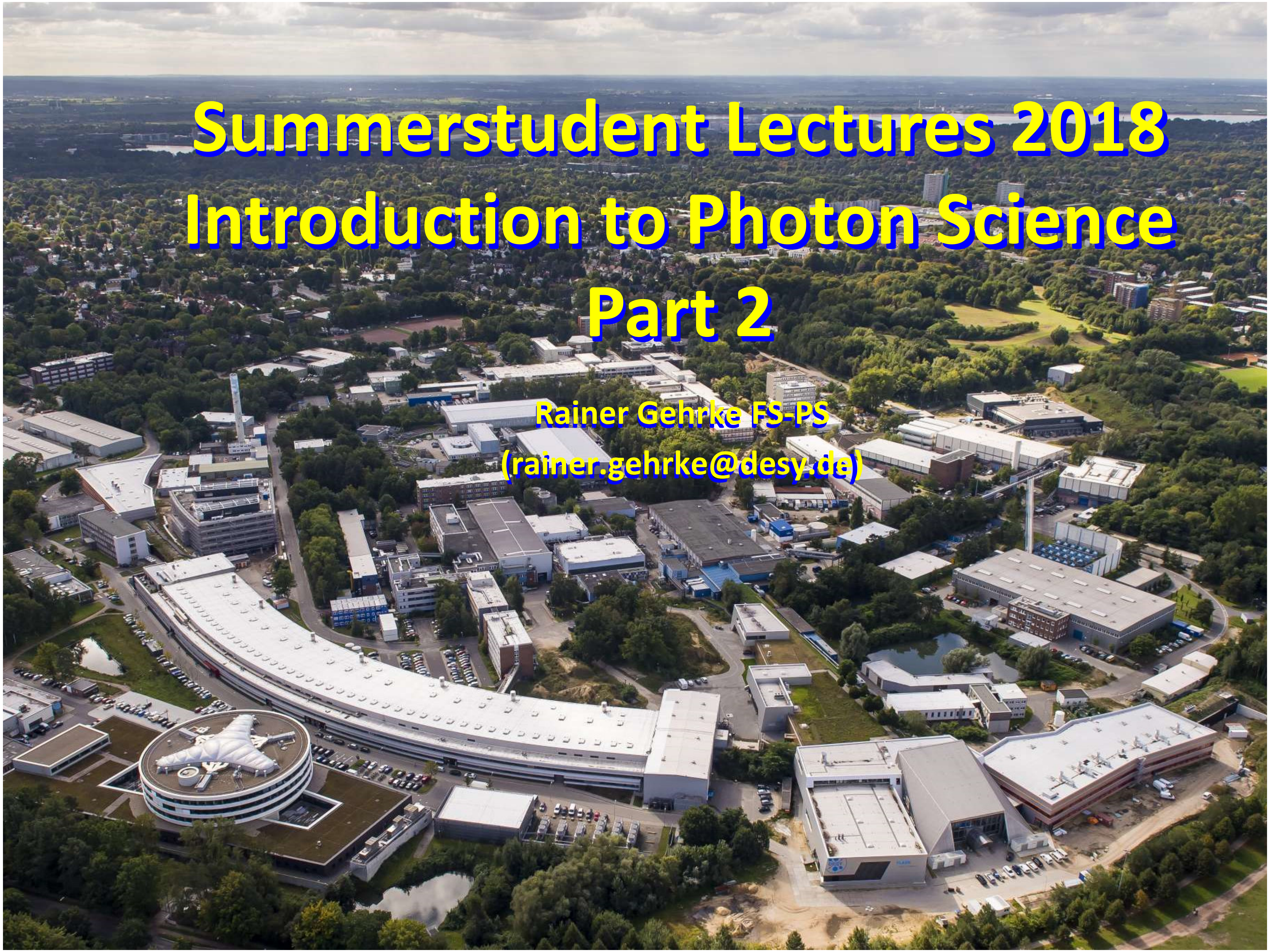
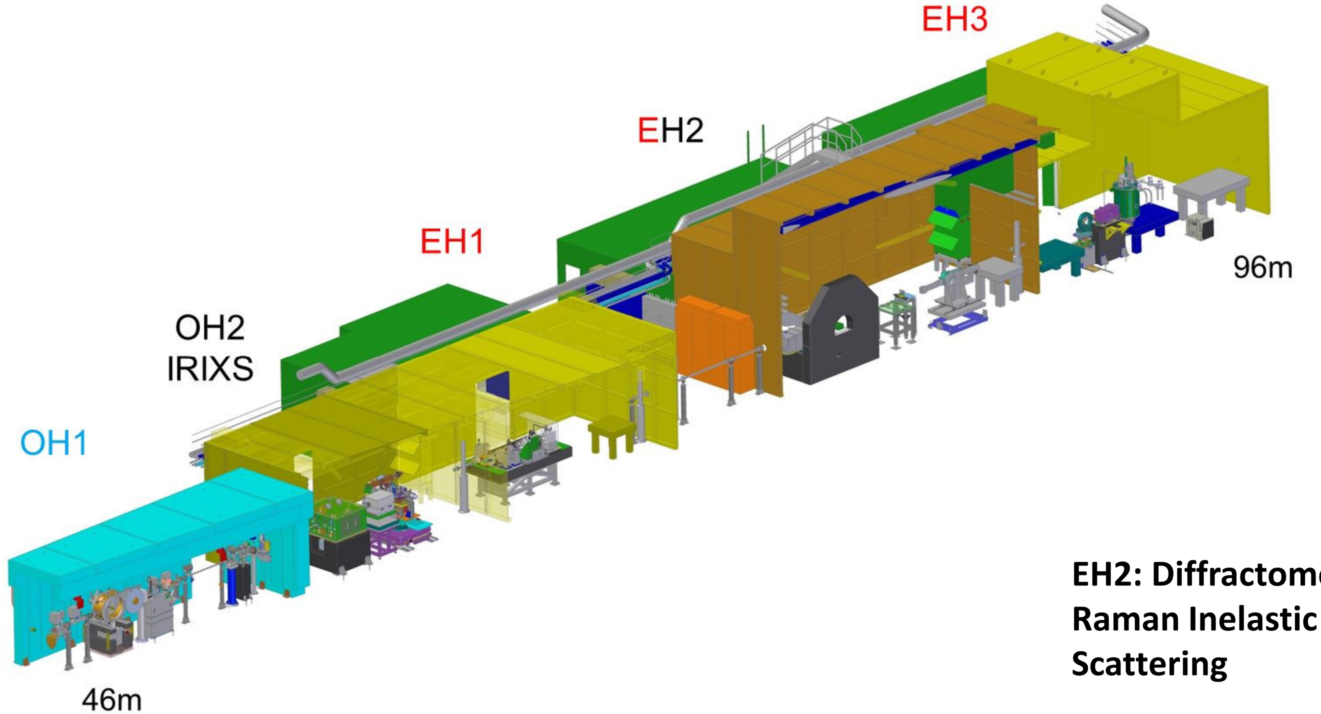


Summerstudent Lectures 2018
Introduction to Photon Science
Part 2

Rainer Gehrke FS-PS
(rainer.gehrke@desy.de)

Optics and Detectors

PETRA III Beamline Layout Example: P01

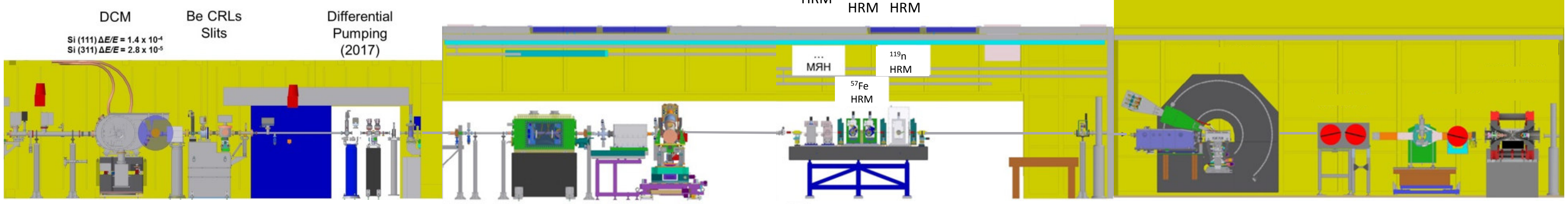


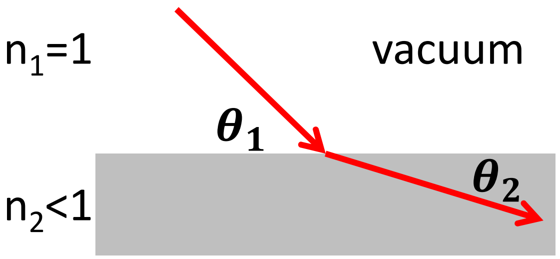
EH2: Diffractometer for Raman Inelastic X-Ray Scattering

EH1: Nuclear Resonant Scattering

OH1: High heat load optics

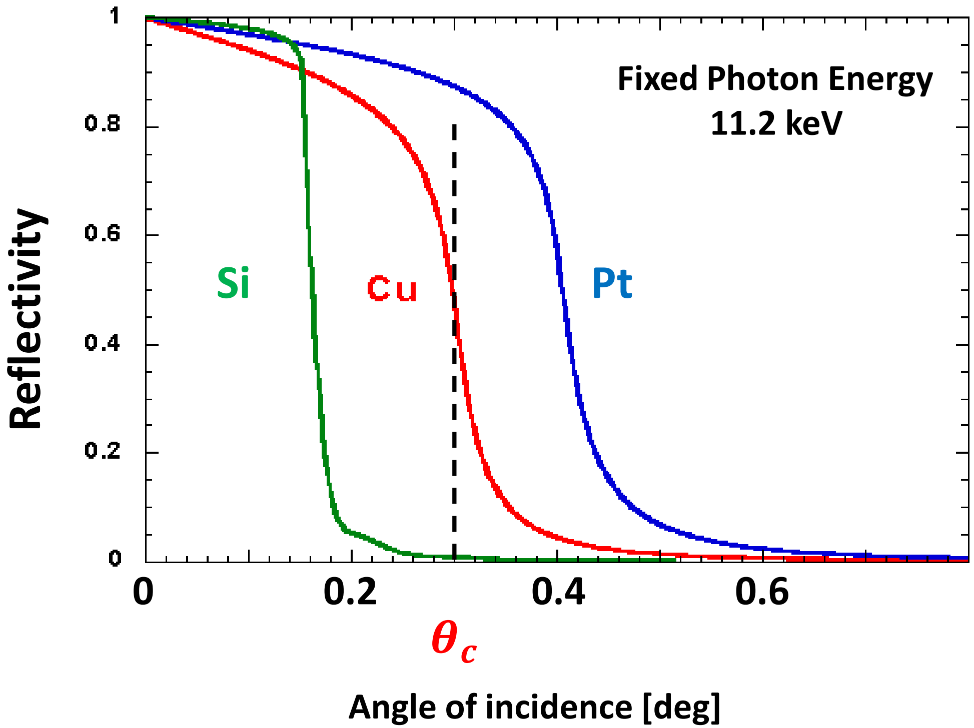
OH2: Resonant Inelastic X-Ray Scattering

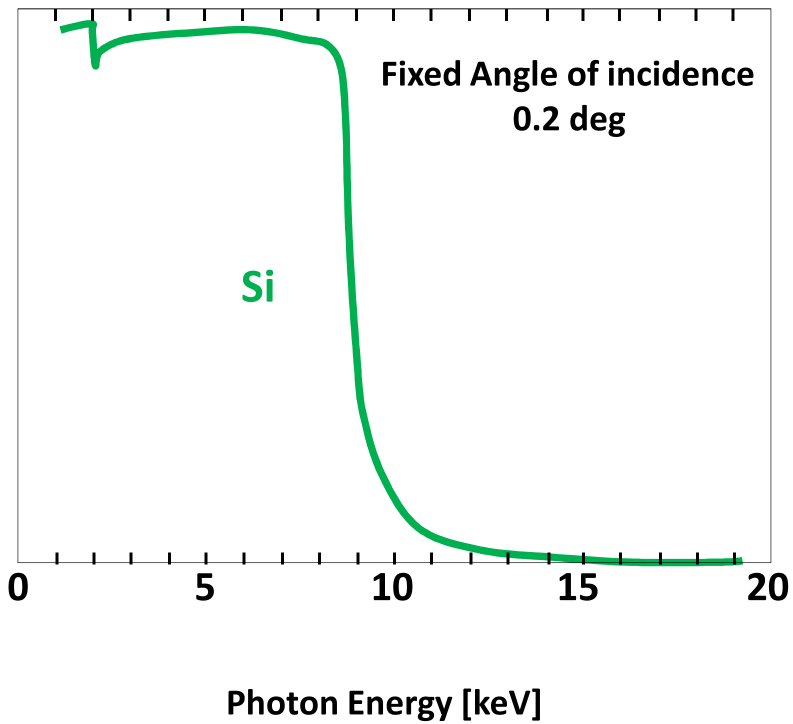




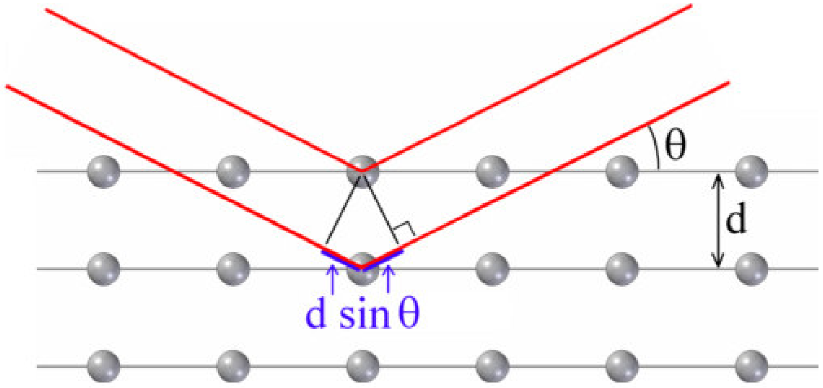
Snell's Law:

$$\frac{\sin\left(\frac{\pi}{2}-\theta_1\right)}{\sin\left(\frac{\pi}{2}-\theta_2\right)} = \frac{n_2}{n_1} = n_2 \xrightarrow[\boldsymbol{\theta_1 = \theta_c}]{\boldsymbol{\theta_2 = 0}} \theta_c \sim \sqrt{2(1-n_2)}$$

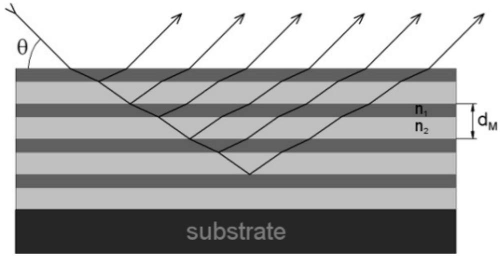




Crystal X-Ray Monochromators



Crystal

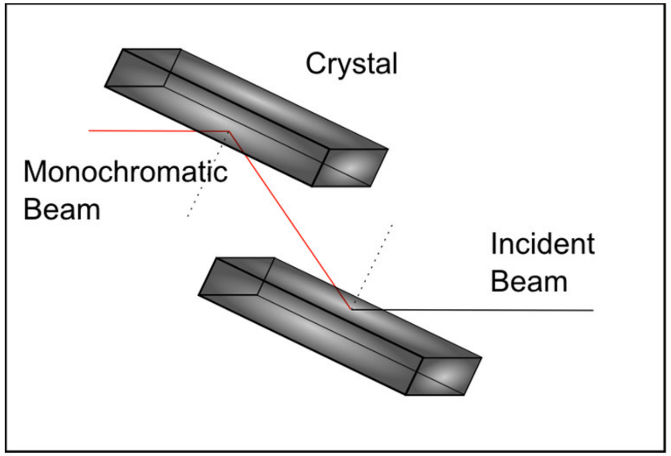


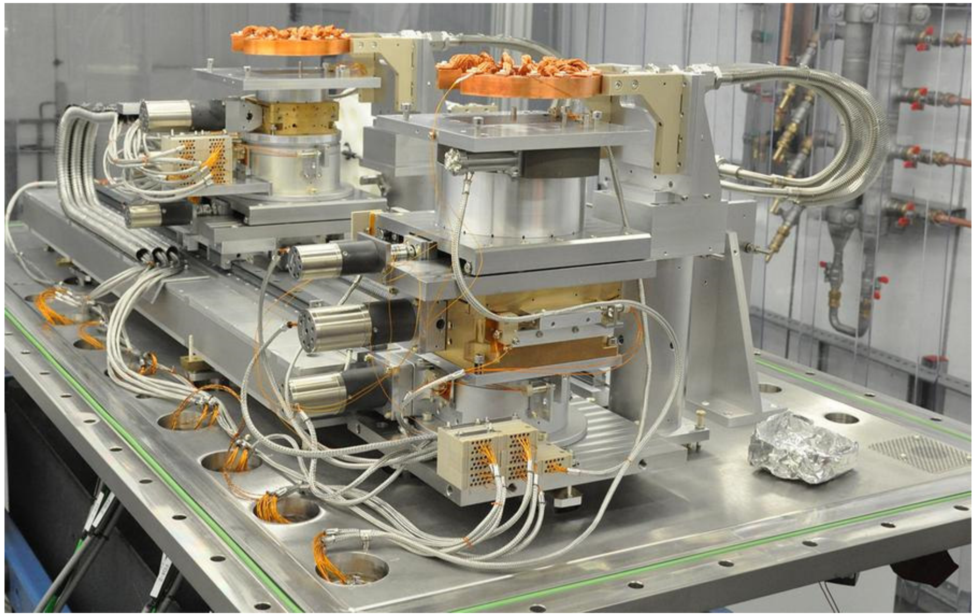
Multilayer
E.g.
SiC/W

$$n \cdot \lambda = \frac{2d}{n} \cdot \sin\theta \qquad , n \epsilon N$$

Fixed exit double crystal monochromator arrangement

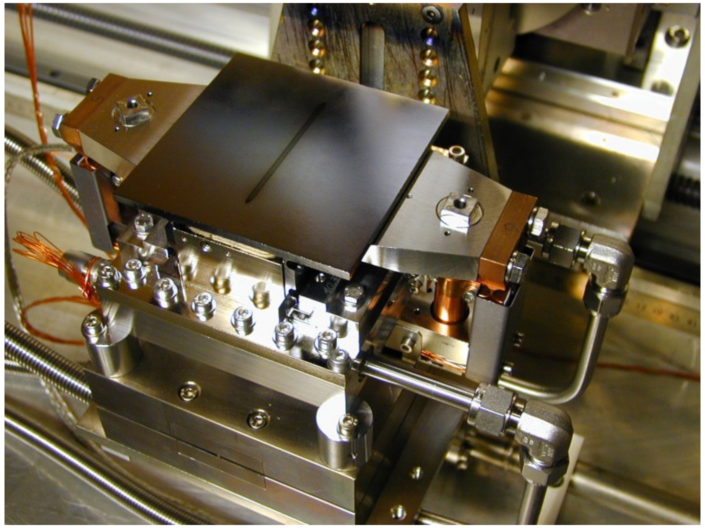
Example:
Silicon (111)
Energy resolution
$\Delta E/E \approx 10^{-4}$





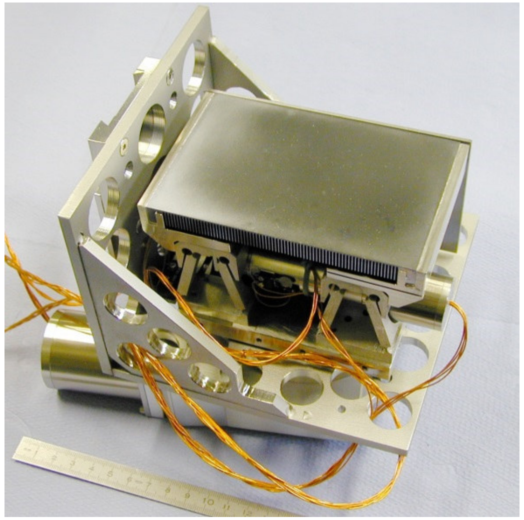
Cryogenically cooled double Laue monochromator at a PETRA III beamline (vacuum vessel removed).

Beamline Components: Monochromators



Torii: adaptable high heat load monochromator at W1, W2, BW1, BW2, BW4
MPG-BW6
PSI-Material Science
Maxlab-Material-Science-I811 (licensed to ACCEL)

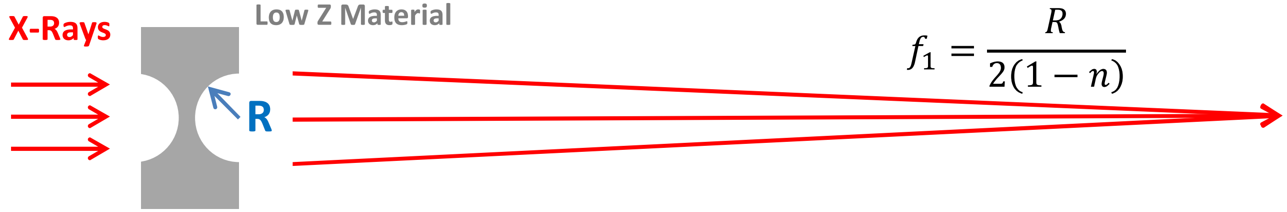
H. Schulte-Schrepping, G. Materlik, J. Heuer, Th. Teichmann, „Monochromatorkristall-Einrichtung für Synchrotronstrahlung“, Patent Nr. 4425594



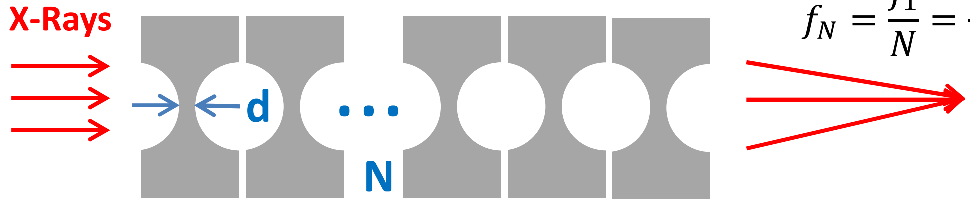
Sagittal bender adapted from ESRF design. Si-111,220, and 311 assembly available for high energy electron spectroscopy at BW2

Diamond crystal and holder at the PETRA-II undulator beamline. Attached to the water-cooled heat exchanger ΔT=5K measured at the crystal support

Compound Refractive X-Ray Lenses



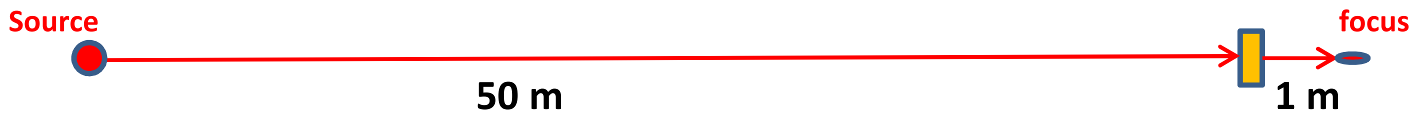
$$f_1 = \frac{R}{2(1-n)}$$



$$f_N = \frac{f_1}{N} = \frac{R}{2N(1-n)}$$

Example:
Material: Beryllium
R = 0.25 mm
f_N = 1 m
d = 20 µm

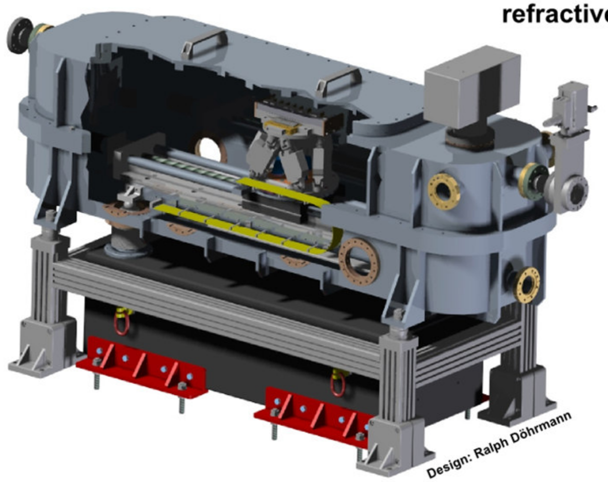
E [keV]	1-n [10^{-6}]	N
8	5.26	24
20	0.85	147
40	0.21	587



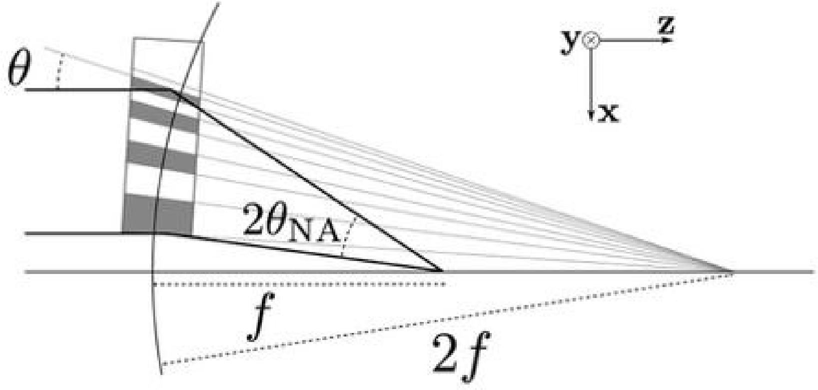
Source demagnification 1:50

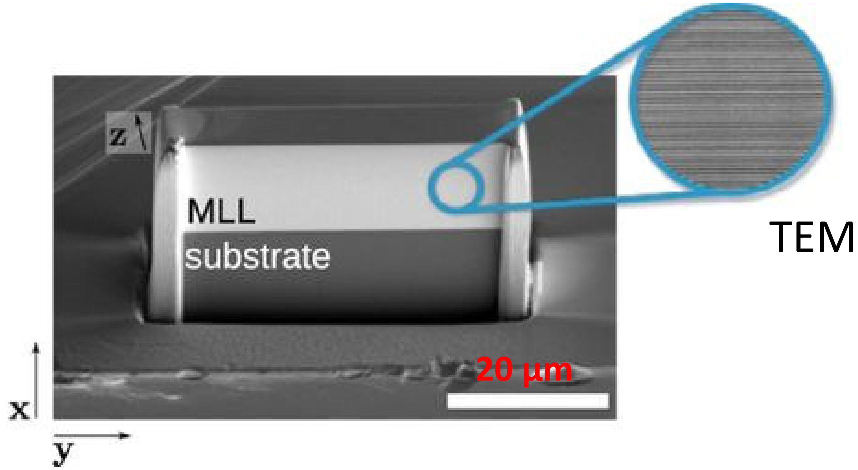
Stack of 2D Beryllium lenses

Housing for beryllium compound refractive lenses



1D Multilayer Laue Lenses



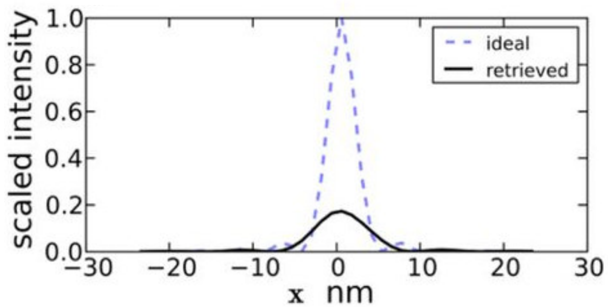


Volume Zone Plate: Simultaneously follow zone-plate condition and obey bragg‘s law

Magnetron sputtering of 2750 SiC/W bi-layers on substrate (total height 17.5 µm)
Layer thickness follows zone-plate condition for target wavelength and focal length
Layer gradient (controlled by shadowing mask) follows Bragg condition
Period variation 15.9 nm to 3.7 nm
Cutting a Slice perpendicular to z-direction (thickness 6.5 µm) using focused ion beam. Width 40 µm

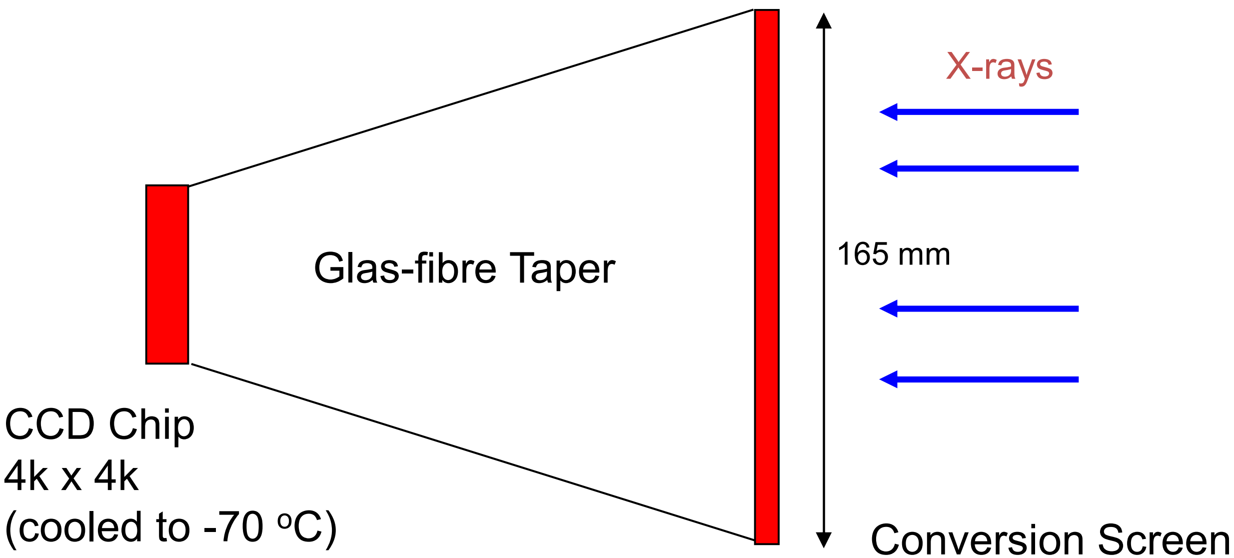
Design parameters: focal length 1.2 mm at 17 keV
➔ focal spot size 5 nm

Recovered spot intensity profile: 8.2 nm @ 22 keV

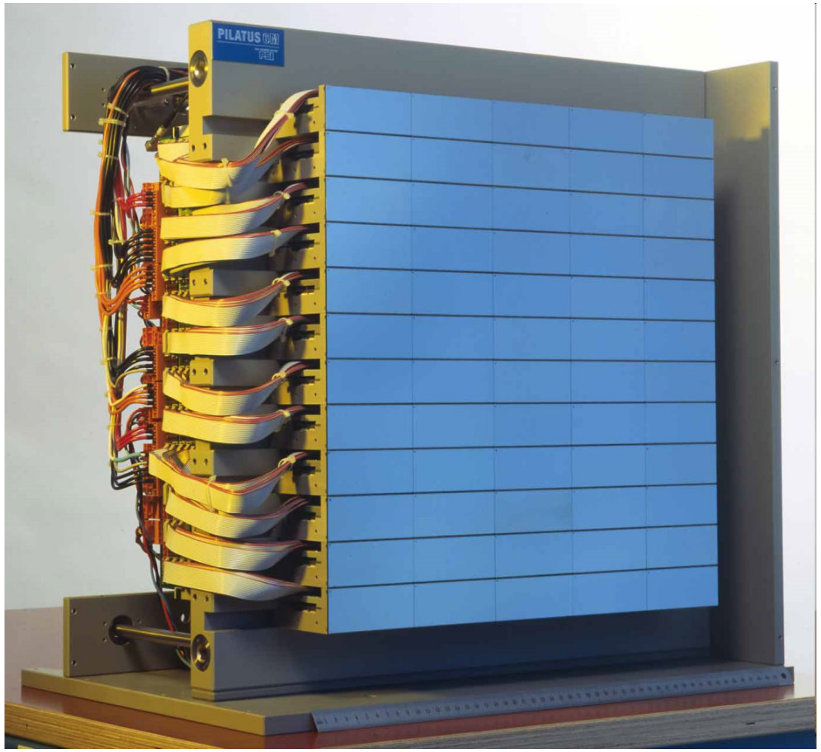


Beamline Components: Detectors

Integrating pixel detector
Readout time 2.5 s
Dark current 0.01 e^-/pixel/s
Readout noise 10 per pixel
Dynamic range 10^4
(limited by dark current and
pixel saturation)



Beamline Components: Detectors



Pilatus 6M

2-D Hybrid Pixel Array

Single Chip: 60 x 97 pixel (pixel size 0.17 mm)
Each pixel with preamp, treshhold adjustment, and 20-bit counter (count rate 1.5 MHz)

Single Module: 8 x 2 Chips
Parallel readout (readout time 2 ms)

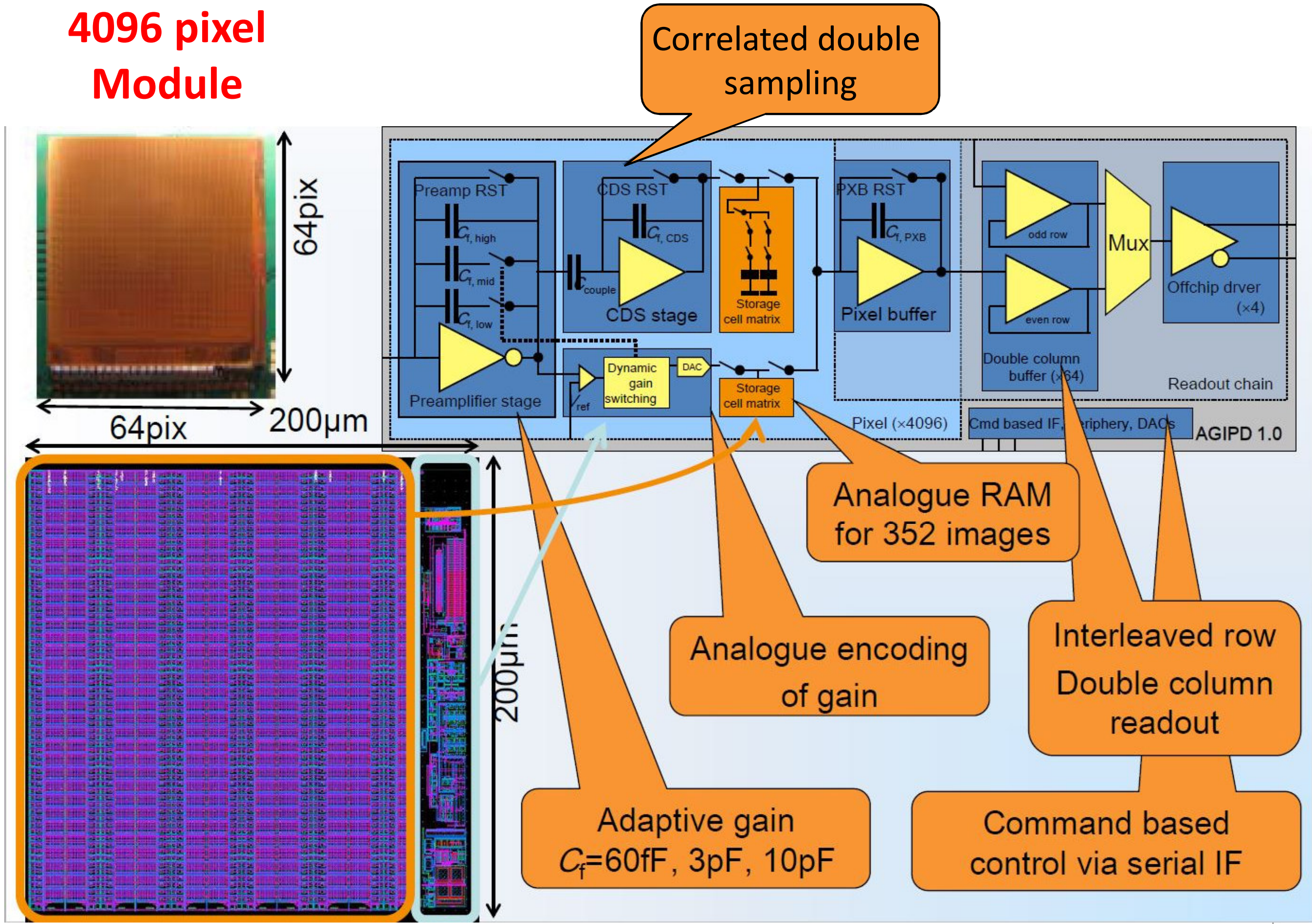
6M Detector: 12 x 5 Modules
(2463 x 2527 pixel)

Efficiency: 100% @ 8 keV, 50% @ 16 keV

Single photon counting pixels:
- No readout noise
- Discrimination of fluorescence background
- High dynamic range (10^6, limited by counter)

Adaptive Gain Integrating Pixel Detector (AGIPD)

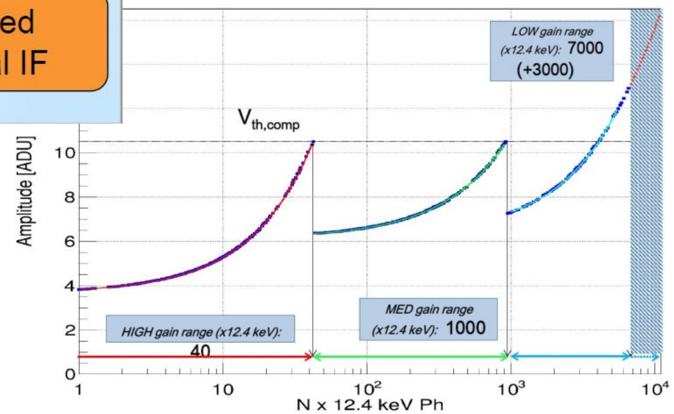
4096 pixel Module



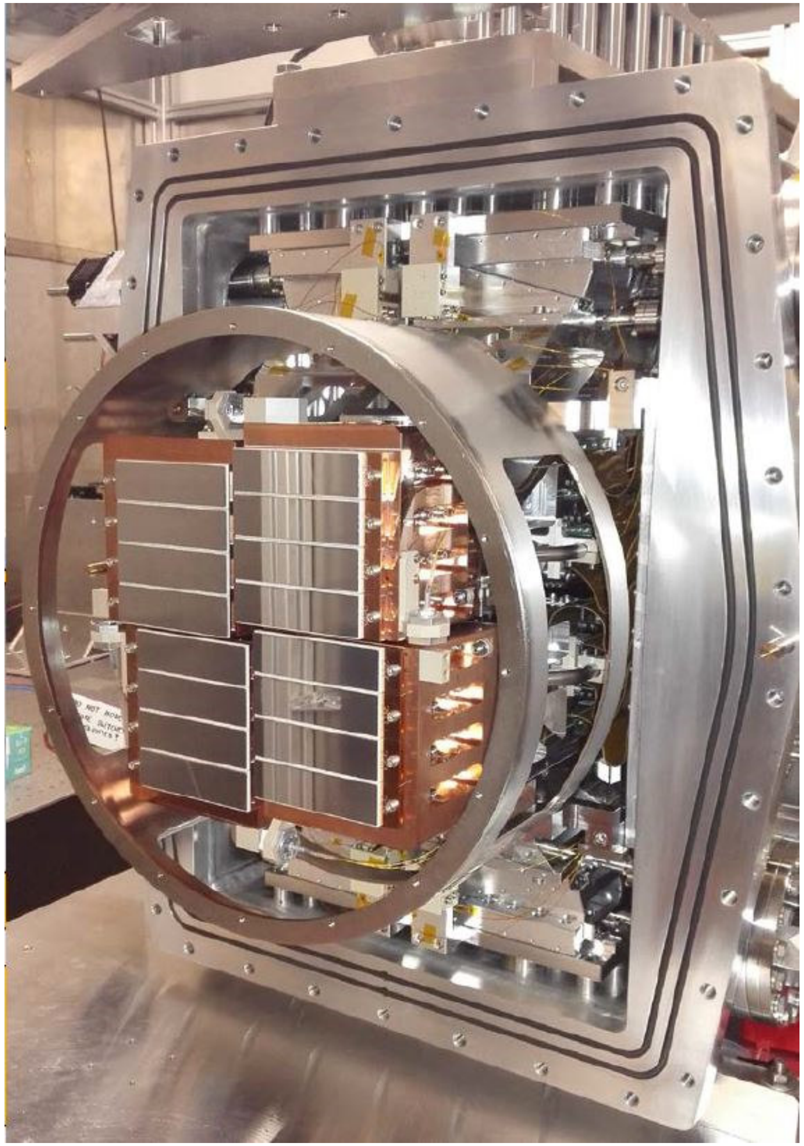
Single Pixel
size 200 x 200 µm²

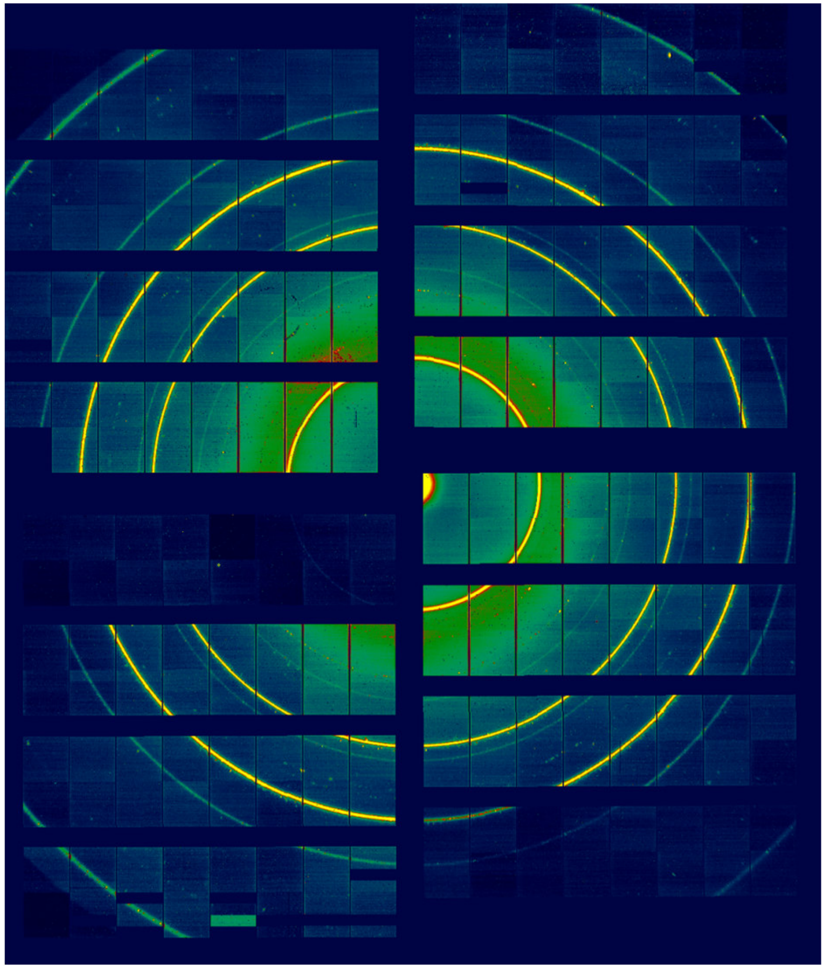
AGIPD ASIC

- 1 to 10^4 photons/pixel and pulse (three gain stages)
- at 12 keV
- 4.5 MHz frame rate
- Capable to store 352 frames Prior to readout



The AGIPD



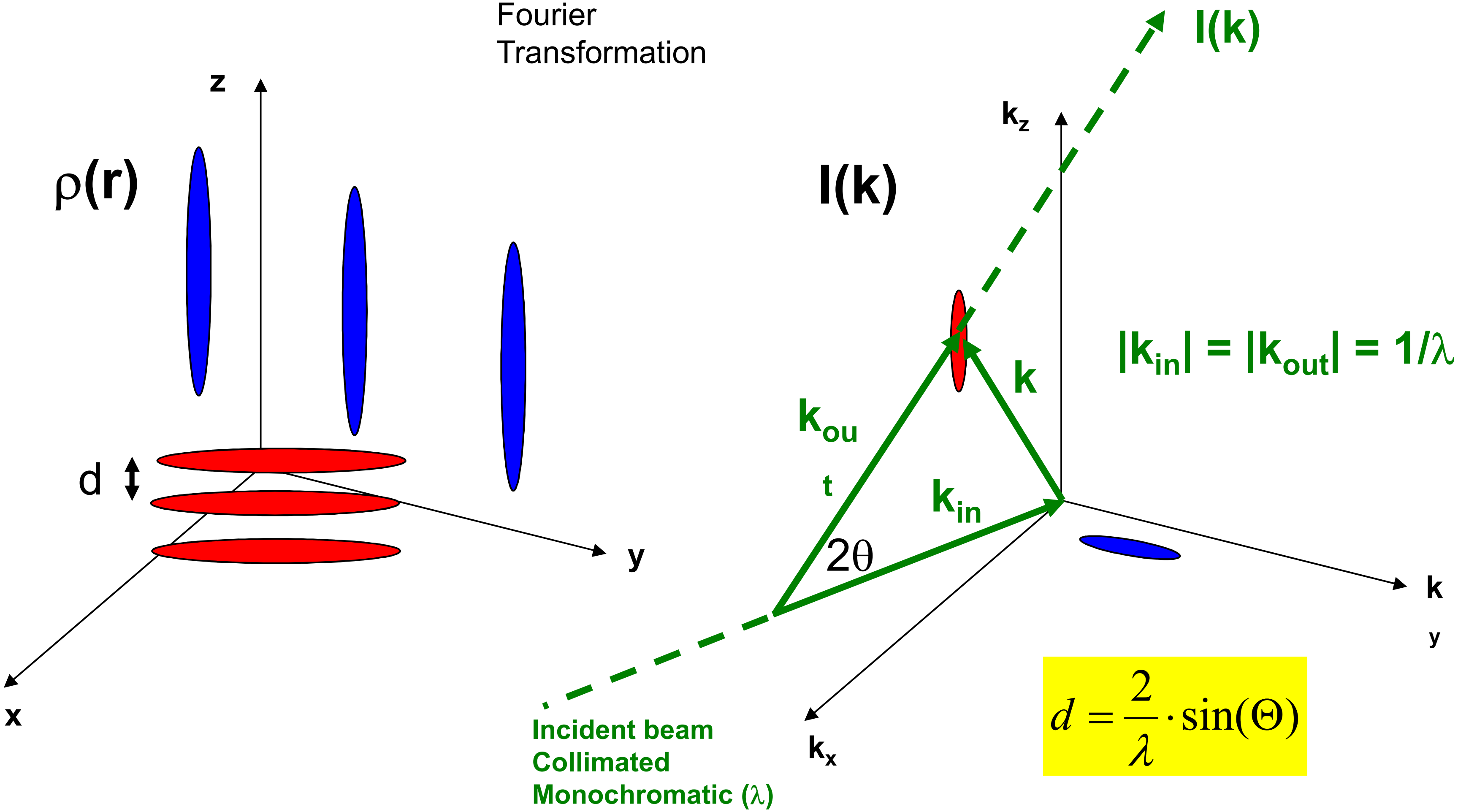


Four movable Quadrants, each with four stripes of
2 x 8 modules in each stripe → 1024 x 2024 pixel (1 Mpix)
Housing under vacuum
Optimize coverage of scattering plane, guide primary beam alongside the sensitive area

Scientific Experiments
Scattering & Diffraction

Basics of an Elastic Scattering Experiment

$$\rho(\mathbf{r}) \xrightarrow{\text{Fourier Transformation}} I(\mathbf{k}) = I_e \cdot |F[\rho(\mathbf{r})]|^2$$

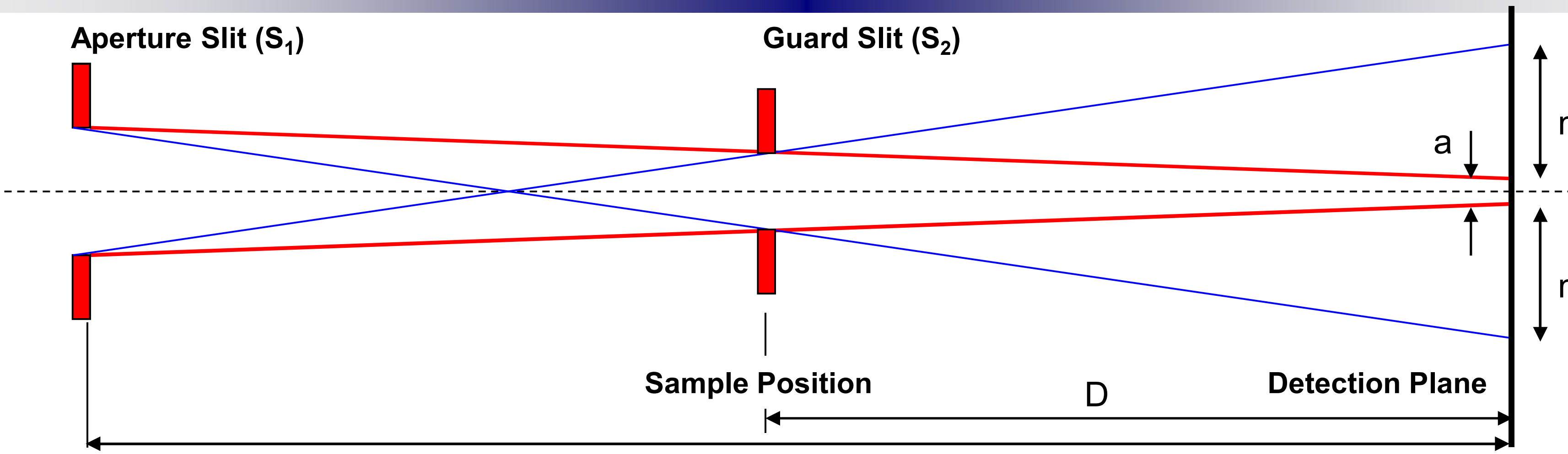


Real space

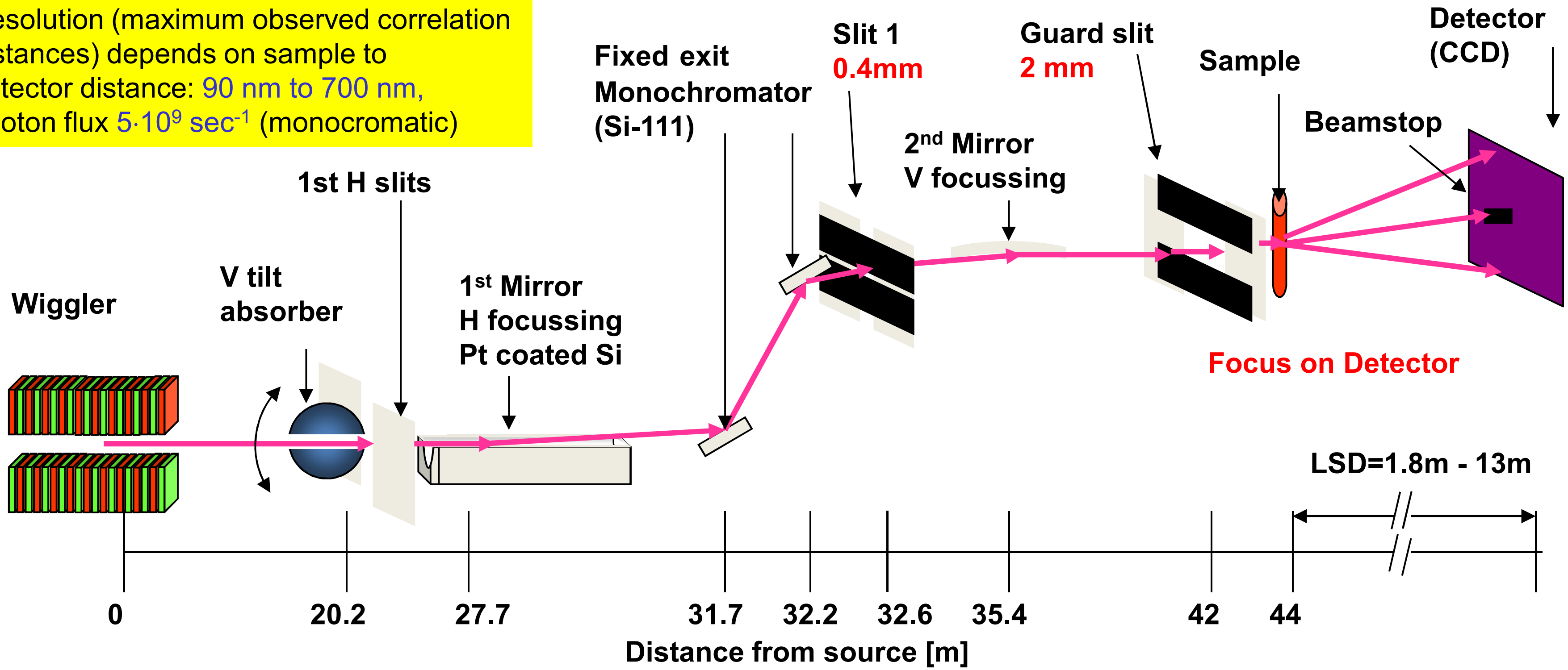
Reciprocal space

No direct back-transformation possible, because phase is lost!

Layout of a SAXS Instrument

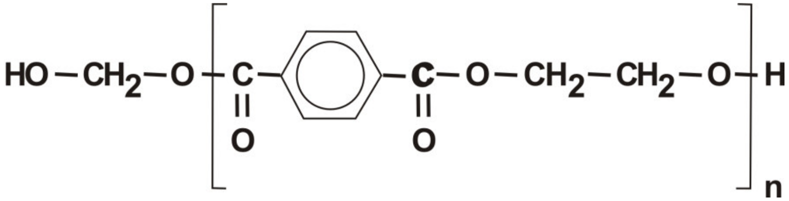


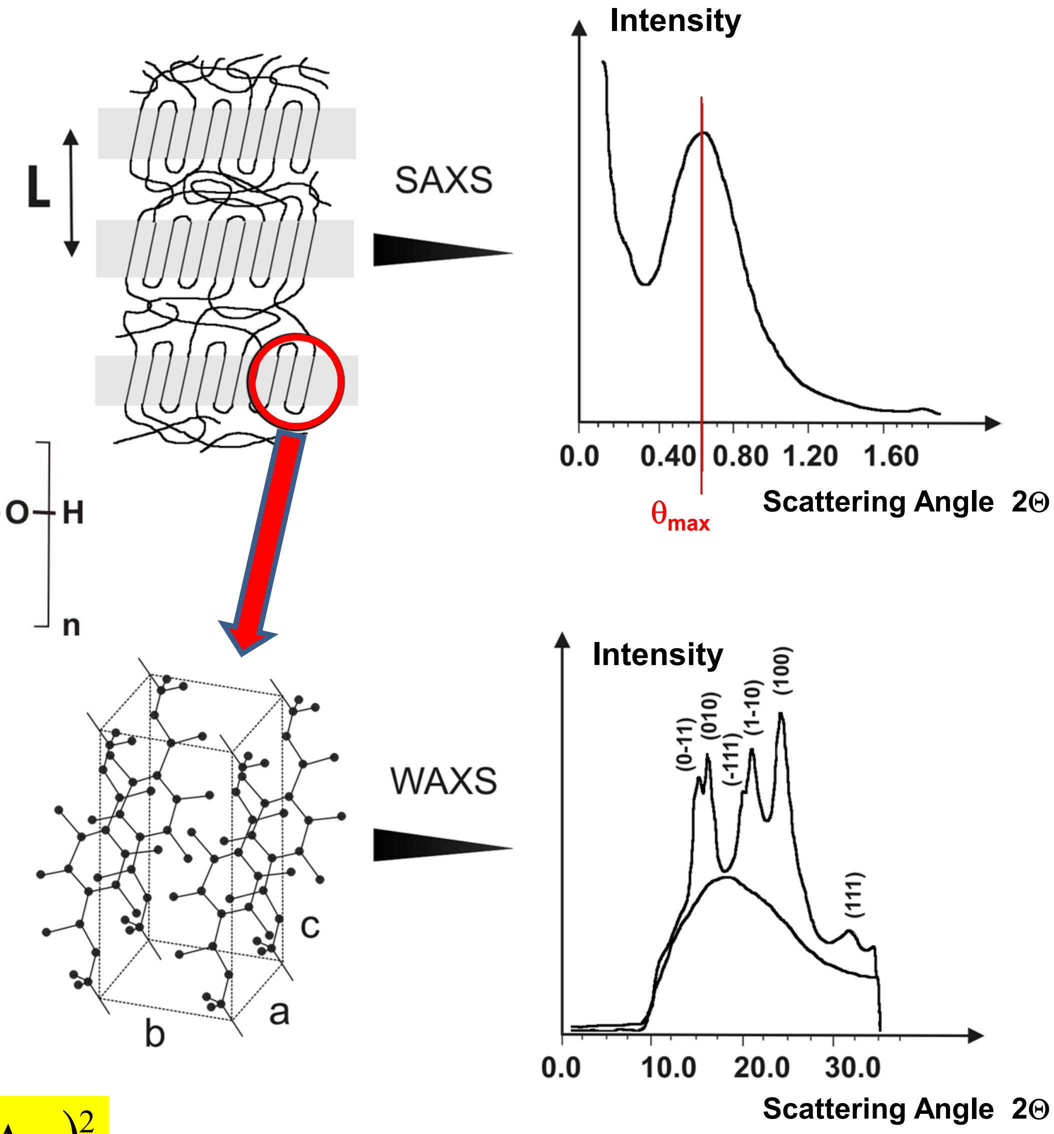
Resolution (maximum observed correlation distances) depends on sample to detector distance: 90 nm to 700 nm, photon flux $5 \cdot 10^9$ sec^{-1} (monocromatic)



SAXS/WAXS at lamellar polymer systems

Example:
Polyethylenterephtalate (PET)





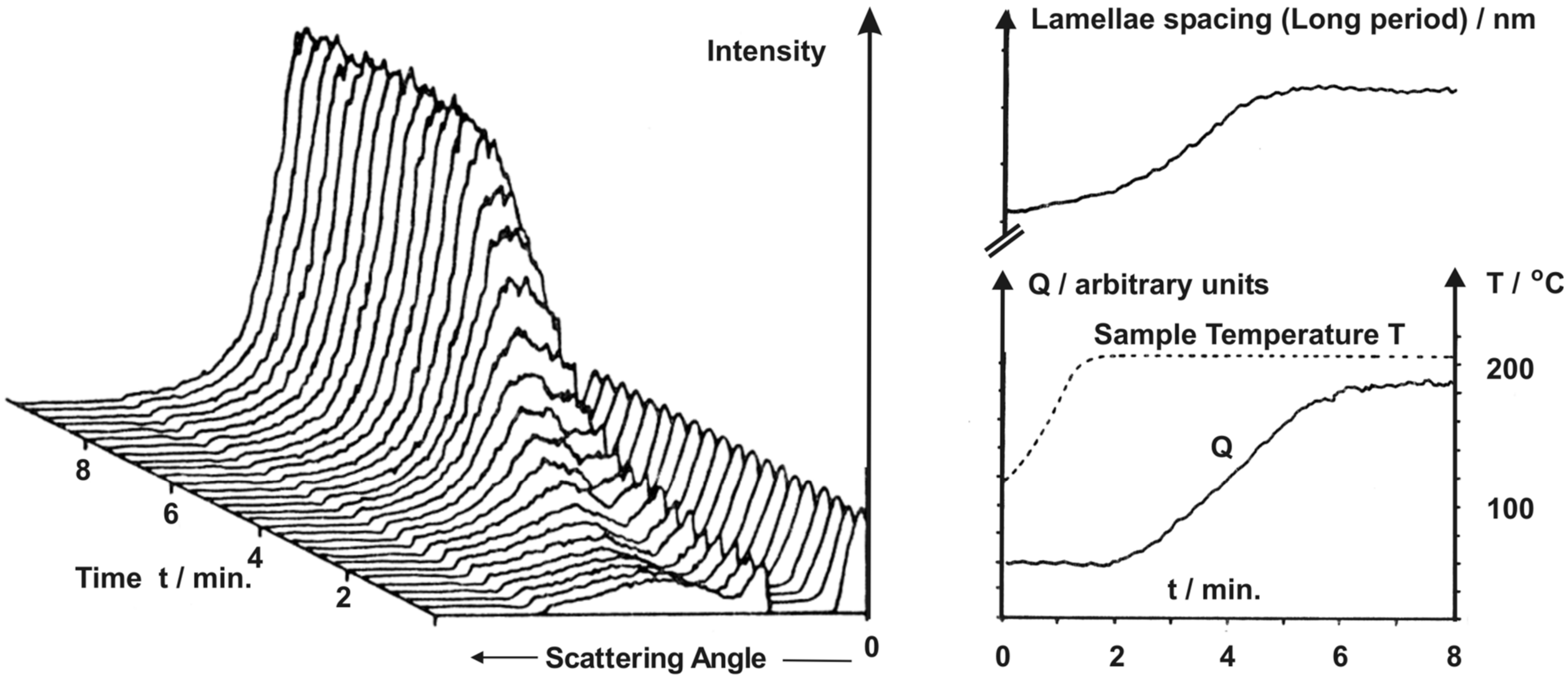
$$q_{\max} = \frac{4\pi}{\lambda} \cdot \sin(\theta_{\max}) \approx \frac{4\pi}{\lambda} \cdot \theta_{\max}$$

Long Period

$$L = \frac{2\pi}{q_{\max}}$$

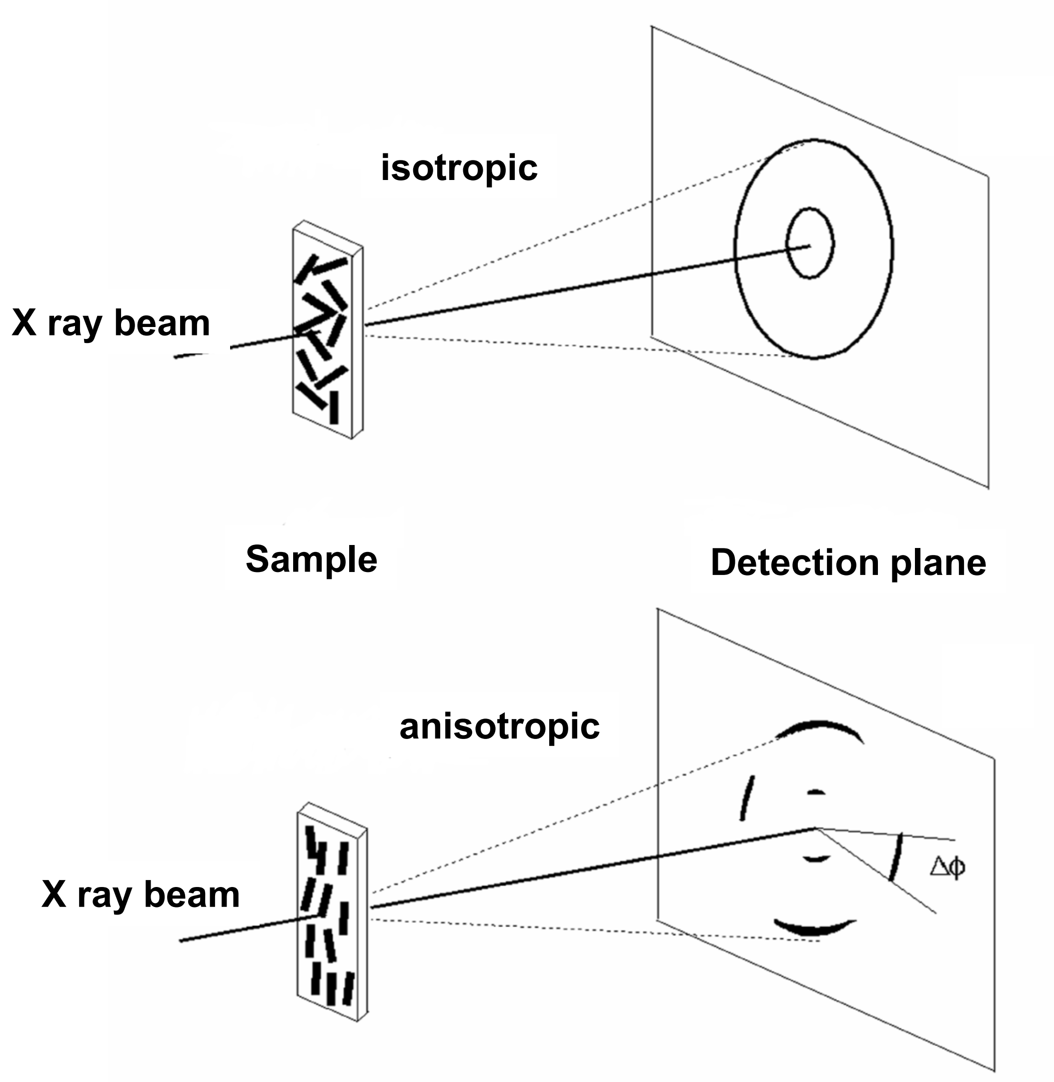
$$Q = \int I(q) q^2 dq = 2\pi^2 \cdot \Phi \cdot (1 - \Phi) \cdot (\Delta\rho)^2$$

Time resolved in-situ SAXS: Recrystallisation of PET



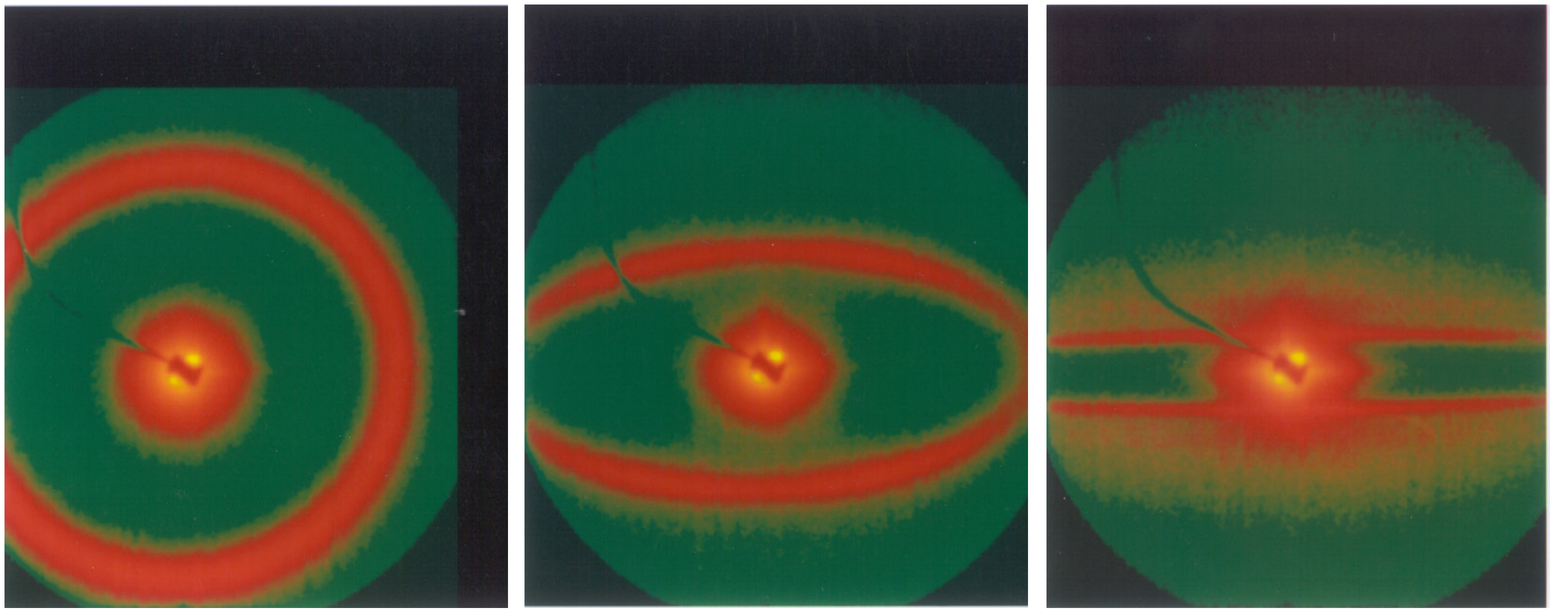
Annealing of PET, previously crystallised at T_1=130°C,
recrystallisation at T_2=230°C

Scattering of Anisotropically Oriented materials



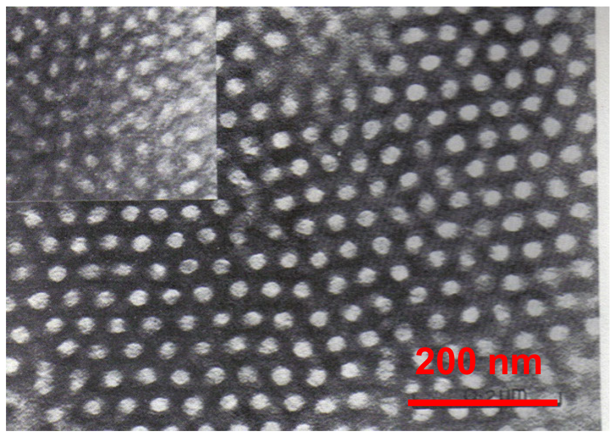
Deformation of an SBS-Triblock Copolymer (Thermoplastic Elastomer)

$$\lambda \equiv \frac{L_{final}}{L_{initial}} = 1 \qquad \lambda = 2 \qquad \lambda = 6$$



PS PB PS

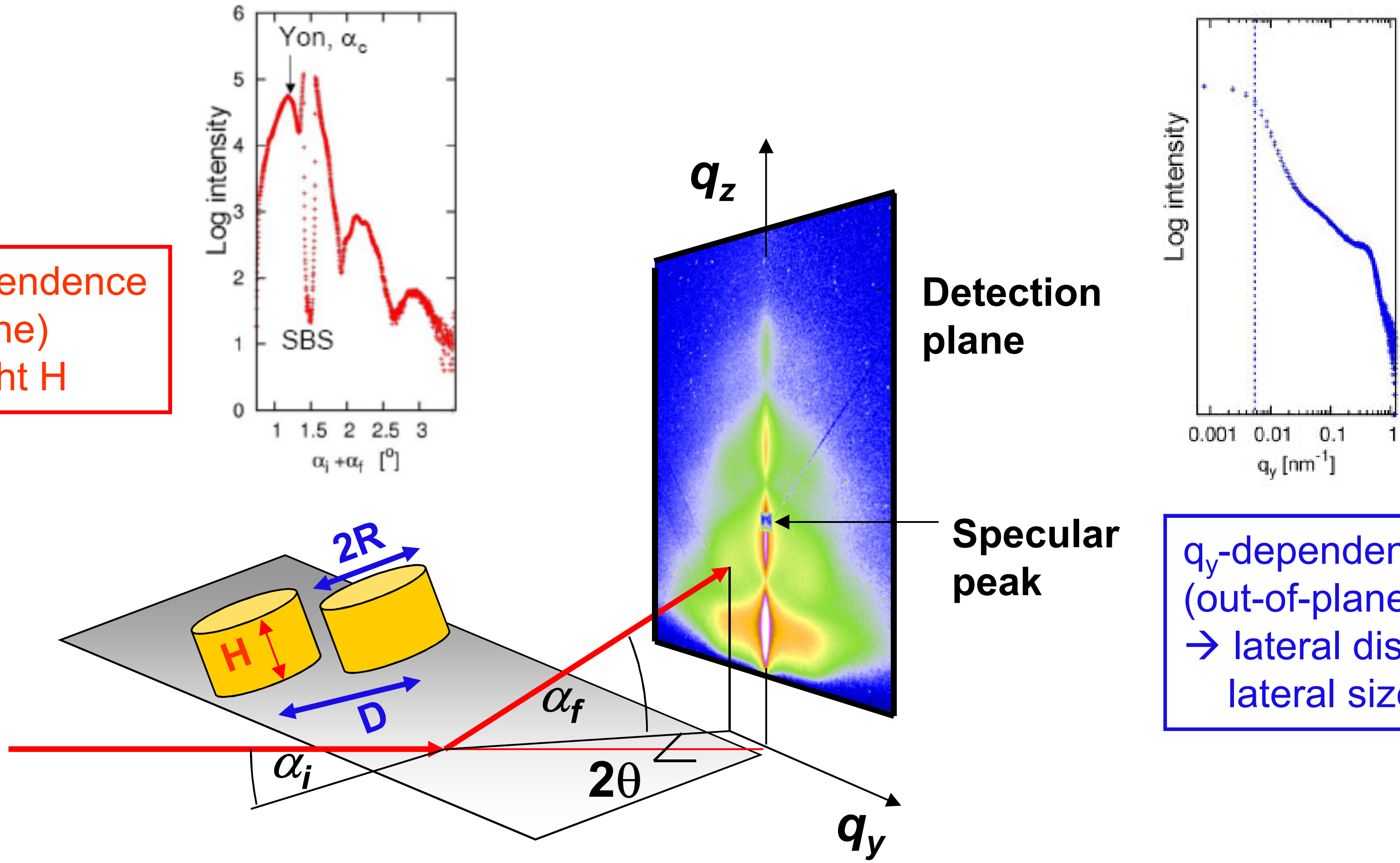
80% Polystyrene
20% Polybutadiene
→ PS cylinders in PB-Matrix



Electron micrograph

Grazing Incidence Small Angle Scattering (GISAXS)

q_z-dependence
(in-plane)
→height H

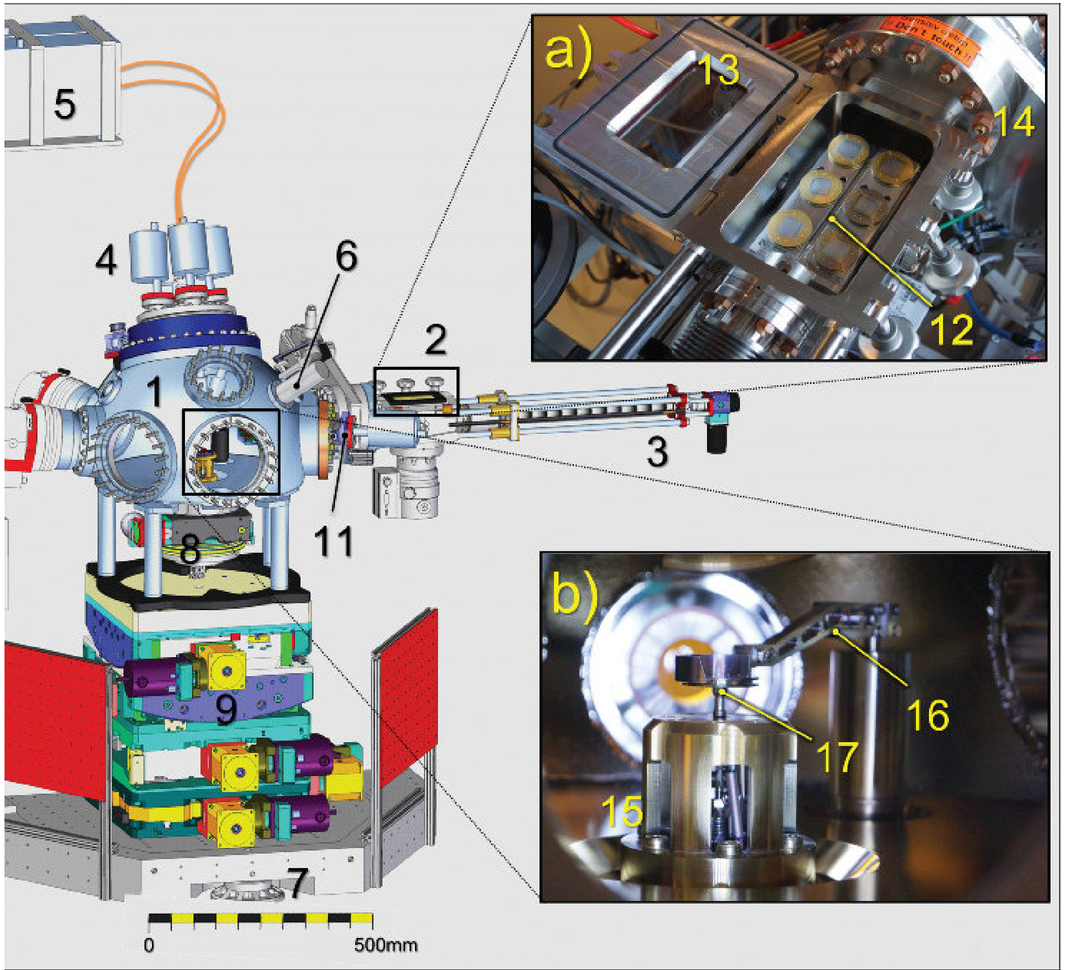


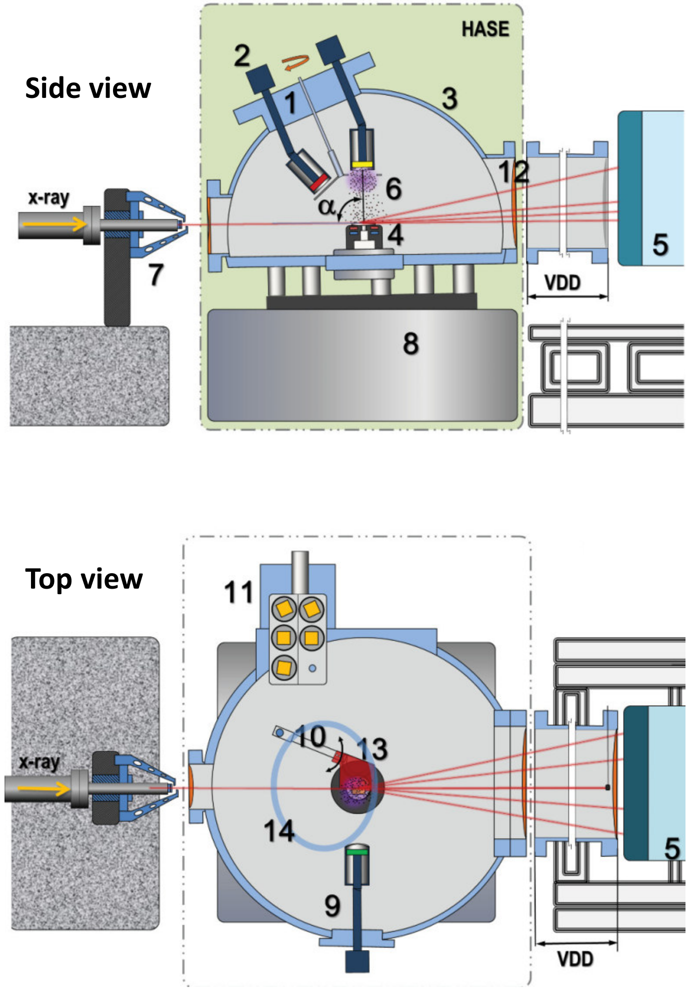
q_y-dependence
(out-of-plane)
→ lateral distance D,
lateral size R

$q_z = 2\pi/\lambda \sin(\alpha_i + \alpha_f)$
$q_y = 2\pi/\lambda \sin(2\theta)\cos(\alpha_f)$

Method only probes thin layer on surface

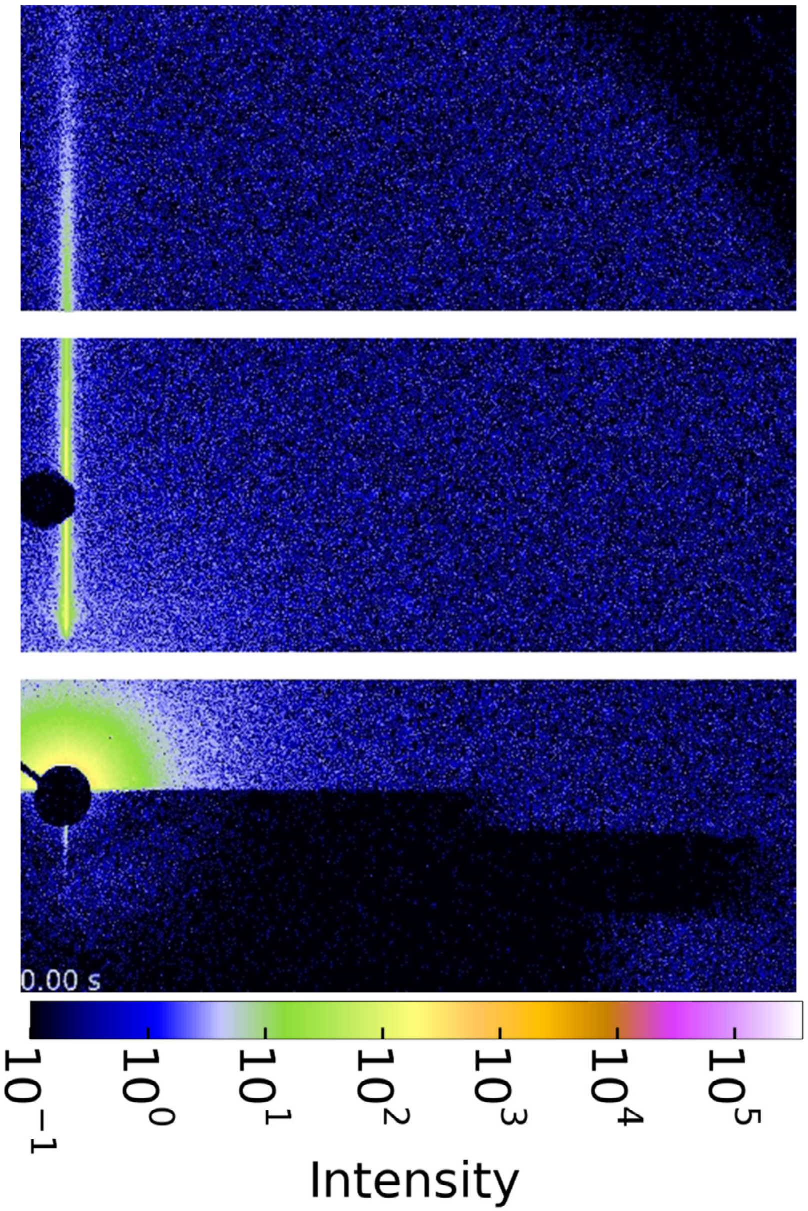
In-Situ Sputtering Equipment at PETRA III (P03)





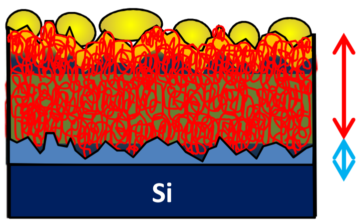
In-Situ Real-Time GISAXS Experiment

Au on PS – the kinetic movie



Nanostructural evolution *during* fabrication with subpicometer resolution

- wavelength = 0.09445 nm
- D_{SD} = 1836 mm
- PILATUS 300K: Frame rate = 10 fps
- Deposition time: 1013 s
- Deposition rate: 0.0082 nm/s
- RF; P = 100 W; p_{Ar} = 15 µbar
- Lateral sample movement



δ(Au) ≈ (8 ± 1) nm

δ(**PS**) ≈ (91 ± 2) nm

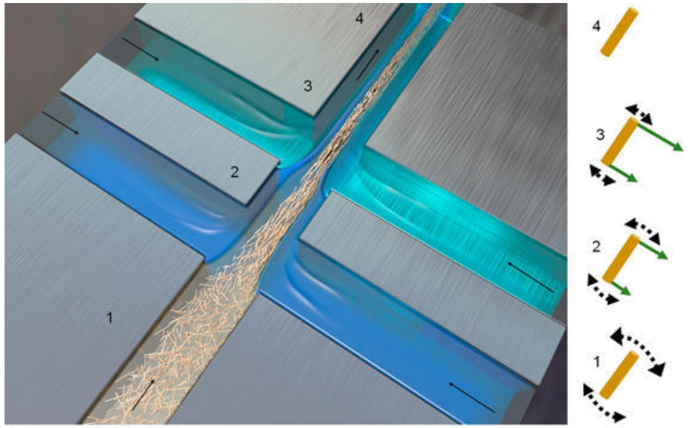
δ(**SiOx**) ≈ (6 ± 2) nm

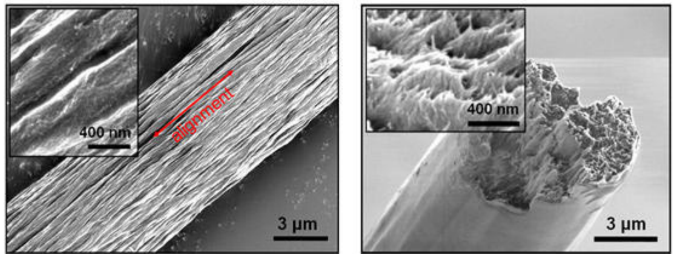
spin casted polystyrene thin film (M_w = 270 kDa) on acid cleaned silicon with correlated roughness

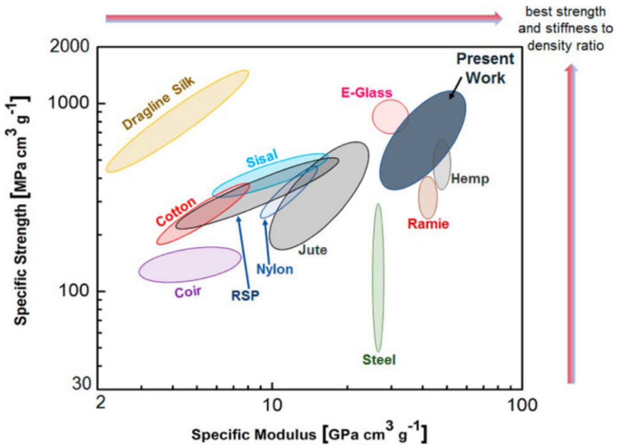
Schwartzkopf et al., ACS Appl. Mater. Interfaces 7, 13547 (2015).

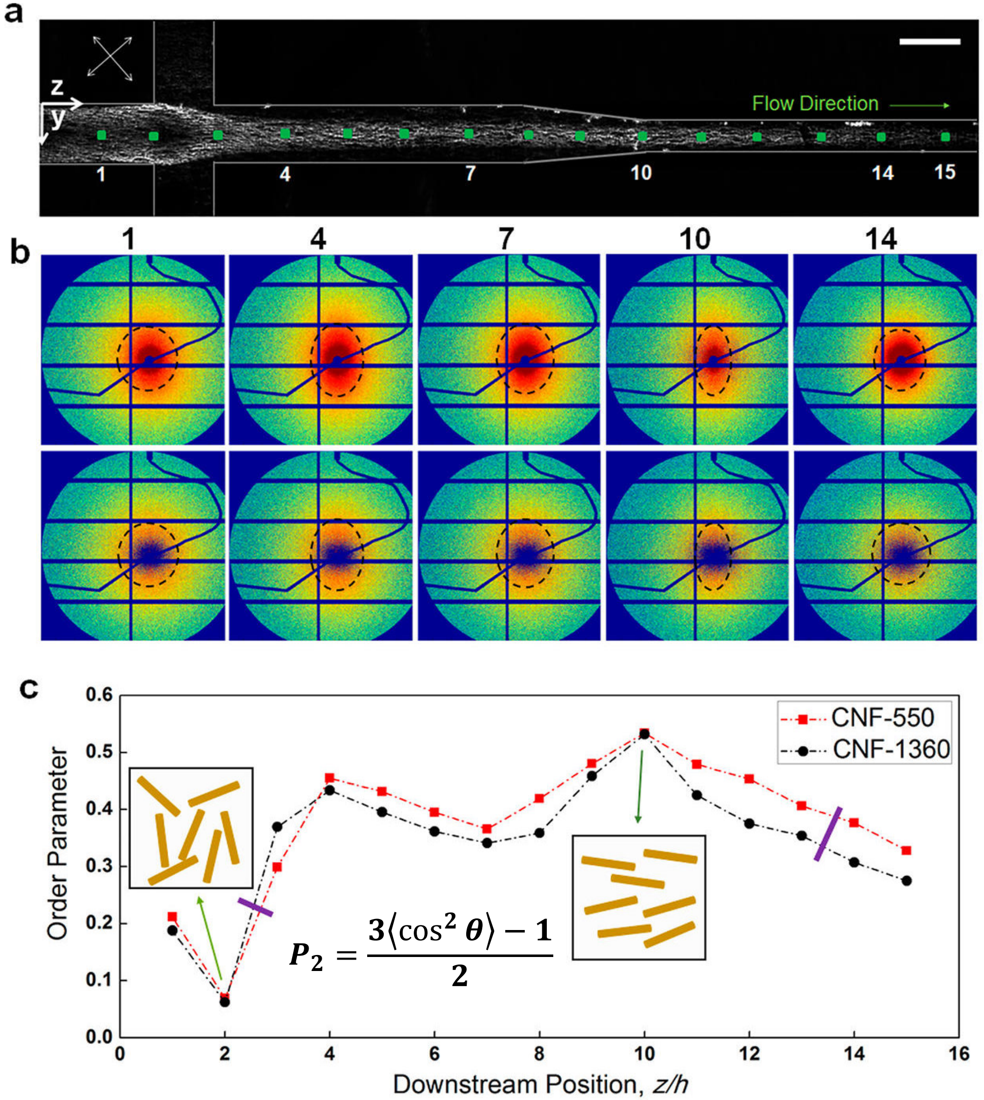
Microfluidics: Flow Assisted Assembly of Nanocellulose

Orientation of cellulose nano-fibrils by means of hydrodynamic focusing and pH reduction







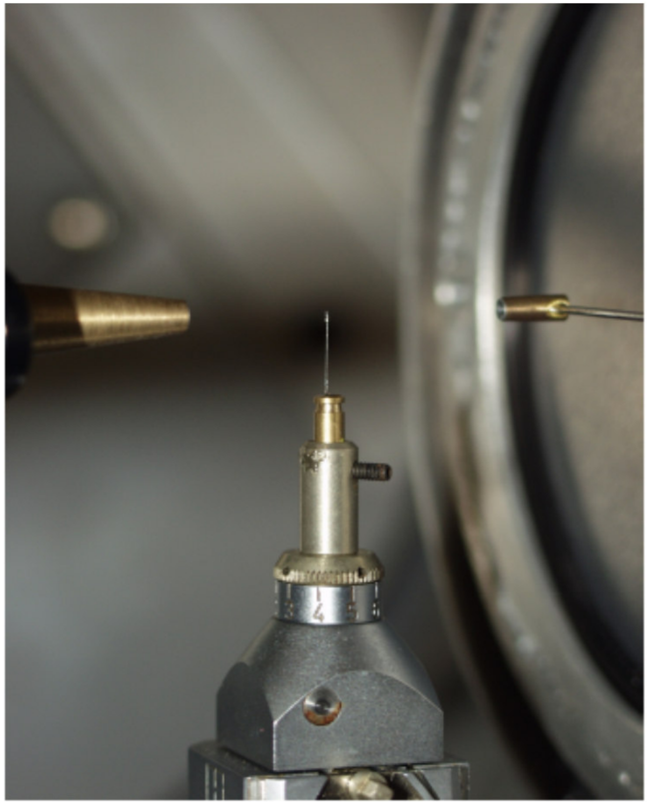


N. Mittal et. al., ACS Nano (2018)

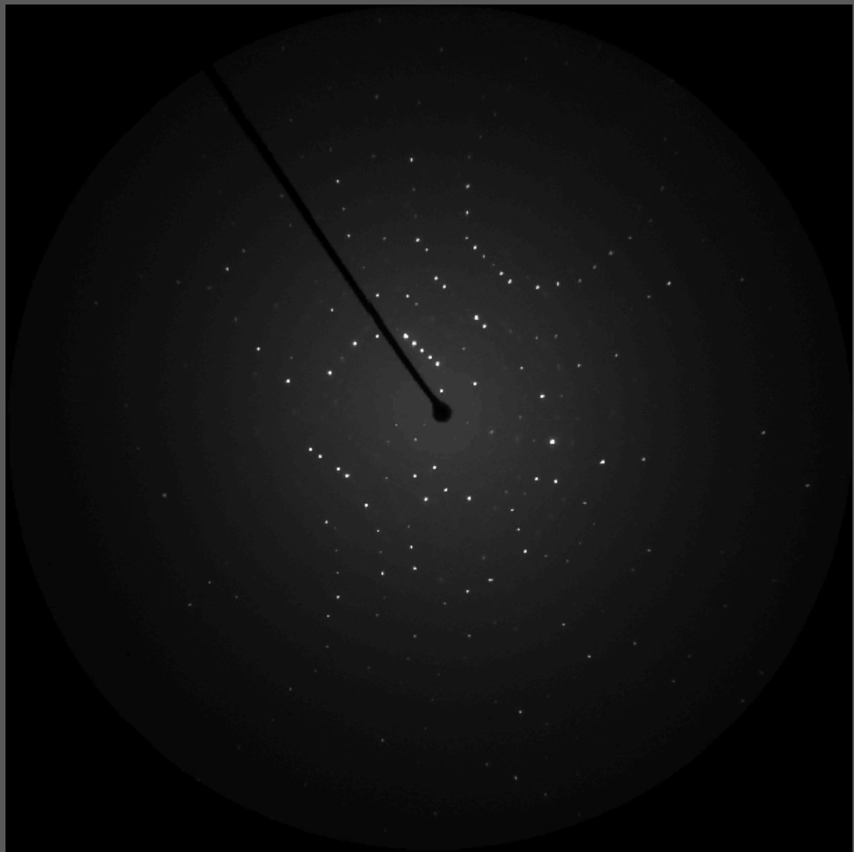
Protein Crystallography (PX)

Tiny samples
Huge unit cells
Light elements
Sensitive to radiation damage → High brilliance required
High resolution necessary

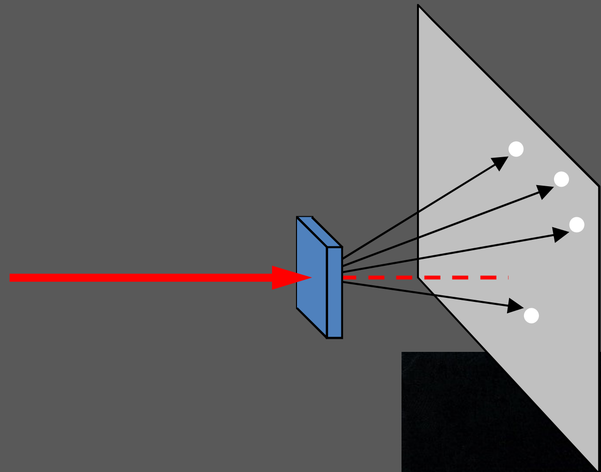
- narrow energy band
- high degree of collimation

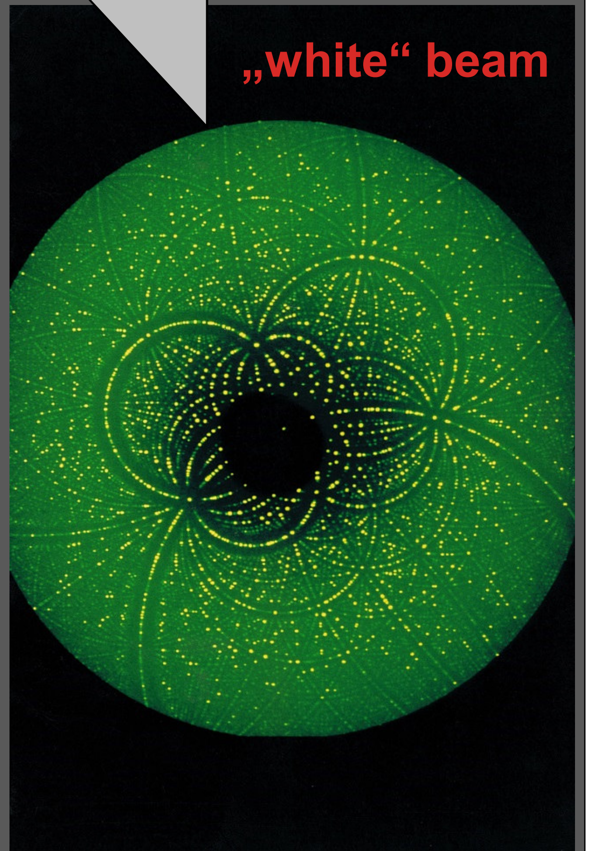



Protein Crystallography (PX)



Monochromatic beam



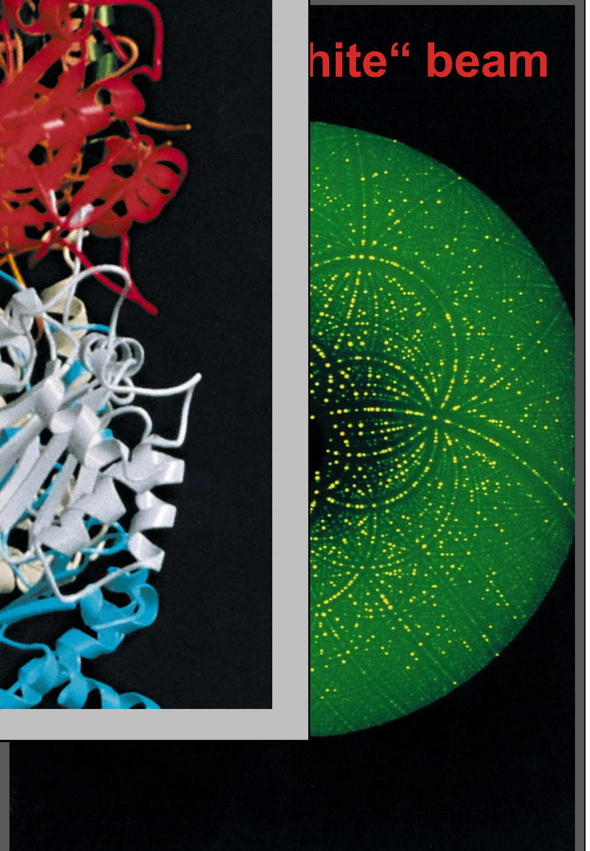


„white“ beam

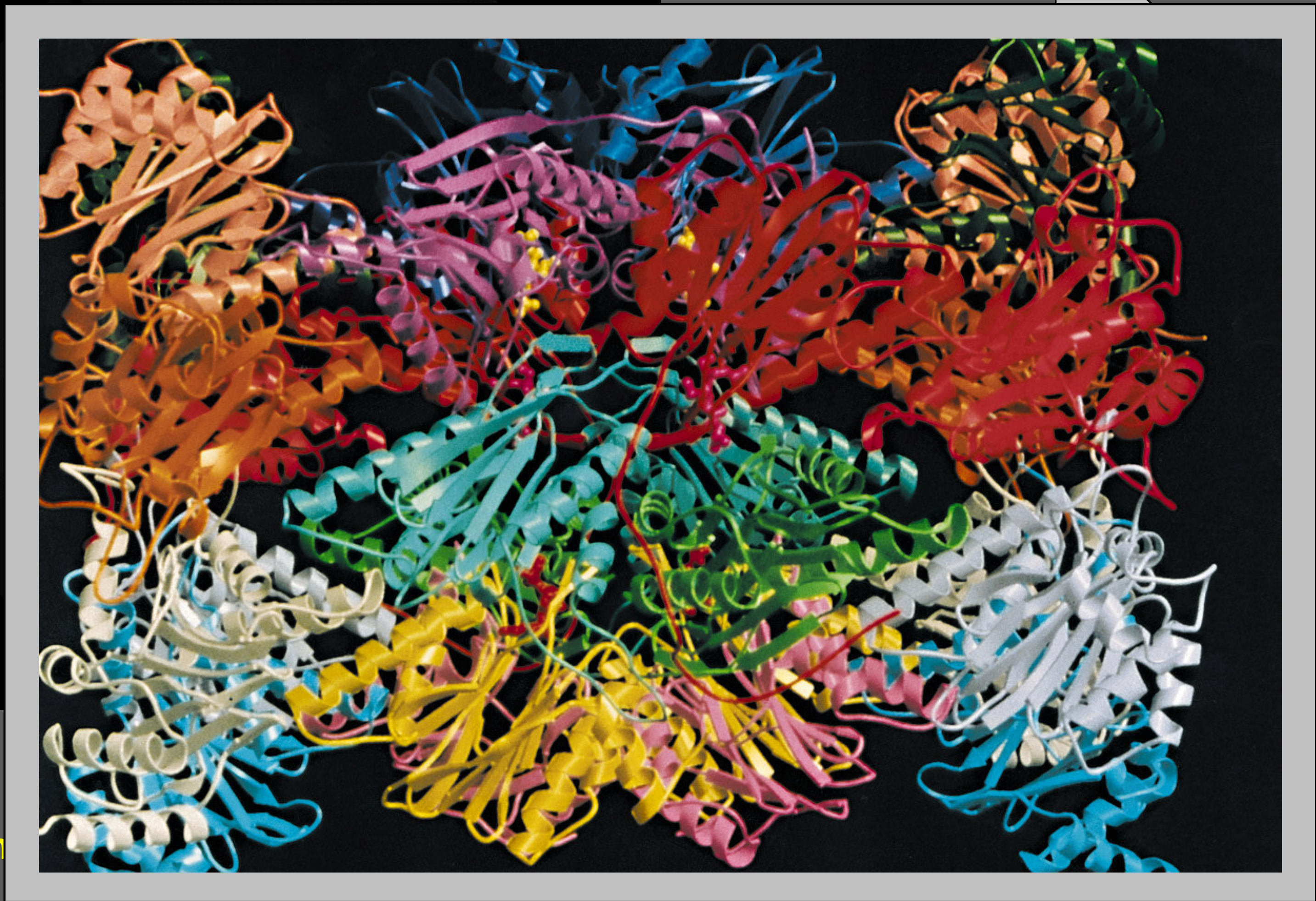
Protein crystal: Yeast Proteasome (50000 Atoms/unit cell)

Resolution 0.09 nm, mean position error 0.001 nm
Even Position of Hydrogene Atoms resolved!

Protein Crystallography (PX)



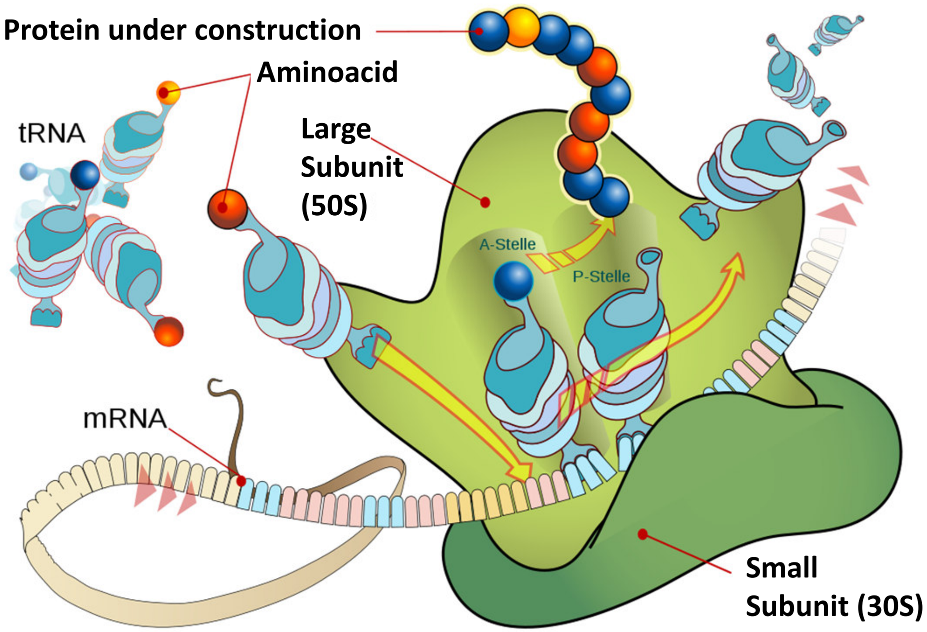
hite“ beam

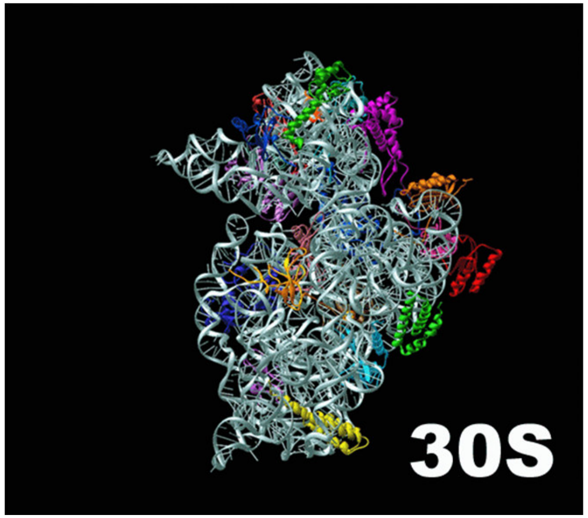


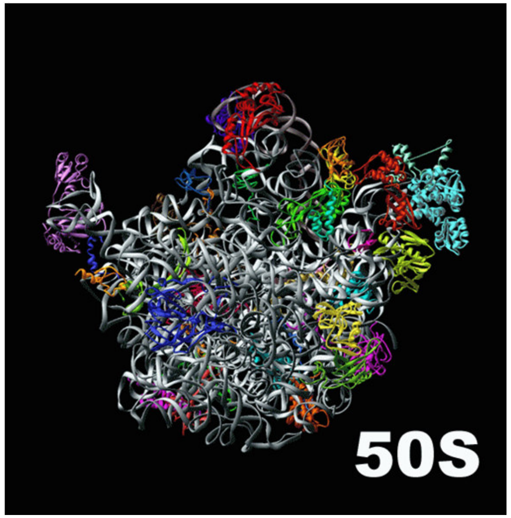
Protein

Resolution 0.09 nm, mean position error 0.001 nm
Even Position of Hydrogene Atoms resolved!

Revealing Structure and Dynamics of Ribosome





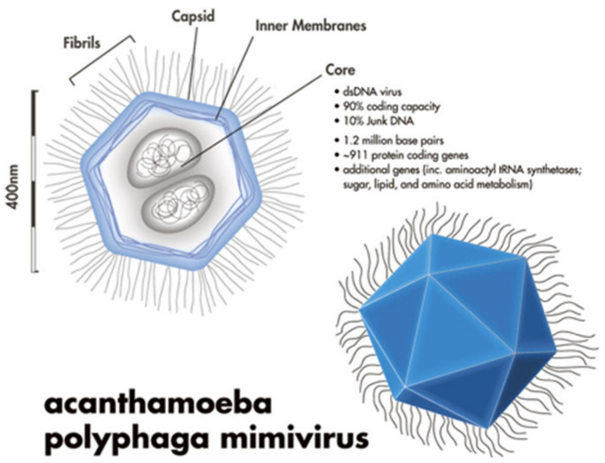


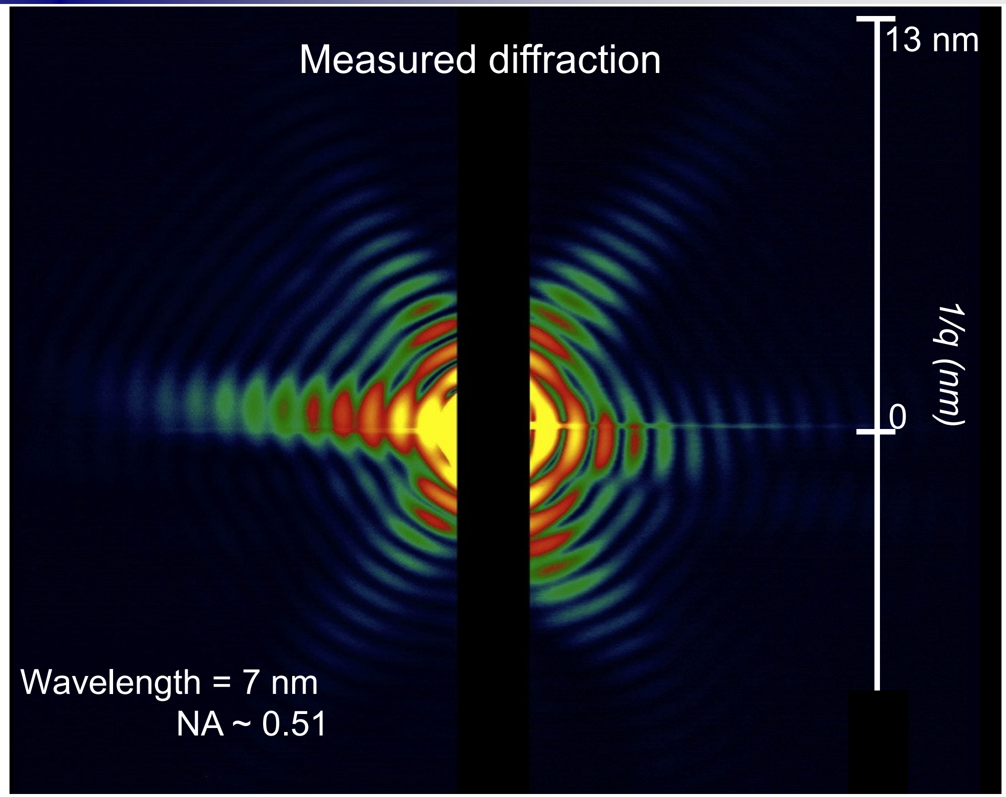
Ada Yonath:
- **Head of the MPG-work group „Structure of the Ribosome" at DESY, 1986 - 2004**
- **Nobelprize Chemistry 2009**

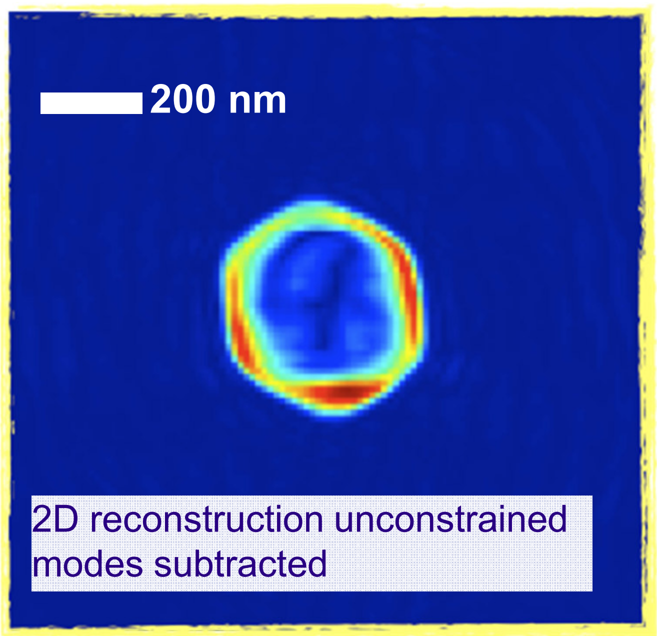
  With T. Steitz and V. Ramakrishna

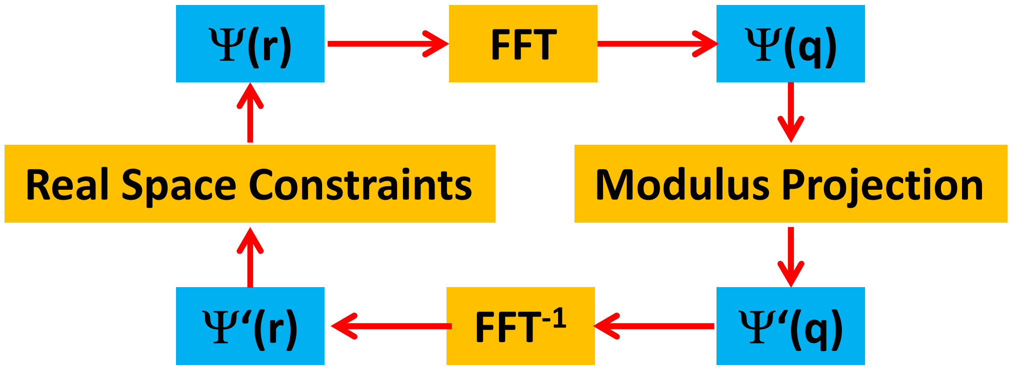
Schlünzen, ..., Yonath, Cell, 102(2000)615

Coherent Diffraction Imaging of a Mimivirus





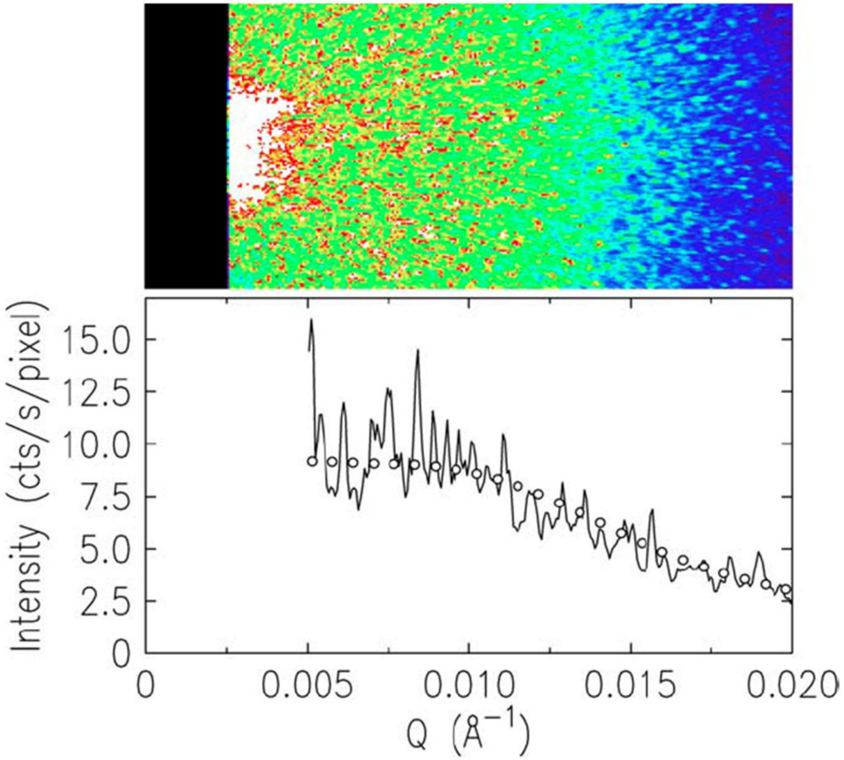




Samples: Uppsala University and CNRS, Aix-Marseille Université
FEL experiments: CFEL @ DESY, Uppsala, SLAC, MPMI

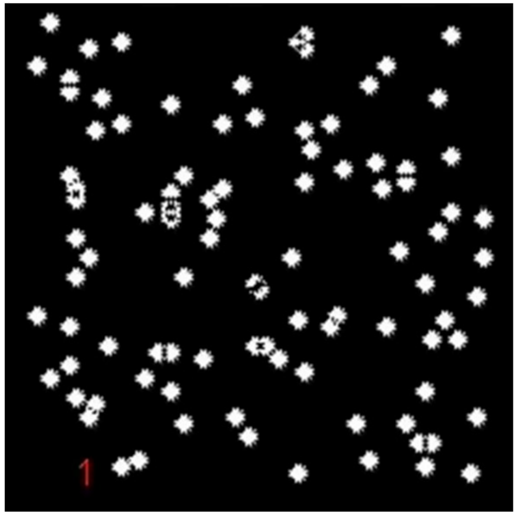
Chapman, Hajdu et al.

X-ray photon correlation spectroscopy (XPCS)

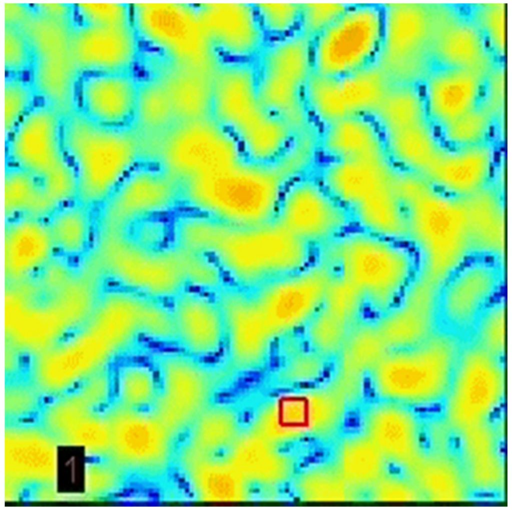


Diffraction of coherent light from a disordered sample leads to a 'grainy' diffraction pattern (speckles)

Simulation of Brownian motion



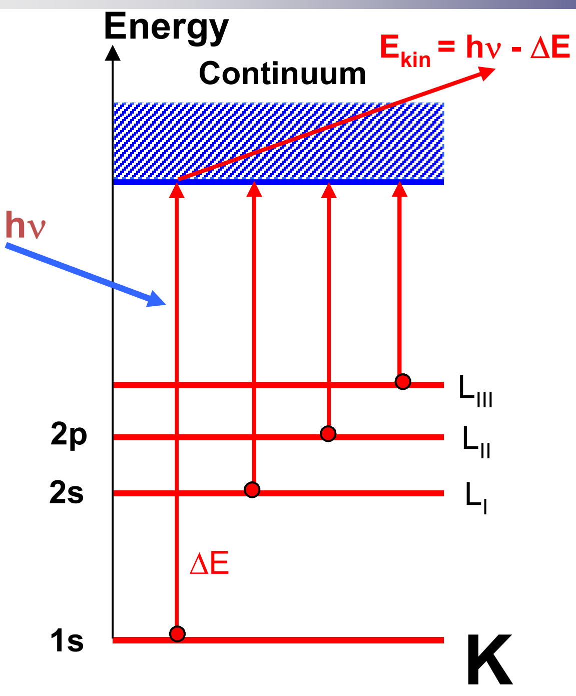
Real space

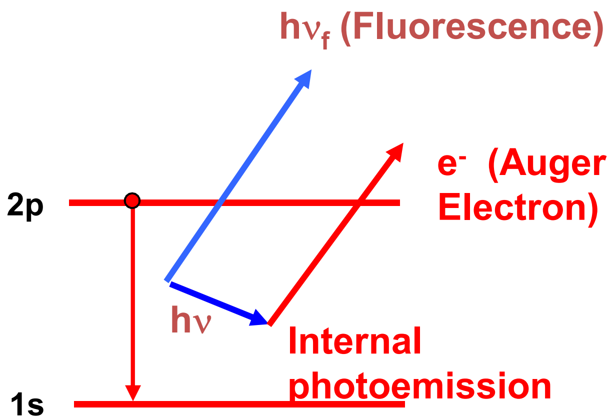


Diffraction pattern

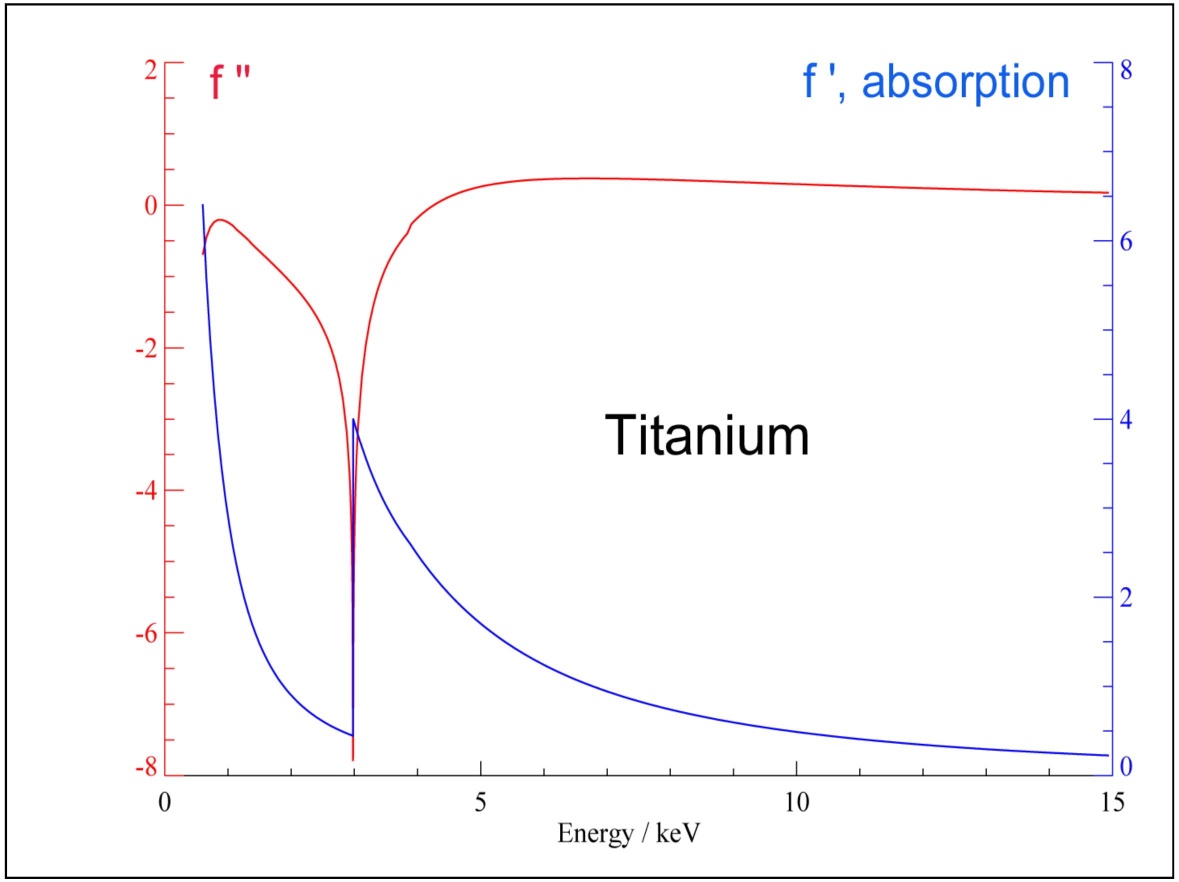
Scientific Experiments
Spectroscopy

Basics of X-ray Spectroscopy





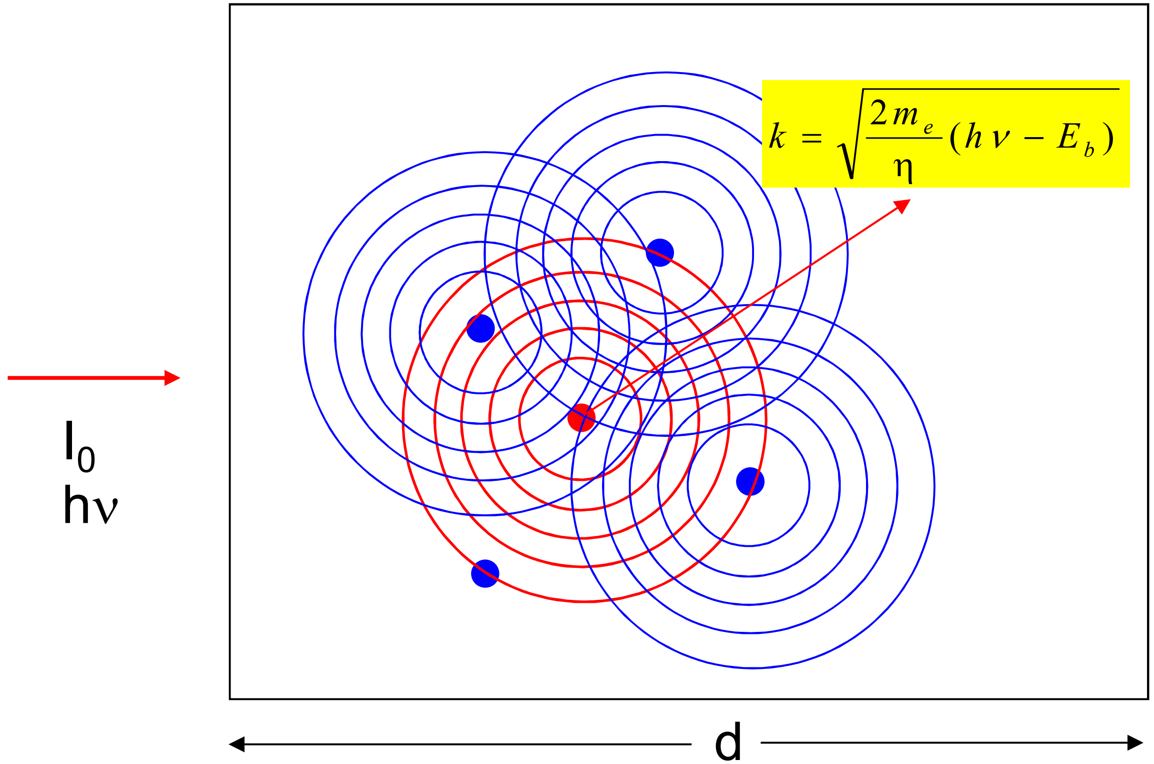
X-Ray Absorption Edges (example Titanium K-Edge)



$$f(s,E) = f_0 + f'(E) + i\,f''(E)$$

Atomic scattering Amplitude

X-Ray Absorption Spectroscopy (XAS)



$$k = \sqrt{\frac{2m_e}{\eta}(h\nu - E_b)}$$

Linear absorption coefficient μ

$$I = I_0 \cdot e^{-\mu d}$$

I_1
$h\nu$

Detected Signal: Absorption or Fluorescence as function of energy of incident radiation

Photoionisation of a K-electron in the „red“ ad-atom →

Free electron: $E_{kin} = h\nu - E_{binding}$
→ Electron Wave with wavevector

$$k = \sqrt{\frac{2m_e}{\eta}E_{kin}}$$

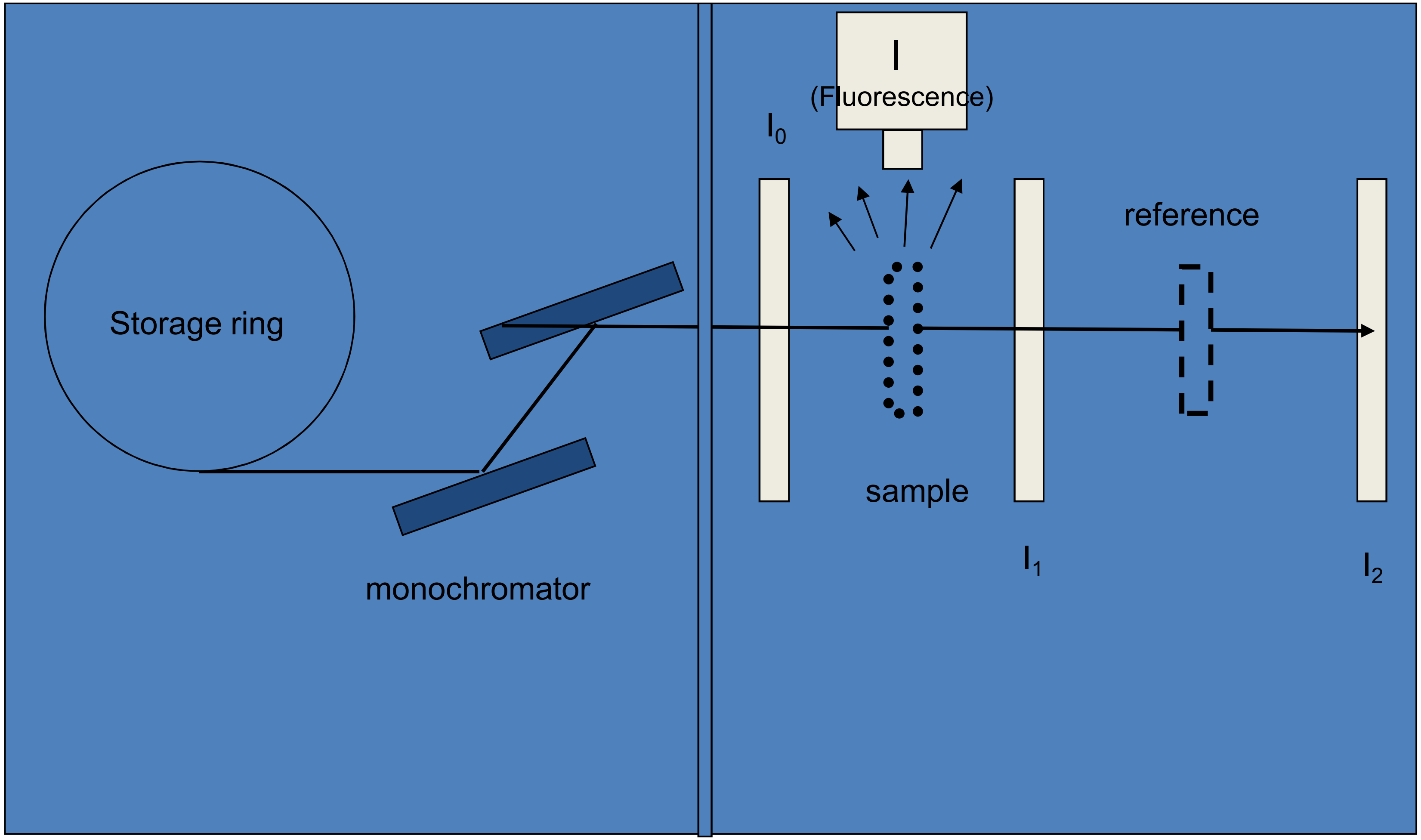
Fermis „Golden Rule“

$$\mu \propto |\langle\Phi_f|\, H\, |\Phi_i\rangle|^2 \cdot \delta(E_f - E_i - \hbar\omega)$$

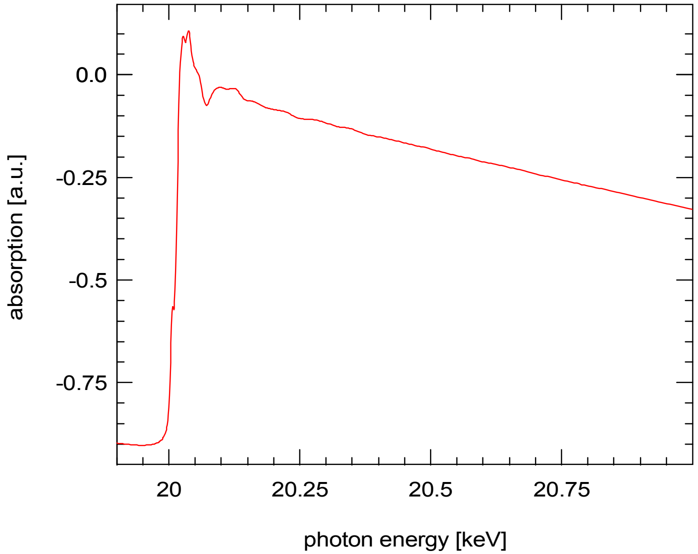
Initial state

final state $\Phi_f = \Phi_{outgoing} + \Phi_{backscattered}$

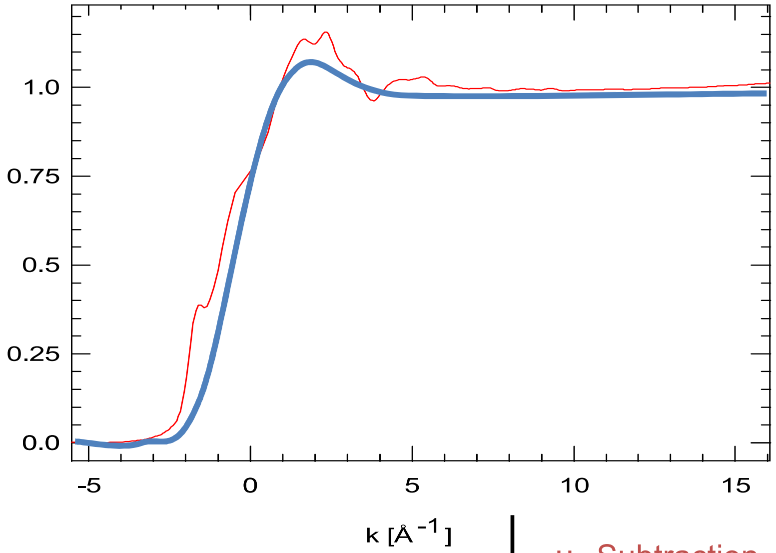
X-Ray Absorption Spectroscopy Experiment Setup



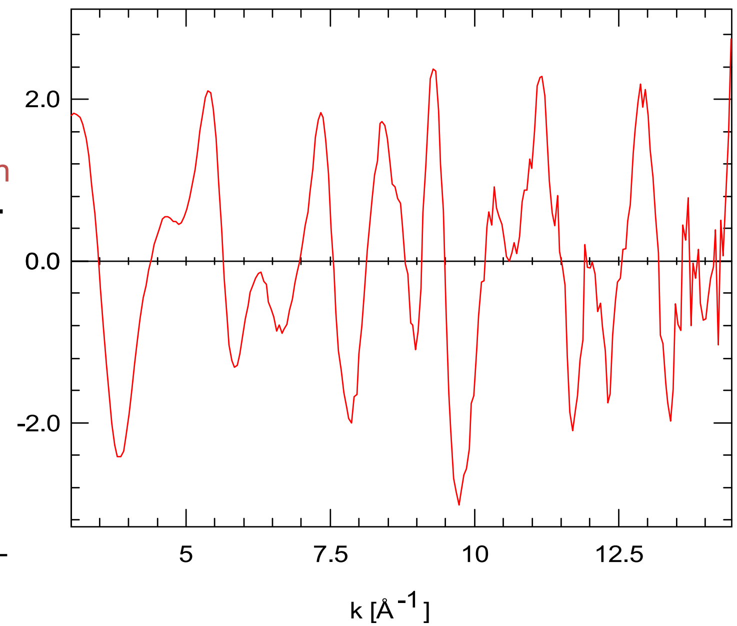
X-Ray Absorption Spectroscopy (EXAFS)



Energy calibration
Background subtraction
Normalization
E to k conversion

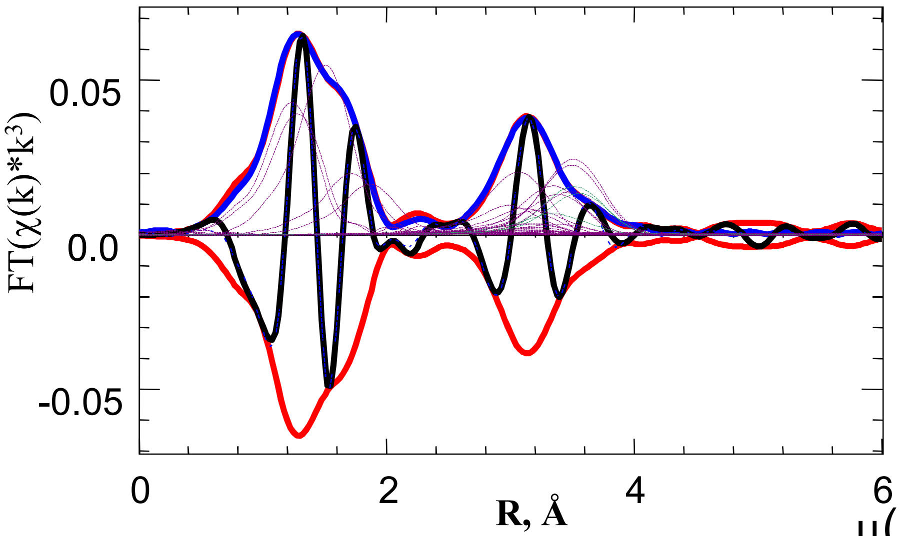


μ_0 Subtraction



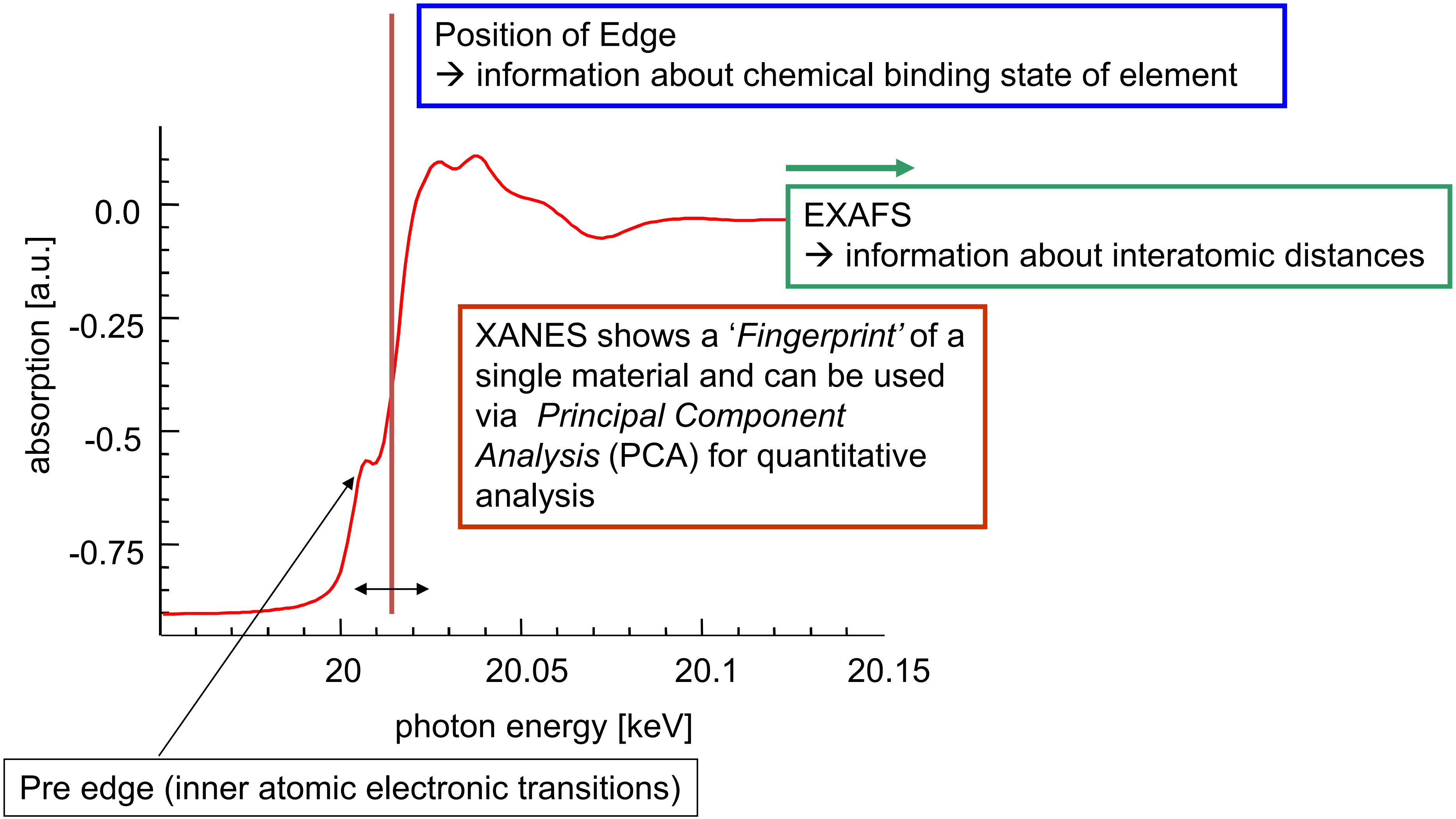
Fourier-Transformation

$\chi(k)$



$$\chi(k) = \frac{\mu(k) - \mu_0(k)}{\mu_0(k)}$$

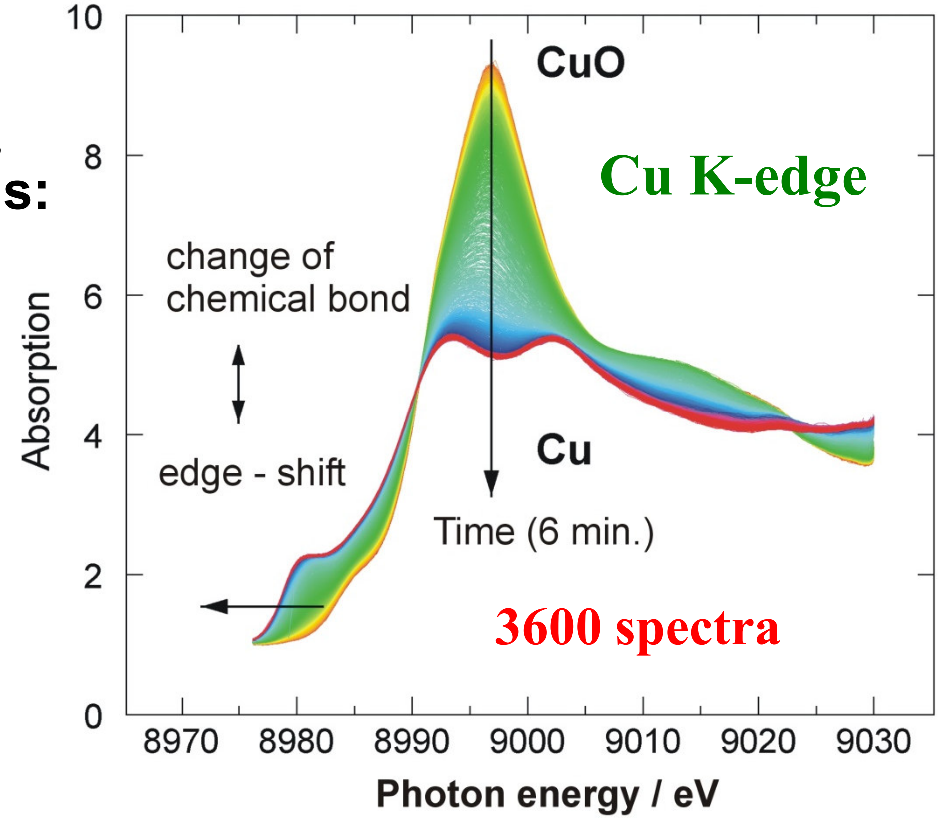
Near Edge X-Ray Absorption Spectroscopy (XANES)



Instrumental development: QEXAFS (piezo scanning)
Study of solid state transformations in catalysis

Activation of a $CuO/ZnO/Al_2O_3$ catalyst for methanol synthesis:

- In-situ reduction in H_2 gas flow at elevated temperatures
- 50 ms time resolution
- Detailed analysis of transient chemistry (here Cu_2O)
- Experiment done at BW1



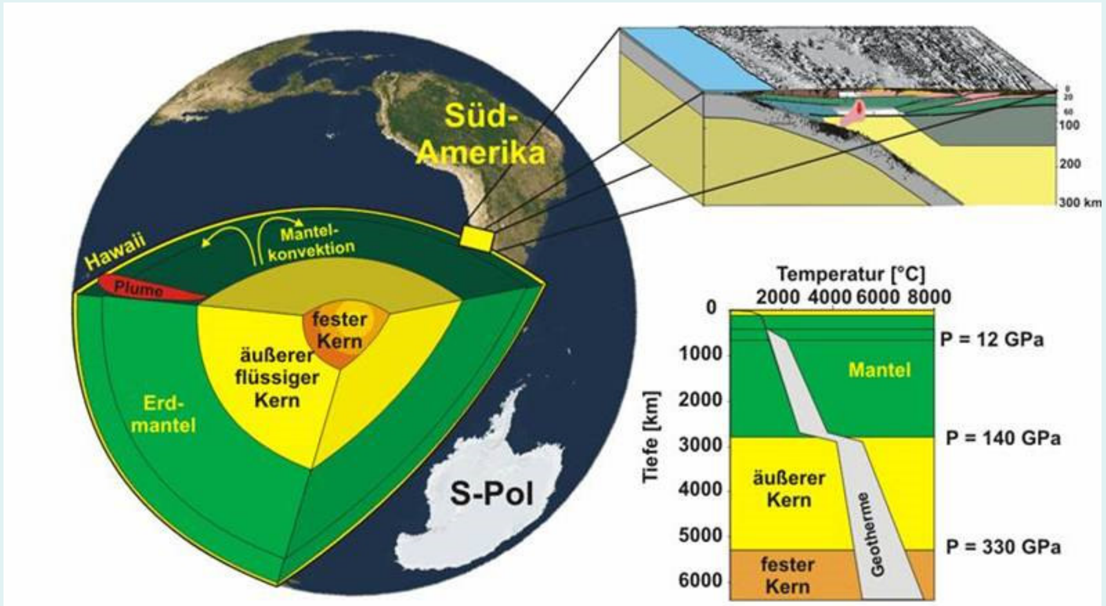
J.-D. Grunwaldt et al., J. Phys. Chem. B **105**, 5161 (2001)

Geo Science Experiments (High p, High T)

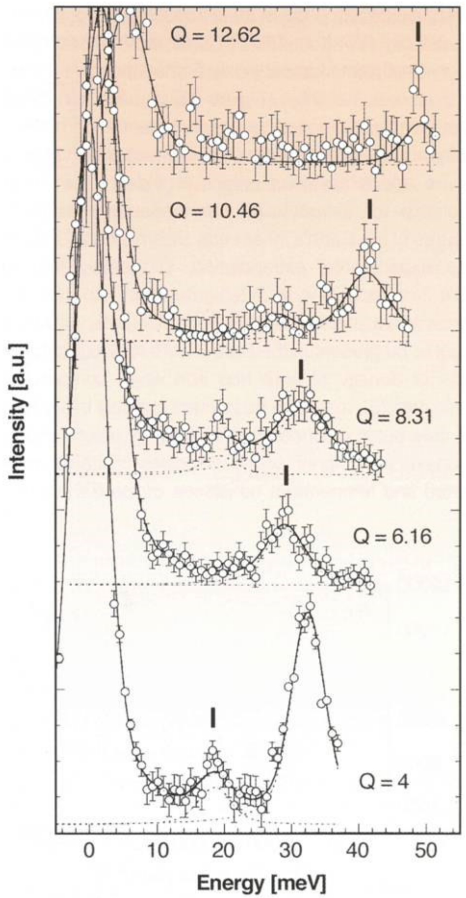
Large volume press of GFZ (Geo Research Center Potsdam) at DESY

1750t press for in situ studies of large sample volumes.
Maximum pressure: **~ 25 GPa**
Temperature: **> 2000 K**

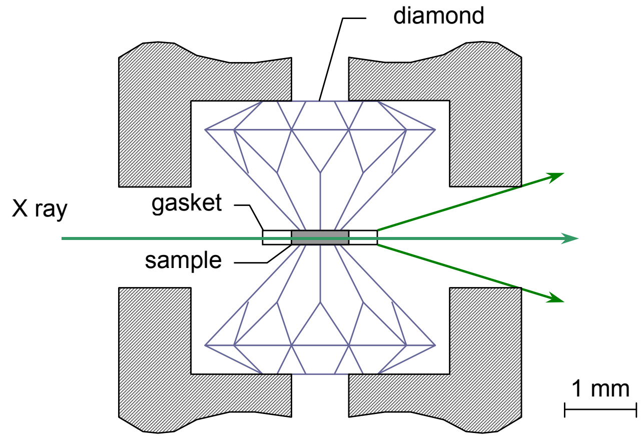
Study of material under the conditions of the earths lower mantle.

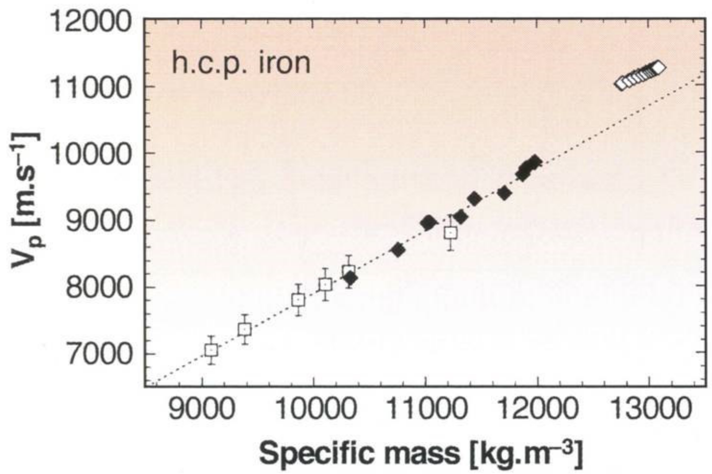


Speed of sound of Fe under pressure (19 to 110 GPa)



P=28GPa

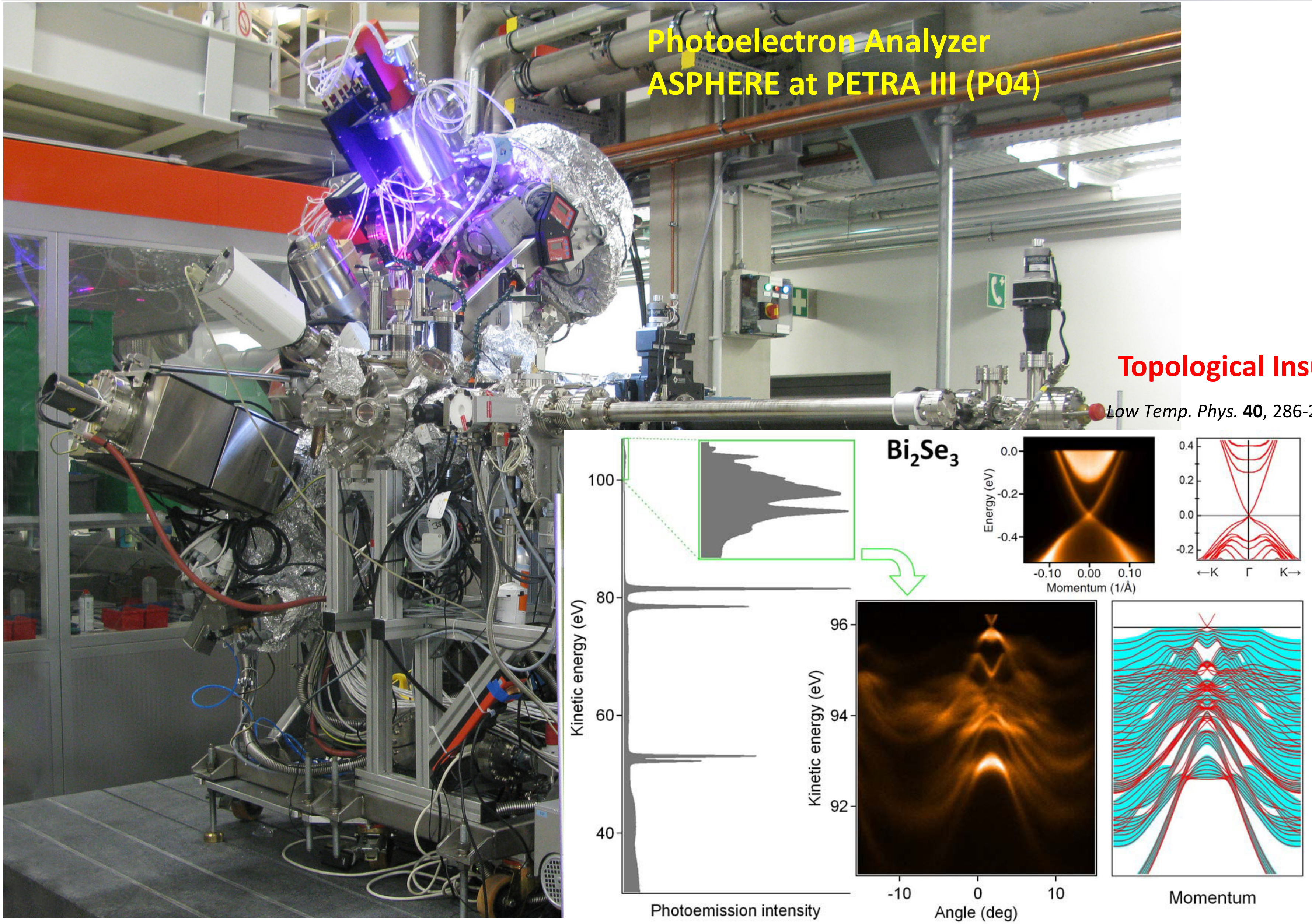




Determining the acoustic phonon dispersion E(Q) at a given pressure by means of inelastic X-ray scattering

G. Fiquet et al., Science (2000)

Angular Resolved Photoelectron Spectroscopy (ARPES)



Topological Insulator

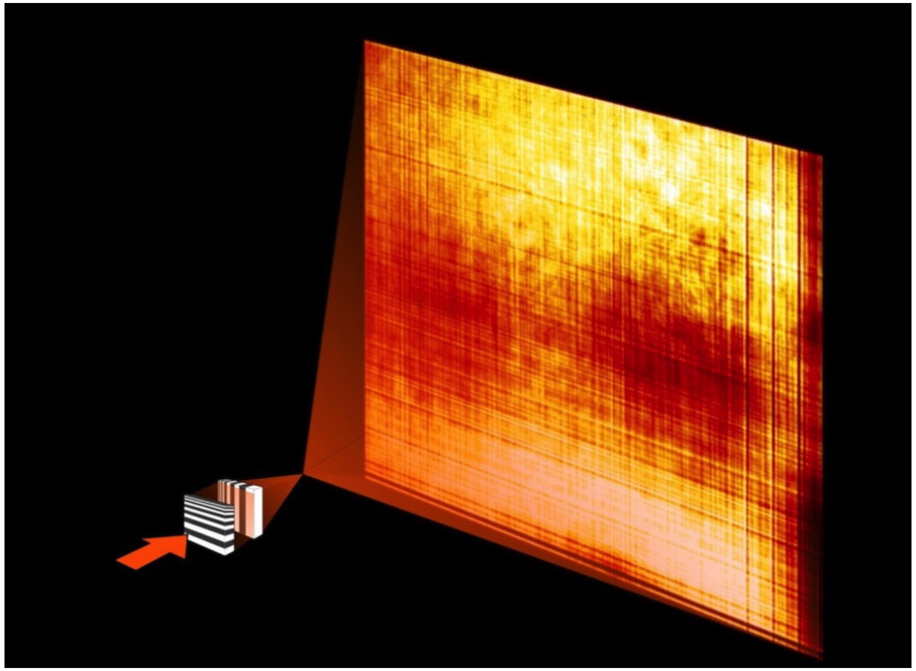
Low Temp. Phys. **40**, 286-296 (2014)

Scientific Experiments
Imaging

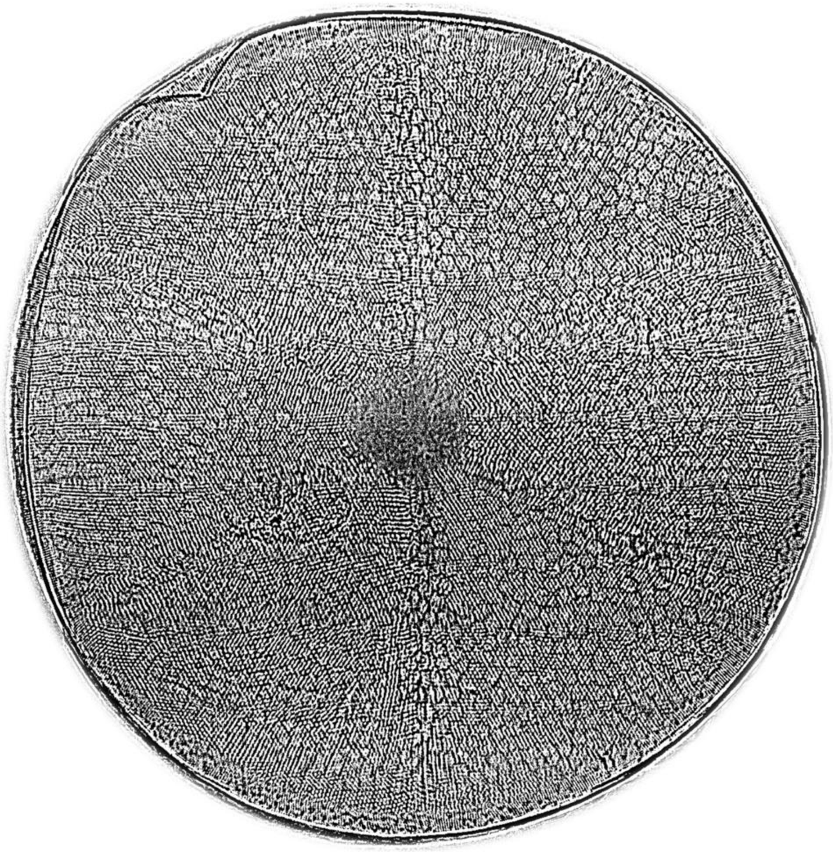
Laue Lenses Towards 1 nm Resolution

Two crossed SiC/WC MLLs
About 20000 layers
Focal lengths 1.4 mm and 2.0 mm

Focus 8.4 nm x 6.8 nm
At 16.3 keV with 80 % efficiency



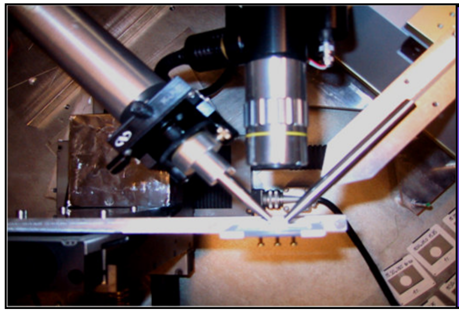
X-Ray hologram of diatomee structure

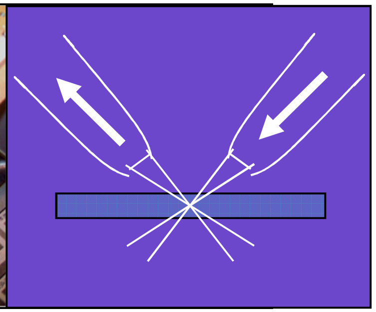


Micro X-ray Fluorescence on Daphnia Magna
(water flea)

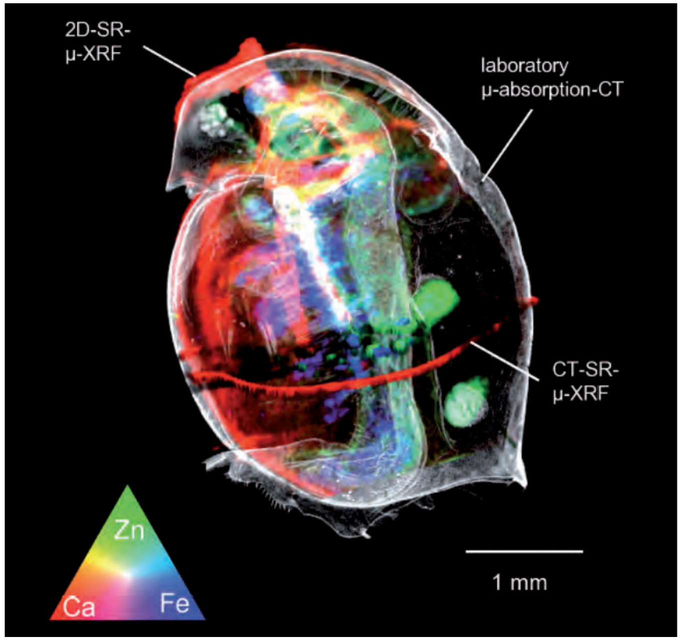
Principle

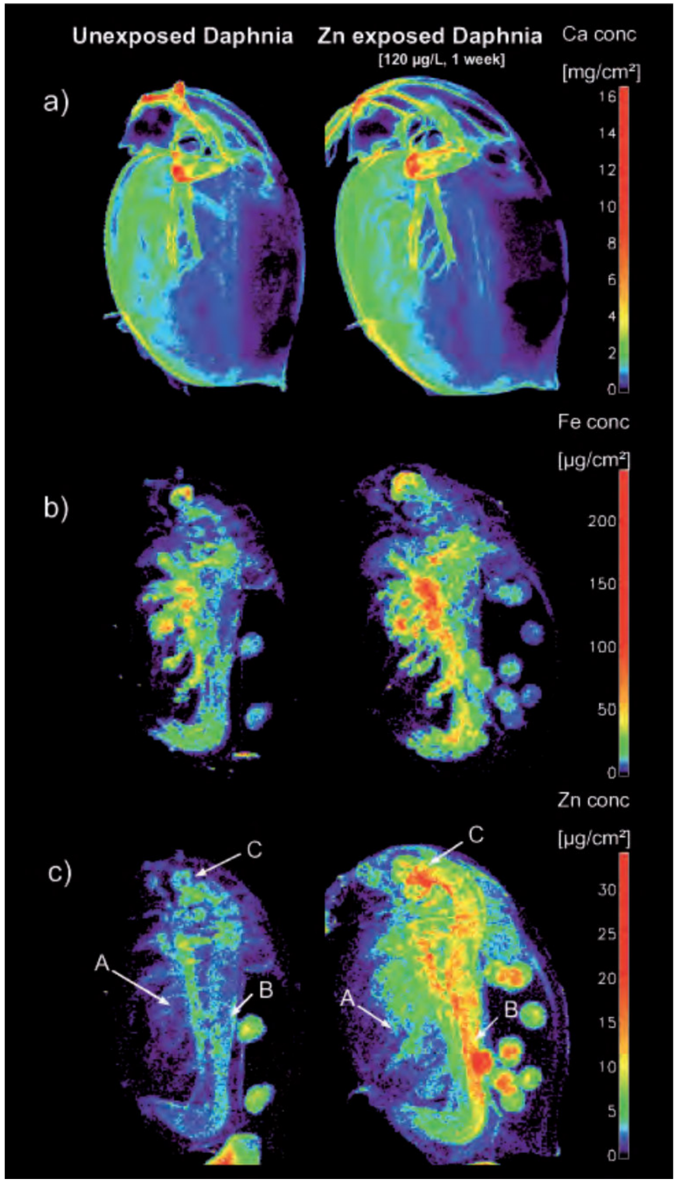
DORIS III beamline L



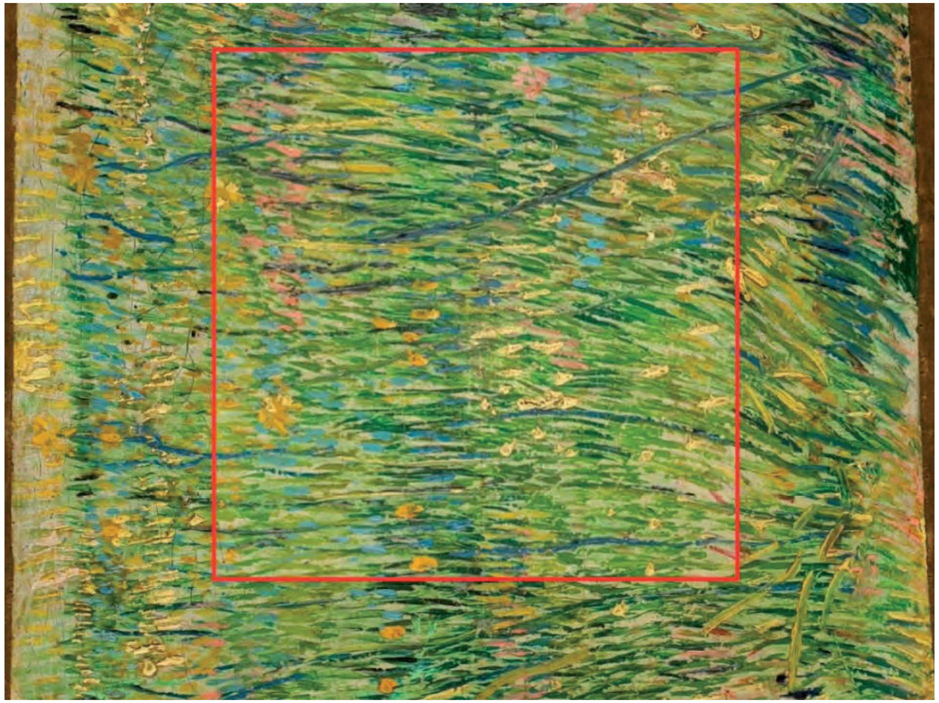


Focus 10 μm

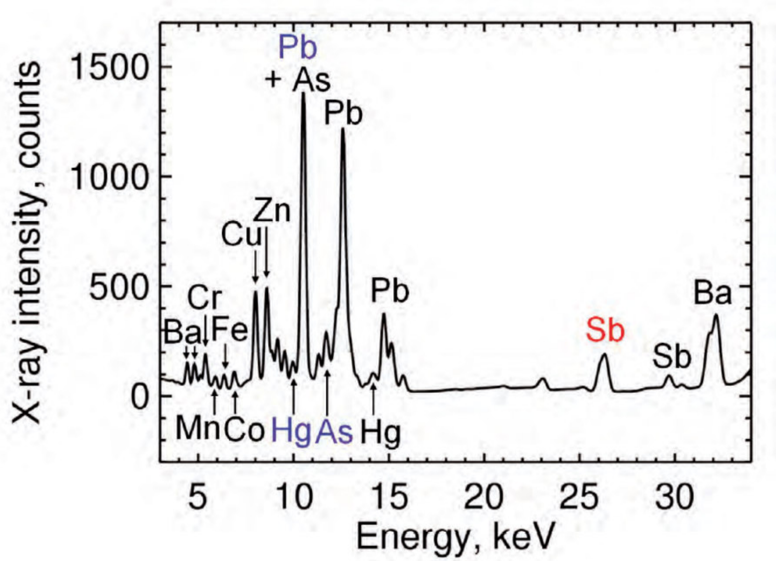




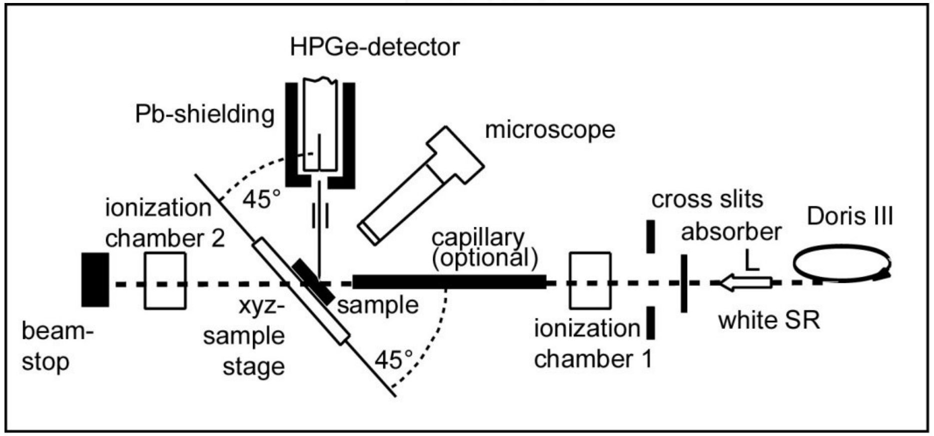
Vincent van Gogh: Meadow with flowers

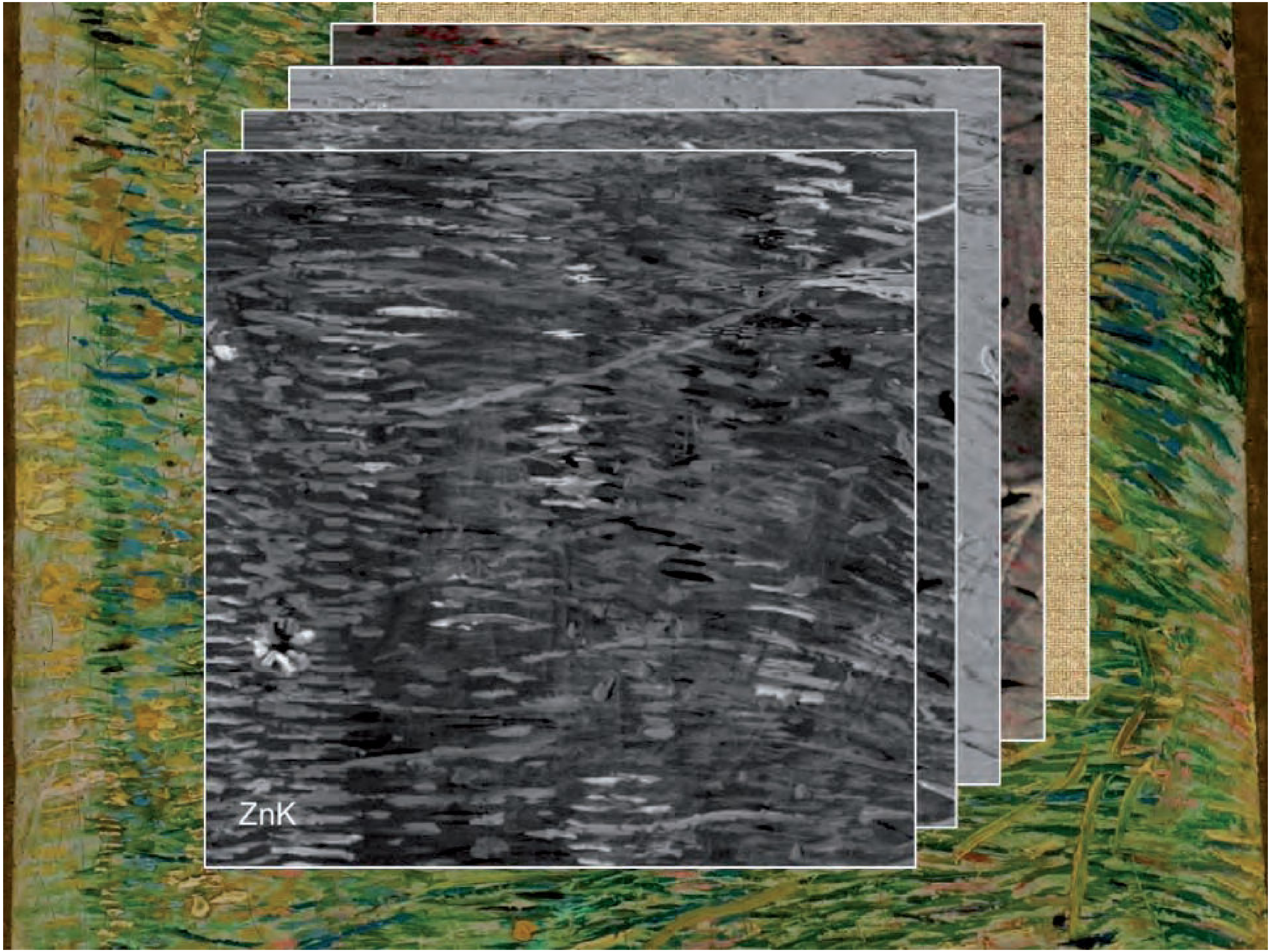


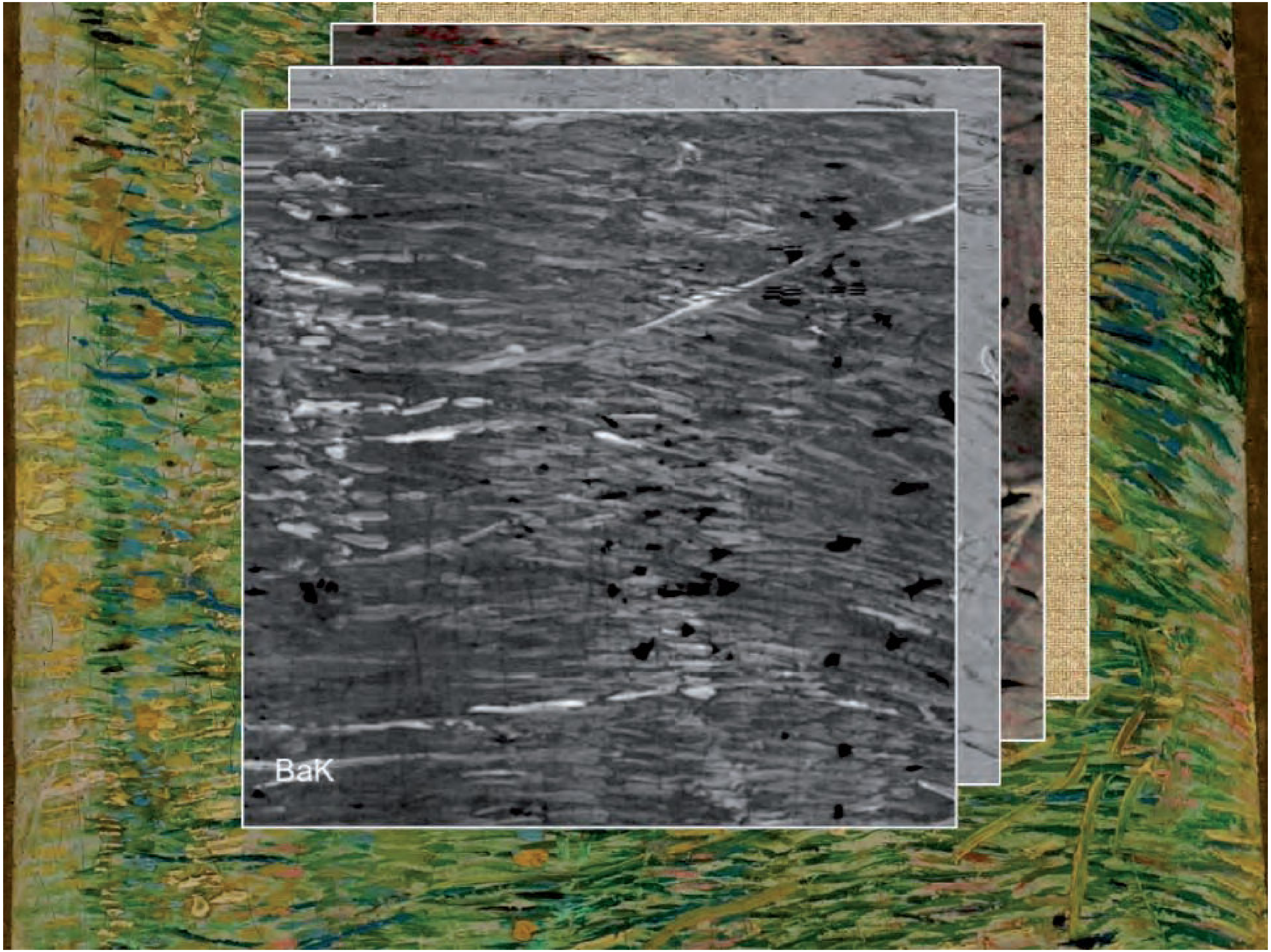
Typical fluorescence spectrum in a single pixel

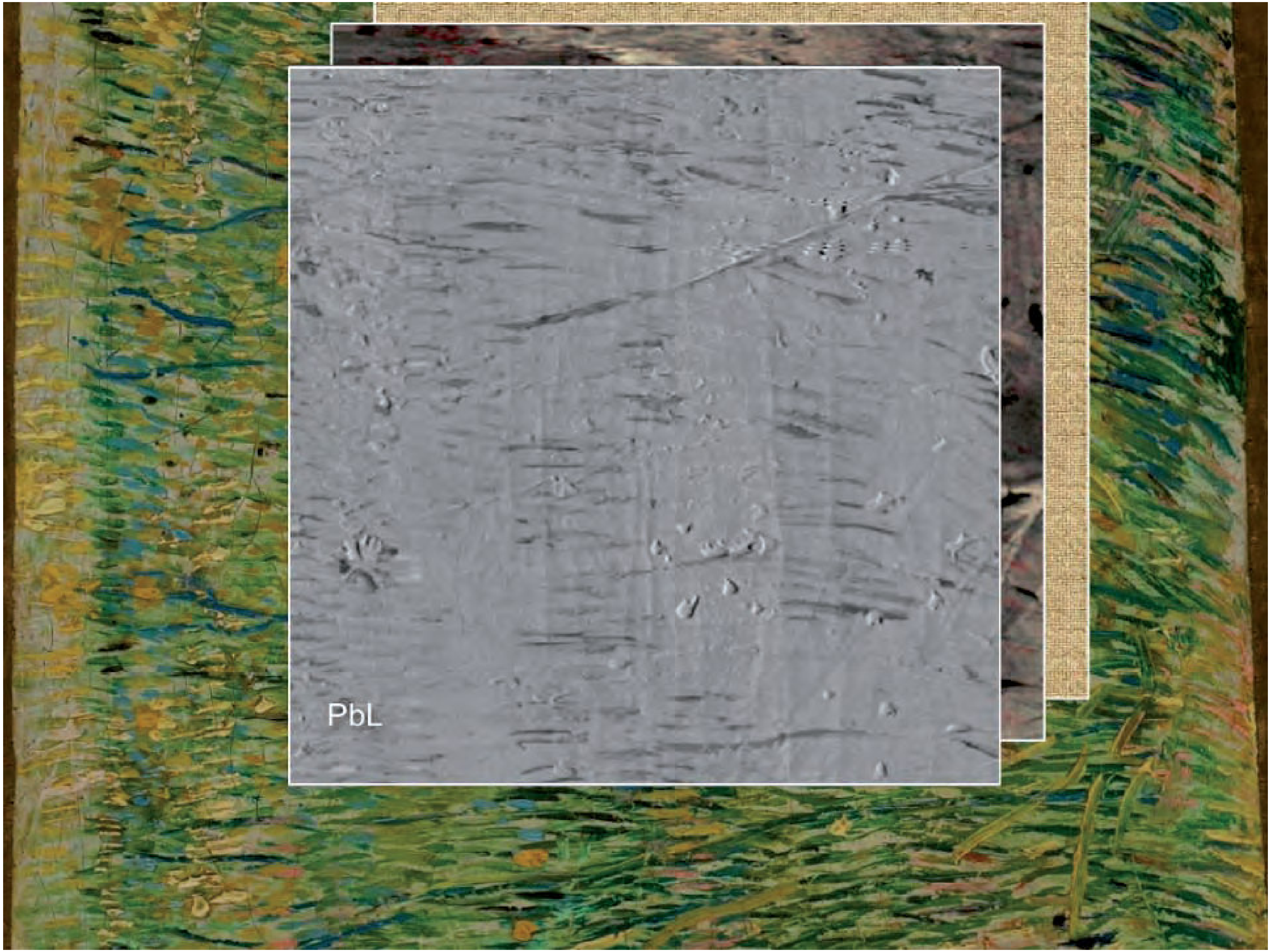


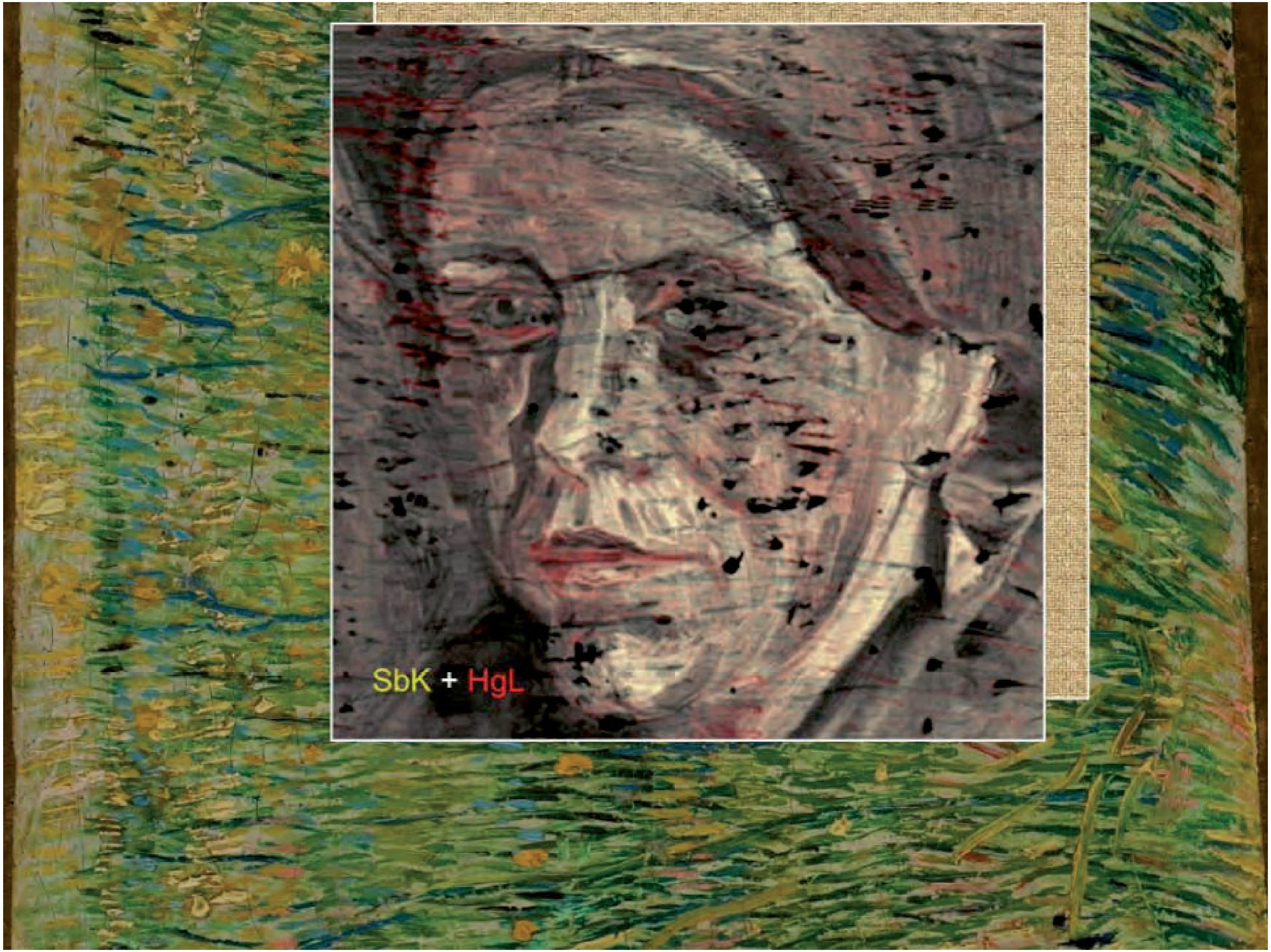
Raster scanning along 90000 pixels with 0.5 mm resolution




ZnK

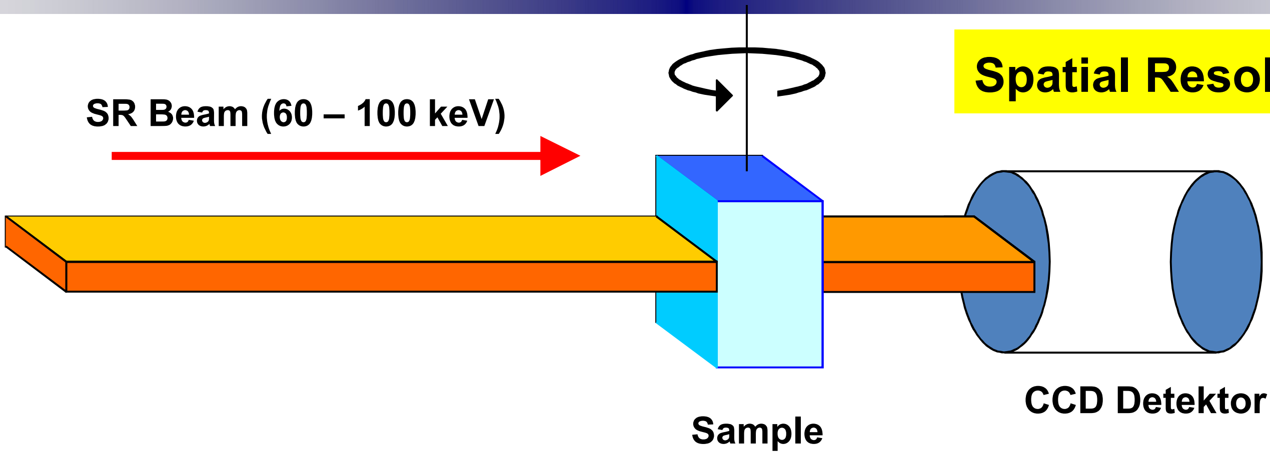

BaK

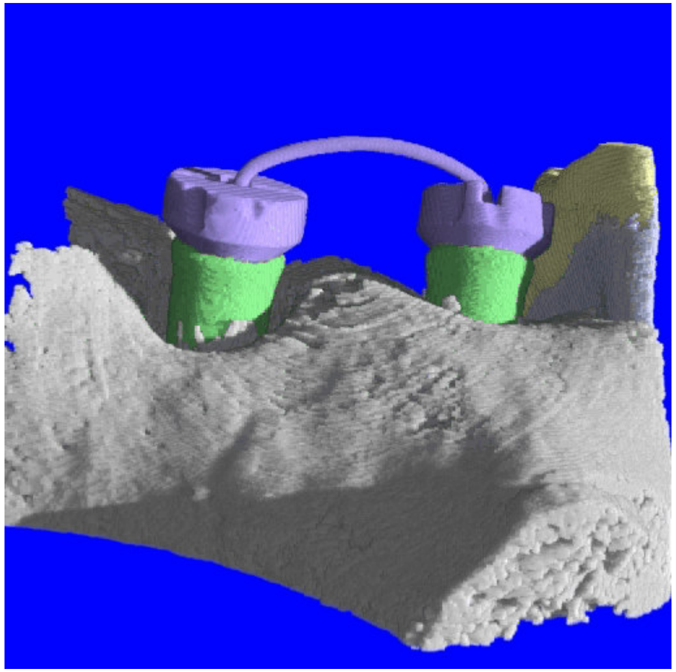

PbL


SbK + HgL

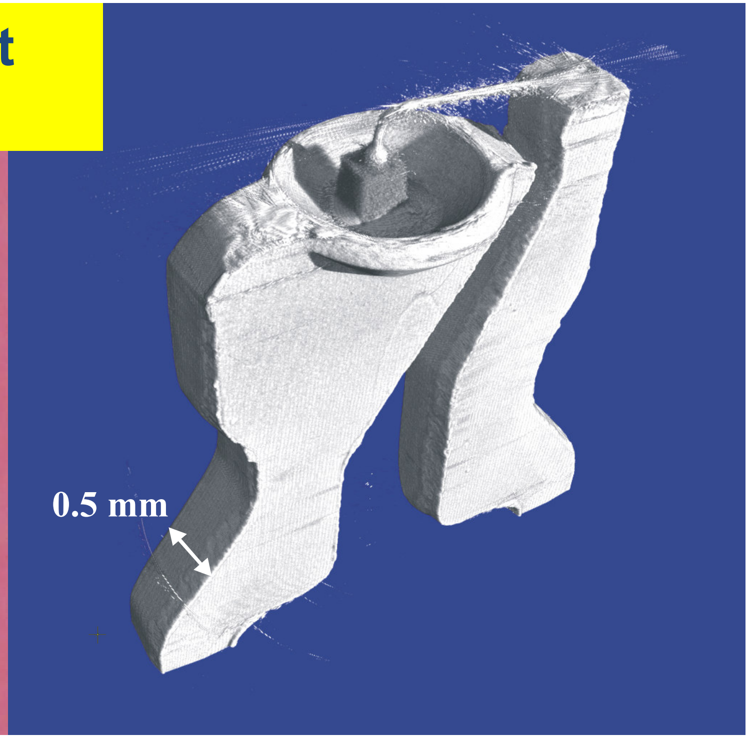
Parallel Beam X-Ray Tomography

Spatial Resolution: 10 µm





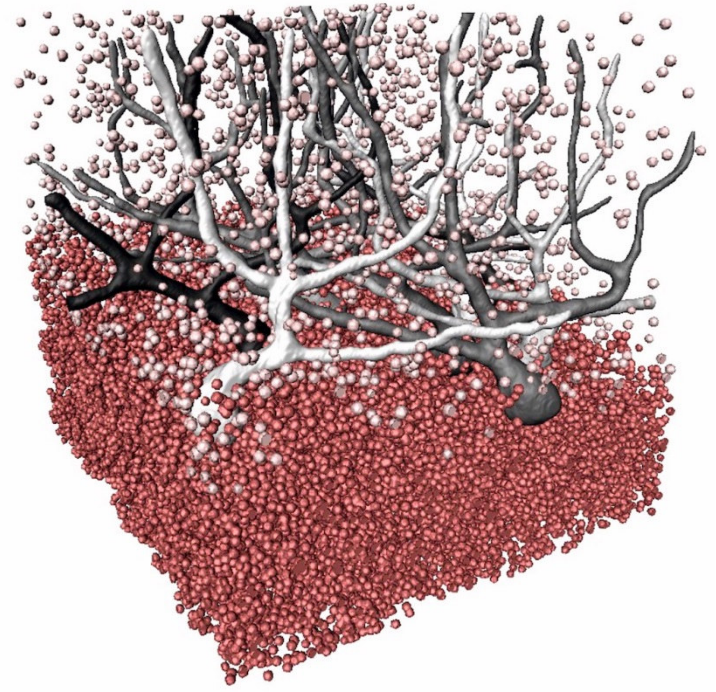
Sample: LED at 60 keV

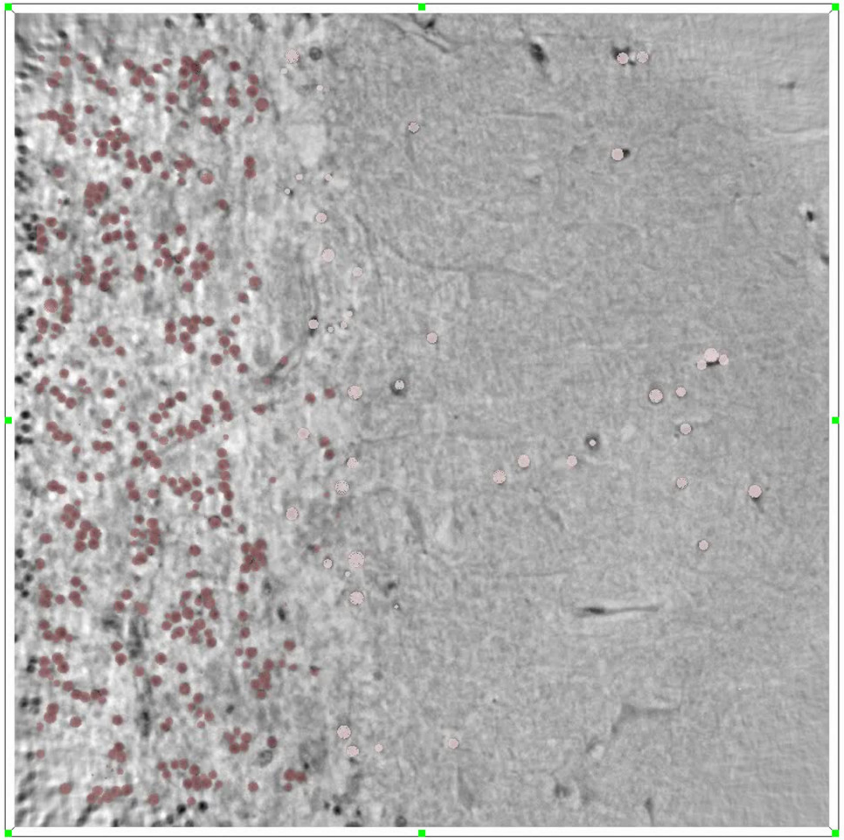


Phase Contrast Tomography of Neurons in Brain Tissue

3D virtual histology at beamline P10
Highly divergent nanometre focus (waveguide)
Photon energy 8 keV

Automatic cell segmentation
Rendering of 1.8·10^{6} neurons.

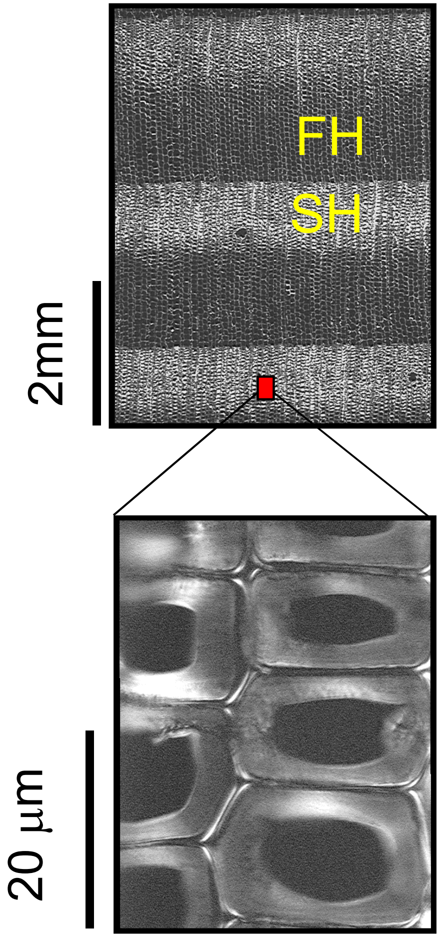


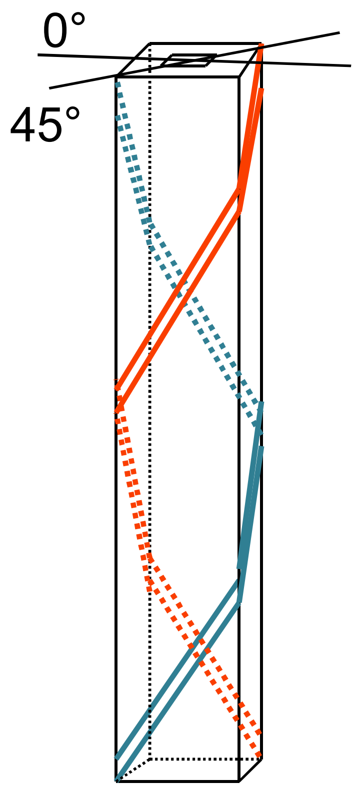


M. Töpperwien, F. van der Meer, C. Stadelmann, T. Salditt; „PNAS“, 2018

Wood - Determination of the microfibril angle (Microfocus SAXS and WAXS)

Picea abies (spruce)

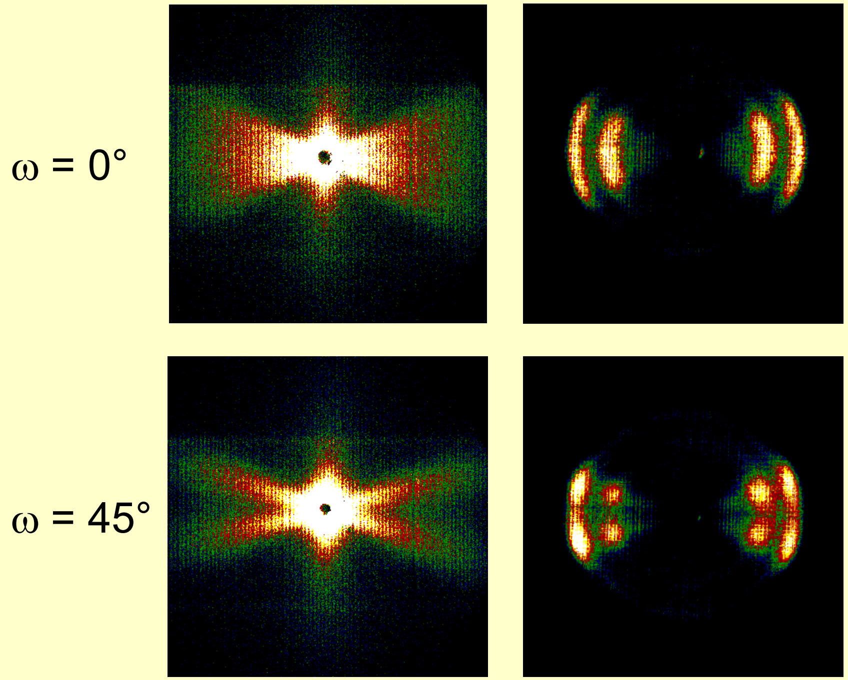




Focus 2x2µm^2

Fiber geometry

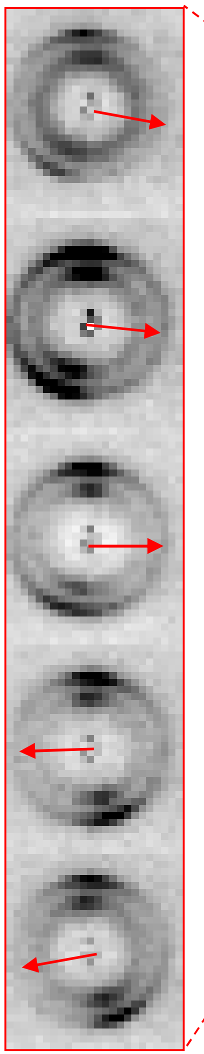
	SAXS	**WAXS**

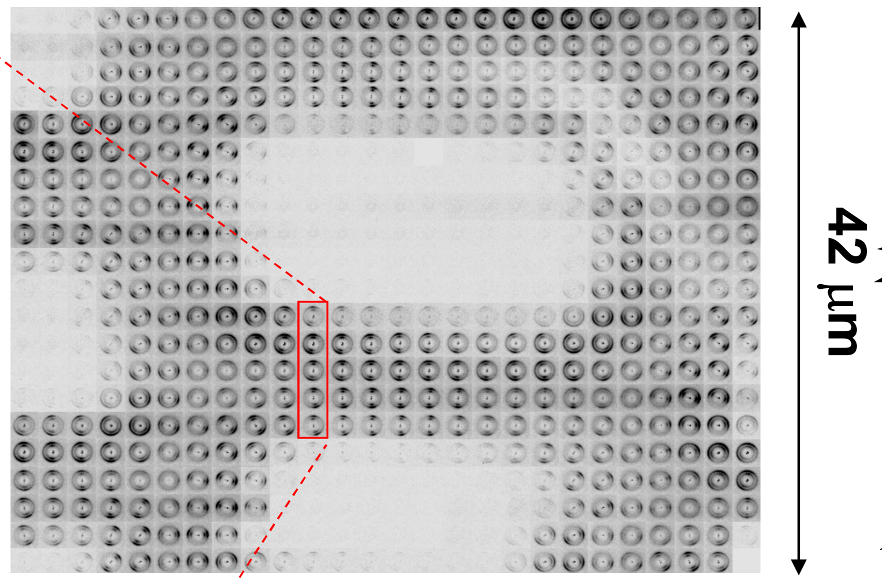


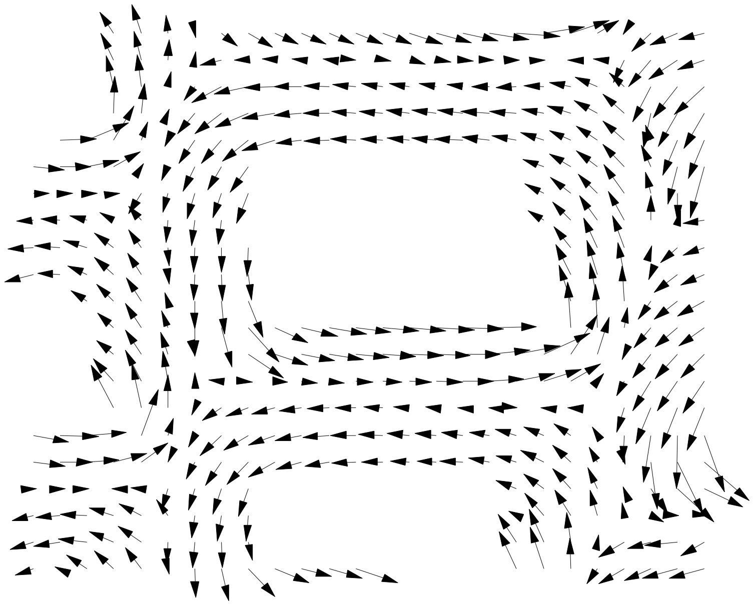
$\mu = 20.4°(\pm 0.6°)$ $\mu = 20.0°(\pm 0.6°)$

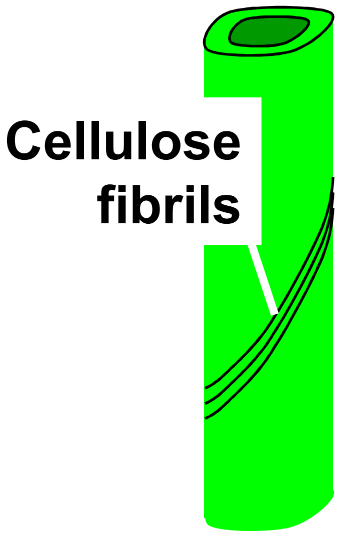
Lichtenegger et al. *J. Struct. Biol.* (1999)

Helical arrangement of cellulose fibers in the wood cell wall (Scanning Microfocus SAXS)







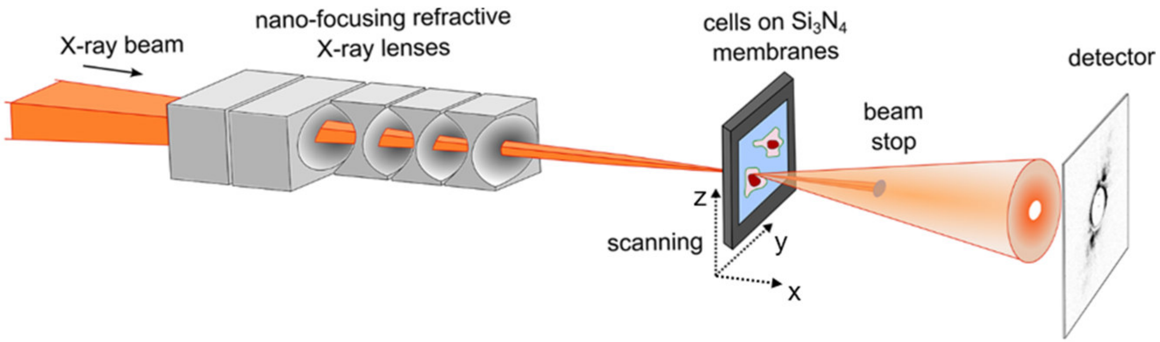


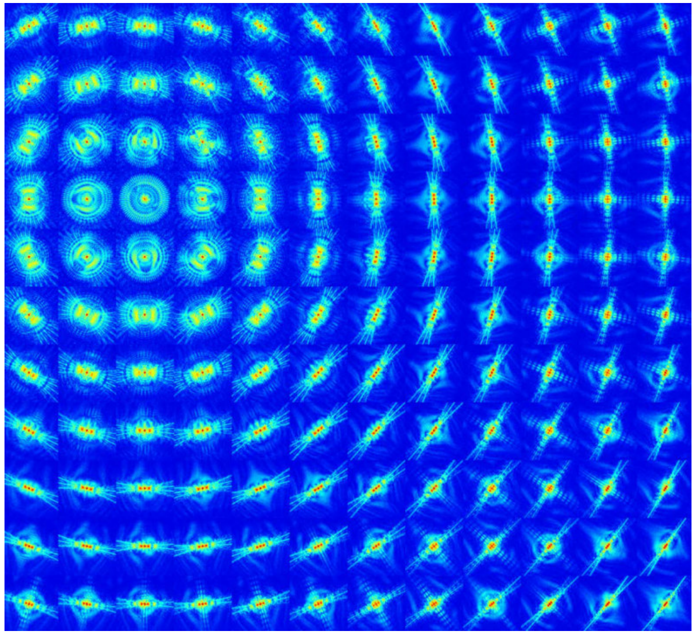
Focus 2x2µm^2

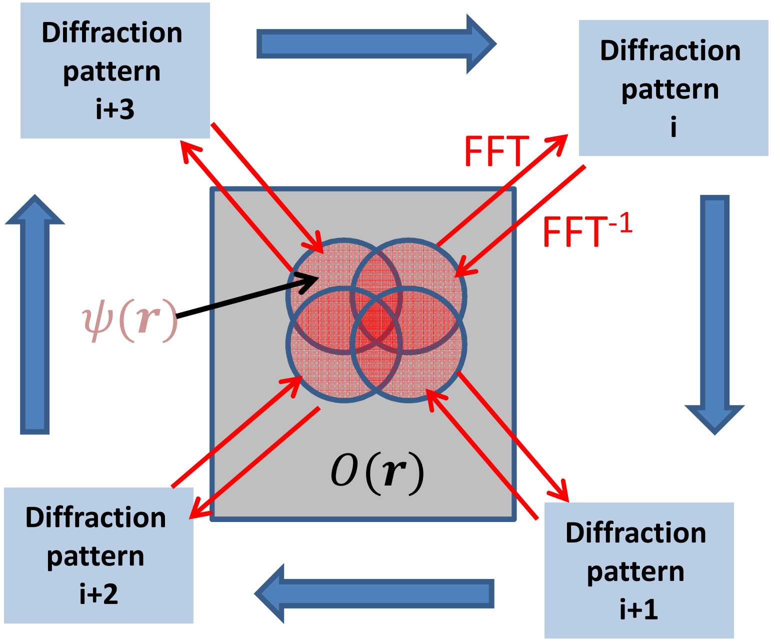
Z-Helix

Lichtenegger, Müller, Paris, Riekel & Fratzl
J. Appl. Cryst. (1999)

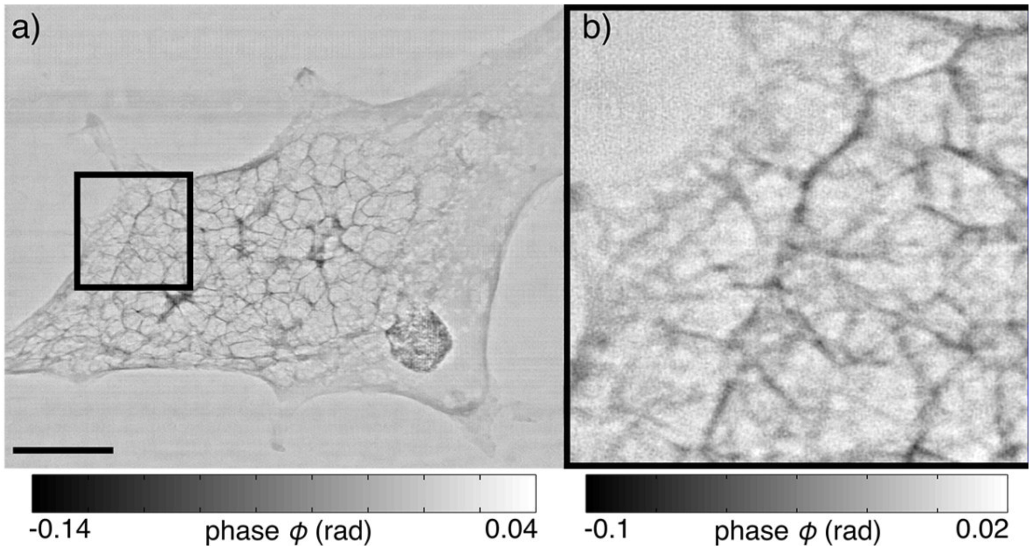
Ptychography







Energy 14.9 keV
Beamsize 100 nm
Stepwidth 10 nm
Resolution a) 65 nm
Resolution b) 10 nm



$$O^{n+1}(\boldsymbol{r}) = O^{n}(\boldsymbol{r}) + \beta_0 \frac{\overline{p^n}}{max|\overline{p^n}|^2}\left(\psi_j^{n+1} - \psi_j^{n}\right)$$

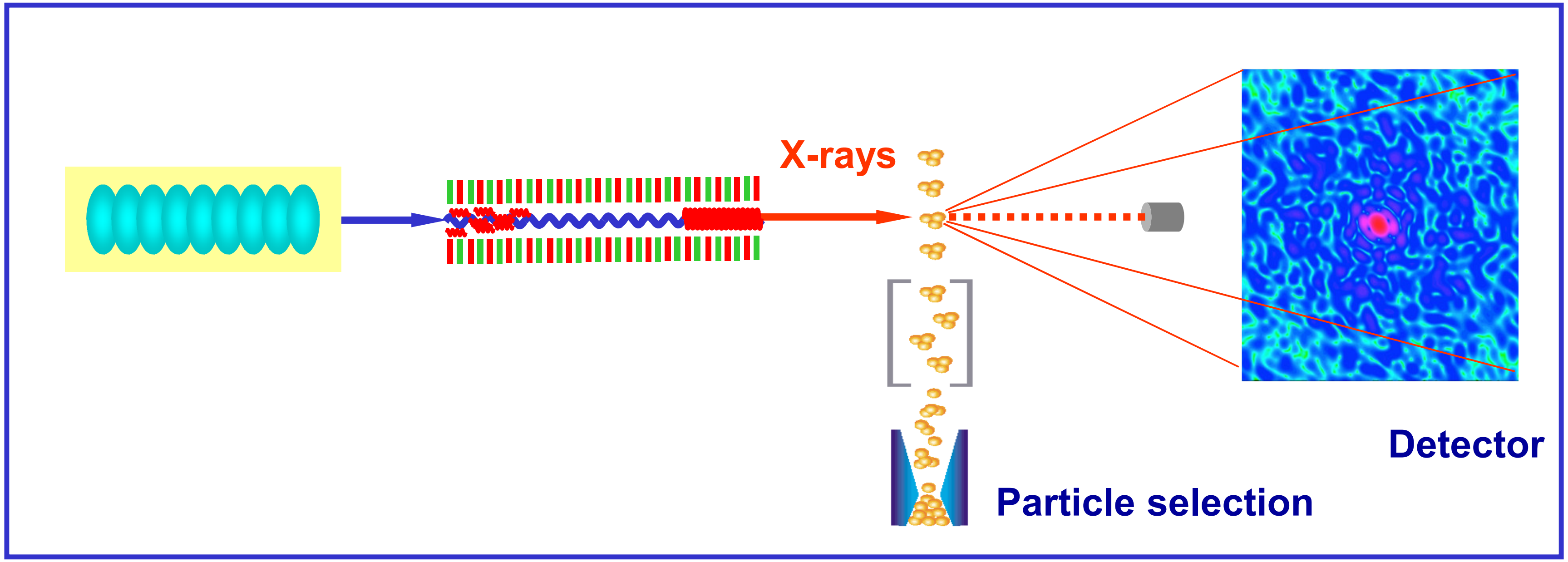
$$\psi_j(\boldsymbol{r}) = P(\boldsymbol{r} - \boldsymbol{r}_j) \cdot O_j(\boldsymbol{r})$$ Exit surface wave

Photon Science Facilities on DESY Campus

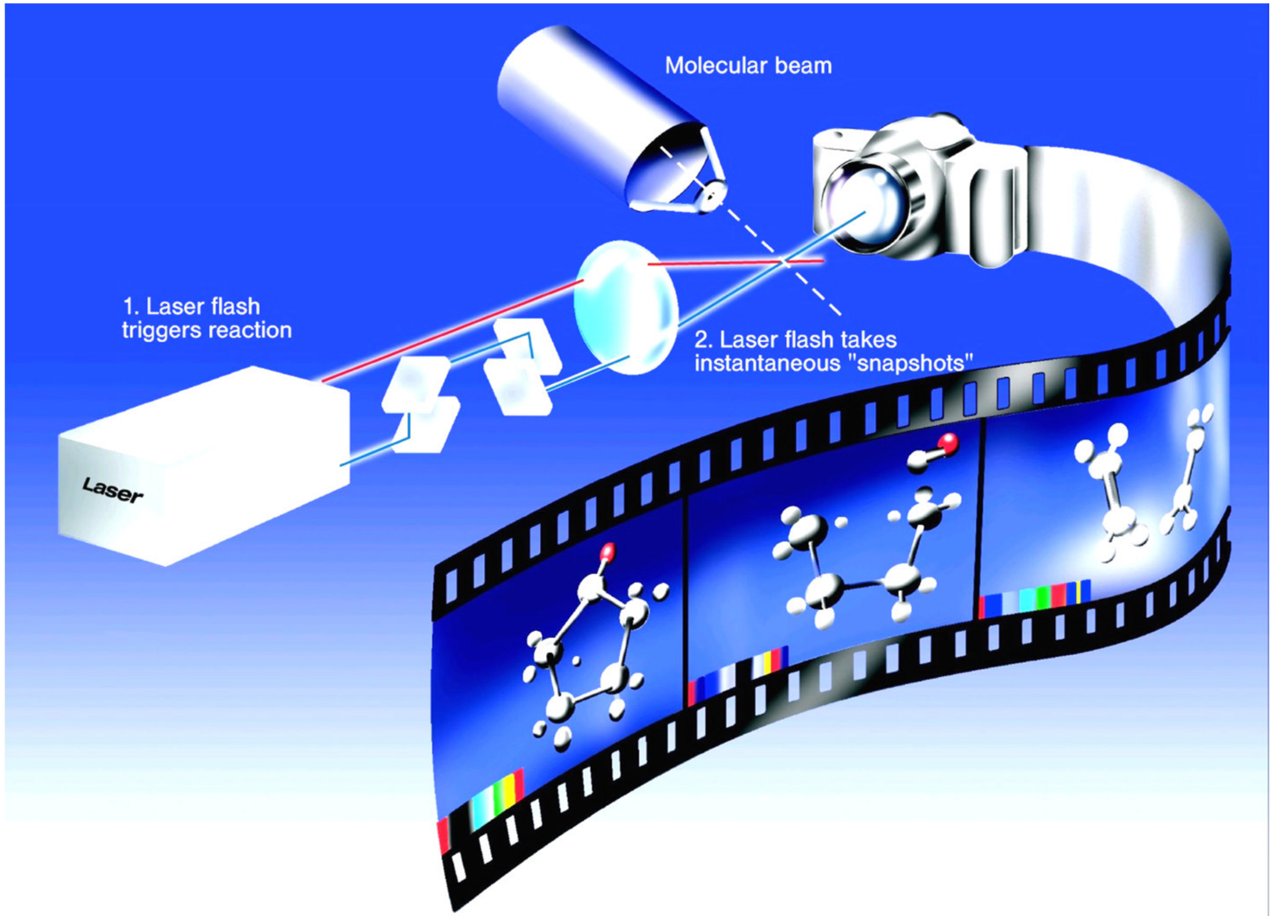
CXNS = Center for X-Ray and Nano Science
CFEL = Center for Free Electron Laser Science (DESY, MPI, UniHH)
CHyN = Center for Hybrid Nanostructures
CSSB = Centre for Structural Systems Biology
MPSD = Max-Planck Inst. For Structure and Dynamics of Matter

Experiments at FELs

The Ultimate Goal: Single-Molecule Diffraction

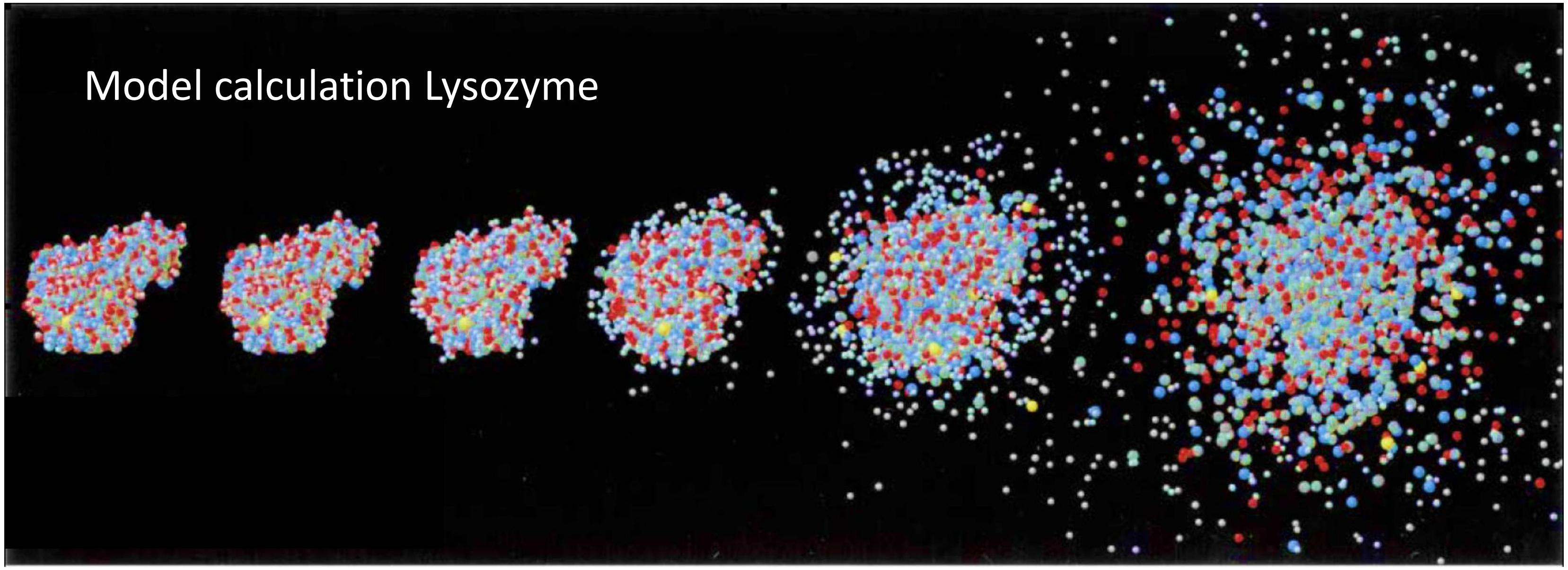


The Ultimate Goal: Recording the "Molecular Movie"



Snapshots for different times after excitation (pump-probe spectroscopy) → "motion picture" of the reaction

Coulomb Explosion

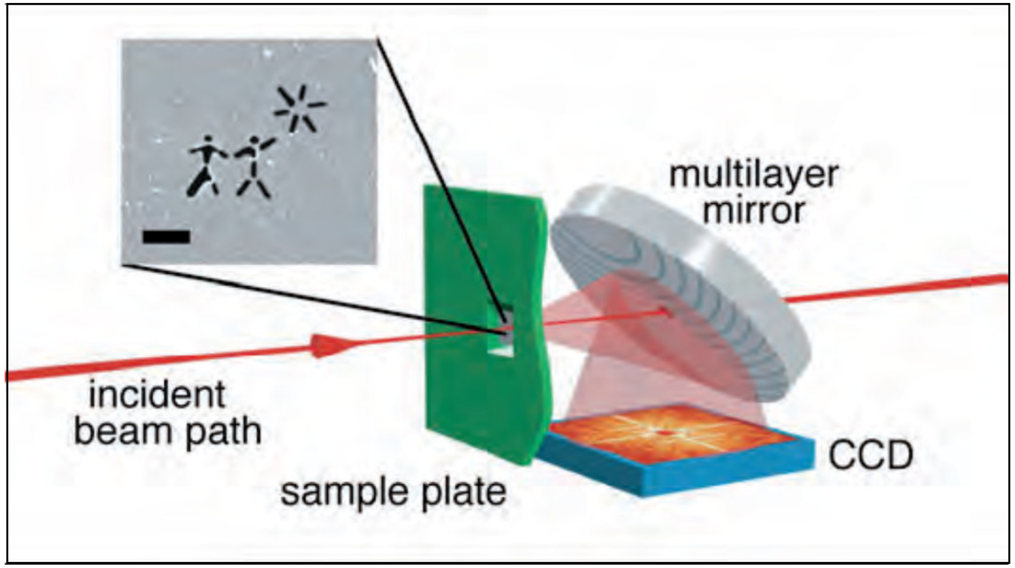


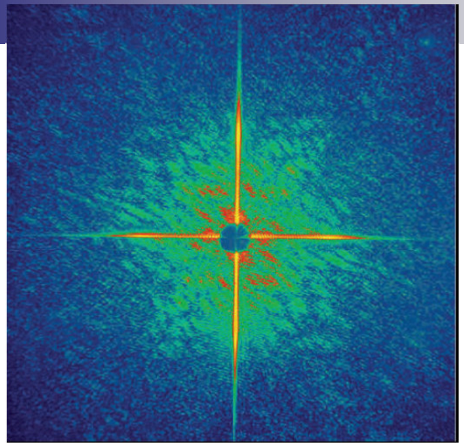
Time = 0 2 5 10 20 50 femtoseconds

Coulomb Explosion of a molecule in the strong electric field of an FEL

Ultrafast Coherent Diffractive Imaging at FLASH

Resolution 50 nm





1st shot

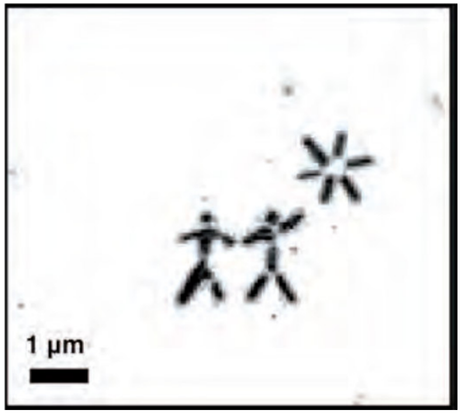
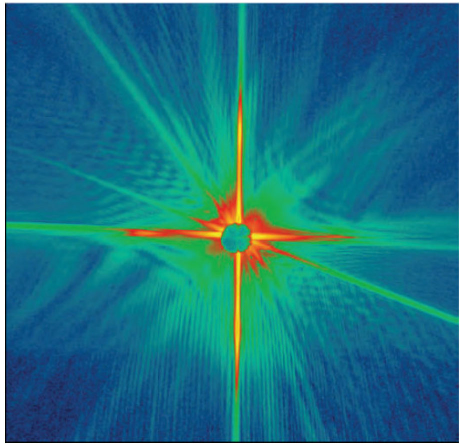
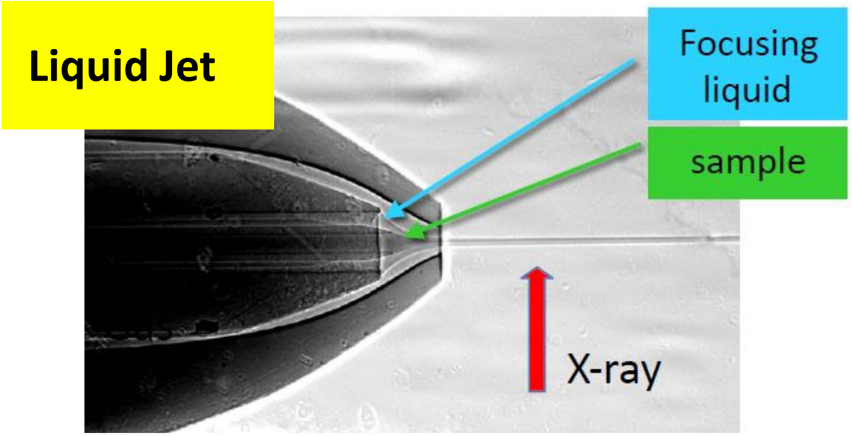


Image Reconstructed From 1st shot

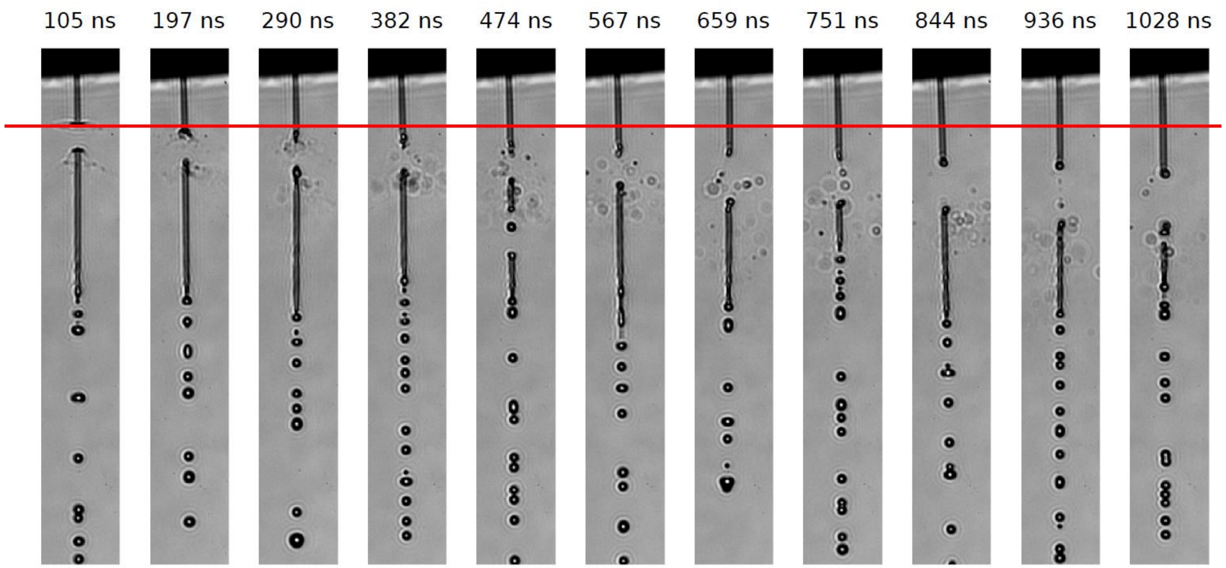


2nd shot
(target destroyed)

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



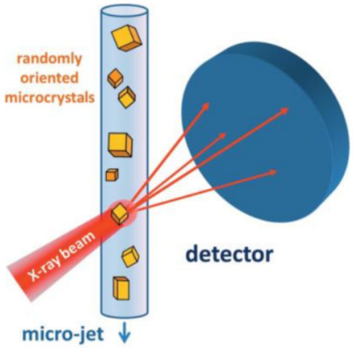
Fast liquid jet for sample delivery

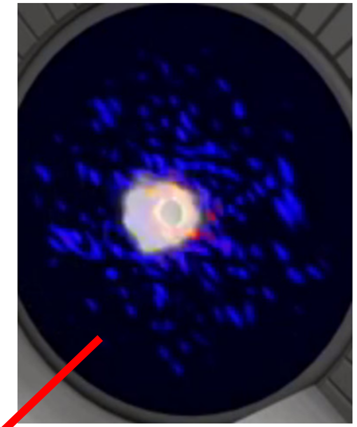


FLASH

Serial Femtosecond Crystallography SFX

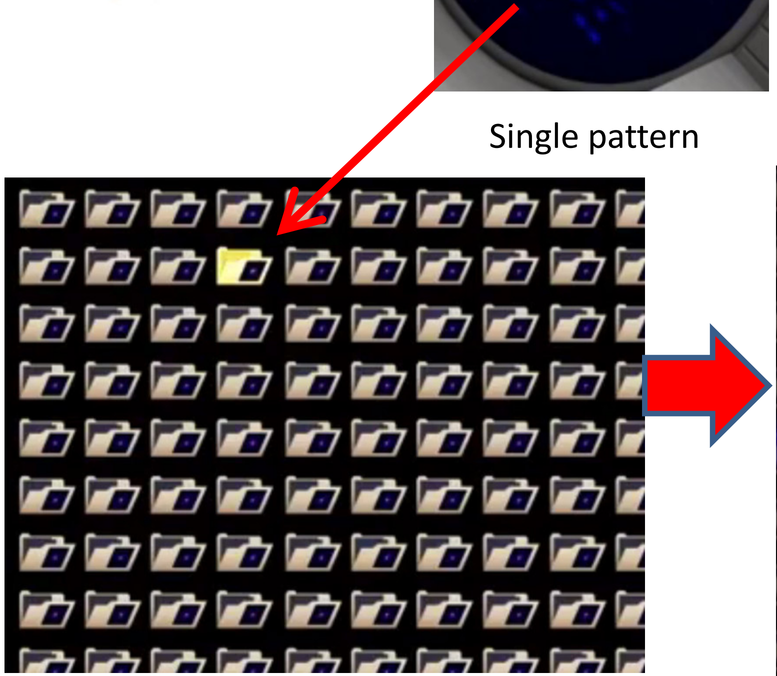
„Diffraction before Destruction“



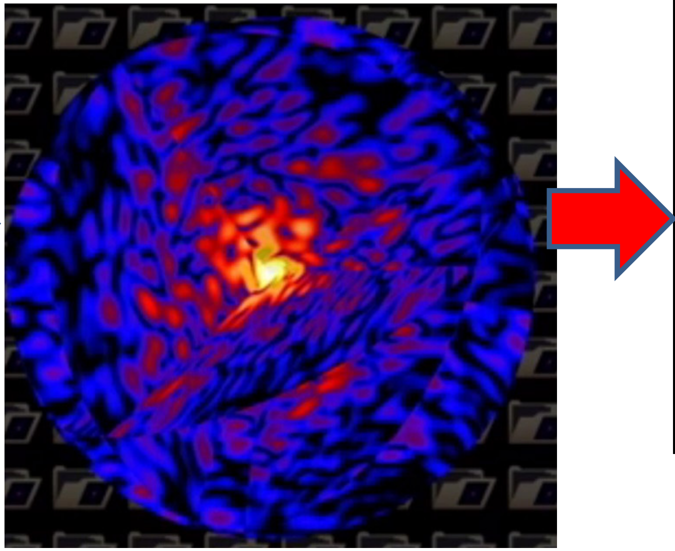


Single pattern

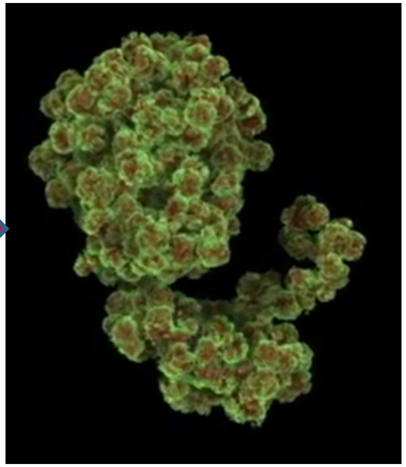
1. **Acquisition of scattering patterns**
2. **Classification and accumulation**
3. **Distribution in 3D reciprocal space**
4. **Reconstruction of structure in real space**



(accumulated) orientation classes



Intensity in reciprocal space



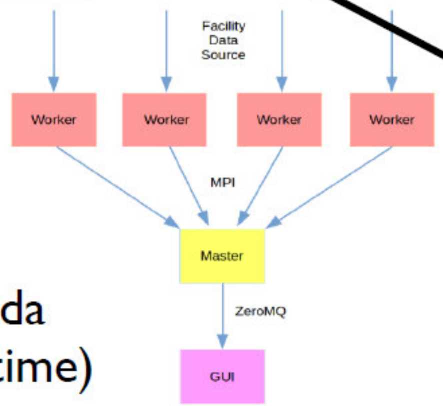
Electron density
in real space

SFX Data Processing Stages

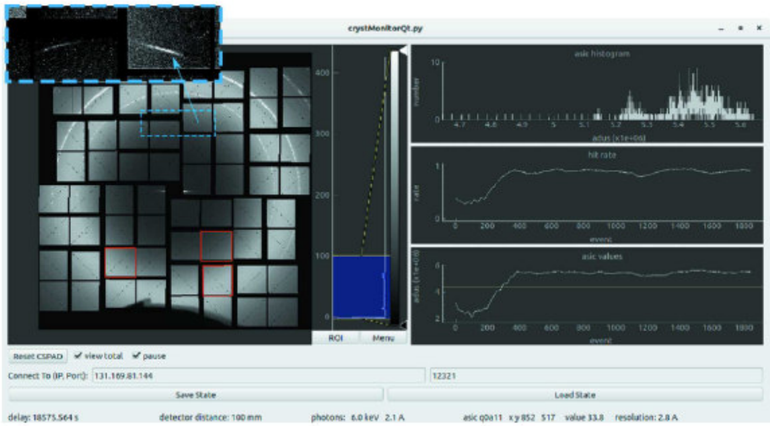
Steve Aplin, Anton Barty

On site → On site → Off site

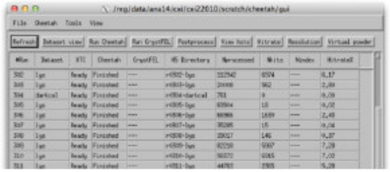
European XFEL



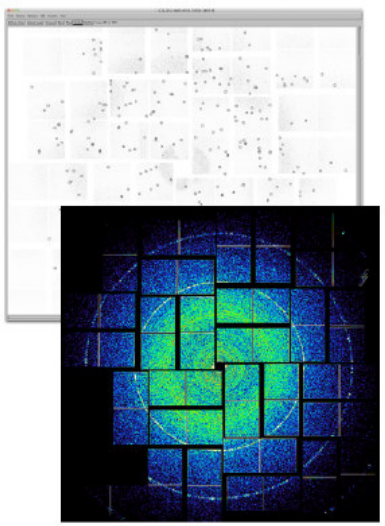
Onda
(real time)



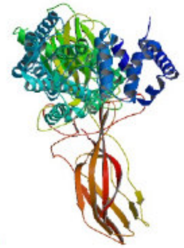
Valerio Mariani



Cheetah
Full analysis and
data reduction



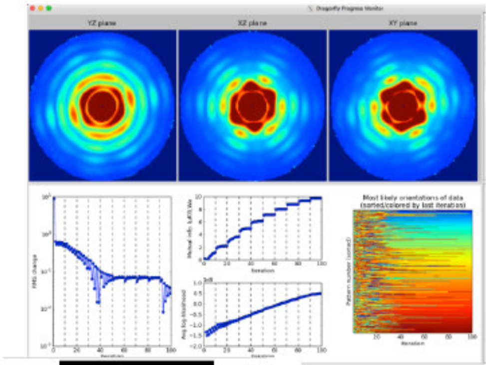
Anton Barty, Oleksandr Yefanov

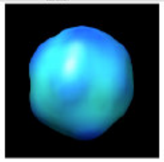


CrystFEL
(SFX analysis)

Tom White

Single particle imaging

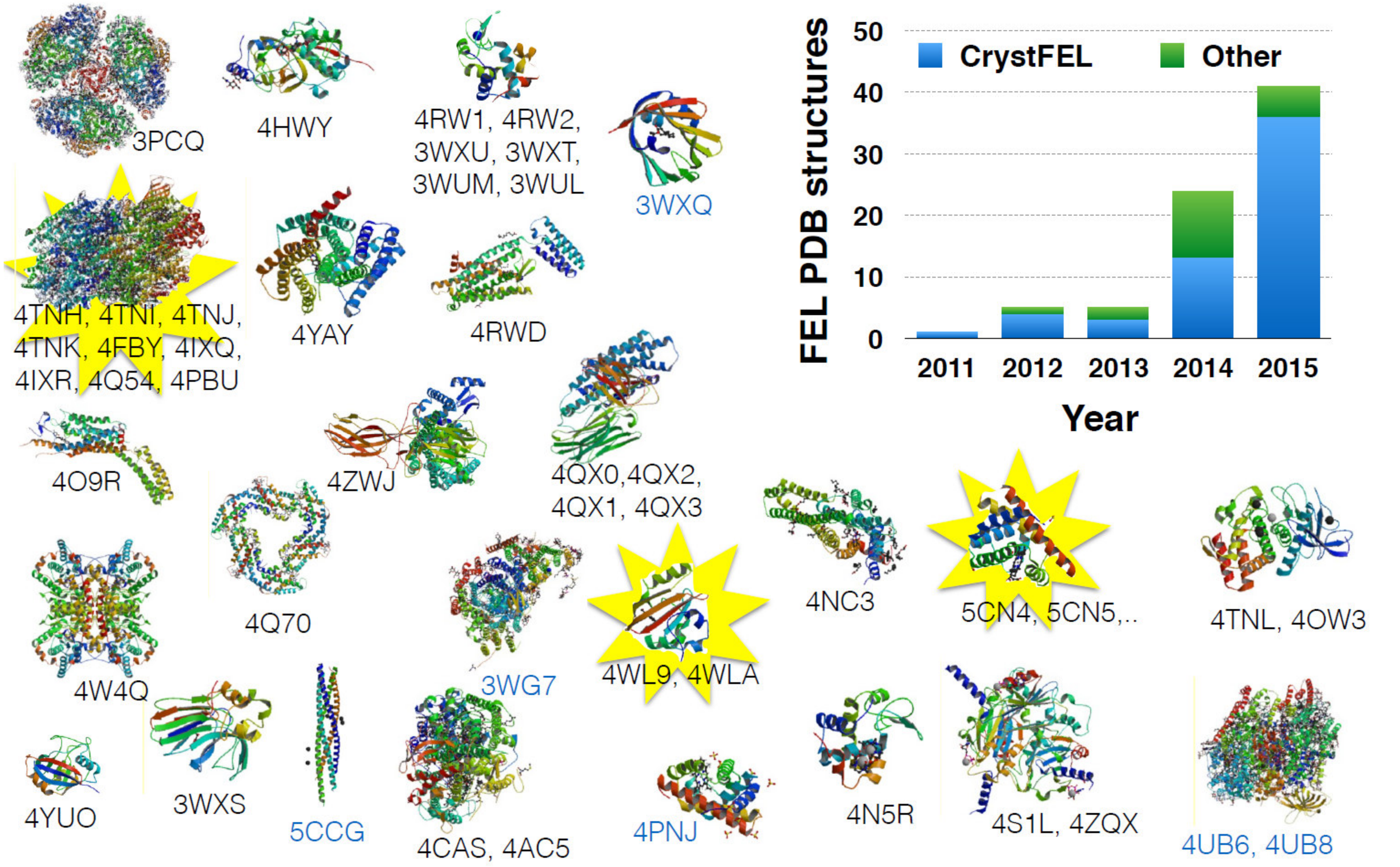




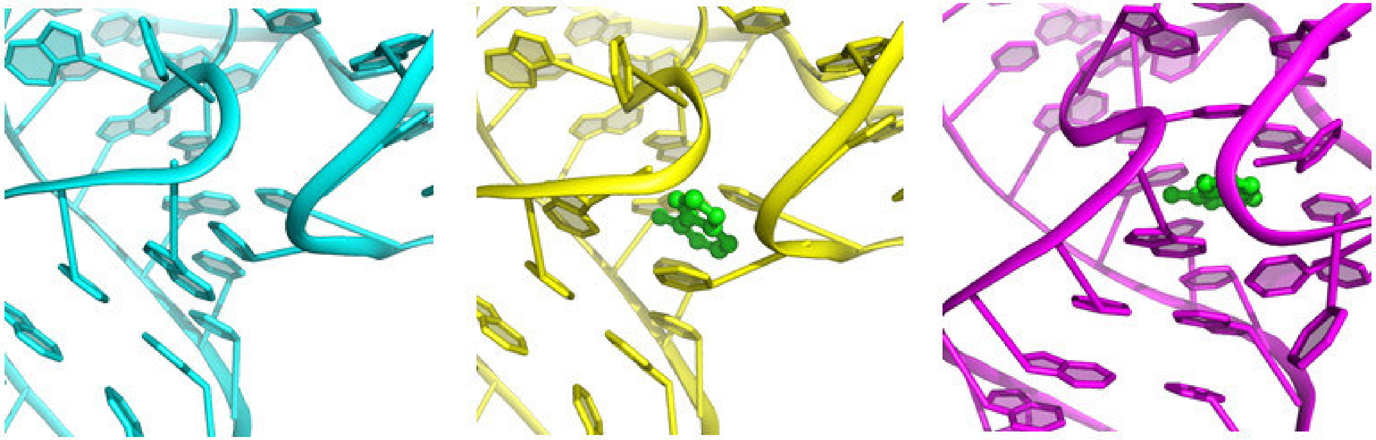
Kartik Ayyer

Andrew Morgan

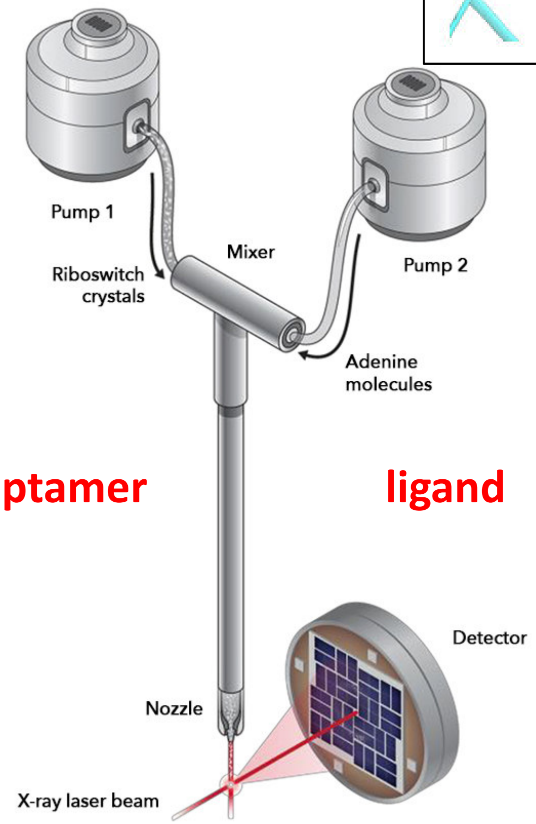
FEL Depositions in the Protein Databank (>200 in 2015)



A Riboswitch at Work



Mix and inject concept



Riboswitch from the bacterium **Vibrio vulnificus**
(a close relative of the cholera germ)
Active centre is aptamer = sequence of nucleic acids (easy to synthesize into nanocrystals

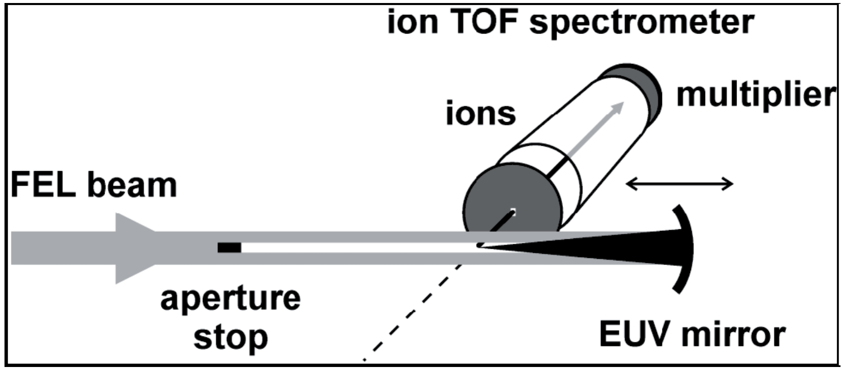
Activated by signal molecule (ligand) Adenine (in green)

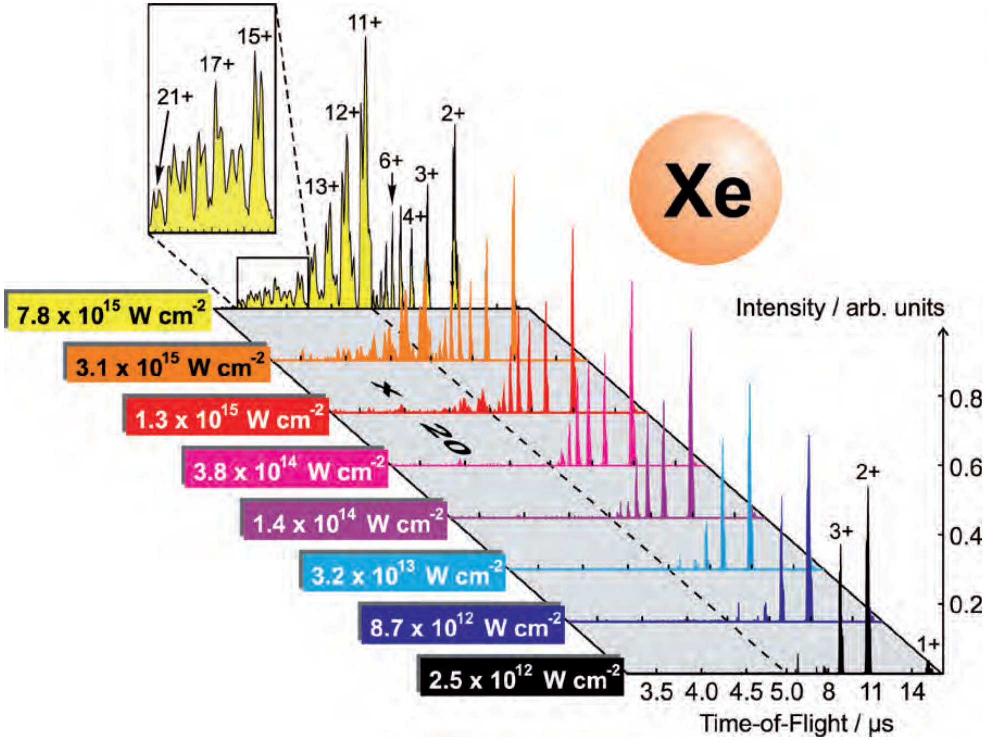
After activation the gene related to the switch in not read out anymore

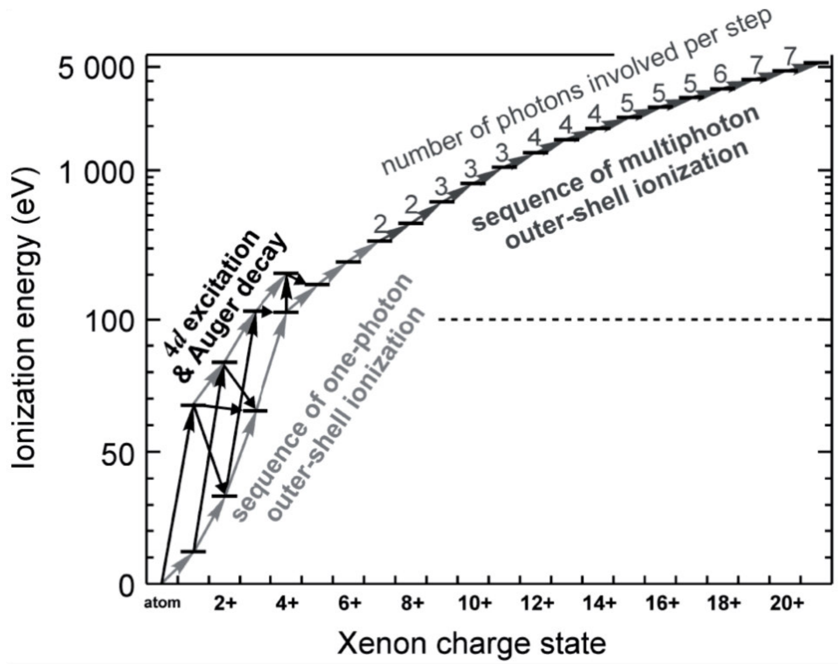
- Delay adjustment allows to follow intermediate states of reaction
- Tiny crystals are required , larger crystals would decompose upon the involved conformational changes and ligand diffusion would be too slow and uneven

Yun-Xing Wang, Nature (2014)

Photoelectric Effect at Ultrahigh Intensities (FLASH, 13.5 nm]







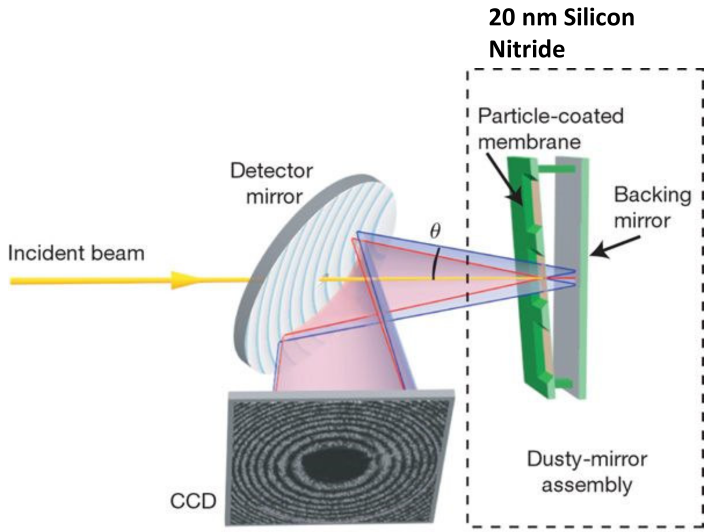
13.5 nm ≡ 91.8 eV

Dramatic changes in the ion charge state at high power densities

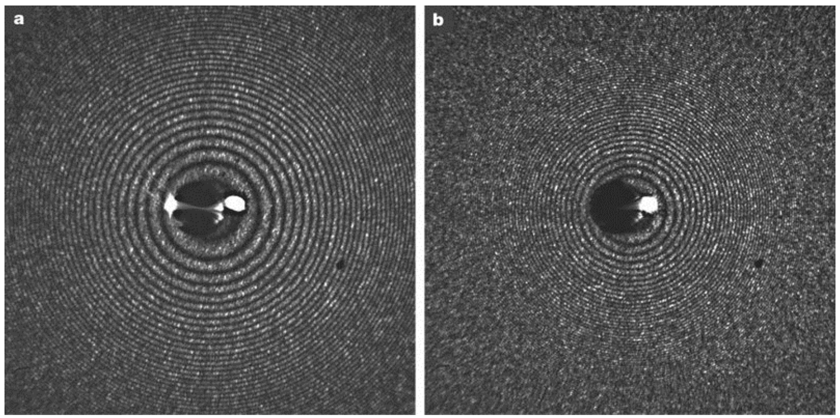
One atom has to absorb more then 50 photons

Phys. Rev. Let. 99, 213002 (2007)

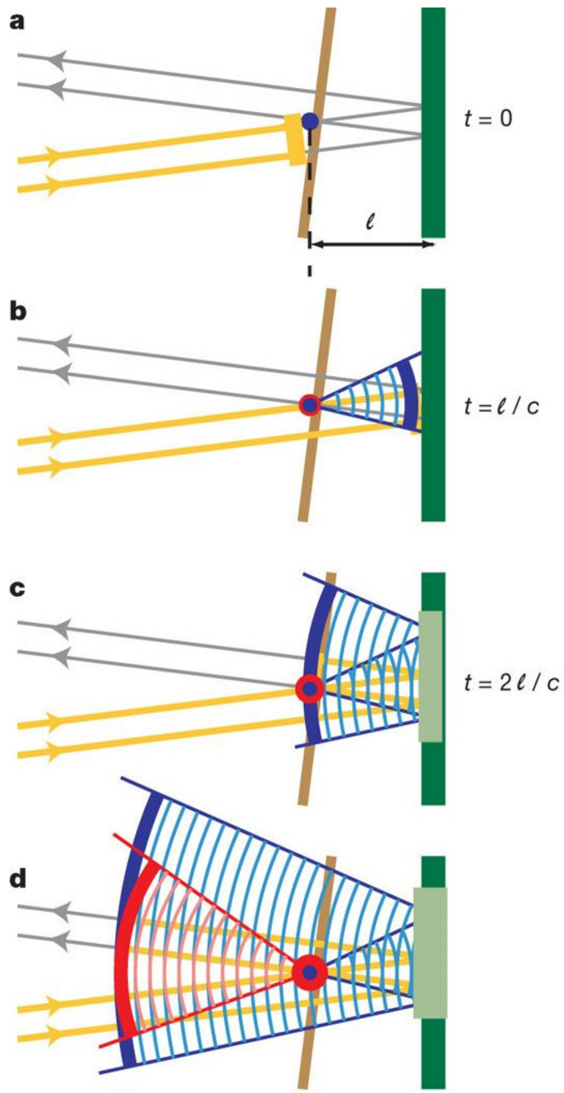
Femtosecond time-delay X-ray holography



25 fs pulses at λ = 32 nm, $0.5\cdot10^{14}$ W cm^{-2}



Δt=348 fs　　Δt=733 fs



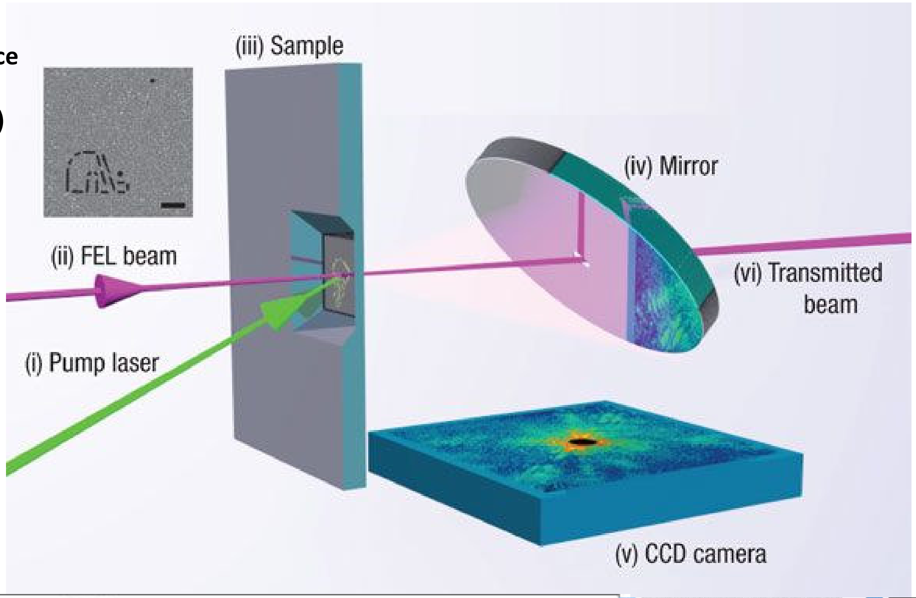
H. Chapman et. al., Nature **volume 448**, pages 676–679 (09 August 2007)

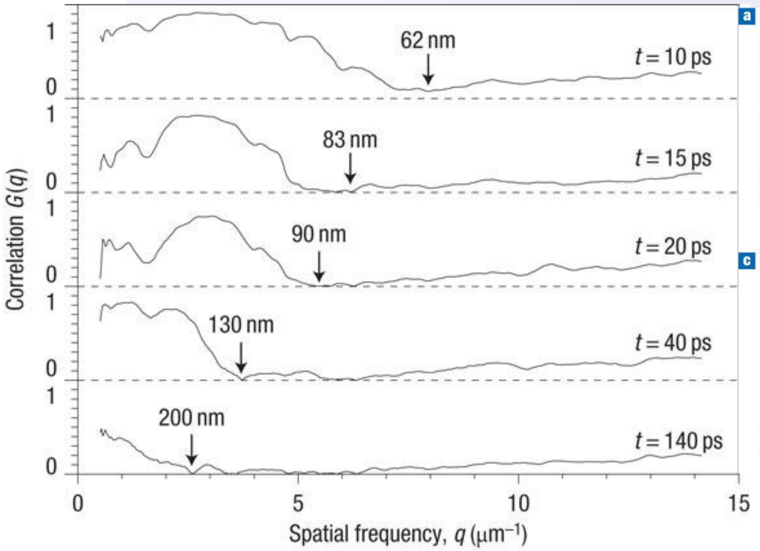
Pump Probe Experiment

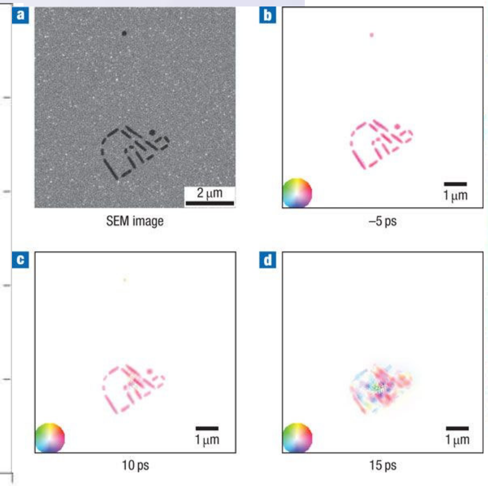
Reference Object (Sample)

13.5 nm

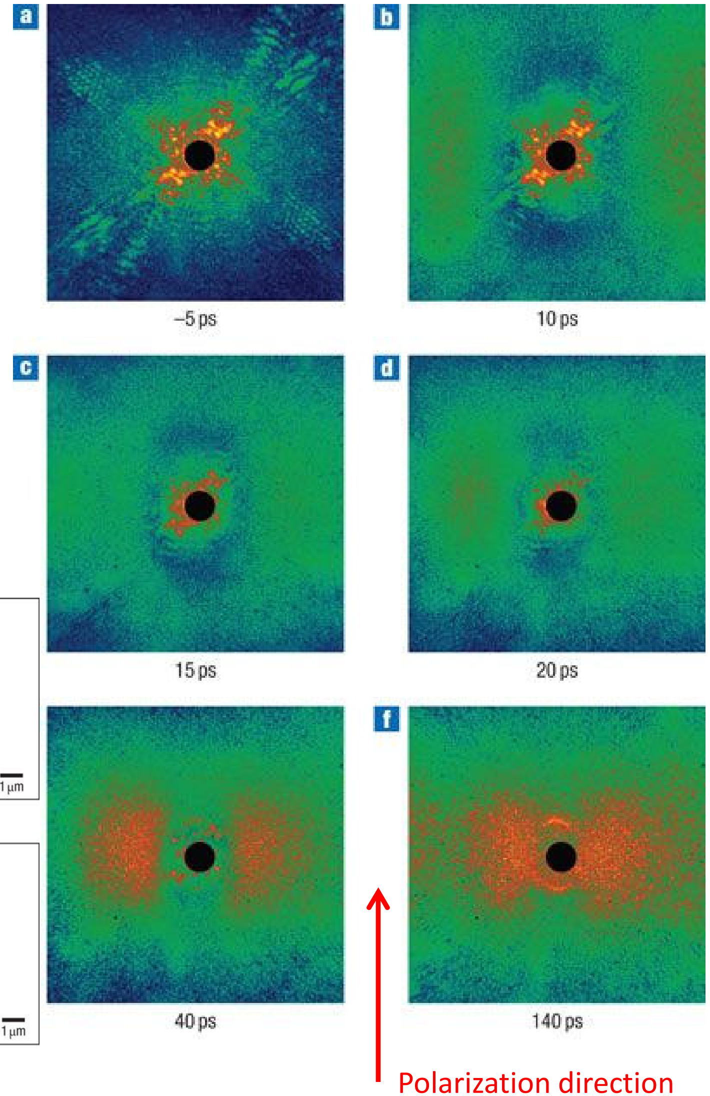
523 nm







Reconstructed images (phase retrieval)



Polarization direction

Correlation between actual and initial pattern quantifies progressive loss of mesoscale order (explosion speed about 5000 m·s^{-1})

A. Barty et. al., Nature Photonics **volume 2**, pages 415–419 (2008)