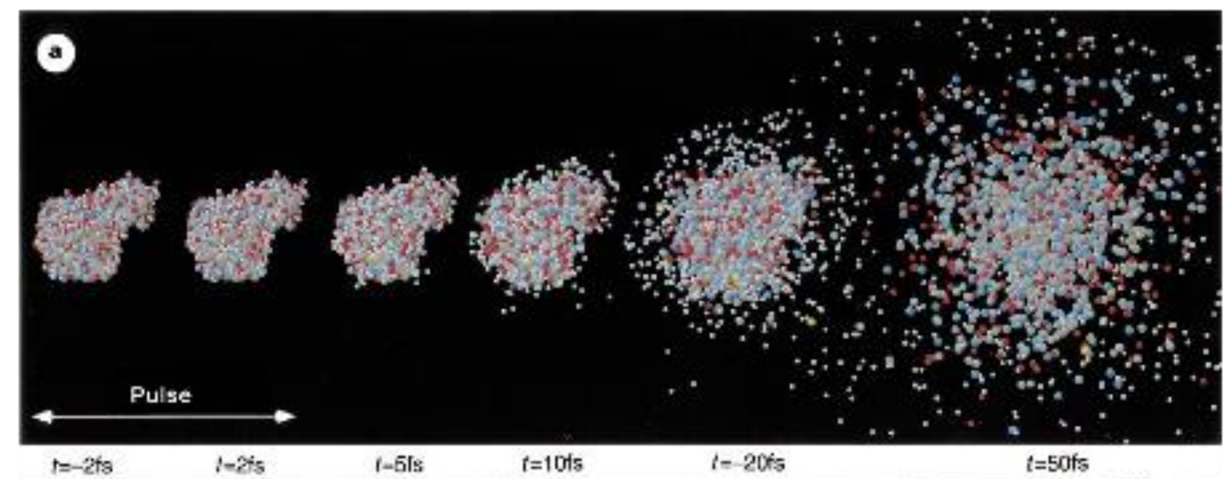


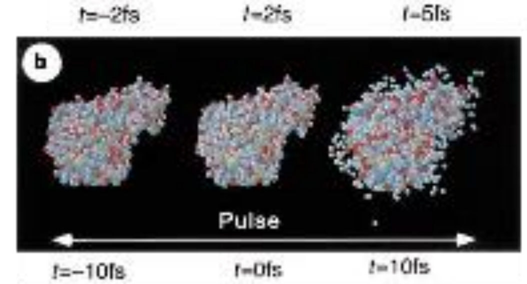


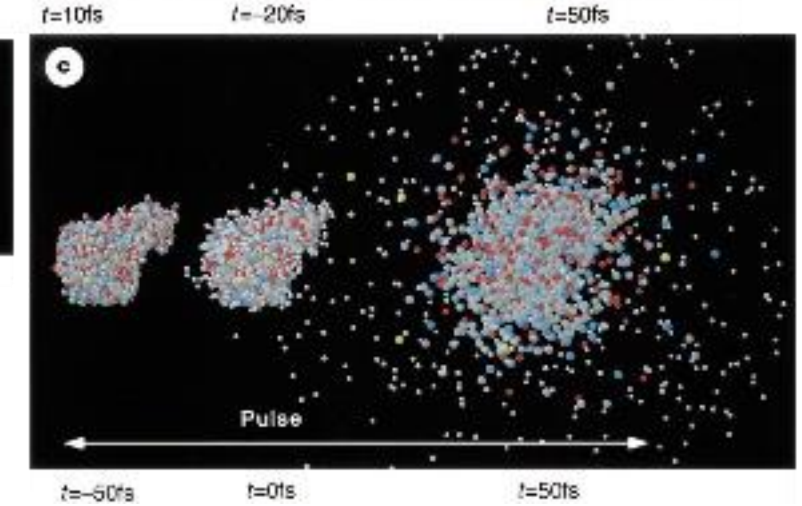
Cytochrome c oxidase is very sensitive to radiation damage

Long X-ray exposure leads to errors in the active part of the protein

H. Aoyama et al. *PNAS* **106**, 2165 (2009)







Shorter X-ray pulses should at least avoid measuring the structural damage

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

Speculative: nonlinear X-ray spectroscopy ?

Hypersatellite lines

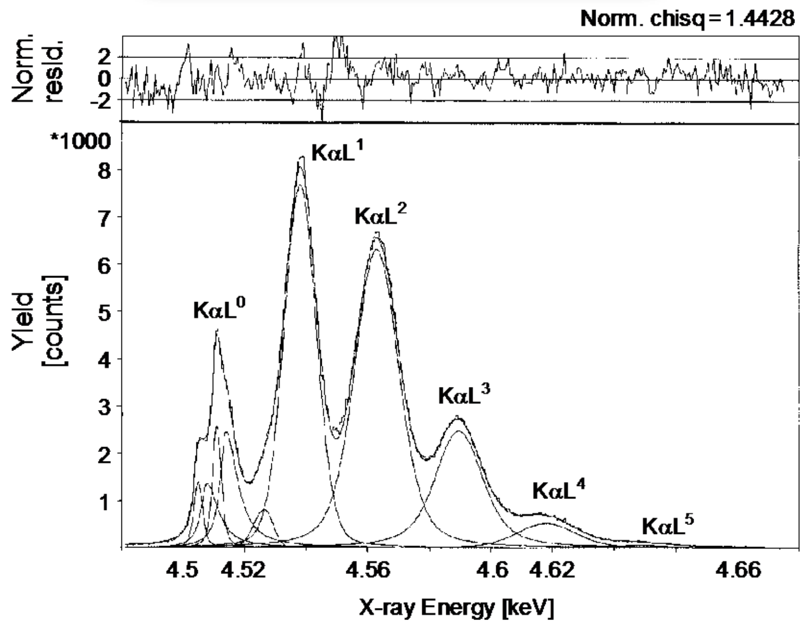
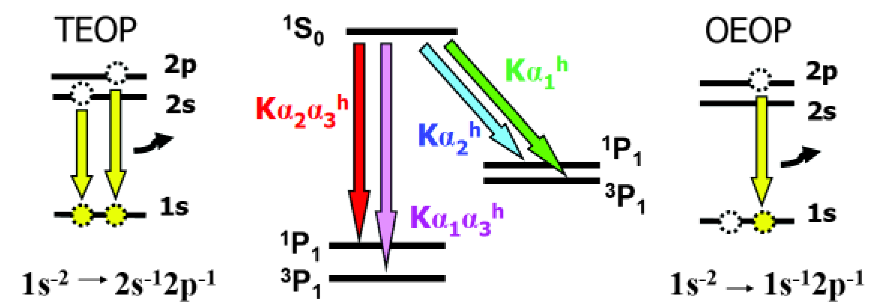


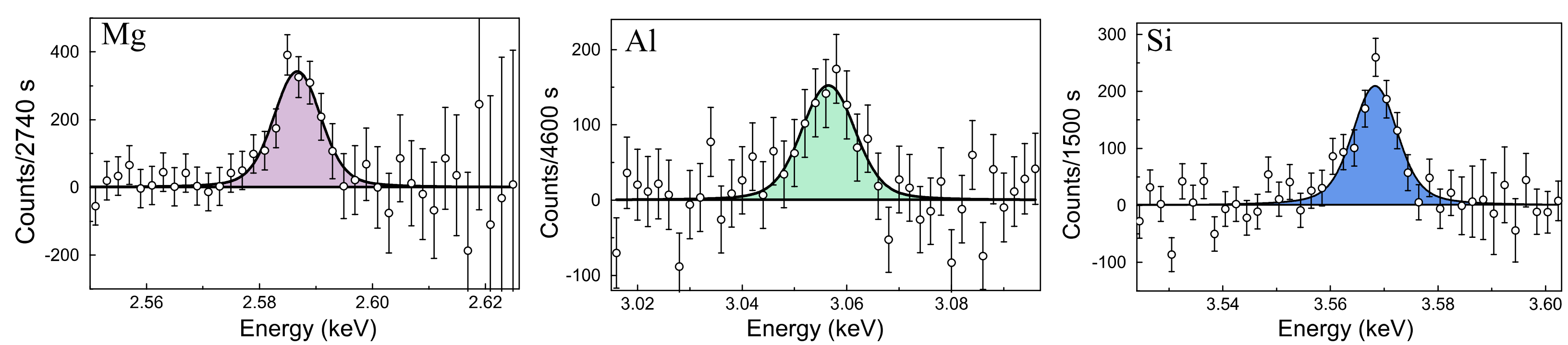
Figure 1. K$_{\alpha}$ L^{N} satellite spectrum of Ti ionized by 64 MeV O ions. The spectrum was fitted with the model presented in the text. Voigt profiles were used for the satellite lines. The KL^{0} line was fitted with two Voigt functions of fixed widths obtained from the photoinduced spectra and two asymmetric lines describing the M satellites.

M. Kavčič et al. *X-ray Spectr.* **32**, 381 (2003)

Low-probability core-hole processes might become measurable with high peak powers

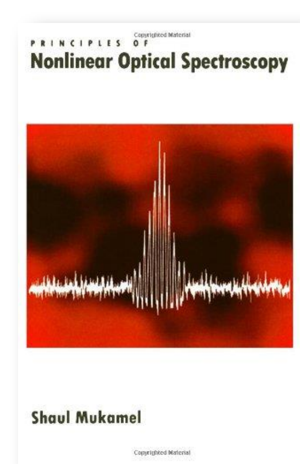
Two-electron One photon

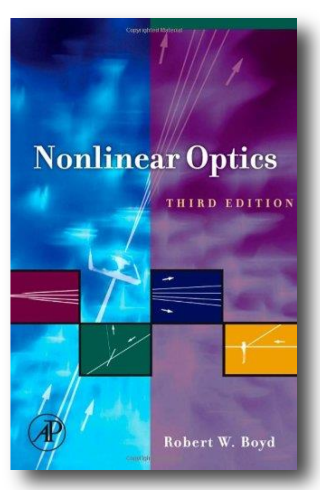




J. Hoszowska et al. *Phys. Rev. Lett.* **107**, 053001 (2011)

There's also significant ideas available from nonlinear optics and nonlinear spectroscopy in the optical regime





What are the priorities ?

~~What do we want to measure ?~~

How do I get the X-rays onto my experiment ?

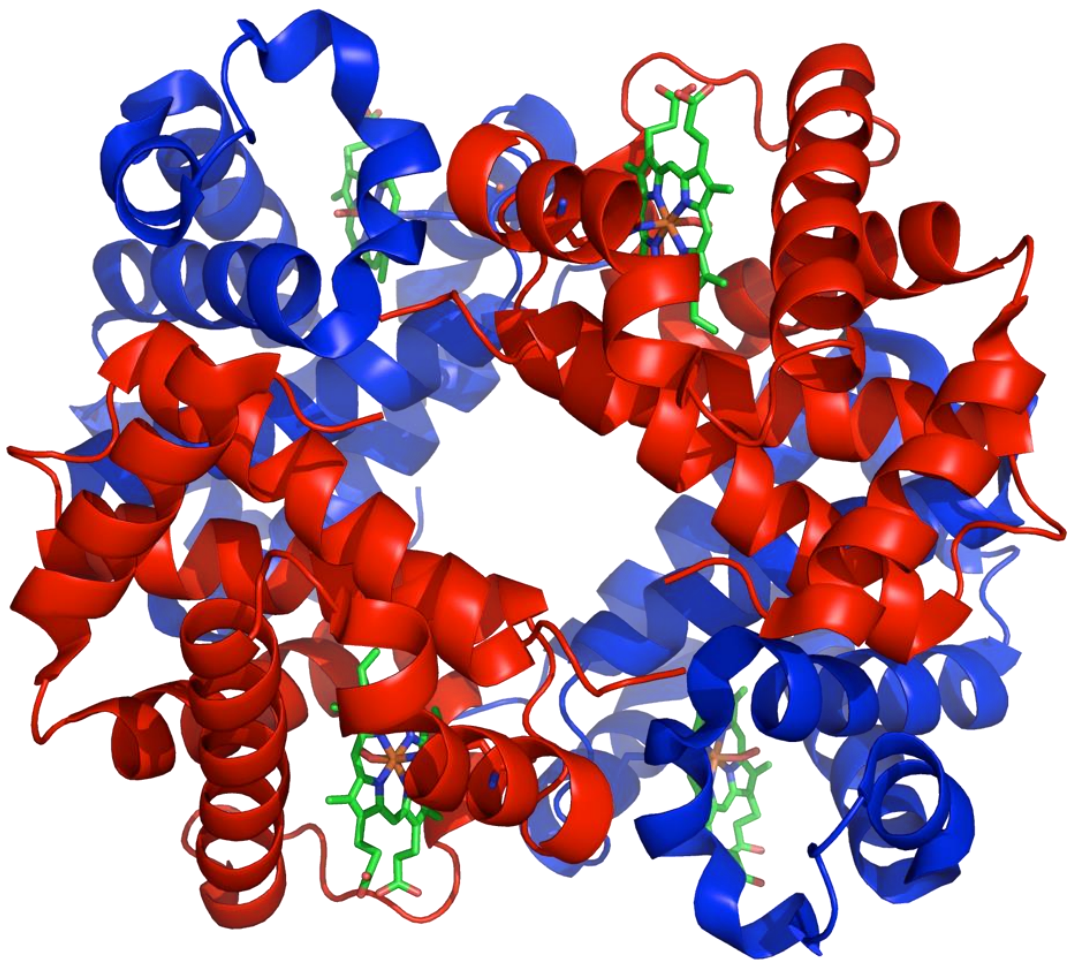
How do I know anything about the X-rays ?

What kind of X-rays and laser do I need ?

Is function structure or dynamics ?

Structure

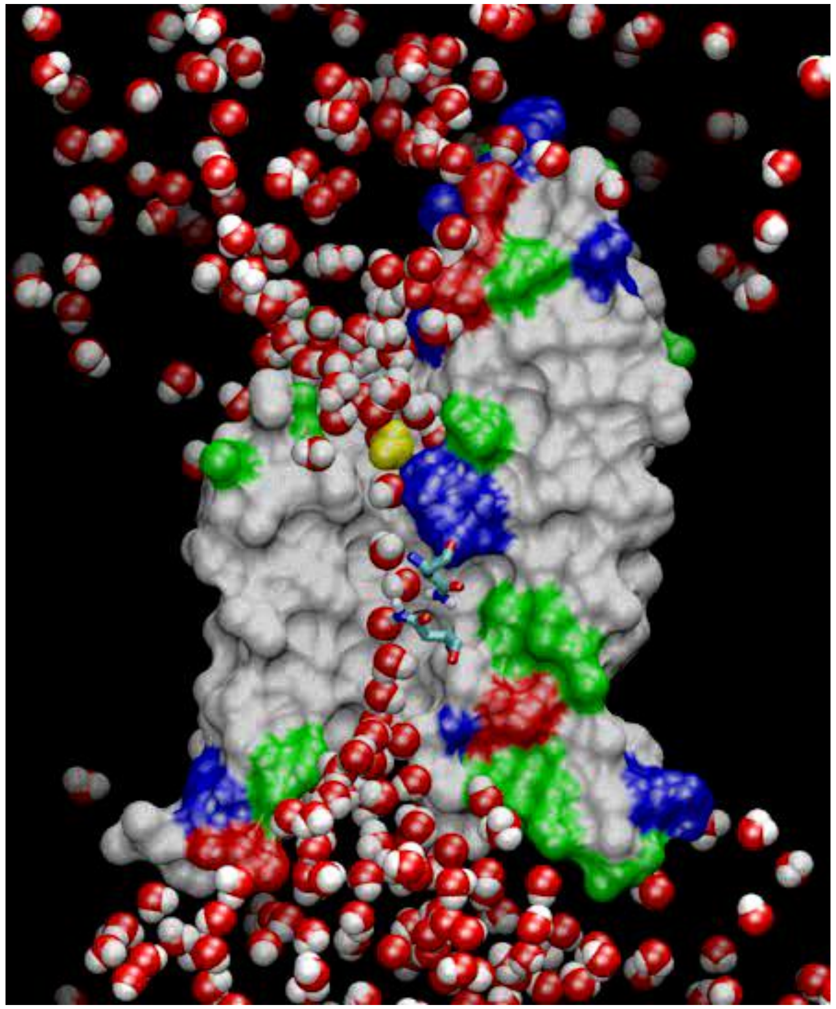
- X-ray crystallography
- electron microscopy
- atomic force microscopy
- electron diffraction
- X-ray absorption spectroscopy
- NMR



Protein structure of human hemoglobin in the T-state with oxygen bound at all 4 hemes (from PDB 1GZX Wikipedia)

Dynamics

- Laser spectroscopy
- NMR
- time-resolved diffraction
- X-ray absorption spectroscopy



Water transport through an aquaporin channel in a cell membrane
http://www.ks.uiuc.edu/Research/aquaporins/
Tajkhorshid, E., Nollert, P., Jensen, M.O., Miercke, L.J., O'Connell, J., Stroud, R.M., and Schulten, K. (2002). Science 296, 525-530

On what timescale do we want to measure structure ?

X-rays

laser plasma

X-FELs

laser-slicing

synchrotrons

high-harmonic generation

electronics

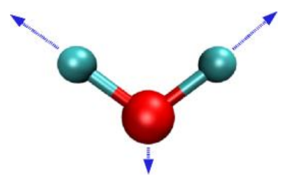
lasers

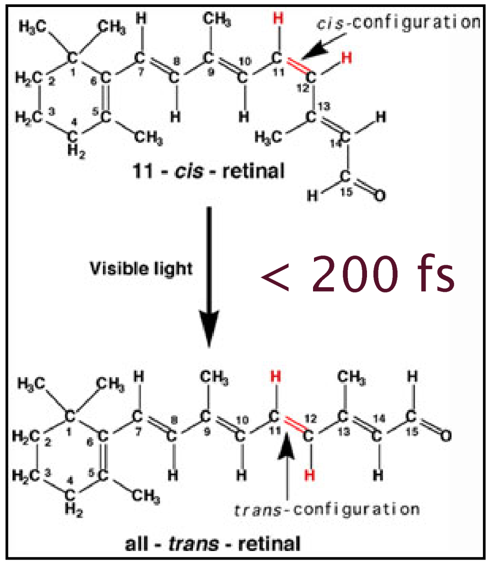
attoseconds 10^{-18} s	femtoseconds 10^{-15} s	picoseconds 10^{-12} s	nanoseconds 10^{-9} s	microseconds 10^{-6} s	milliseconds 10^{-3} s
electron motion core-hole lifetimes	motion of nuclei molecular vibrations optical phonons molecular bond formation	rotation of molecules molecular librations vibrational dephasing	electronic relaxation fluorescence thermal effects	large-scale protein motion phosphorescence	protein folding chemical kinetics

110 as delay between electron emission from conduction band and lower-lying states in Tungsten upon irradiation

The Fe K-edge core-hole lifetime is 4 fs

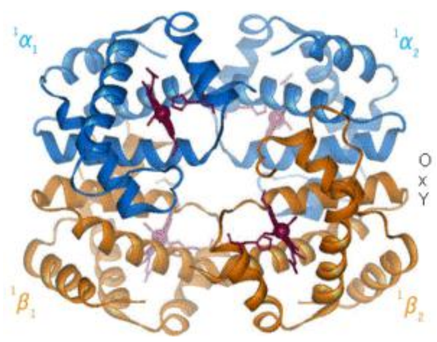
period of the symmetric stretch in H_2O is 10 fs





< 200 fs

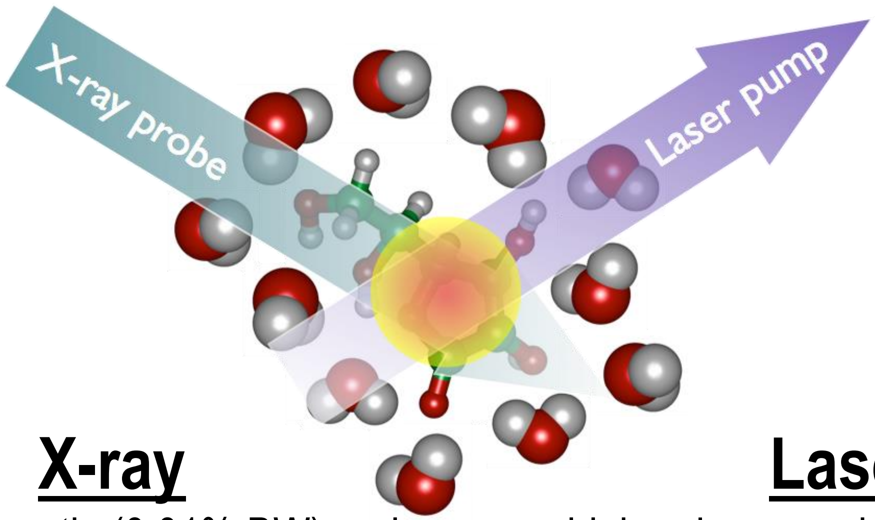
Hemoglobin R→T transition takes microseconds



Camera shutter speeds range from ms through to seconds

Hb animation from Wikipedia

X-ray probe techniques for disordered systems



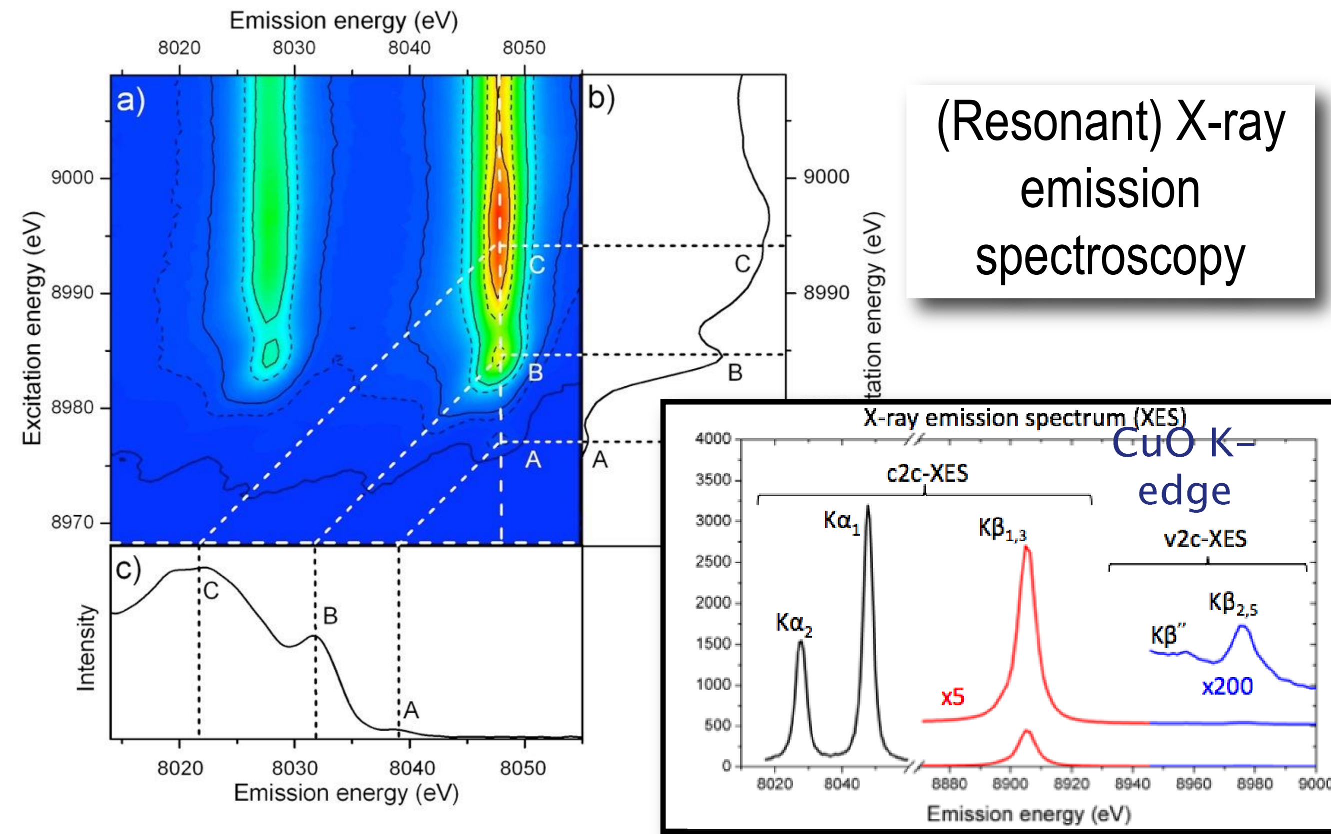
X-ray

- monochromatic (0.01% BW) and broadband (1-4%)
- variable focus (1-100 μm)
- tuneable energy (2-12.4 keV)
- ultrashort pulse durations (<1 fs to 50 fs)

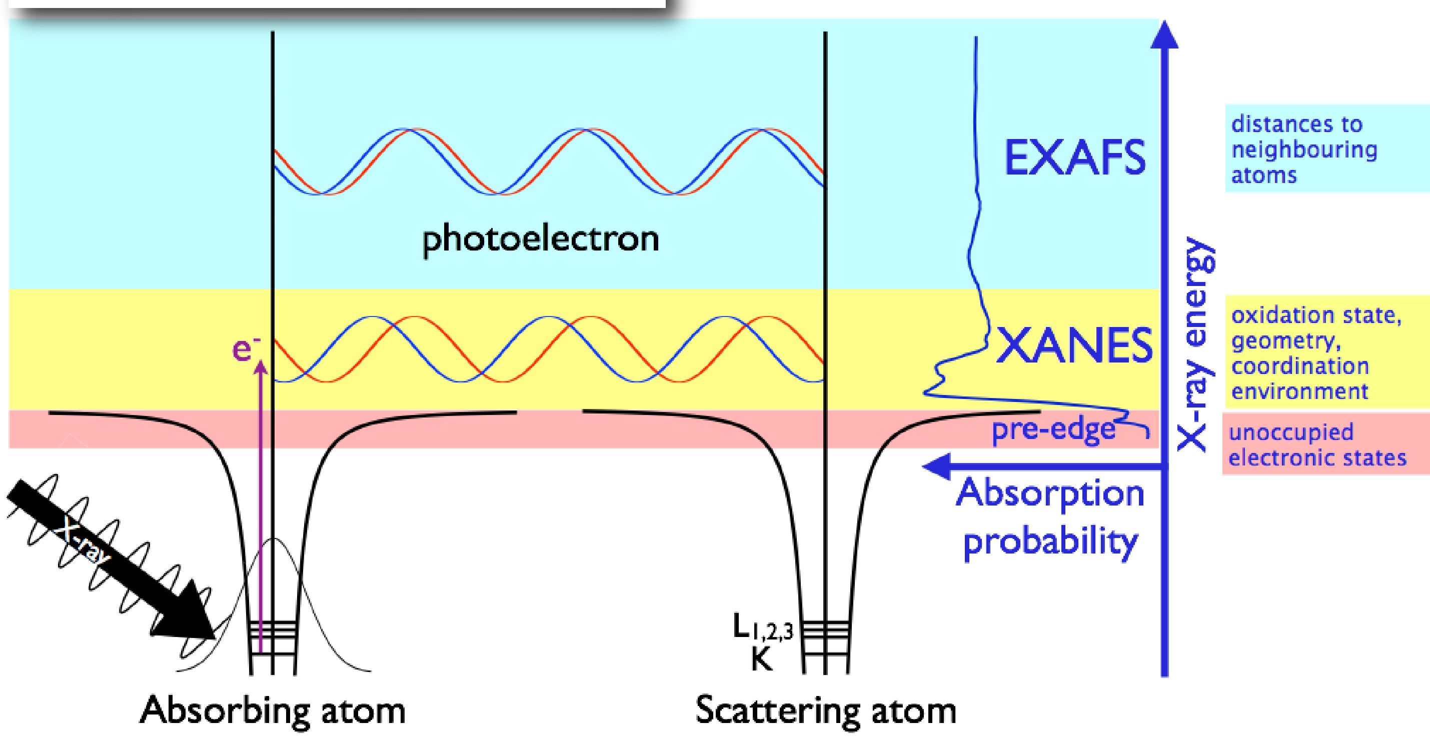
Laser

- high pulse energies (5-10 mJ)
- short pulses (20-50 fs)
- tuneable wavelengths including IR, visible, and UV
- preparation for THz and <10 fs

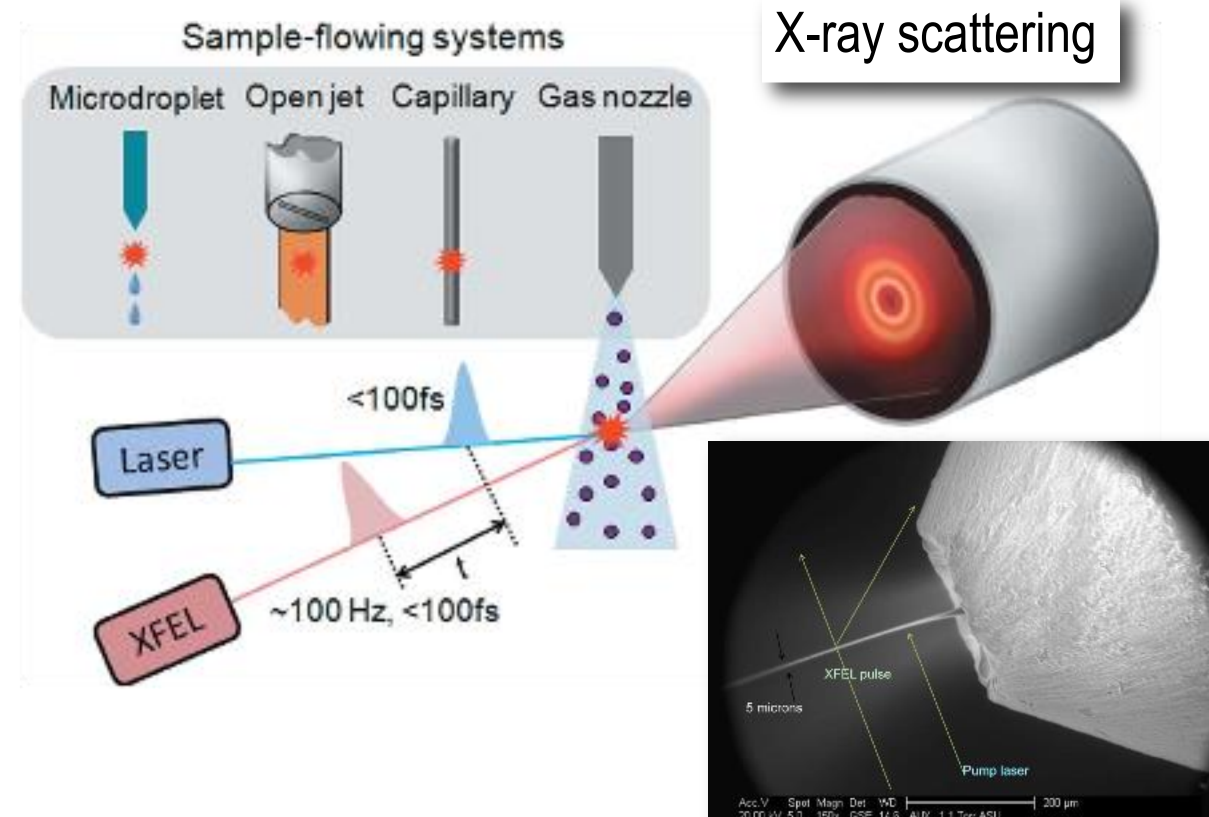
(Resonant) X-ray emission spectroscopy



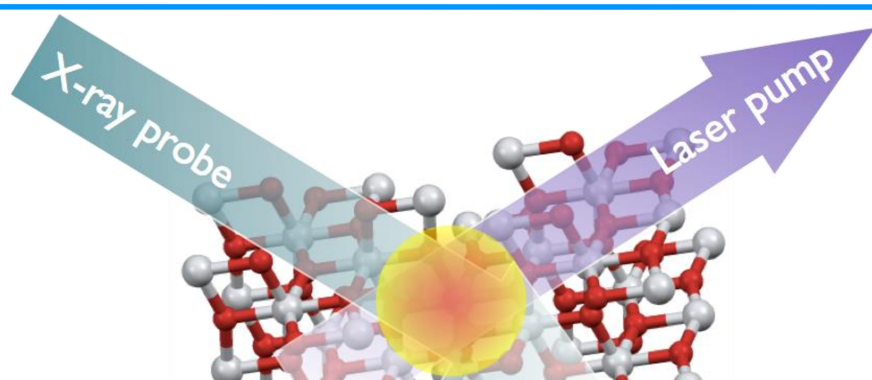
X-ray absorption spectroscopy



X-ray scattering



X-ray probe techniques for ordered systems



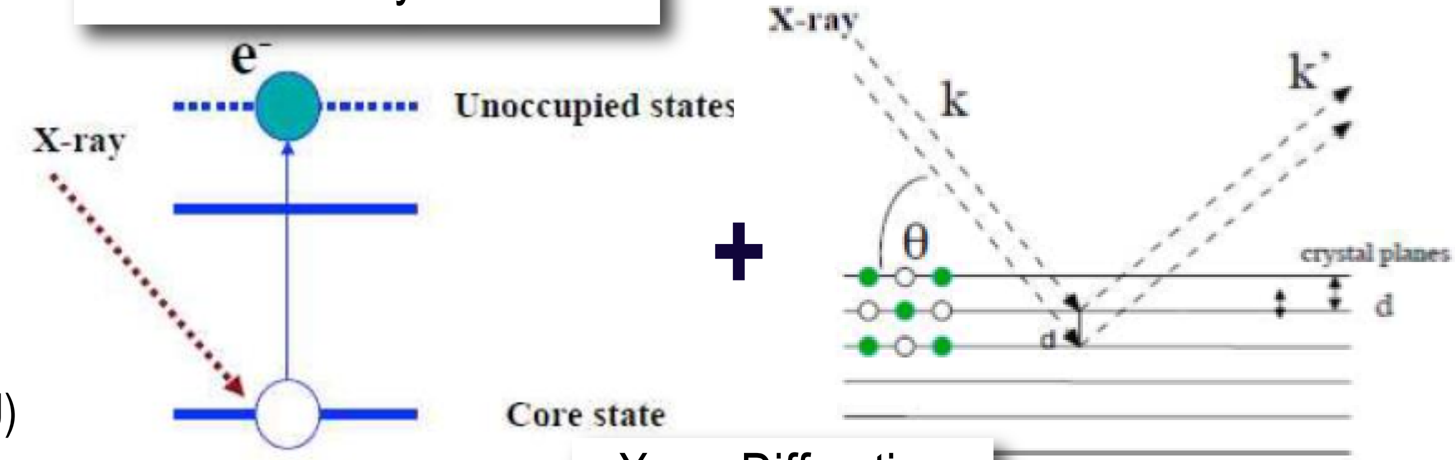
X-ray

- monochromatic Si(111) and Si(311) and broadband (1-4%)
- variable KB mirror focus 1-100 µm
- tuneable energy (2)5-12.4 keV
- ultrashort pulse durations (<1 fs to 50 fs)

Laser

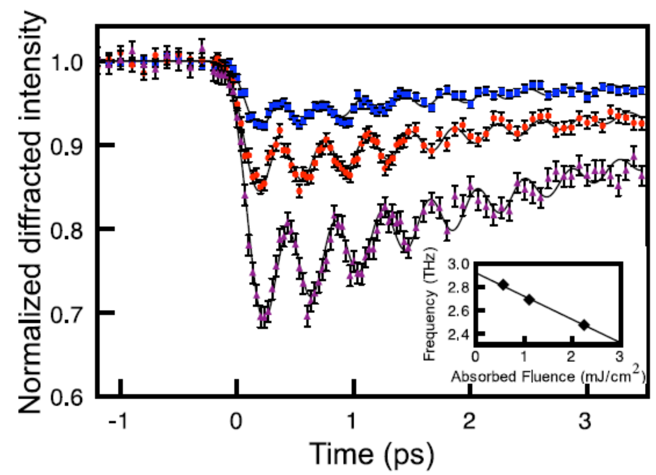
- high pulse energies (5-10 mJ)
- short pulses (10-20 fs)
- flexible pump setup (polarization and wavelength UV to NIR)
- THz excitation and <10 fs

Resonant X-ray Diffraction



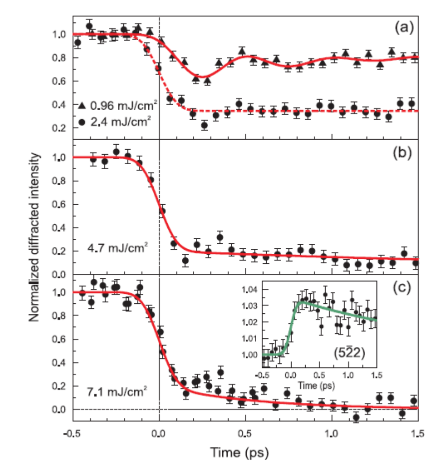
X-ray Diffraction

Peierls system: coherent phonons (Bi)



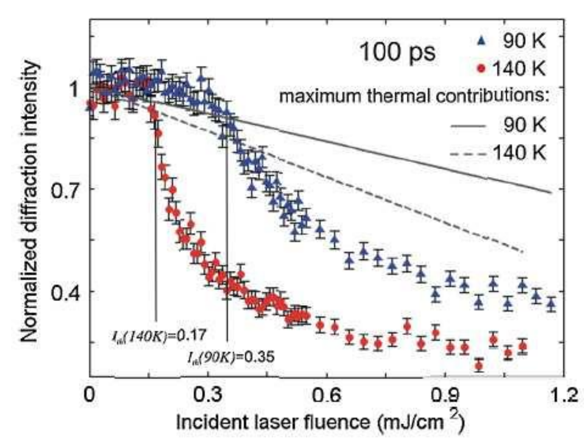
[Johnson PRL 2008]

Jahn-Teller dynamics (LCMO)



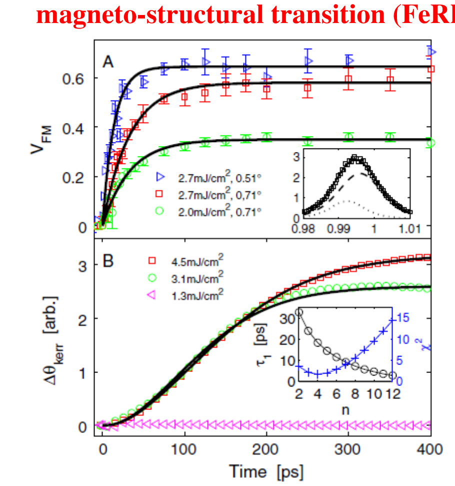
[Beaud PRL 2009]

excitonic CDW system ($TiSe_2$)



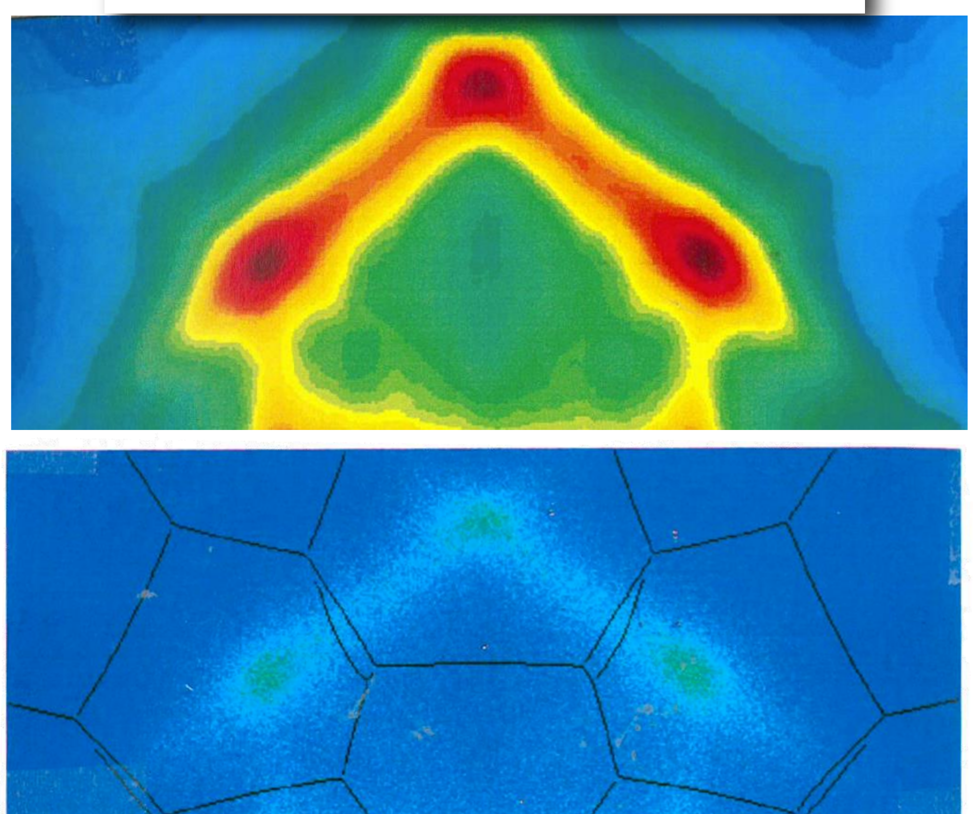
[Vorobeva PRL 2011]

magneto-structural transition (FeRh



[Mariager PRL 2012]

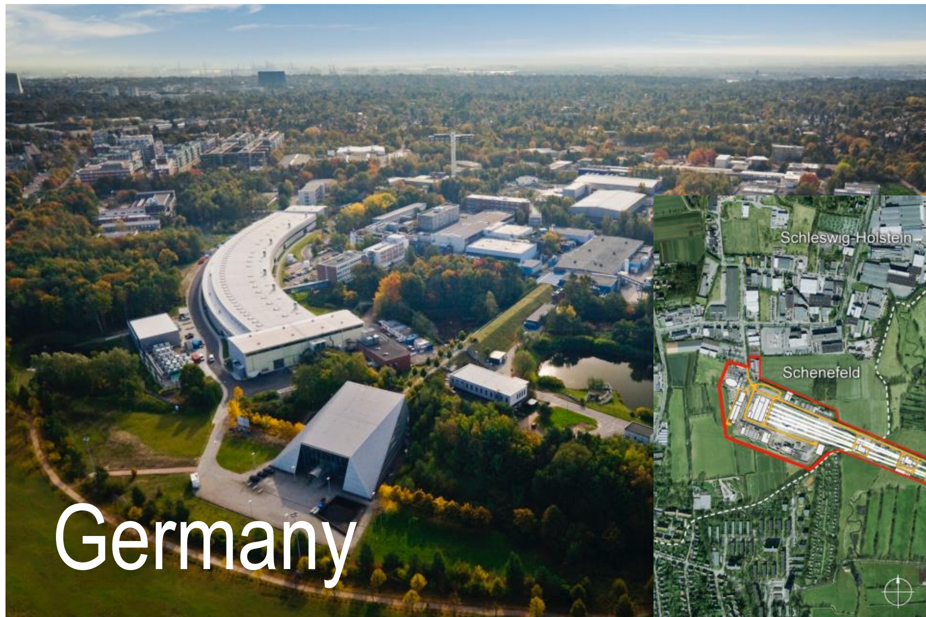
Elastic X-ray Diffuse Scattering

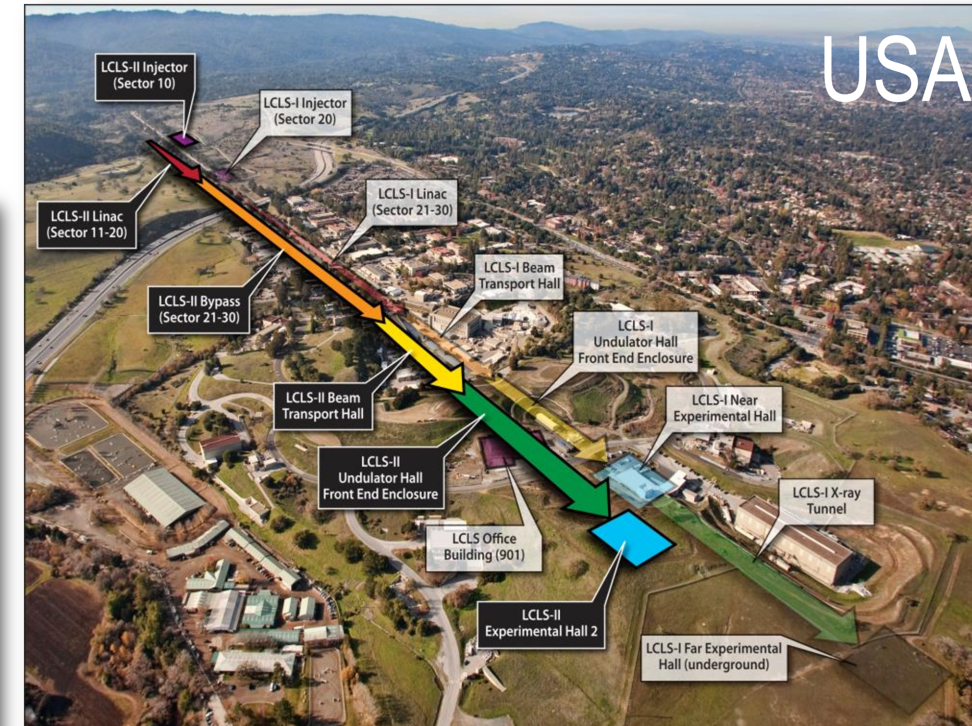


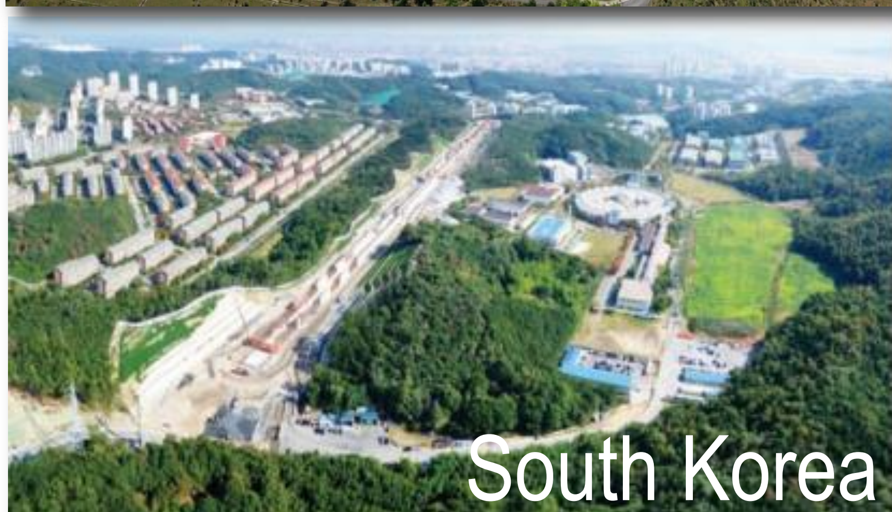
TDS from Si(100) measured at the SLS (courtesy J. Johnson)

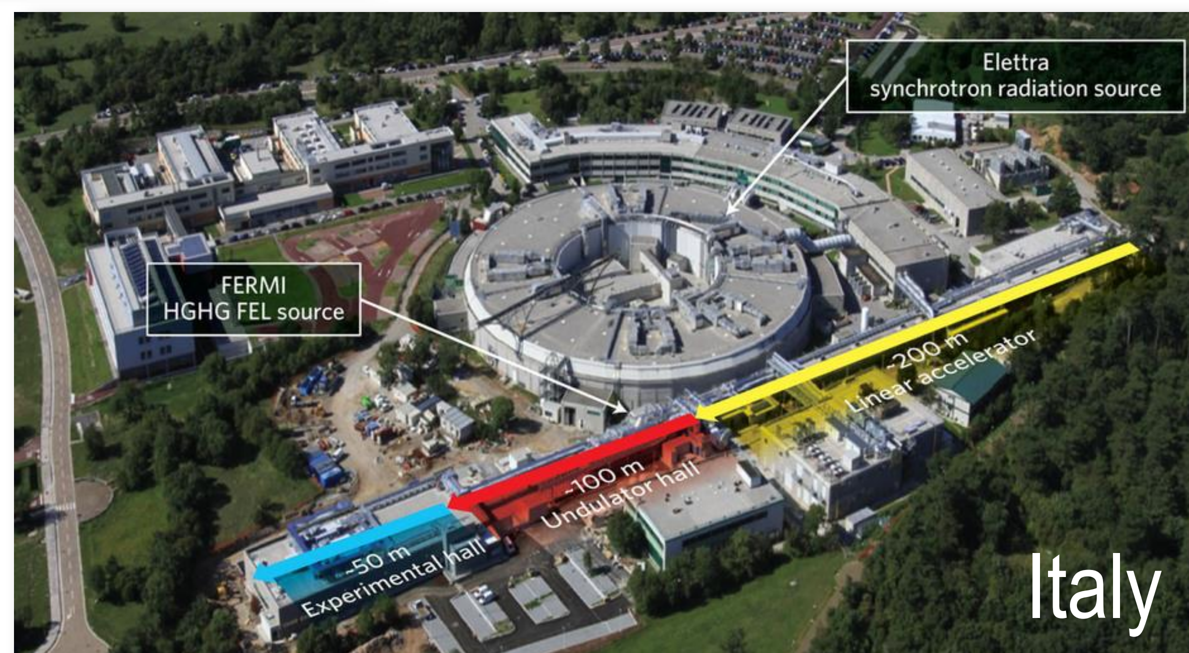

What are the priorities ?
~~What do we want to measure ?~~
How do I know anything about the X-rays ?
How do I get the X-rays onto my experiment ?
~~What kind of X-rays and laser do I need ?~~

Now we just need an XFEL…





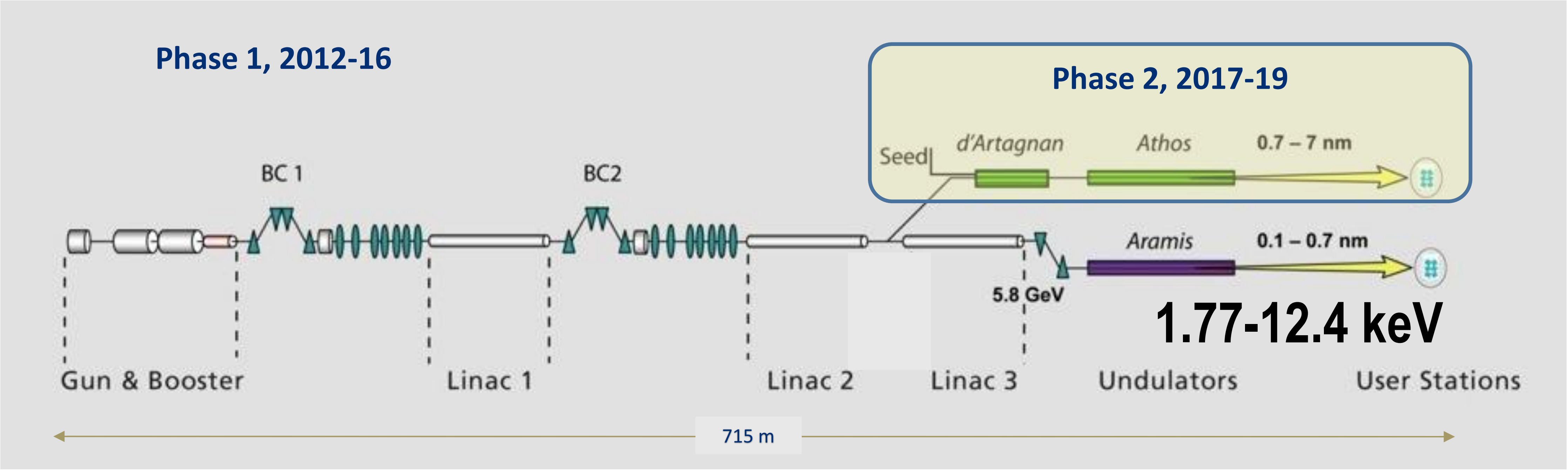




But these ones are all much too far away….

Why don't we build our own ?

SwissFEL



Aramis: 1-7 Å hard X-ray SASE FEL,
In-vacuum , planar undulators with variable gap.
User operation from mid 2017

Athos : (2nd phase) 7-70 Å soft X-ray FEL for SASE & Seeded operation .
APPLE II undulators with variable gap and full polarization control.
To be implemented after 2017

SwissFEL parameters

Wavelength from	1 Å - 70 Å
Photon energy	0.2-12 keV
Photon / pulse (1Å)	7.3×10^{10}
Pulse duration	1 fs - 20 fs
Energy bandwidth	0.05-0.16%
e^- Energy	5.8 GeV
e^- Bunch charge	10-200 pC
Repetition rate	100 Hz

Reflective X-ray Optics

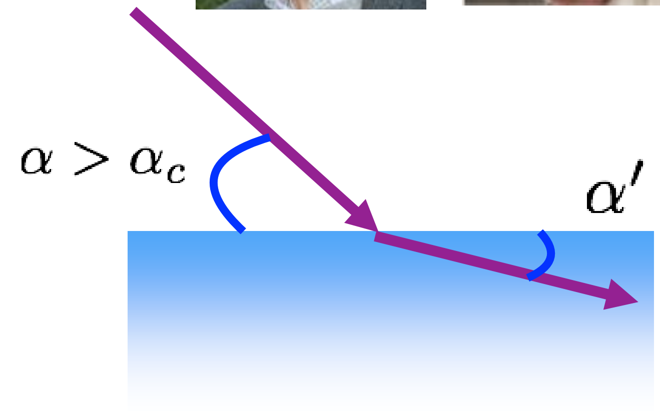


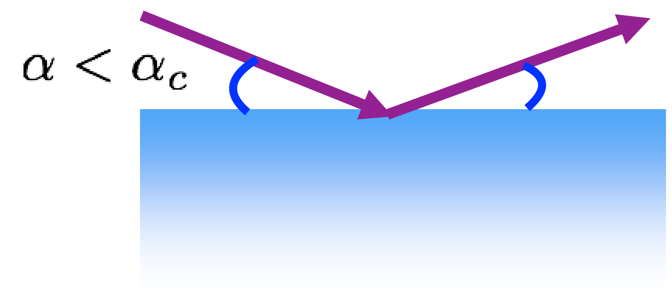
We need to steer the X-rays to the experiment

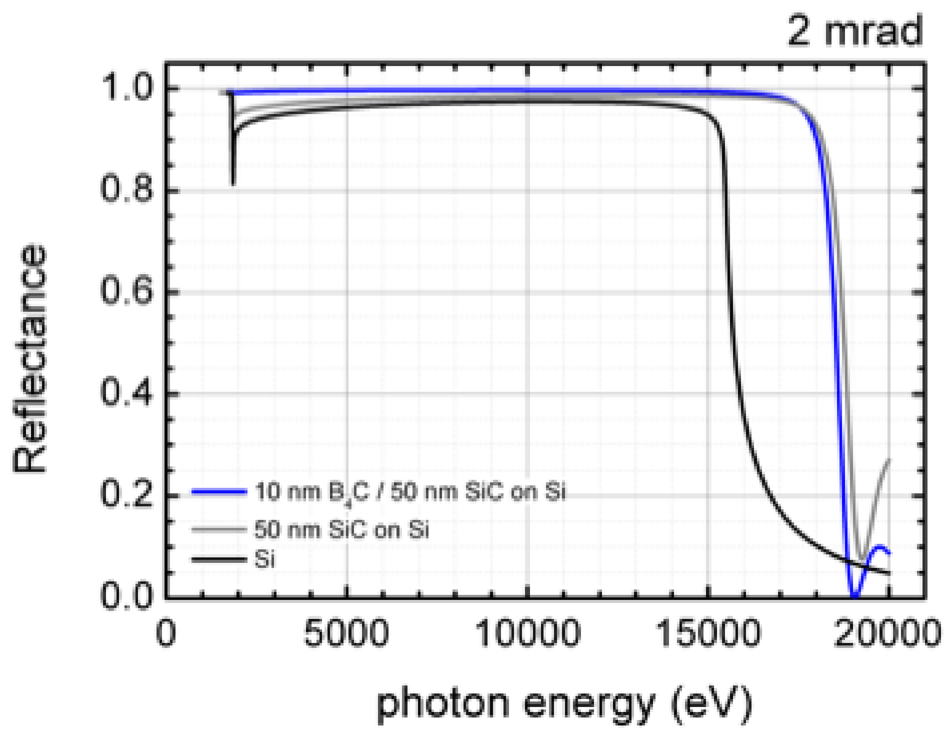
$$\frac{\cos\alpha}{\cos\alpha'} = n_R$$

$$\cos\alpha_c = n_R$$

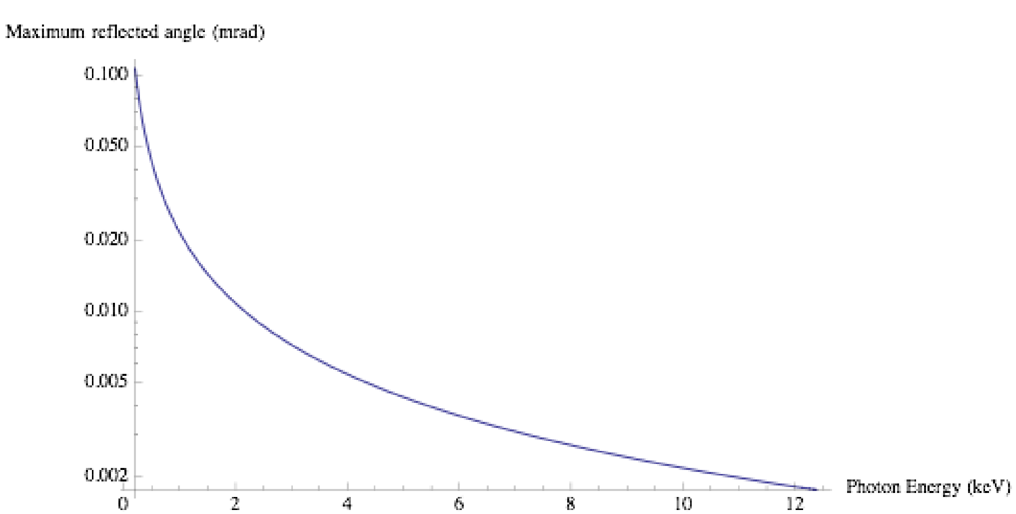
At grazing angles below the critical angle we get total external reflection







$$\alpha[degrees] \sim \lambda[\text{Å}]/10$$



Courtesy of Rolf Follath

X-ray mirrors for XFELs

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We need to **know**:
- Photon energy range
- Beam size on the mirror
- Pulse energy and average power

We need to **optimize**:
- No absorption edges
- Maximum throughput
- Reflected angle
- Mirror length

28.11.2012, updated. 23.9.2013

200 pC, 21 fs (rms)

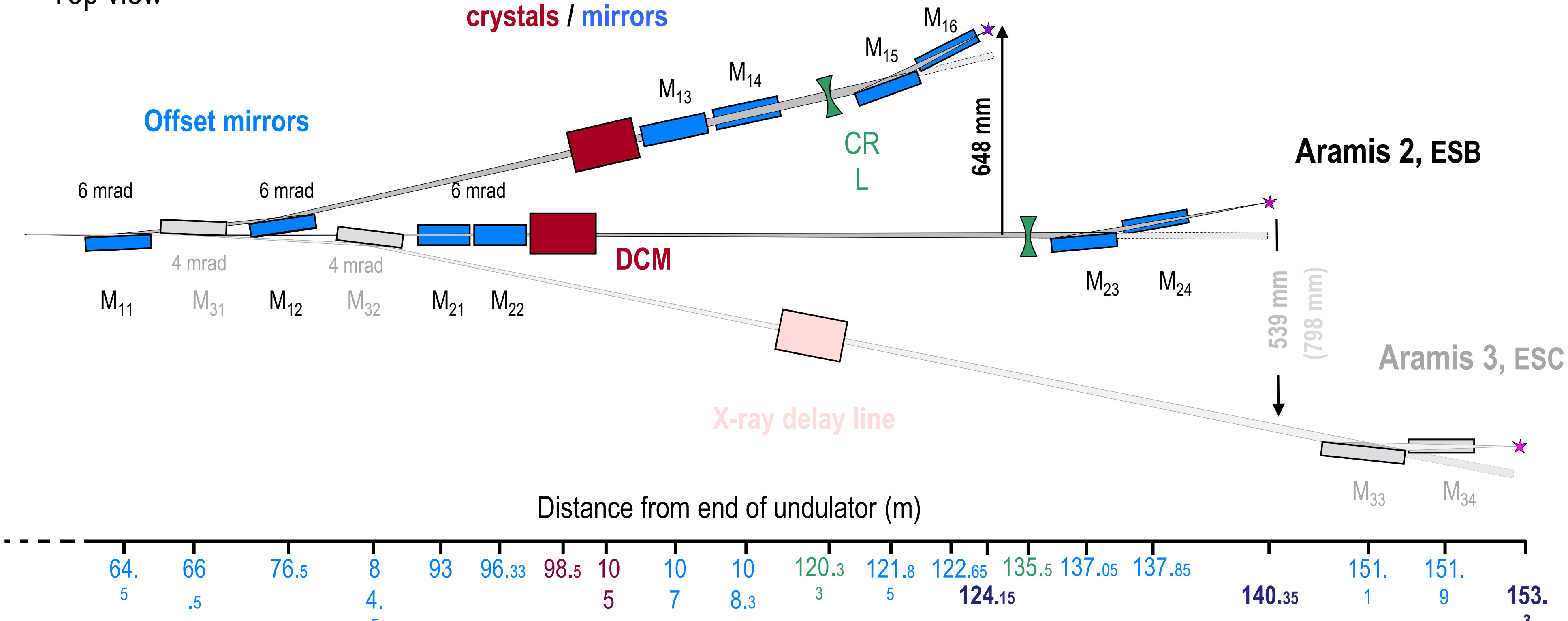
Photon energy		1770	3540	5320	7090	8860	10600	12400
Source size*	μm	44	31	29	28	27	26	25
~ divergence*	μrad	6.4	3.4	2.3	1.8	1.5	1.3	1.1
Pulse energy	mJ	0.20	0.15	0.16	0.18	0.18	0.18	0.16
Spectr. Bandw.*	%	0.17	0.09	0.09	0.07	0.06	0.06	0.06
Size at M_{11}*	mm	0.41	0.22	0.15	0.12	0.10	0.099	0.075
Fluence at M_{11}*	mJ/cm²	38	99	226	408	578	755	912
Footprint on M_{11}* (3 mrad)	**mm**	**97**	**52**	**35**	**28**	**23**	**21**	**18**
Fluence at M_{33}*	mJ/cm²	6.6	17	41	75	107	142	176
Footprint on M_{33}* (4 mrad)	**mm**	**173**	**92**	**62**	**49**	**41**	**35**	**30**

rms-values

M_1 at 64 m, M_3 at 153 m

S. Reiche, 27. Nov. 2012: https://intranet.psi.ch/Swiss_FEL/SwissFELSimAramis-Design

Optics should accept 5 rms



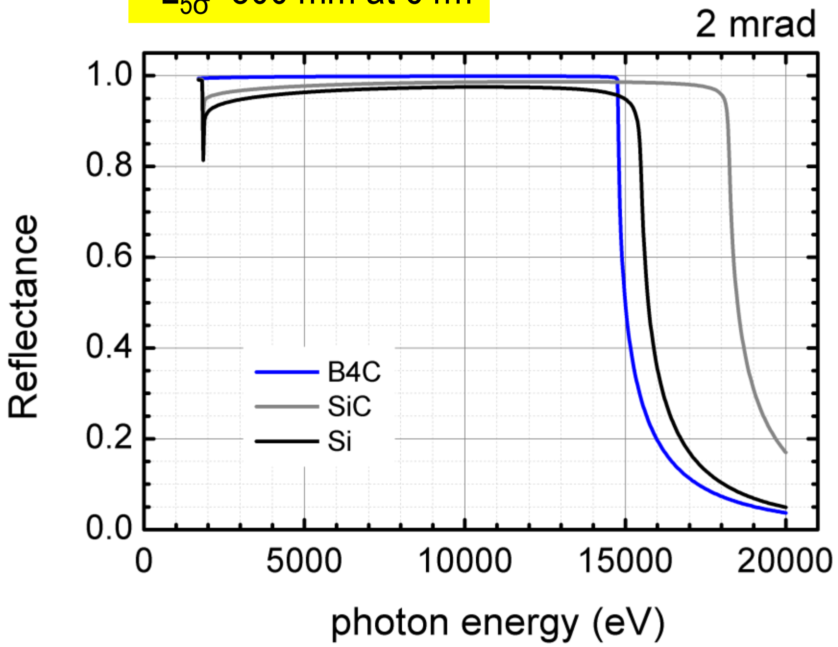
X-ray mirrors for SwissFEL Aramis



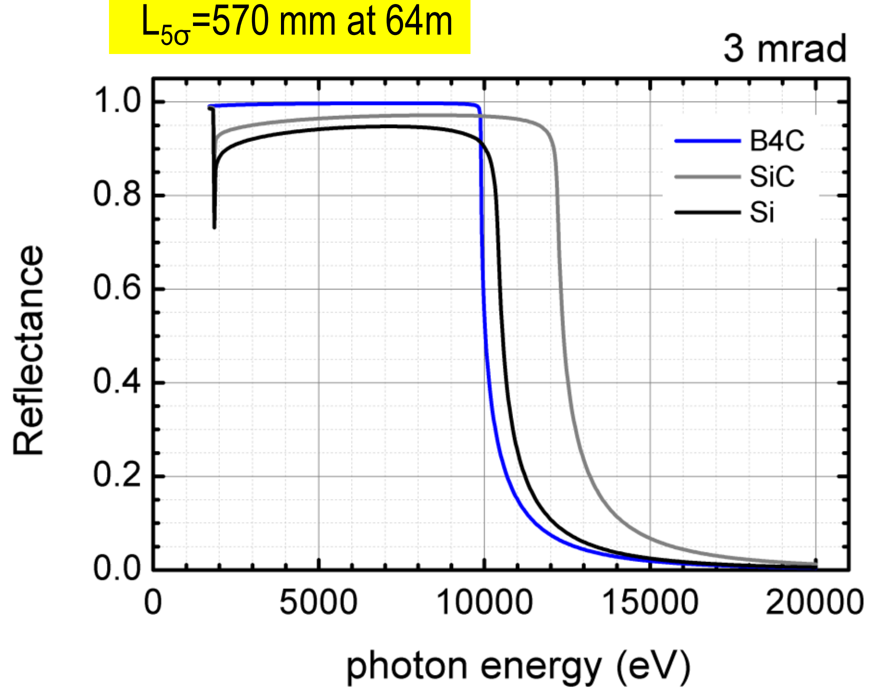
Small grazing incidence angle
+ high reflectance
- long mirrors

Mirror should accept 5σ of beam
(at least 4σ)

$L_{5\sigma}$=800 mm at 64m



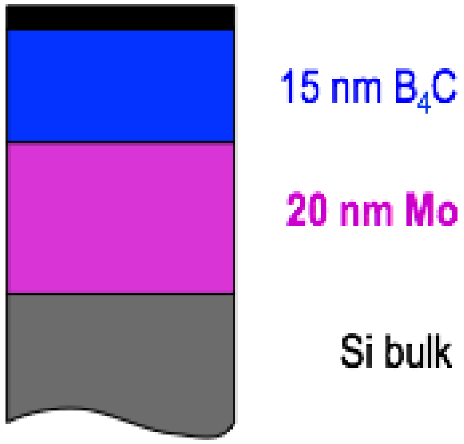
$L_{5\sigma}$=570 mm at 64m

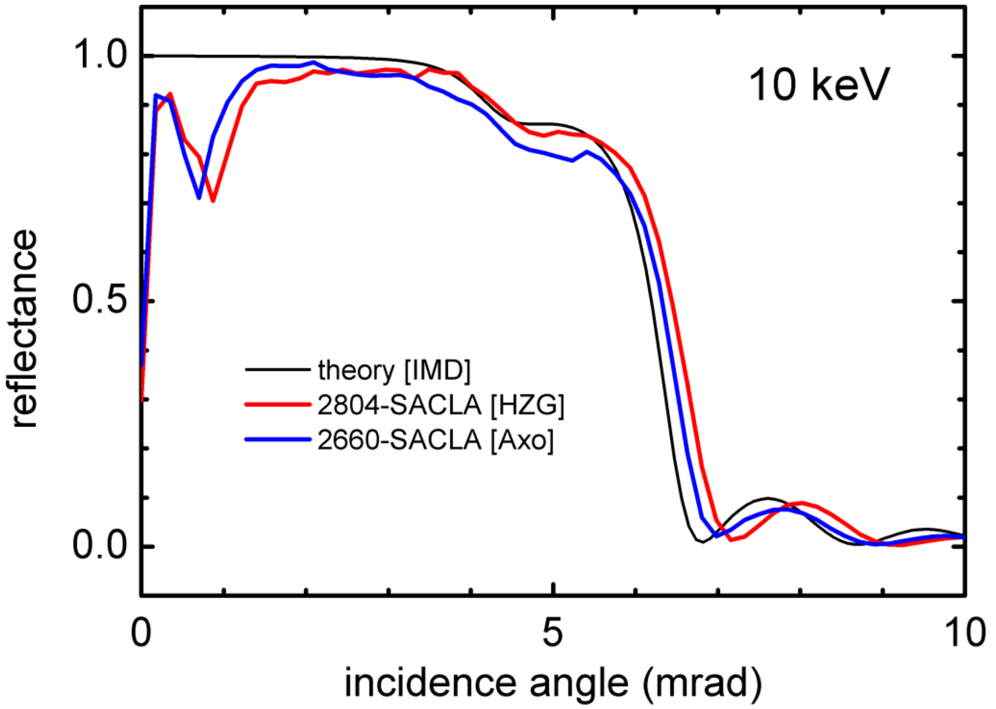


B_4C Looks OK at low energies, but angle is low and high energy cut-off is too

What about a dual-layer coating ?

B_4C/Mo looks better at higher energies and still good at lower energies

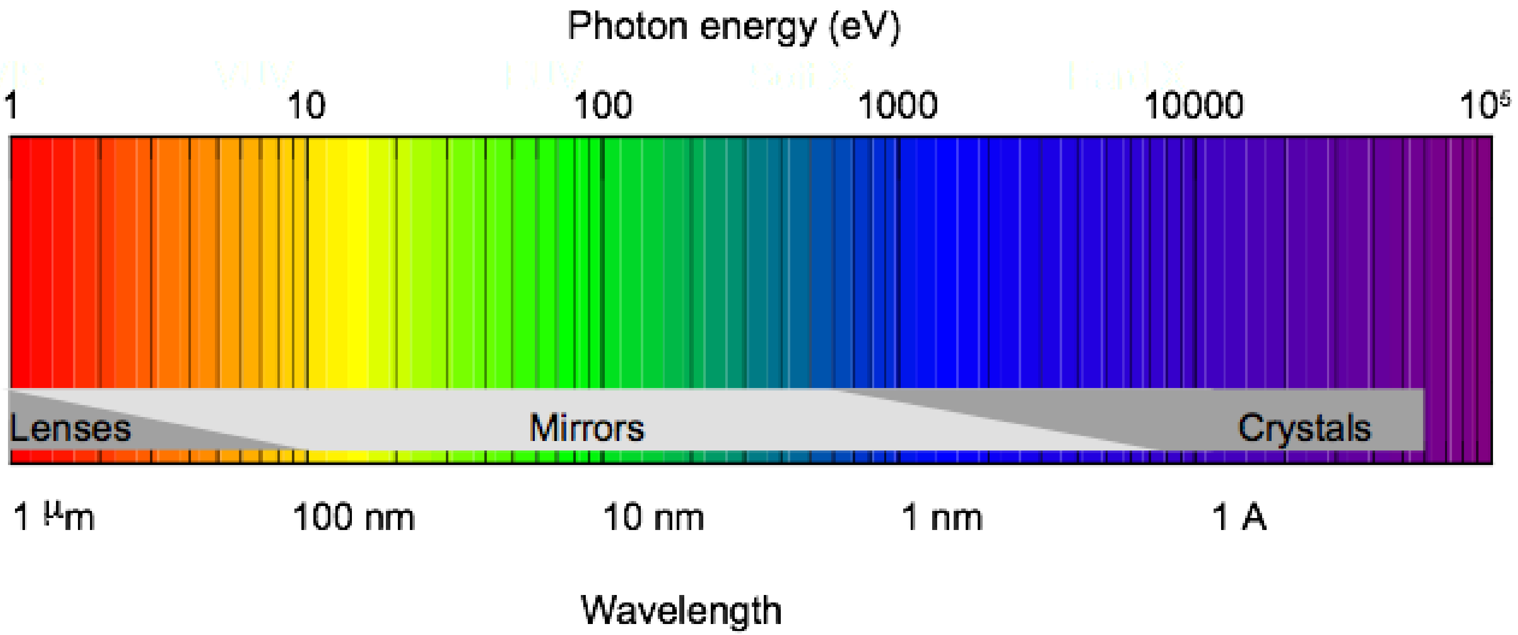




Courtesy of Rolf Follath

X-ray monochromators



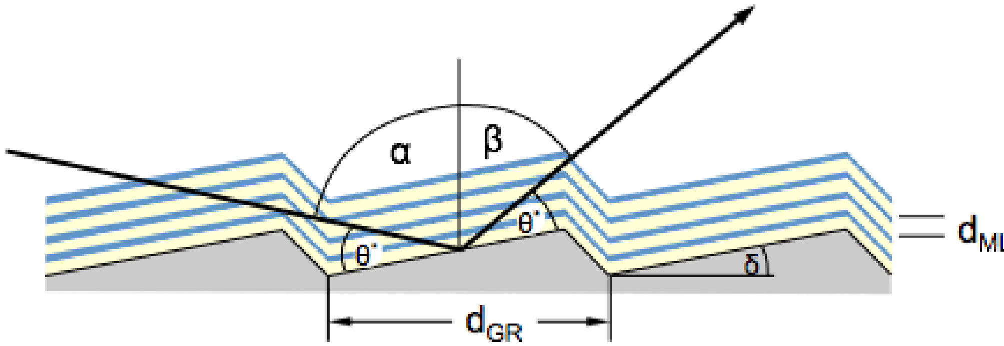


Normal incidence monos

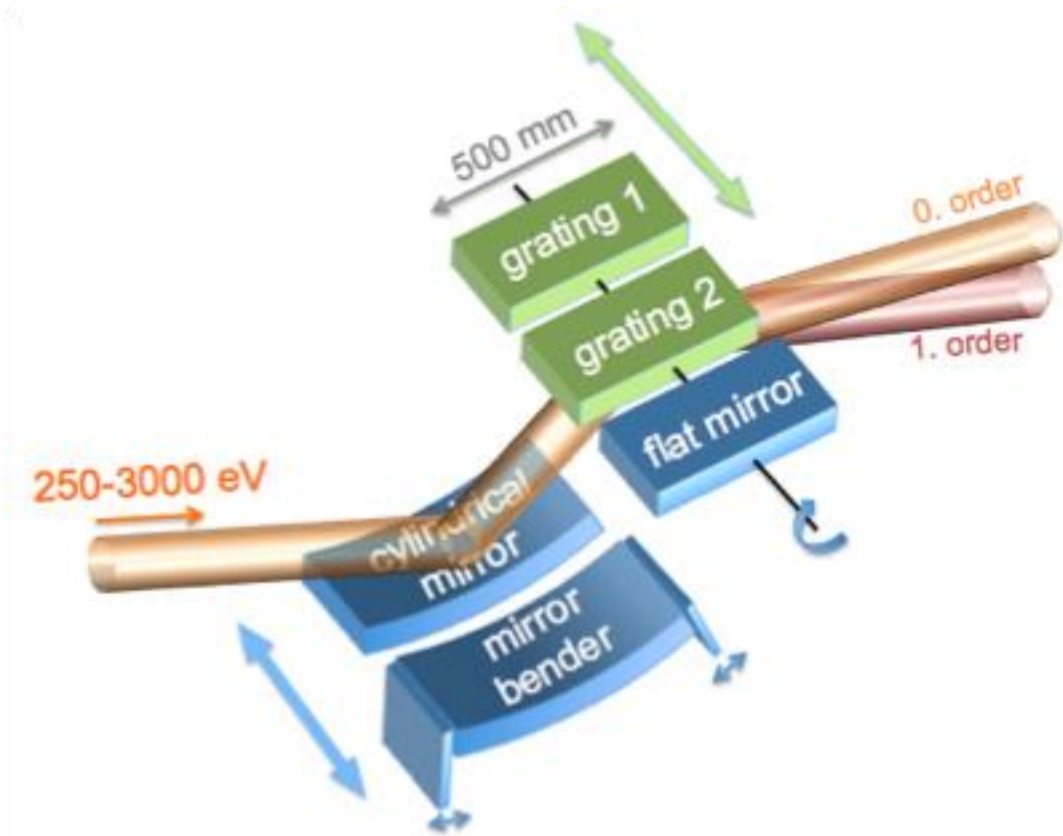
Grazing Incidence monos

Crystal monochromators

Soft X-ray grating monochromators

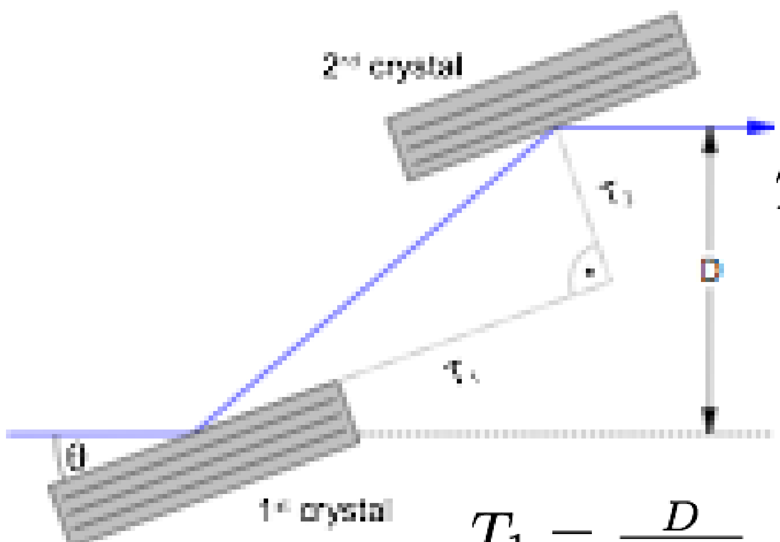


$$n\lambda = d_{gr}(\sin\alpha + \sin\beta)$$



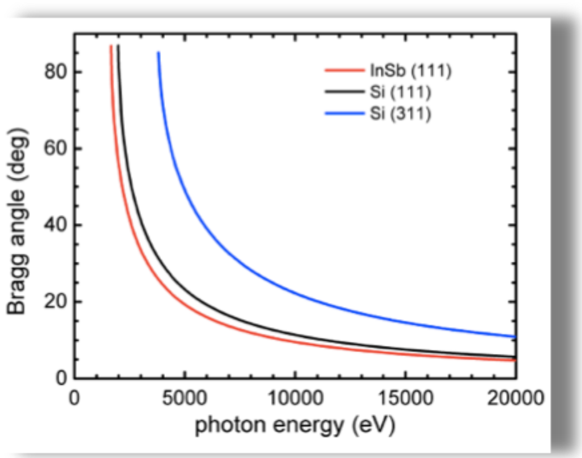
Courtesy of Rolf Follath

Hard X-ray crystal monochromators



$$T_2 = \frac{D}{2\cos\theta}$$

$$T_1 = \frac{D}{2\sin\theta}$$

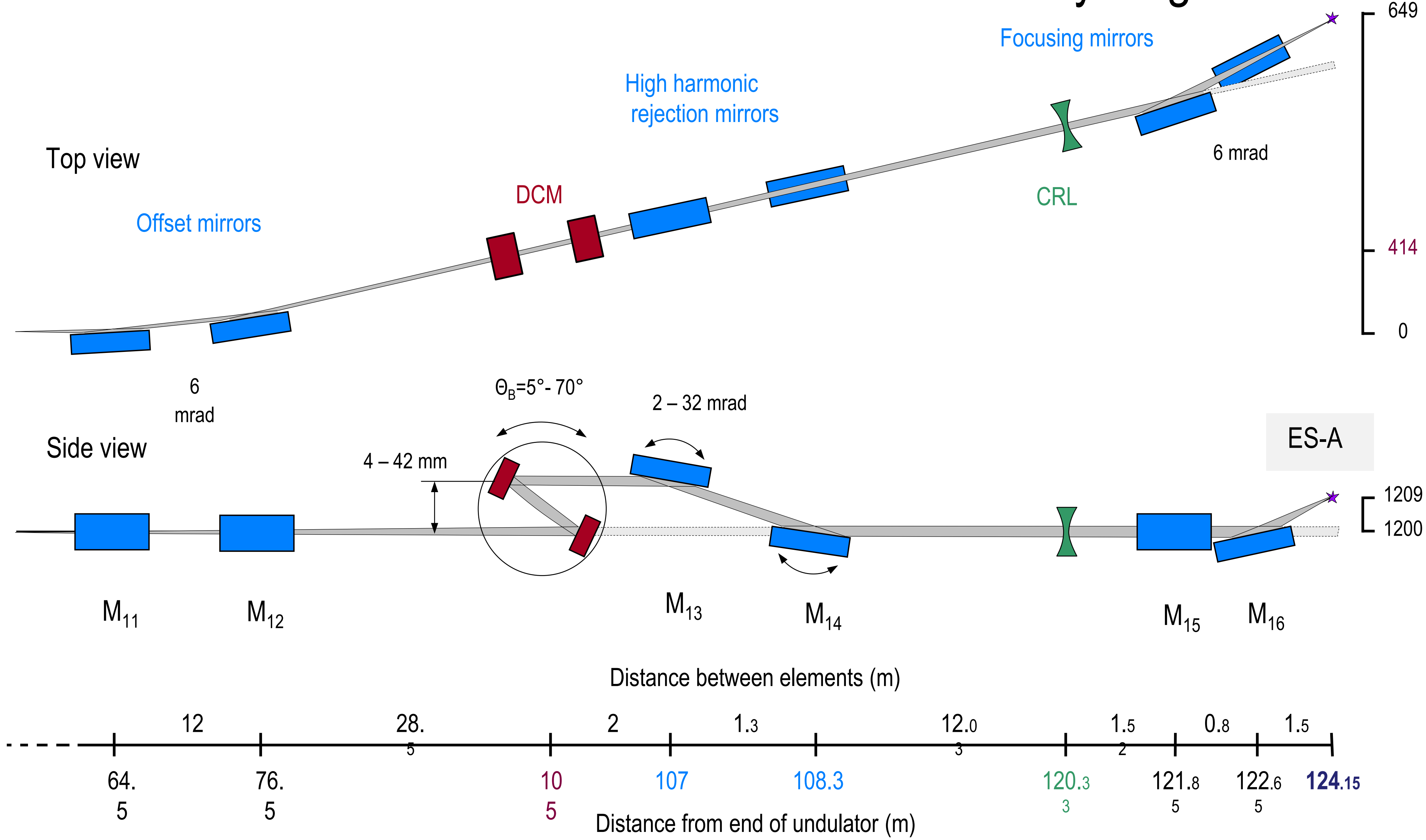


$$n\lambda = 2d_{hkl}\sin\theta$$

Switching from pink beam to monochromatic

We want to be able to switch easily between the two modes

BUT we don't want to have to move everything when we switch



Solution: Two vertical offset mirrors to correct for the monochromator*

*Added bonus: harmonic rejection !

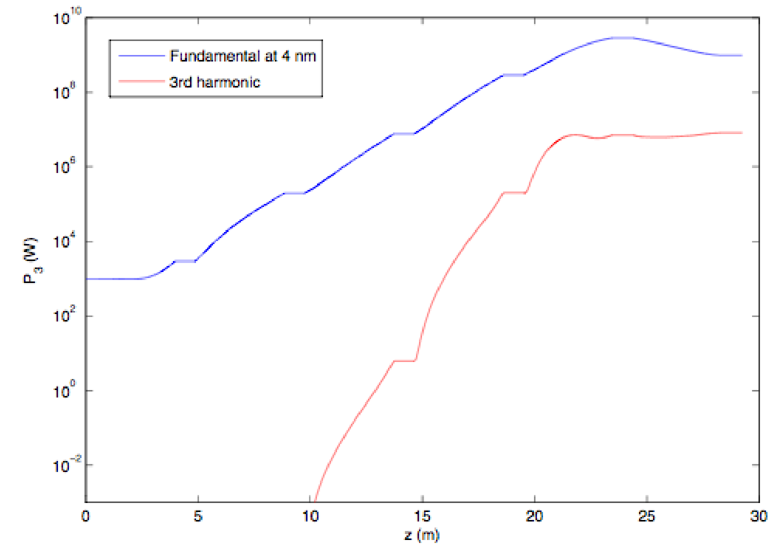
Harmonic rejection

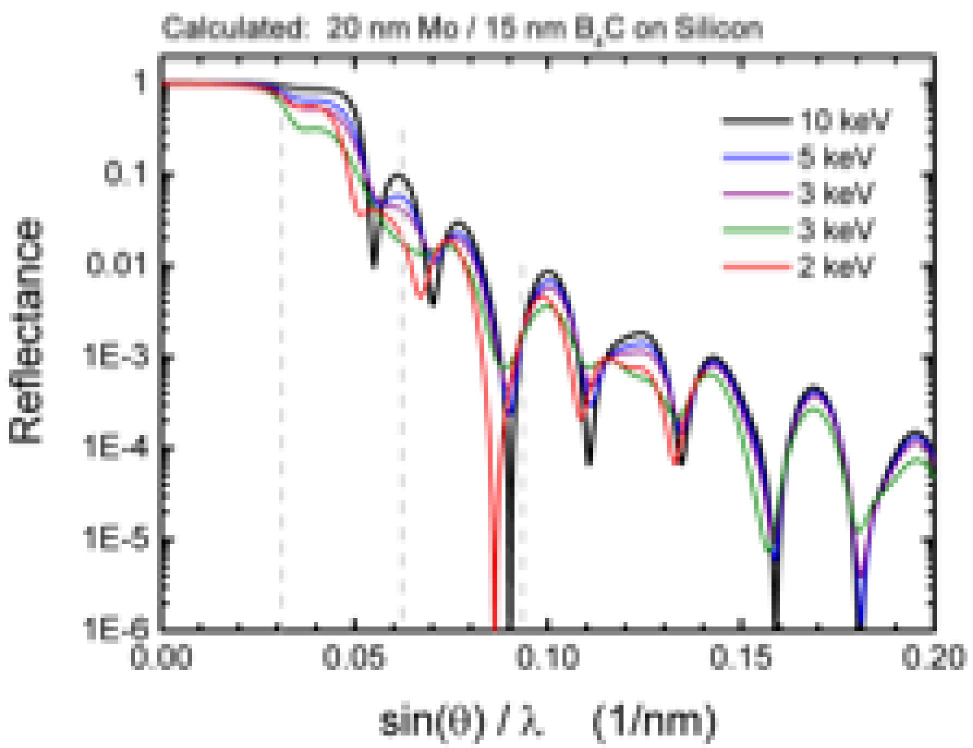


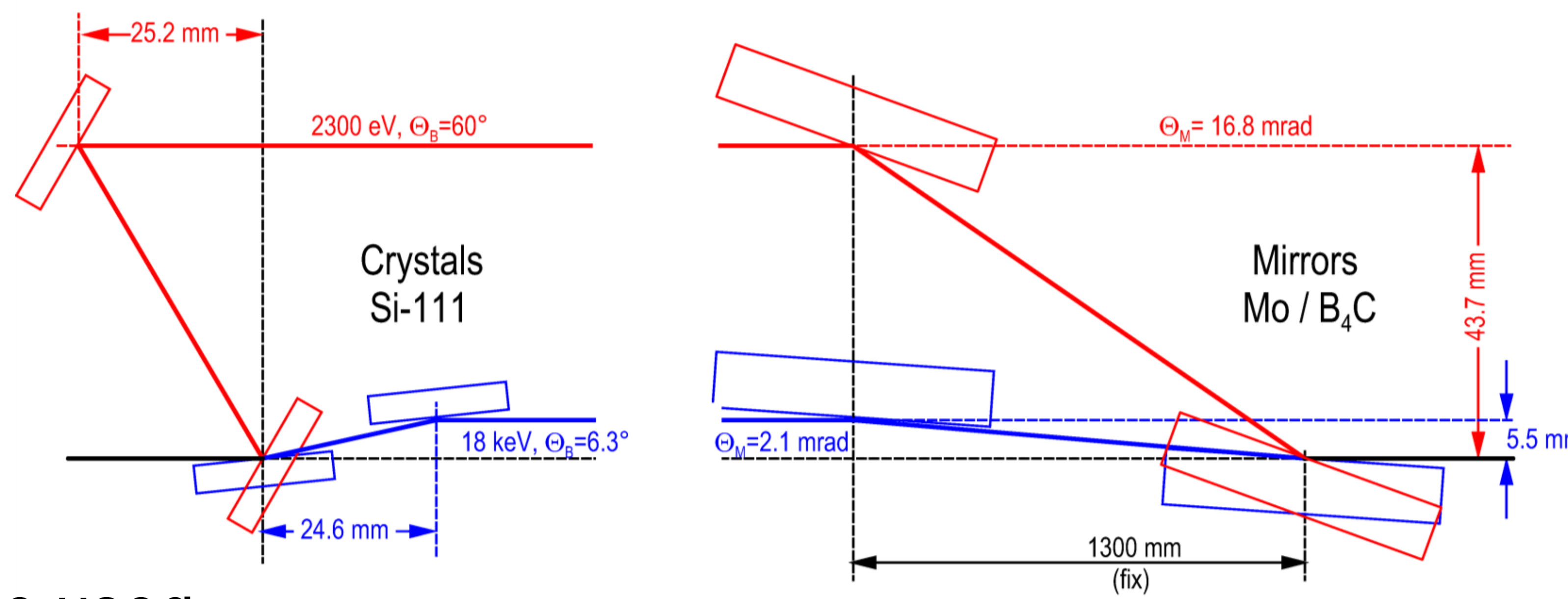
The harmonic contribution is about 1% for each subsequent harmonic

So if we have 10^{12} photons per pulse at 5 keV we also have about 10^{10} photons at 15 keV

That's not so small







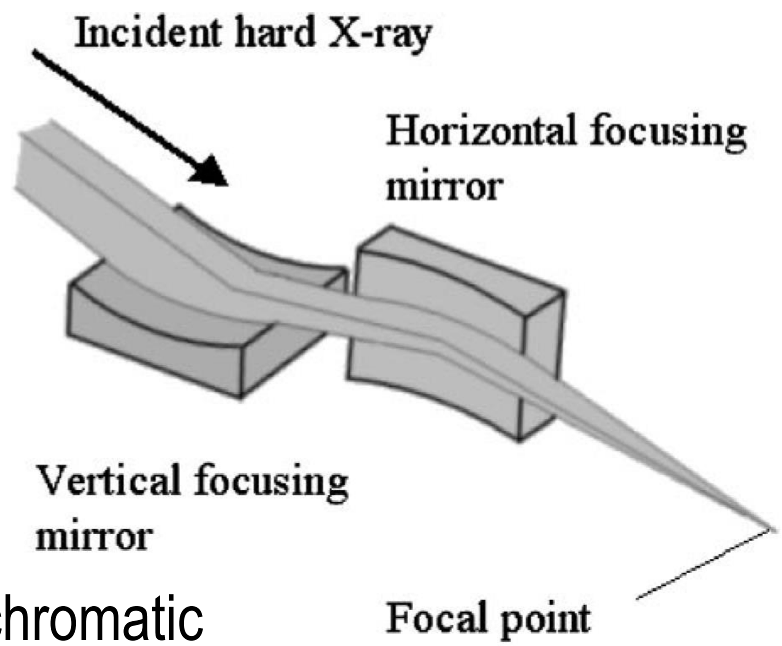
But the second set of mirrors can be used to cut-off further orders of magnitude of the higher-order harmonics

Courtesy of Rolf Follath

Experimental X-ray focus

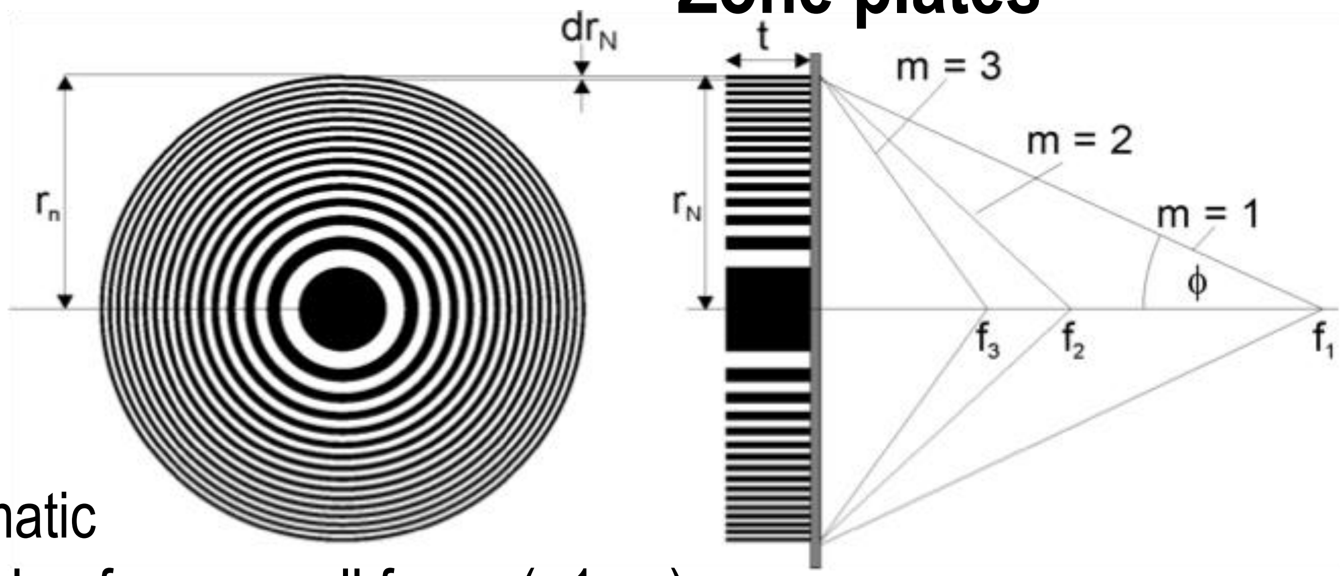
There are lots of ways to focus X-rays onto your sample

KB mirrors



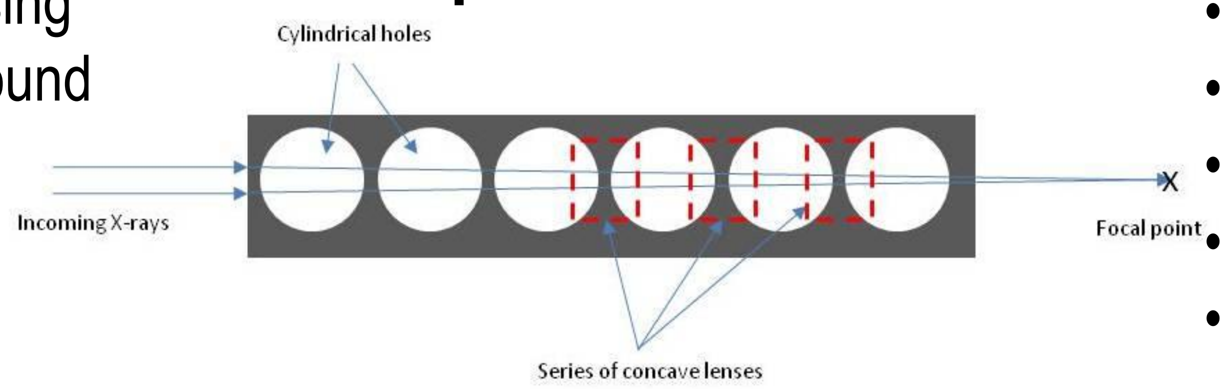
- Achromatic
- Small focus (~1μm) with reasonable working distance (~1 m)
- Asymmetric focus possible
- Capable of de-focussing
- Moves X-ray spot around

Zone plates



- Chromatic
- Capable of very small focus (<1μm)
- Short working distance
- Easy alignment
- Inflexible (specific focus at specific X-ray energy)

Compound refractive lenses



- Chromatic
- Beam always on-axis
- Very long working distance (meters)
- Easy alignment
- Focus profile depends on quality of lens

You need to pick what's right for the experiment

What are the priorities ?

~~What do we want to measure ?~~

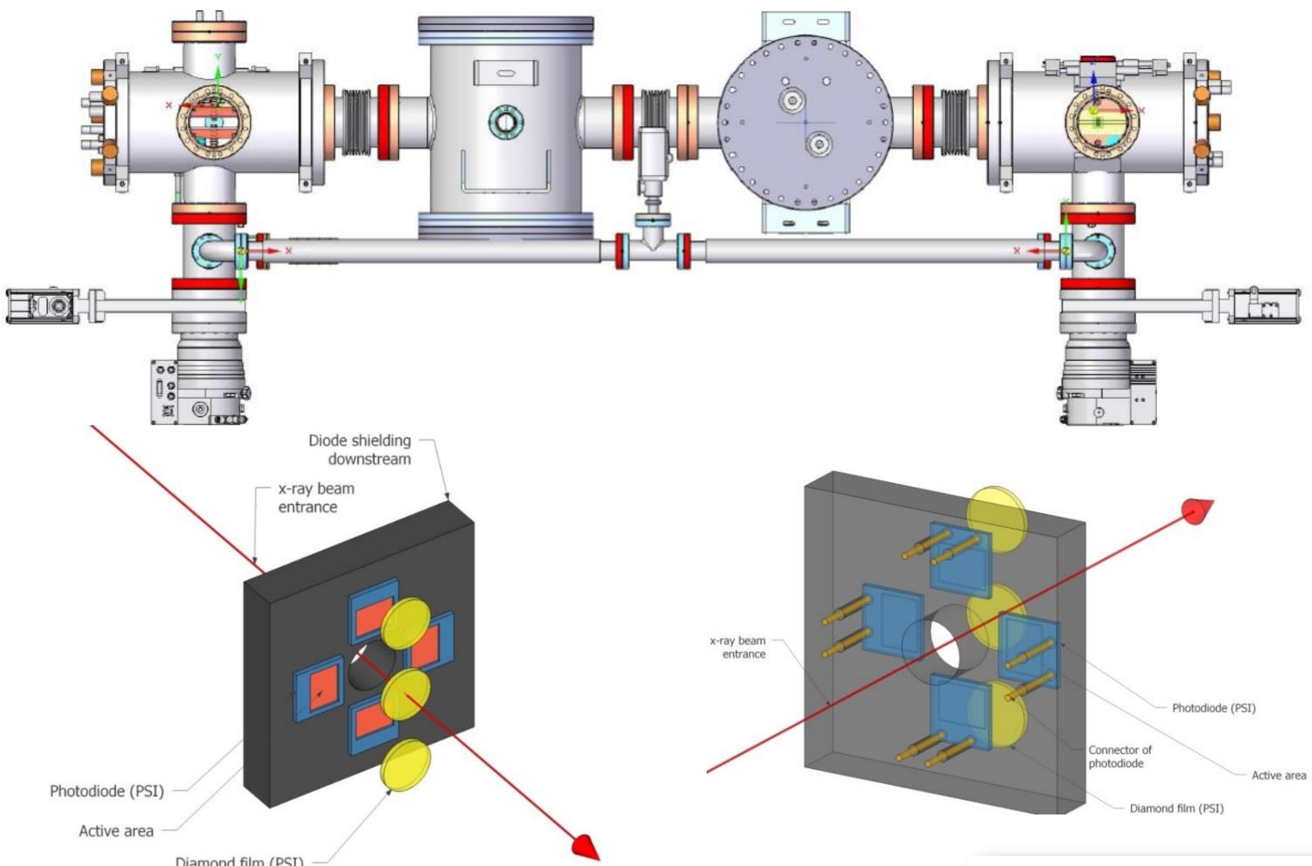
How do I know anything about the X-rays ?

~~How do I get the X-rays onto my experiment ?~~

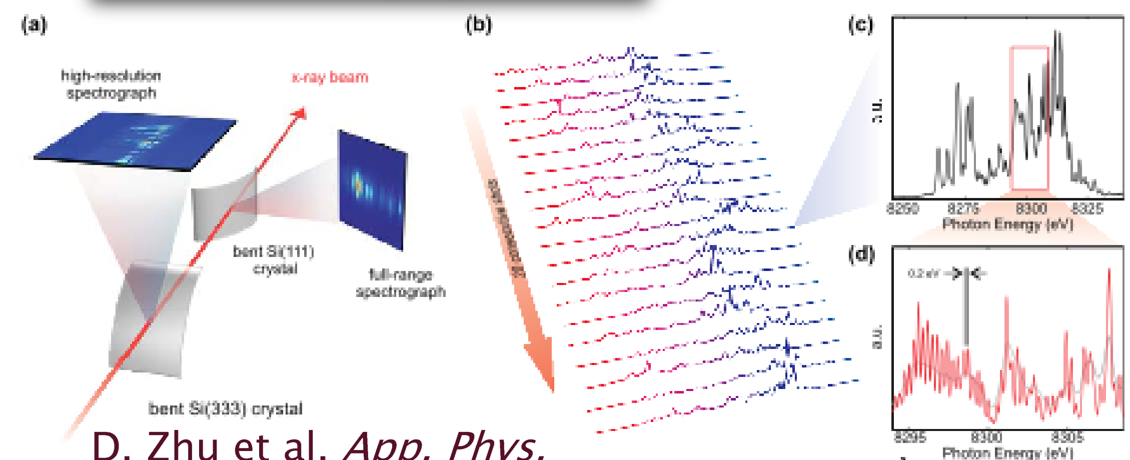
~~What kind of X-rays and laser do I need ?~~

X-ray Photon Diagnostics

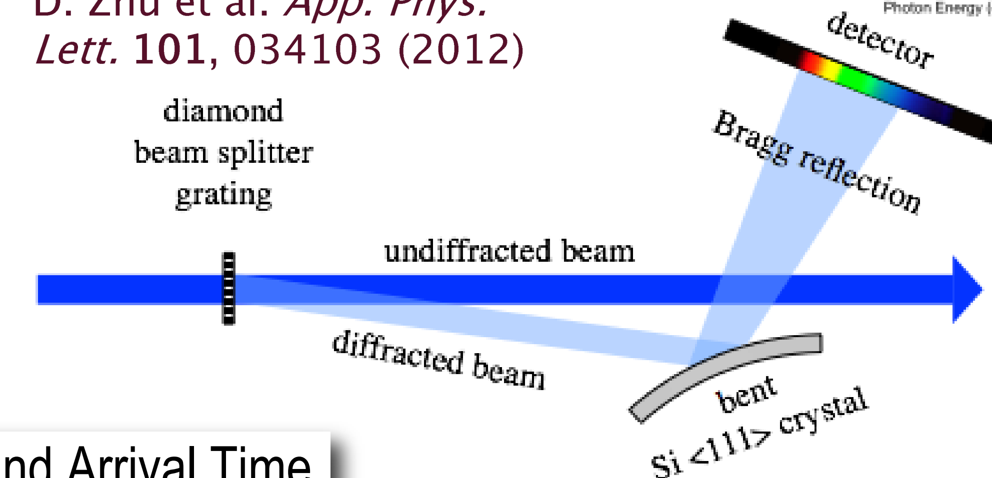
Position and Intensity Monitors



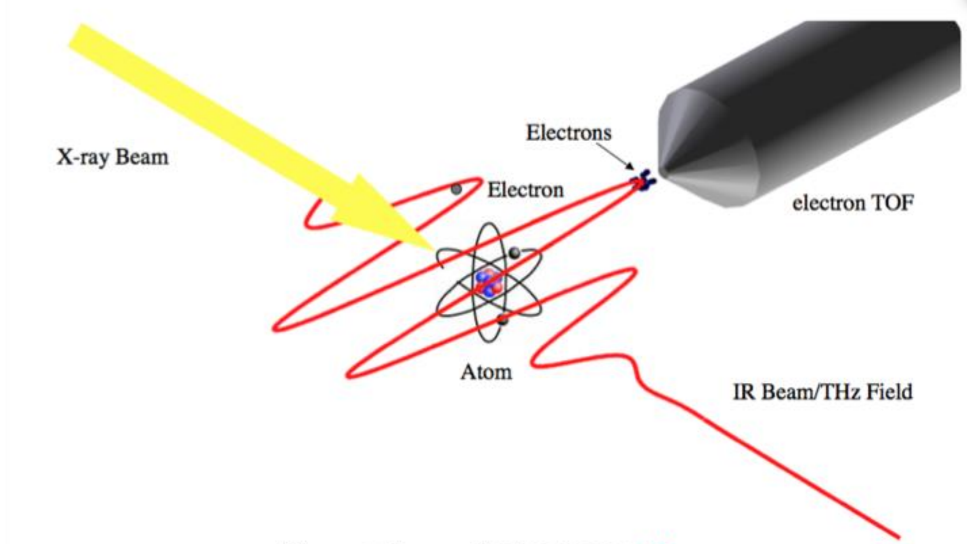
Incident X-ray Spectrum



D. Zhu et al. *App. Phys. Lett.* **101**, 034103 (2012)

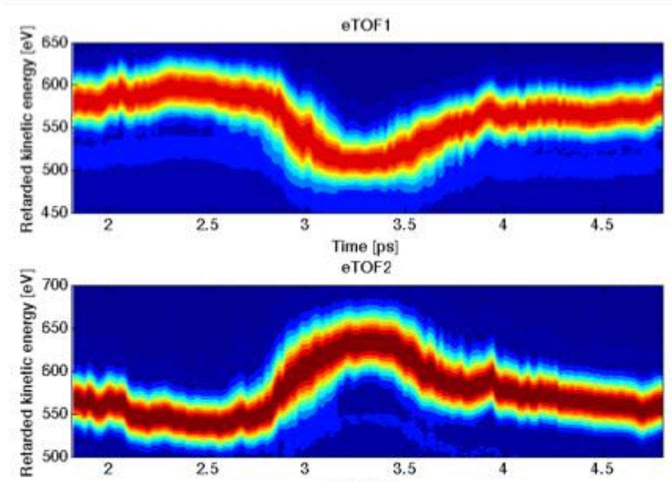


- Use a THz streak camera concept to measure the arrival time and length of the photon pulse.

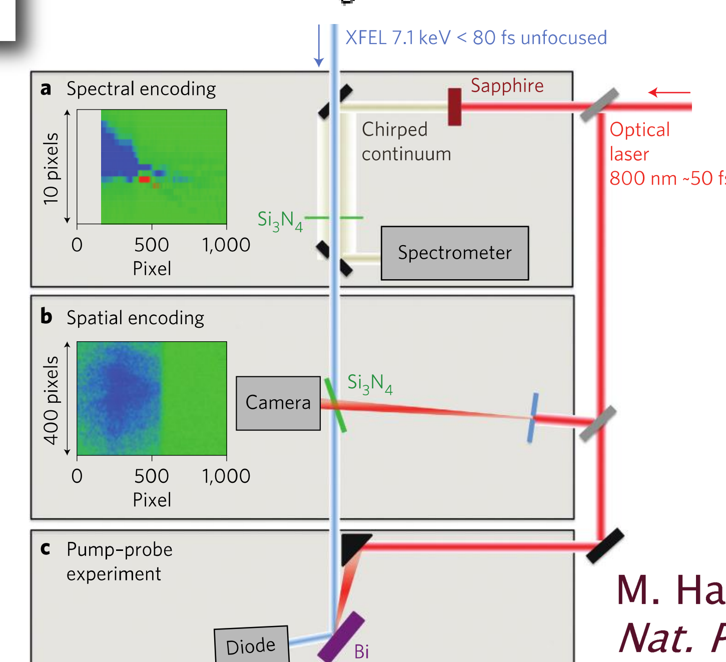


$KE_{electron} = E_{photon} - \text{Binding Energy} \pm W$

X-ray Pulse Duration and Arrival Time



Streak scan for the Xe $2p_{3/2}$ electrons at a photon energy of 10 keV



M. Harmand et al. *Nat. Phot.* 7, 215 (2013)

Courtesy of Luc Patthey and Pavle Juranic

PAUL SCHERRER INSTITUT

What are the priorities ?

~~What do we want to measure ?~~

~~How do I know anything about the X-rays ?~~

~~How do I get the X-rays onto my experiment ?~~

~~What kind of X-rays and laser do I need ?~~

Prioritisation

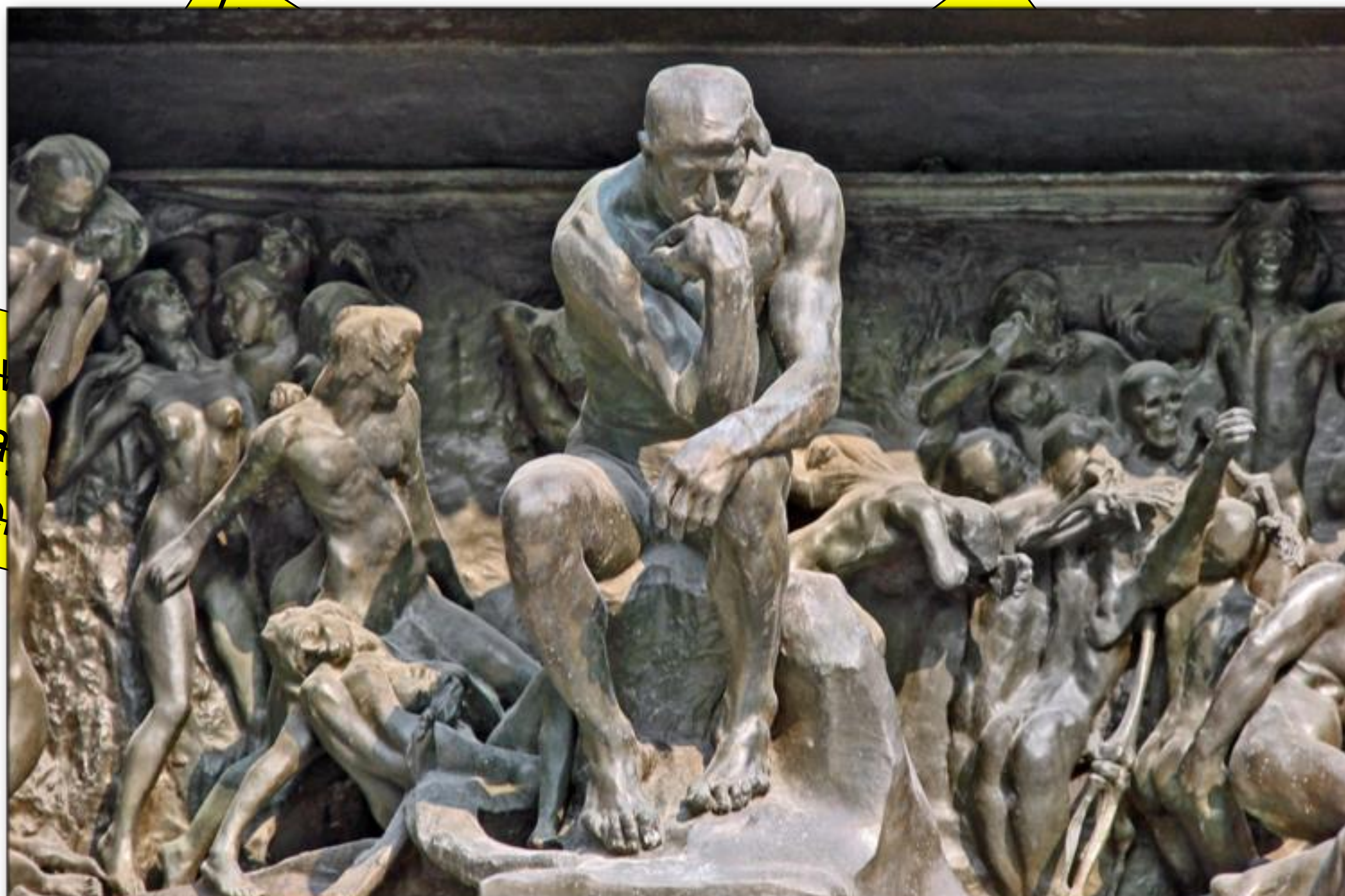
This is a multi-dimensional problem and needs to be established through discussion with potential uses, external review, and realistic projections

Experimental Station A at SwissFEL

October 2008:	XFEL Workshop at EPFL in Lausanne to stimulate ideas
January 2010:	Science case document published (available online)
September 2011:	Open meeting in Bern for all potential interested users in Switzerland and elsewhere to provide details on current status of project
March 2012:	ESA Instrumentation Workshop to figure out what should be implemented at ESA
mid-2012:	ESA budget numbers established
June 2013:	Completion of ESA Conceptual Design Report and presentation of concept to Photon Science Advisory Committee
January 2014:	ESA Advisory Committee Meeting to obtain specific and technical feedback on plans prior to preparation of Technical Design Report

At this point we know what we should prioritize, who is interested in what techniques, how we're going to do it, and the general time-frame

So call all your friends, let's go do an experiment !

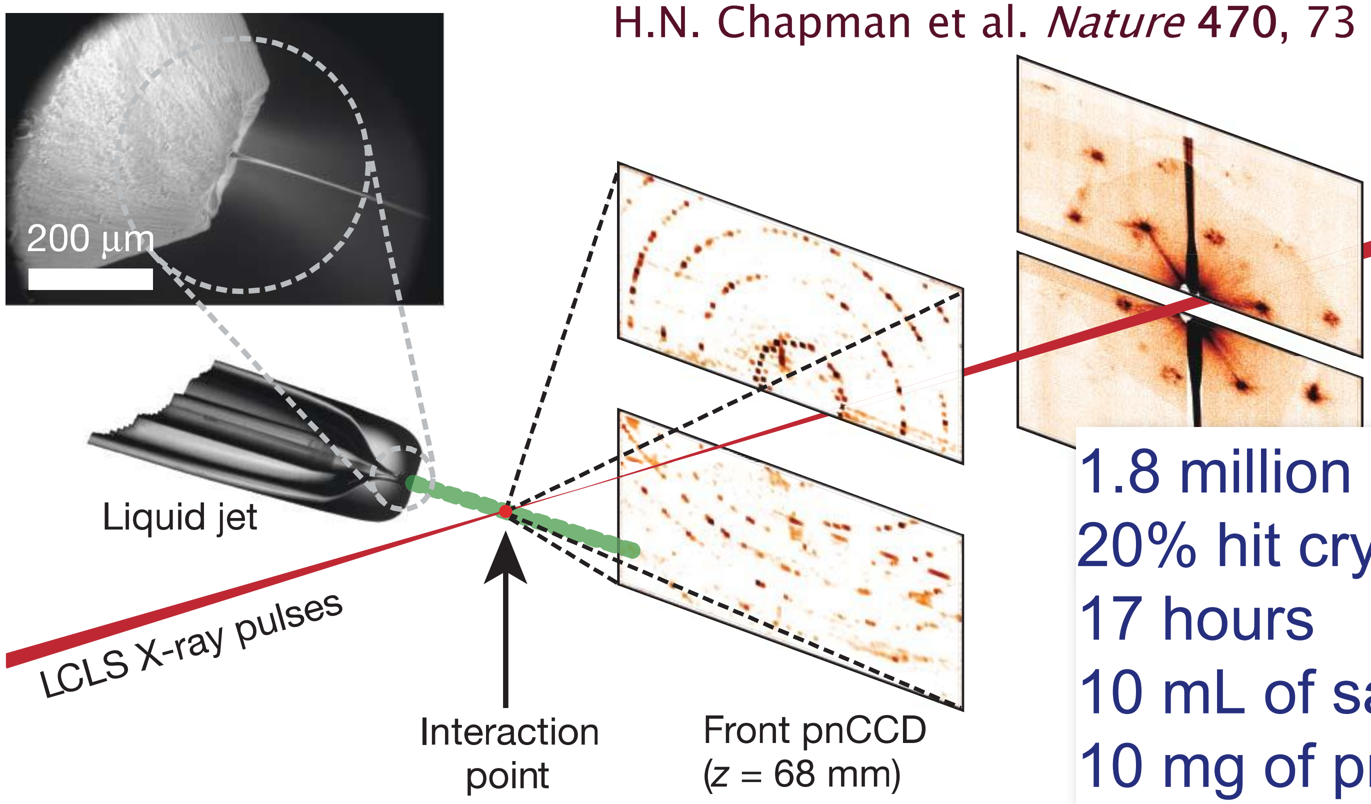


Experiment #1

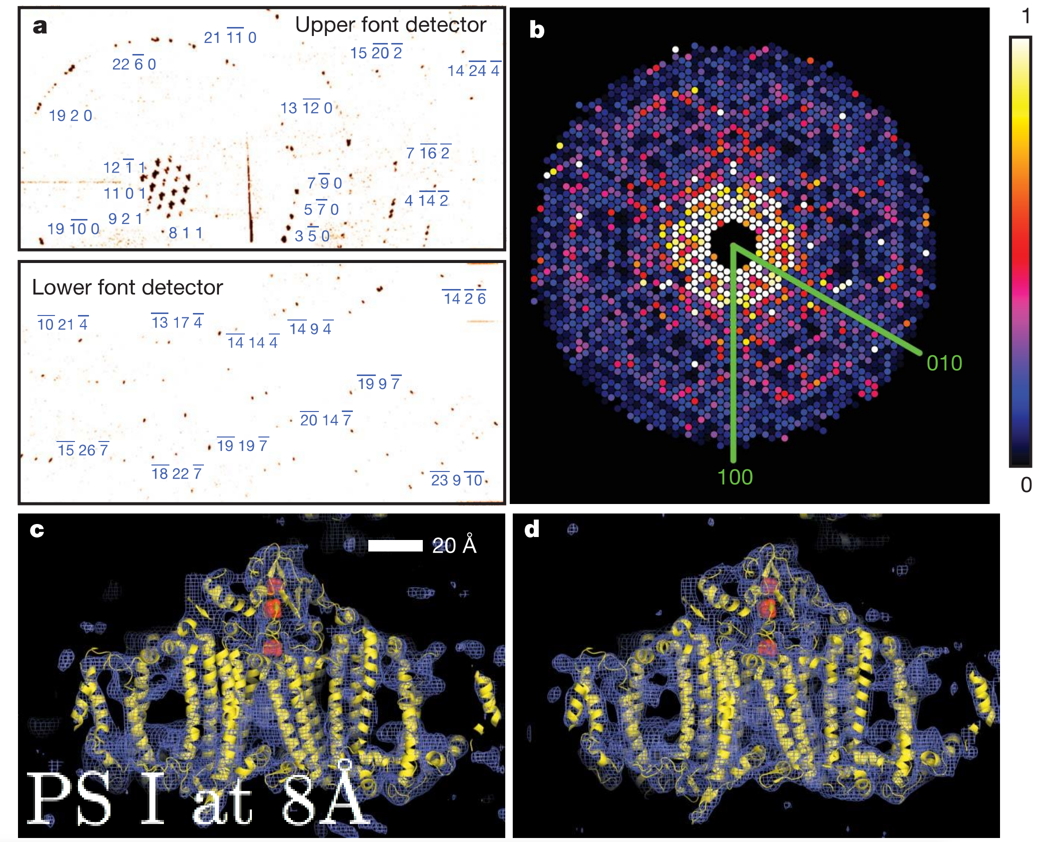
Serial Femtosecond Crystallogrpahy

X-ray nanocrystallography: Retrieving crystalline structure

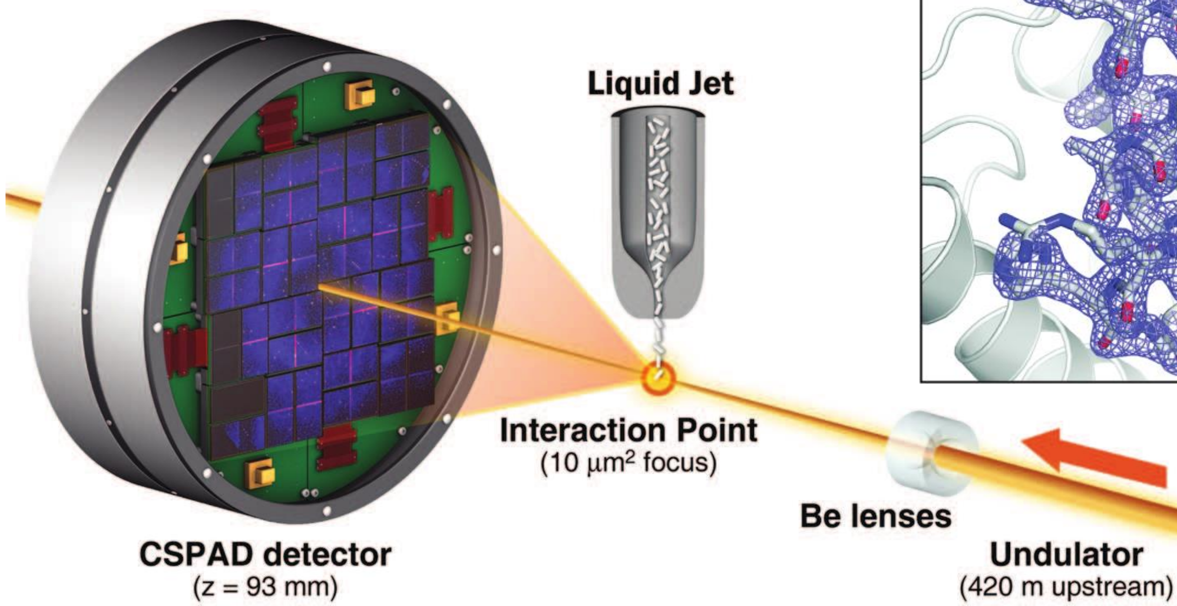
H.N. Chapman et al. *Nature* **470**, 73 (2011)

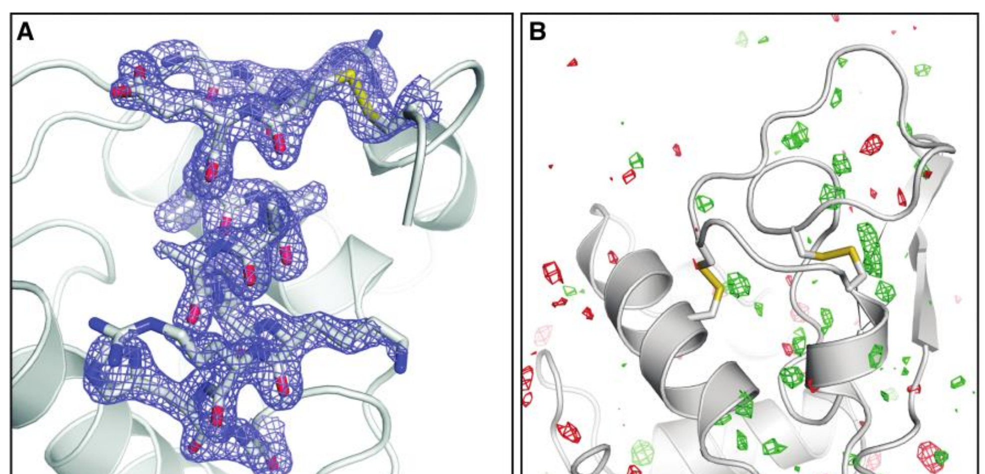


1.8 million shots
20% hit crystals
17 hours
10 mL of sample
10 mg of protein



S. Boutet et al. *Science* **337**, 362 (2012)



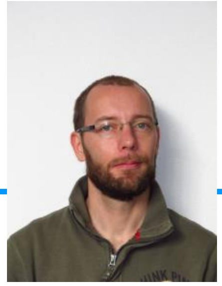


lysozyme at 1.9Å

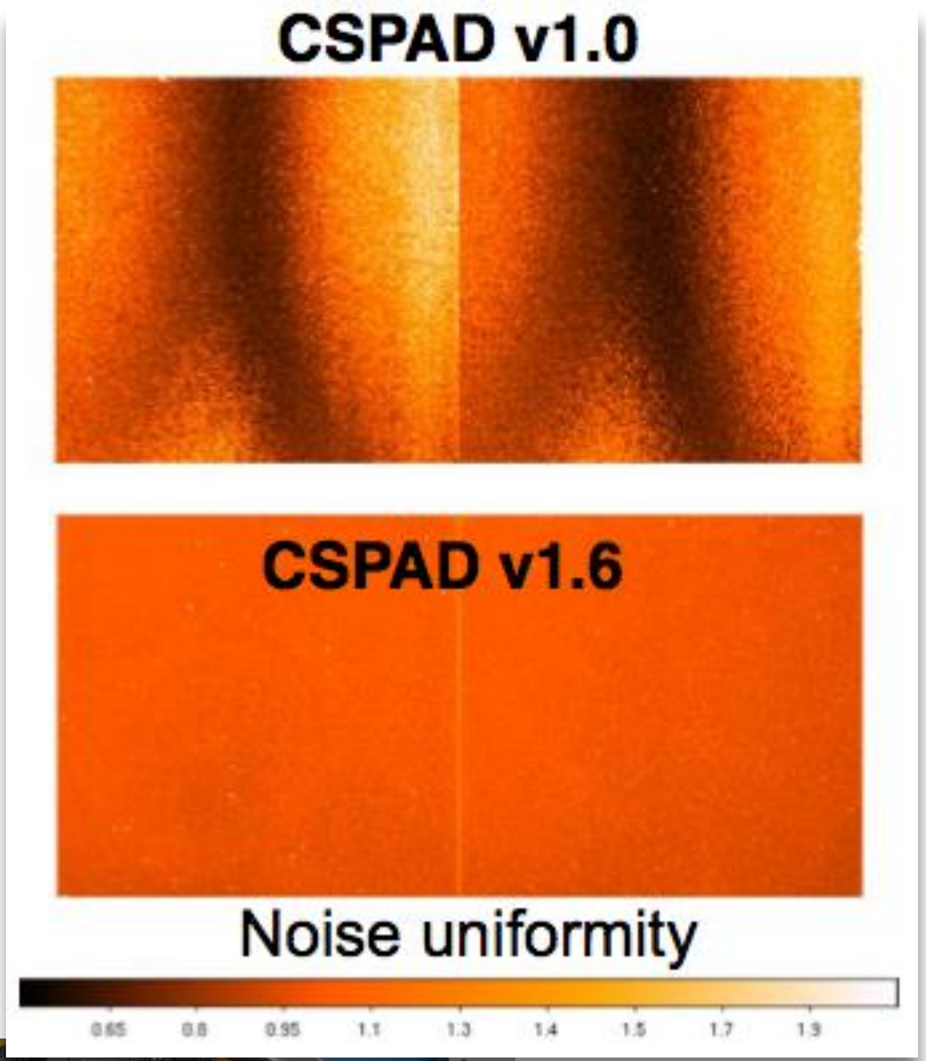
1.5 million shots
4% hit crystals
0.007% were useable
3-4 hours

X-ray diffraction gives information on the positions of all the atoms in the crystal

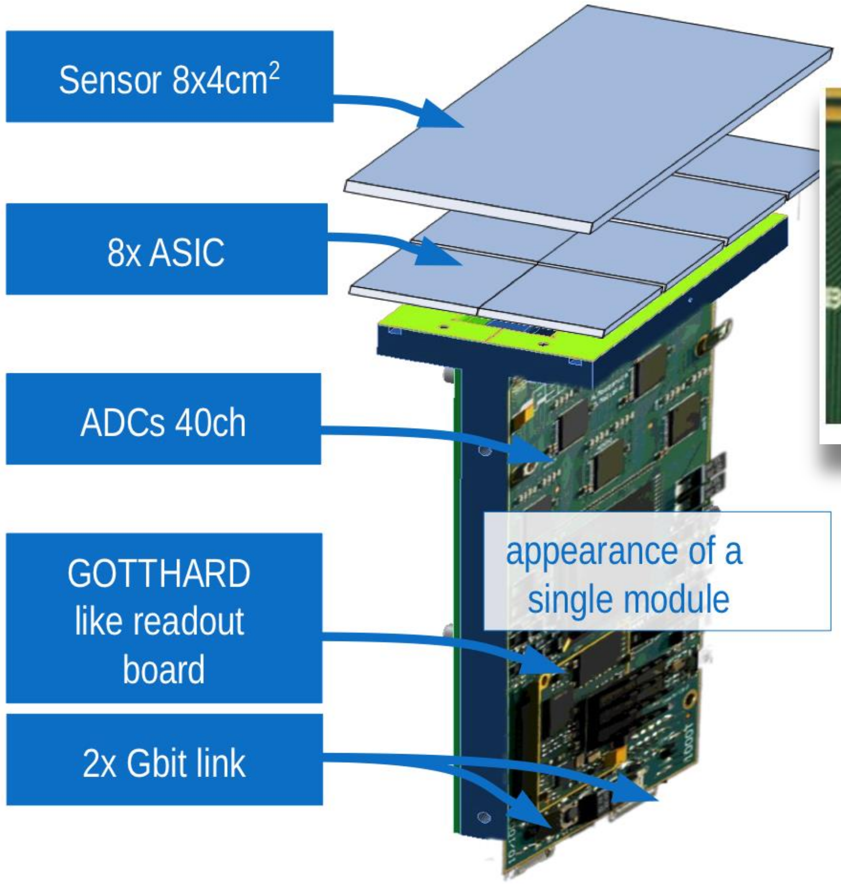
J.C.H. Spence et al. *Rep. Prog. Phys.* **75**, 102601 (2012)

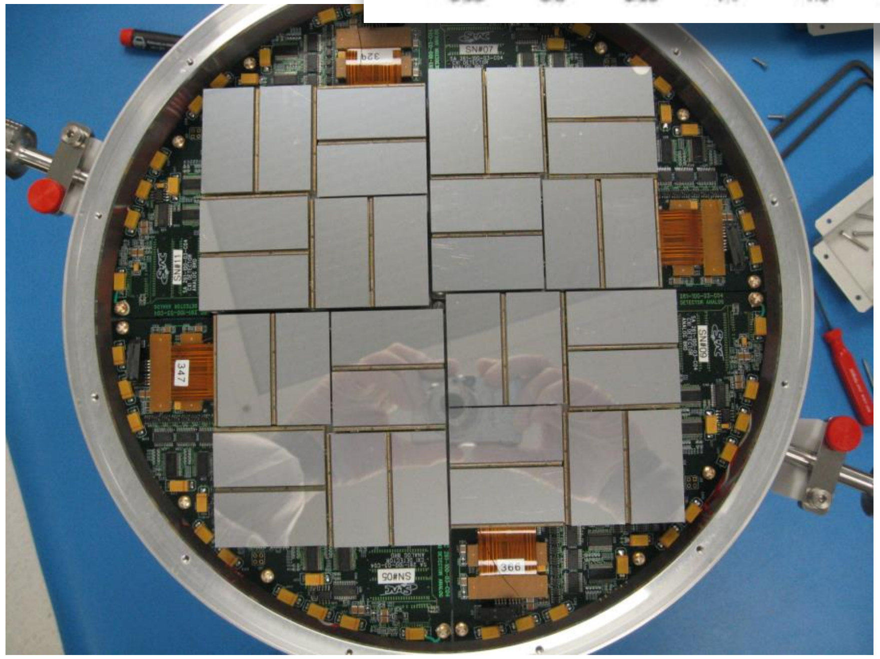


CS-PAD

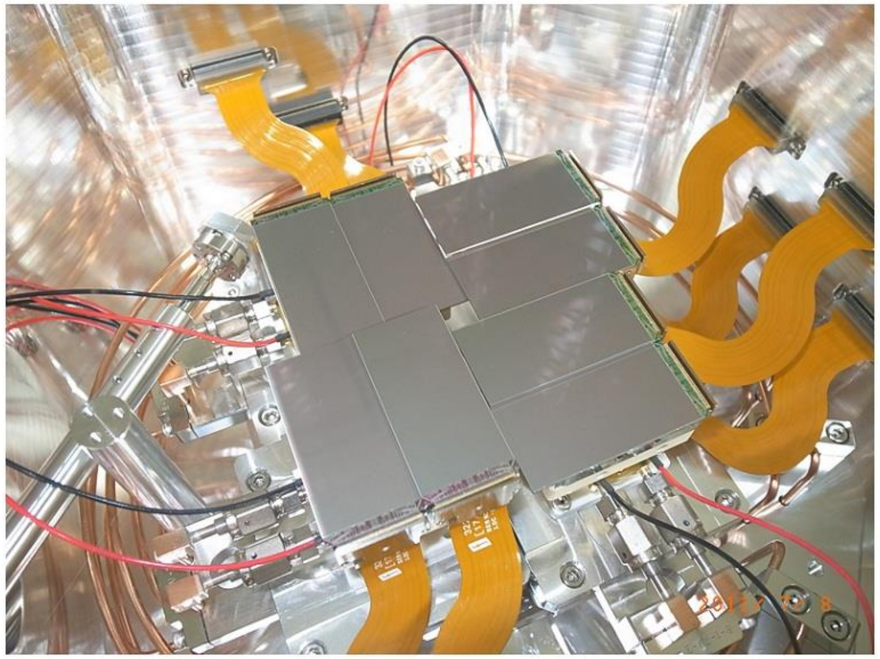


Jungfrau

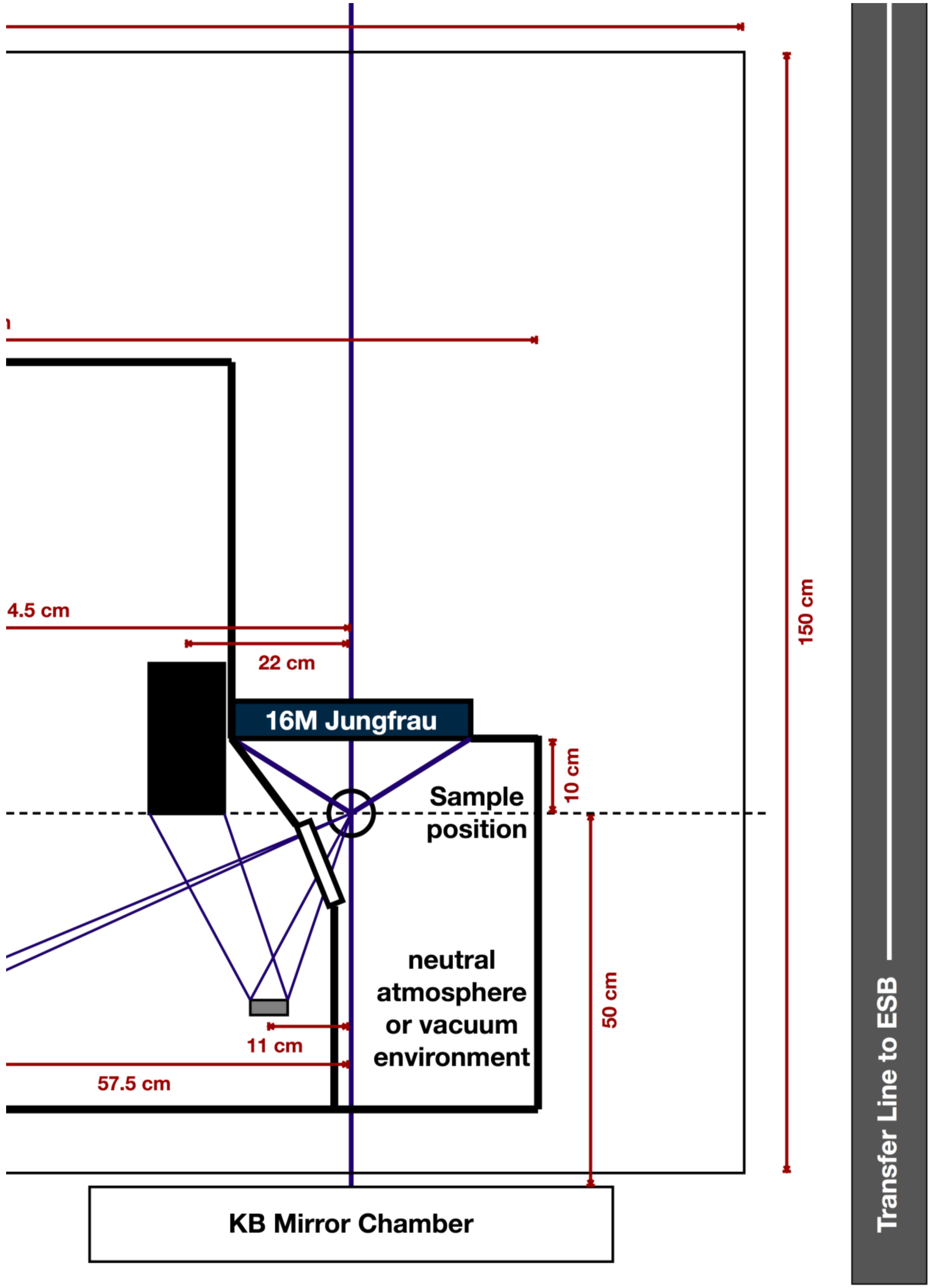


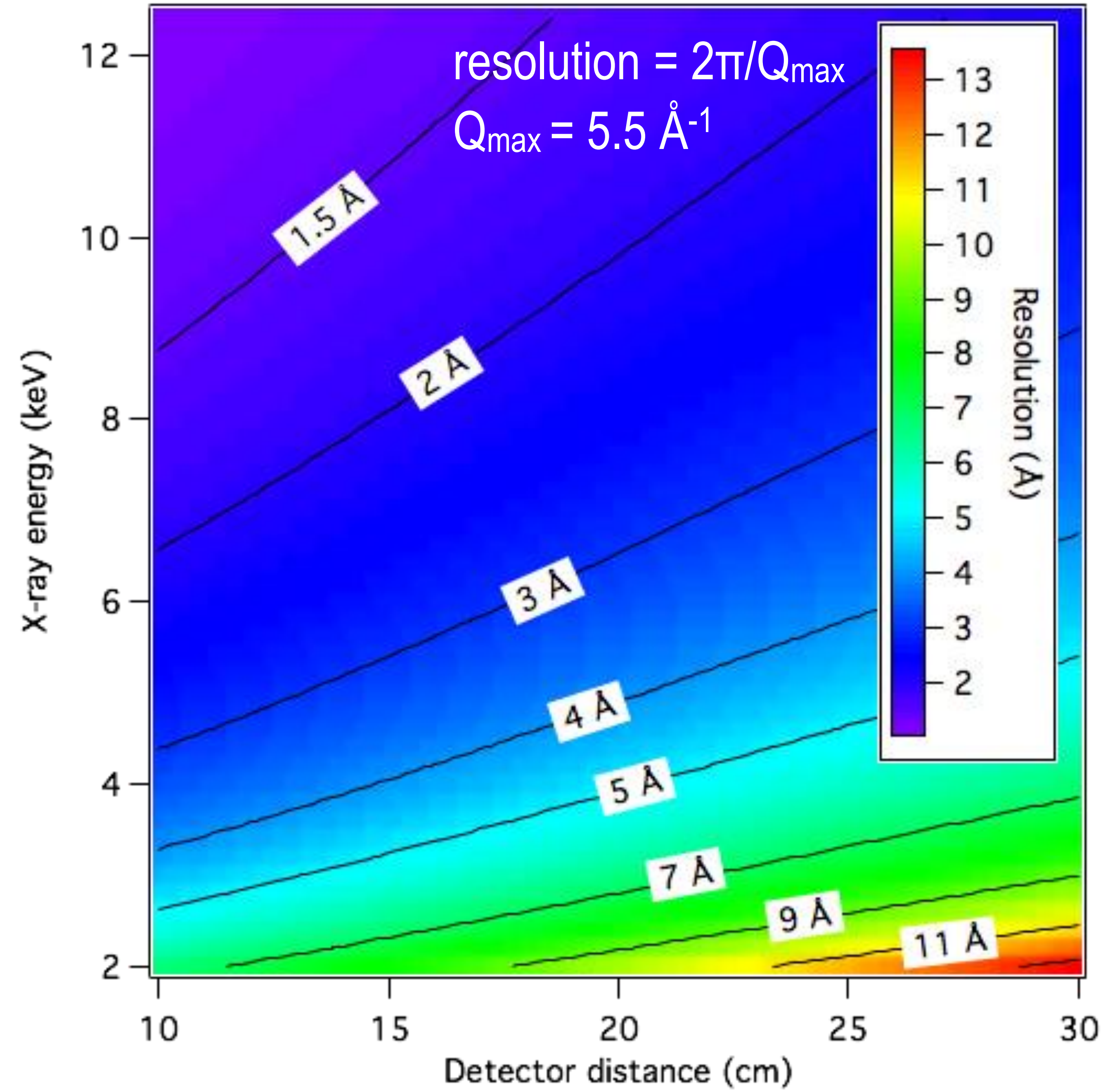


MPCCD



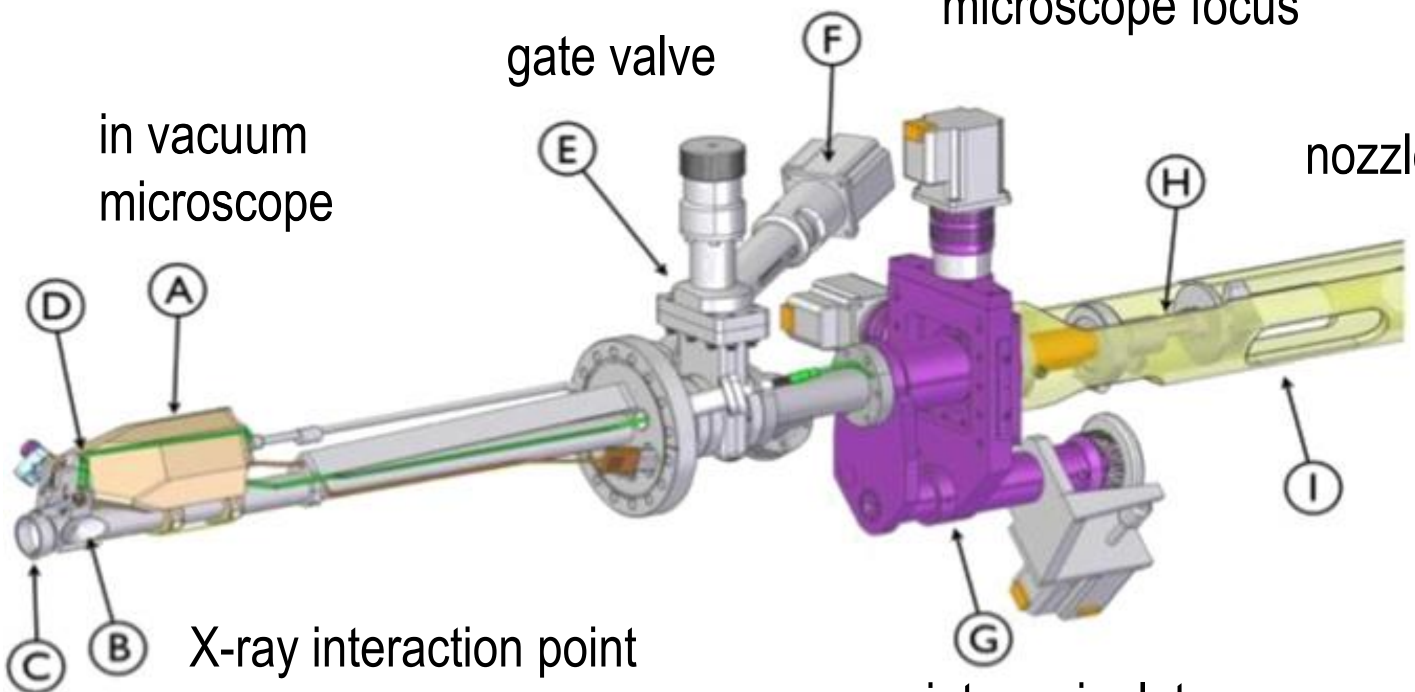
SFX measurements



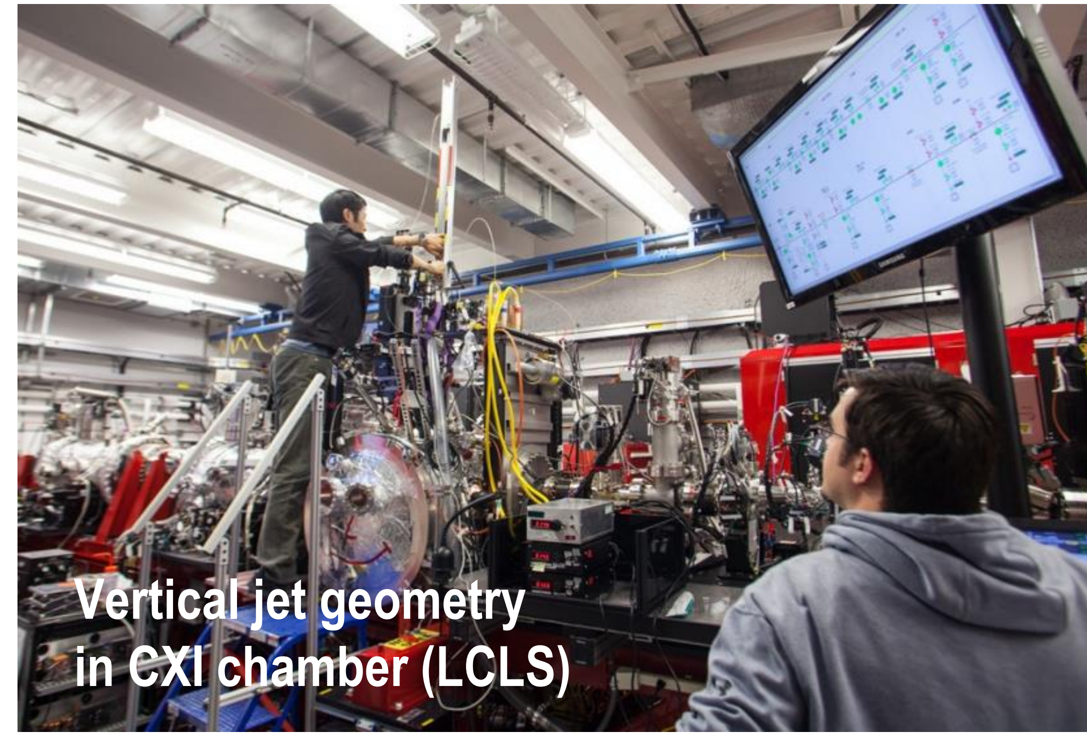


- 16M Jungfrau
- 75 um pixels
- 30 x 30 cm^2 area with a hole for the direct beam
- vacuum compatible
- adaptive gain for high flux measurements

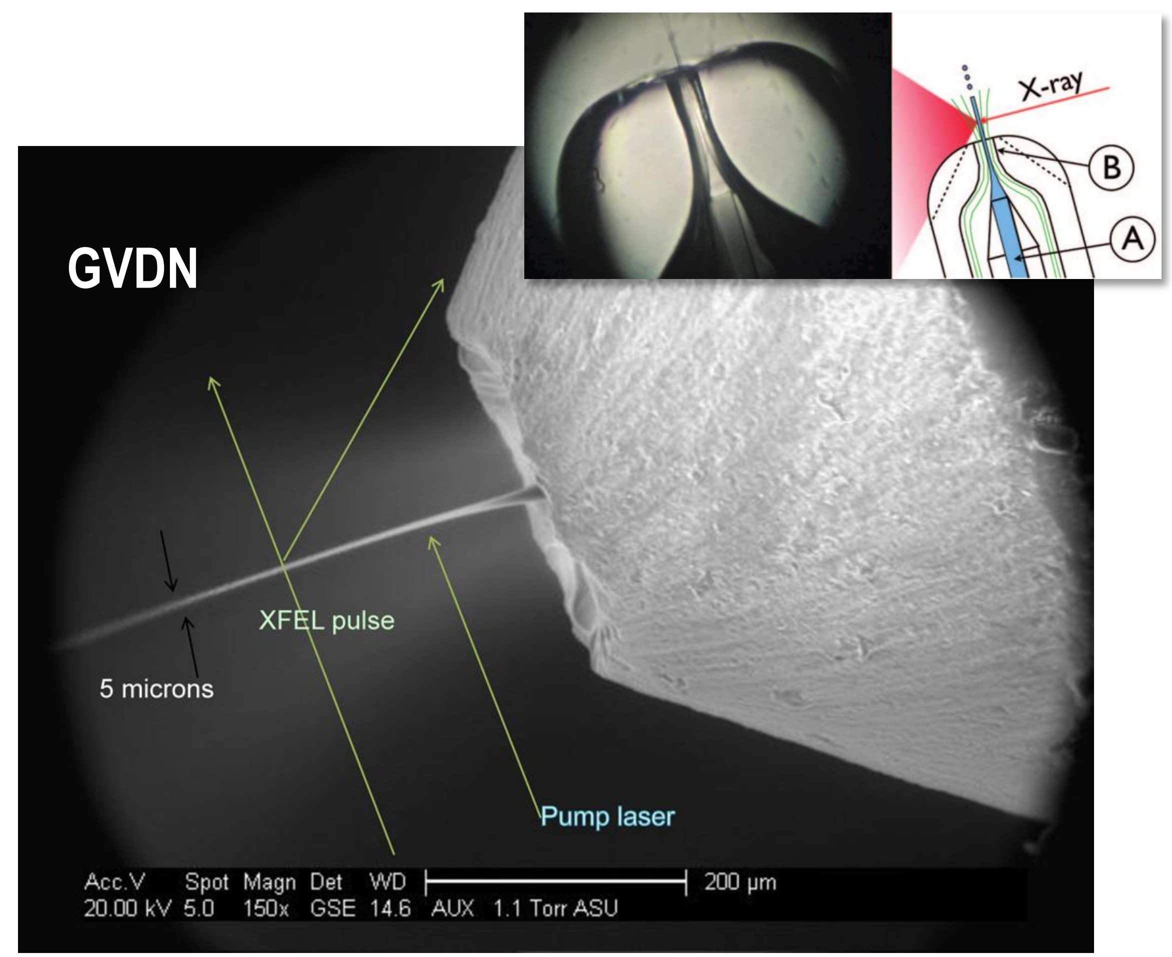
Sample delivery: liquid jet for SFX and WAXS



U. Weierstall et al. *Rev. Sci. Instr.* **83**, 035108 (2012)

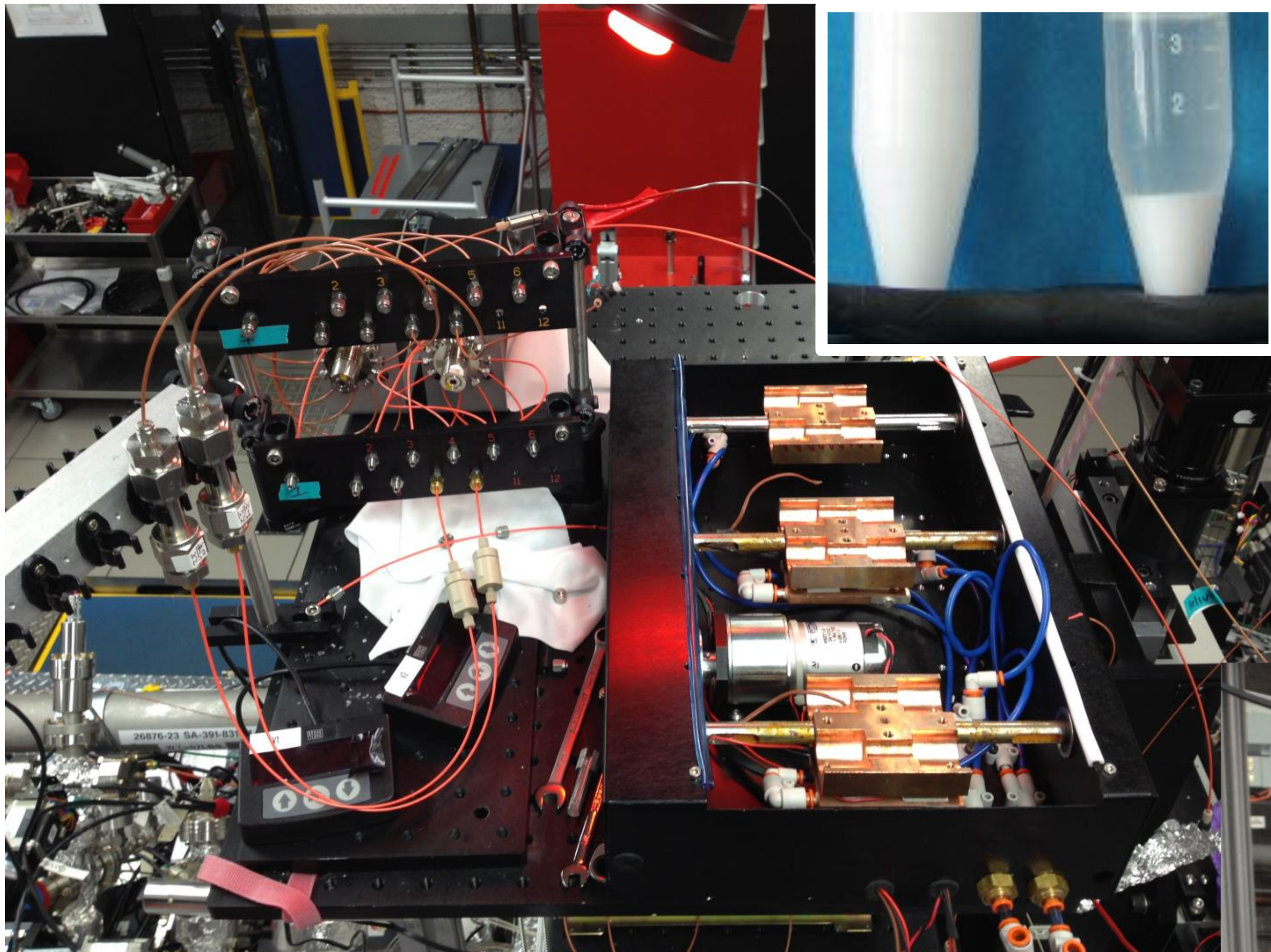


- Based on Gas Virtual Dynamic Nozzles (Spence et al.)
- Liquid jet injector for nanocrystalline samples
- In use at CXI (LCLS) and SACLA
- Also useable for photochemistry (WAXS)



Massive drawback: working in vacuum is a headache

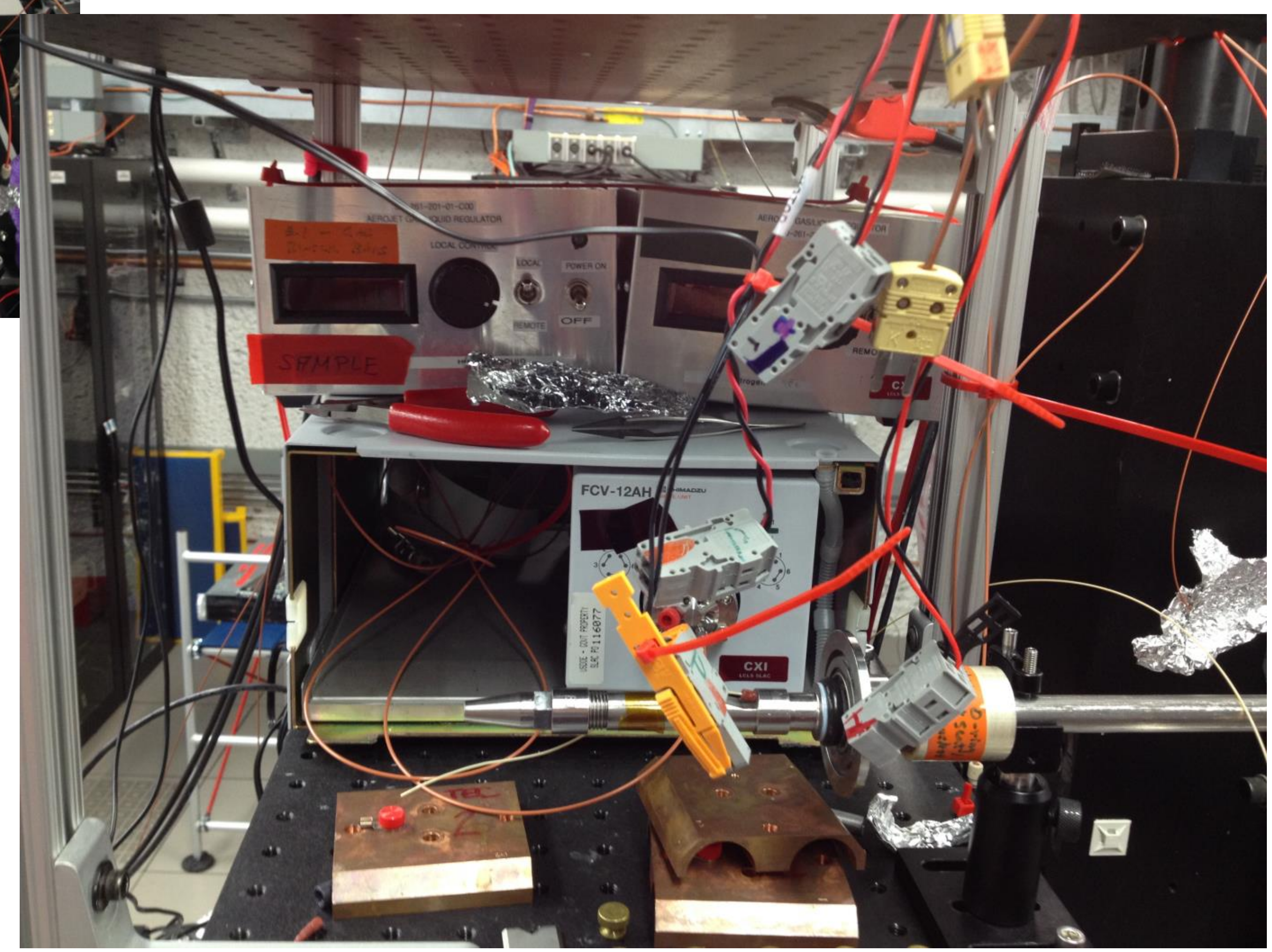
Liquid jet accessories



Gas pressure control

High-pressure liquid chromatography connections everywhere

Gas tanks for pressurization or HPLC pumps

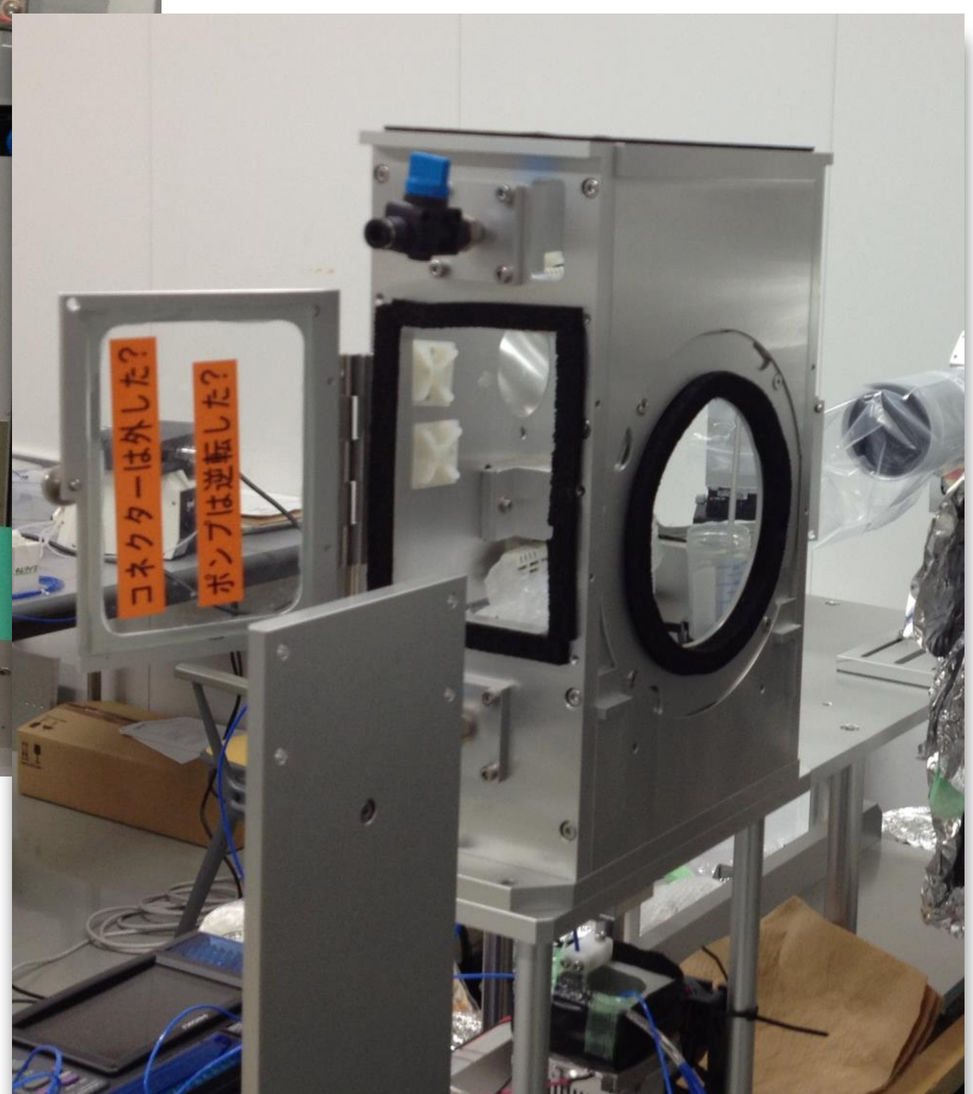


Anti-settling tank (prevent crystals from settling in container

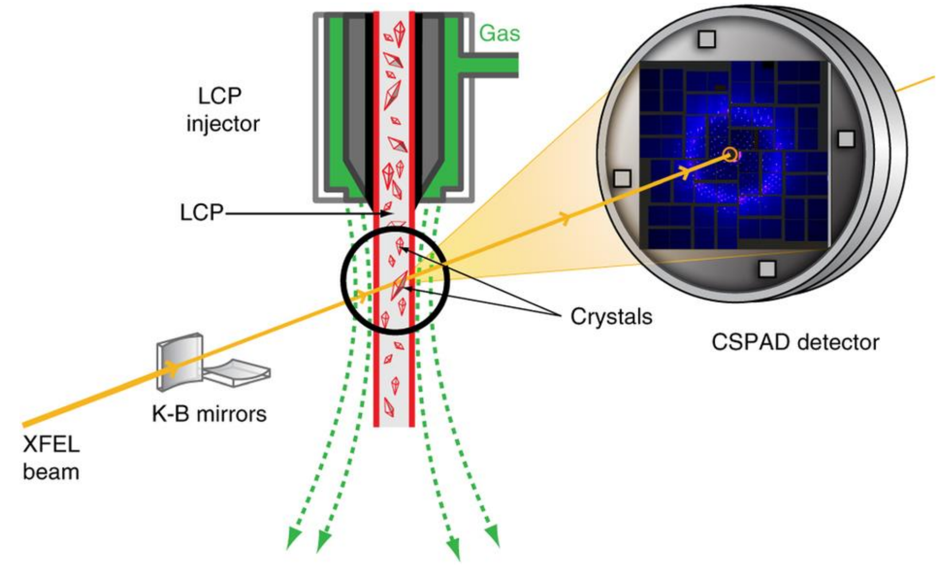
Remote controlled valves to switch between samples, buffer, water

I. Schlichting, J. Miao *Curr. Op. Struct. Bio.* 22, 613-626 (2012)

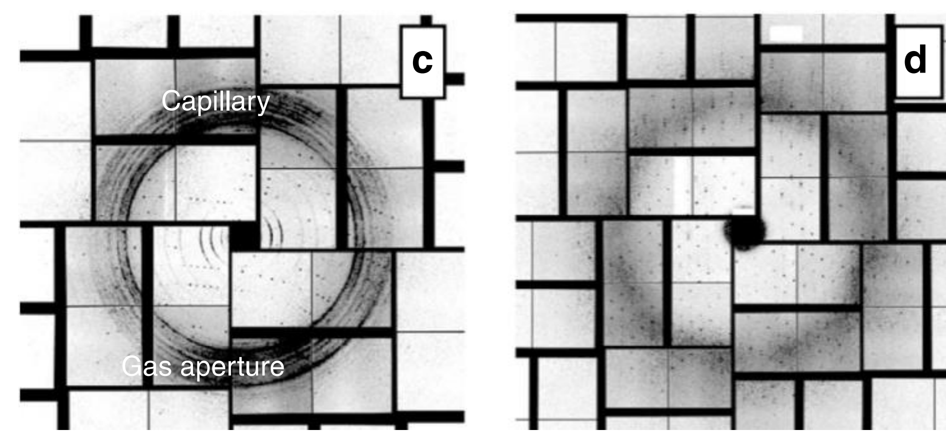
SFX sample delivery: Making things simpler



SACLA development uses GVDN in compact helium box



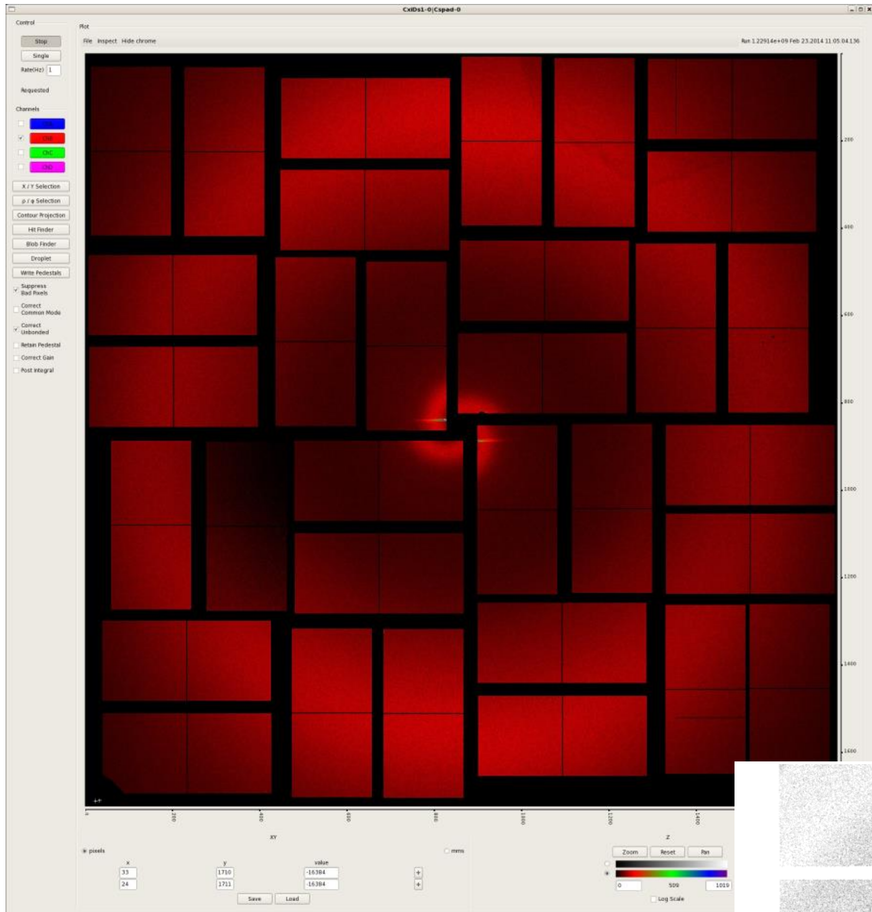
U. Weierstall et al. *Nat. Commun.* 5:3309, doi: 10.1038/ncomms4309 (2014)



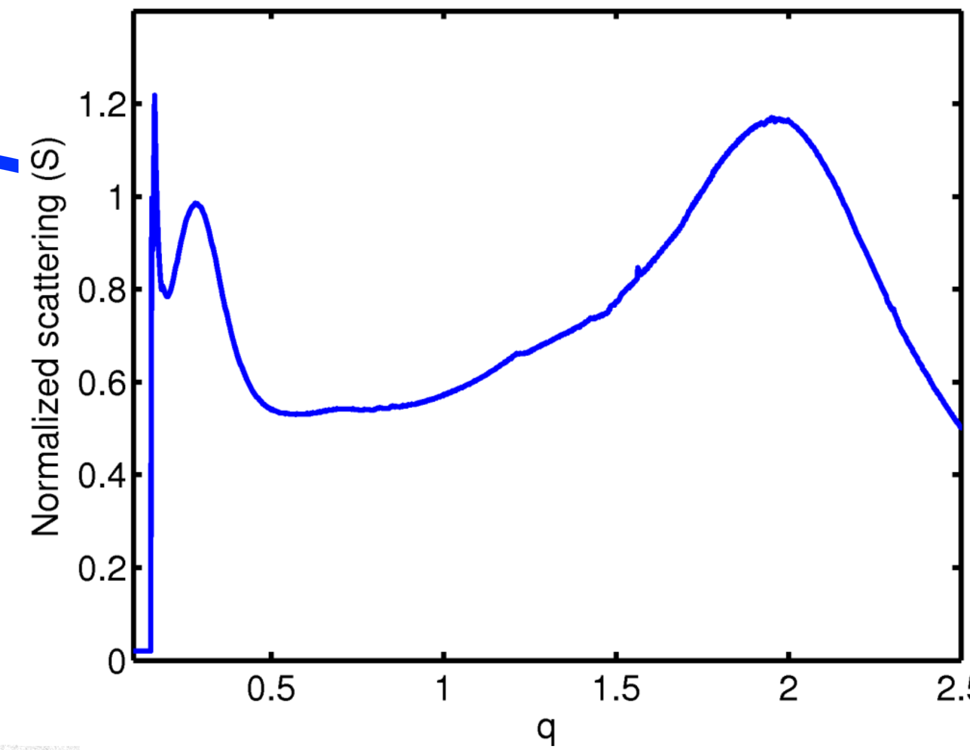
Lipidic Cubic Phase jet uses less sample and is more stable

SFX Data Processing

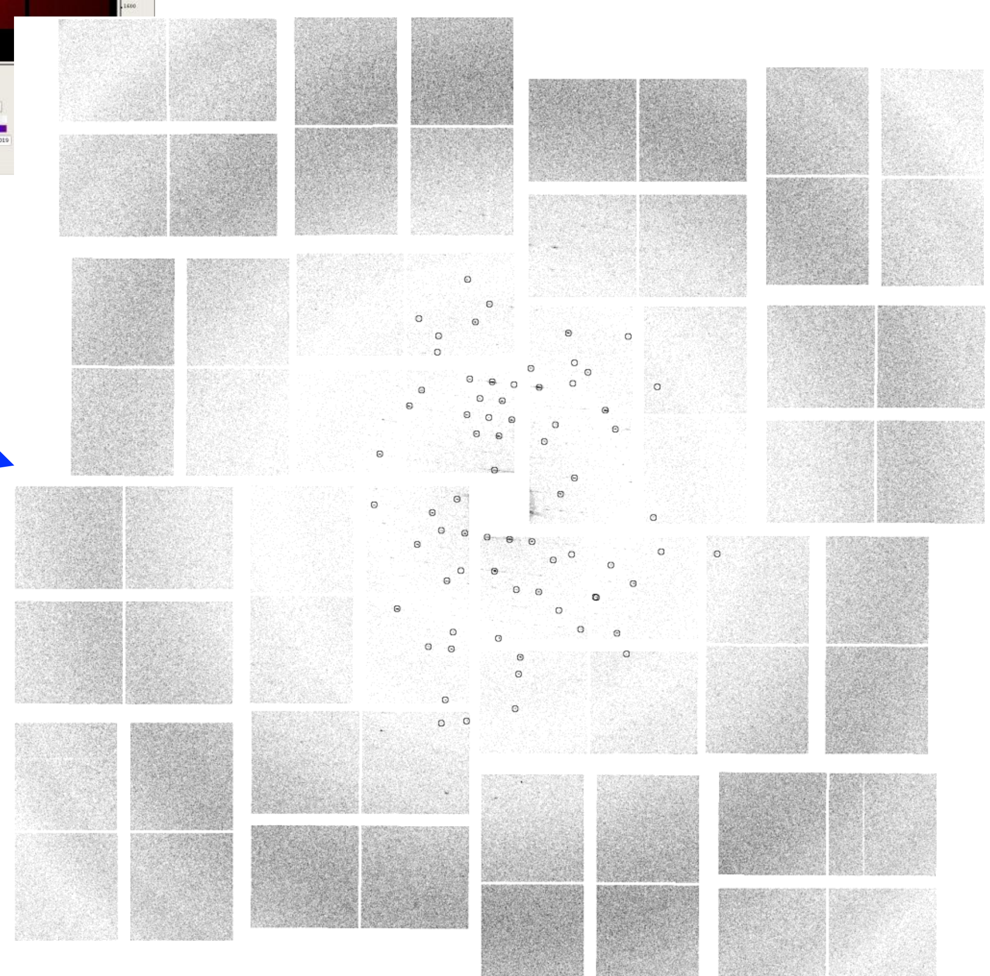
http://www.desy.de/~twhite/crystfel/



Radial integration (WAXS)



Hit finding followed by peak finding (SFX)



***CrystFEL*: a software suite for snapshot serial crystallography**

Thomas A. White,[a*] Richard A. Kirian,[b] Andrew V. Martin,[a] Andrew Aquila,[a] Karol Nass,[a,c] Anton Barty[a] and Henry N. Chapman[a,c]

J. Appl. Cryst. (2012). **45**, 335–341

Crystallographic data processing for free-electron laser sources

Thomas A. White,[a*] Anton Barty,[a] Francesco Stellato,[a] James M. Holton,[e,f] Richard A. Kirian,[a,c] Nadia A. Zatsepin[d] and Henry N. Chapman[a,b]

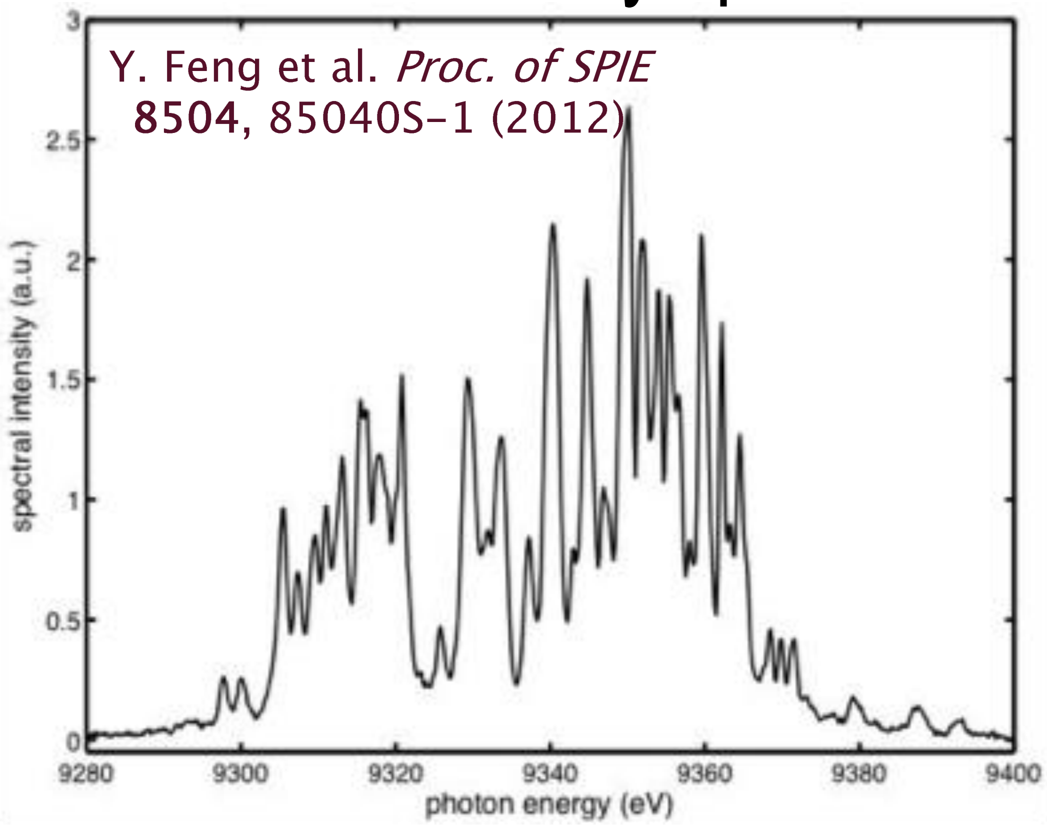
Acta Cryst. (2013). D**69**, 1231–1240

Experiment #2

X-ray spectroscopy experiments

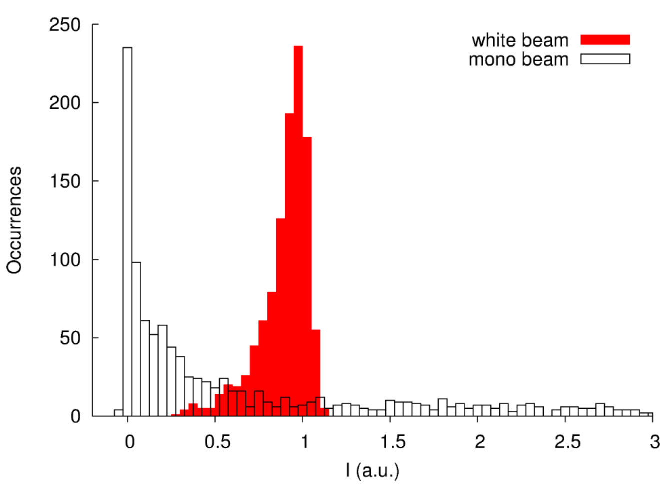
X-ray spectroscopy experiments at XFELs

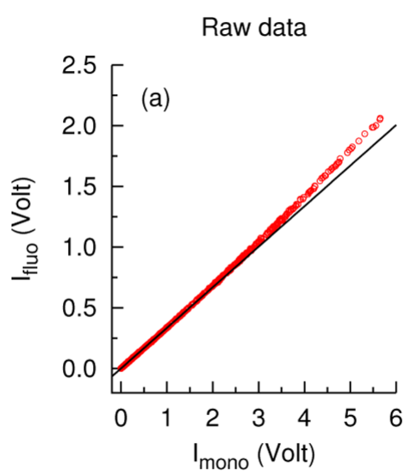
Stochastic X-ray spectrum

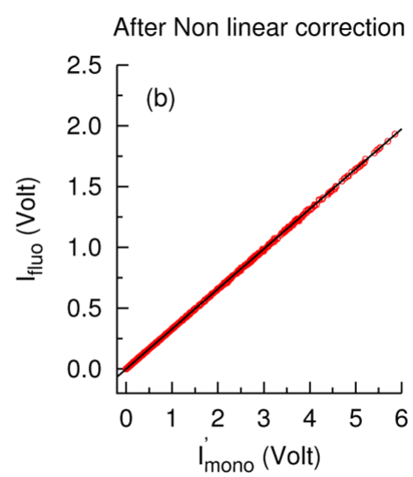


Y. Feng et al. *Proc. of SPIE* **8504**, 85040S-1 (2012)

Spectral instability and detector linearity

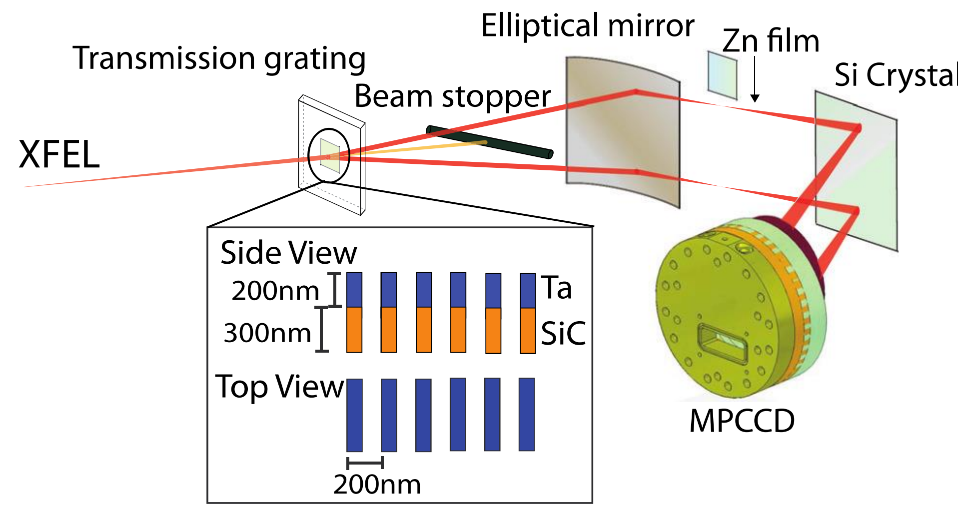


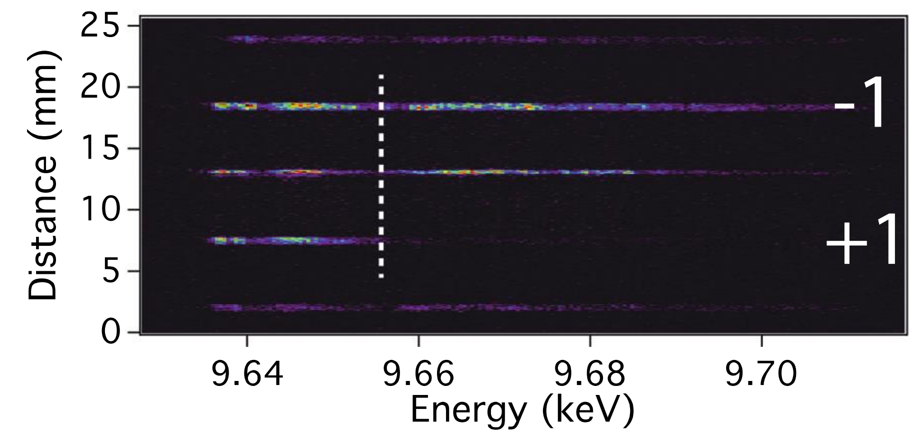


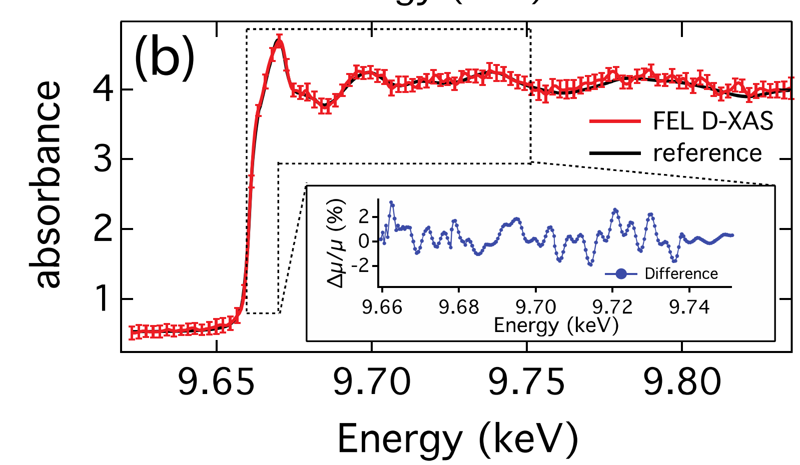


H.T. Lemke et al. *JPCA* **117**, 735 (2013)

Single-shot XAS



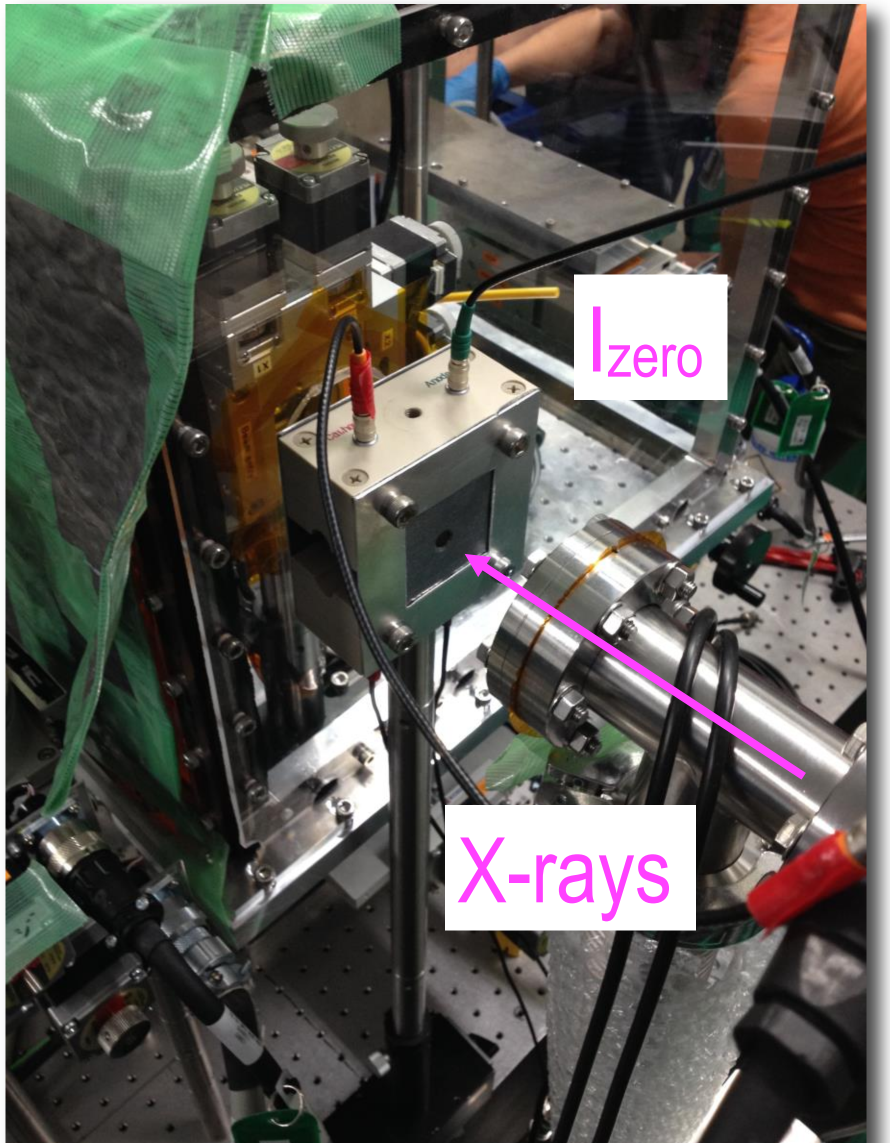


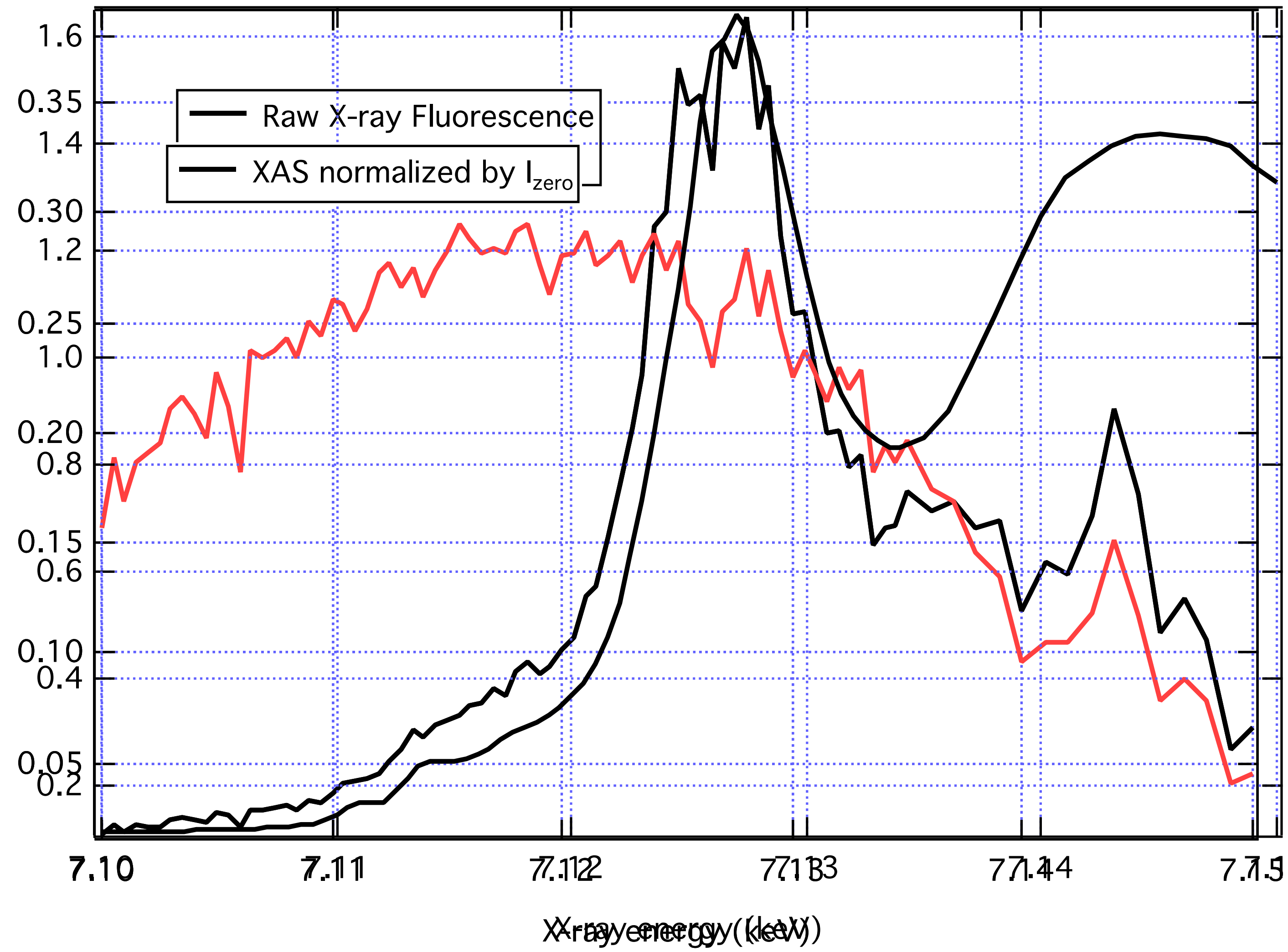


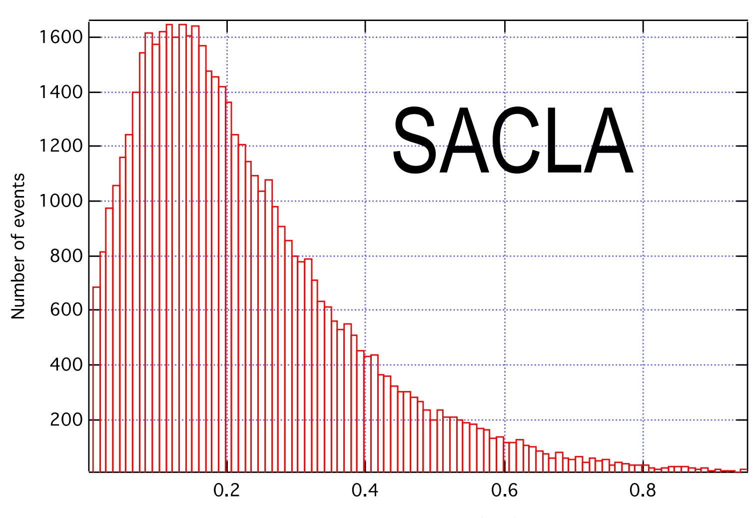
T. Katayama et al. *App. Phys. Lett.* **103**, 131105 (2013)

Shot-by-shot diagnostics and techniques have significant advantages at XFELs

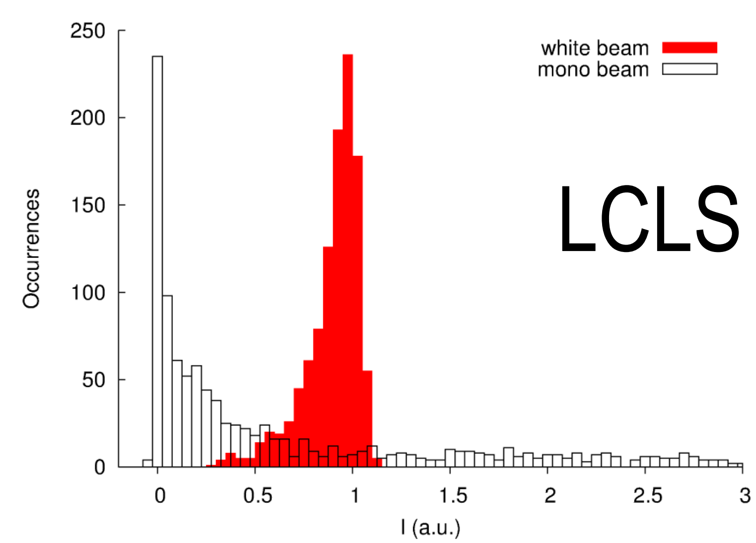
X-ray intensity normalization for scans



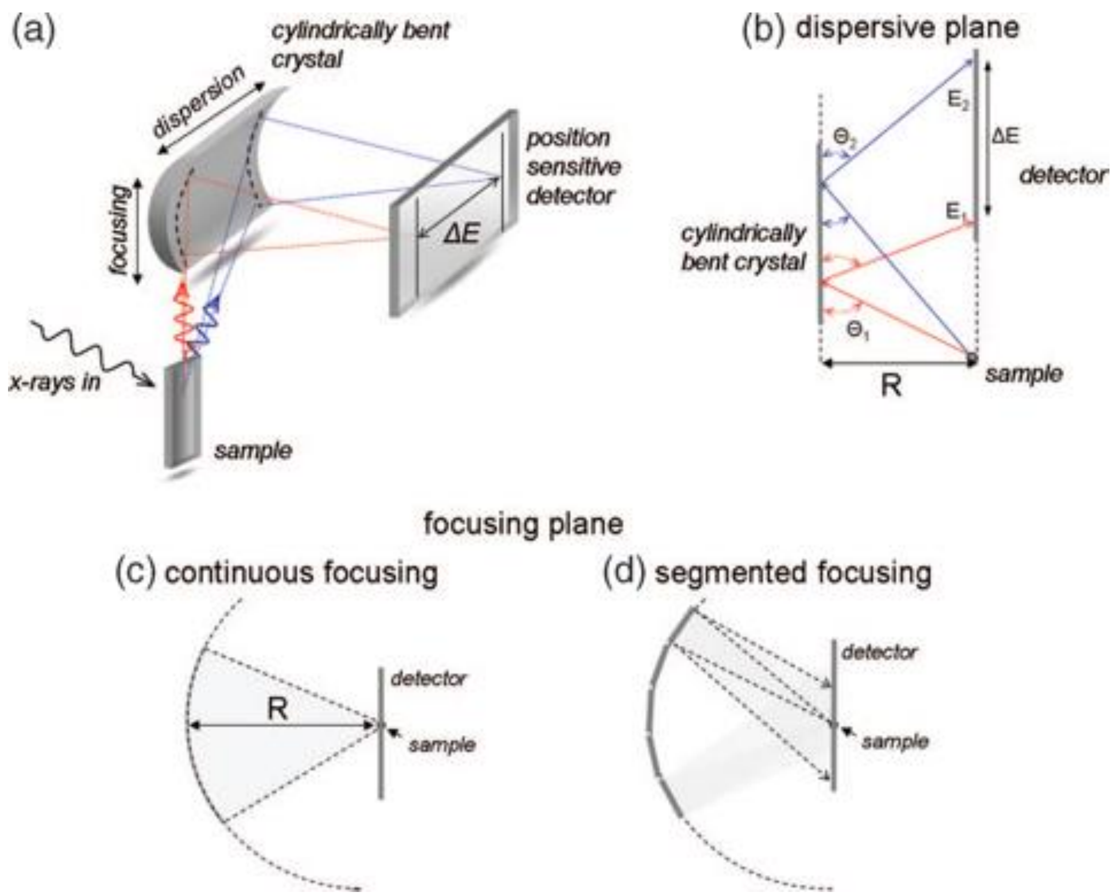


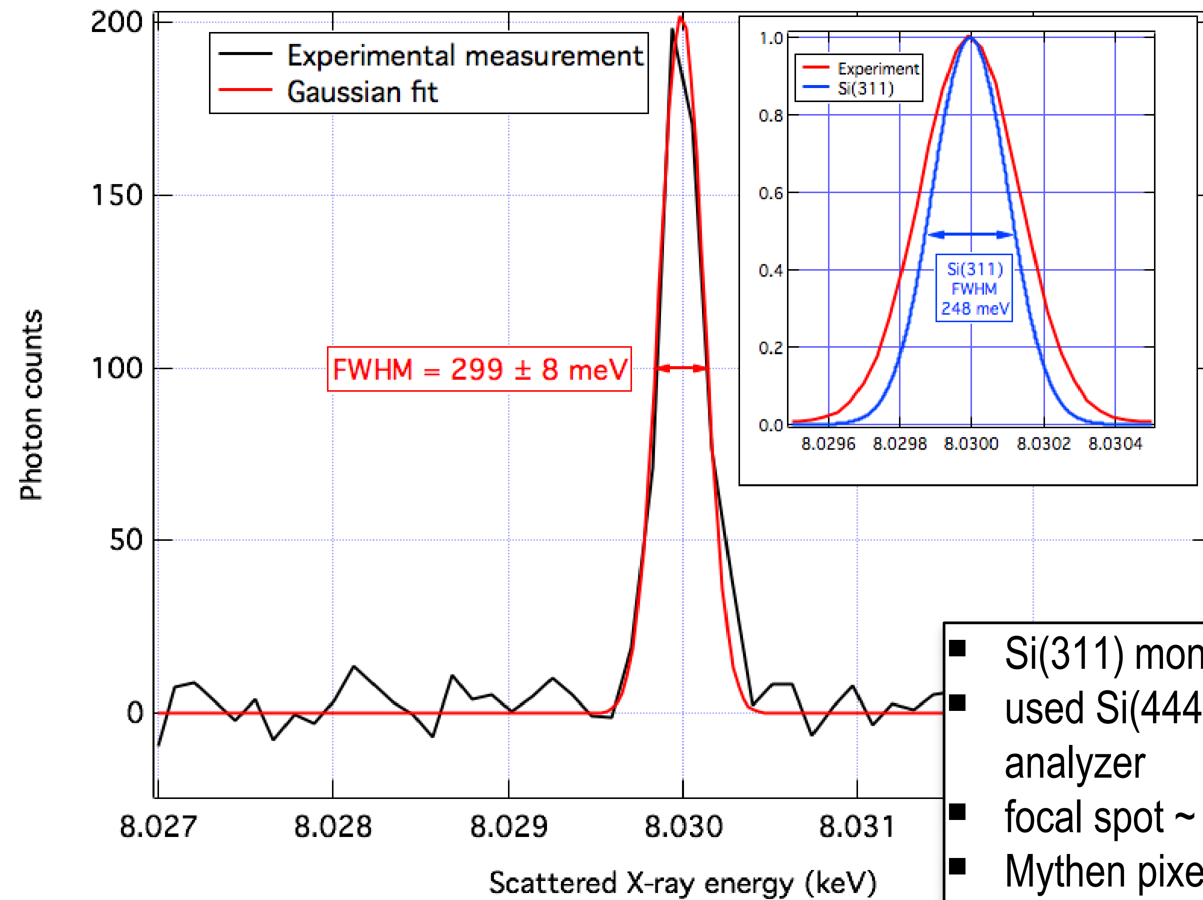


Vs



Introduction to von Hamos X-ray spectrometers





- Si(311) mono
- used Si(444) diffraction from analyzer
- focal spot ~ 80 μm
- Mythen pixels 50 μm
- Resolution: 100 meV

von Hamos geometry

- Dispersive so you get a complete energy range per XFEL pulse
- 25 cm radius of curvature crystals means compact setup
- Scan-free setup means no moving parts
- Development of segmented crystals provides excellent energy resolution
- Scales easily with additional crystal+detector pairs to cover other energies

HoXS: Holochromatic X-ray Spectroscopy

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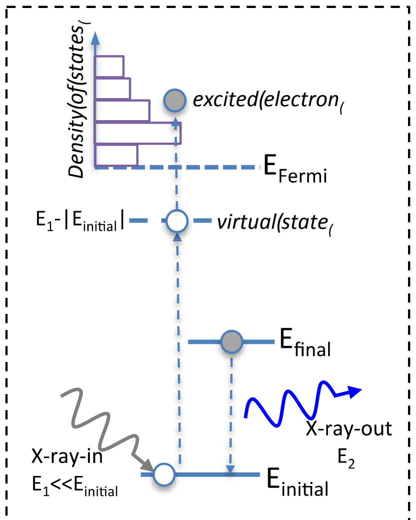
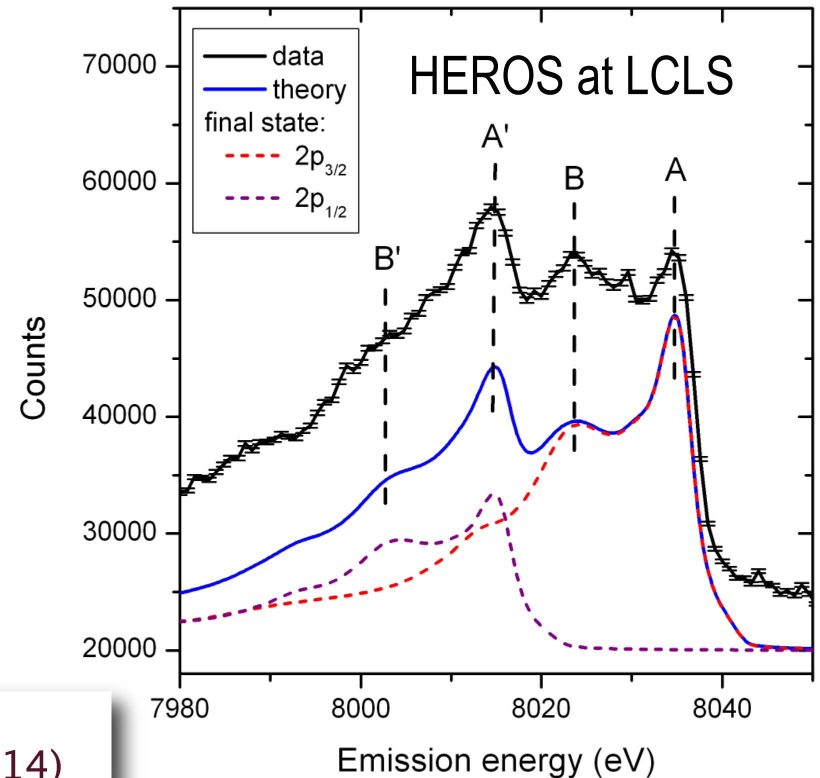
Goal: The ability to obtain a holistic picture of the sample

High energy resolution off-resonant spectroscopy (HEROS)

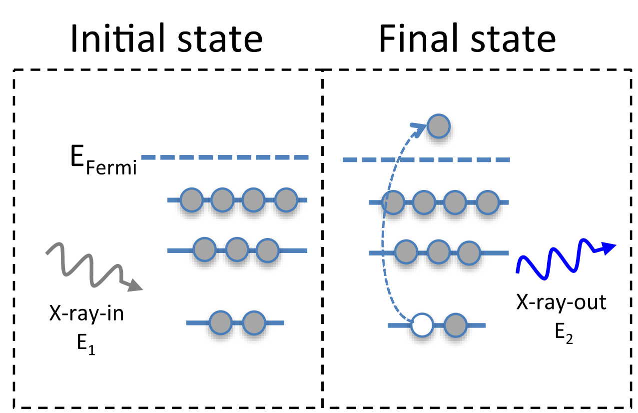
Energy diagram level

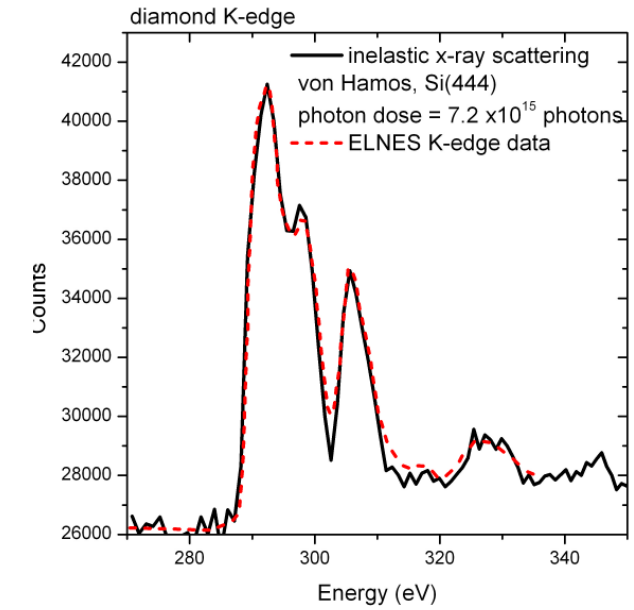


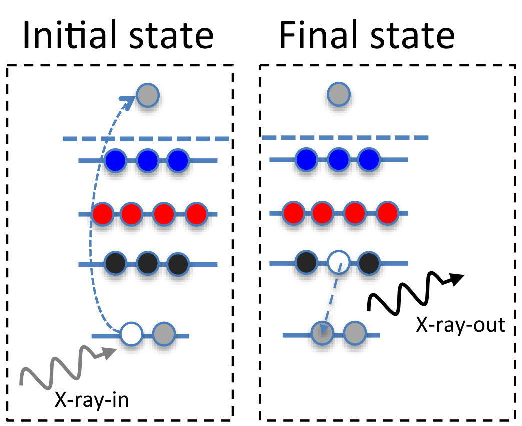


HEROS: J. Szlachetko et al. *Struct. Dyn.* 1, 021101(2014)

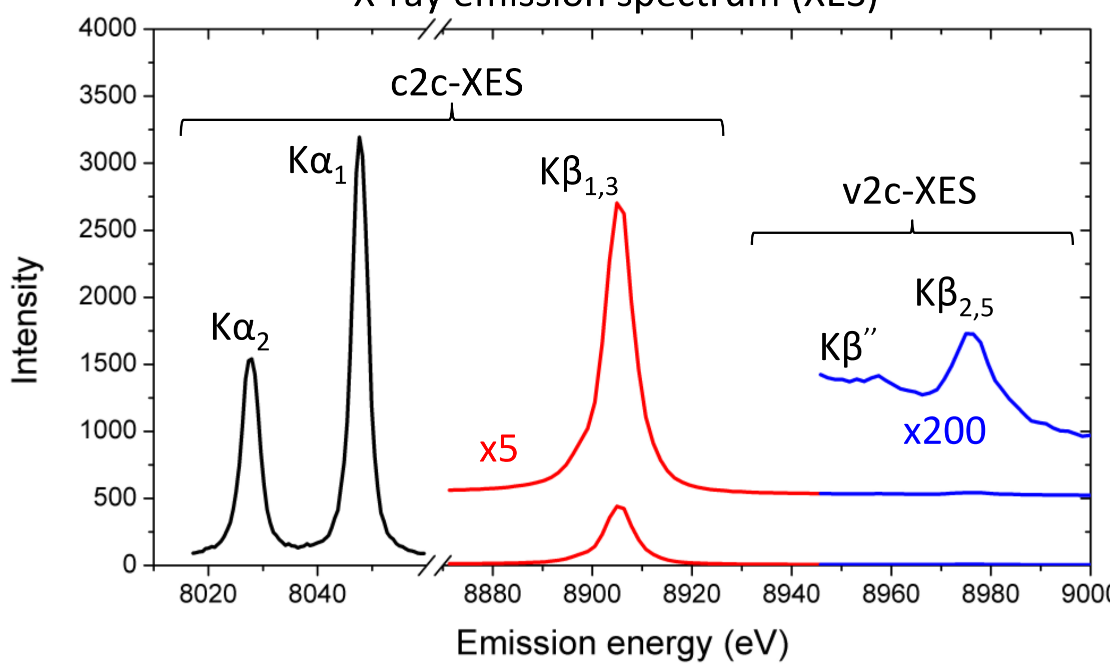
Inelastic x-ray scattering (IXS)



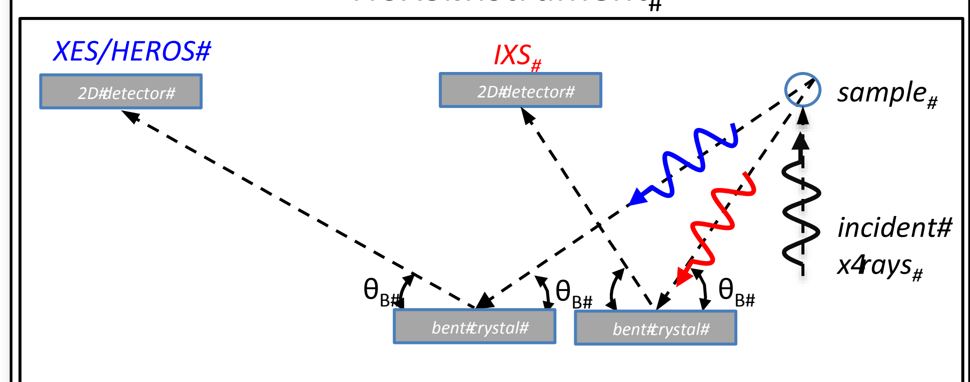


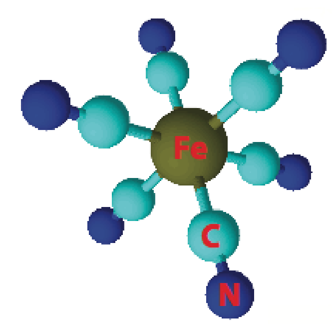


X-ray emission spectrum (XES)



HoXS instrument



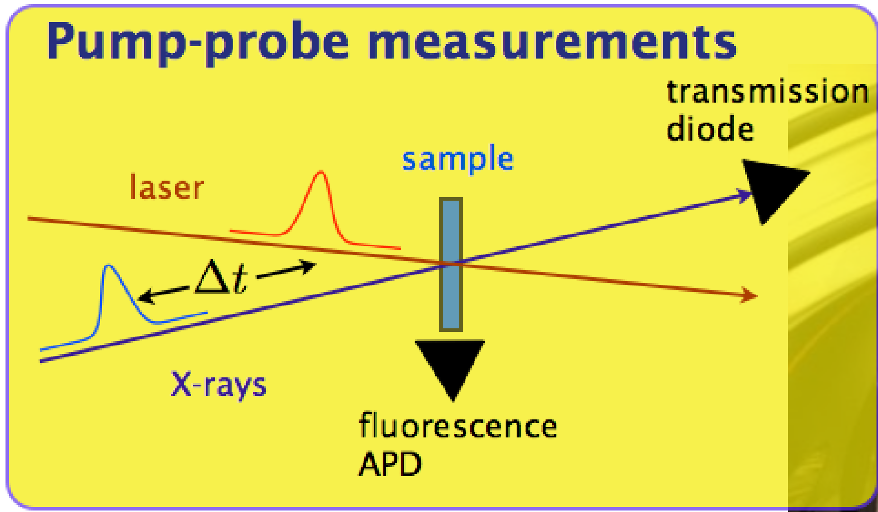


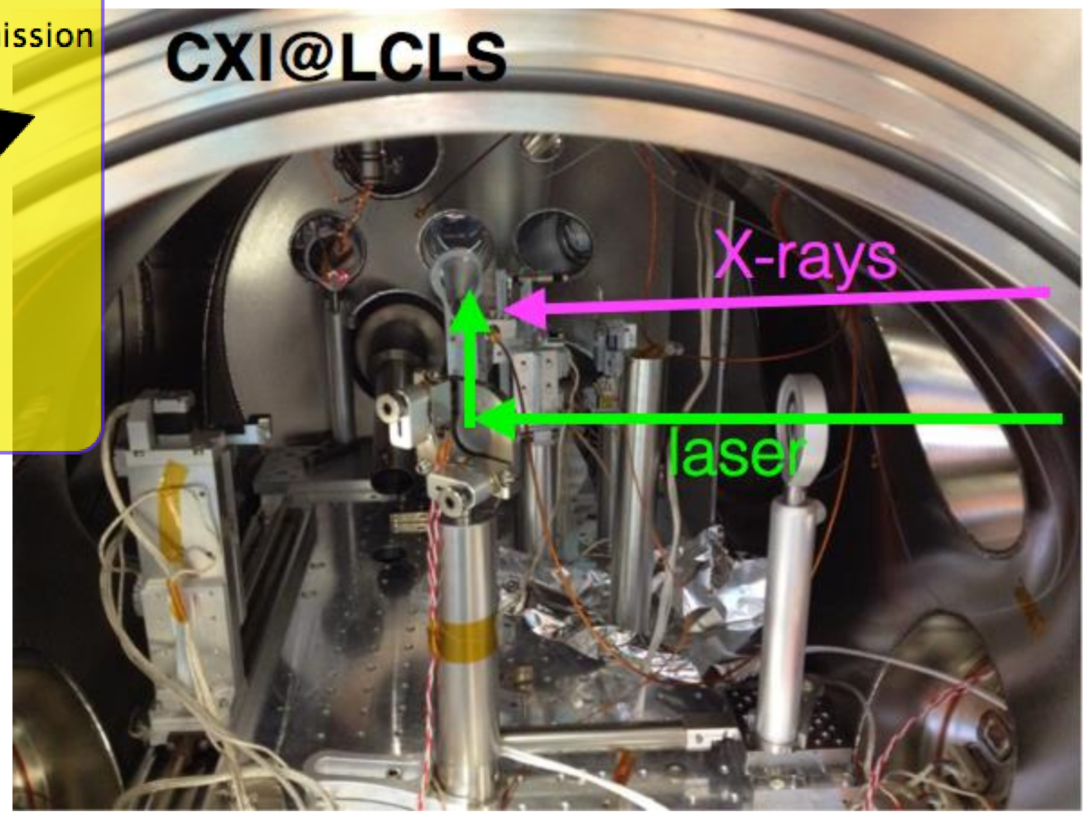
For example Fe Kβ XES and C or N K-edge IXS measured simultaneously

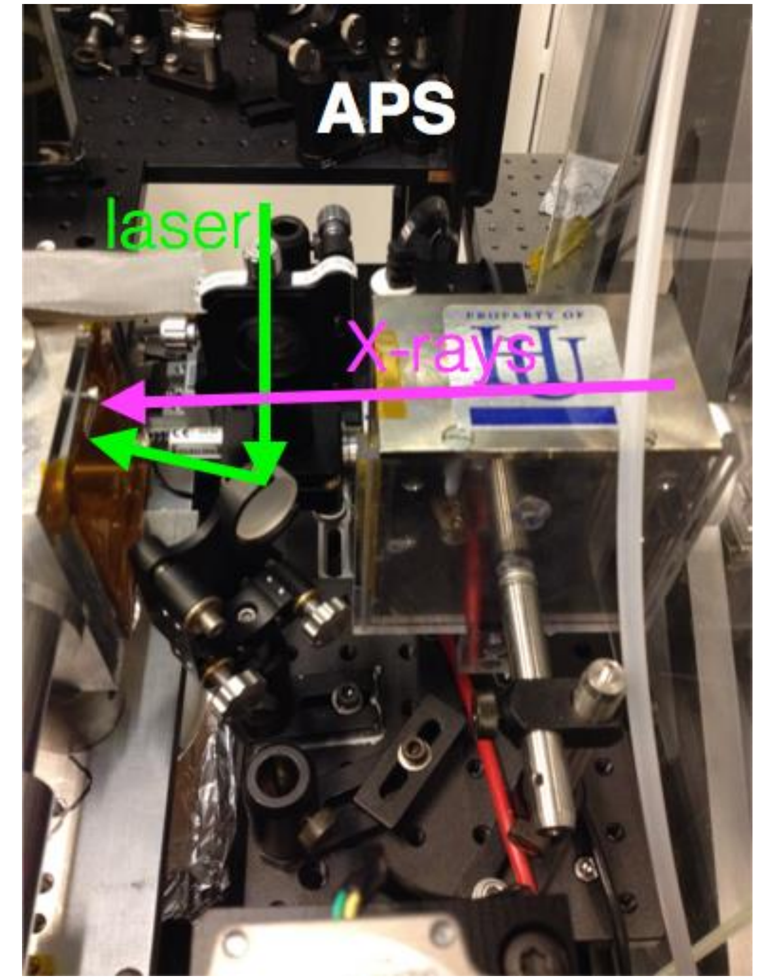
Experiment #3

Pump-probe experiments

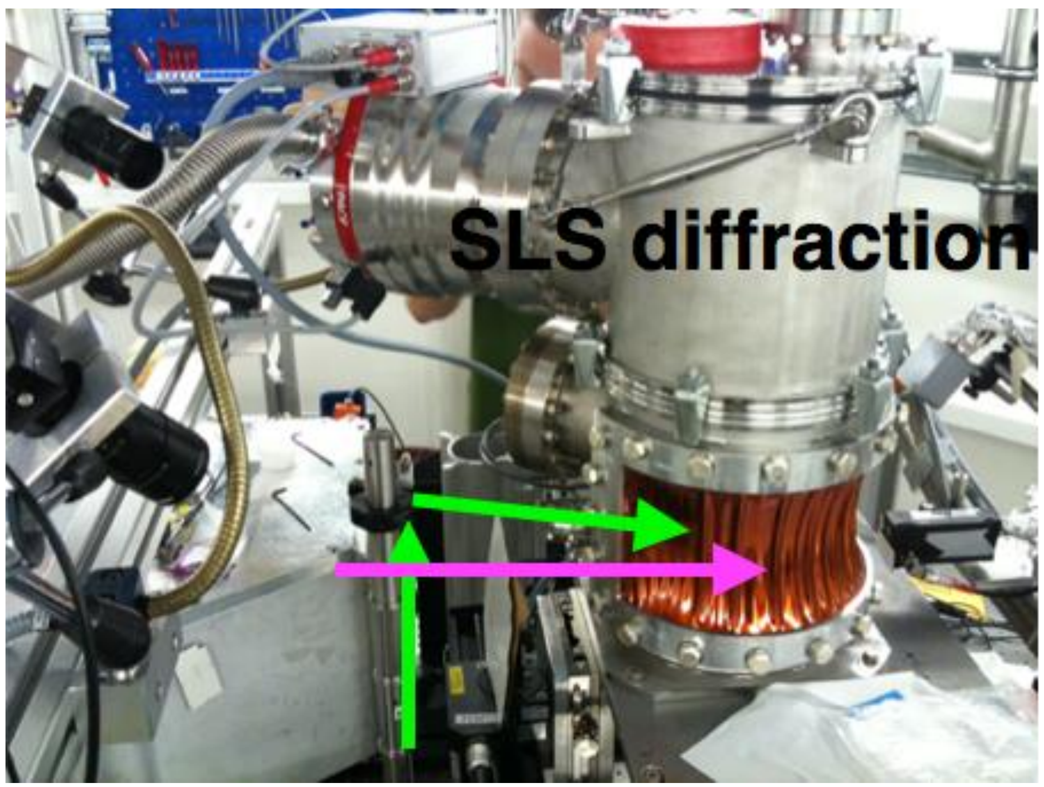
Pump-probe measurements

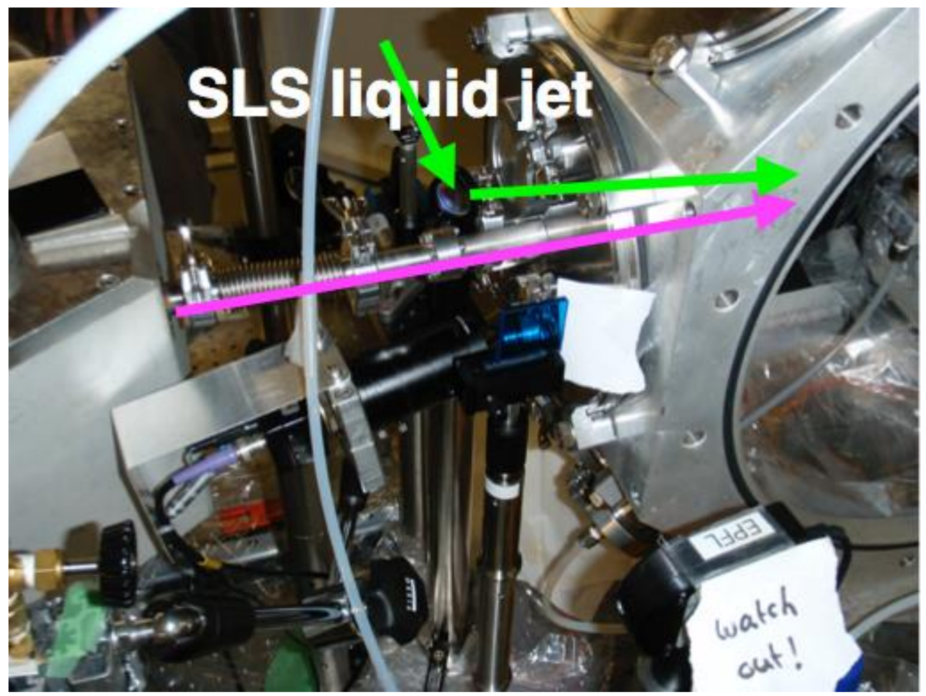






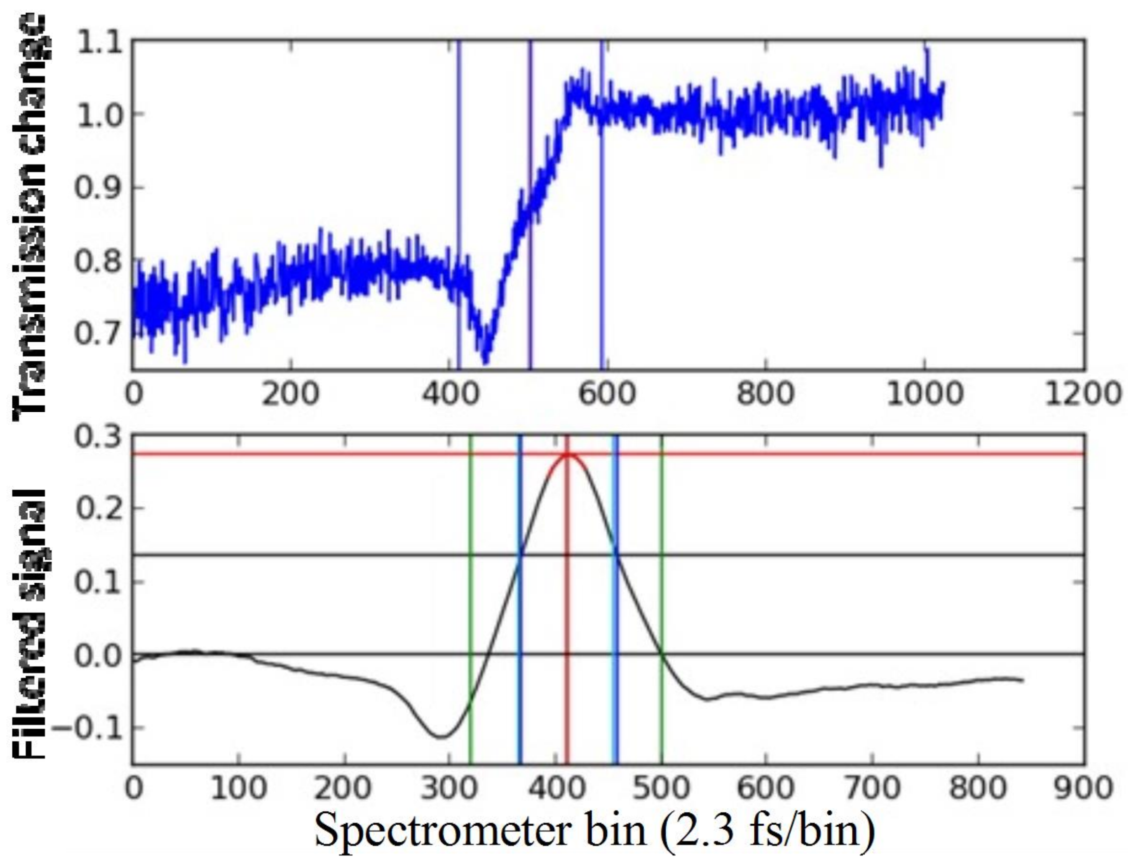
First we need to get the laser into the setup

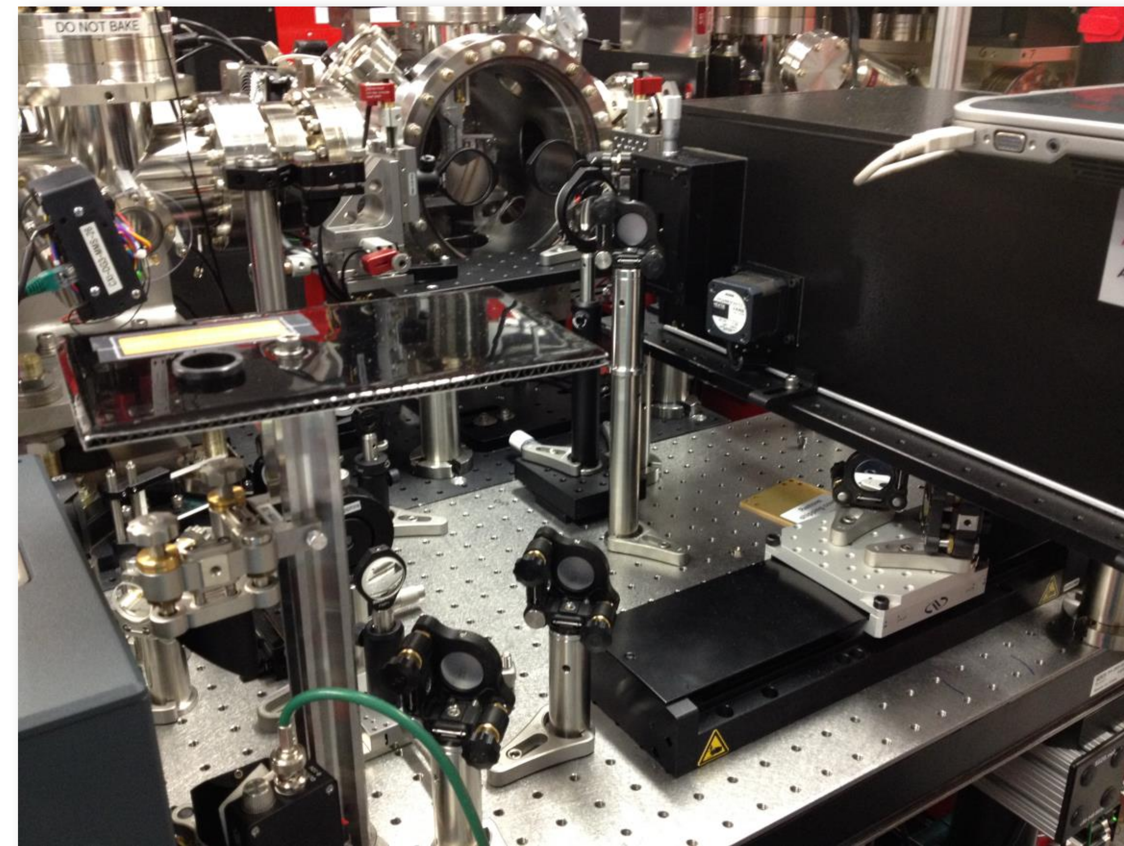


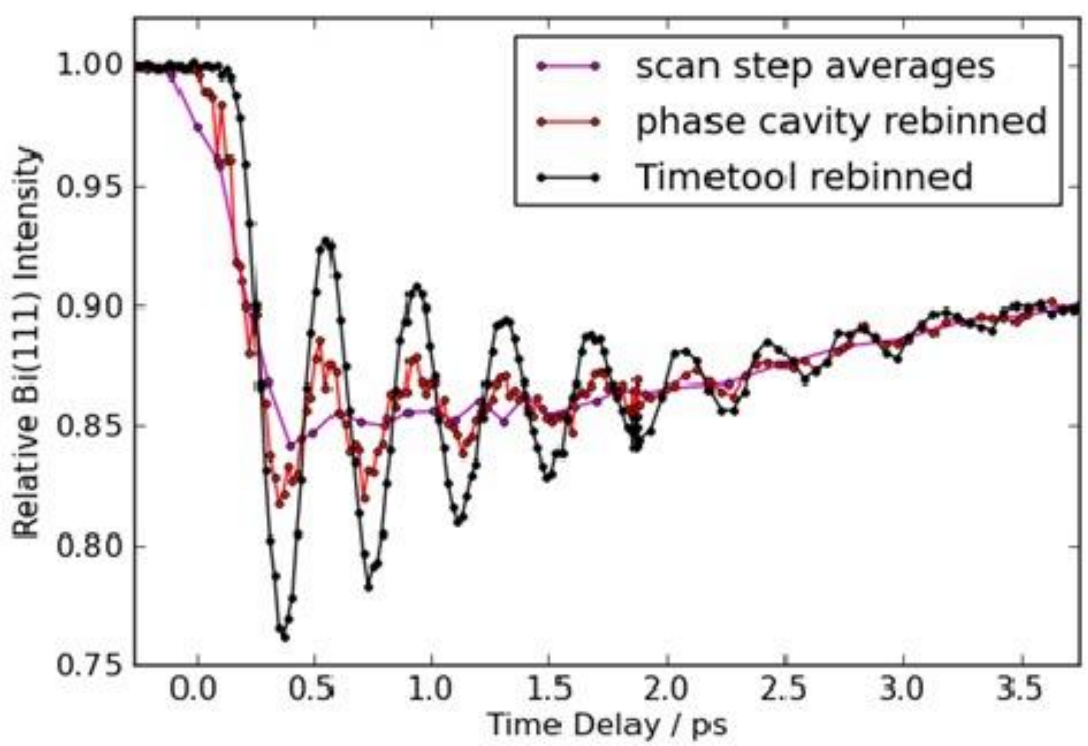


Control over the laser is pretty simple so this is quite flexible

Pump-probe jitter correction

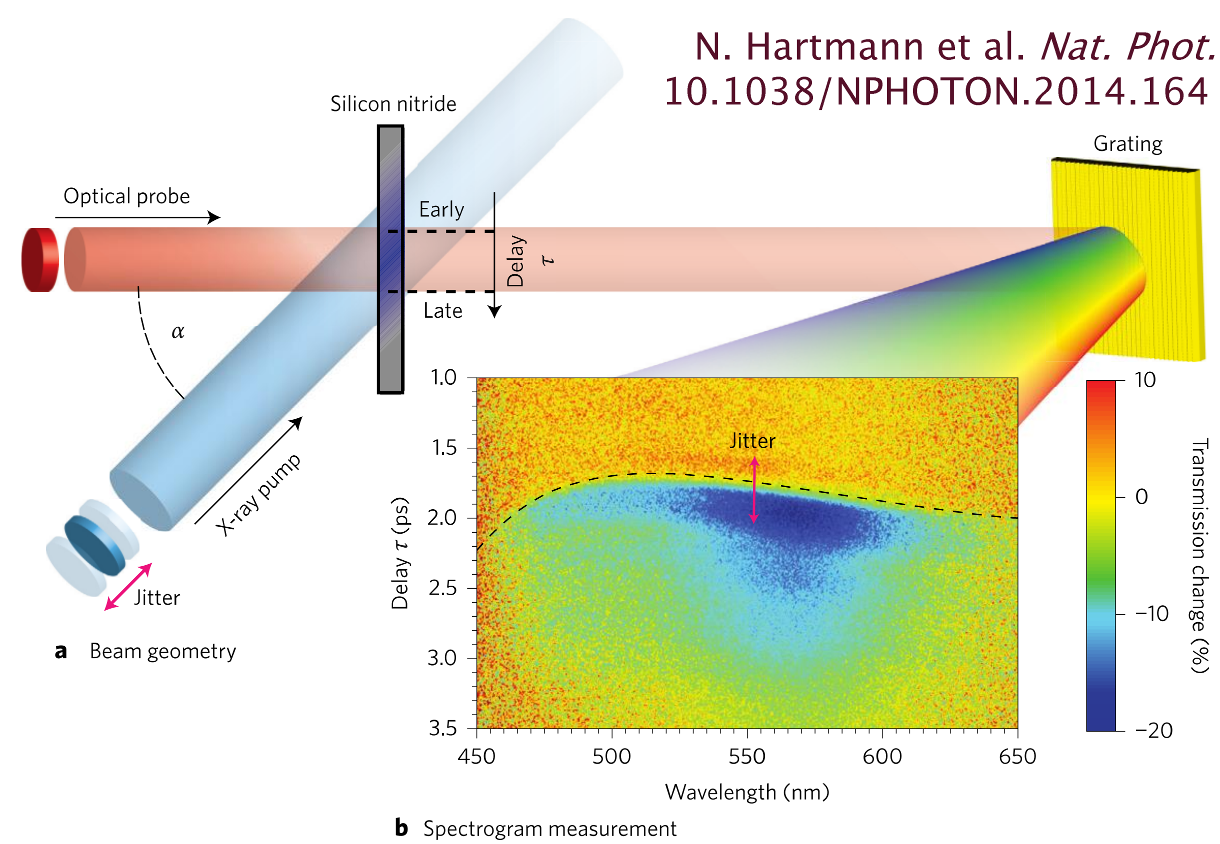






H.T. Lemke et al. *Proc. of the SPIE* **8778**, 87780S-1 (2013)

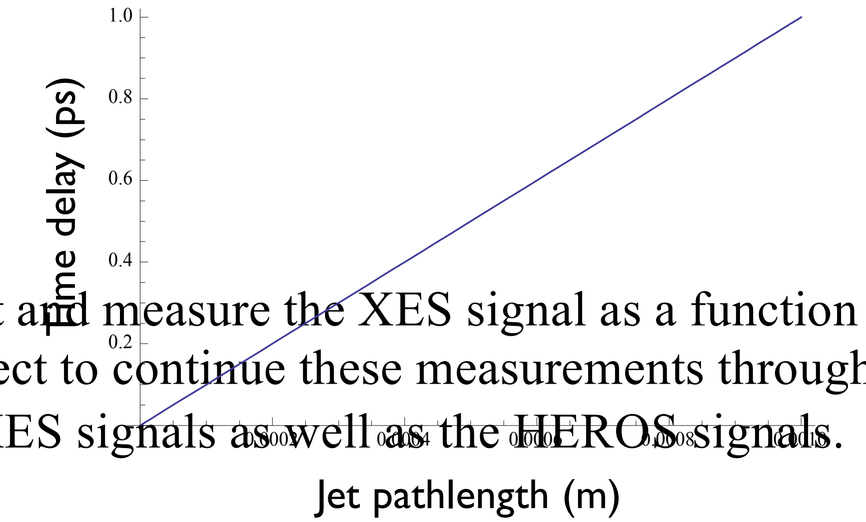
N. Hartmann et al. *Nat. Phot.* DOI: 10.1038/NPHOTON.2014.164 (2014)



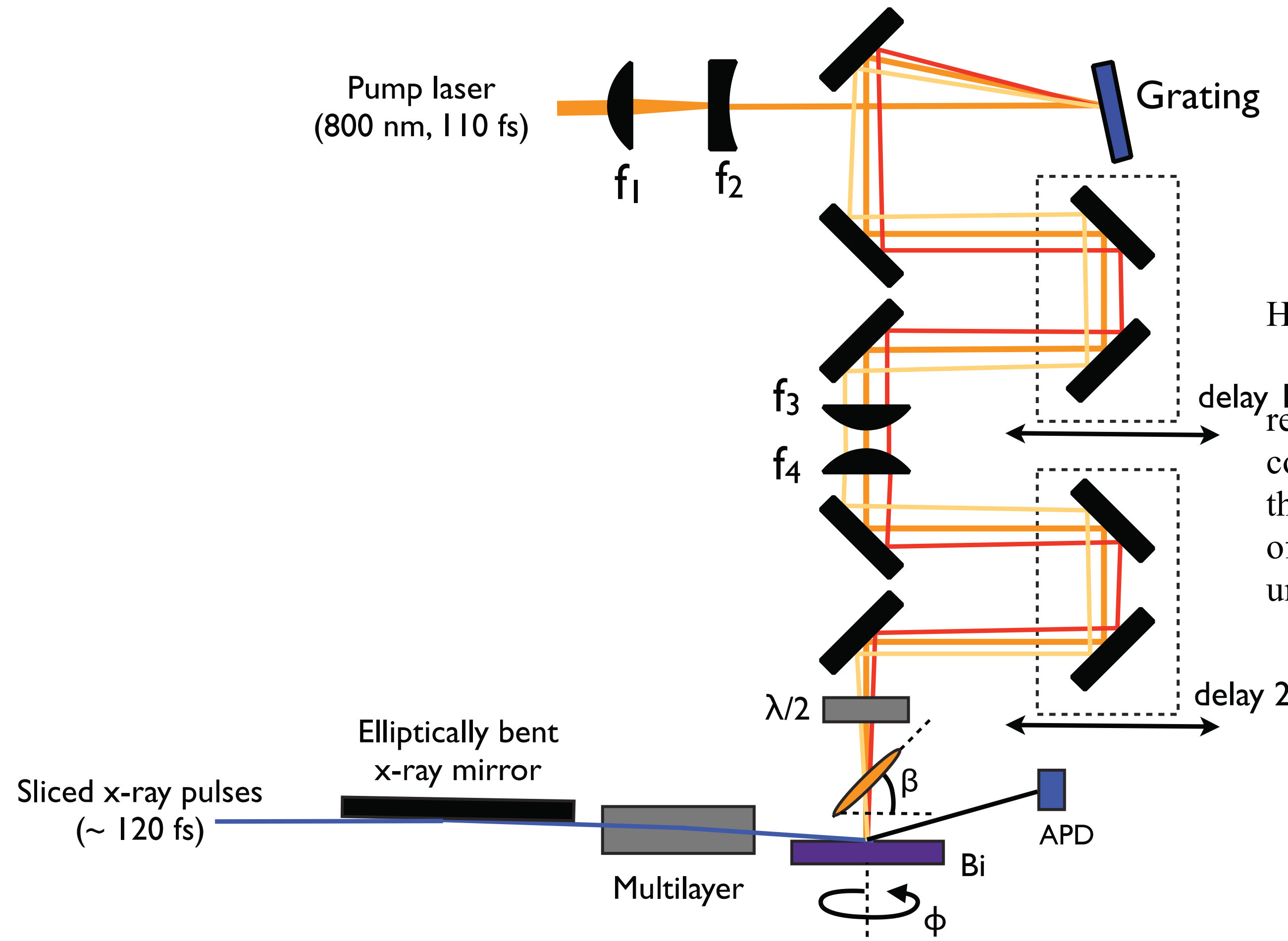
Getting the best pump-probe time resolution

The time resolution can be affected by many things, including geometry and sample index of refraction

$$\tan(\beta) = \frac{\lambda_0}{d_g M \sqrt{1-(\lambda_0/d_g - \sin\Theta)^2}}$$



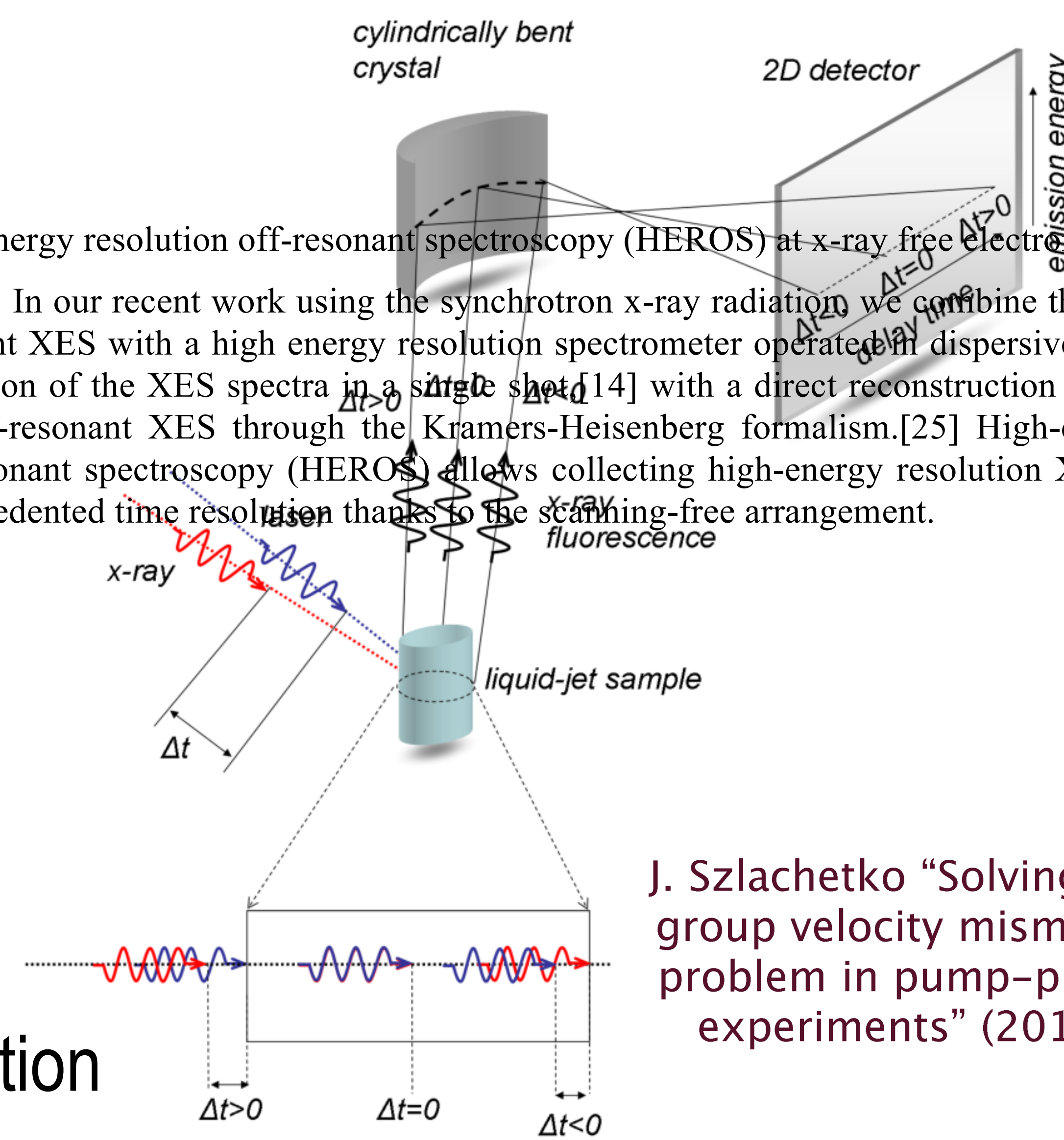
the jet to obtain the 1 mm footprint and measure the XES signal as a function of time between laser and X-rays. We expect to continue these measurements through the thi shifts to measure the Ka and Kb XES signals as well as the HEROS signals.



S.L. Johnson et al. *Phys. Rev. B* **87**, 054301 (2013)

High energy resolution off-resonant spectroscopy (HEROS) at x-ray free electron lasers

In our recent work using the synchrotron x-ray radiation we combine the conce resonant XES with a high energy resolution spectrometer operated in dispersive mode[collection of the XES spectra in a single shot[14] with a direct reconstruction of the X the off-resonant XES through the Kramers-Heisenberg formalism.[25] High-energy r off-resonant spectroscopy (HEROS) allows collecting high-energy resolution XAS spe unprecedented time resolution thanks to the scanning-free arrangement.



J. Szlachetko "Solving the group velocity mismatch problem in pump-probe experiments" (2013)

In other words it's pretty easy to wreck time resolution

Conclusions

This is obviously a SwissFEL perspective, but it's by no means unique

Experiments: Users will definitely use the XFEL in ways we haven't thought of

Optics: Optics can wreck things very quickly (acceptance, coherence)
Changing the photon energy needs to be as simple as possible

Diagnostics: The most important diagnostic is the one closest to the experiment

Priorities: XFEL experimental stations need to duplicate many storage ring beam

XFEL Science Success Stories

- Serial Femtosecond Crystallography (Biology)
- Pump-probe Experiments (Chemistry, Biology, Condensed Matter Physics)
- Nonlinear X-ray Experiments (AMO, Condensed Matter Physics, Chemistry)

PAUL SCHERRER INSTITUT

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