

Development of Experimental Techniques: Sharp Views into the Nano Cosmos

Christian G. Schroer

DESY & Universität Hamburg



HELMHOLTZ

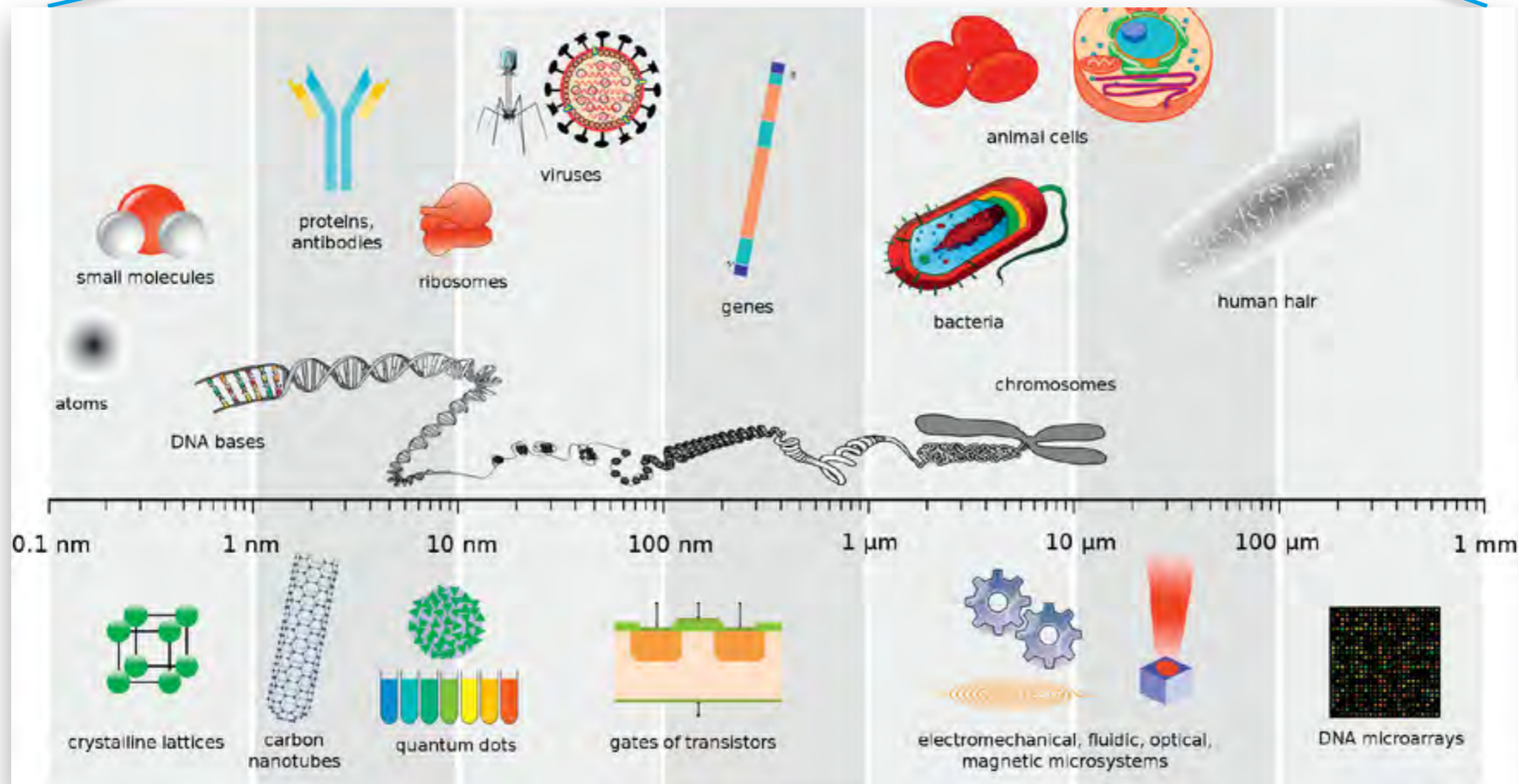
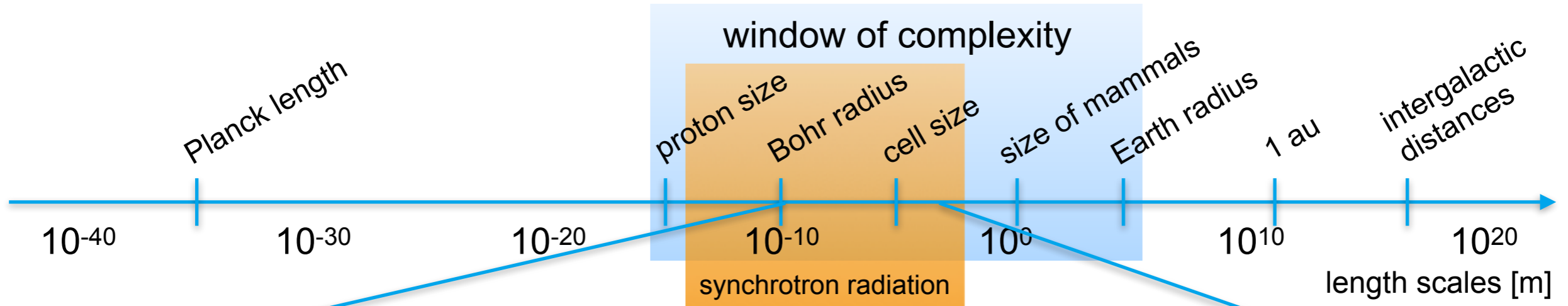
RESEARCH FOR GRAND CHALLENGES



Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG



Complexity in Nature: Characteristic Length Scales



What we need to do:

Quantitative in-situ measurement of physical properties of matter

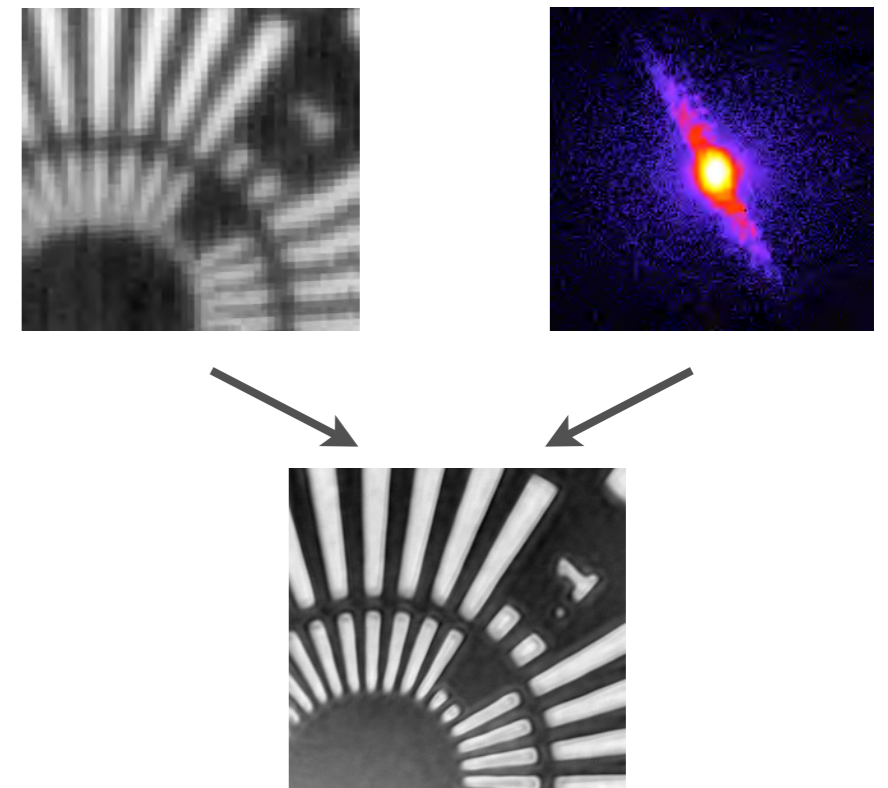
- > on all relevant length scales → (in principle) from Å to millimeters
- > on all relevant time scales

Key technology: bright, coherent x-rays with time structure

Requirements:

- > high coherent flux
 - x-ray free-electron lasers
 - diffraction-limited storage rings (PETRA IV)
- > efficient nanofocusing
 - optics
- > stability on nanometer scale

Fusion of real and reciprocal space!



DESY: Accelerator-Based Light Sources

Cooperation partners
UHH · MPG · EMBL · HZG
CSSB partner institutes
Sweden · India · Russia



X-Ray Free-Electron Laser
fs dynamics of complex matter
on the atomic scale

CHyN

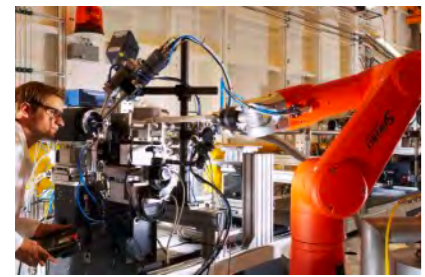
HARBOR

MPI-SD

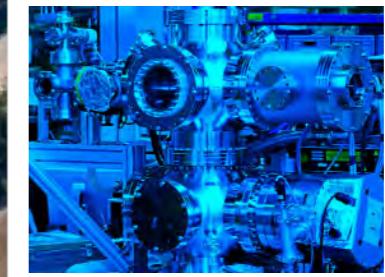


CSSB
Centre for Structural
Systems Biology

CXNS
NanoLab



PETRA III
**Synchrotron Radiation
of Highest Brightness**
atomic structure of
complex matter



FLASH
**VUV & Soft X-ray
Free-Electron Laser**
fs dynamics of complex
matter (spectroscopy)

DESY's bright synchrotron radiation source

History of PETRA:

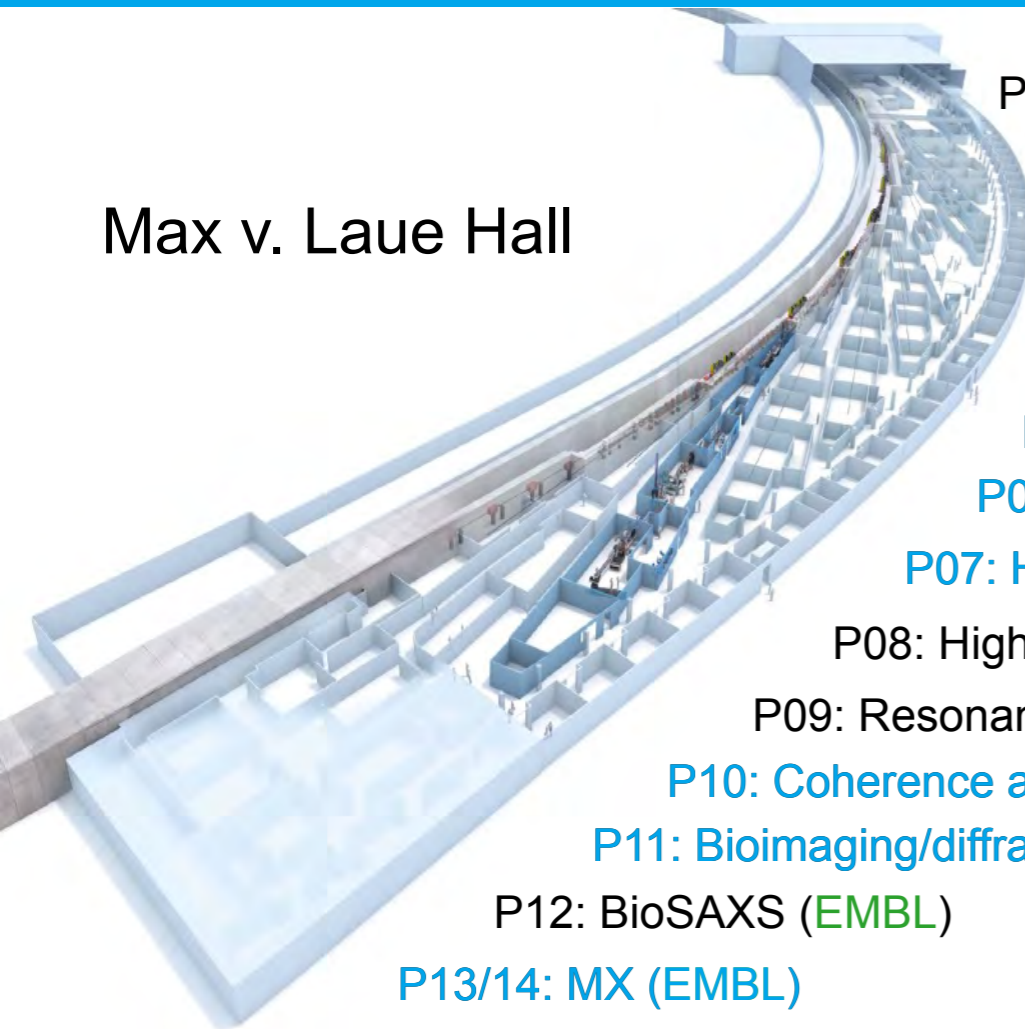
- > 1978 built for high-energy physics, first direct observation of the gluon, since 1988 pre-accelerator for HERA
- > starting July 2007: rebuilding PETRA as a synchrotron radiation source (PETRA III)
- > Sept. 2010: start of user operation with the first three beamlines
- > End of 2013: all 15 beamlines fully operational in Max v. Laue Hall
- > Mar. 2014 - Apr. 2015: Shutdown for extension project after the DORIS III shutdown
- > 2016: First beamlines in the extension operational



- > electron energy: **6 GeV**
- > stored current: **100 mA (top-up)**
- > emittance: **1.2 nm rad**
- > circumference: **2304 m**
- > photon energy range: **250 eV — 150 keV**
- > beamlines in operation: **21**
- > beamlines under construction: **3**
- > beamlines in planning: **2**
- > user operation (hours/year): **5000 h (4000 h)**

PETRA III Beamlines

Max v. Laue Hall



P01: Dynamics beamline, IXS, NRS

P02.1: Powder diffraction & total scattering

P02.2: Extreme conditions

P03: Micro-, nano-SAXS, WAXS

P04: Variable polarisation XUV

P05: Micro-, nano-tomography (HZG)

P06: Hard X-ray micro-, nanoprobe

P07: High-energy materials sci. (HZG, DESY)

P08: High-resolution diffraction

P09: Resonant scattering/diffraction

P10: Coherence applications

P11: Bioimaging/diffraction

P12: BioSAXS (EMBL)

P13/14: MX (EMBL)

Imaging

P21: Swedish materials science beamline

P22: Hard X-ray photoelectron spectroscopy

P23: In-situ and nano diffraction beamline

P24: Chemical crystallography

P25: HIMAX, NRS (in planning)




Ada Yonath Hall

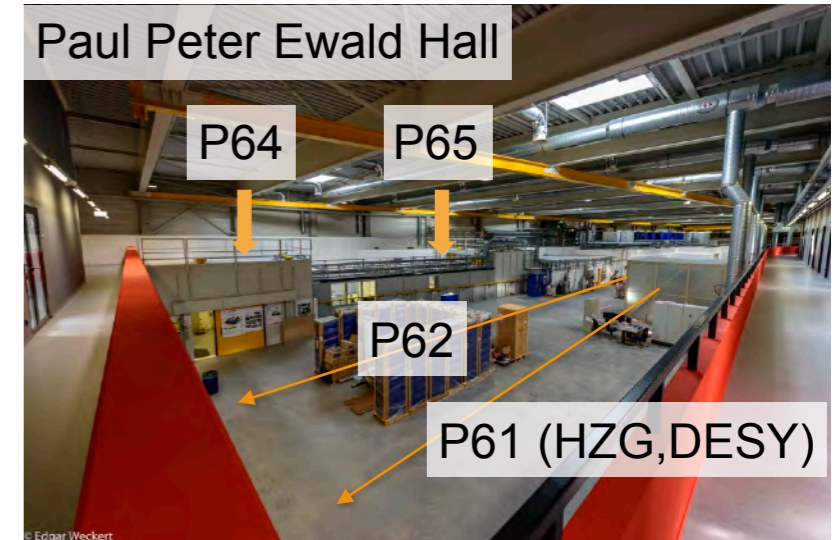
P22 

P21 

P24

P23 

Verbundforschung



Paul Peter Ewald Hall

P64

P65

P62

P61 (HZG, DESY)

P61: High-energy wiggler beamline (HZG, DESY operational 2019)

P62: Small-angle X-ray scattering (under construction)

P63: MPG catalysis (in planning)

P64: Advanced XAFS

P65: Applied XAFS

P66: Time-resolved luminescence spectroscopy (operational 2021)



X-ray Scanning Microscopy

Broad field of applications:

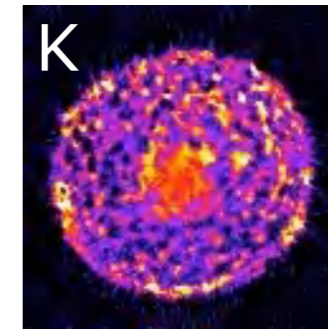
- > Main advantage: large penetration depth
 - *in-situ* and *operando* studies
 - 3D bulk analysis without destructive sample preparation
- > X-ray analytical contrasts: XRD, XAS, XRF, ...
 - elemental, chemical, and structural information

Today: „mesoscopic gap“

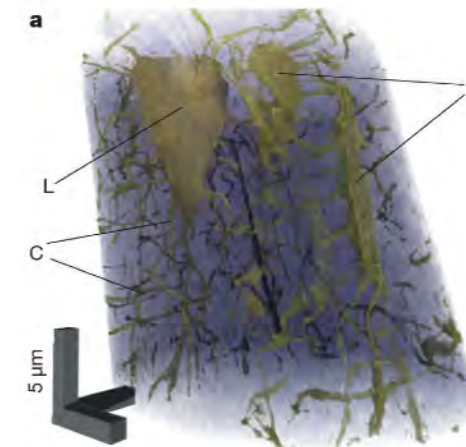
real-space resolution: down to about 10 nm

XRD and XAS: atomic scale

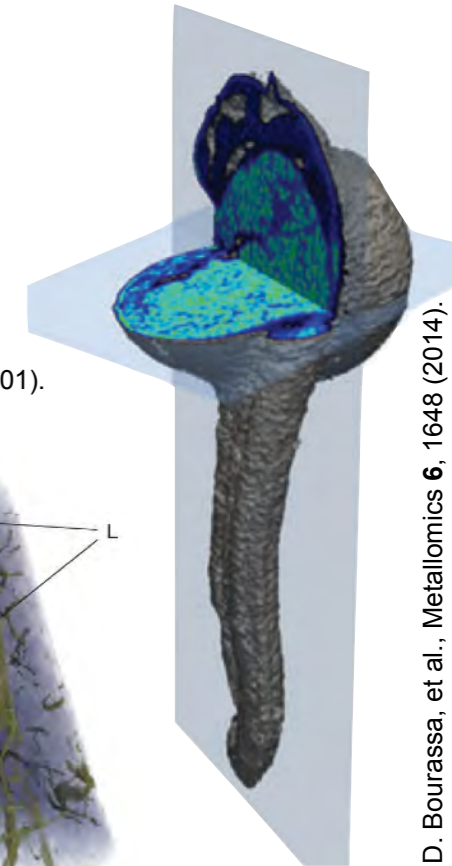
Many interesting physics and chemistry (e. g. catalysis)
at the 1 - 10 nm scale!



C. G. Schroer, APL **79**, 1912 (2001).

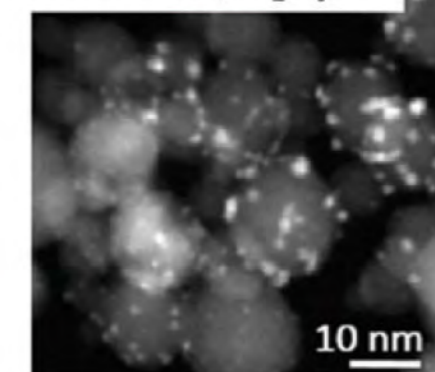


M. Dierolf, et al., Nature **467**, 436 (2010).



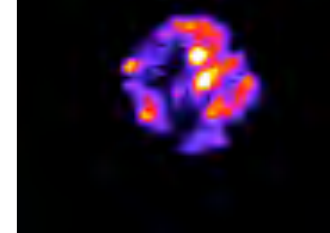
D. Bourassa, et al., Metallomics **6**, 1648 (2014).

2.5%Pt-2.5%Rh/Al₂O₃



catalysts

Cu(I)₂O



C. G. Schroer, et al., APL **82**, 3360 (2003).

X-ray Microscopy

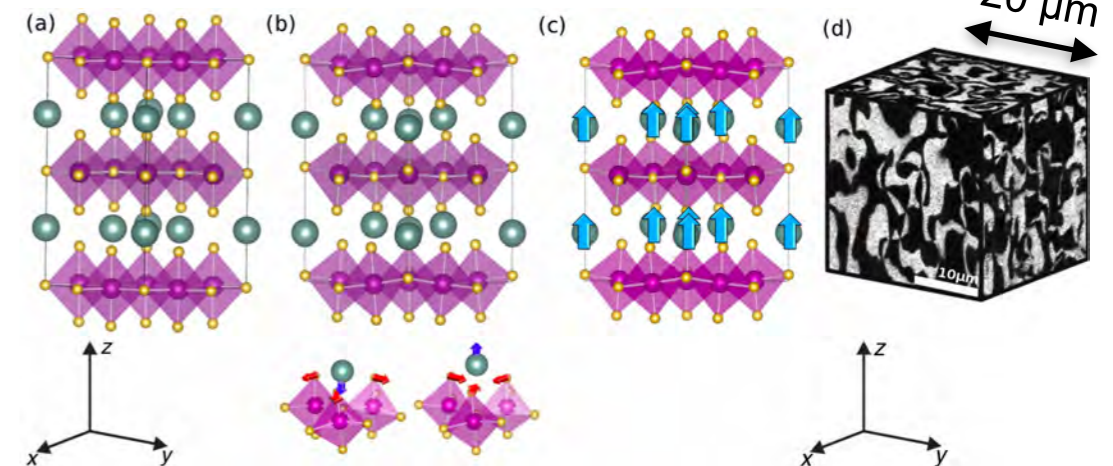
Many interesting physics and chemistry questions:

investigate local states:

- > individual defects (0D): changes in electron density, charge ordering
- > (structural) domain boundaries (2D), e. g., in multiferroics
- > mesoscopic dynamics at (solid-state) phase transitions
- > catalytic nanoparticles (under reaction conditions)
- > ...

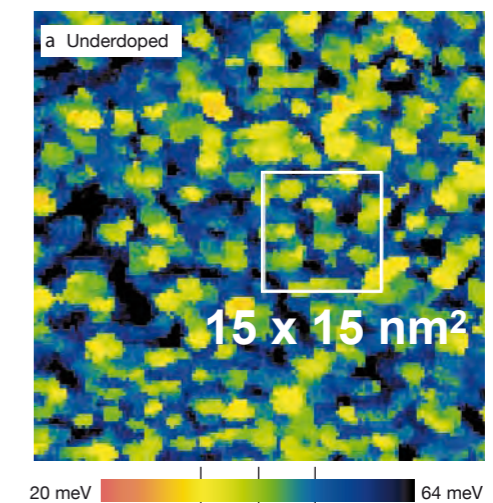
Mesoscale also very important for nanotechnology (e. g., defects in devices)!

ferroelectric phase transition



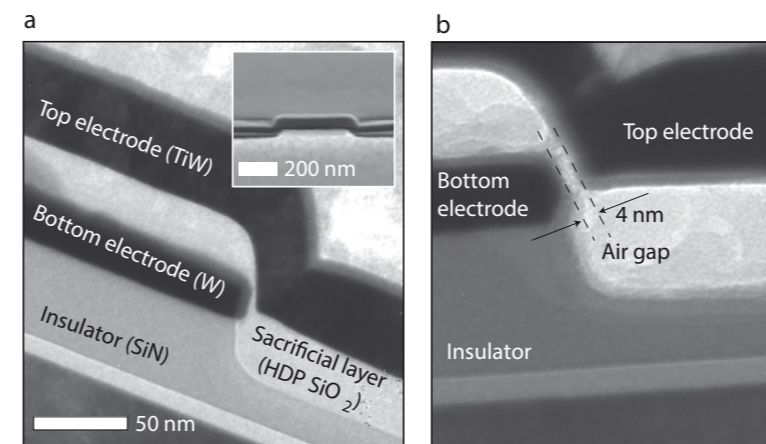
Griffin, et al., PRX **2**, 041022 (2012).

variation of supercond. gap



Lang, et al., Nature **415**, 412 (2002).

nanoelectromechanical switch



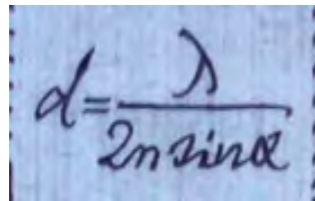
Lee, et al., Nature Nanotech. **8**, 36 (2012).



Current State of X-Ray Microscopy

Conventional x-ray microscopy

- optics limit spatial resolution: diffraction limit


$$d = \frac{\lambda}{2n \sin \alpha}$$

(typically: a few tens of nanometers)

optics are technology limited!

Theoretical extrapolation of x-ray optical performance to the atomic level.

[PRB **74**, 033405 (2006); H. Yan, et al., PRB **76**, 115438 (2007)]

Coherent x-ray imaging techniques (CXDI, ptychography)

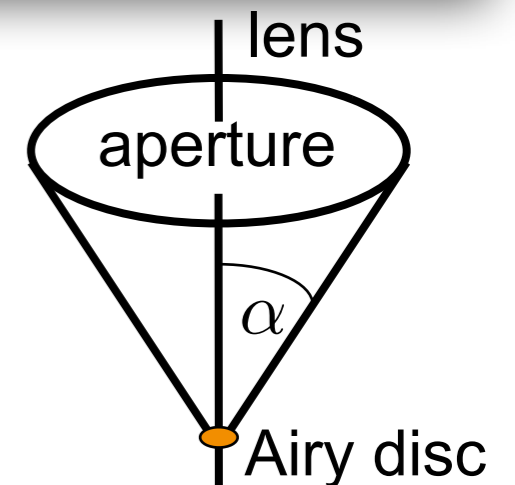
- no imaging optics needed!
- limited by statistics of far-field diffraction patterns ...

highest resolution: a few nanometers, focusing coherent beam

[PRL **101**, 090801 (2008); Y. Takahashi, et al., PRB **80**, 054103 (2009);
A. Schropp, et al., APL **100**, 253112 (2012)]

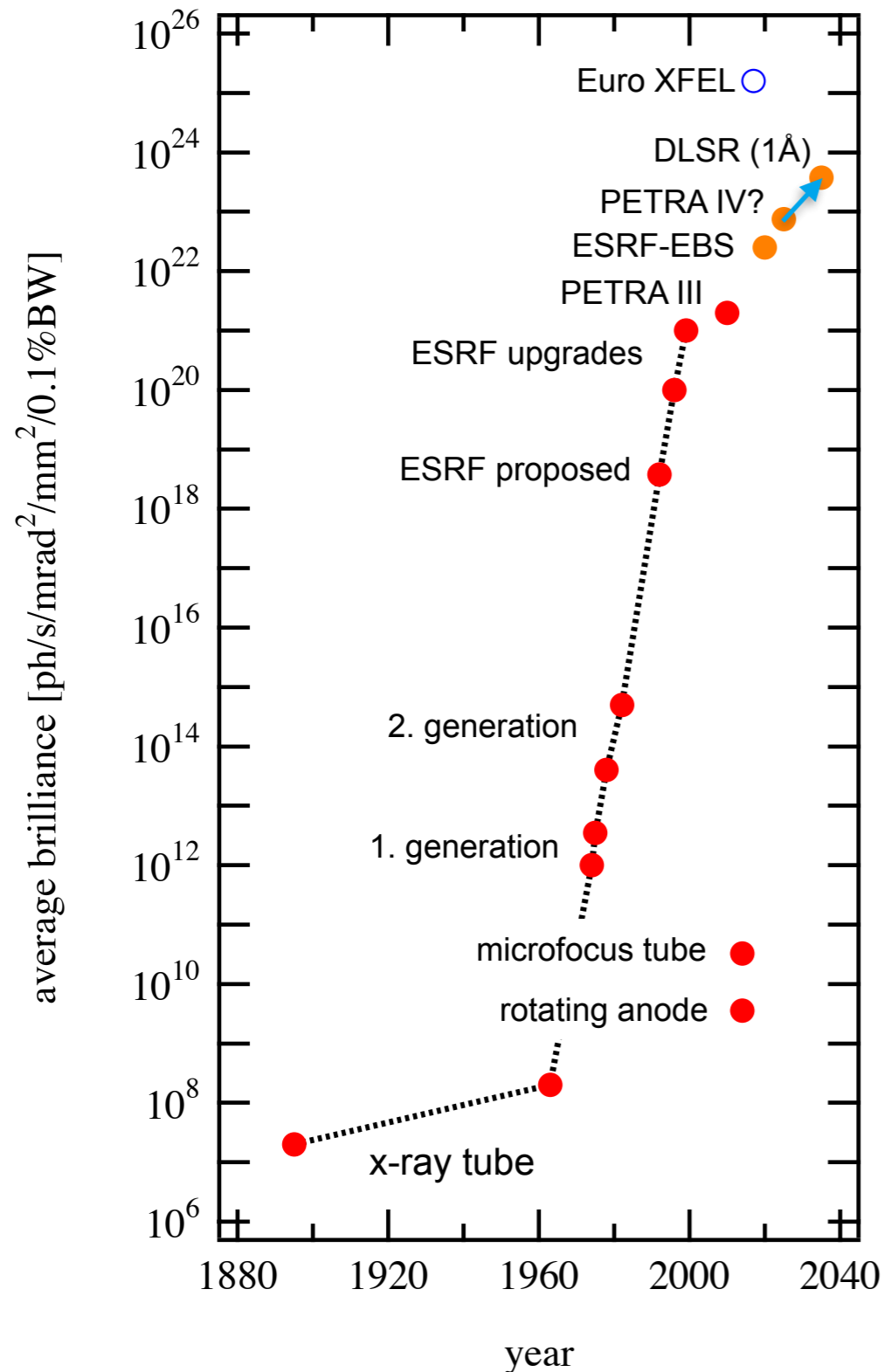


Ernst Abbe



Spectral Brightness

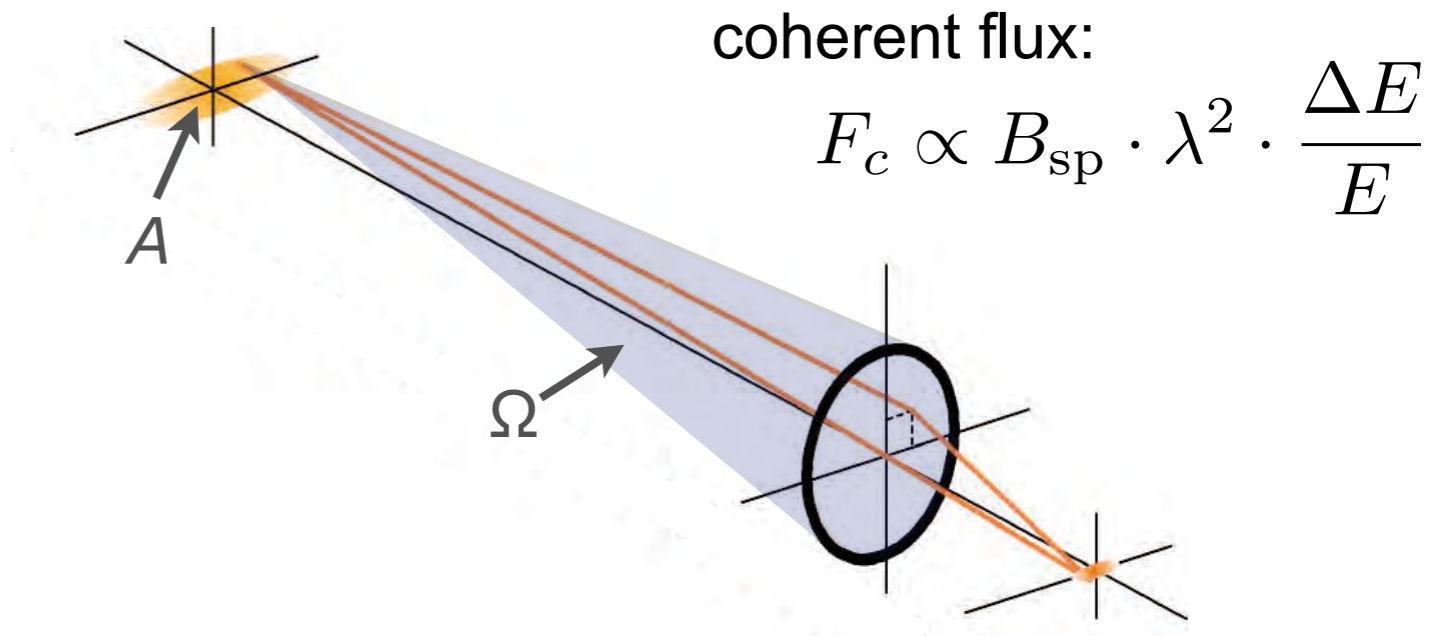
10000x more “light” per decade (since 1965)!!



Spectral brightness:

$$B_{\text{sp}} = \frac{F}{\Omega \cdot A \cdot \Delta E / E}$$

Flux per phase-space volume



$$F_c \propto B_{\text{sp}} \cdot \lambda^2 \cdot \frac{\Delta E}{E}$$

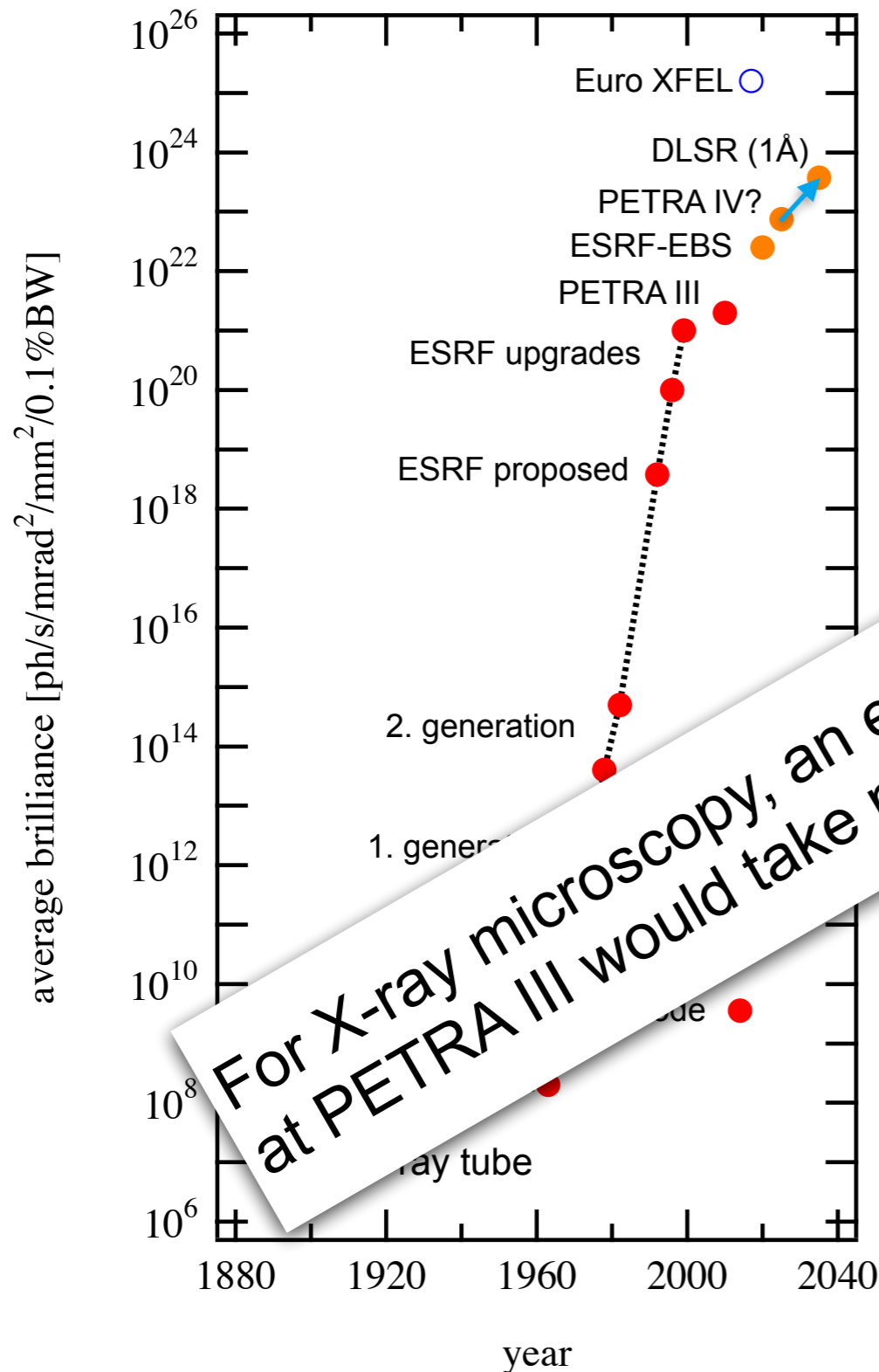
Improvements in brightness:

- > faster measurements (time resolution)
- > nano-imaging (spatial resolution)
- > spectroscopy (energy resolution)



Spectral Brightness

10000x more “light” per decade (since 1965)!!



Spectral brightness:

$$B_{sp} = \frac{F}{\Omega \cdot \lambda^2 \cdot \frac{\Delta E}{E}}$$

Flux per phase

incident flux:

$$F_c \propto B_{sp} \cdot \lambda^2 \cdot \frac{\Delta E}{E}$$

Ω

Improvements in brightness:

- > faster measurements (time resolution)
- > nano-imaging (spatial resolution)
- > spectroscopy (energy resolution)



Nanofocusing Optics

reflection:

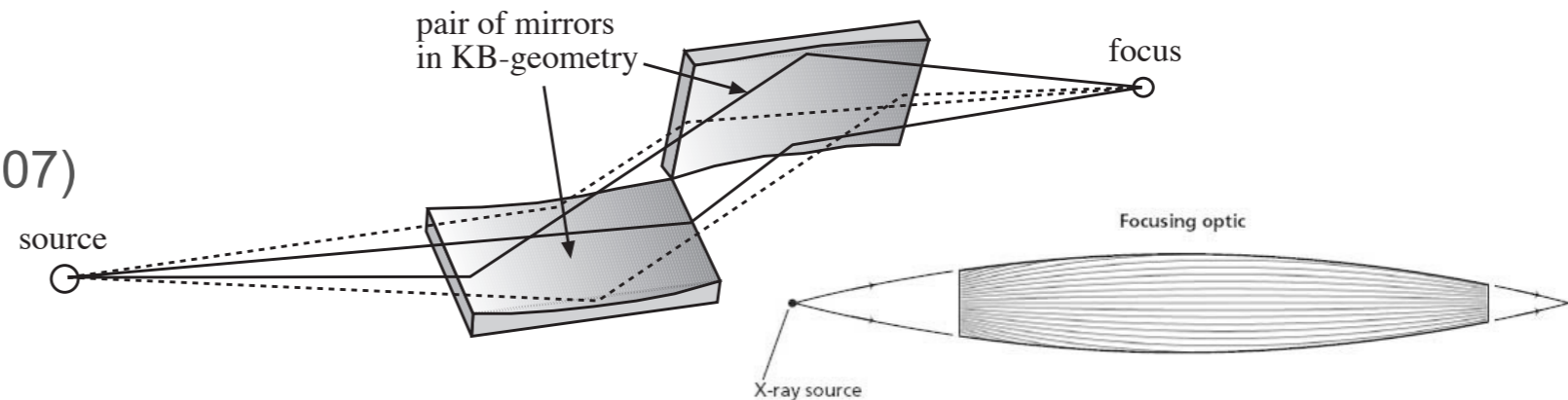
- > mirrors (25 nm)

H. Mimura, et al., APL **90**, 051903 (2007)

- > capillaries

- > wave guides (~10 nm)

S. P. Krüger, et al., J. Synchrotron Rad. **19**, 227 (2012)



diffraction:

- > Fresnel zone plates (< 10 nm)

J. Vila-Comamala, et al., Ultramic. **109**, 1360 (2009)

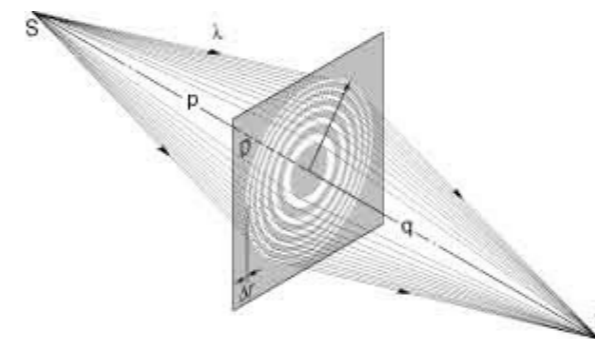
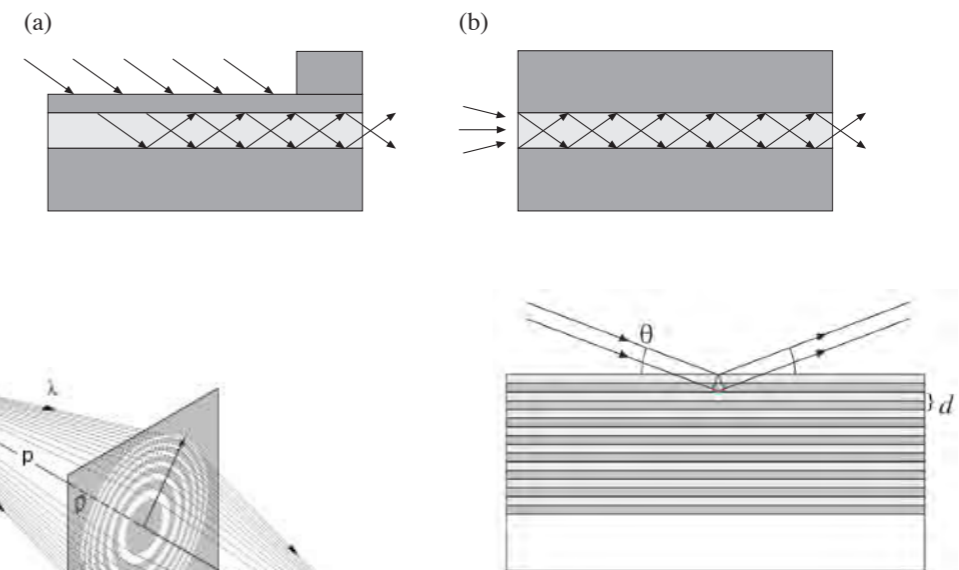
- > multilayer mirrors (7 nm)

H. Mimura, et al., Nat. Phys. **6**, 122 (2010)

- > multilayer Laue lenses (8 nm)

A. Morgan, et al., Sci. Rep. **5**, 09892 (2015)

- > bent crystals

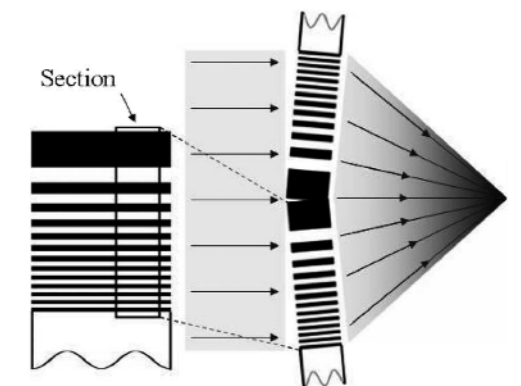
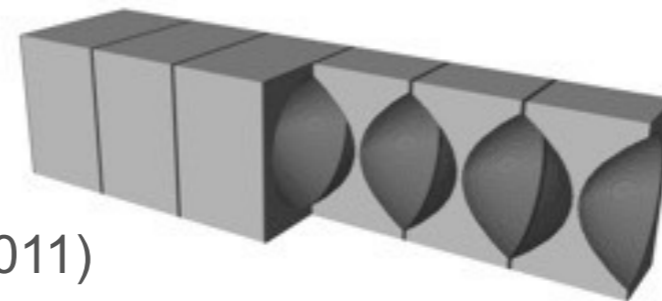


refraction:

- > lenses (43 nm, 18 nm)

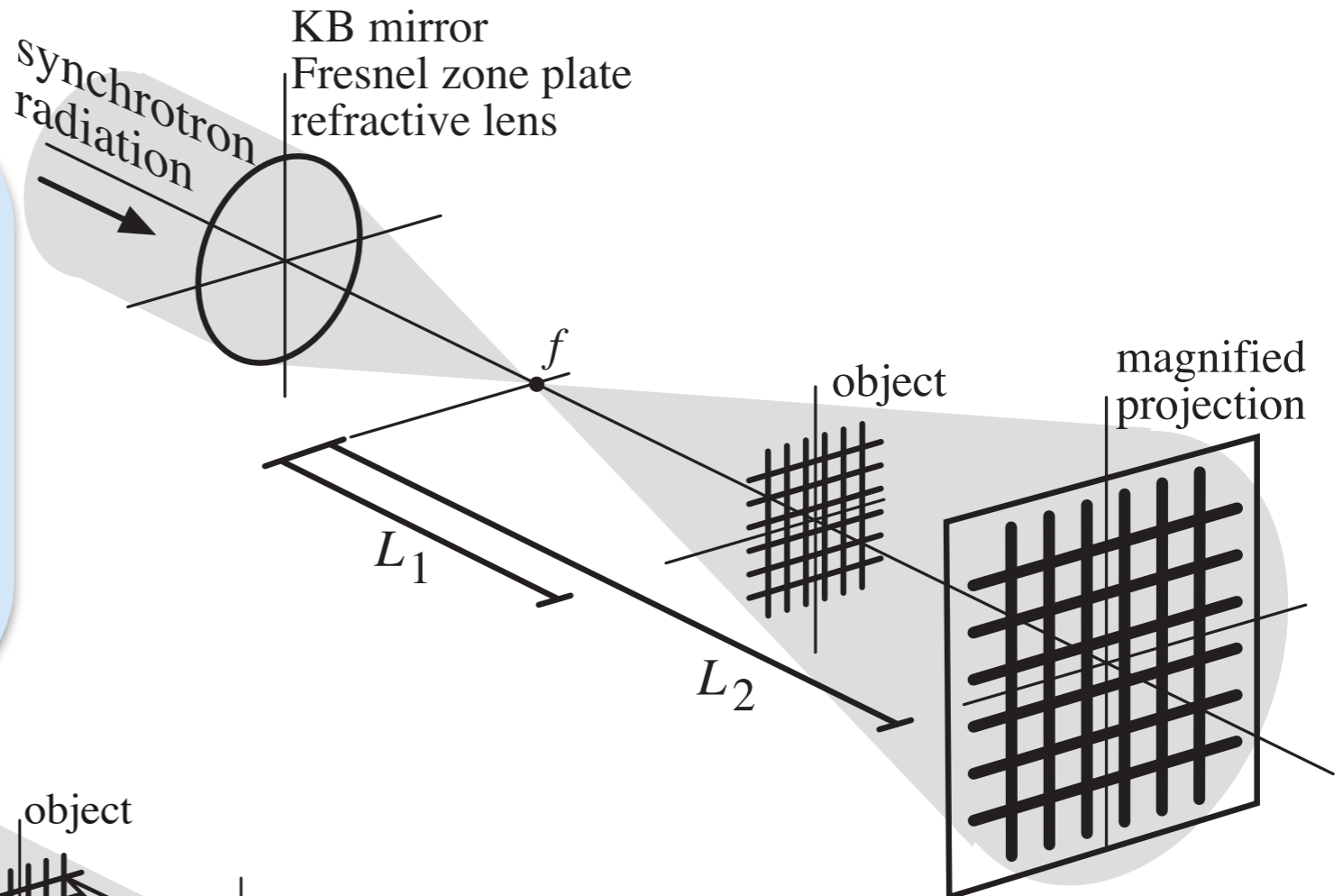
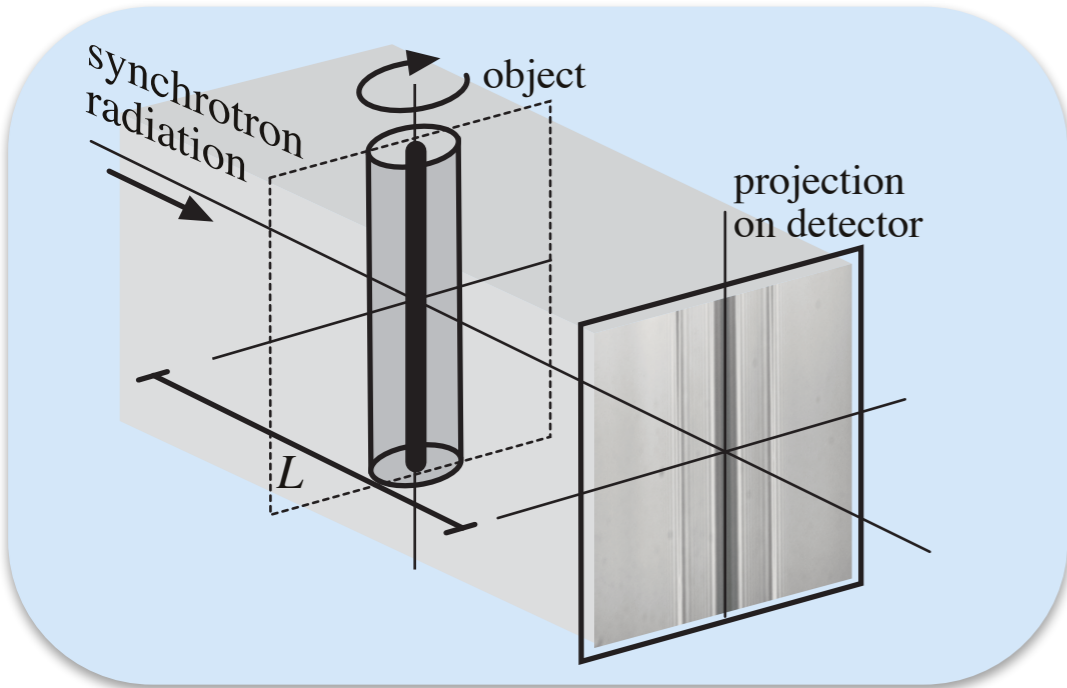
C. G. Schroer, et al., AIP Conf. Ser. **1365**, 227 (2011)

J. Patommel, et al., APL **110**, 101103 (2017)



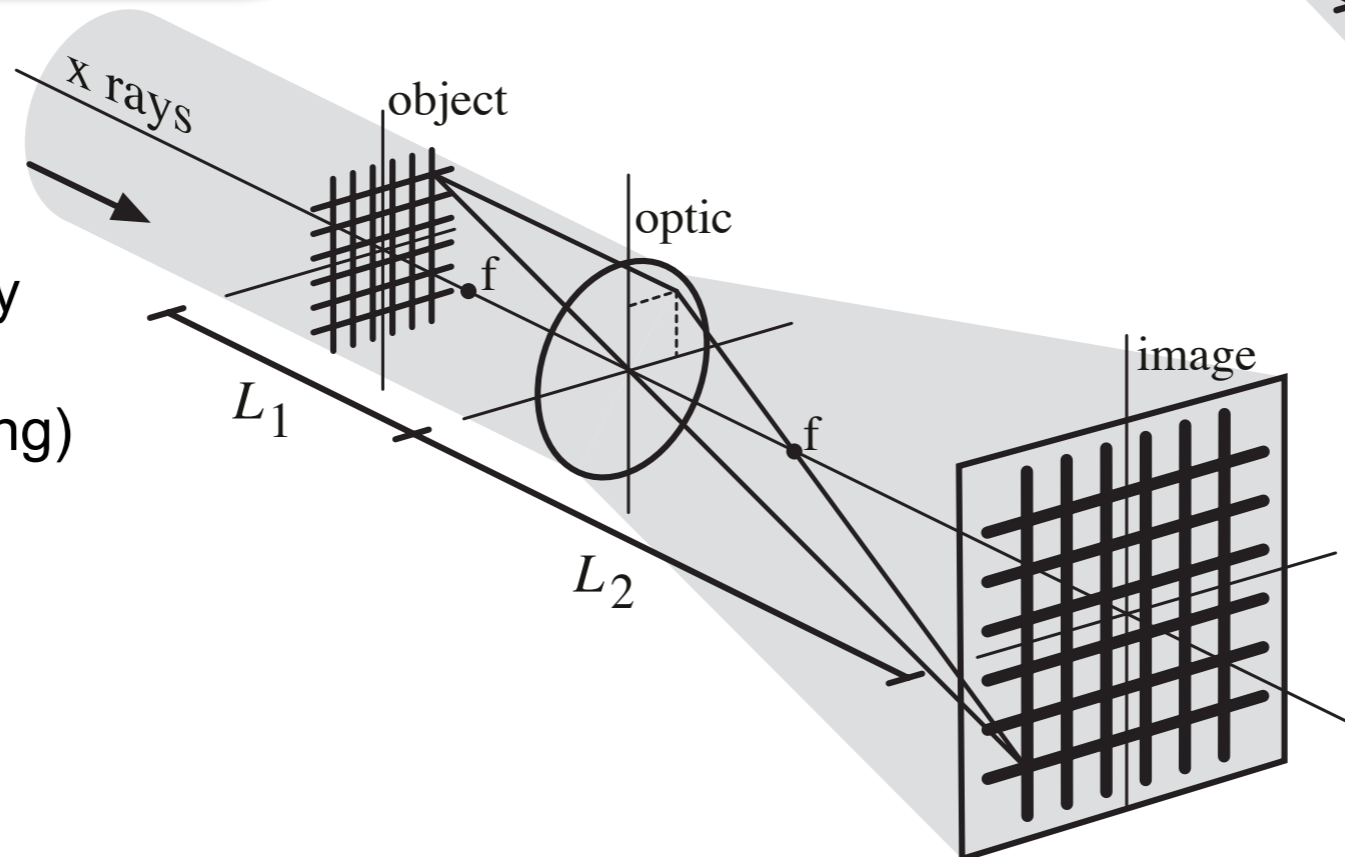
X-Ray Microscopy Techniques: Full-Field Imaging

Projection imaging:



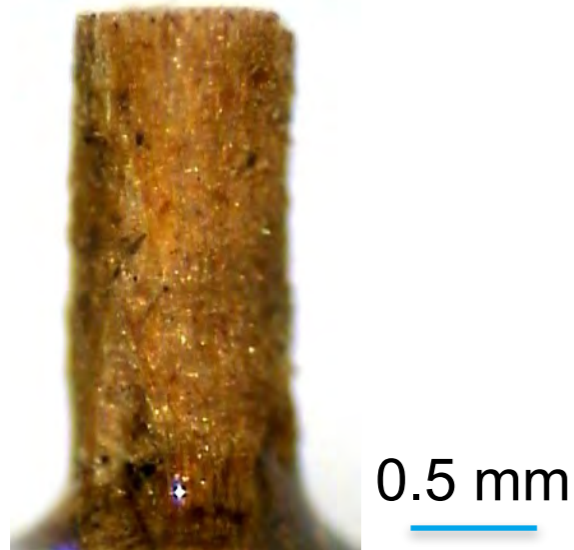
Imaging through objective lens:

x-rays focused by condenser (aperture matching)



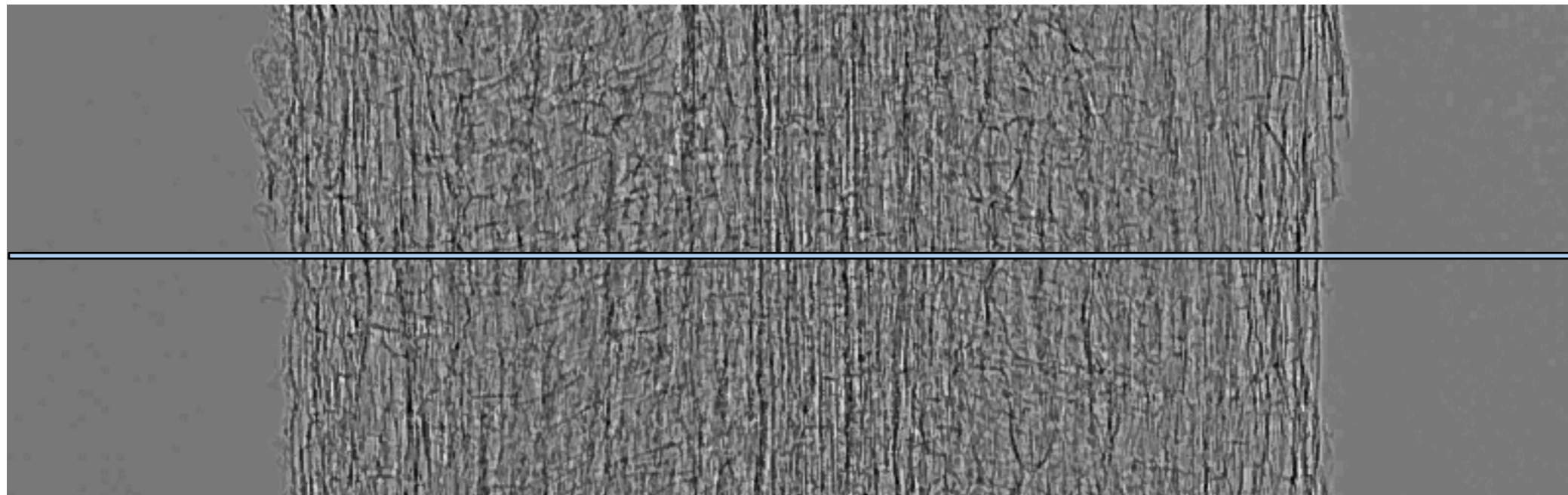
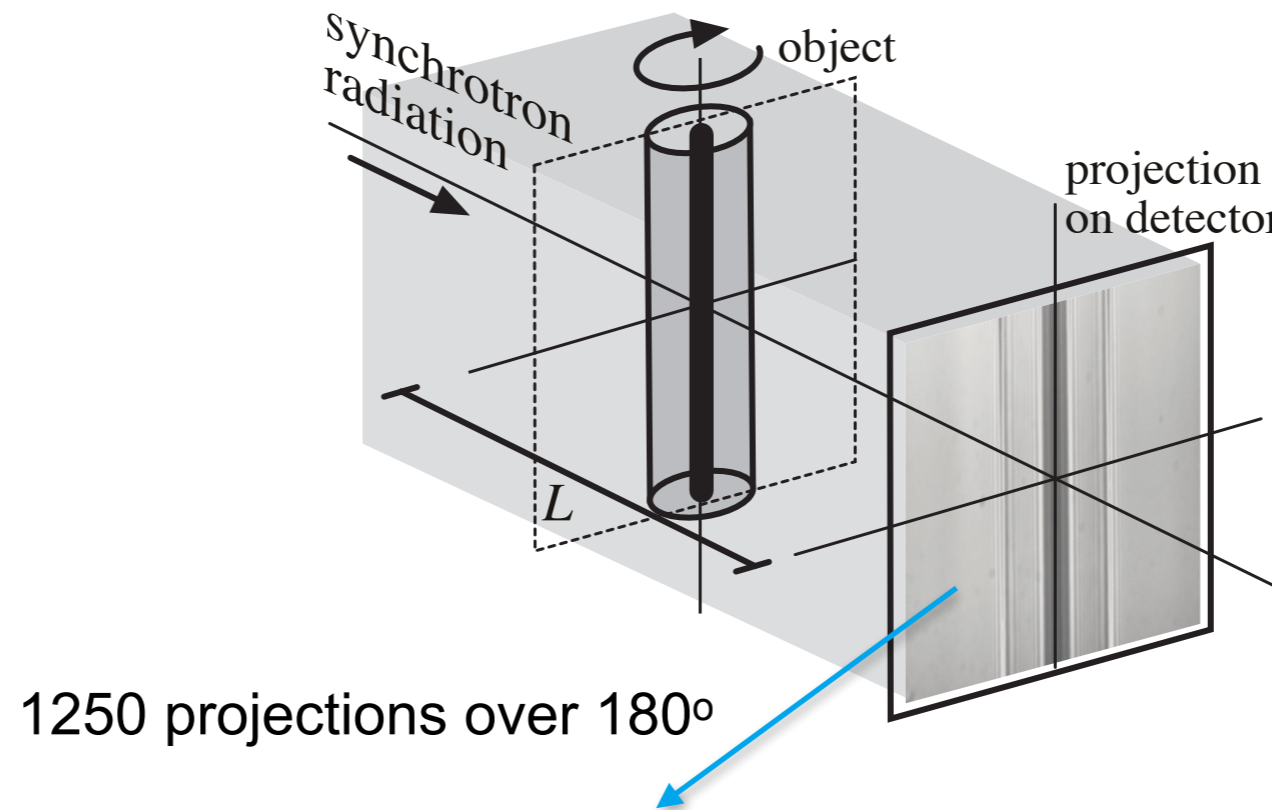
Example: Projection Imaging (Phase Contrast)

Example: plant physiology



root of mahogany tree
(W. H. Schröder, FZ Jülich)

Reveal inner structures of object:



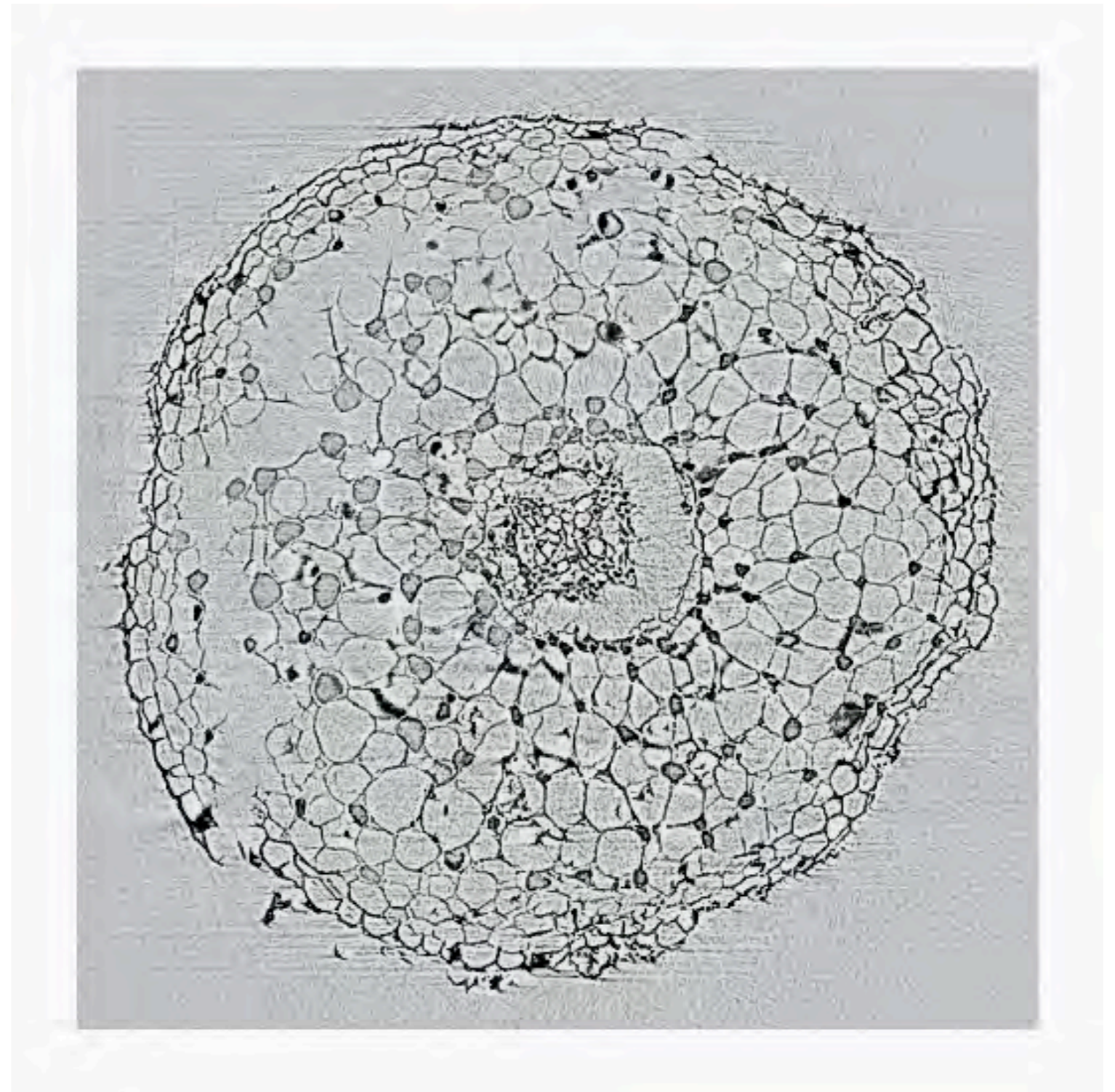
energy: 20 keV
 $L = 50$ mm
pixel size: 1.4 μ m

3D Reconstruction

Many slices:
3D structure



root of mahogany tree
(W. H. Schröder, FZ Jülich)



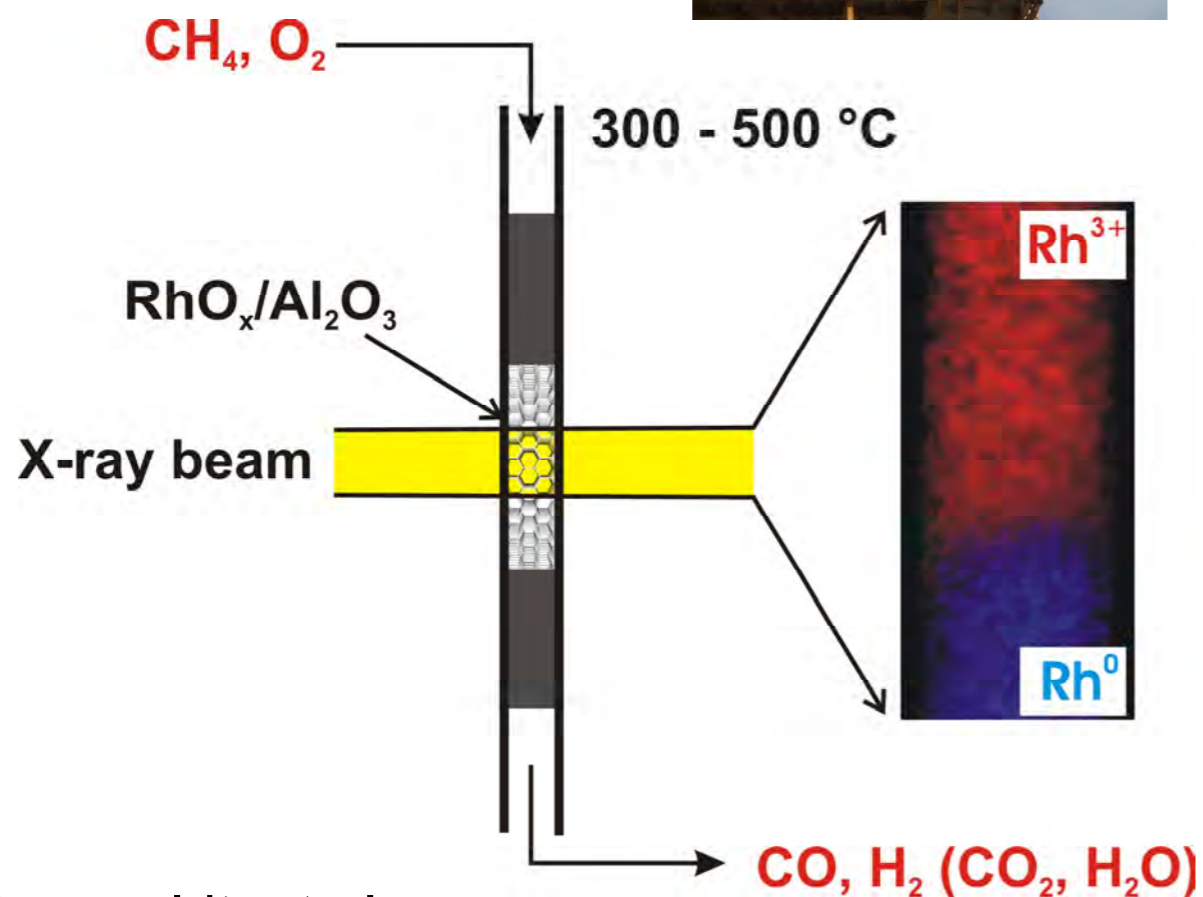
resolution: $\sim 3 \mu\text{m}$

Visualize Catalysts in Action

Methane often wasted during oil production:



First step to convert methane into liquid fuels (syngas production):



Grunwaldt, et al.,
J. Chem. Phys. B **110**, 8674 (2006)

JPCBFK

VOLUME 110
JUNE 15, 2006
NUMBER 23
<http://pubs.acs.org/JPCB>

THE JOURNAL OF PHYSICAL CHEMISTRY

B

2D-Mapping of a Heterogeneous Catalyst inside a Fixed-Bed Reactor by X-Ray Absorption Spectroscopy (see page XXXX)

CH₄ + O₂ Gas 1 Gas 2

CCD-camera

Gas Supply

Inlet By-pass

In situ cell

Monochromator

Synchrotron

Hot air stream

Product Analysis Mass Spectrometer

Rh/Al₂O₃

Absorption / a.u.

Inlet Outlet

Rh^{3+} Rh^0

E / keV

CONDENSED MATTER, MATERIALS, SURFACES, INTERFACES, & BIOPHYSICAL CHEMISTRY

PUBLISHED WEEKLY BY THE AMERICAN CHEMICAL SOCIETY

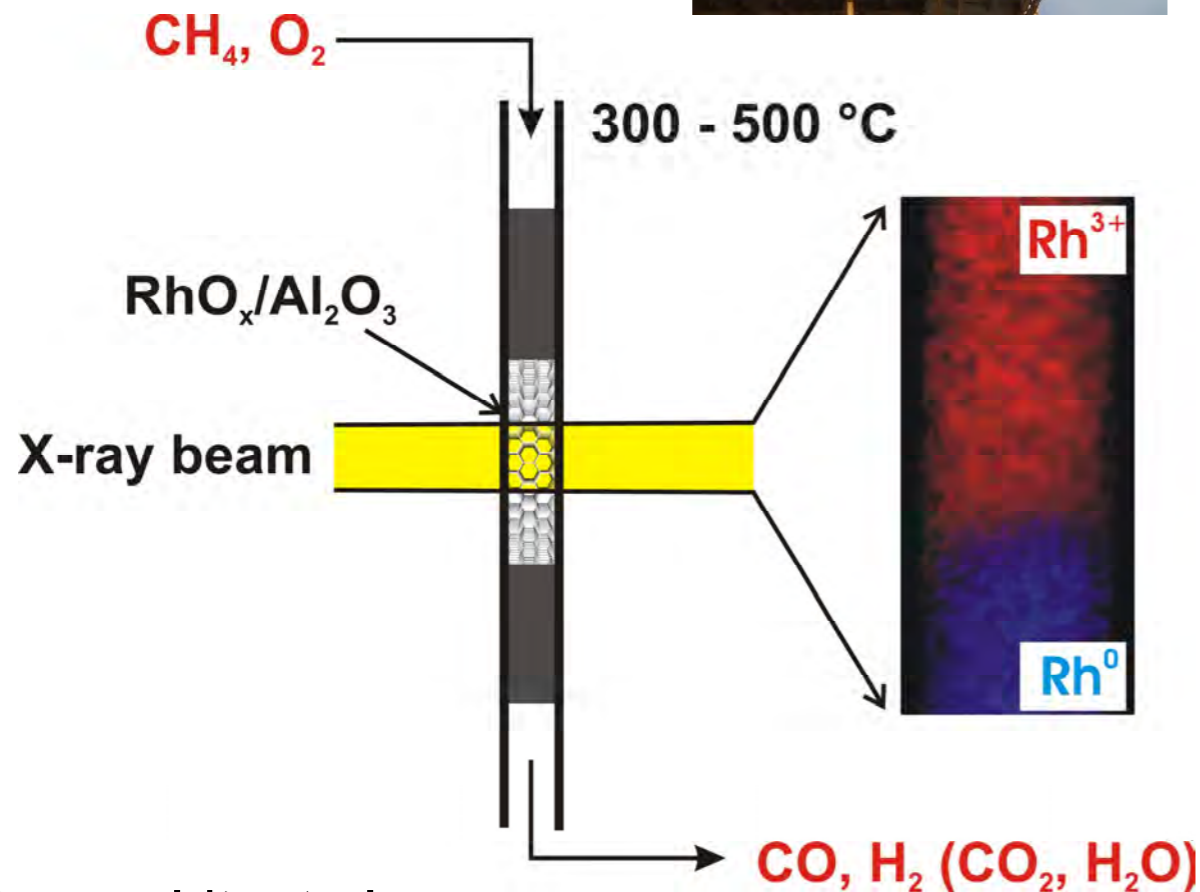


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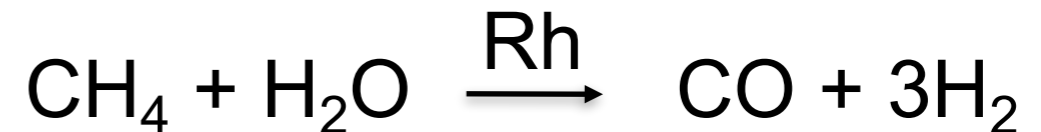
Grunwaldt, et al.,
J. Chem. Phys. B **110**, 8674 (2006)

Combustion of methane:

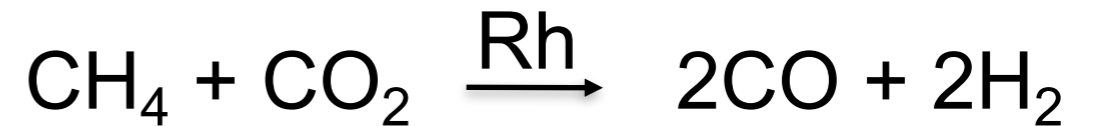


(exothermal: -801,7kJ/mol)

reforming of methane to H₂:



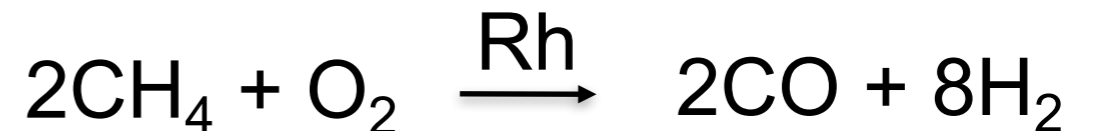
(endothermal: 206.1kJ/mol)



(endothermal: 247,5kJ/mol)

potentially other reaction:

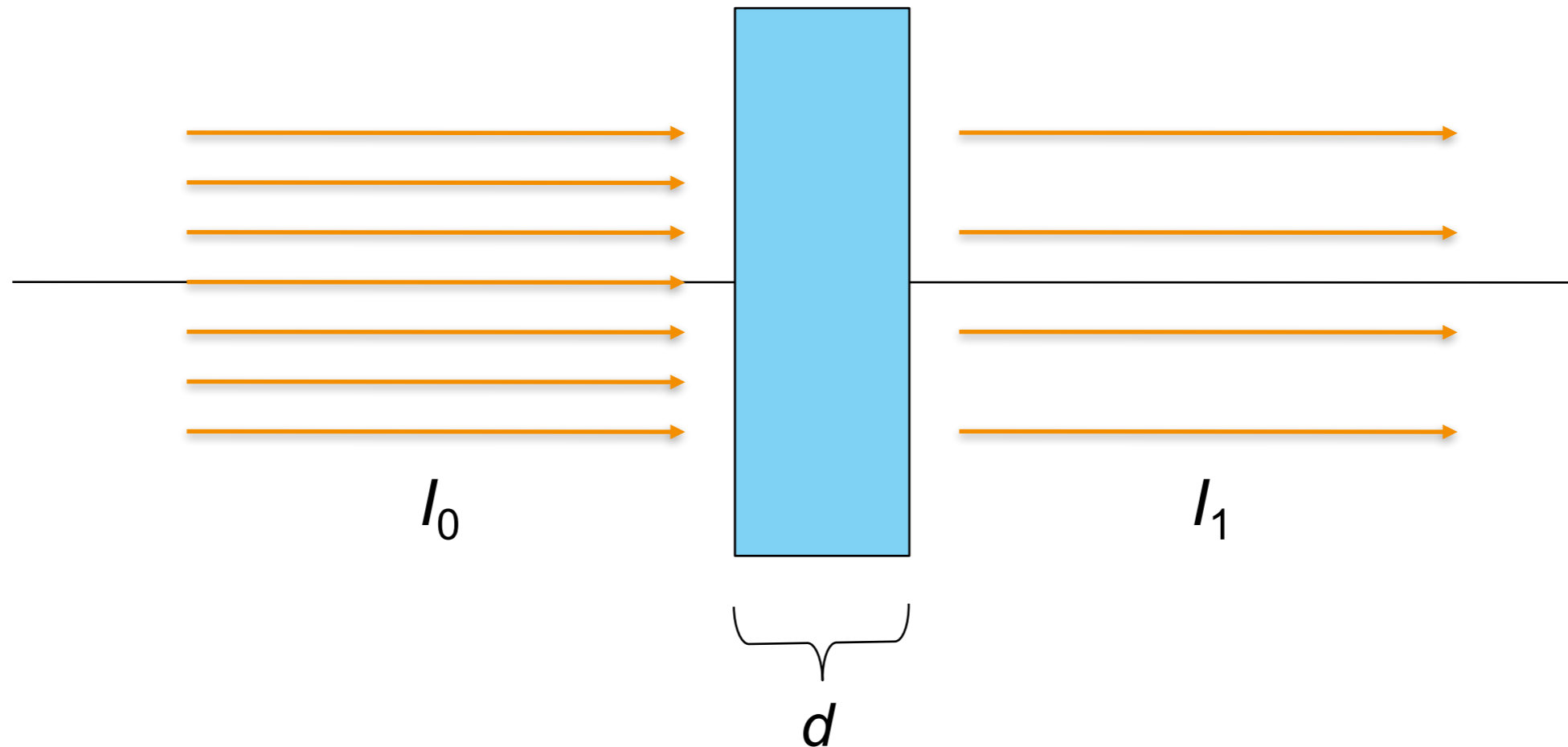
direct partial oxidation:



(exothermal: -35,5kJ/mol)



X-Ray Absorption: Lambert-Beer Law

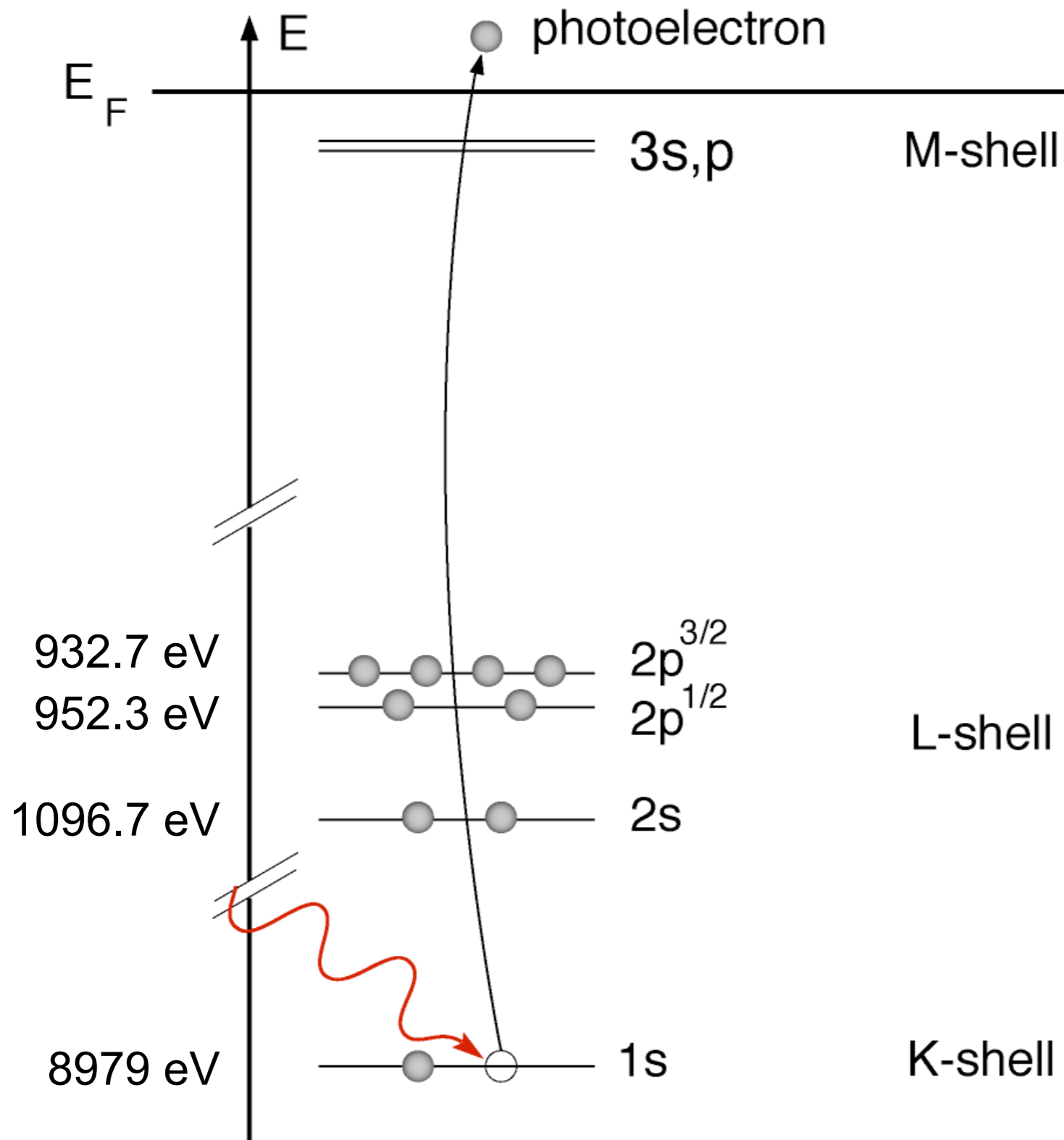


$$I_1(E) = I_0(E) \cdot \exp[-\mu(E)d]$$

$\mu(E)$: linear attenuation coefficient

$$\mu(E) \cdot d = \ln \left(\frac{I_0}{I_1} \right)$$

Photo Absorption



- > electrons populate specific atomic states: binding energies are atom specific: K, L, M
- > atom can absorb x-ray photon if:

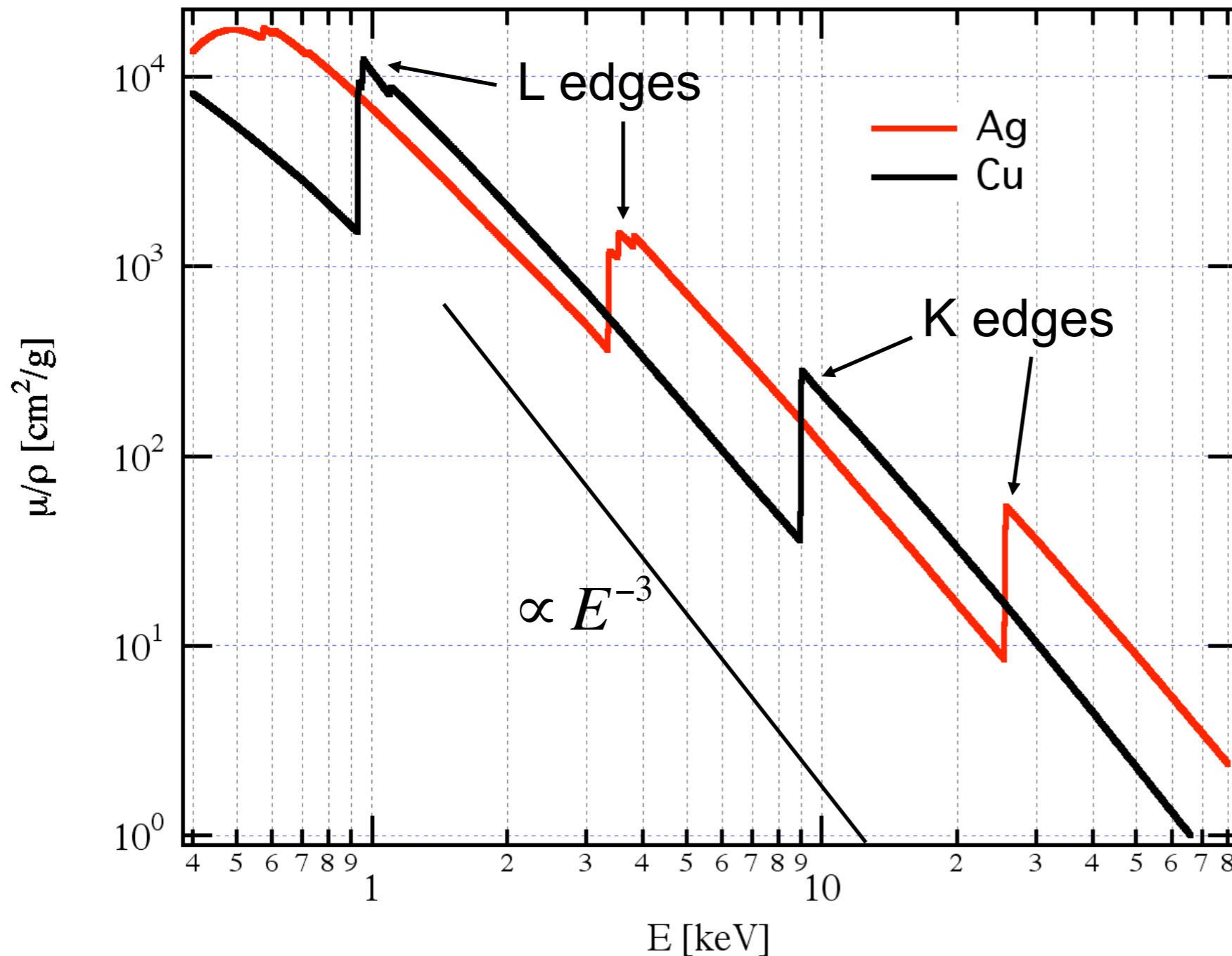
$$E_{\text{photon}} > E_{\text{ionization}}$$

(follows from Pauli principle)



Example: Absorption in Cu & Ag

$\mu(E)$: linear attenuation coefficient



> mainly atomic effect

> strong dependence on x-ray energy:

$$\propto E^{-2.78}$$

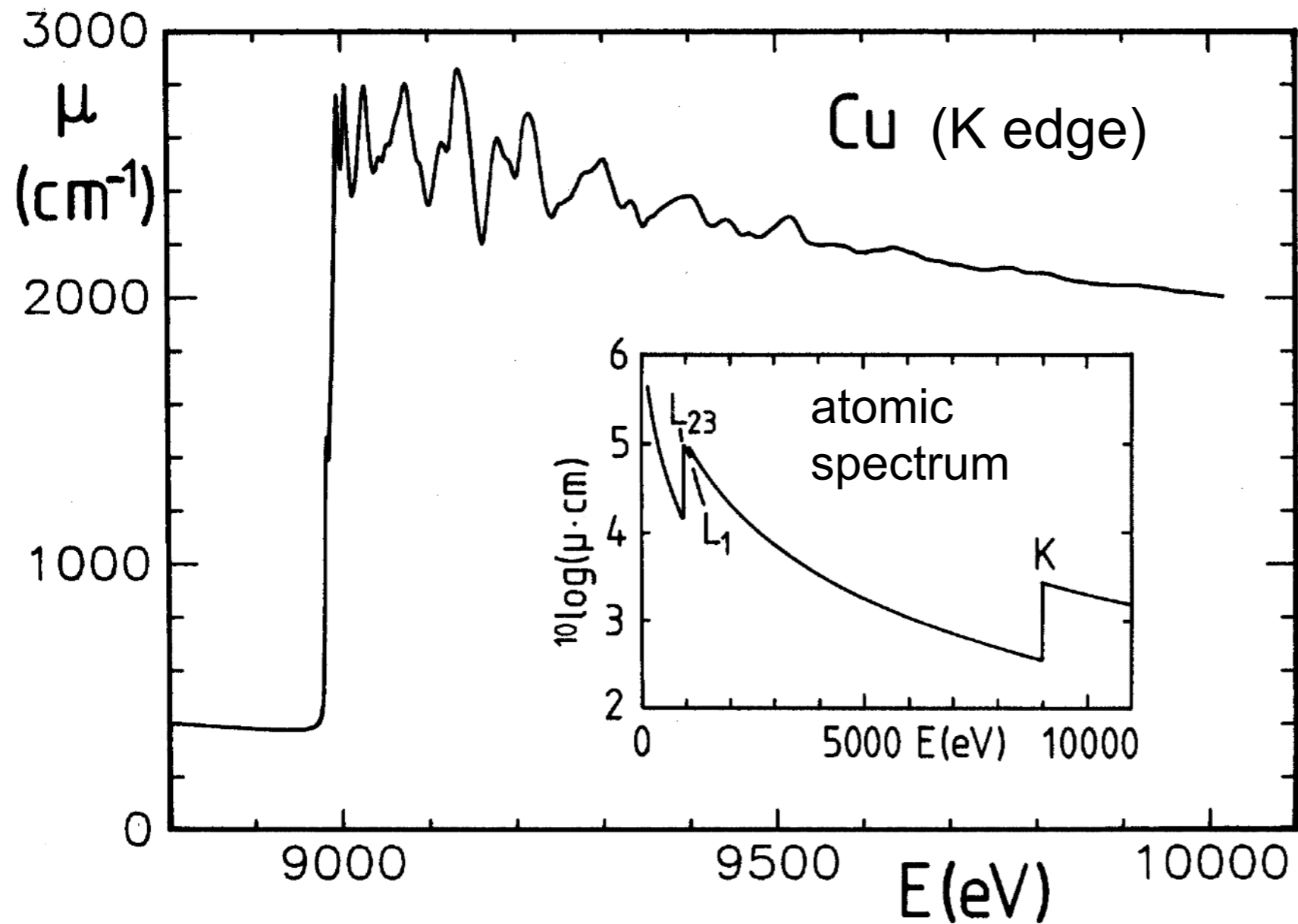
> strong dependence on atomic number:

$$\propto Z^{2.7}$$

> largest contribution from inner shells

Example: Absorption in Cu

$\mu(E)$: linear attenuation coefficient



Metallic Cu:

mainly atomic effect

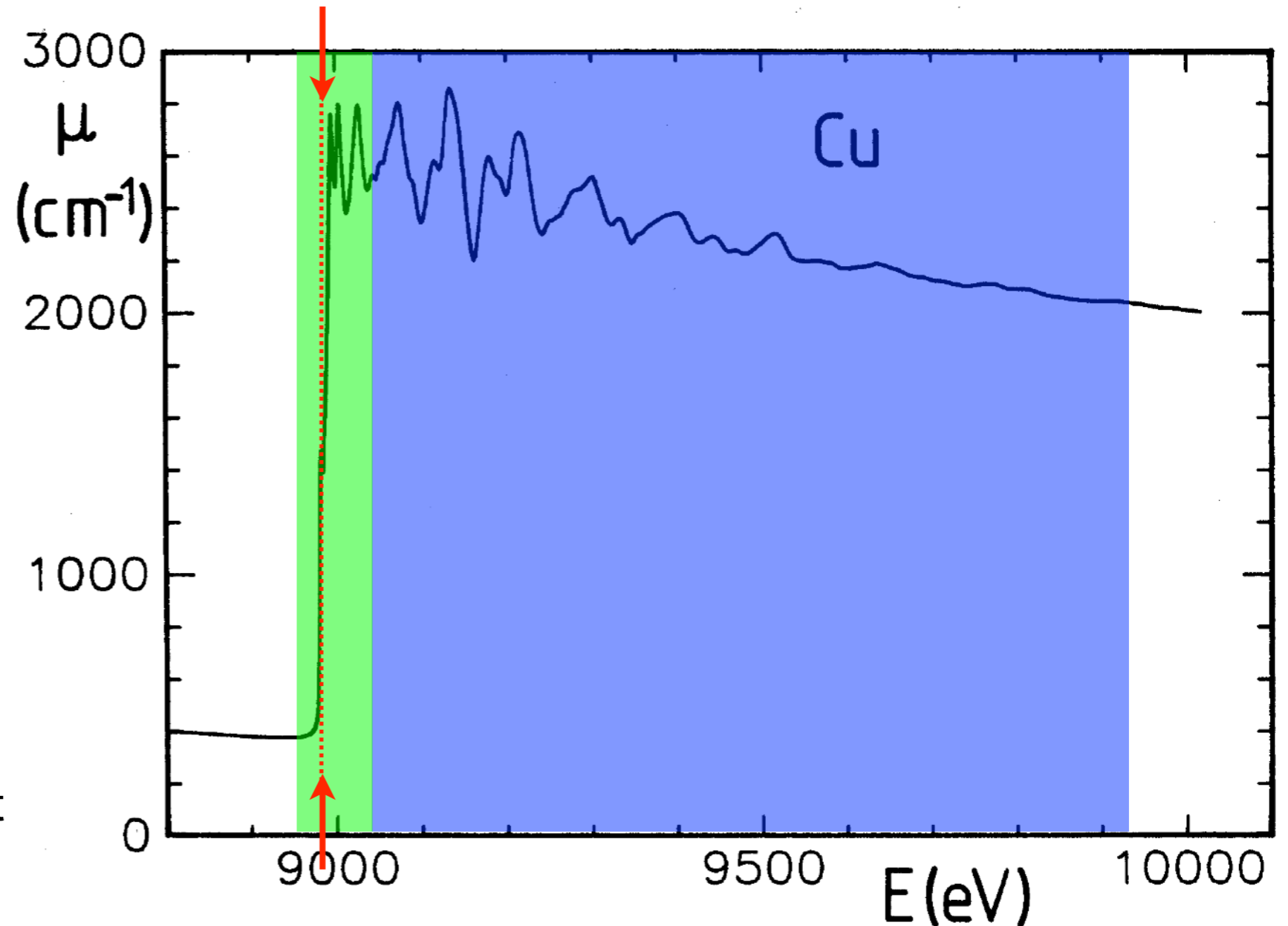
fine structure in solid:

[X-ray Absorption Fine Structure](#)

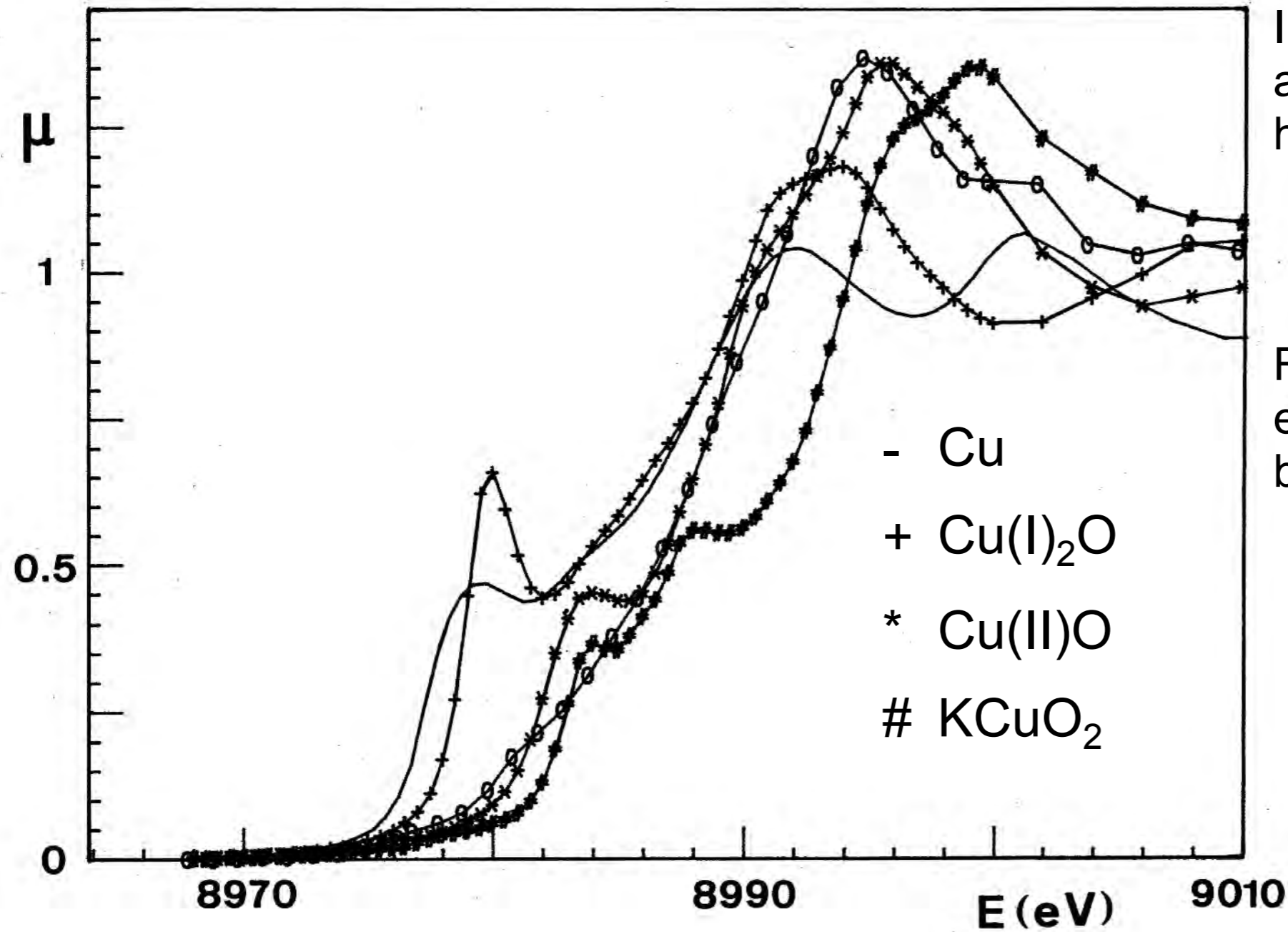
X-ray Absorption Spectrum

Three characteristic features:

- > Energy of absorption edge: oxidation state
- > Near-edge region: (XANES: x-ray absorption near edge structure) local, projected density of states
- > Extended fine structure: (EXAFS: extended x-ray absorption fine structure) local chemical environment of atomic species



Energy of Absorption Edge

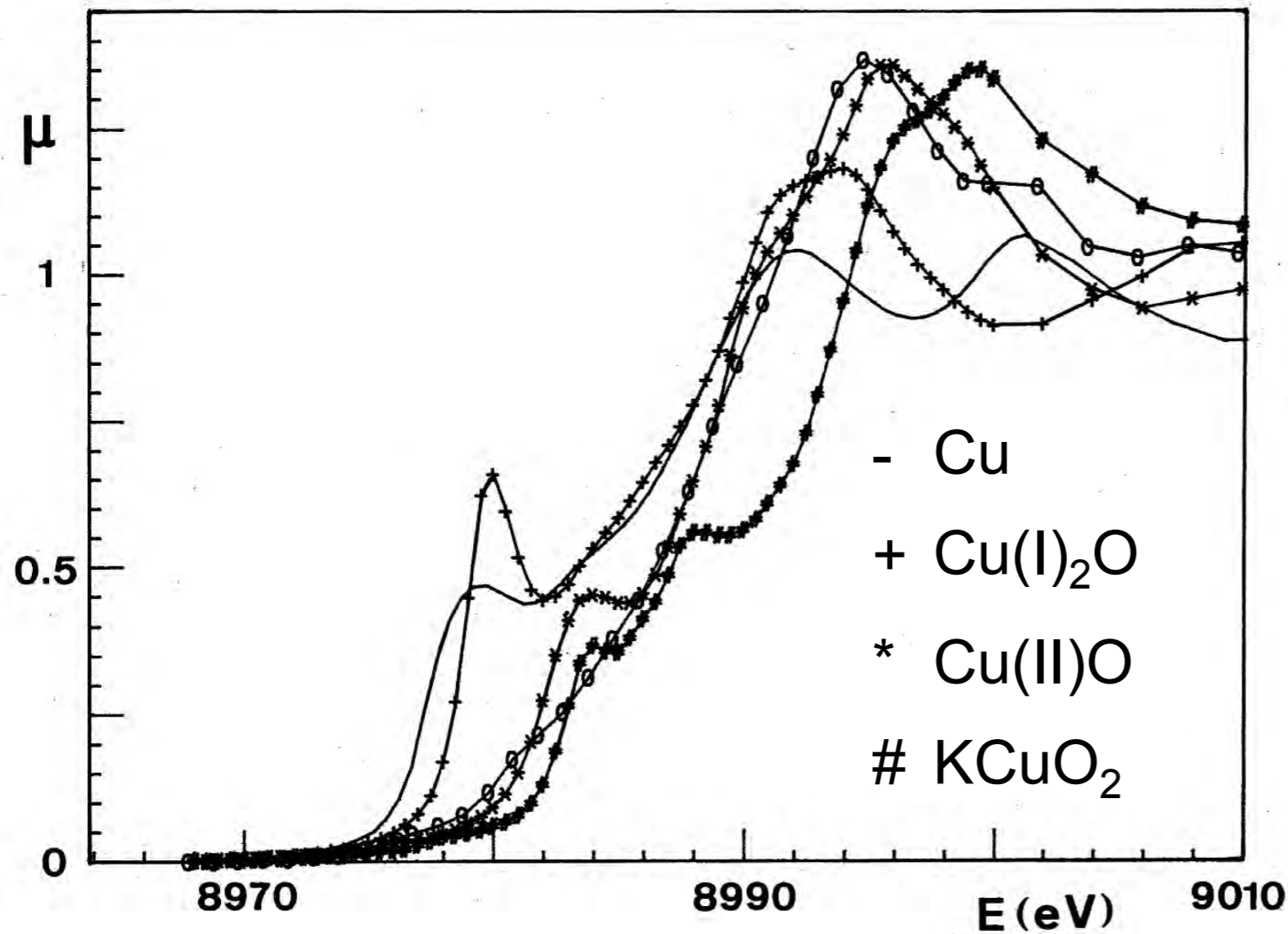


Increasing oxidation state:
absorption edge shifts to
higher x-ray energies

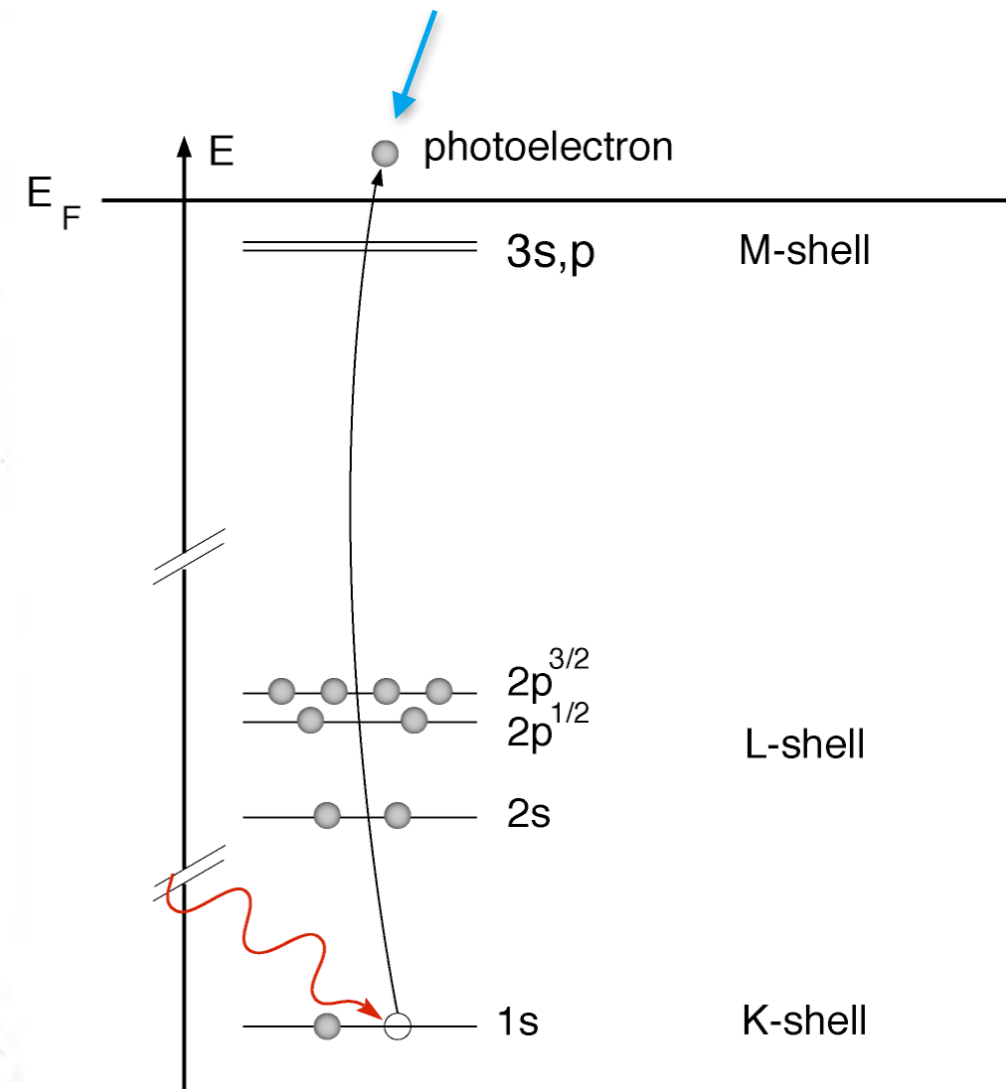
Reduced screening of
electric field of nucleus
by valence electrons:

other electrons more
tightly bound!

Shape of Near-Edge Spectrum



depends on density of states available to photoelectron



Shape of spectrum:

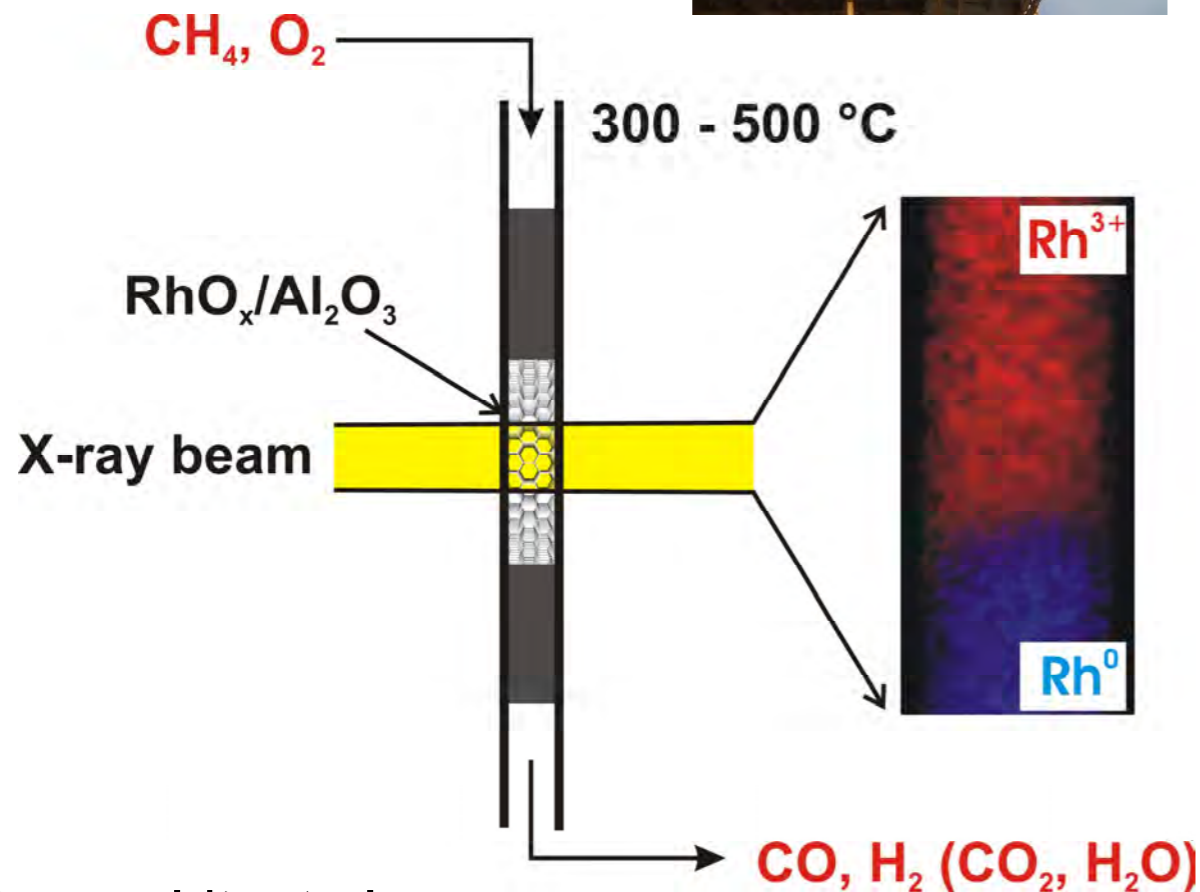
- > can be modeled by methods in theoretical solid state physics
- > can be used as „fingerprint“ to identify a given chemical environment

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Grunwaldt, et al.,
J. Chem. Phys. B **110**, 8674 (2006)

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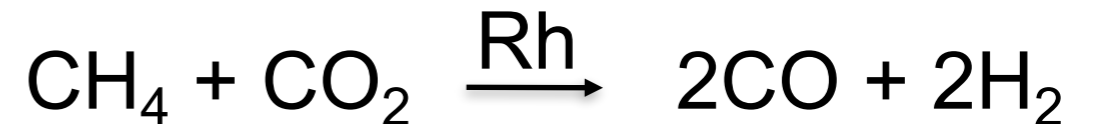


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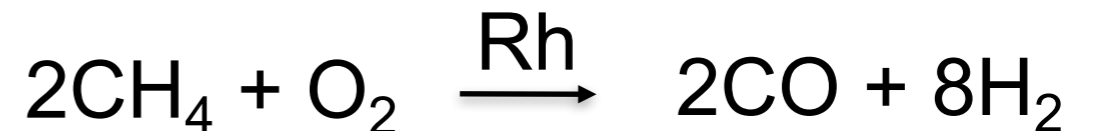
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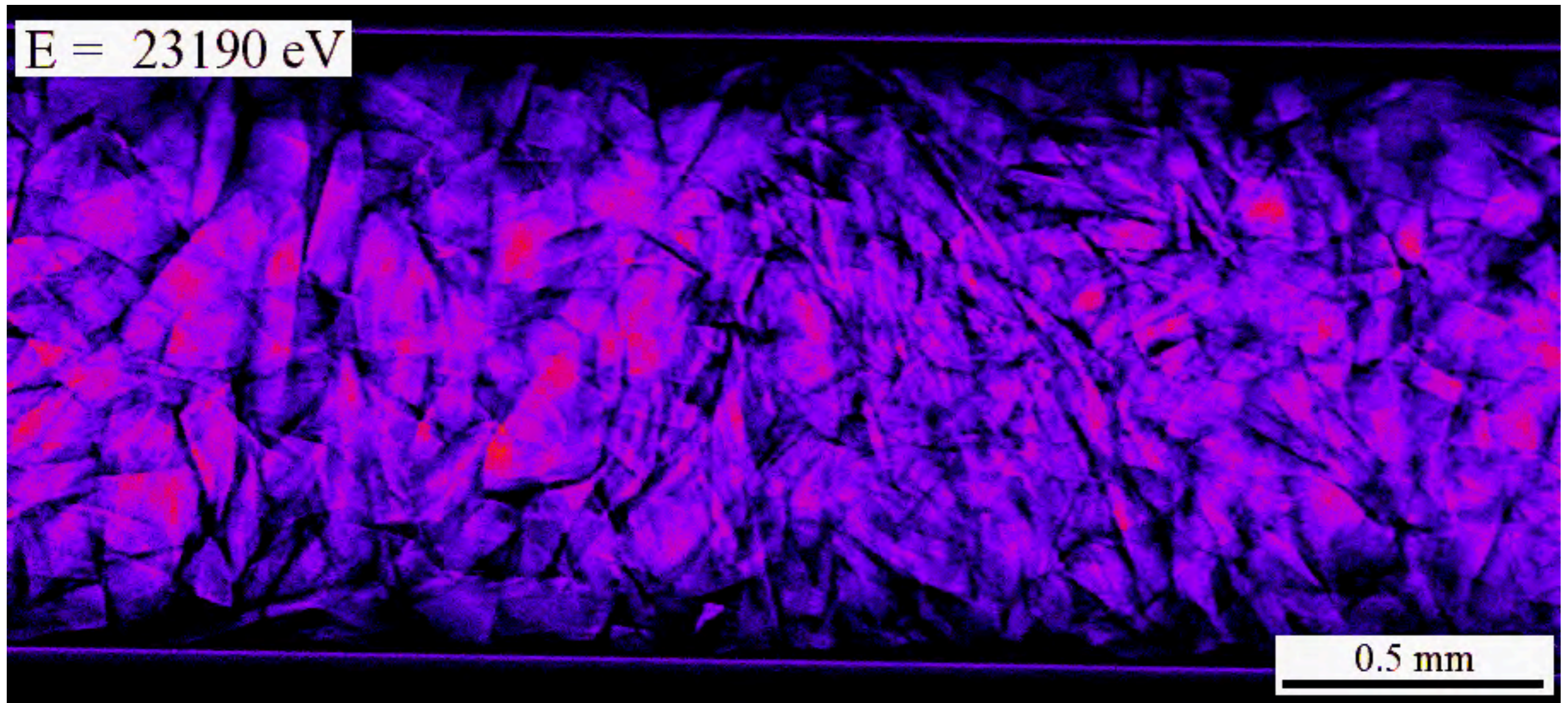


(exothermal: -35,5kJ/mol)



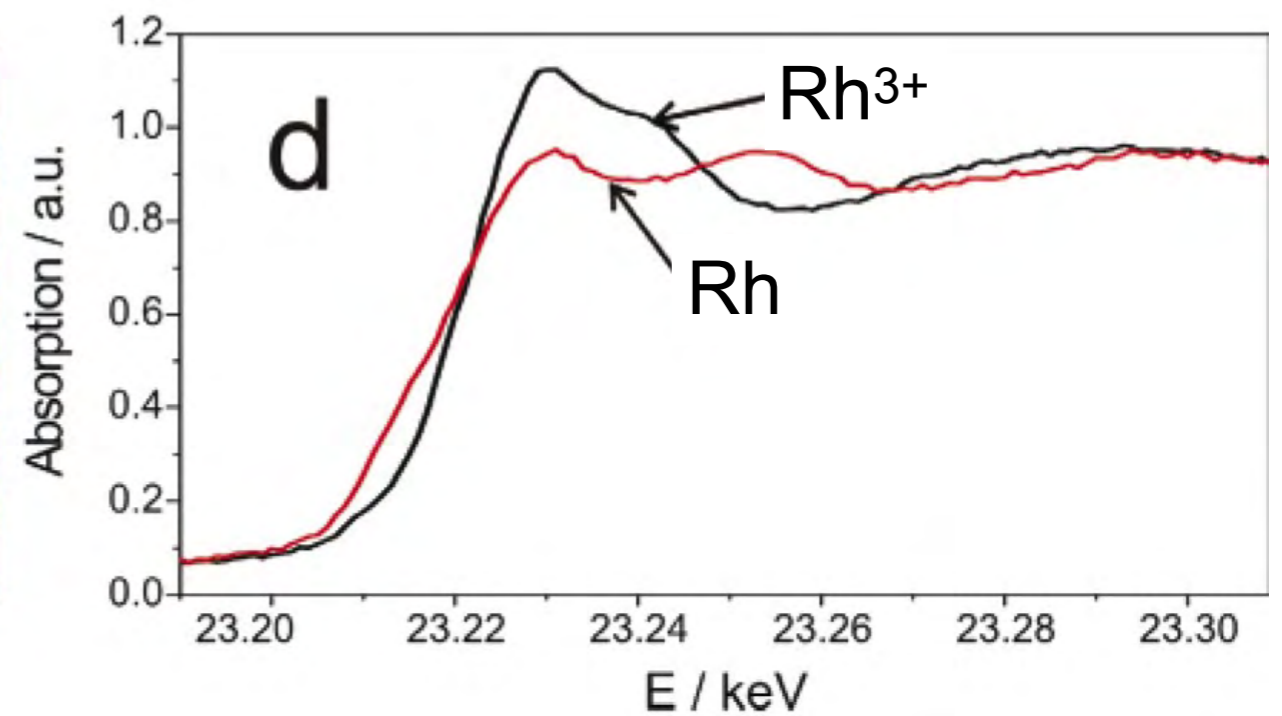
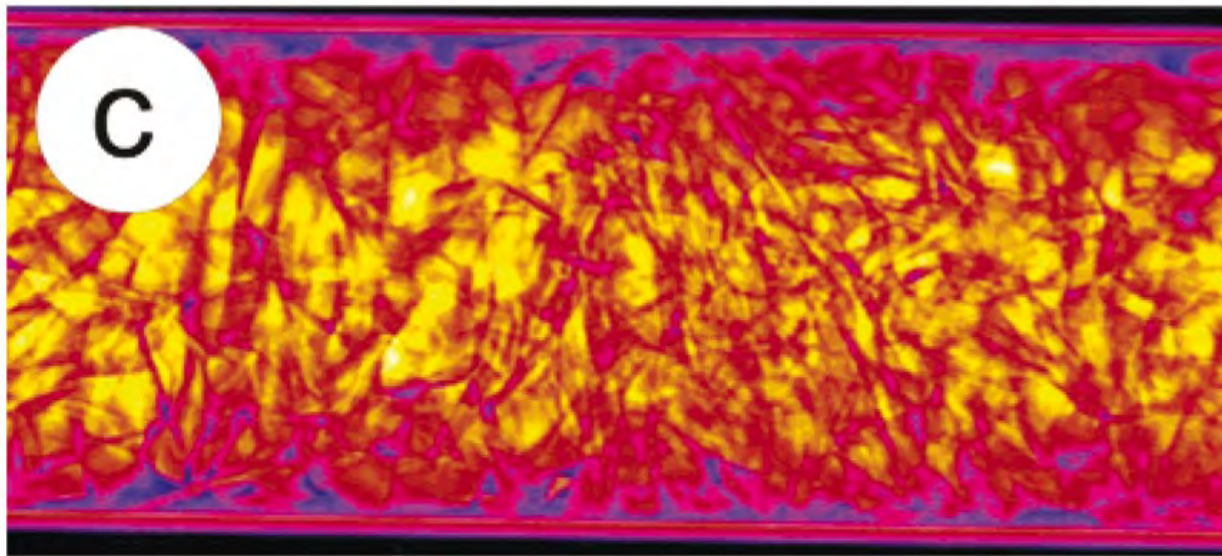
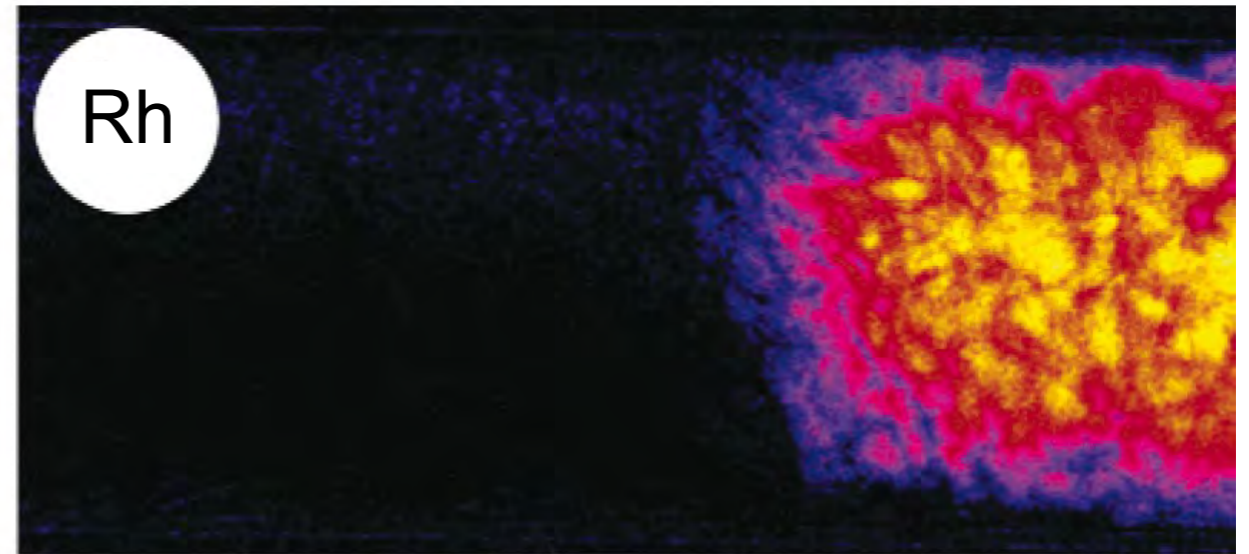
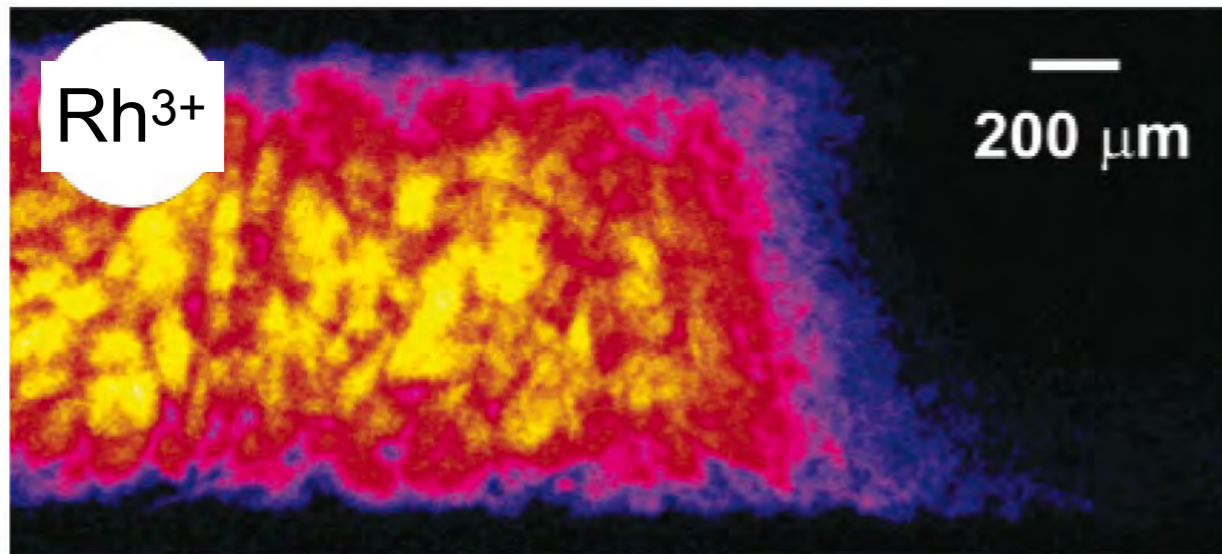
Visualize Catalysis

In-situ transmission imaging of catalyst bed inside chemical reactor



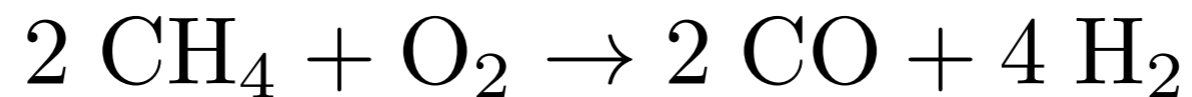
Grunwaldt, et al.,
J. Chem. Phys. B **110**, 8674 (2006)

Visualize Catalysis

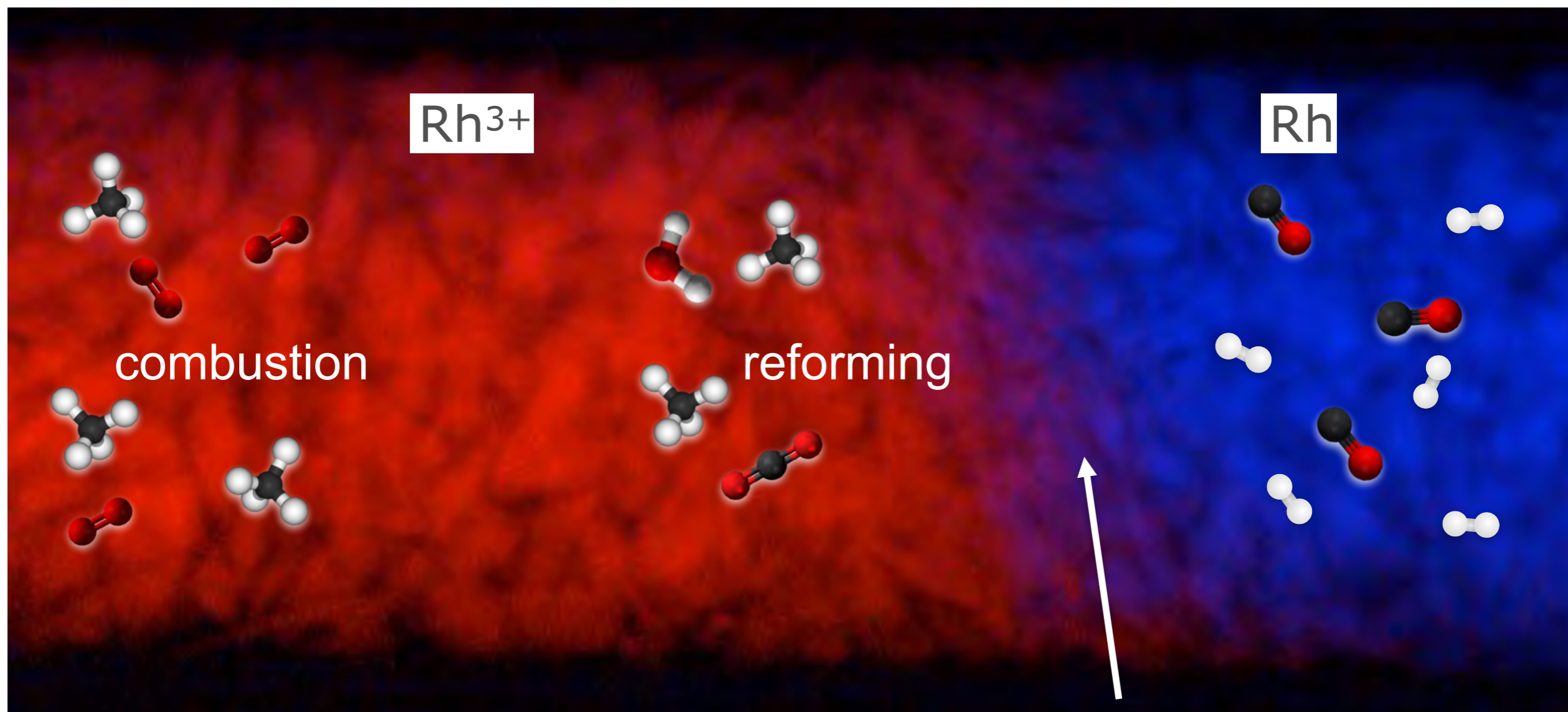


Grunwaldt, et al.,
J. Chem. Phys. B **110**, 8674 (2006)

Visualize Catalysis



direction of flow

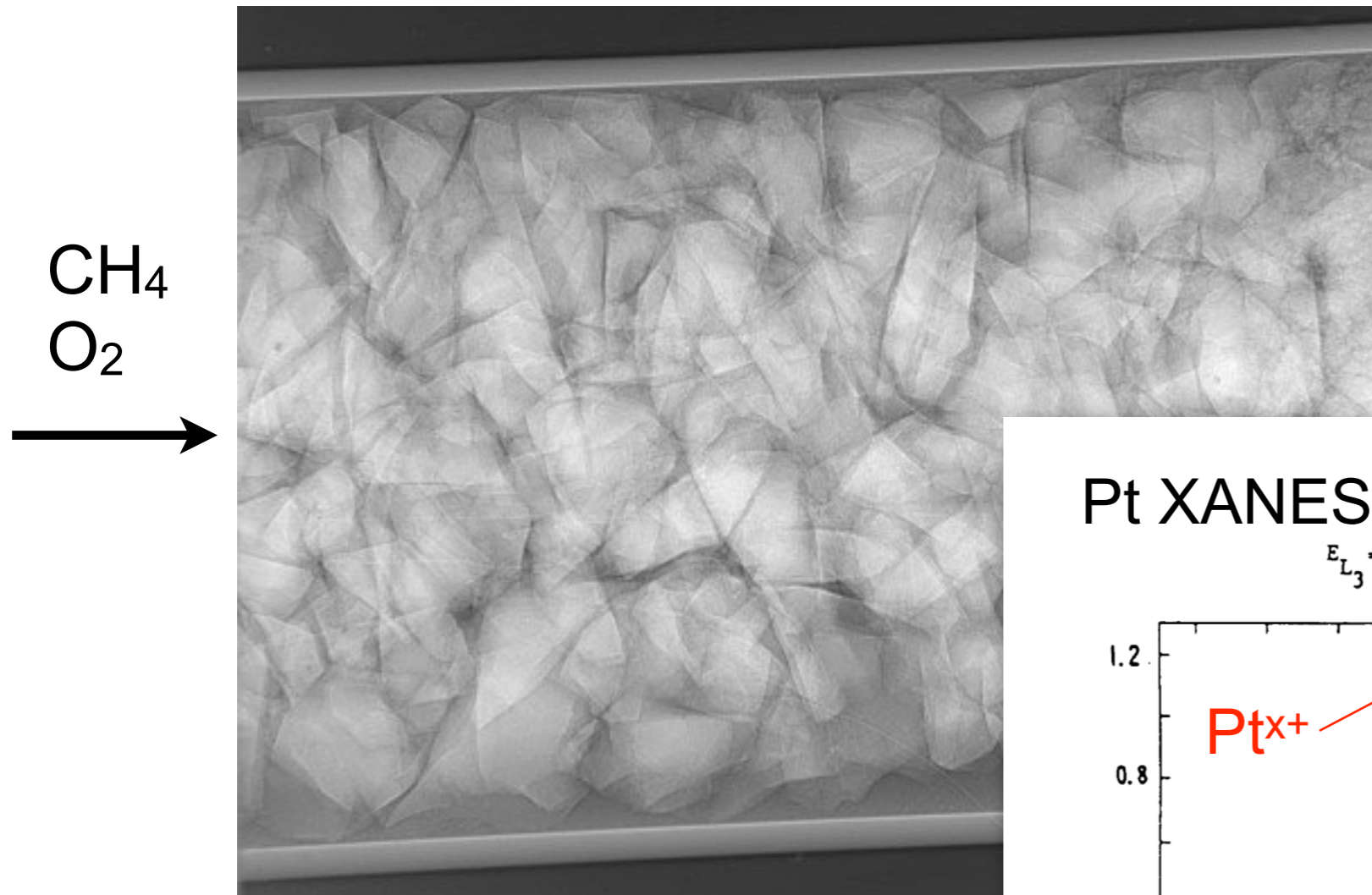


Grunwaldt, et al.,
J. Chem. Phys. B **110**, 8674 (2006)

production of hydrogen
Rh is reduced!

Filming the Ignition of a Catalytic Reaction

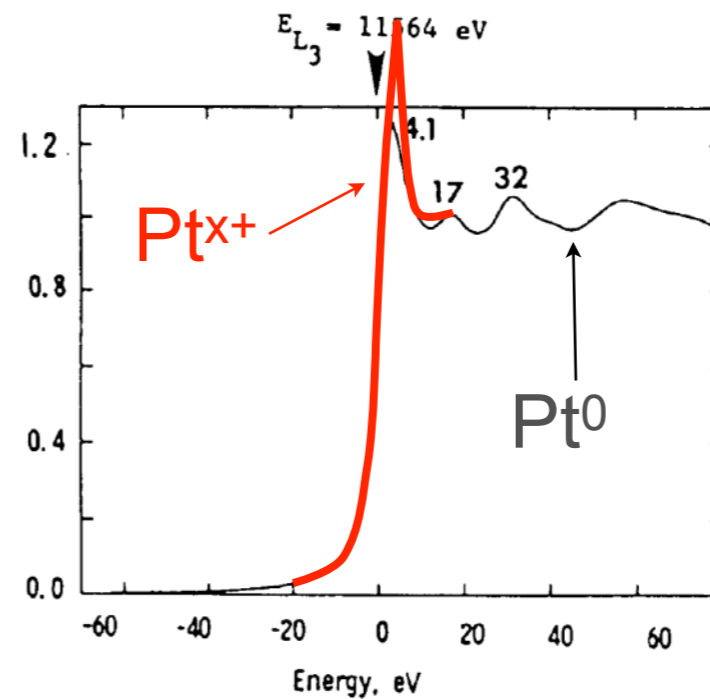
Partial oxidation of Methane by reforming



Pt particles on alumina powder in capillary

$T \sim 350^\circ\text{C}$

Pt XANES:

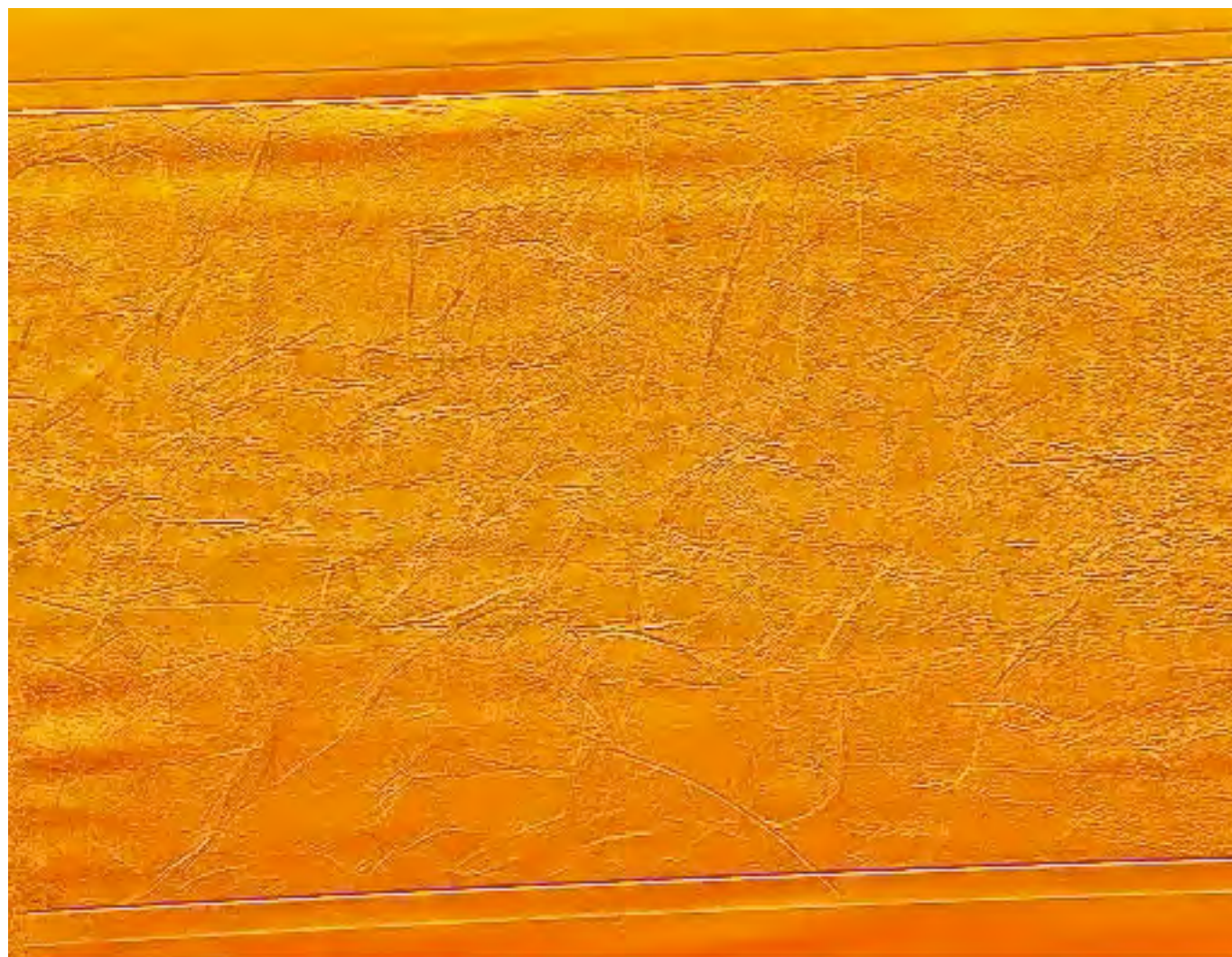


B. Kimmerle, et al.,
J. Phys. Chem. C **113**, 3037 (2009)

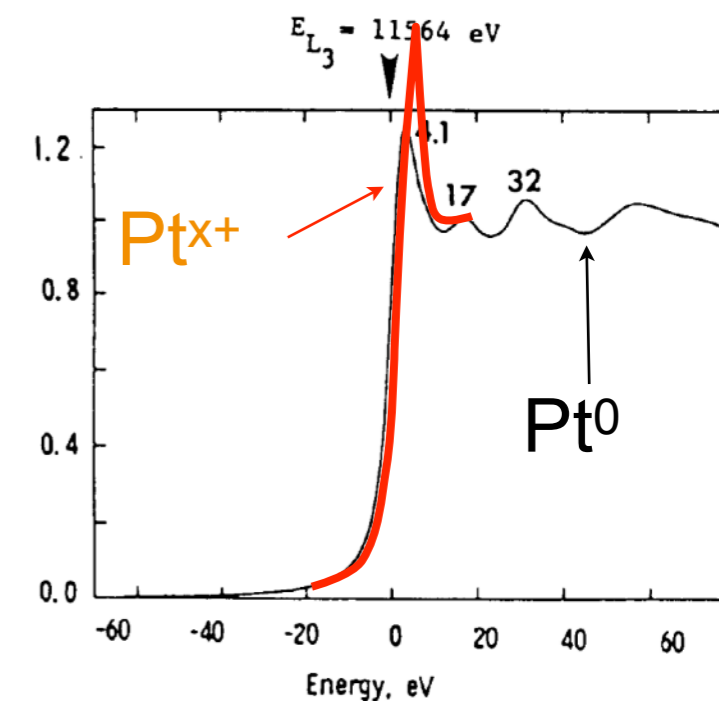


Filming the Ignition of a Catalytic Reaction

Partial oxidation of Methane by reforming
Imaging difference compared to oxidized catalyst:



Pt XANES:

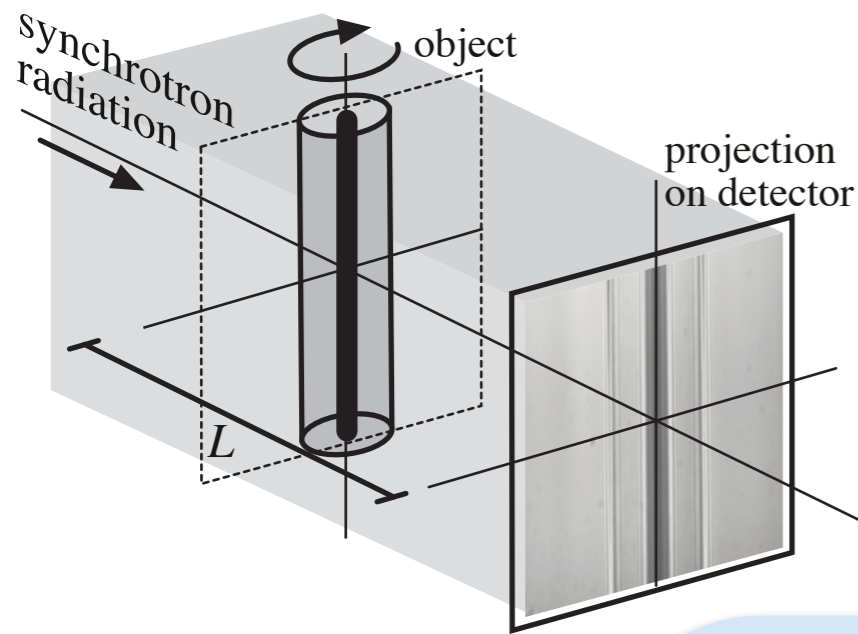


→ Catalyst is being reduced when reforming reaction ignites

B. Kimmerle, et al.,
J. Phys. Chem. C **113**, 3037 (2009)

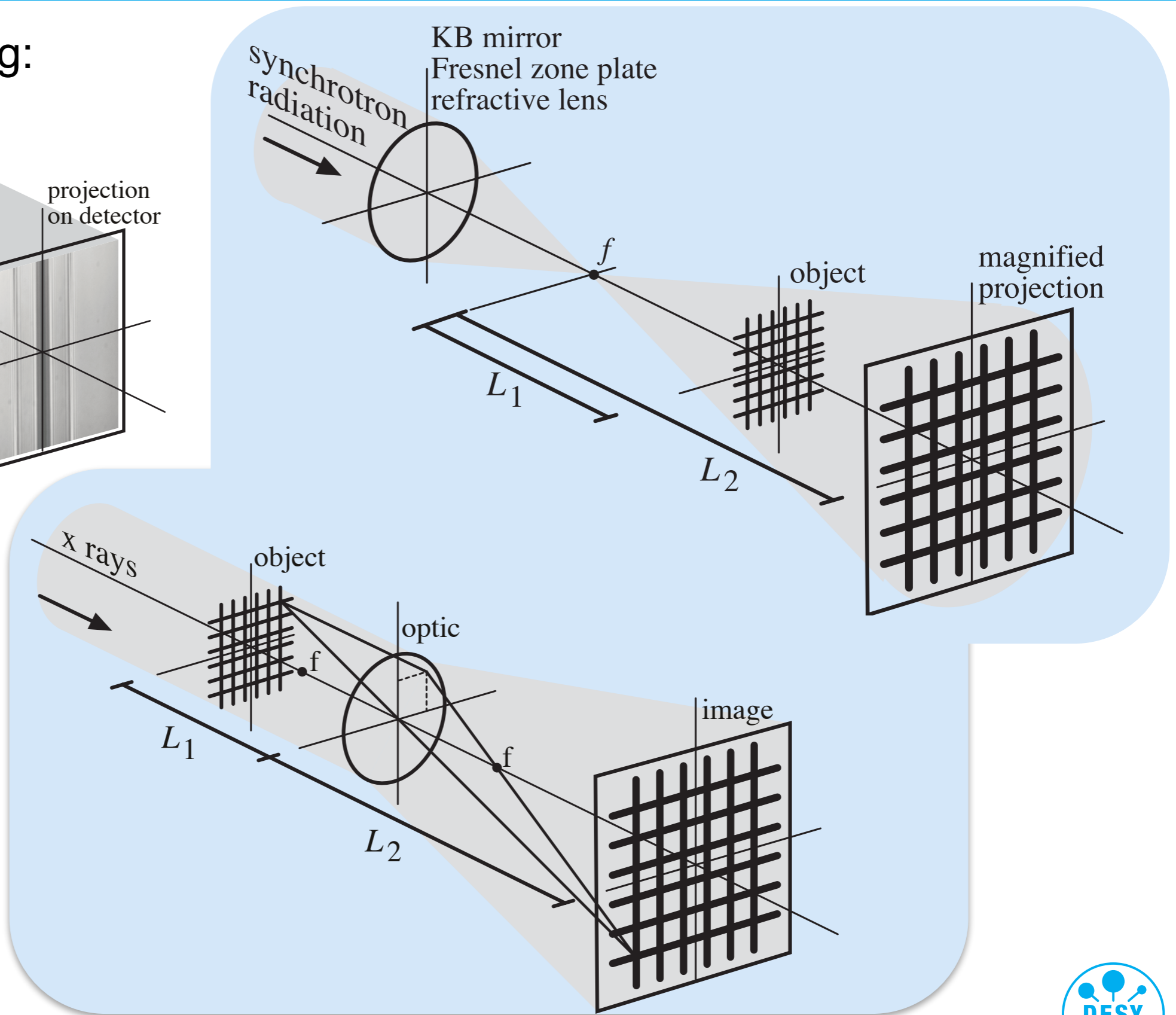
X-Ray Microscopy Techniques: Full-Field Imaging

Projection imaging:



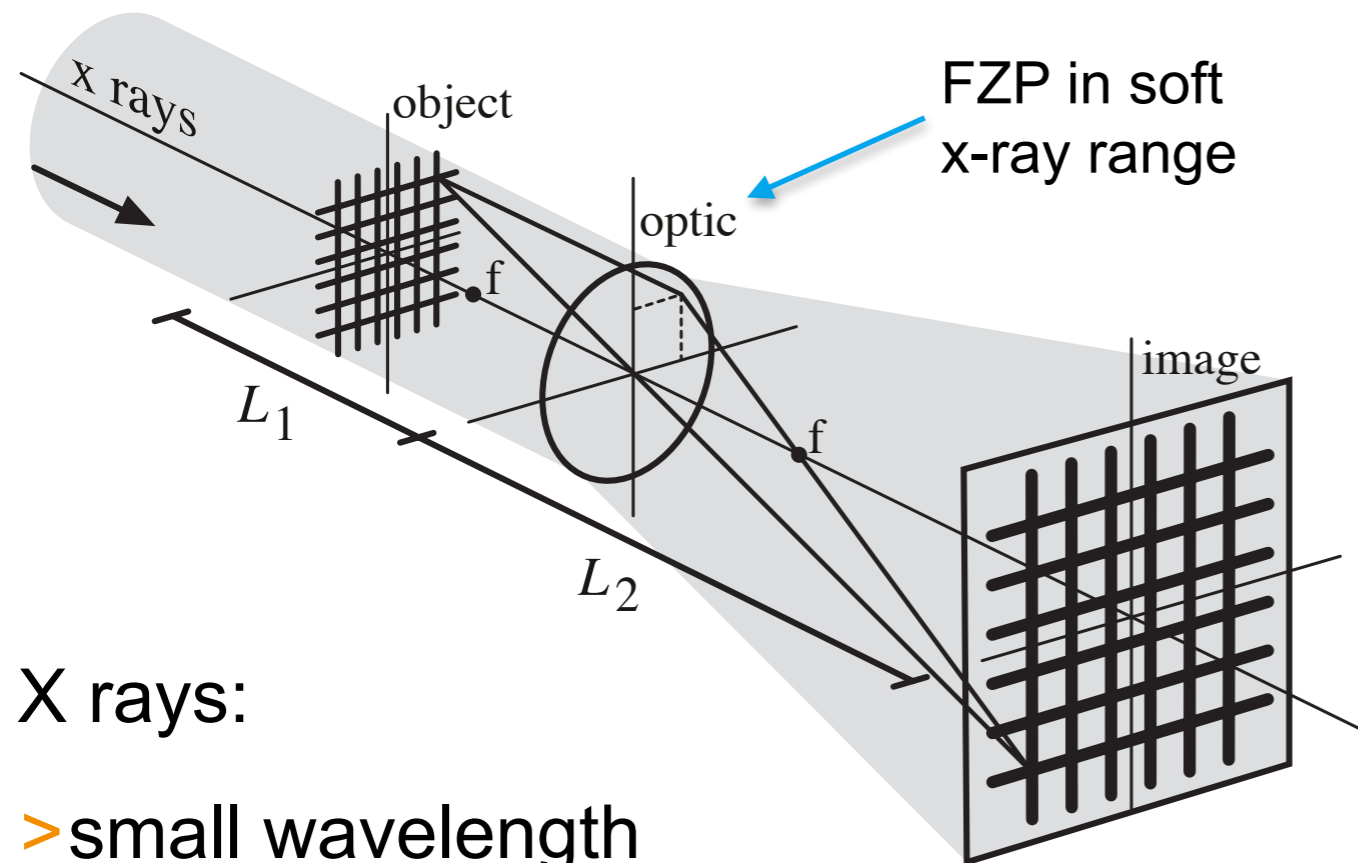
Imaging through objective lens:

x-rays focused by condensor (aperture matching)



Full-Field X-Ray Microscopy

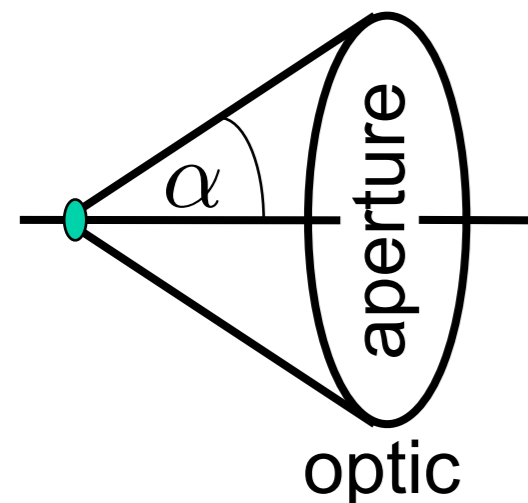
Magnifying imaging by objective:



X rays:

> small wavelength

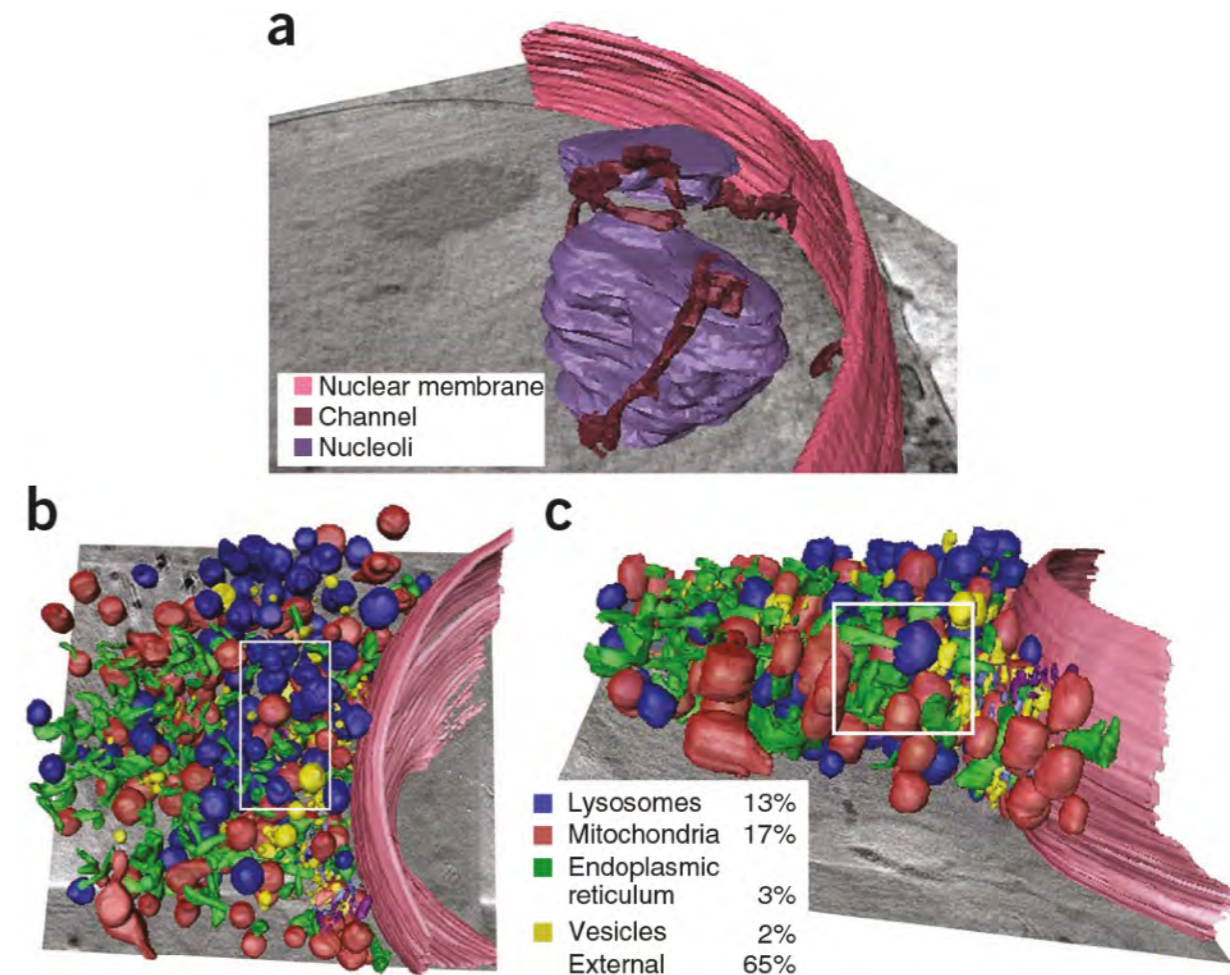
> limited numerical aperture α



resolution (E. Abbe):

$$d = \frac{\lambda}{2 \sin \alpha}$$

Example: soft x-ray imaging of cells (high contrast in water window) adenocarcinoma (mouse)



G. Schneider, et al., Nature Methods (2010). BESSY II, HZB

resolution: ca. 70 nm ($\lambda \approx 2.4 \text{ nm}, \alpha = 3.3^\circ$)

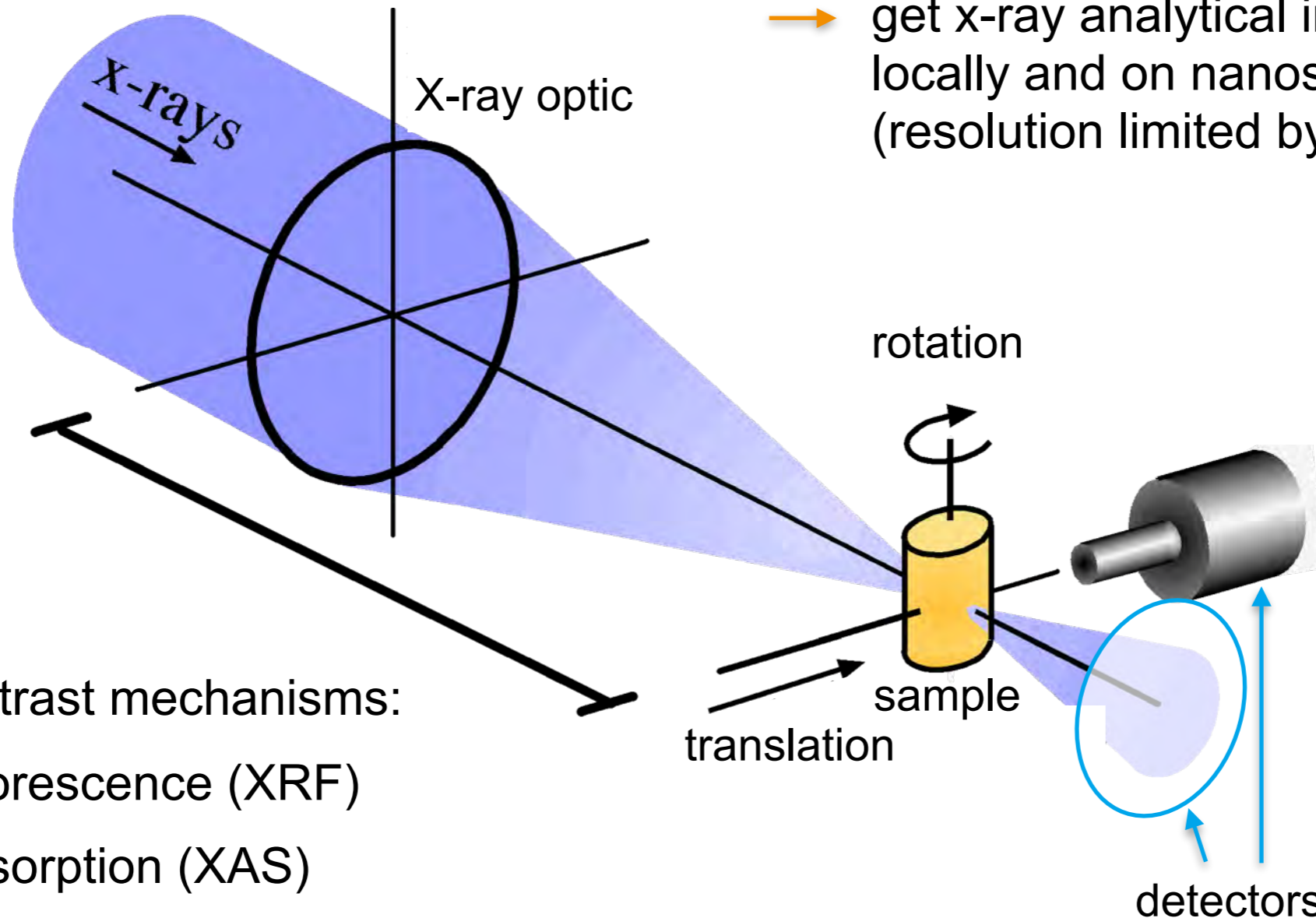


Scanning Microscopy and Tomography: Nanoprobe

X rays are focused onto the sample

raster scan sample through beam:

→ get x-ray analytical information locally and on nanoscale (resolution limited by focus size).

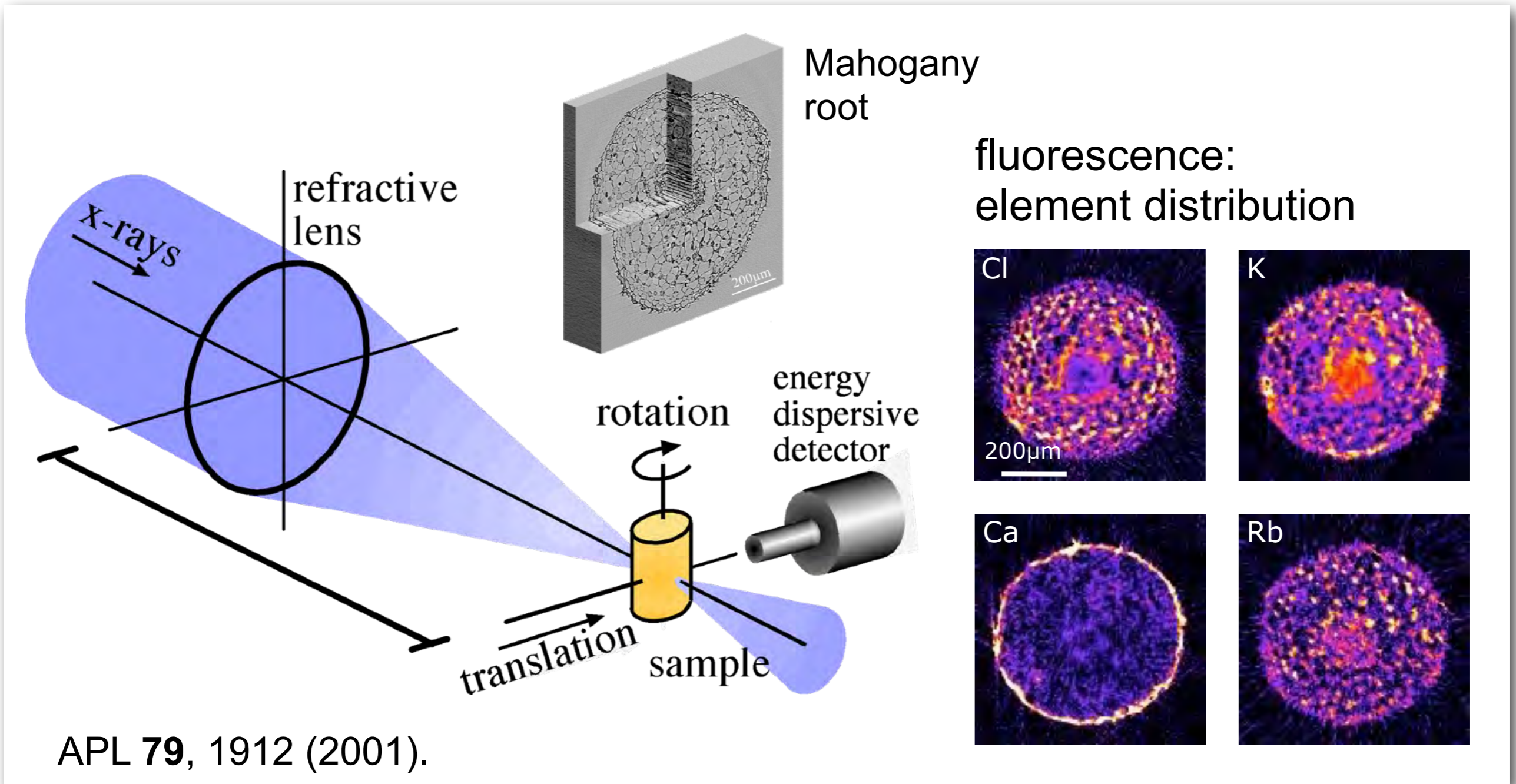


different contrast mechanisms:

- > x-ray fluorescence (XRF)
- > x-ray absorption (XAS)
- > x-ray diffraction (XRD, SAXS, WAXS)
- > maybe in future even IXS (RIXS)
- > ...

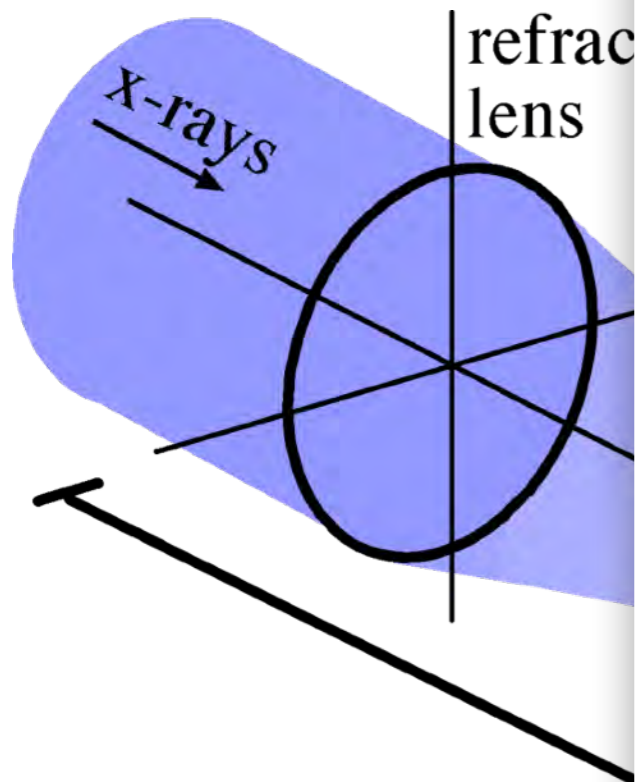
X-Ray Scanning Microscopy and Tomography

> Fluorescence microtomography



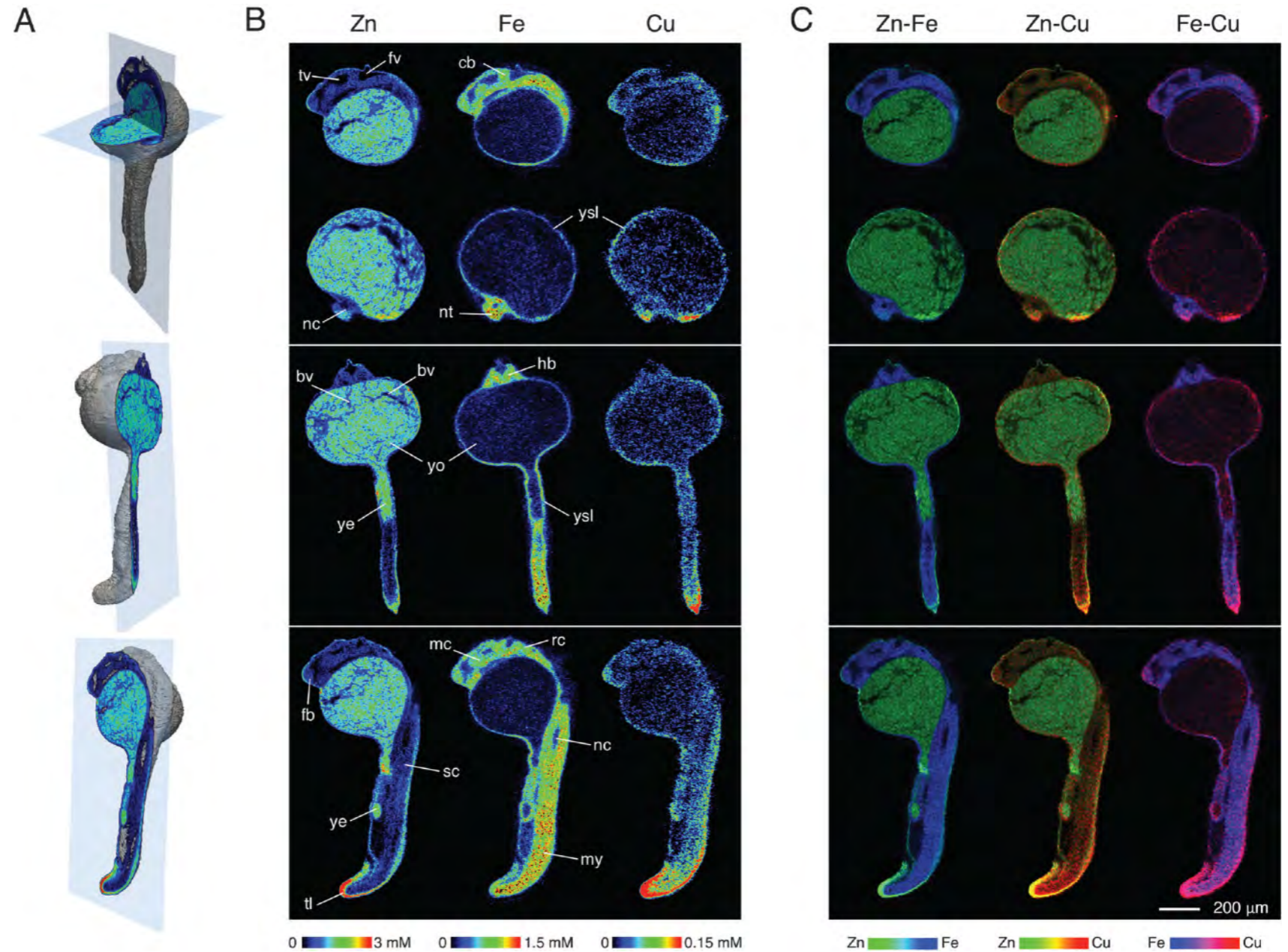
X-Ray Scanning Microscopy and Tomography

> Fluorescence micro



APL 79, 1912 (2001)

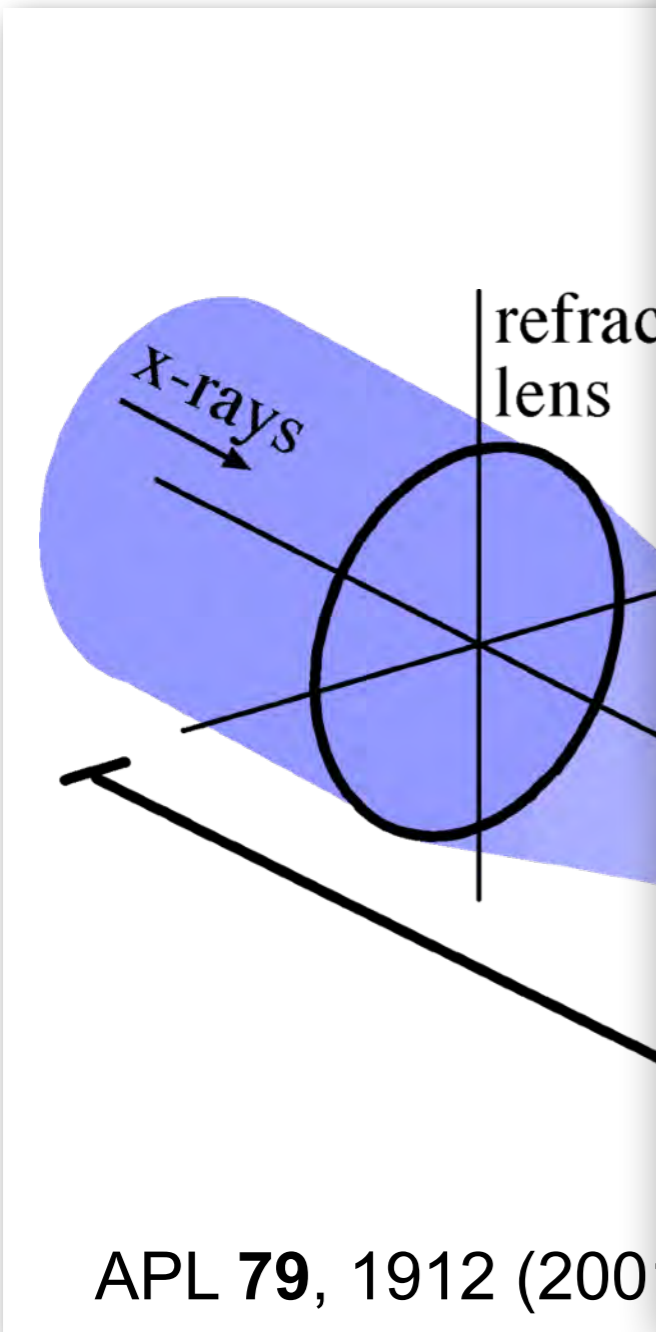
Full fluorescence tomogram of zebra fish embryo



D. Bourassa, et al., Metallomics 6, 1648 (2014).

X-Ray Scanning Microscopy and Tomography

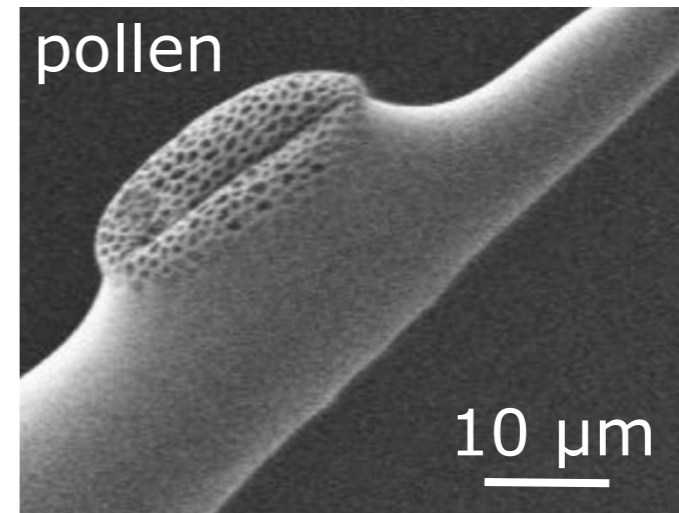
> Fluorescence micro



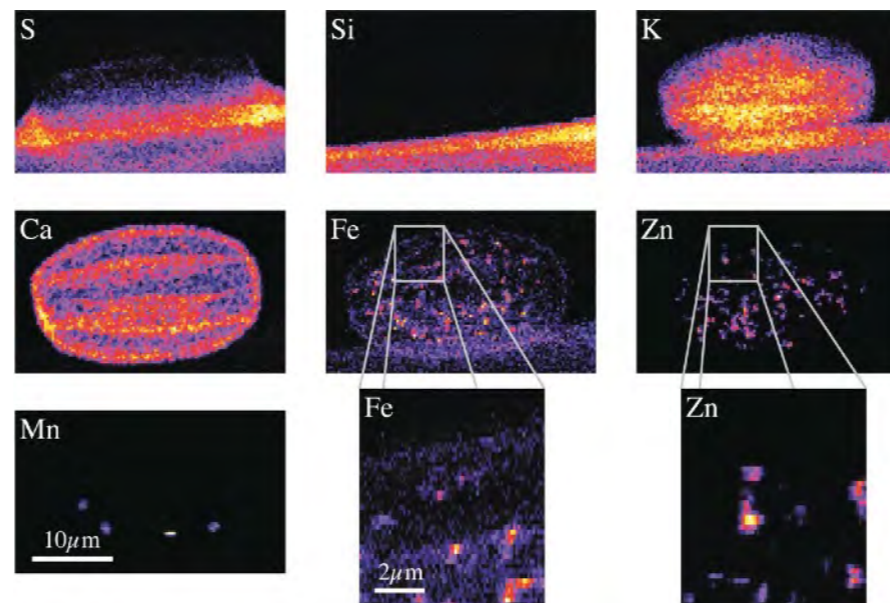
Full fluorescence tomogram of zebra fish embryo

A

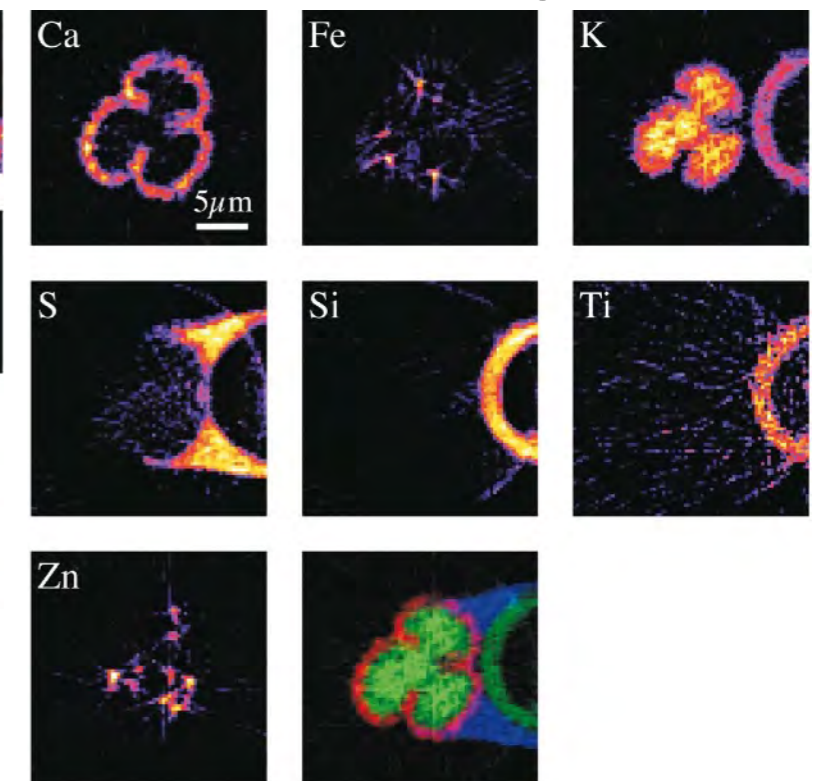
High-resolution fluorescence mapping and tomography



Fluorescence maps



Fluorescence tomogram



pixel size: 100 nm

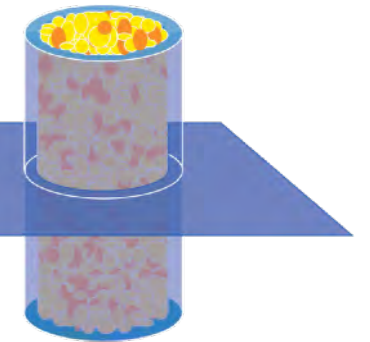
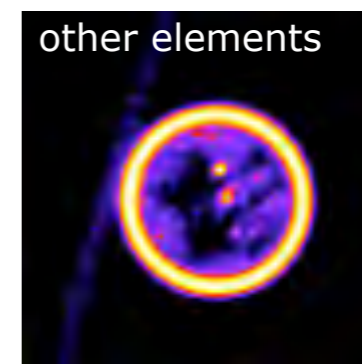
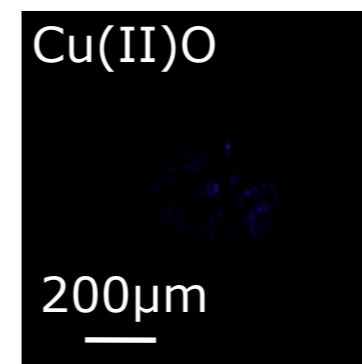
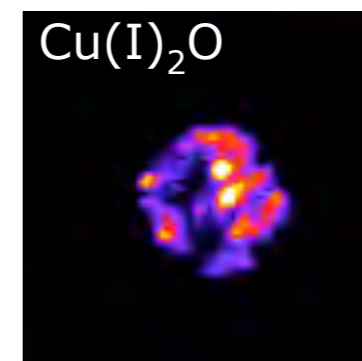
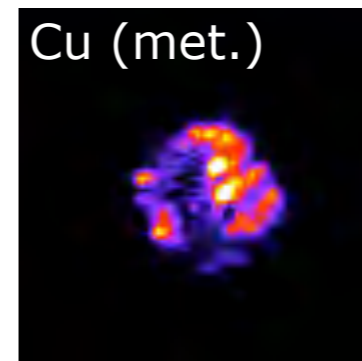
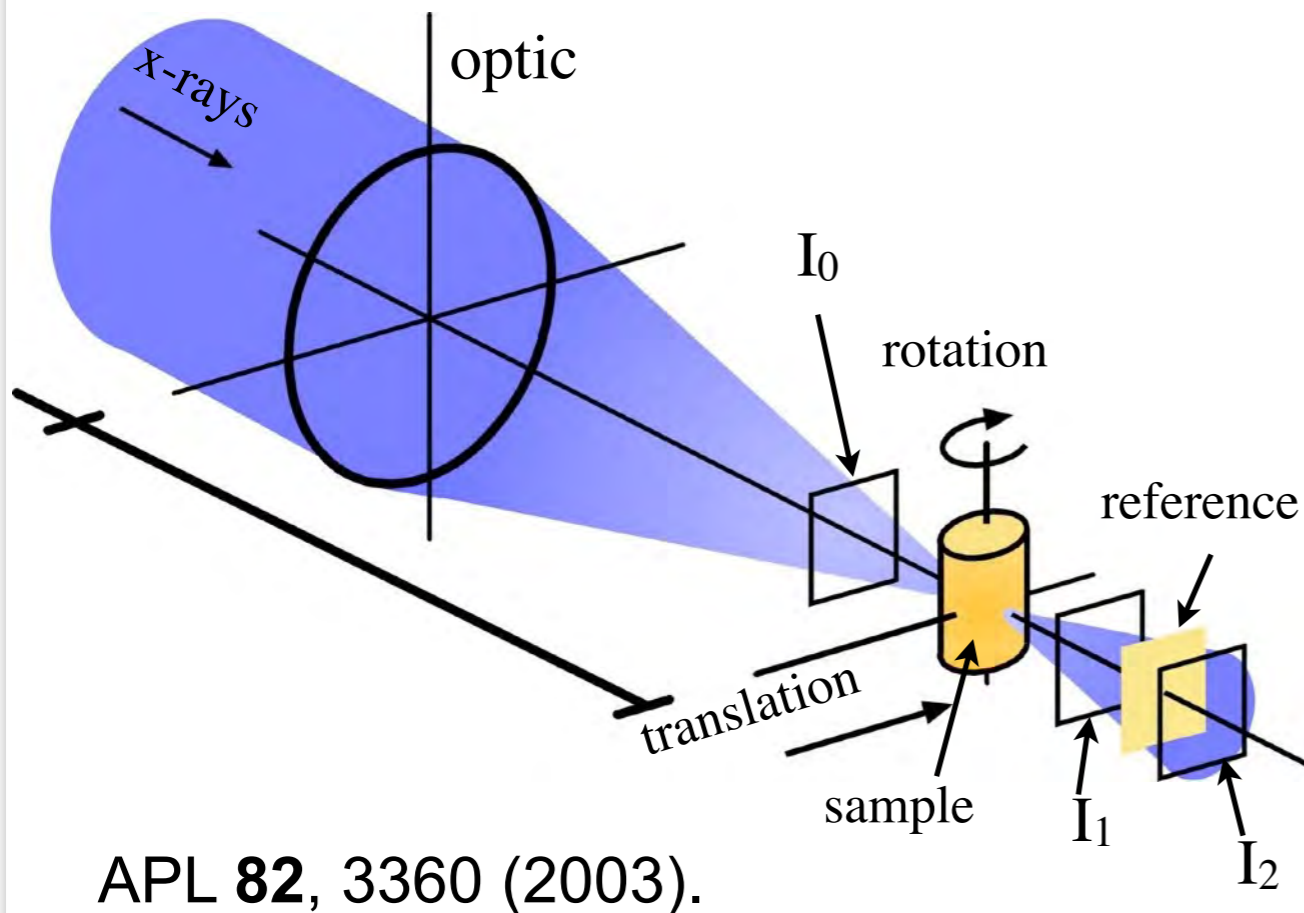
W. Schröder, FZ Jülich



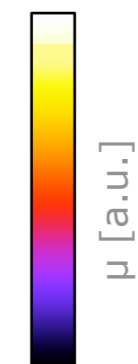
X-Ray Scanning Microscopy and Tomography

- > Fluorescence microtomography
- > Tomographic absorption spectroscopy (XANES tomography)

Absorption spectroscopic tomography distribution of chemical states



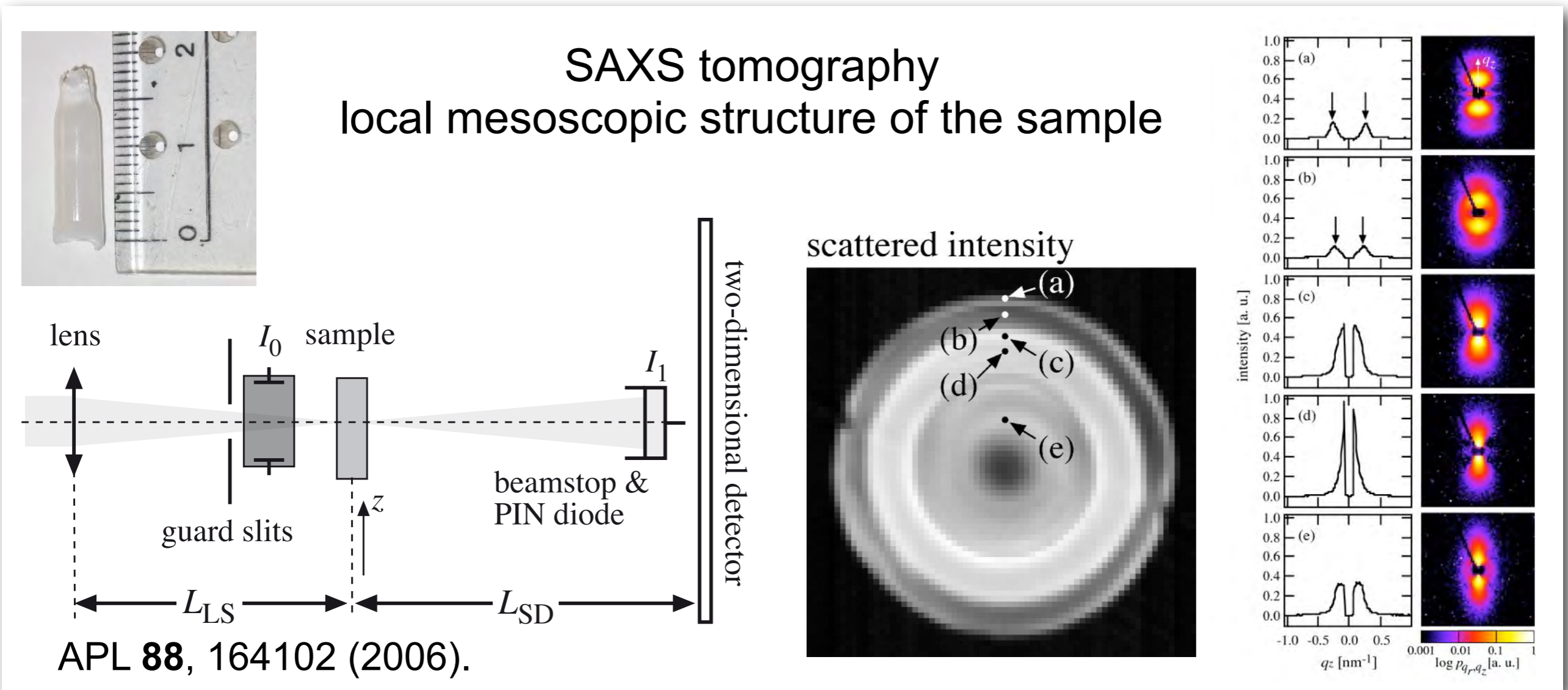
catalyst
in reactor
capillary



APL **82**, 3360 (2003).

X-Ray Scanning Microscopy and Tomography

- > Fluorescence microtomography
- > Tomographic absorption spectroscopy (XANES tomography)
- > Small-angle x-ray scattering tomography (SAXS tomography)



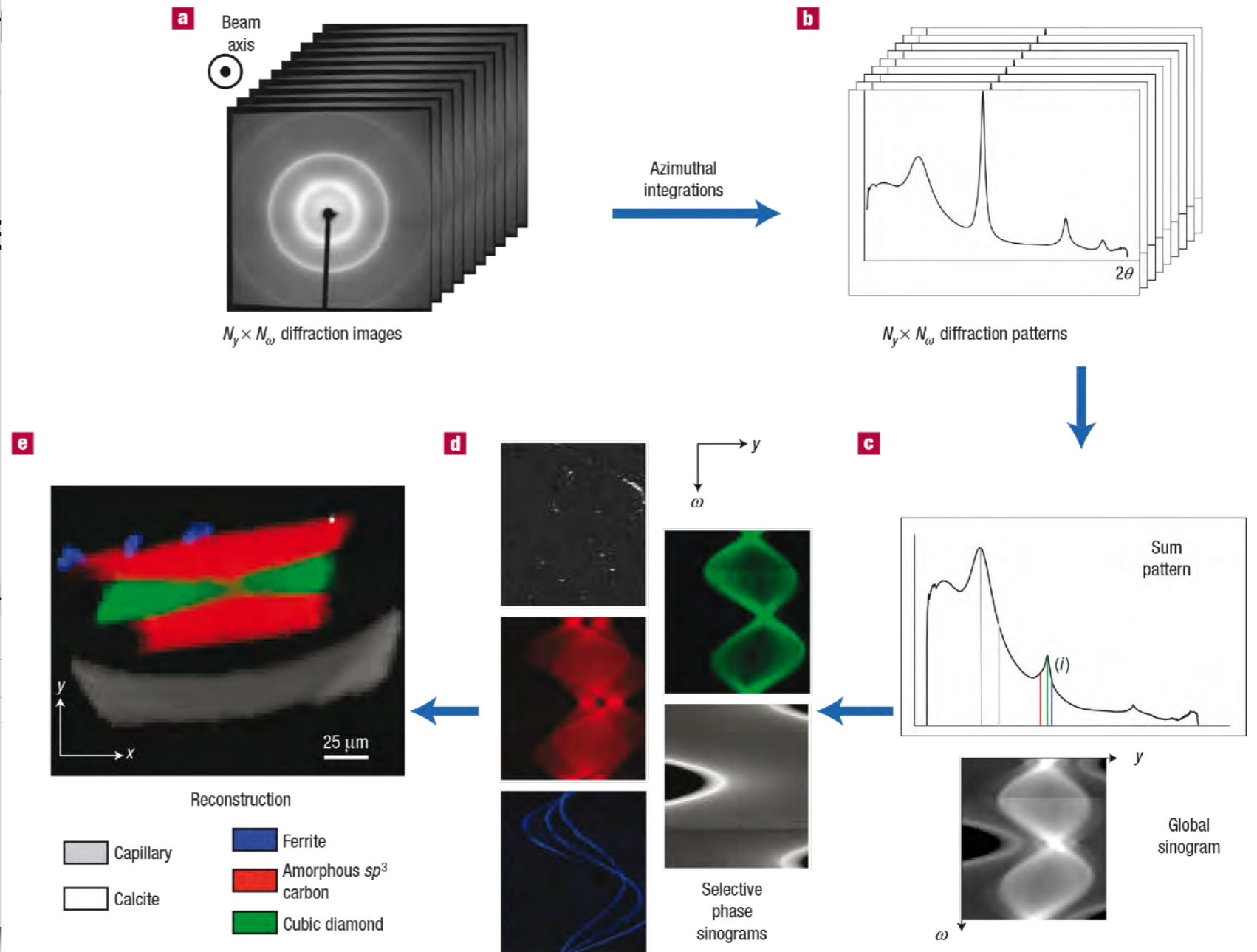
X-Ray Scanning Microscopy and Tomography

- > Fluorescence microtomography
- > Tomographic absorption
- > Small-angle x-ray scattering

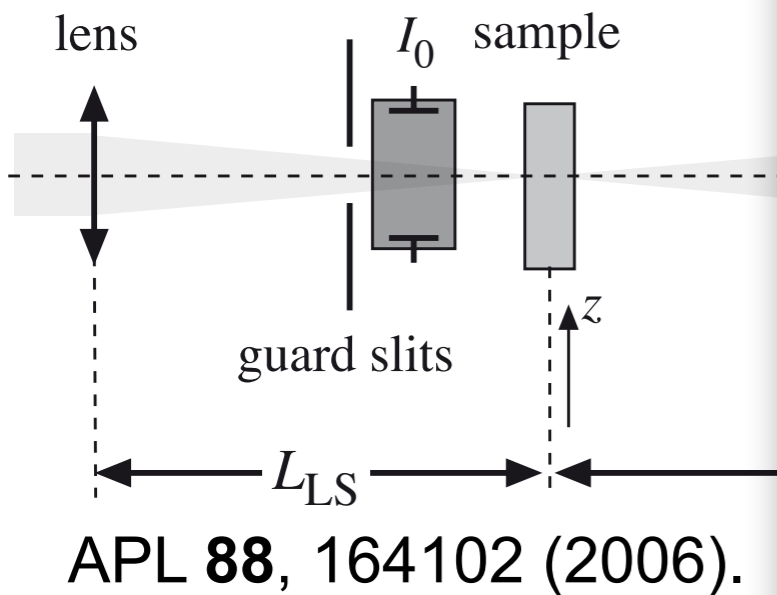


local me

Wide-Angle-X-Ray-Scattering (WAXS) Tomography



P. Bleuet, et al., Nat. Materials 7, 468 (2008).



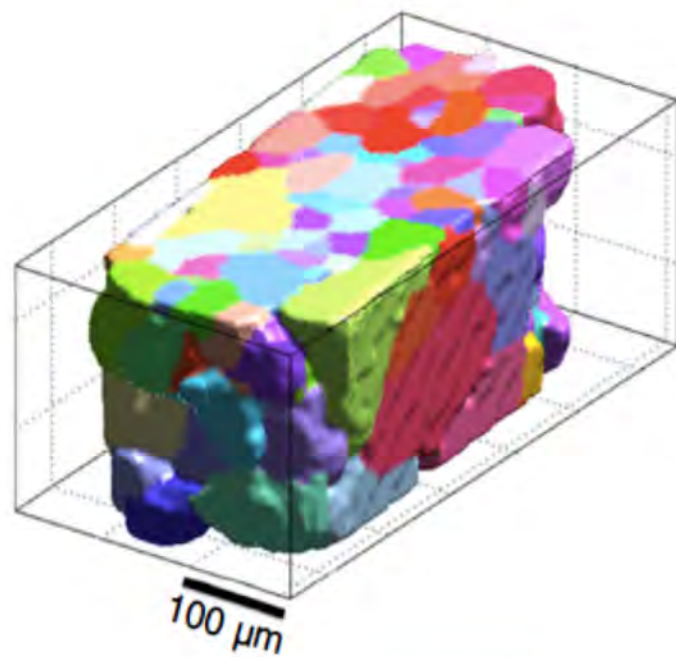
X-Ray Scanning Microscopy and Tomography

- > Fluorescence microtomography
- > Tomographic absorption
- > Small-angle x-ray scattering

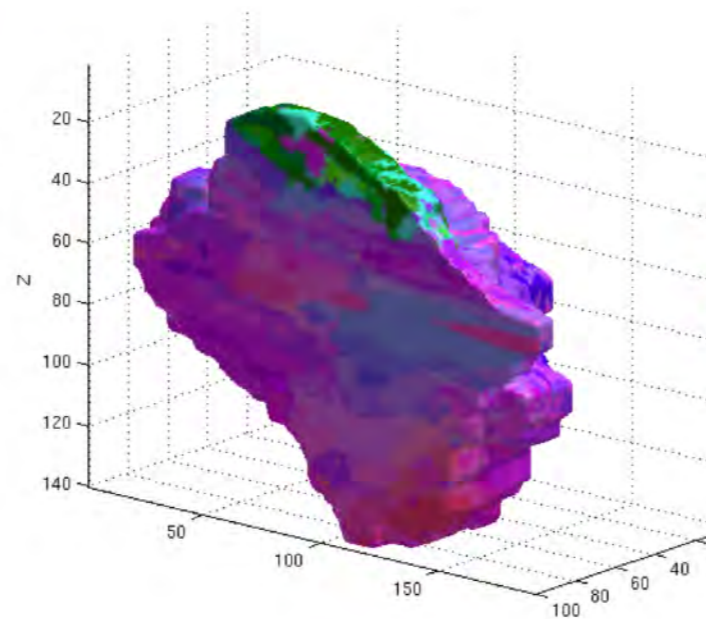
Wide-Angle-X-Ray-Scattering (WAXS) Tomography



3D diffraction microscope (H. Poulsen)



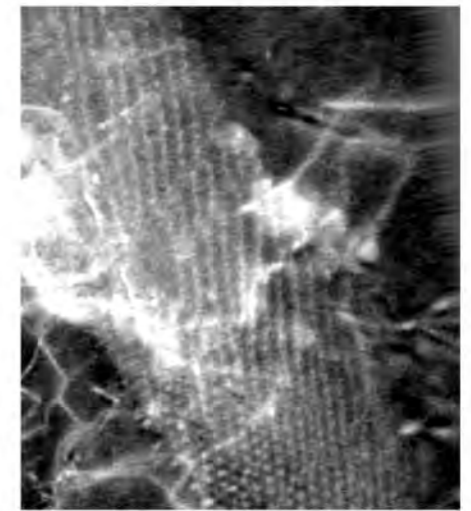
100 μm



10 μm



1 μm



100 nm

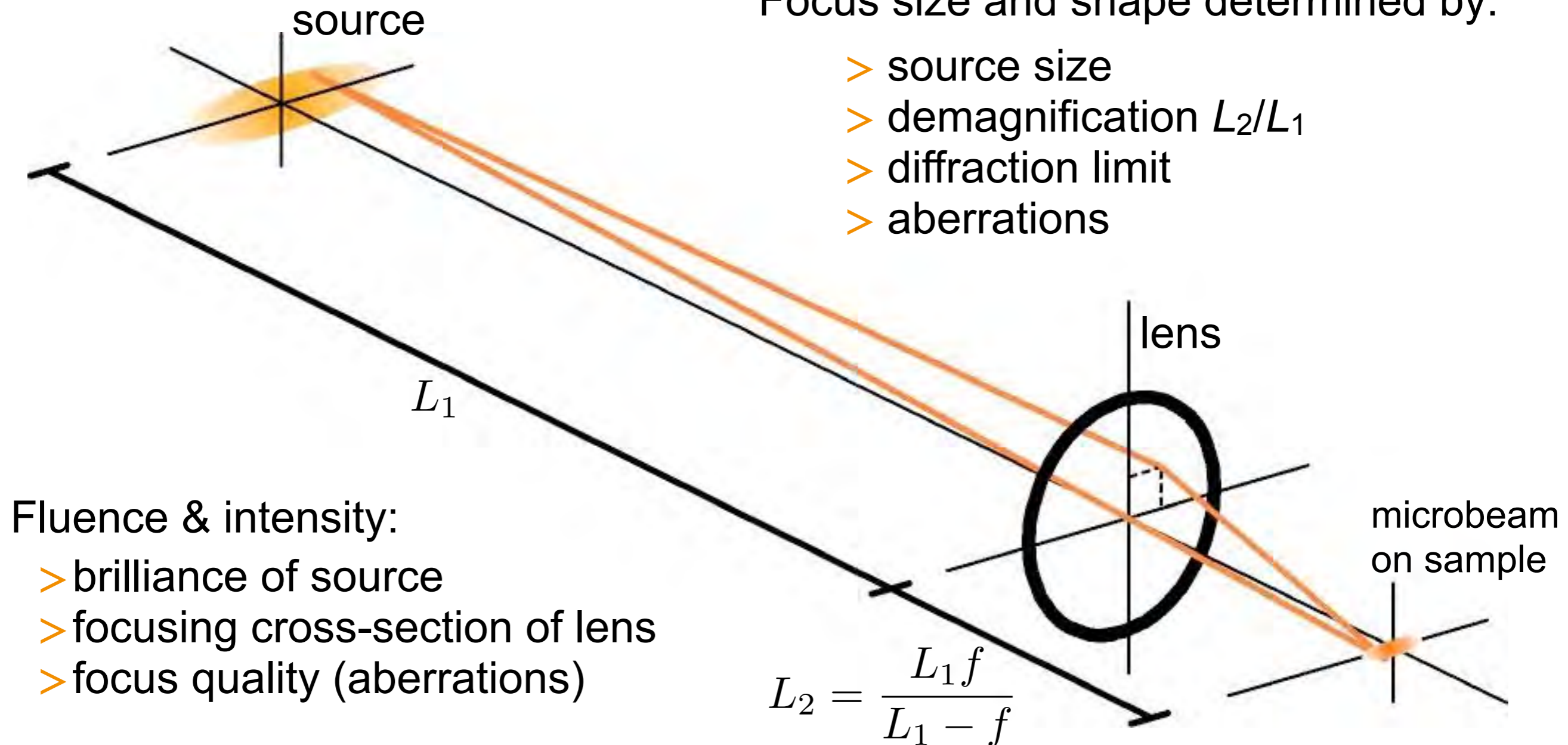
Compare 3D movies directly to 3D modelling on "all scales"

Scanning Microscopy with Hard X-Rays

Source is imaged onto the sample to create an intensive micro-/nanobeam:

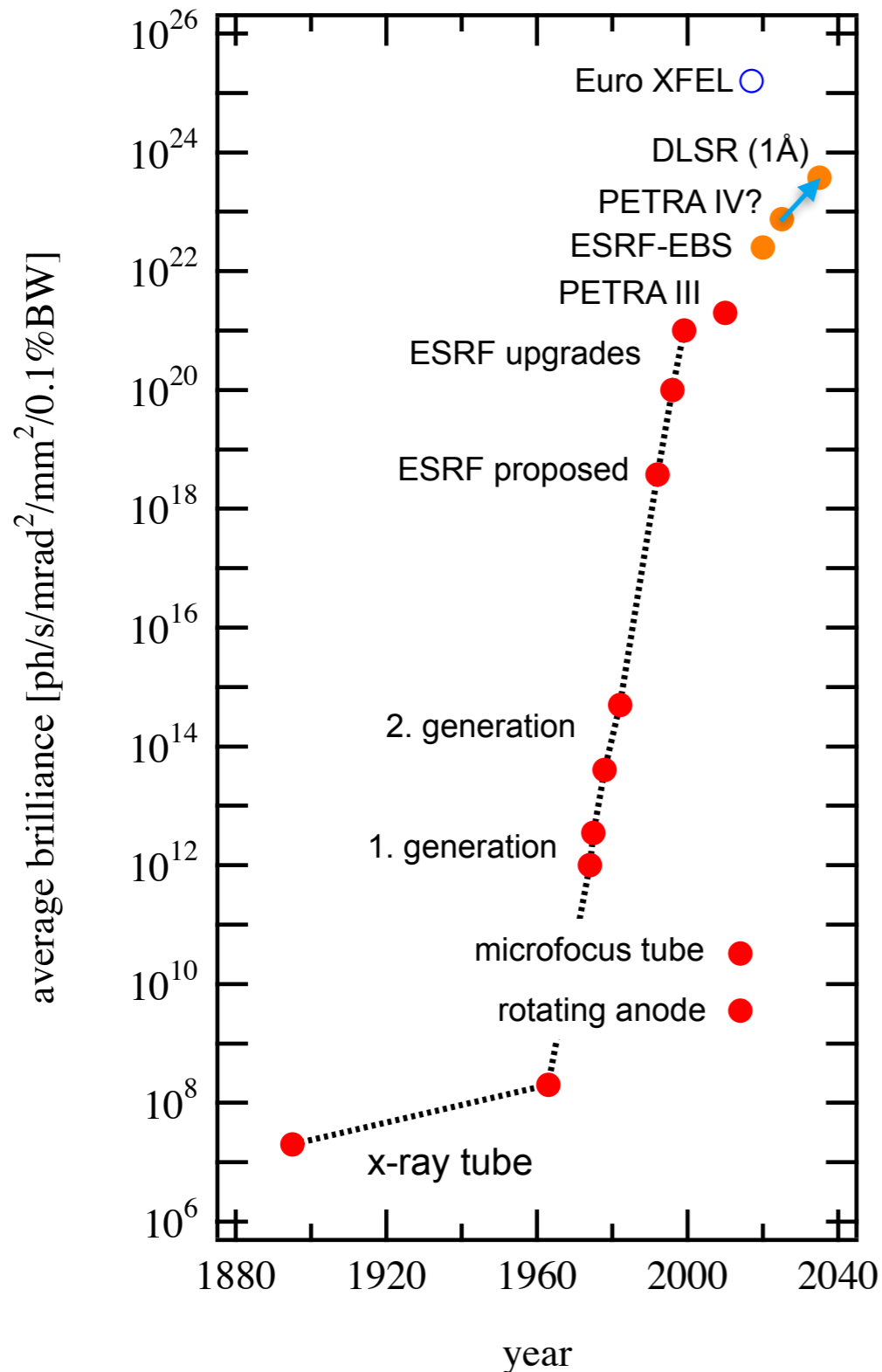
Focus size and shape determined by:

- > source size
- > demagnification L_2/L_1
- > diffraction limit
- > aberrations



Spectral Brightness

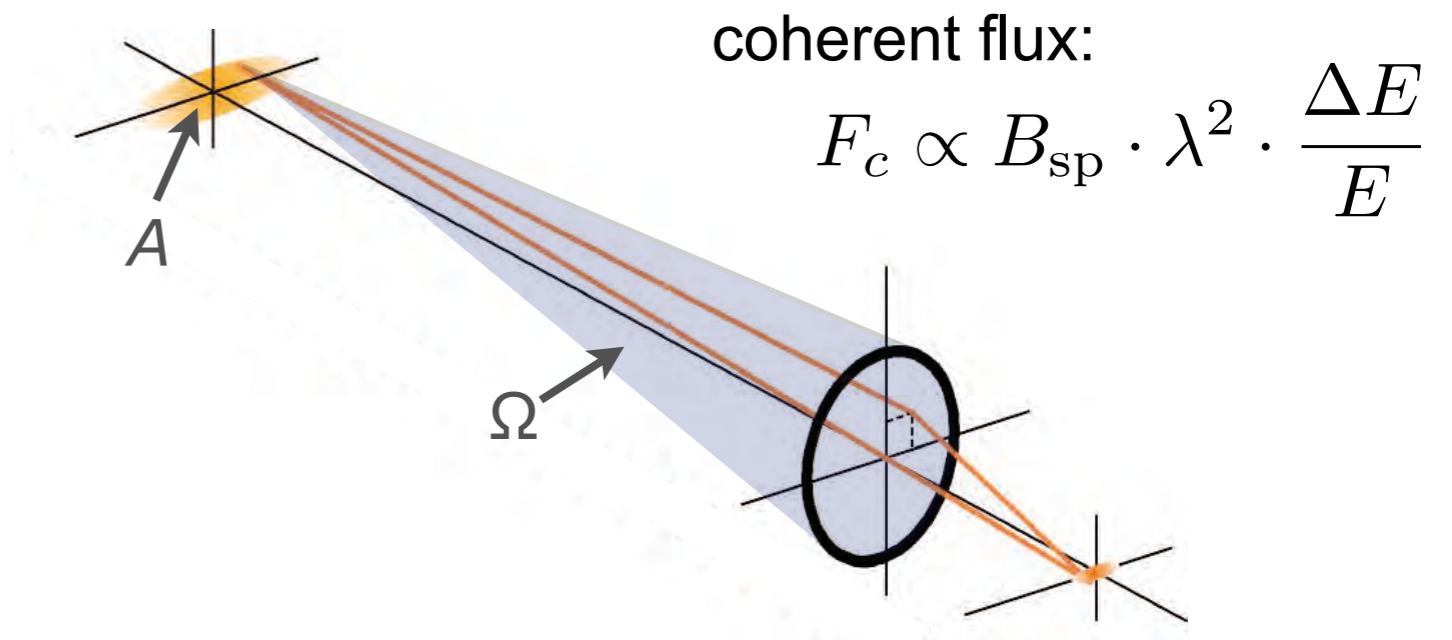
10000x more “light” per decade (since 1965)!!



Spectral brightness:

$$B_{\text{sp}} = \frac{F}{\Omega \cdot A \cdot \Delta E / E}$$

Flux per phase-space volume



Improvements in brightness:

- > faster measurements (time resolution)
- > nano-imaging (spatial resolution)
- > spectroscopy (energy resolution)

Fluorescence Tomography

Example: investigating the ion transport in plants

Fluorescence analysis of plants:

- > strong diffusion of elements
- > cell structure complicated and delicate

Difficult sample preparation

- > cryo sections
- > fracture surfaces

ideal:

nondestructive probe of
inner structures of sample

root of Mahogany
tree

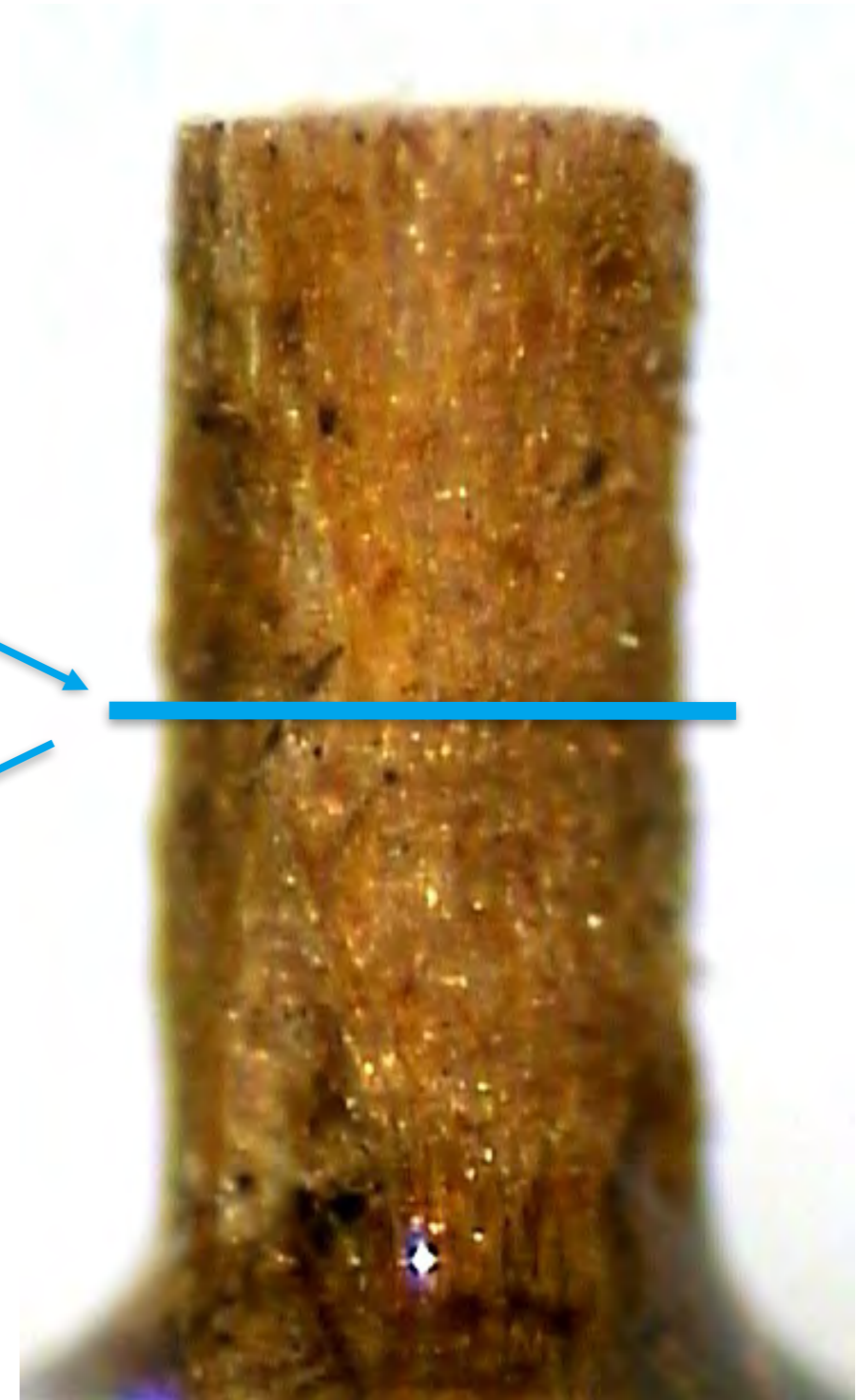
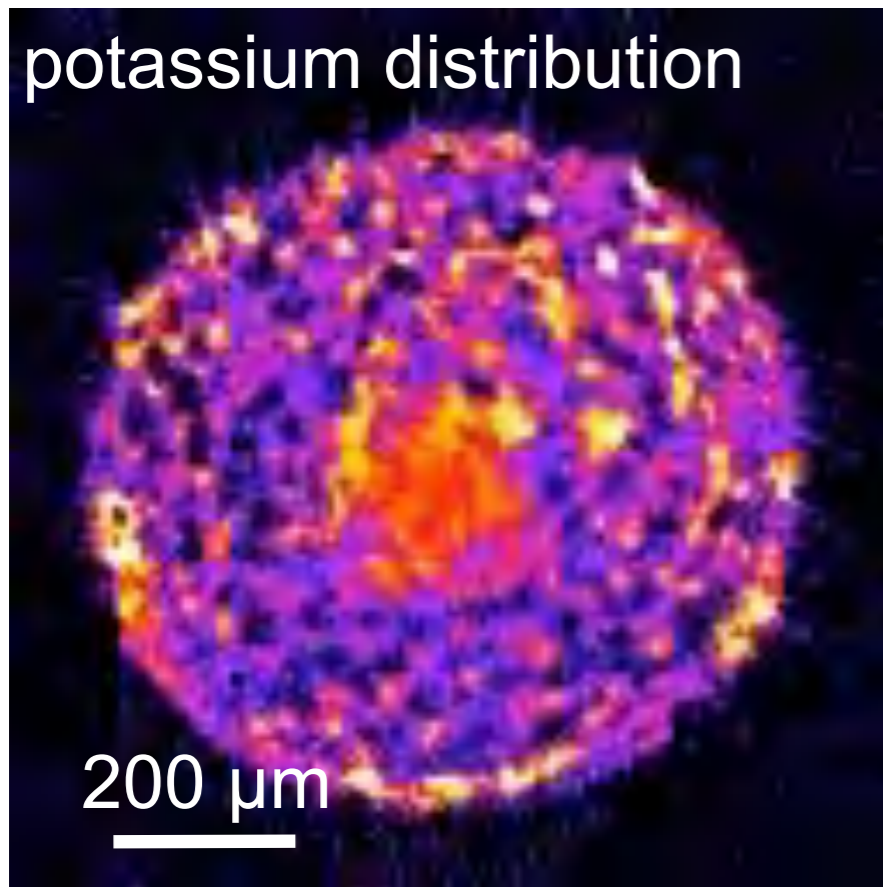


Fluorescence Tomography

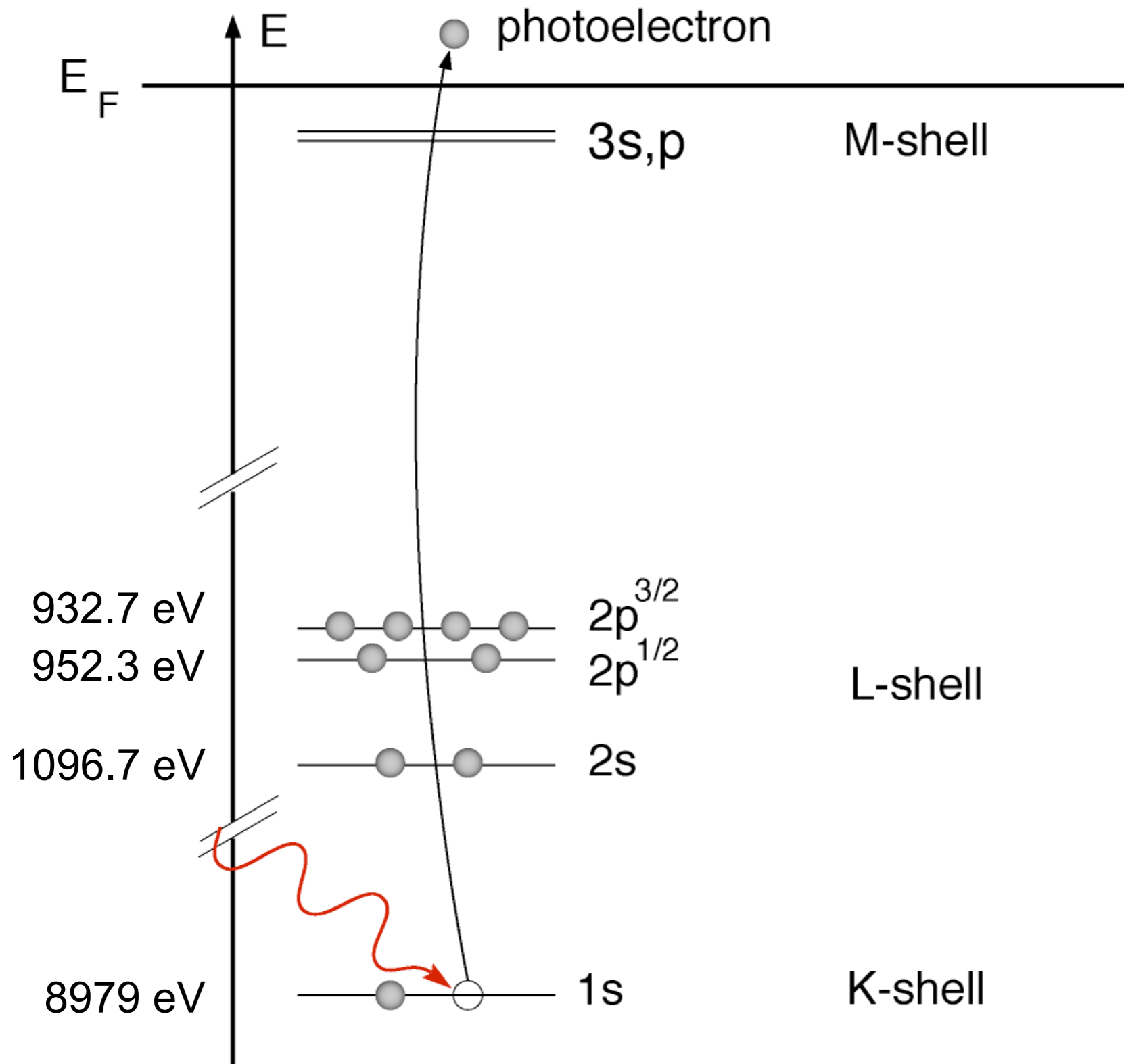
Root of Mahogany tree

element distribution on virtual
section through sample

Example:



X-ray Fluorescence & Auger Prozess



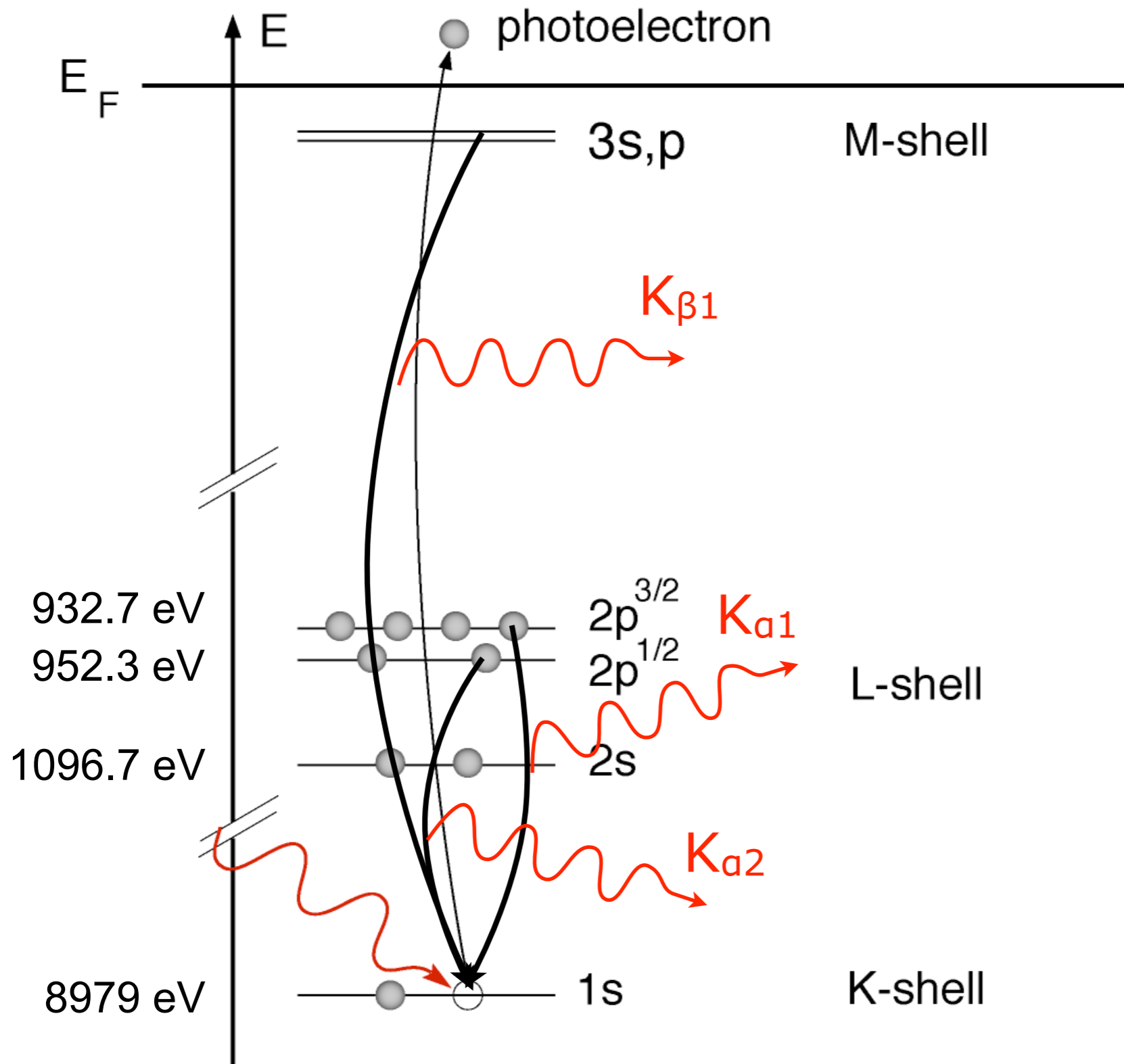
X-ray Absorption

leaves atom in excited state (core hole):

Secondary processes:

> fluorescence

X-ray Fluorescence & Auger Prozess



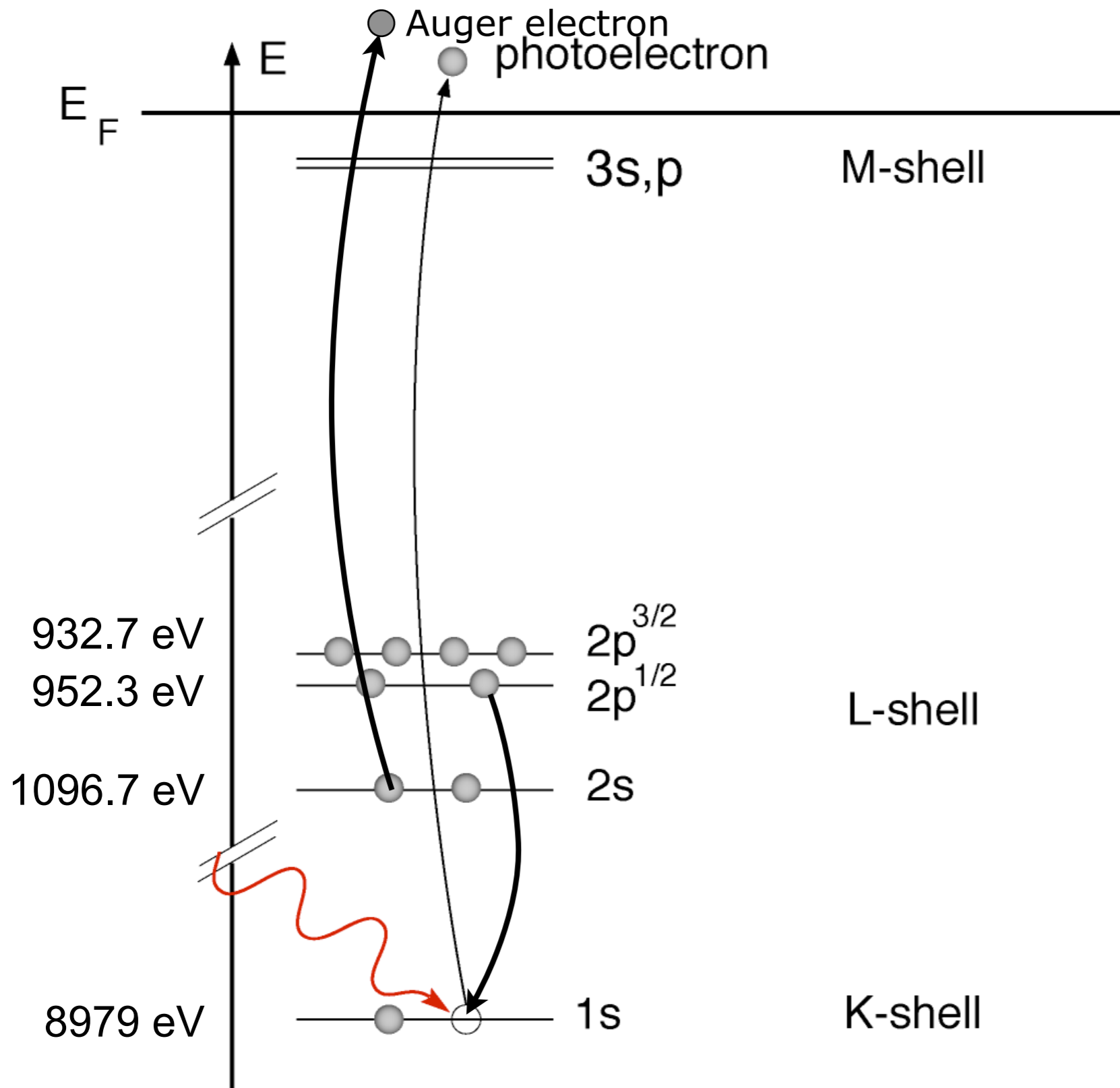
X-ray Absorption

leaves atom in excited state (core hole):

Secondary processes:

> fluorescence

Röntgenfluoreszenz & Augerprozess



X-ray Absorption

leaves atom in excited state (core hole):

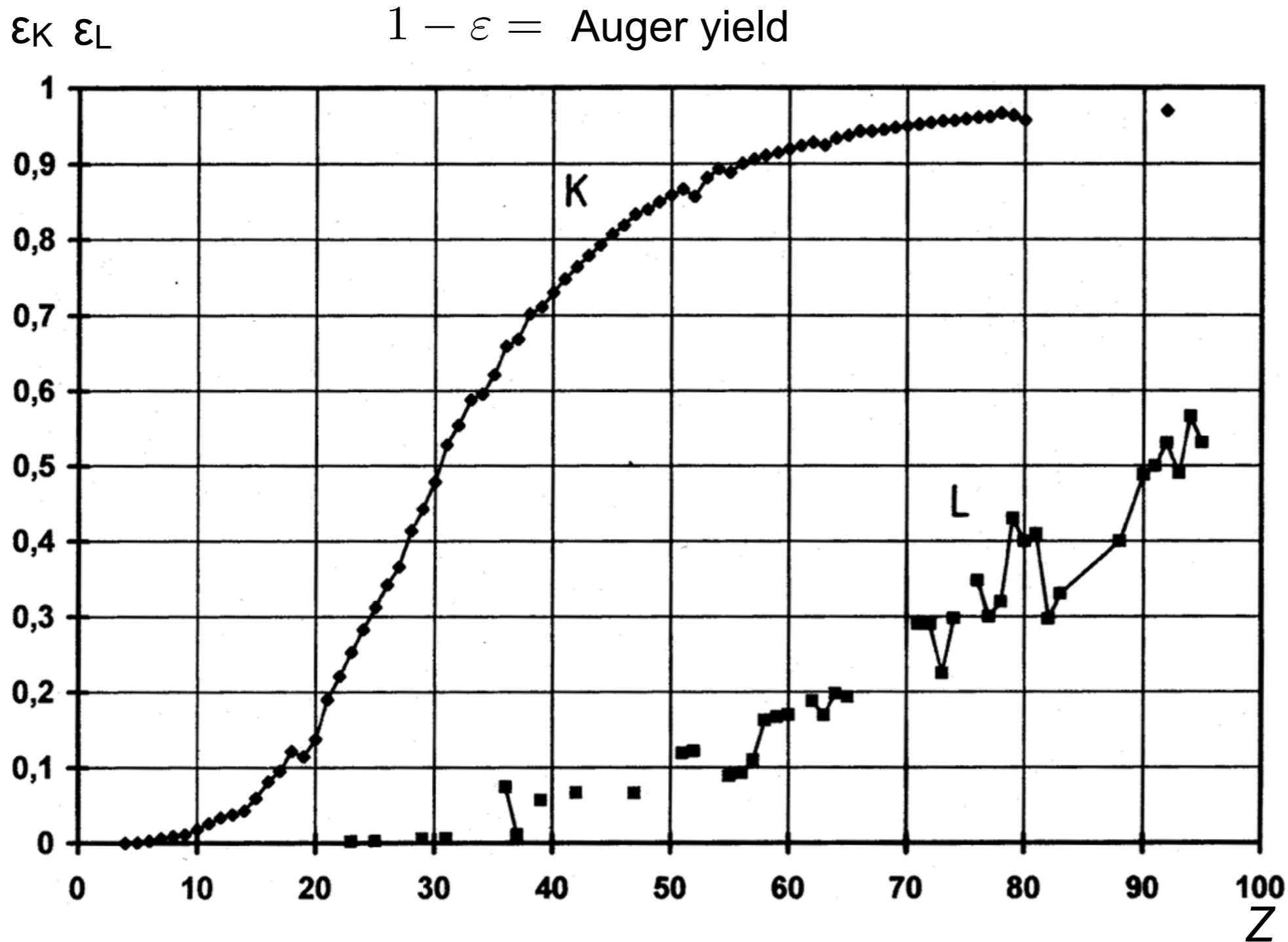
Secondary processes:

> fluorescence

> emission of Auger electron

both processes occur with certain probability

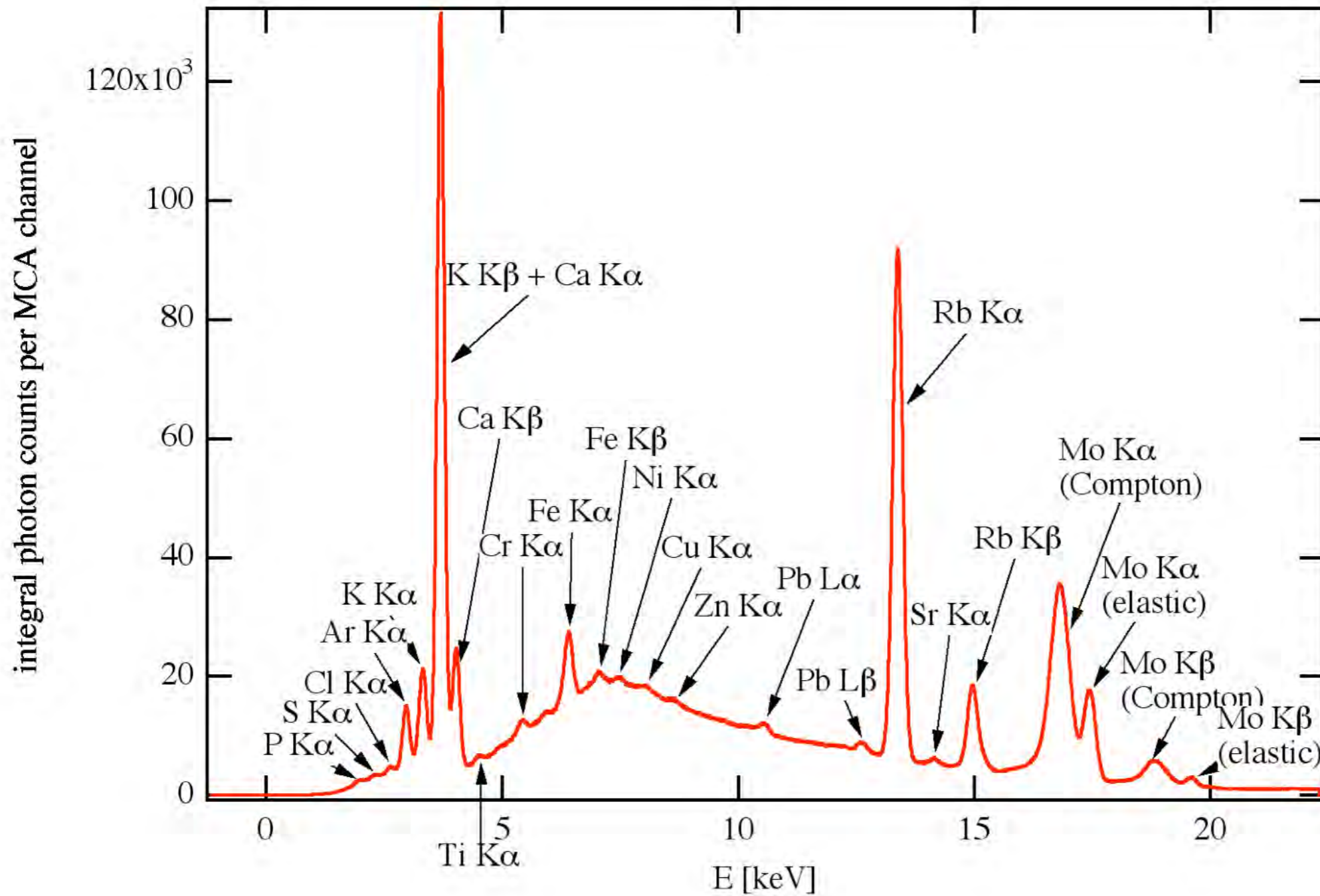
Fluorescence Yield



element
specific

Fluorescence
dominates at higher
binding energies for
core hole excitation
(growing with atomic
number Z)

Fluorescence Spectrum



Illuminated atoms emit characteristic fluorescence radiation!

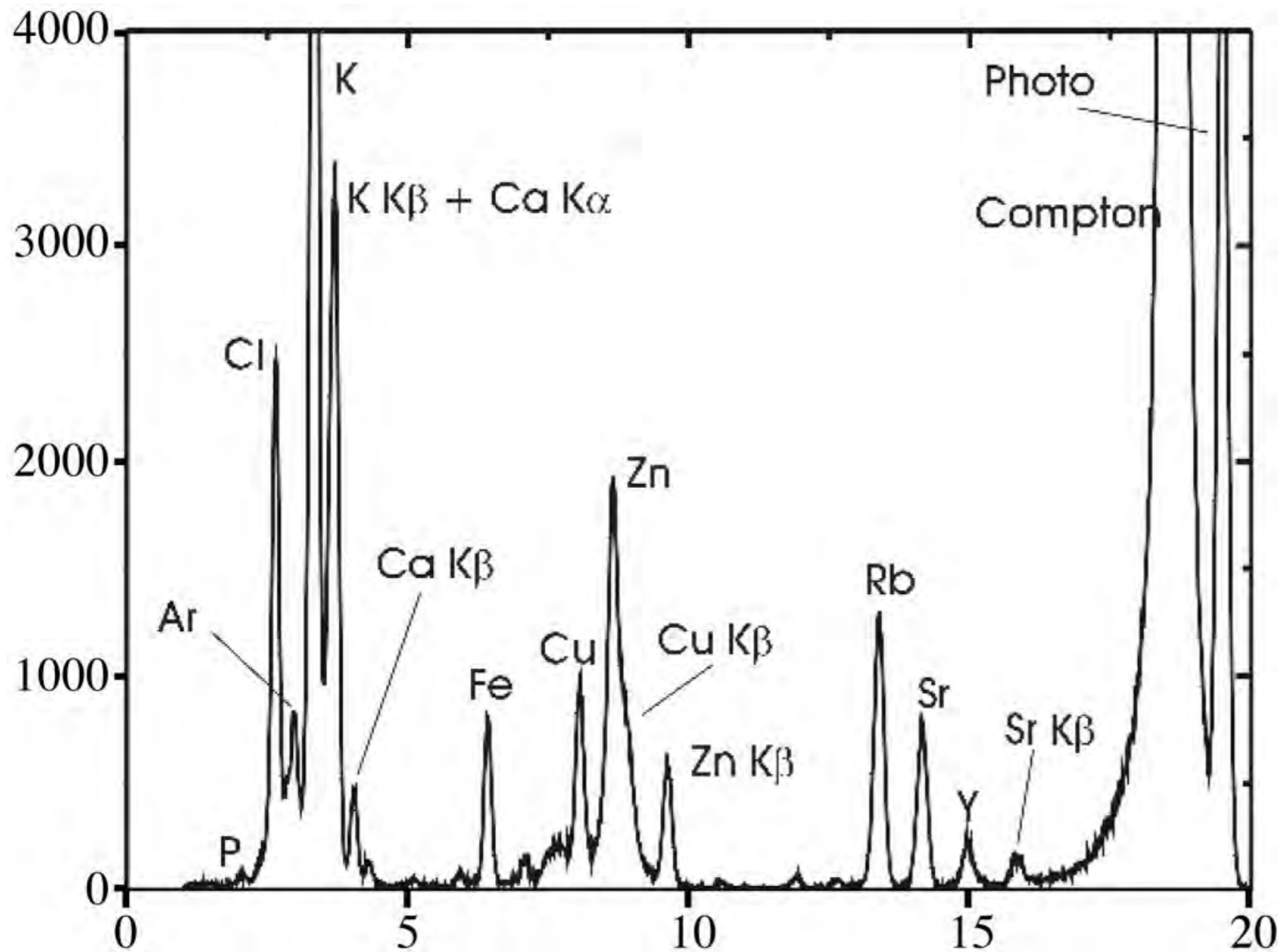
Example spectrum excitation with x-ray tube:

Background due to scattered spectrum of x-ray tube

Limitation of detection limits by background!

Excitation with Monochromatic Synchrotron Radiation

Example: undulator radiation (Si 111 monochrom.): 19.5 keV

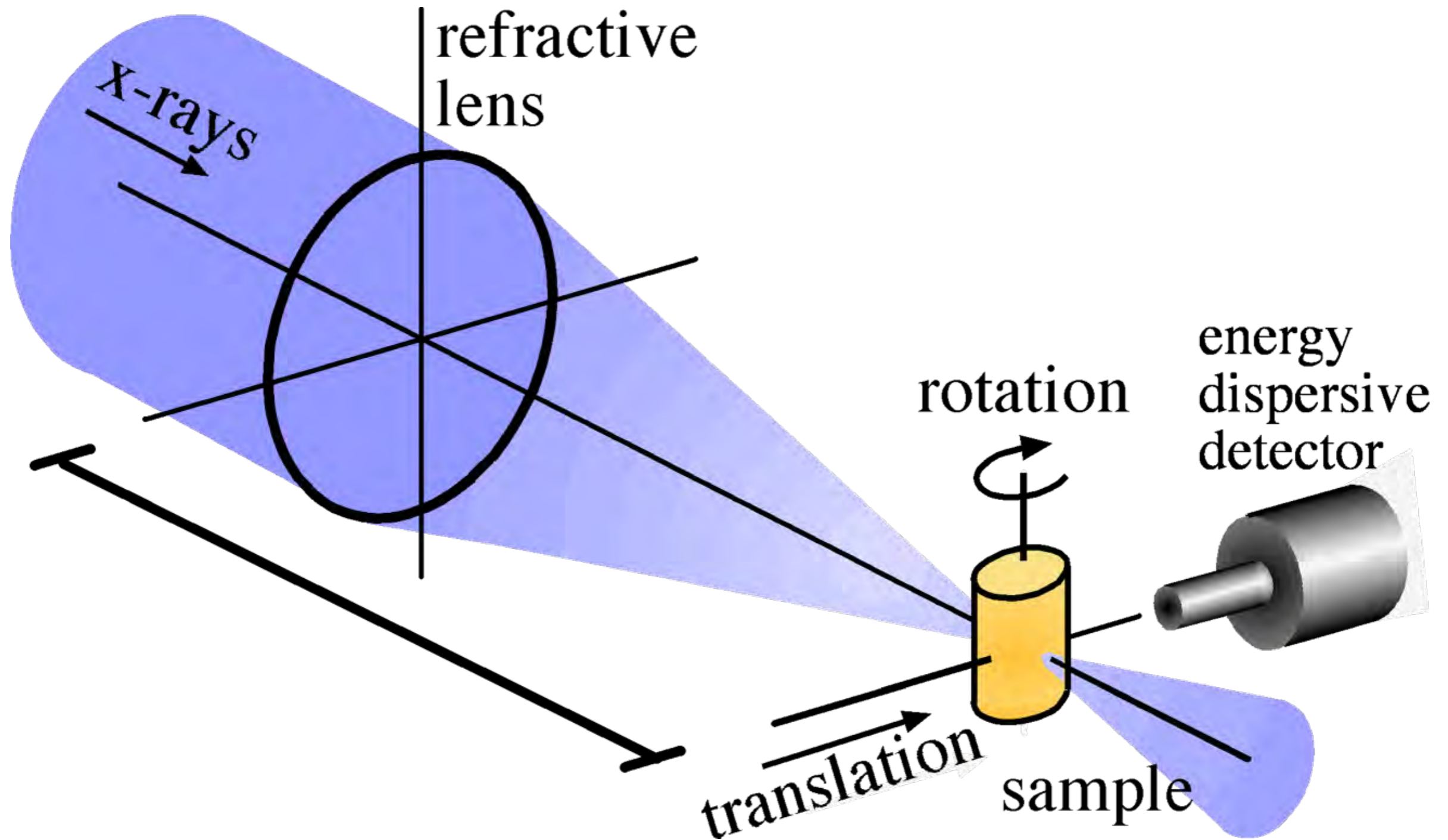


No background due to scattered radiation at fluorescence energy

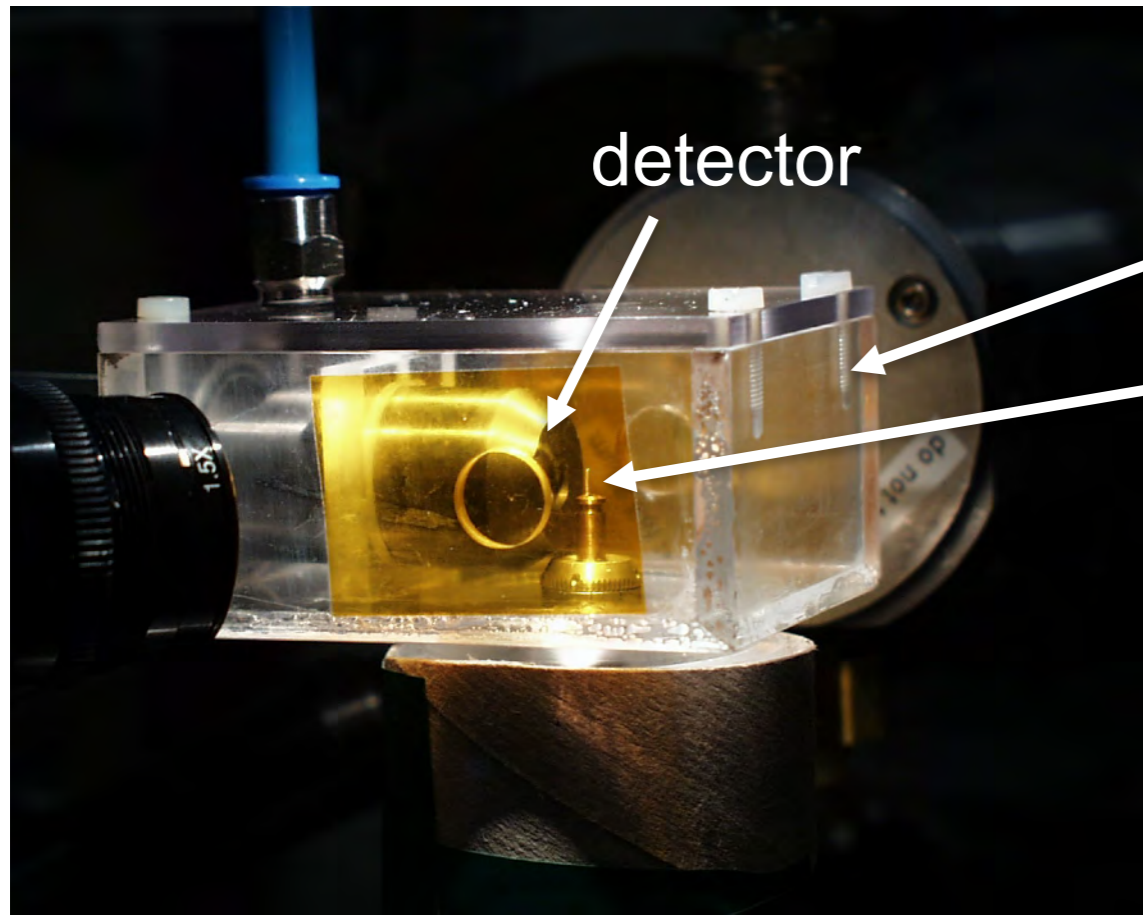
High signal-to-background ratio!!

very low detection limits possible (ppb-level)!

Scanning Probe: Fluorescence Microtomography



Fluorescence Microtomography

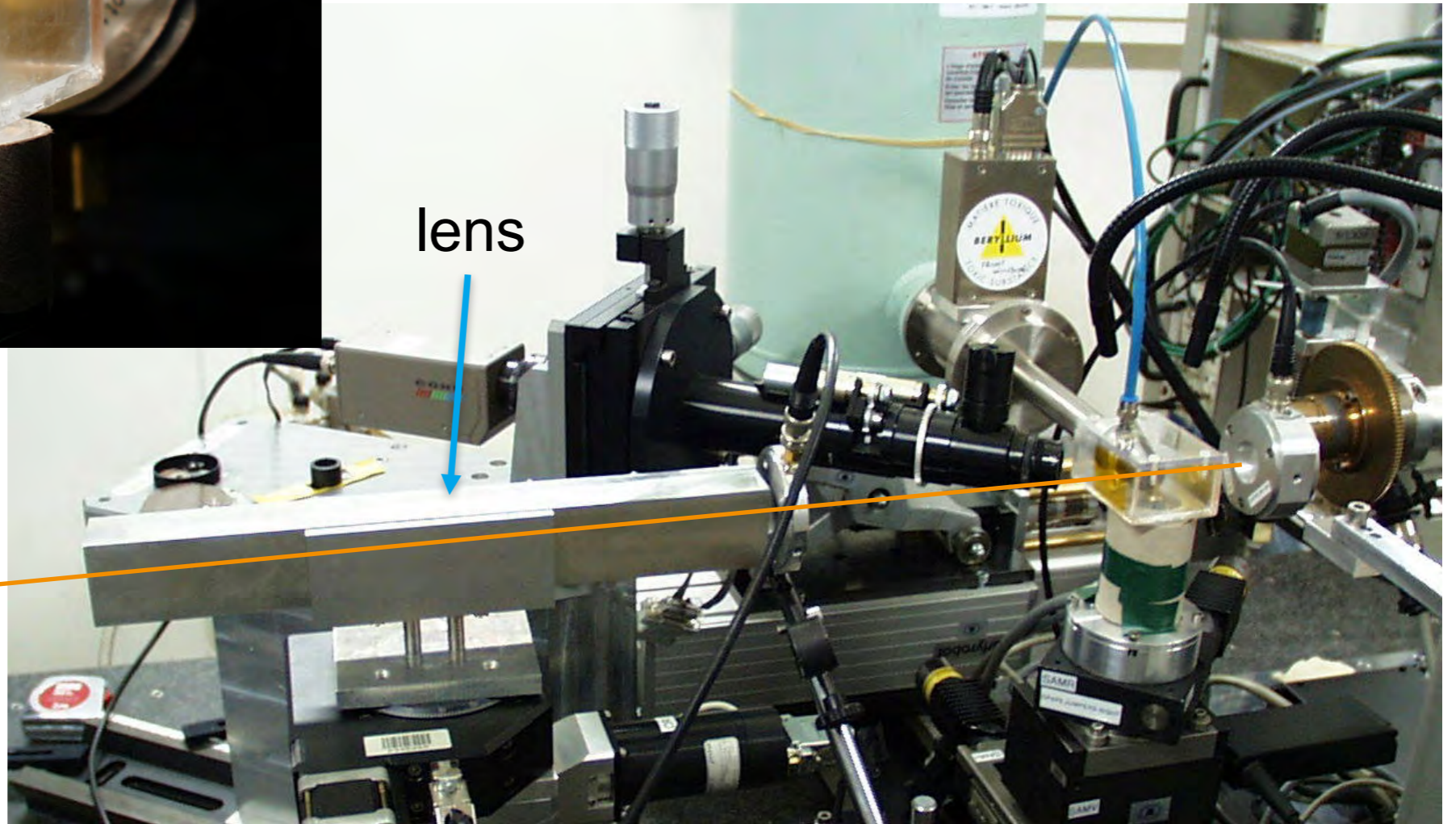


detector

He chamber
sample

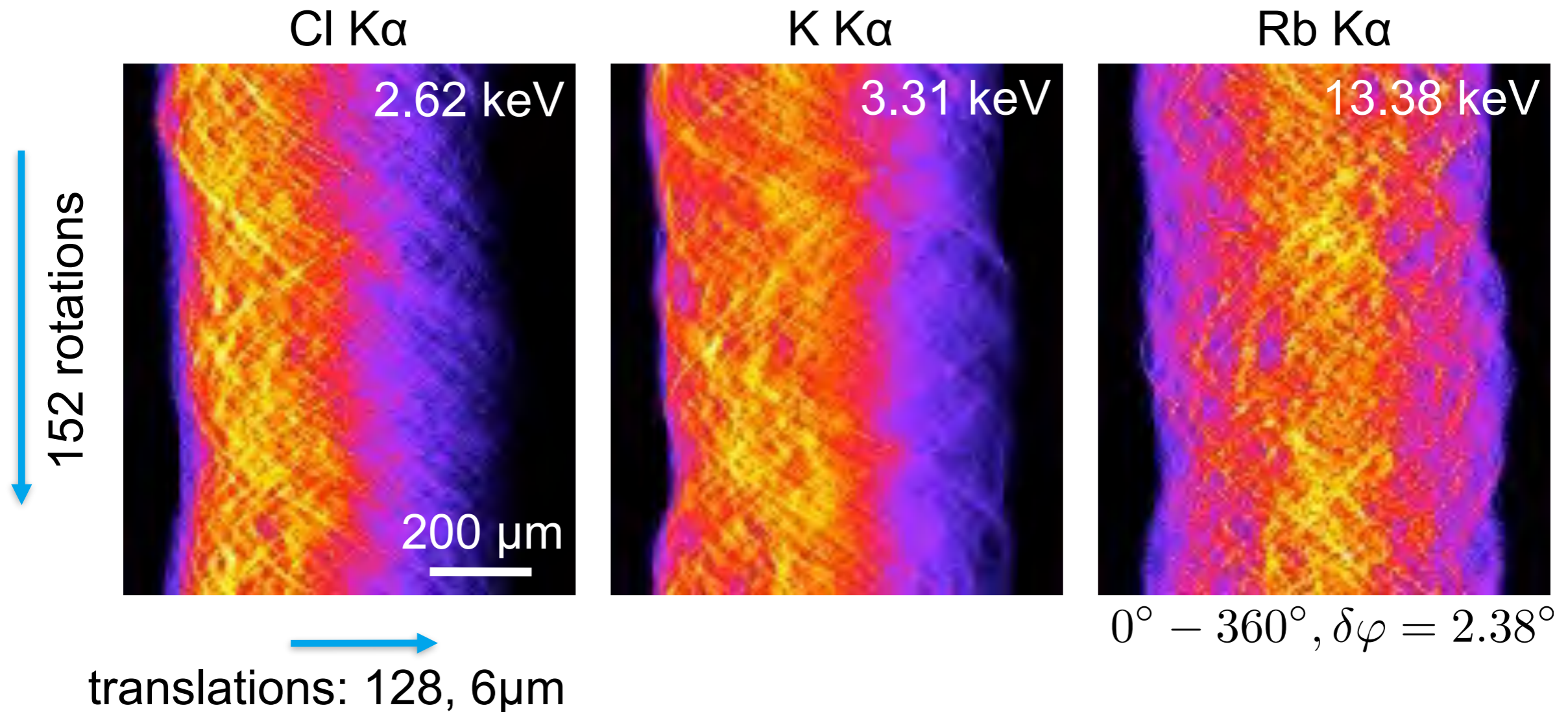
lens

synchrotron
radiation



Fluorescence Tomography: Measured Data

Sinograms:

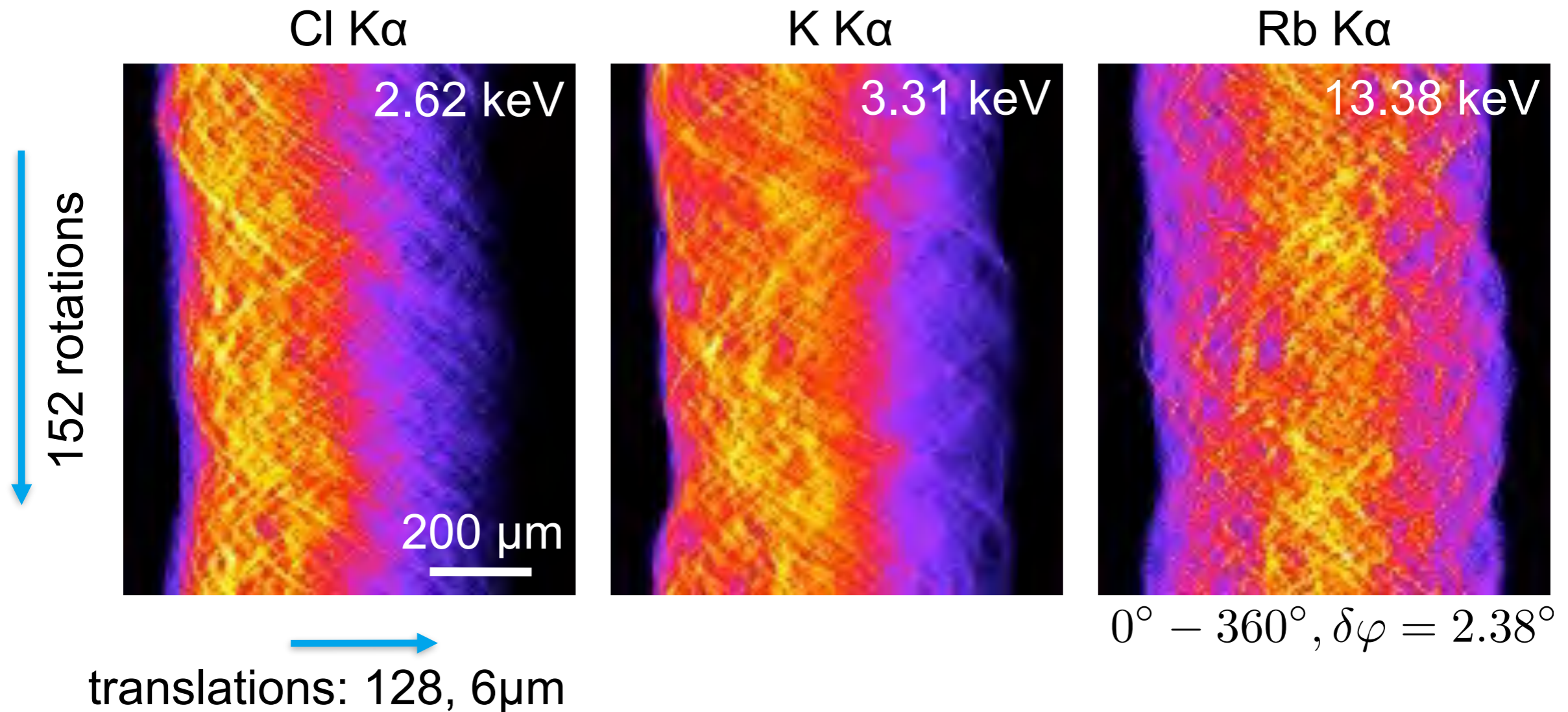


experimental parameters:

- > energy: 19.5 keV
- > refractive lens (Al): $N = 150$, $f = 45.4$ cm, $m = 1/127$
- > beam size: $1.5 \times 6 \mu\text{m}^2$, flux: $1.1 \cdot 10^{10}$ ph/s

Fluorescence Tomography: Measured Data

Sinograms:



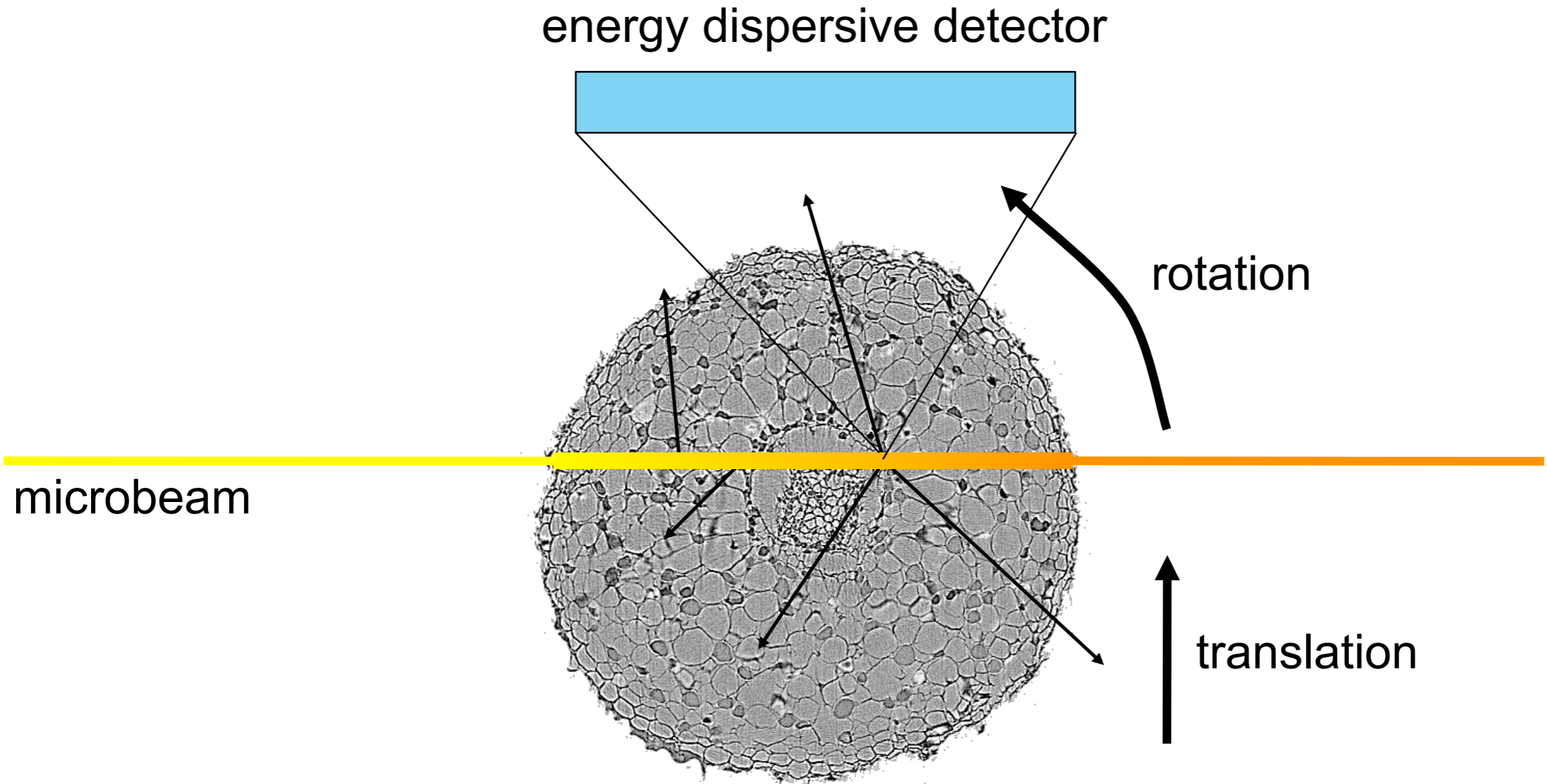
Symmetry:

$$I_{i\nu}(-r, \varphi + \pi) = I_{i\nu}(r, \varphi)$$

only holds for Rb!

Absorption of fluorescence radiation:
asymmetry in sinogram.

Fluorescence Tomography: Model

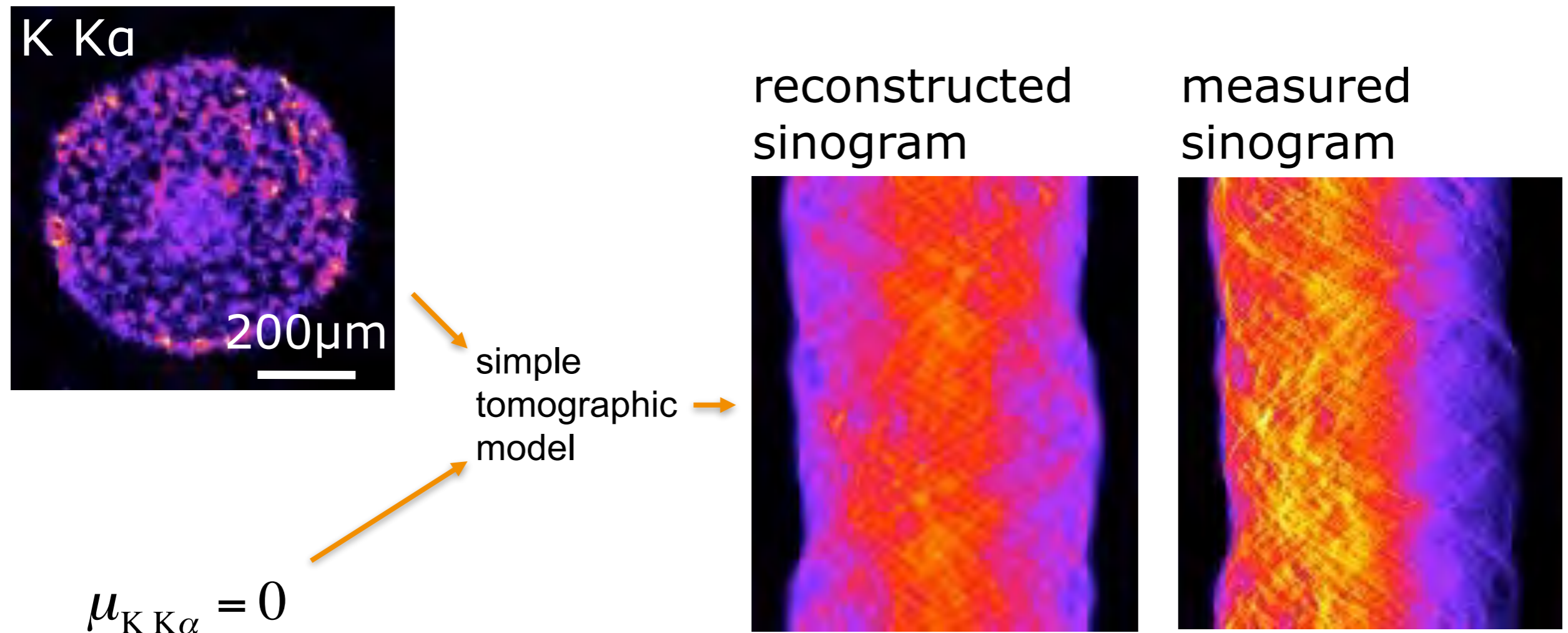


$$I_{i\nu}(r, \varphi) = I_0 \int ds \left[e^{-\int_{-\infty}^s ds' \mu_0(x, y)} \cdot p_{i\nu}(x, y) \cdot \int d\gamma e^{-\int dr' \mu_{i\nu}(x, y)} \right]$$

Absorption Correction

Example: potassium distribution in Mahogany root

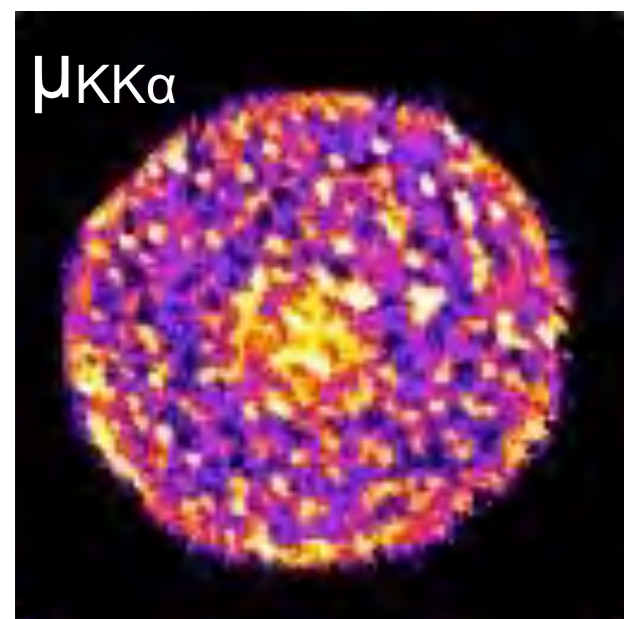
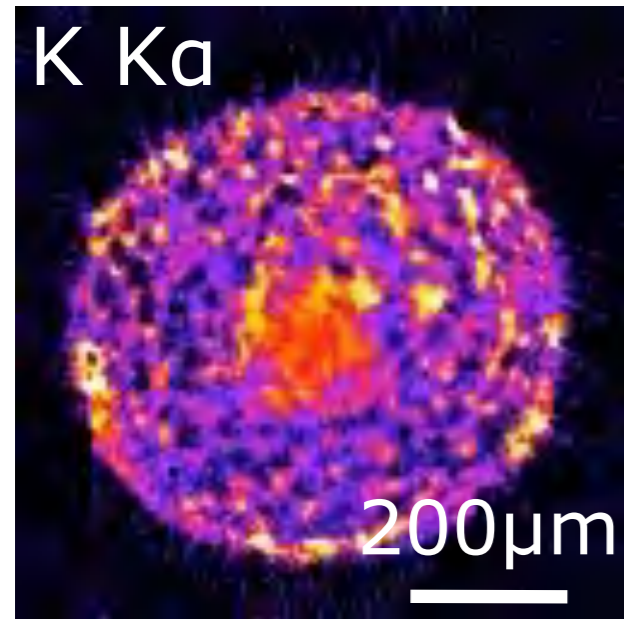
Disregarding attenuation of fluorescence:



Absorption Correction

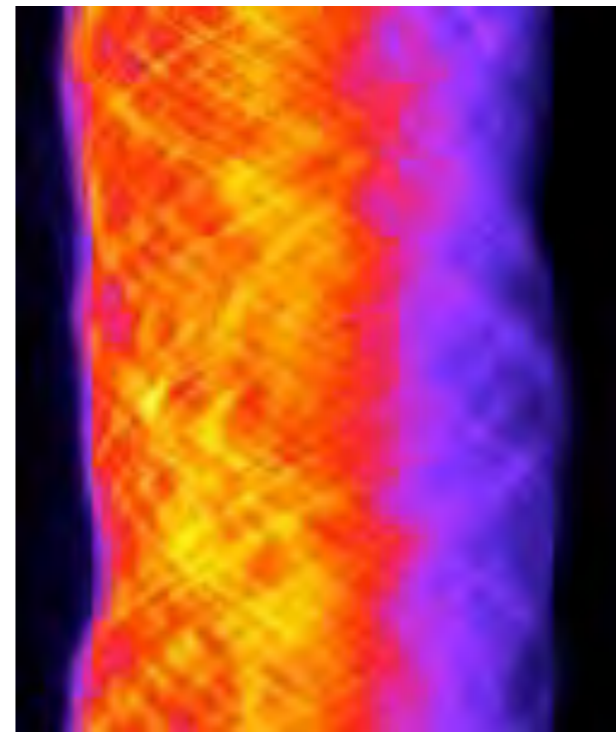
Example: potassium distribution in Mahogany root

Accounting for attenuation of fluorescence:

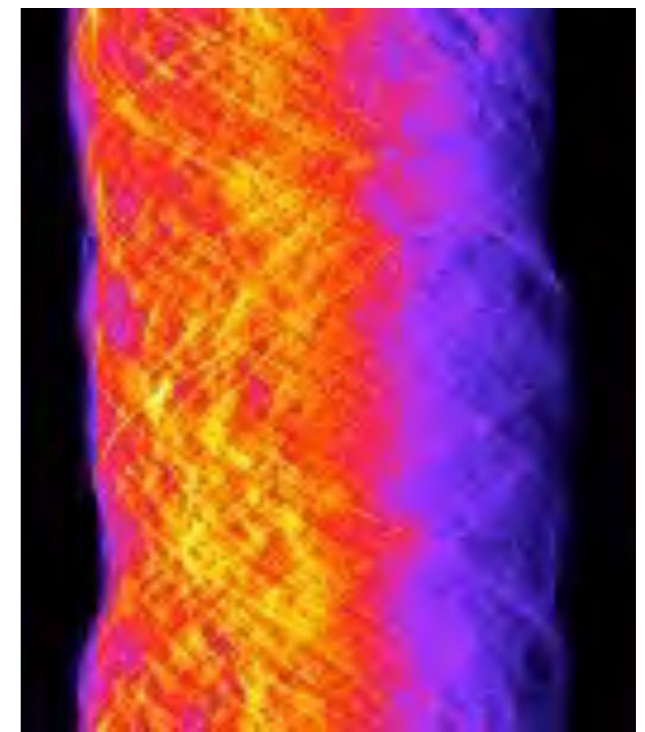


refined
tomographic
model

reconstructed
sinogram



measured
sinogram



C. Schroer, Appl. Phys. Lett. **79**, 1912 (2001).

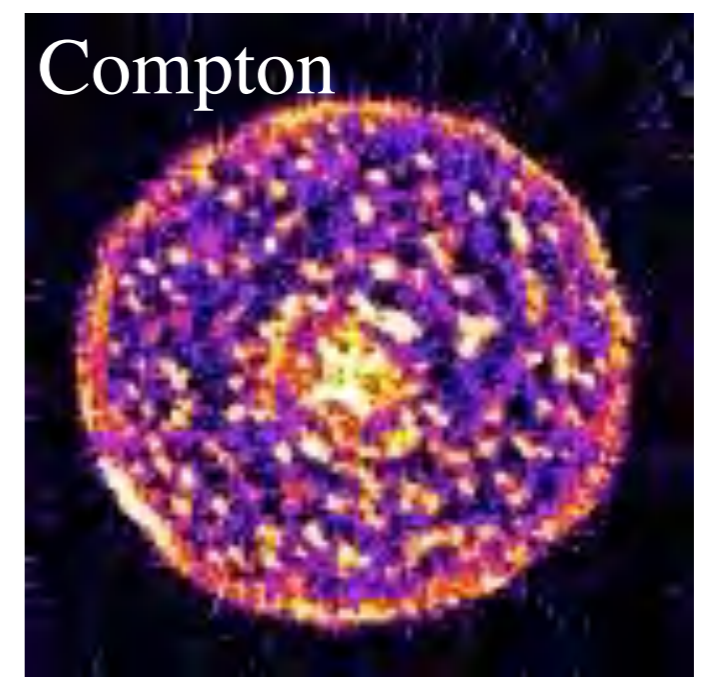
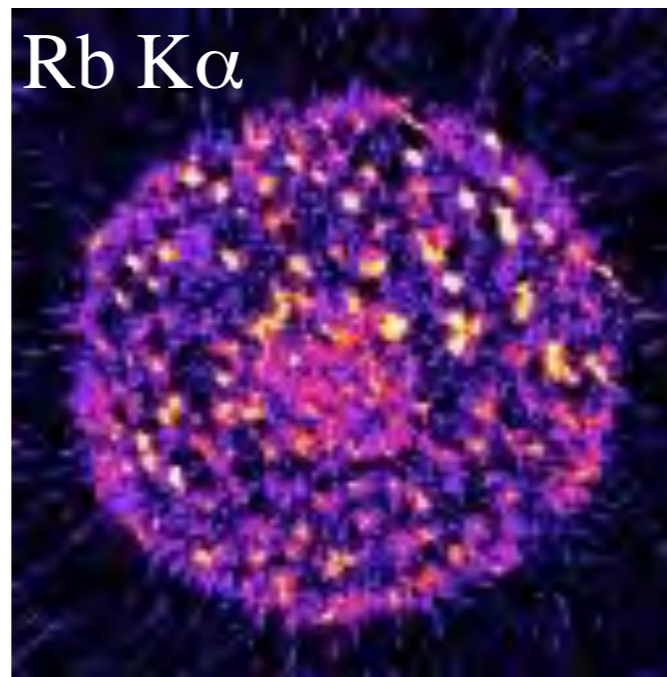
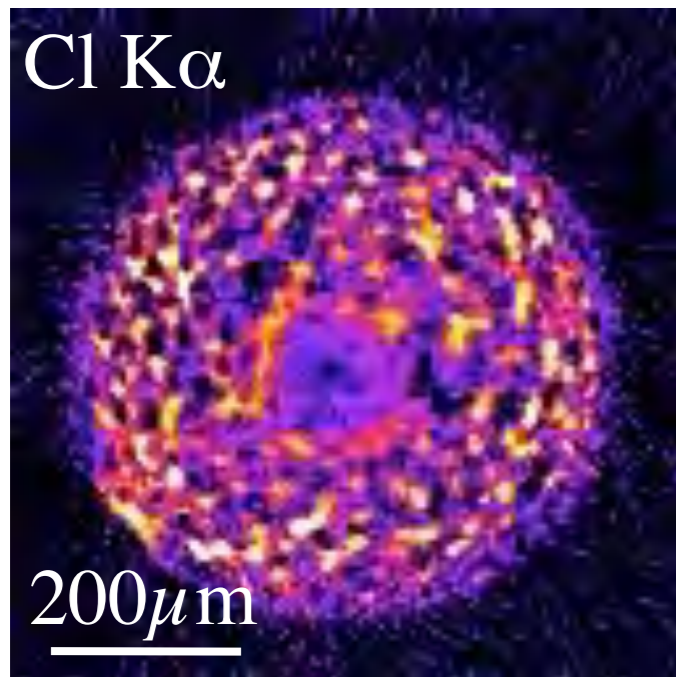
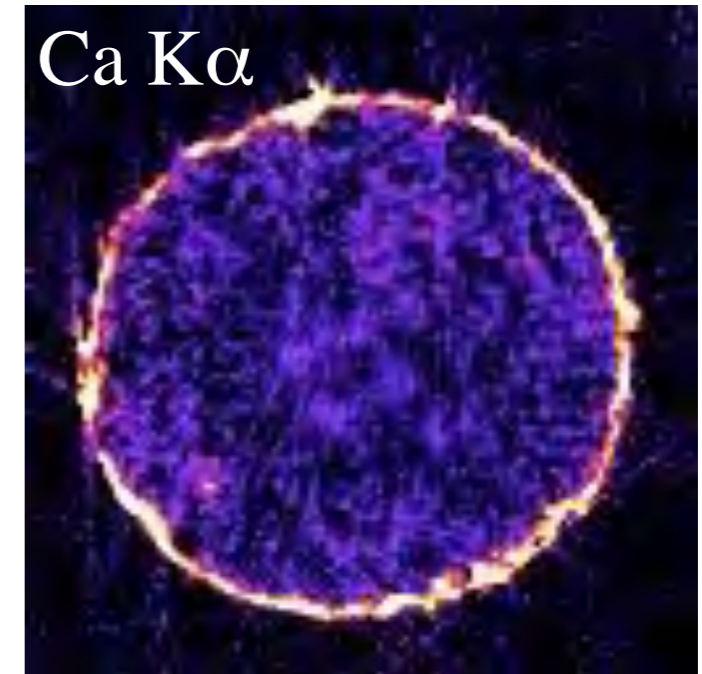
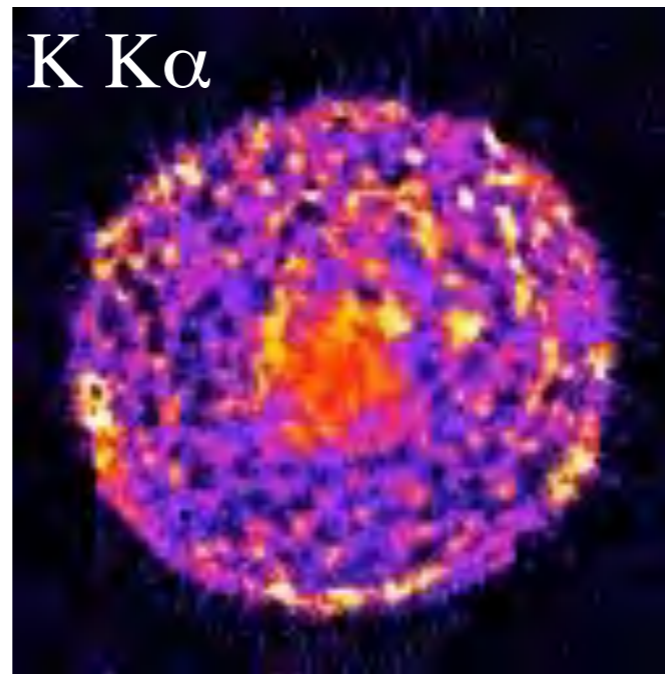
Christian G. Schroer | DESY Summer Student Programme | August 20, 2019 | page 52



Fluorescence Tomography

root of Mahogany
tree

pixel size: 6 μm



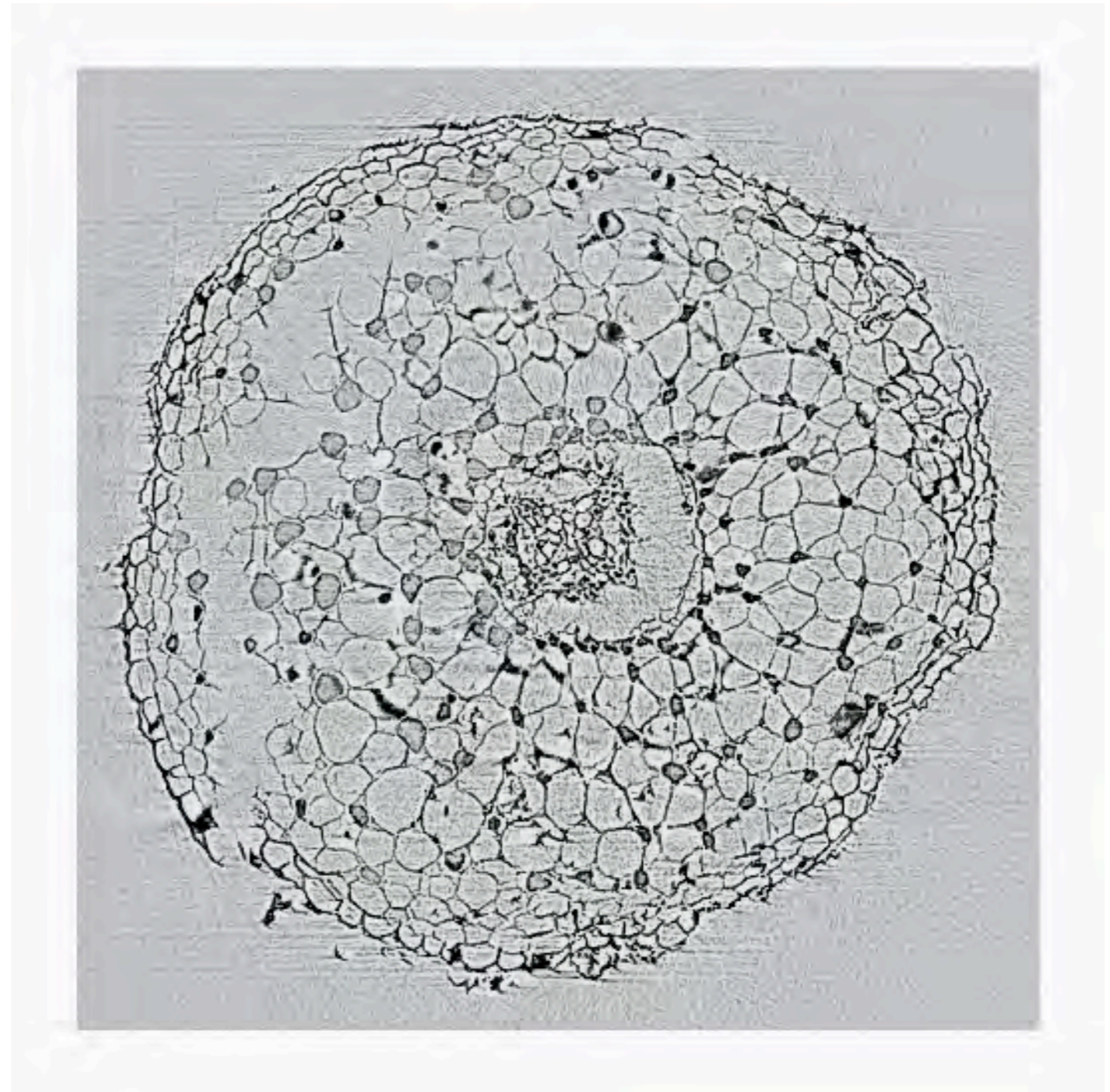
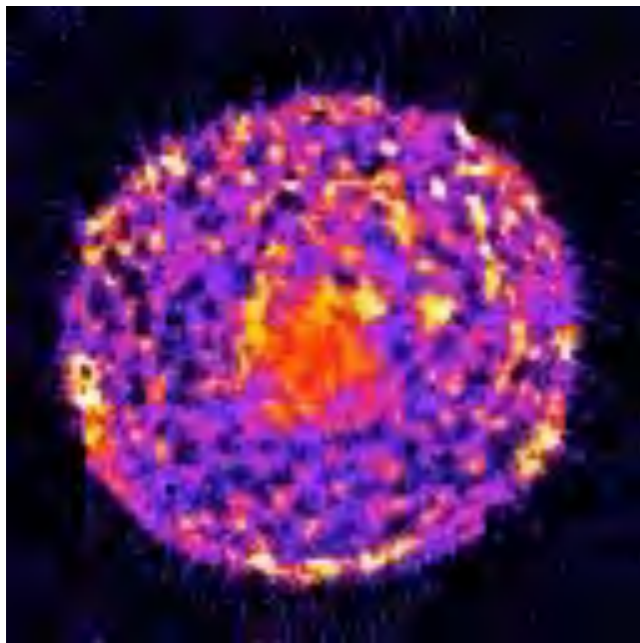
Fluorescence Tomography

Take advantage of:

- > large penetration depth of x-rays
- > element specific contrast

Compare with structural data from transmission tomogram:

K $K\alpha$



SAXS Tomography: Local Nanostructure

SAXS: Small-Angle X-ray Scattering

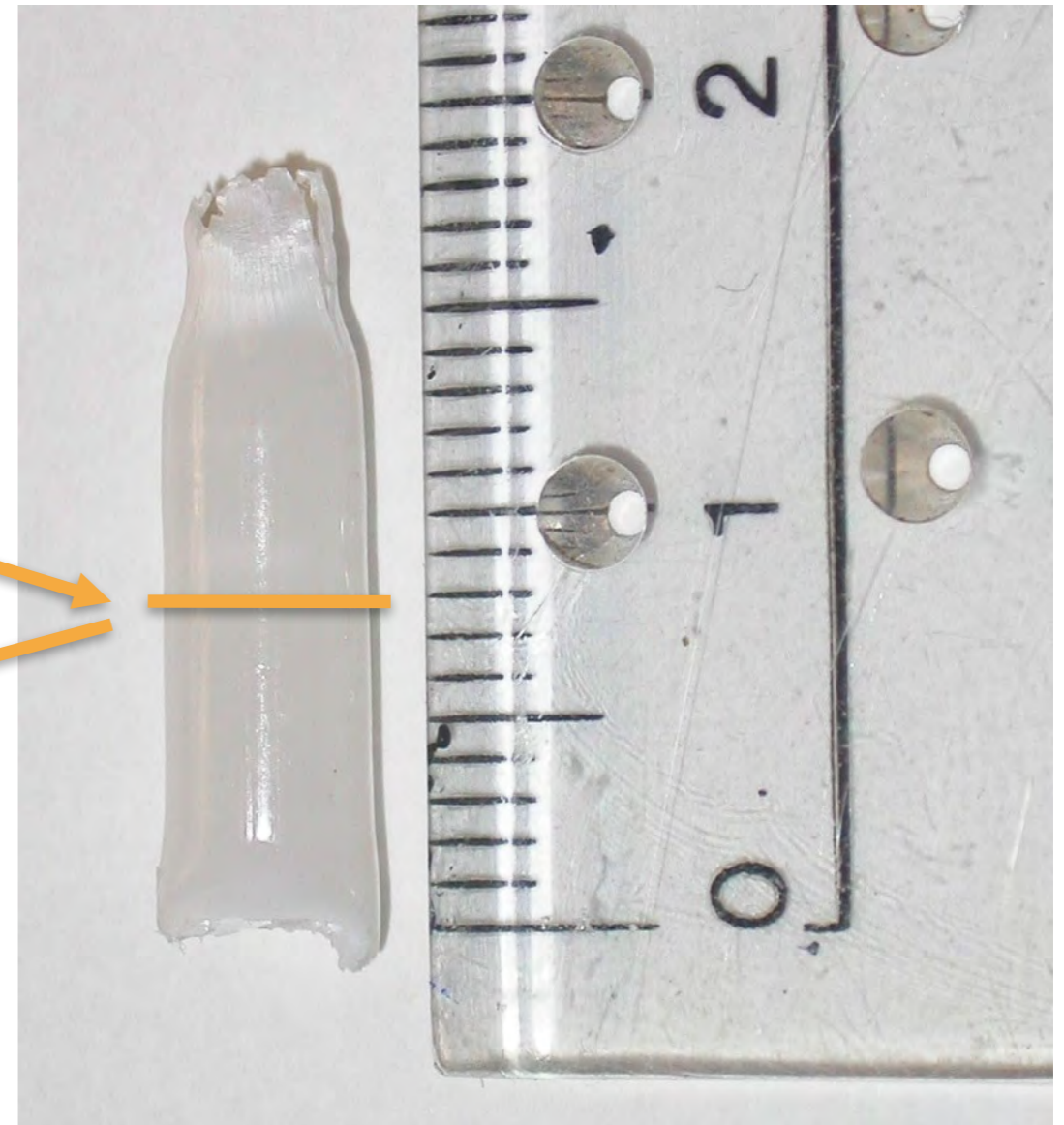
Investigating the local nanostructure on a virtual section through sample

Non-destructive investigation
of inner structure of sample

virtual section

reconstructed SAXS
cross section at each point
on the virtual section

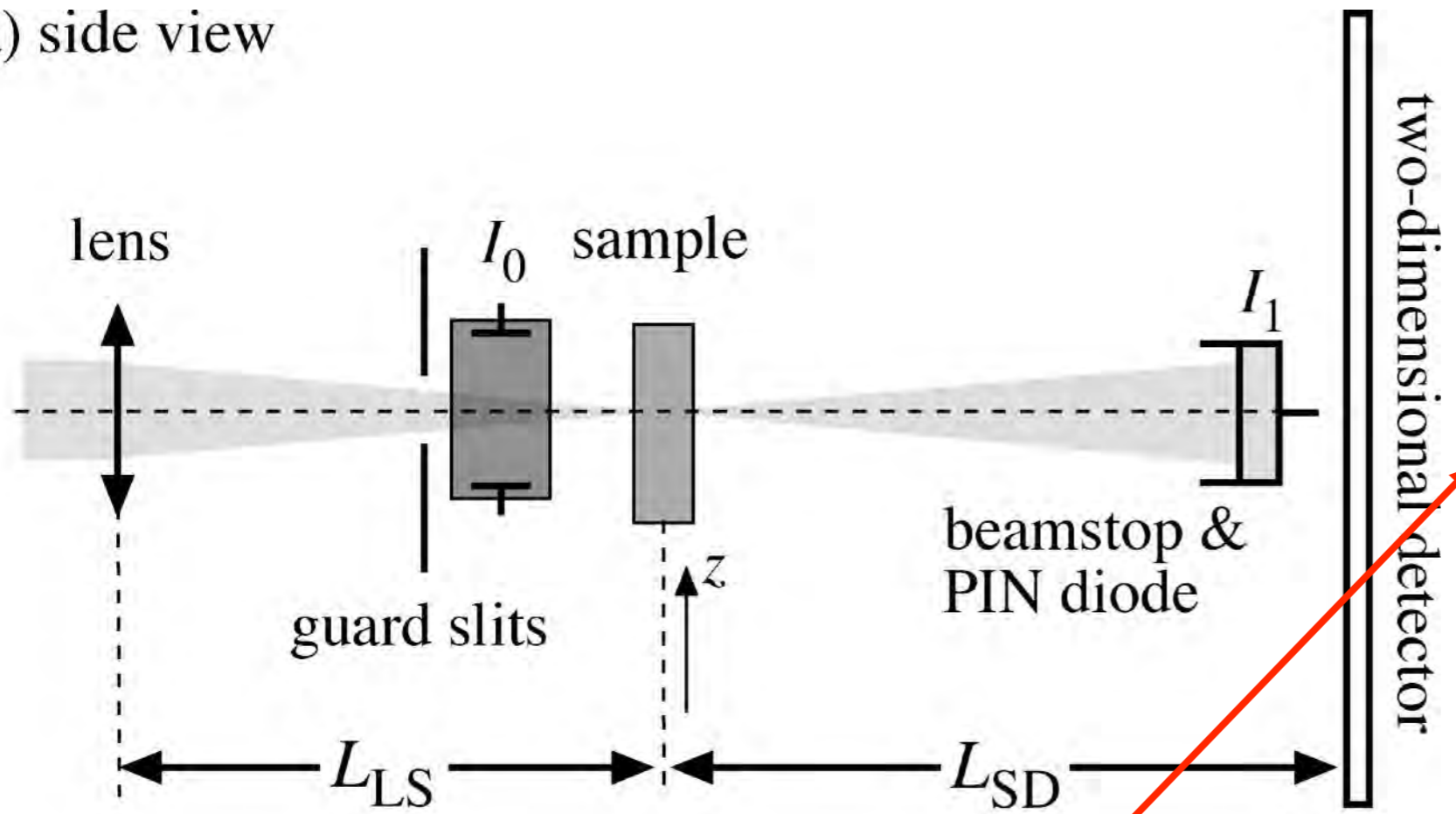
Sample:



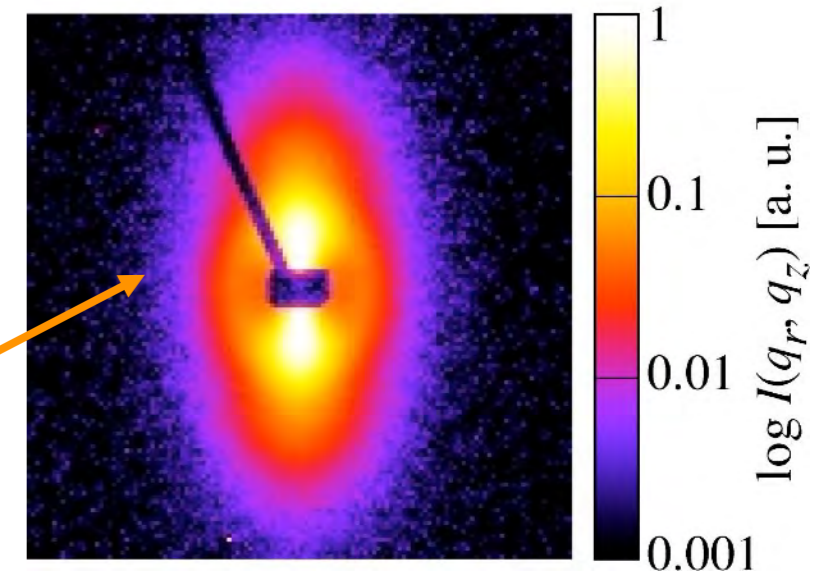
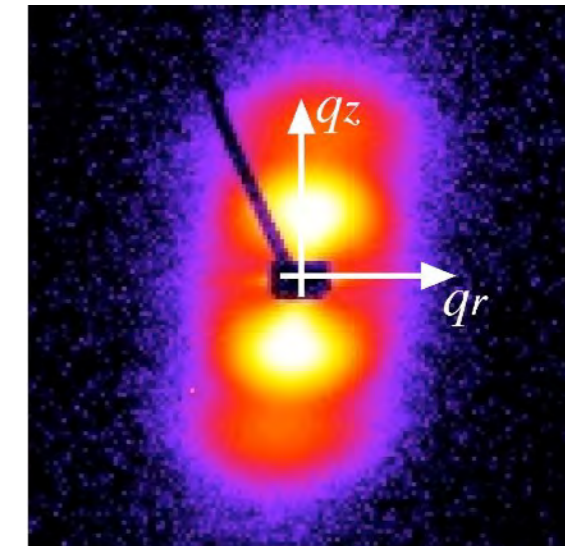
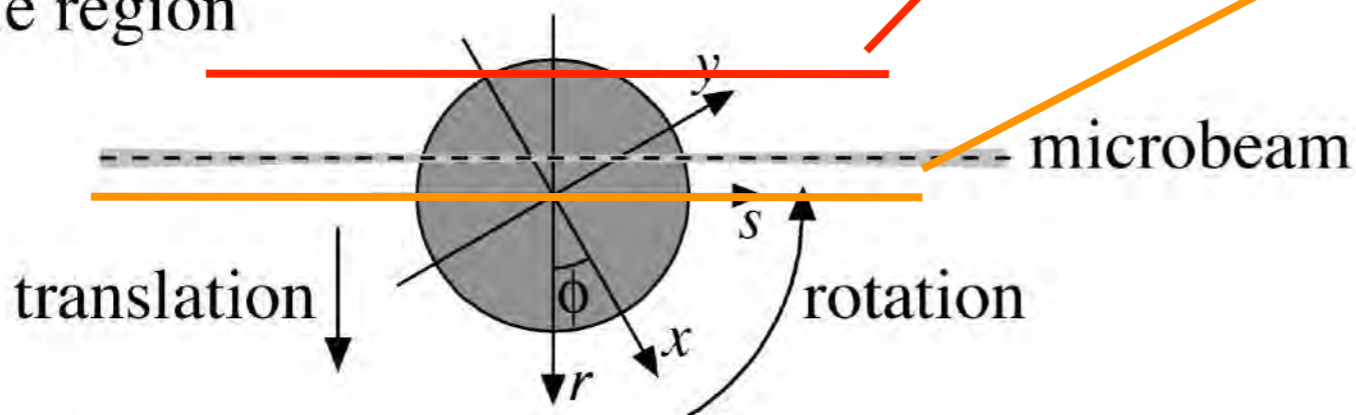
polyethylene rod

Tomographic Small-Angle X-Ray Scattering

(a) side view

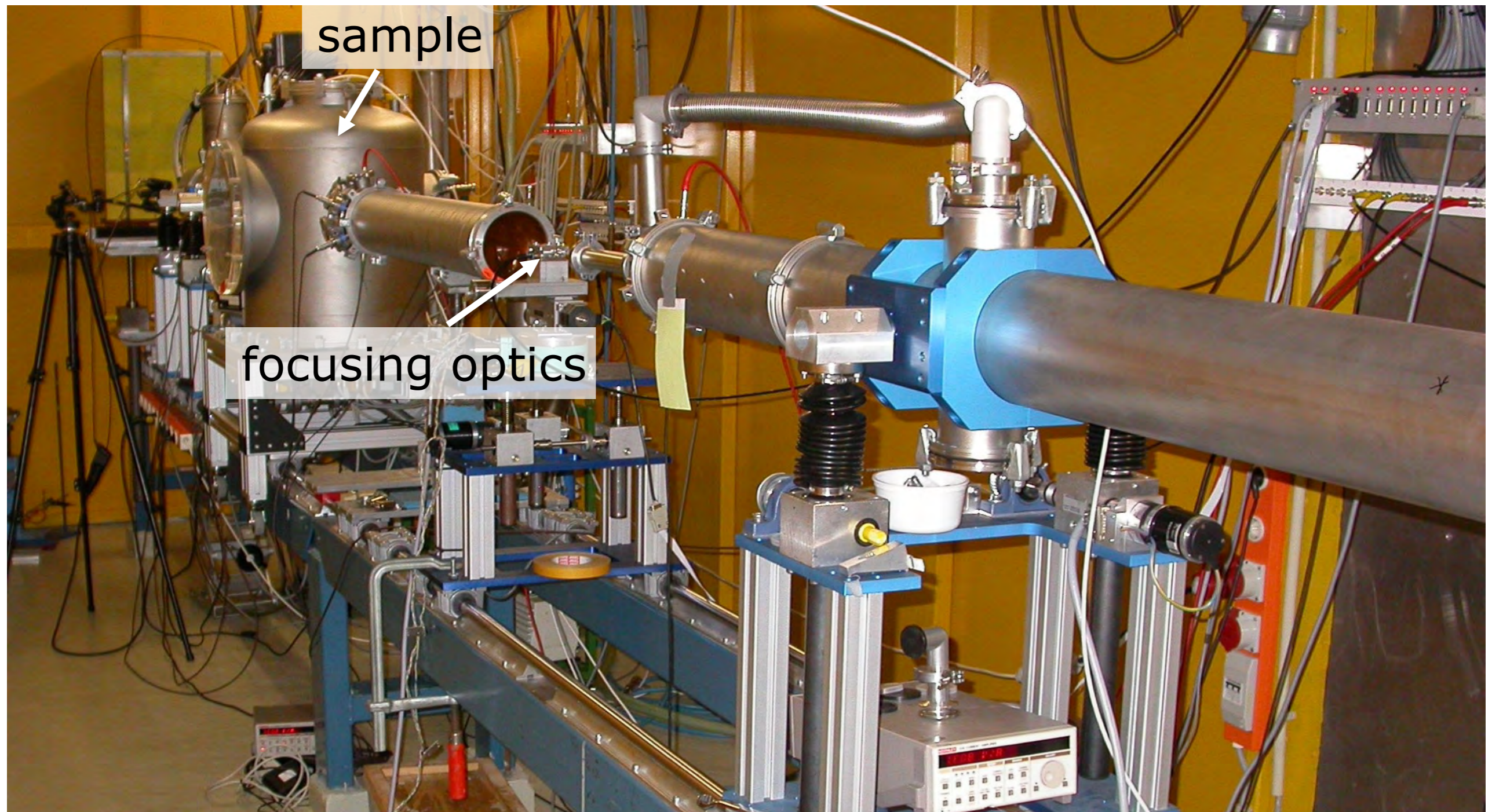


(b) top view of sample region

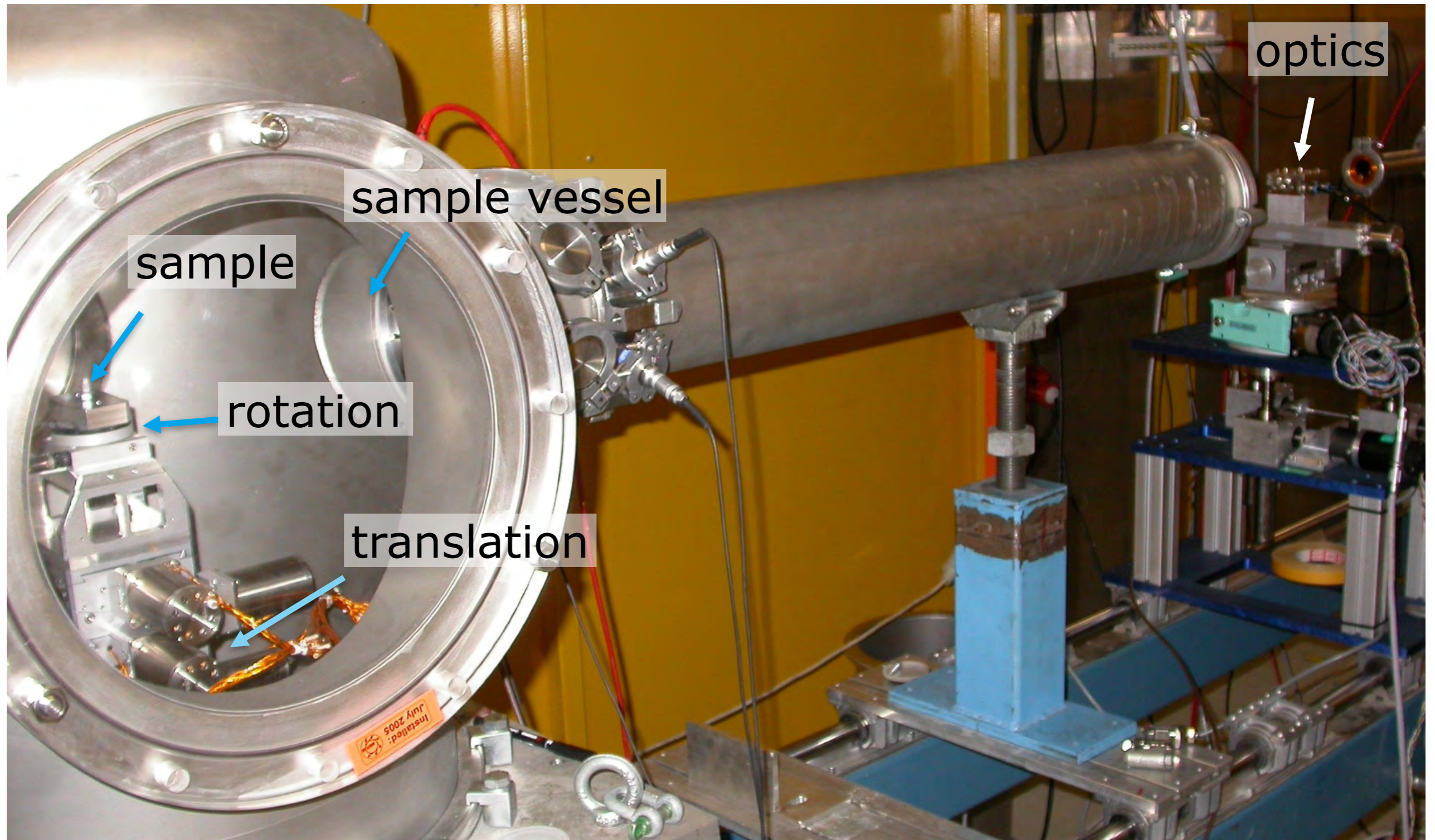


101 projection with
70 steps of
80 μm step size

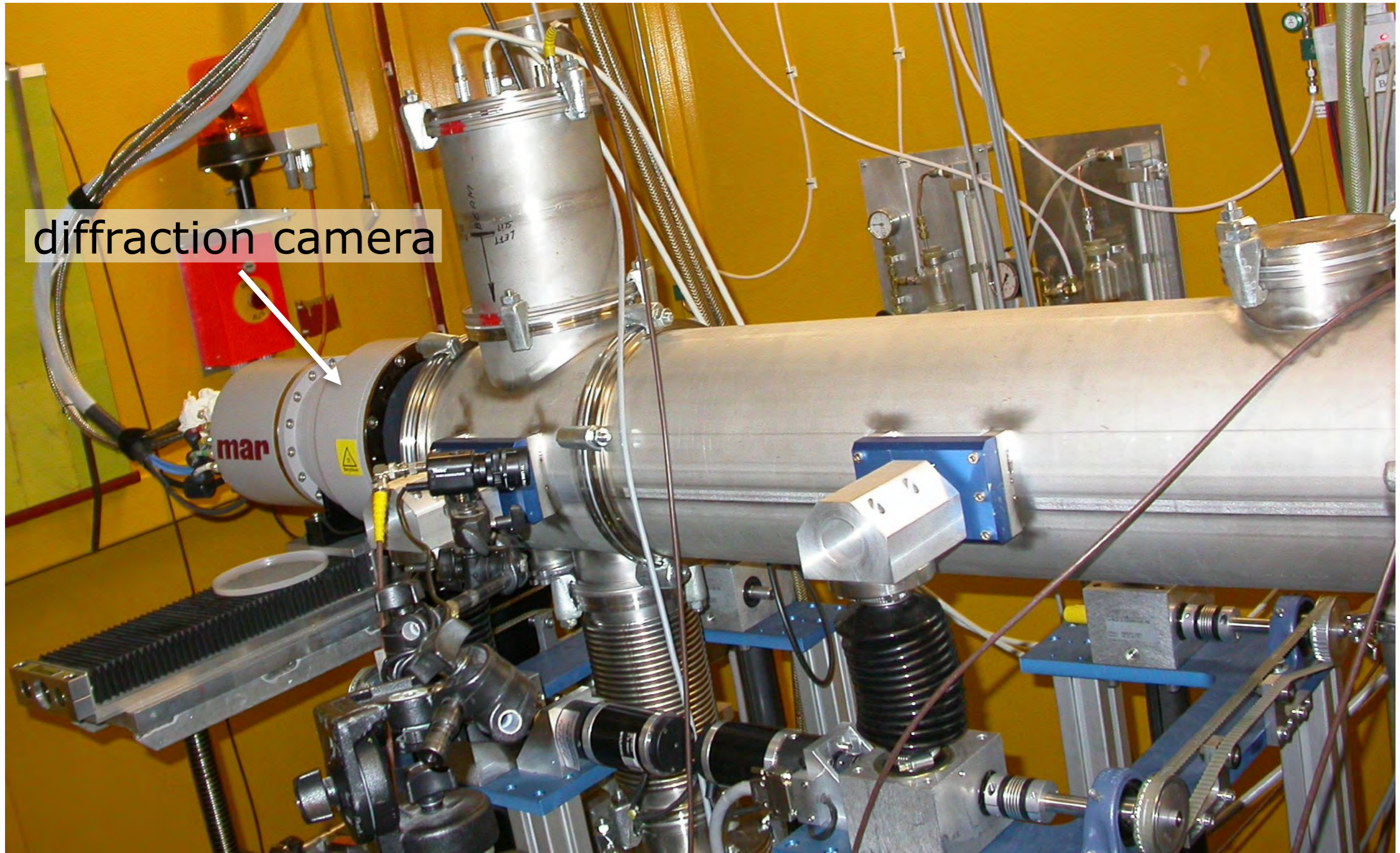
SAXS Tomography at Beamline BW4 at DORIS III



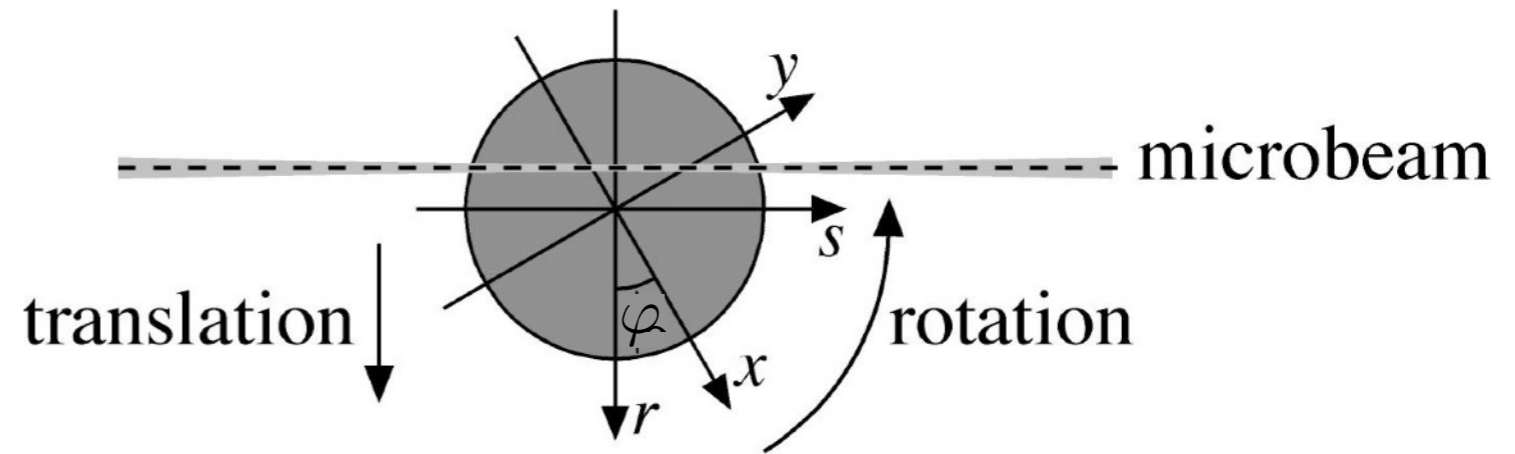
SAXS Tomography at Beamline BW4 at DORIS III



SAXS Tomography at Beamline BW4 at DORIS III



SAXS Tomography



Transmitted beam:

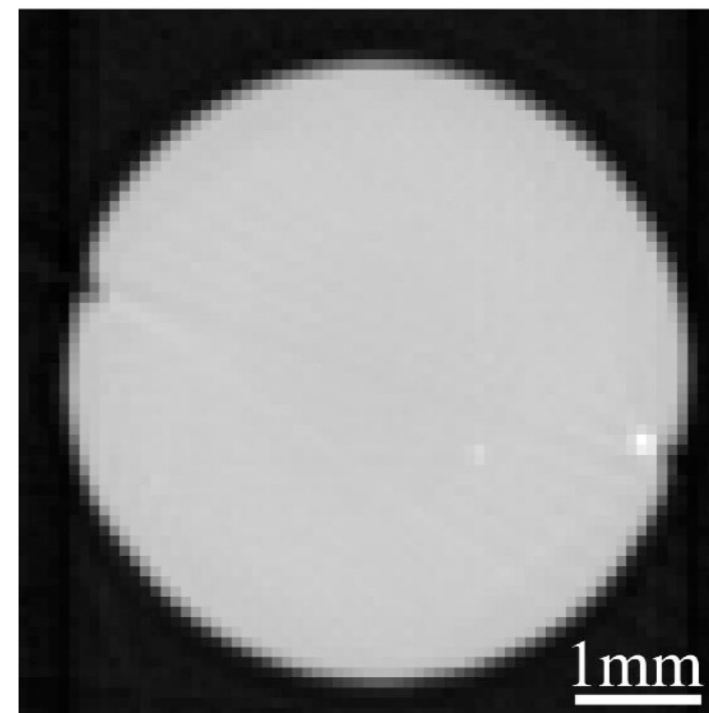
$$I_1(r, \varphi) = I_0 \exp \left\{ - \int ds' \mu [x(s', r), y(s', r)] \right\}$$

Standard tomography:

homogeneous density (polyethylene):

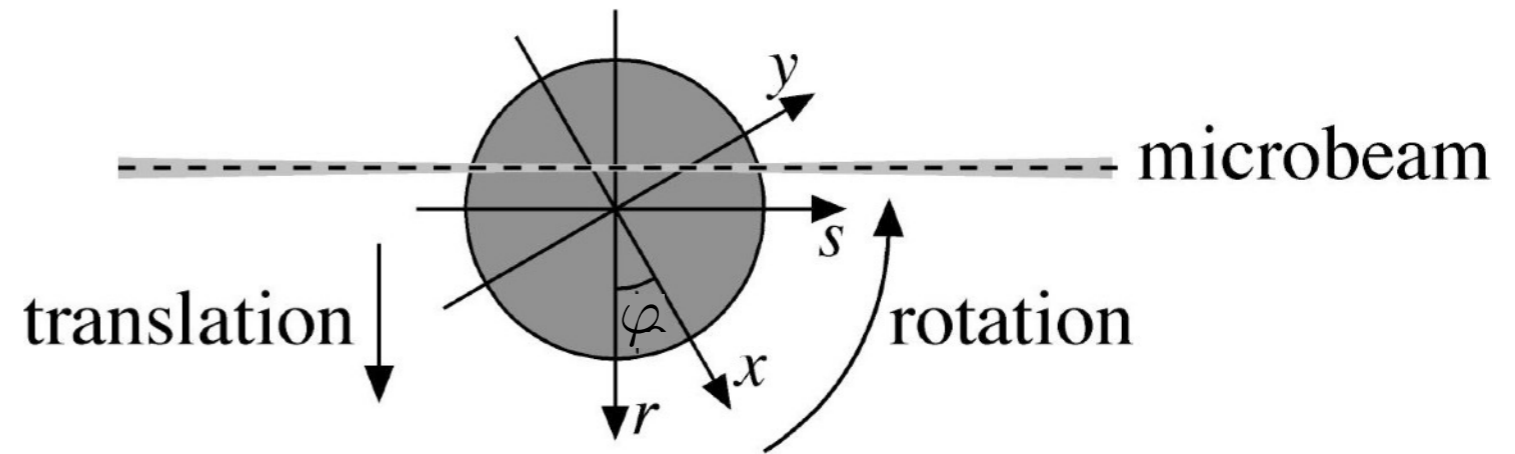
$$\rho = [0.88 \pm 0.04] \text{g/cm}^3$$

attenuation



C. Schroer, et al., Appl. Phys. Lett. **88**, 164102 (2006)

SAXS Tomography



scattered signal:

$$I_{\vec{q}}(r, \varphi) = I_0 \int ds f(\varphi, s, r) p_{\vec{q}, \varphi}(x, y) g(\varphi, s, r)$$

attenuation of primary beam:

$$f(\varphi, s, r) = \exp \left\{ - \int_{-\infty}^s ds' \mu(x, y) \right\}$$

attenuation of scattered beam

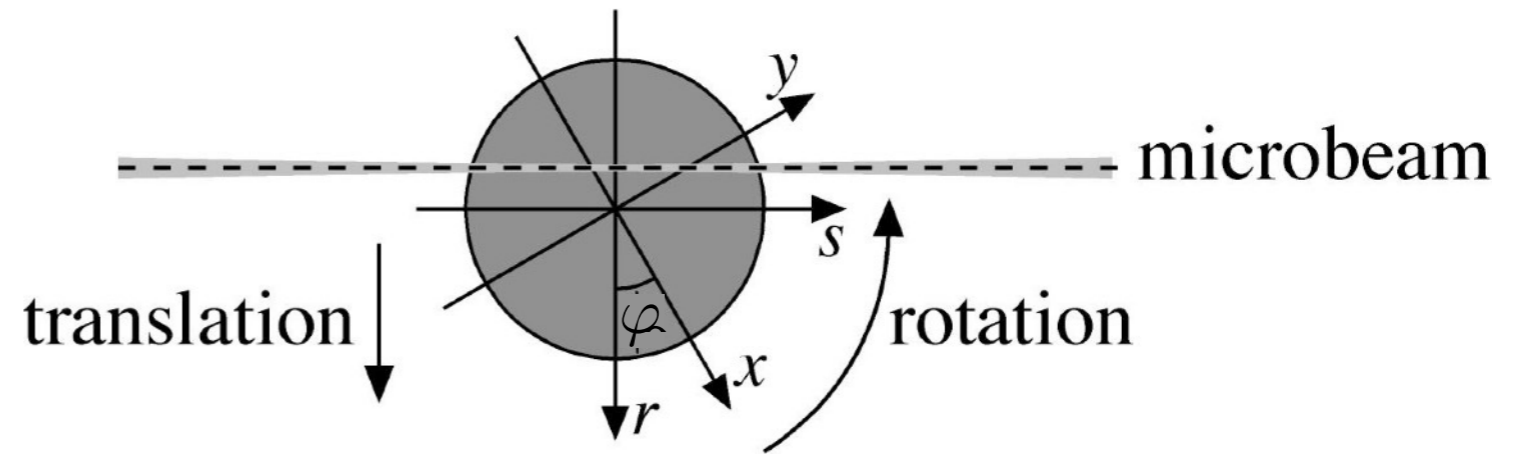
$$g(\varphi, s, r) = \exp \left\{ - \int_s^{\infty} ds' \mu(x, y) \right\}$$

Diffraction signal in forward direction:

$$I_1(r, \varphi) = I_0(r, \varphi) \cdot f(\varphi, s, r) \cdot g(\varphi, s, r) \quad \text{independent of } s$$

C. Schroer, et al., Appl. Phys. Lett. **88**, 164102 (2006)

SAXS Tomography



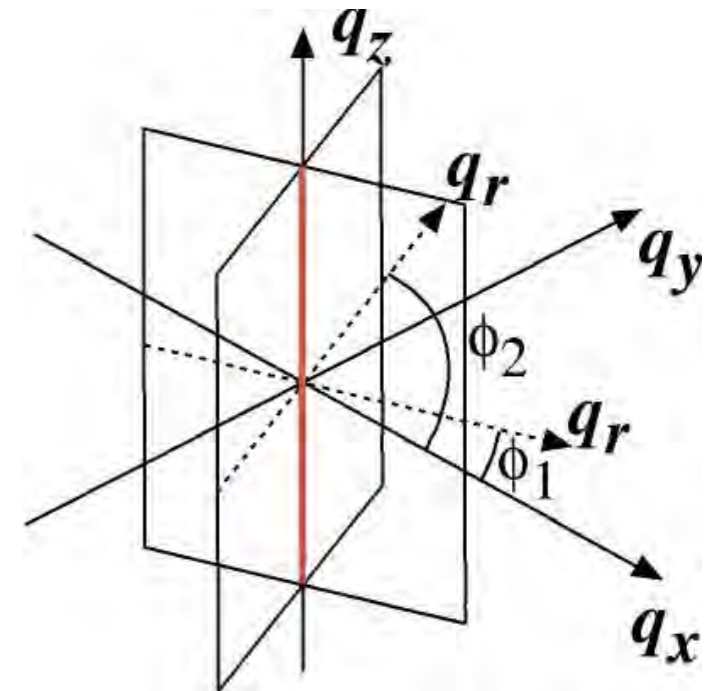
scattered signal:

$$I_{\vec{q}}(r, \varphi) = I_1 \int ds p_{\vec{q}, \varphi}(x, y)$$

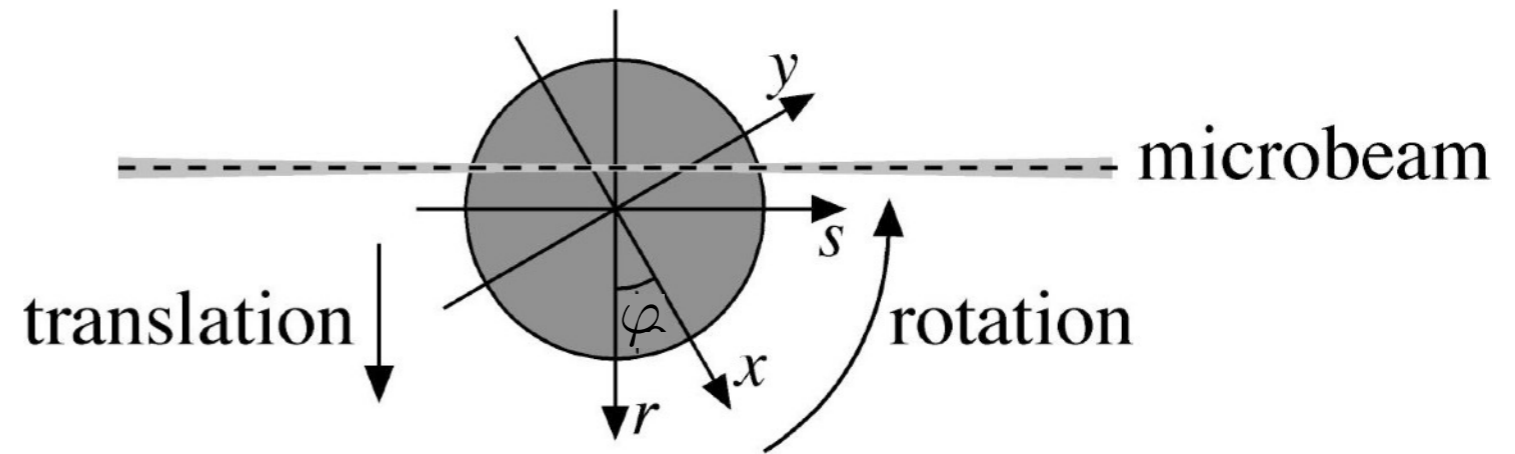
tomography works only if $p_{\vec{q}, \varphi}(x, y)$ is independent φ

general case: $p_{\vec{q}, \varphi}(x, y)$ complicated function

reconstruction only for $q_r = 0$
(q along rotation axis)



SAXS Tomography



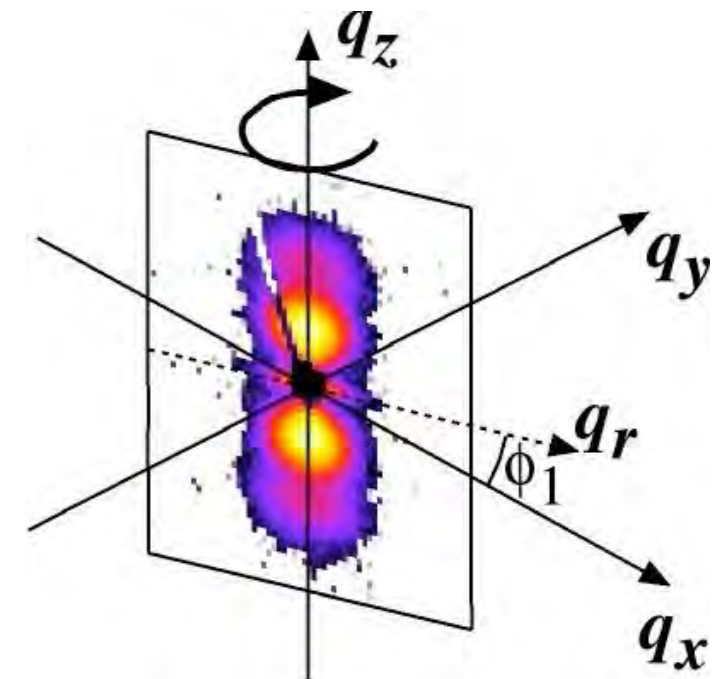
scattered signal:

$$I_{\vec{q}}(r, \varphi) = I_1 \int ds p_{\vec{q}, \varphi}(x, y)$$

tomography works only if $p_{\vec{q}, \varphi}(x, y)$ is independent φ

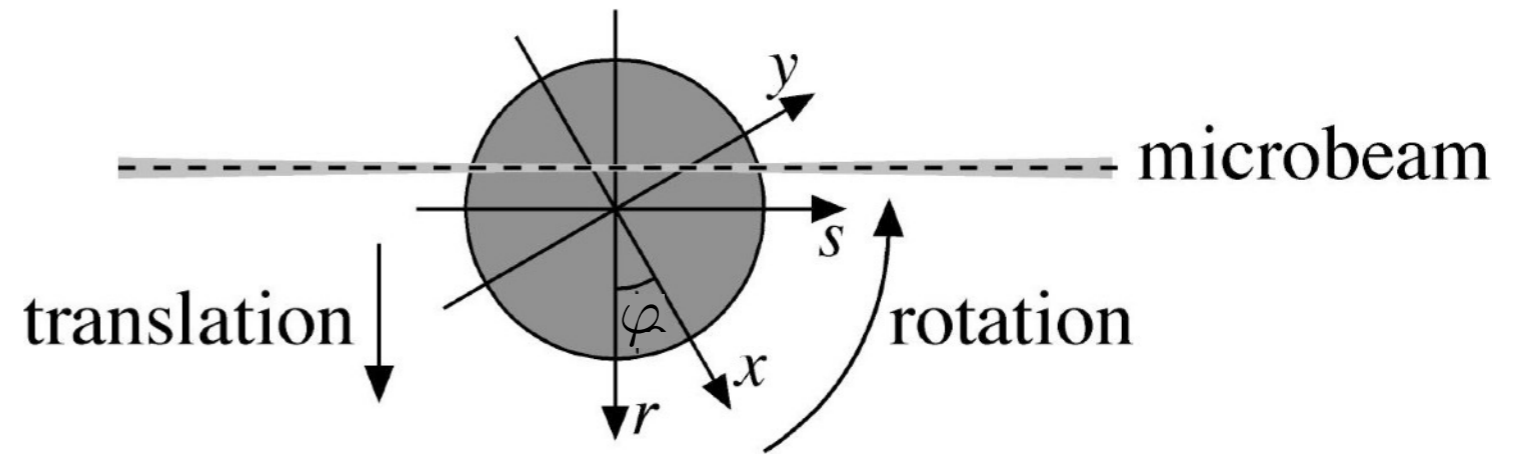
Special case: $p_{\vec{q}, \varphi}(x, y)$ has rotation symmetry
around rotation axis

reconstruction of full SAXS cross section
in the vicinity of $q = 0$

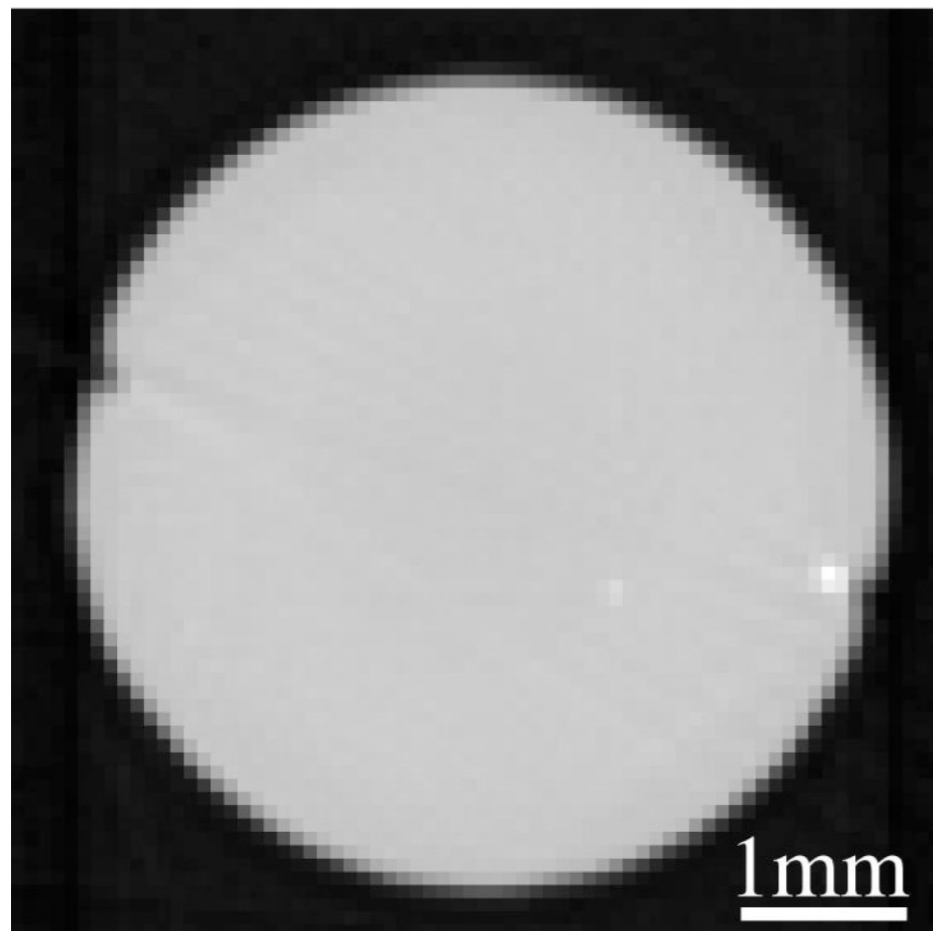


SAXS Tomography

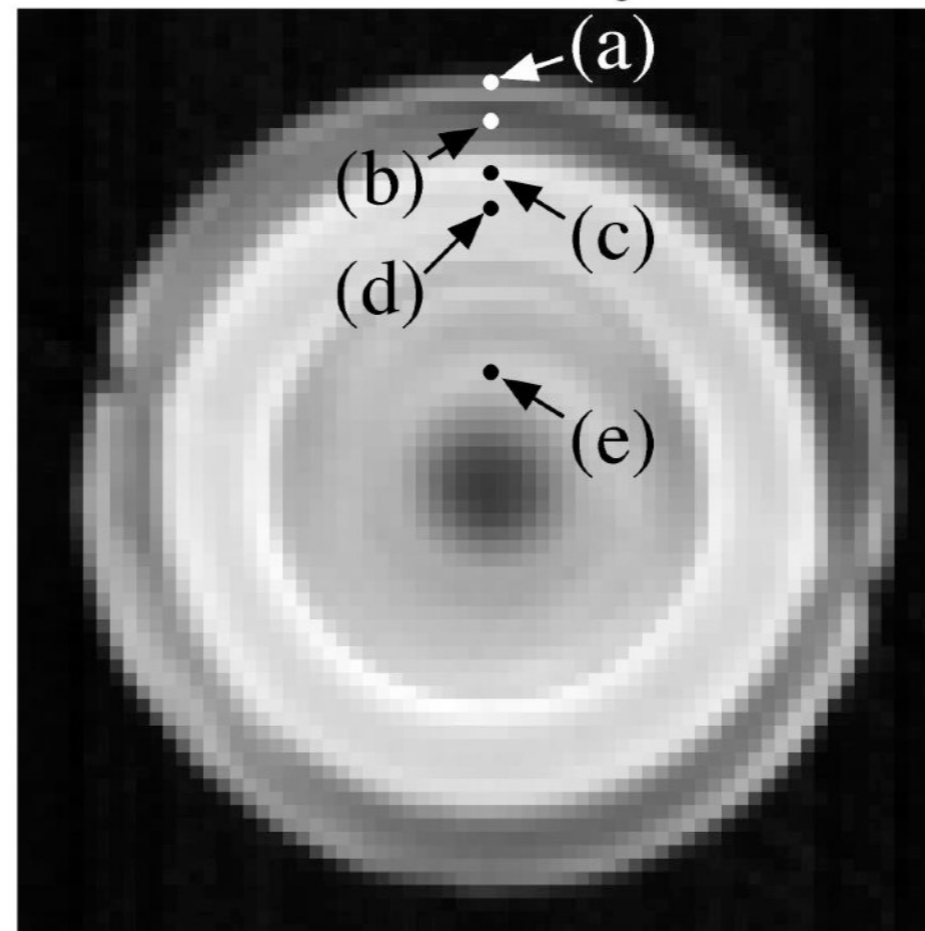
reconstruction:



attenuation



scattered intensity

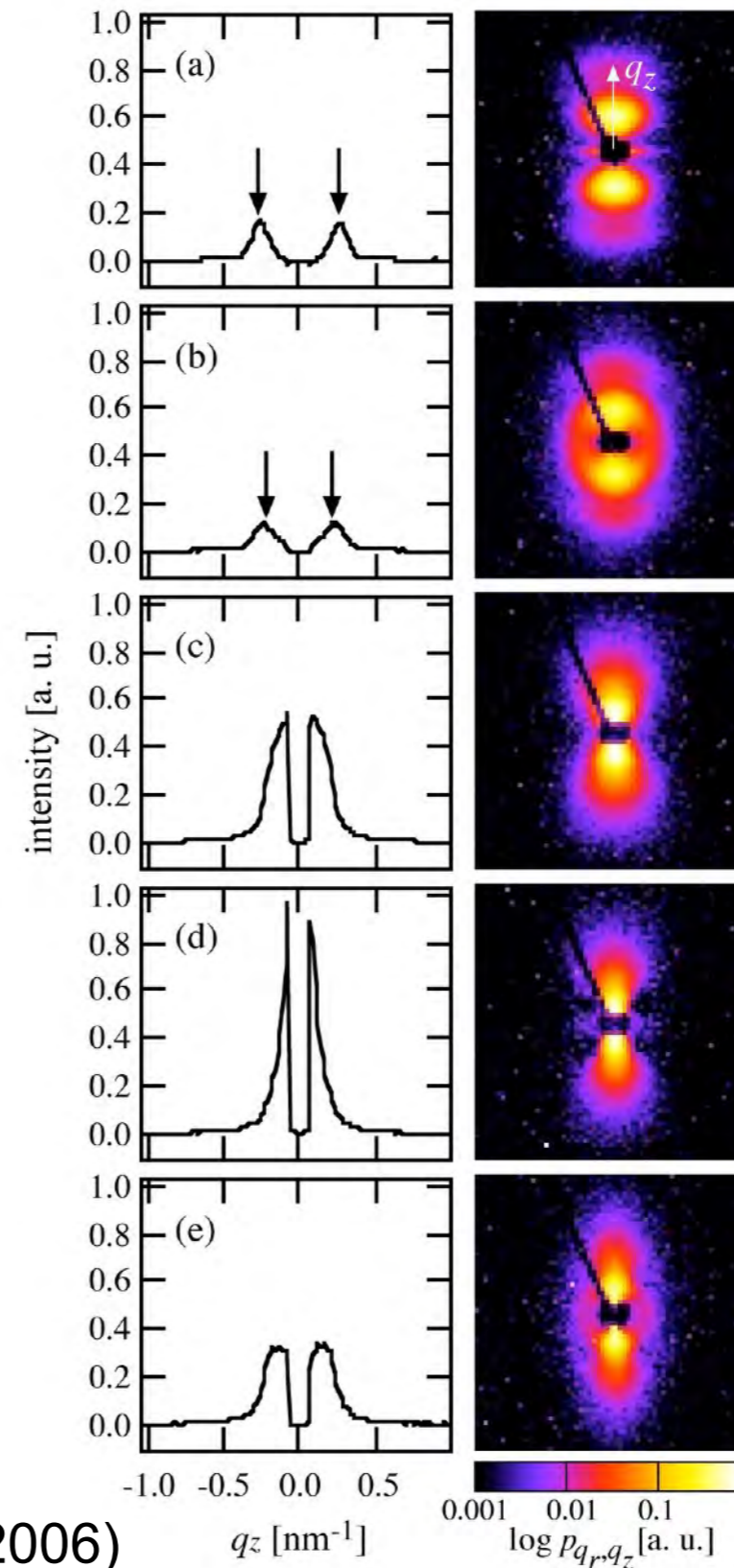
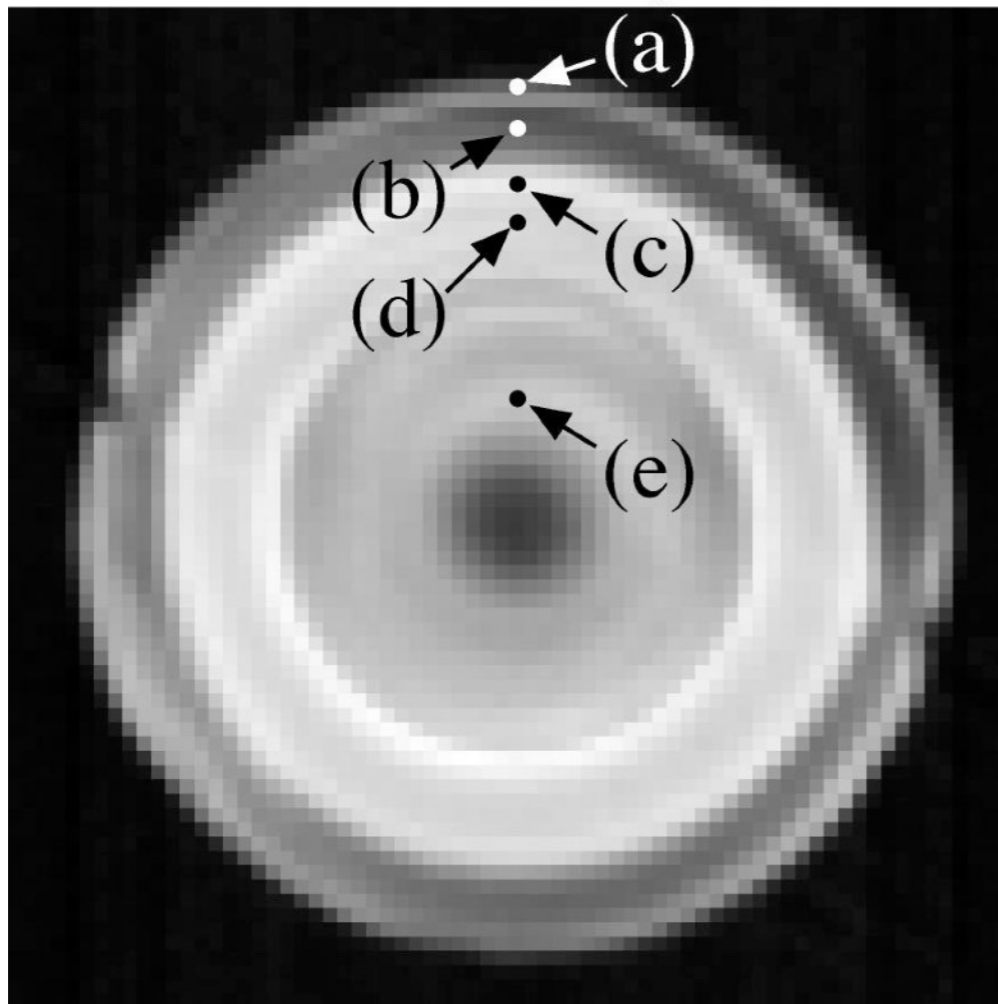


integral scattering cross section along rotation axis

SAXS Tomography

Sample with fibre texture:

scattered intensity



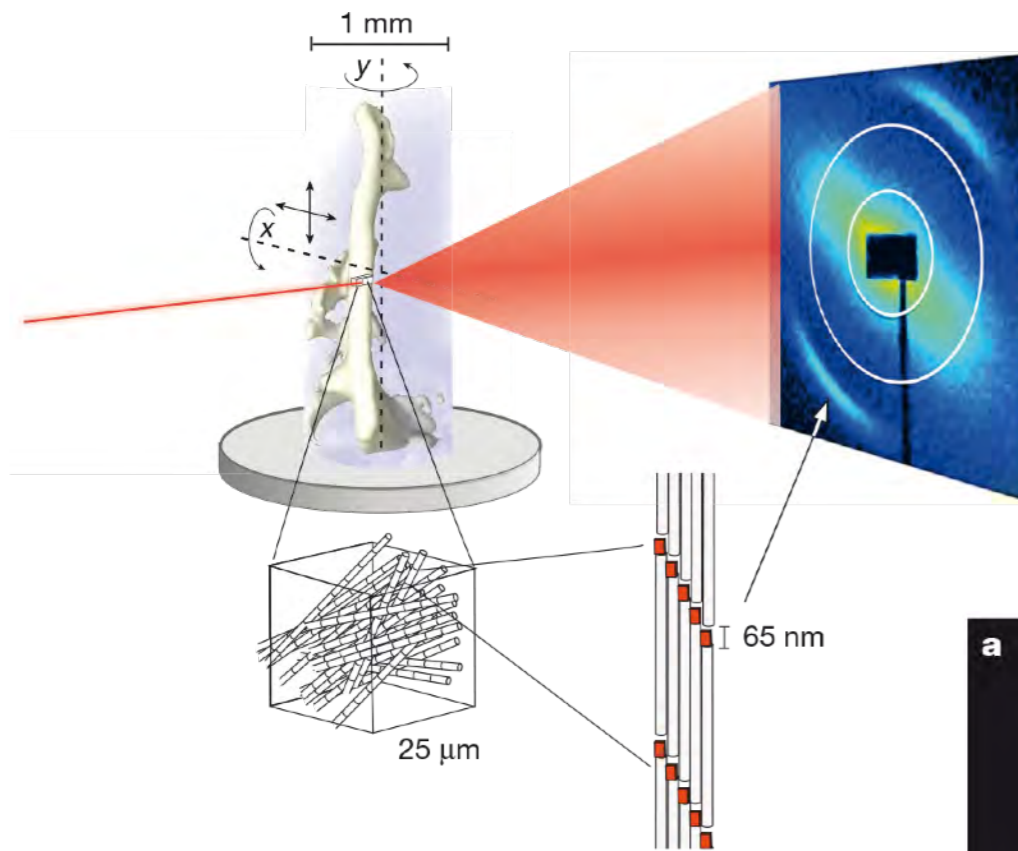
inhomogeneous nanostructure

scattering cross section in each pixel (rotation symmetry)!

C. Schroer, et al., Appl. Phys. Lett. **88**, 164102 (2006)



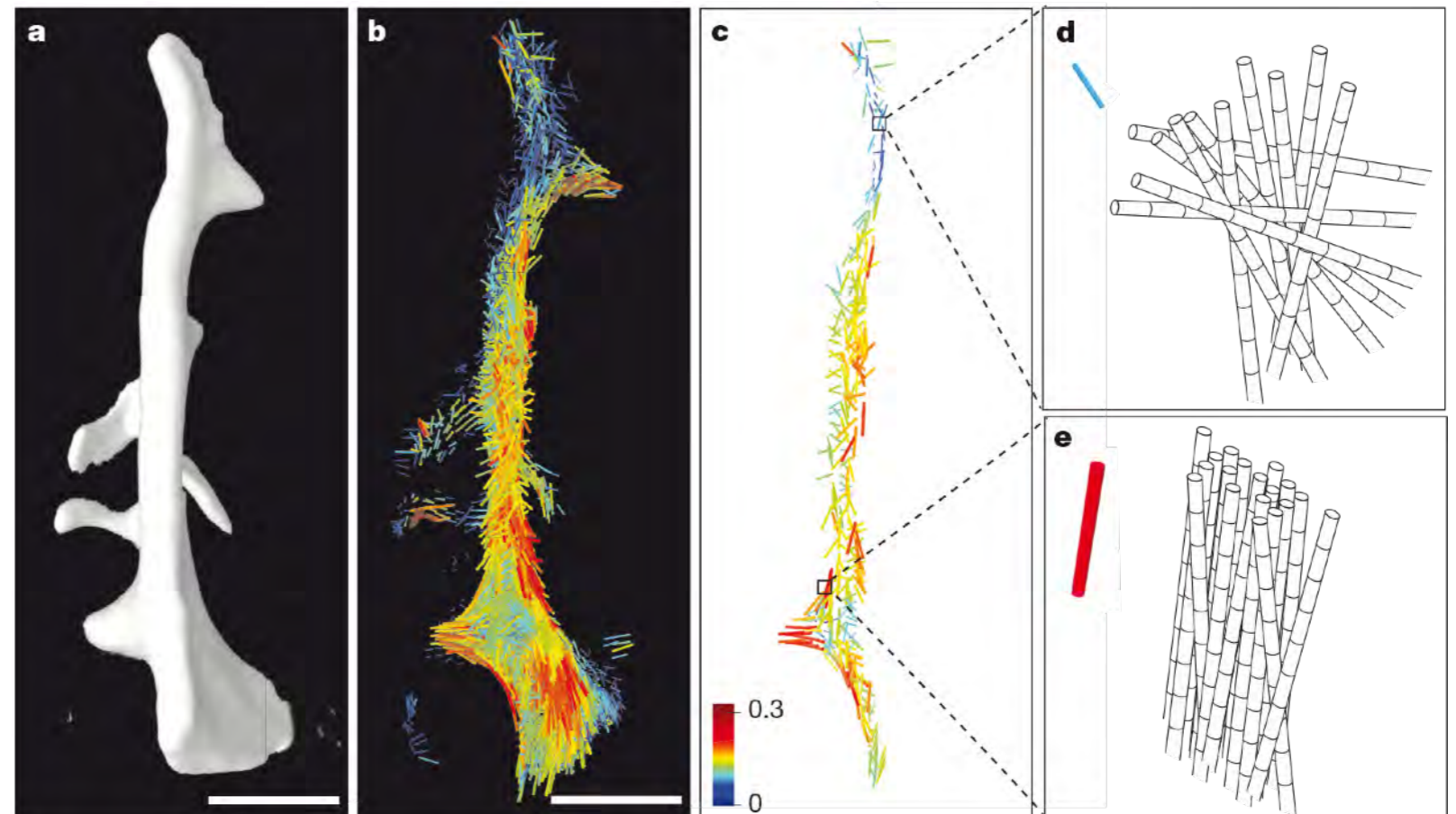
SAXS Tomography in 3D



general SAXS-tomographic problem

in general: measure 6 dimensional information!
Scan in 4 dimensions and record 2D patterns
(coarse mesh due to time limitations)

Liebi, M., et al.,
Nature, **527**(7578),
349–352. (2015).



Conventional X-Ray Microscopy

X-ray microscopy as a quantitative local measurement:

> Full-field microscopy: attenuation and phase contrast

→ measure complex refractive index of sample

> scanning microscopy:

→ all x-ray analytical techniques can be used as contrast:

> x-ray fluorescence (XRF): chemical composition (quantitative analysis)

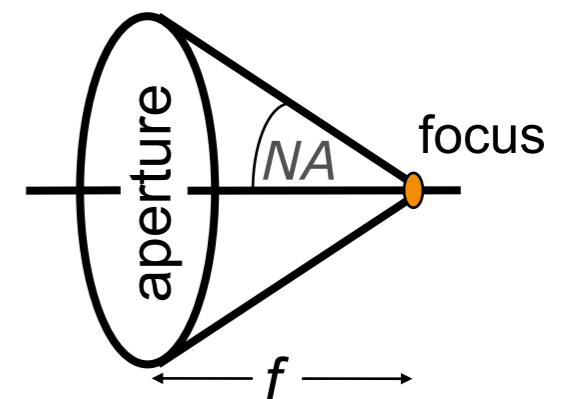
> x-ray absorption spectroscopy (XAS): chemical state of given element (e. g. oxidation)

> x-ray diffraction and scattering (SAXS & WAXS): local nanostructure

> ...

Full-field and scanning microscopy require x-ray optics

→ resolution limited by numerical aperture of optics



Next time: what are the limits and how can we overcome them?