Synchrotron X-ray imaging

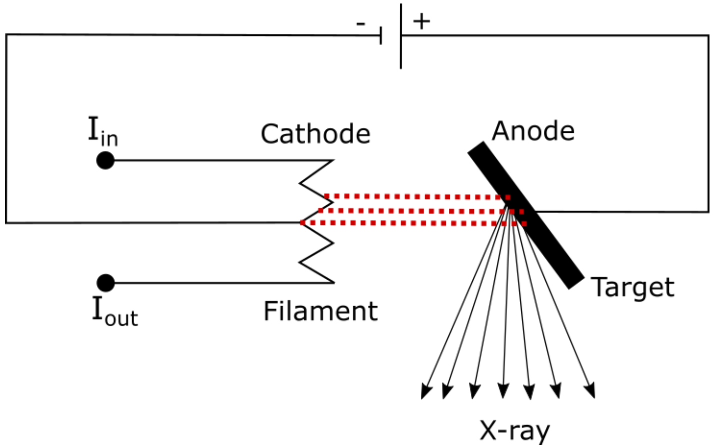
IRT Doctoral school – 09.04.2024

Jaianth Vijayakumar

Beamline scientist – BM18 and BM05

X-ray source

Lab-based source



Filament – W
Target – W, Mo or heavy metals

Synchrotron source

Magnets and IDs | SESAME | Synchrotron-light for Experimental Science and Applications in the Middle East

U100 - Helmholtz-Zentrum Dresden-Rossendorf, HZDR

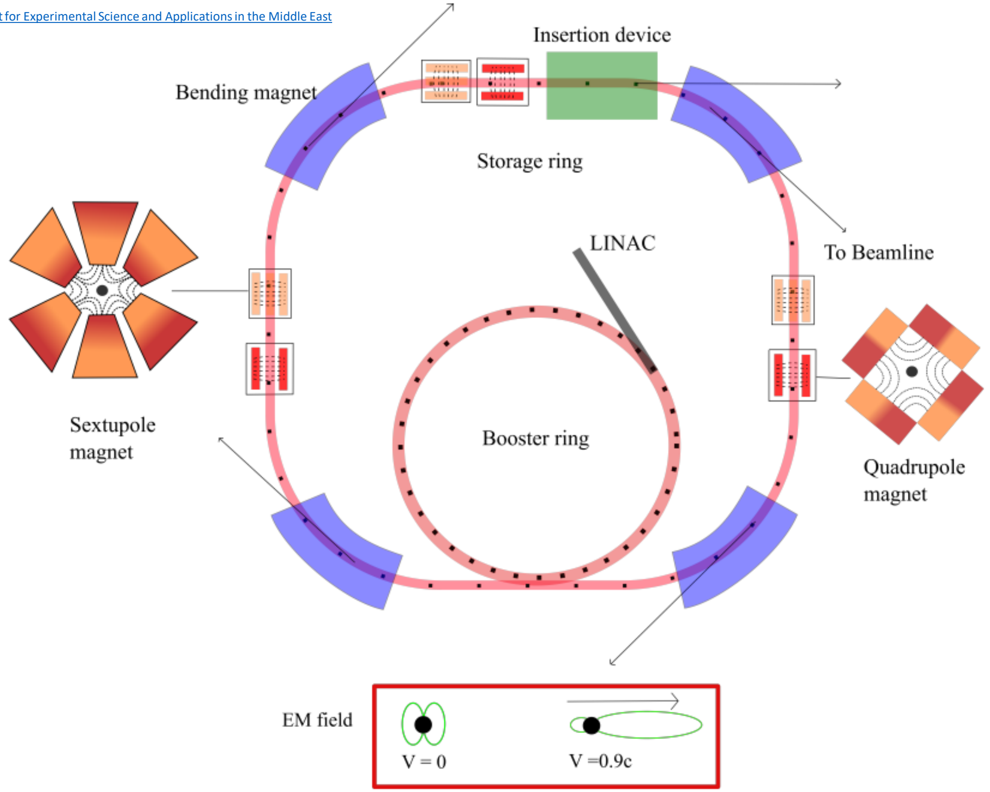
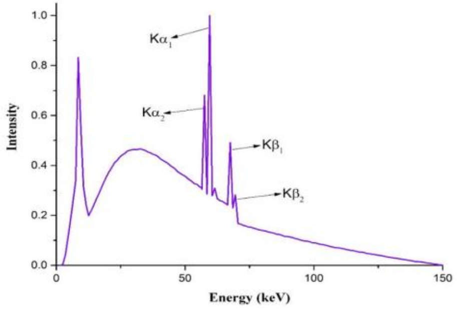


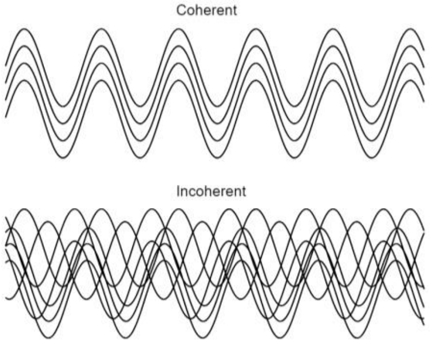
Figure 3.7: Schematic representation of a synchrotron facility.

https://edoc.unibas.ch/70259/1/All_Chapters.pdf

X-ray source – benefit of a synchrotron

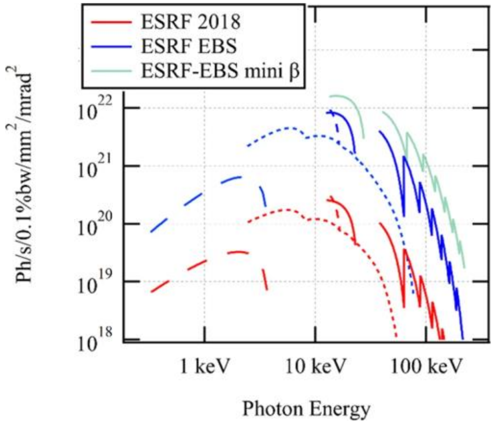
Lab-based source

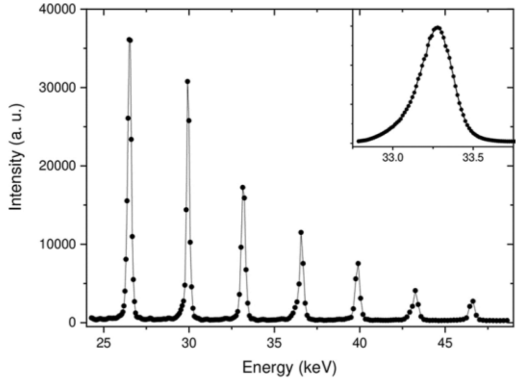




1. Polychromatic
2. Conical beam – large divergence
3. Non-Coherent
4. Low intensity
5. Suitable for only absorption based imaging – e.g. tomography

Synchrotron source





1. Polychromatic/ Monochromatic
2. Parallel beam – less divergence
3. Highly Coherent
4. High intensity – EBS !!
5. Possible to do many imaging modalities ☺

A. Basahel et al., Mathematics 9(11), 1227 (2021)

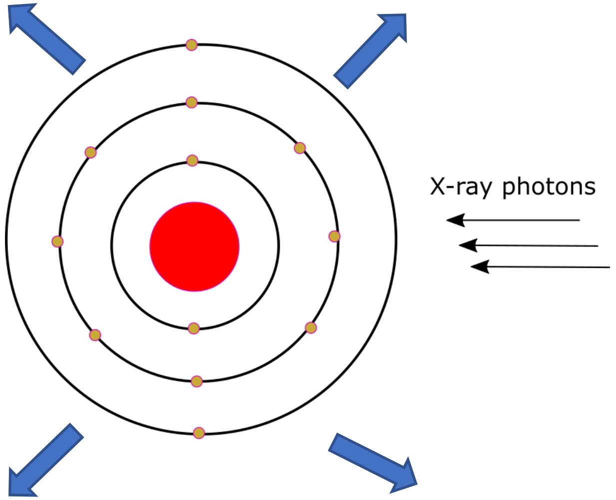
P. Raimondi et al., Communication Physics 6 (82), (2023)

X-ray interaction with matter

Microscopic origin

Elastic scattering

Inelastic scattering

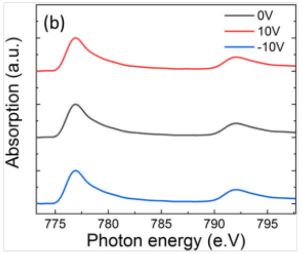


Emission of secondary electron/photoelectric effect

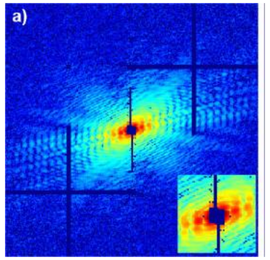
Pair production – electron/positron/nuclear absorption

Macroscopic effect

X-ray absorption (Chemical)

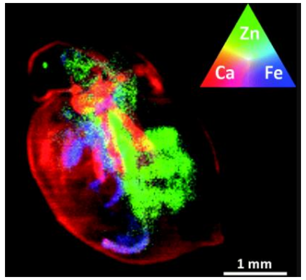


X-ray diffraction (structural)



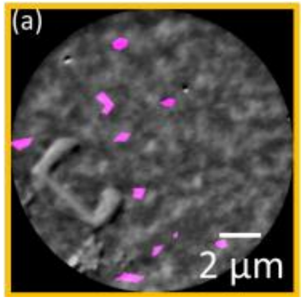
[3D imaging of whole cells using cryocooled coherent X-ray diffraction (esrf.fr)]

Fluorescence (chemical)

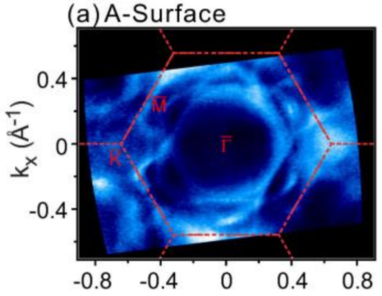


Bojrn et al., JAAS, **34**, 2083-2093 (2019)

Photoemission (Electronic)



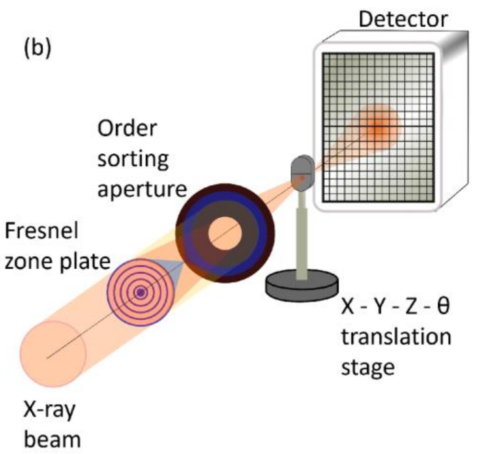
X-ray scattering (form factor, refractive index)



W. Jiang et al., J. Appl. Phys. 128, 135103 (2020)

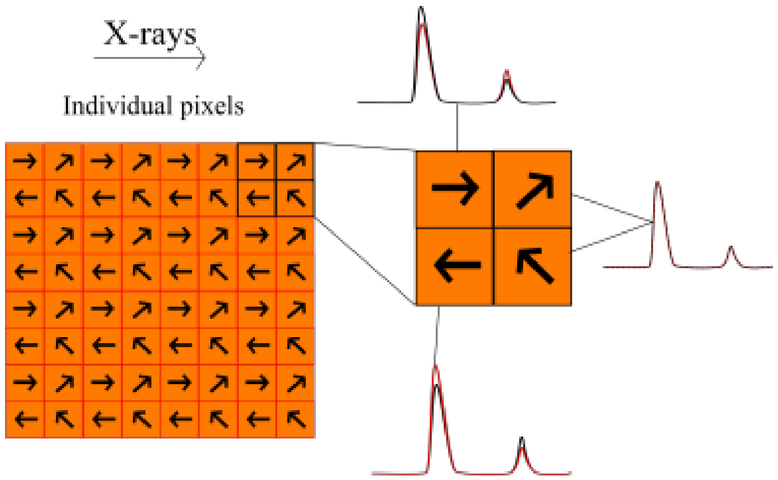
X-ray detector

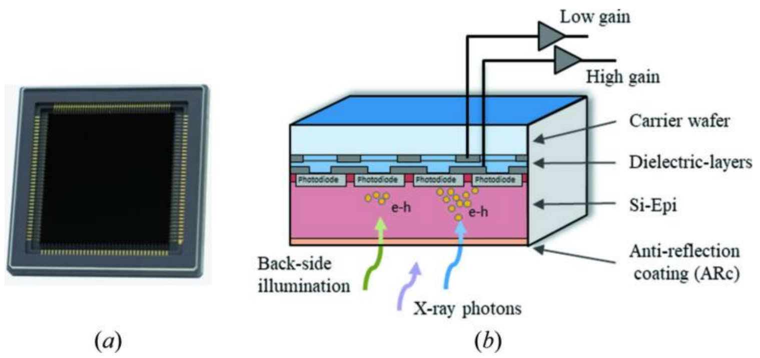
Goal is X-ray imaging is to spatially resolve the X-ray interaction



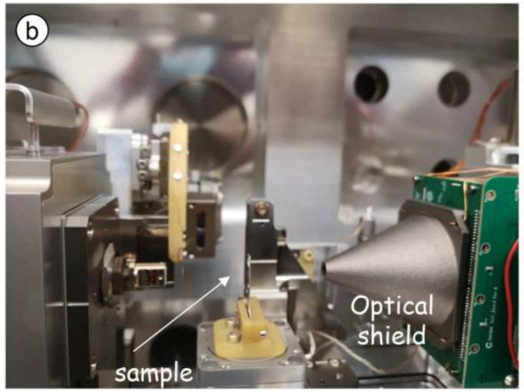
2D array detector

Each pixel is a spectrum,
diffraction/scatting information





CMOS/CCD

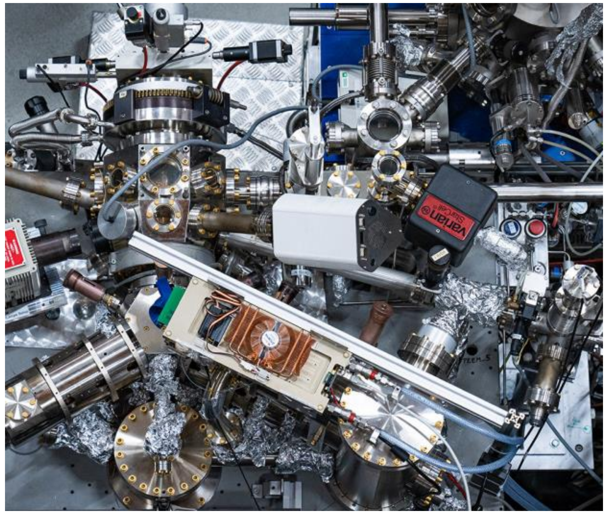


Point detector

Other detectors

1. Scintillator + microscope detector
2. Diode
3. phosphor screen

Modalities – X-ray photoemission electron microscopy



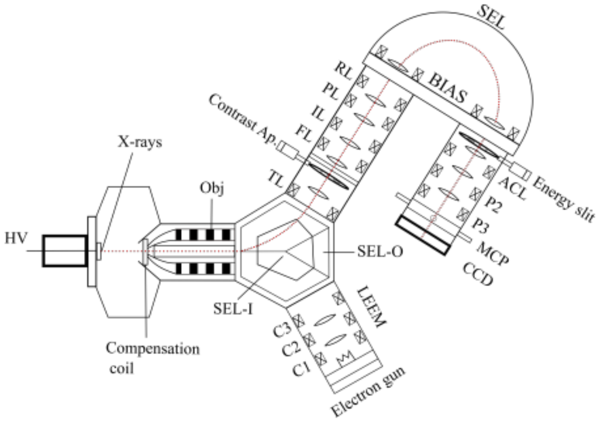
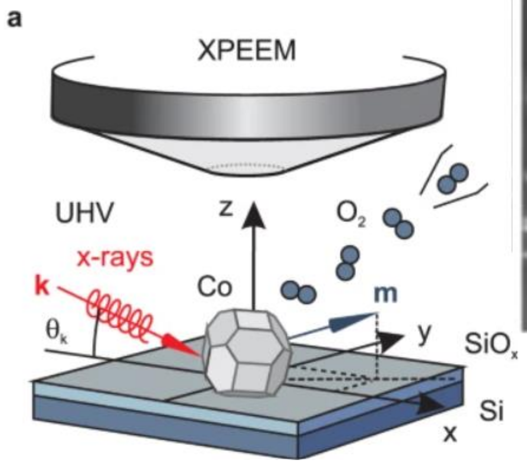
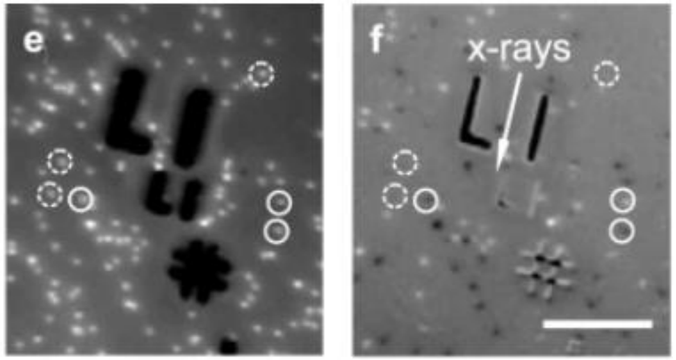


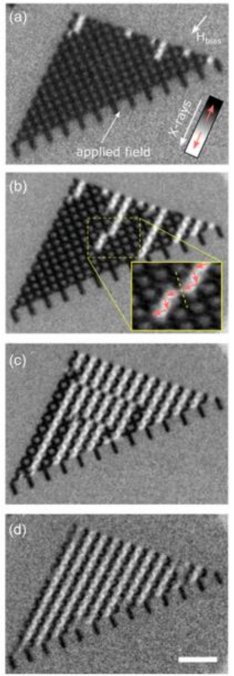
Figure 3.11: ELMITEC XPEEM set-up.

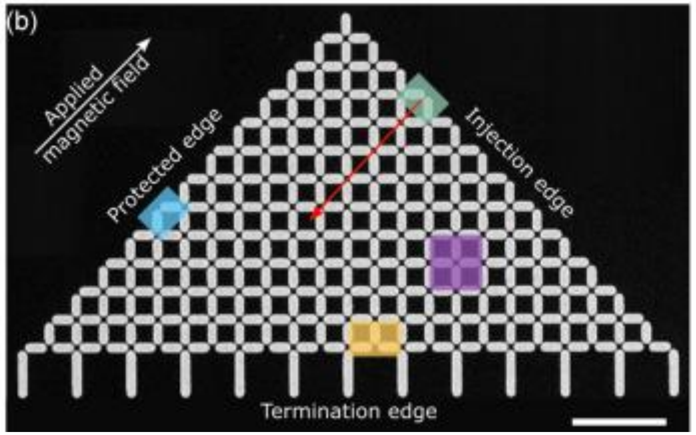
SIM – beamline – SLS, PSI





J. Vijayakumar at al., Nat. Comm. 14, 174 (2023)

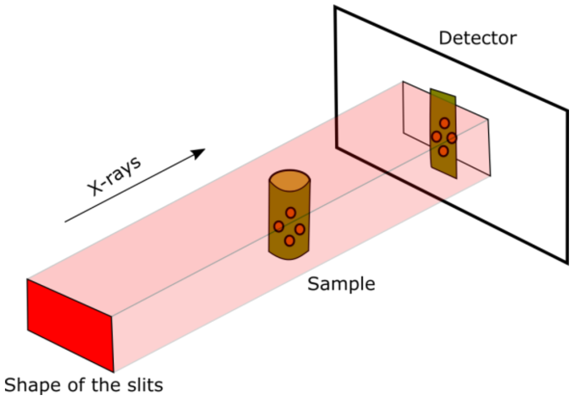


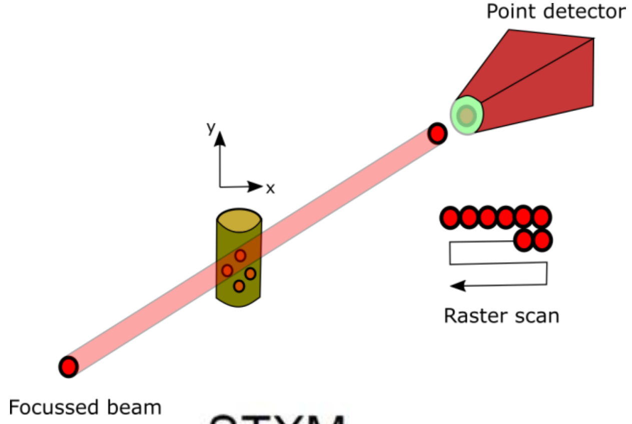


H. Arava et al., PRB, 102, 144413 (2020)

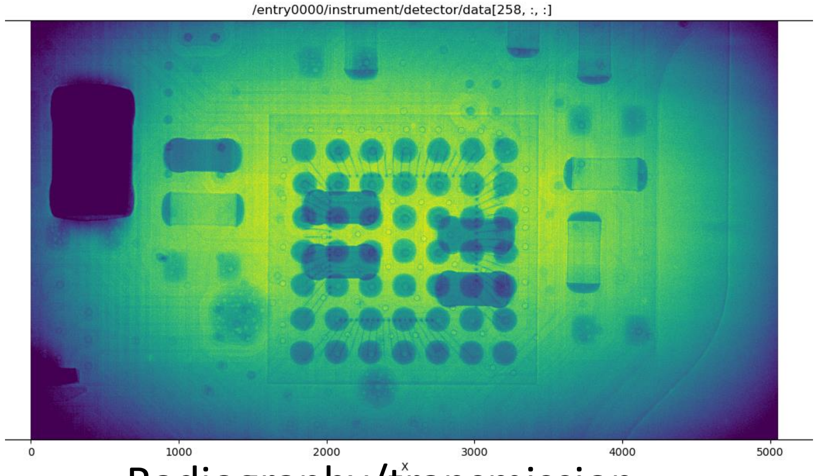
Modalities – X-ray transmission microscopy

Transmission image/ absorption image/ radiography image/ projection image





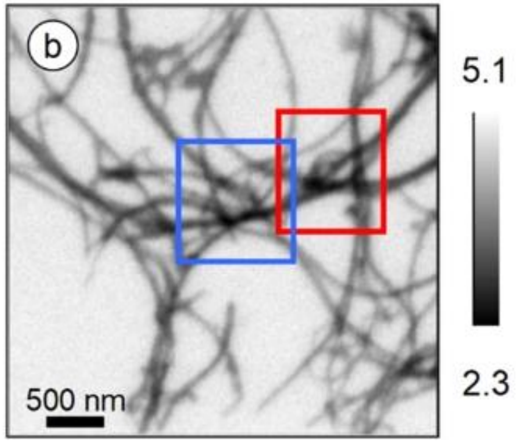
Resolution depends on:
1. Size of the beam (~ 10 nm, default – 60 - 100 nm)
2. Precision of the stage



Radiography/transmission

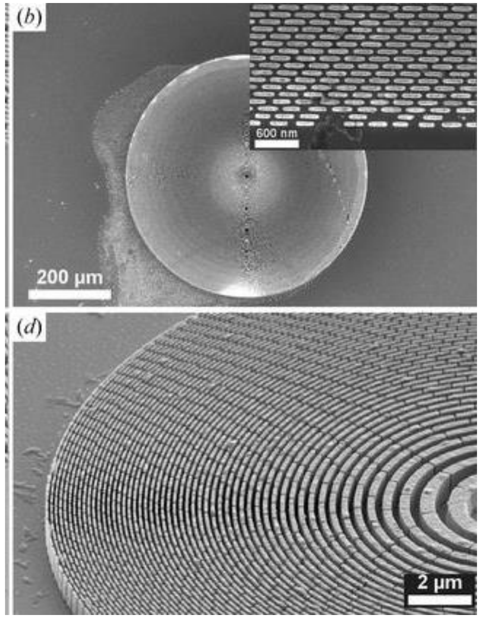
Acquisition time > 50 ms

STXM



Raster scan

Acquisition time >15 min



N. Mille et al., Communications materials, 3,8 (2022)

S. Gorelick et al., JSR,18, 442-446(2011)

ESRF
The European Synchrotron

Modalities – Phase contrast imaging

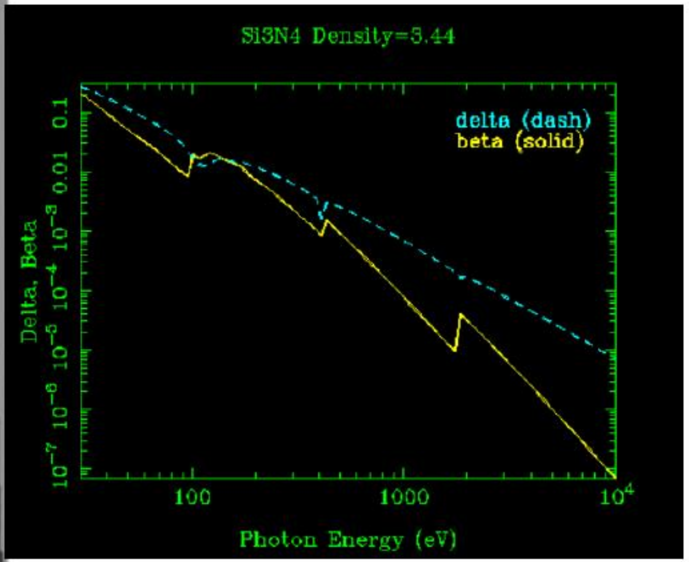
Why phase contrast imaging?
- Measure non-absorbed region
- Suitable for organic materials
- Particularly at high energy

Refractive index = $1 - \delta + i\beta$

Phase shift → $2\pi\ \delta$ thickness/λ

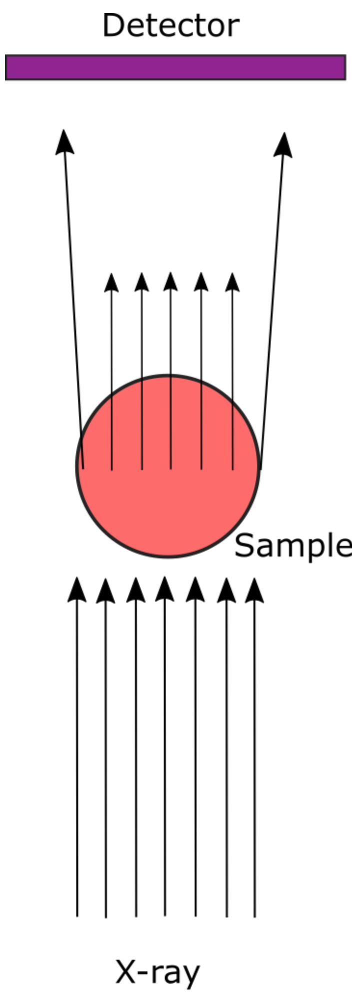
By Zernike phase imaging
Interferometry, analyzer and propagation based methods. At high energy – it the default!

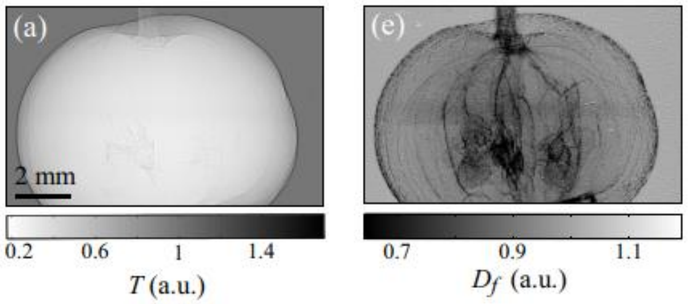
(a) Transmission (b) Phase contrast

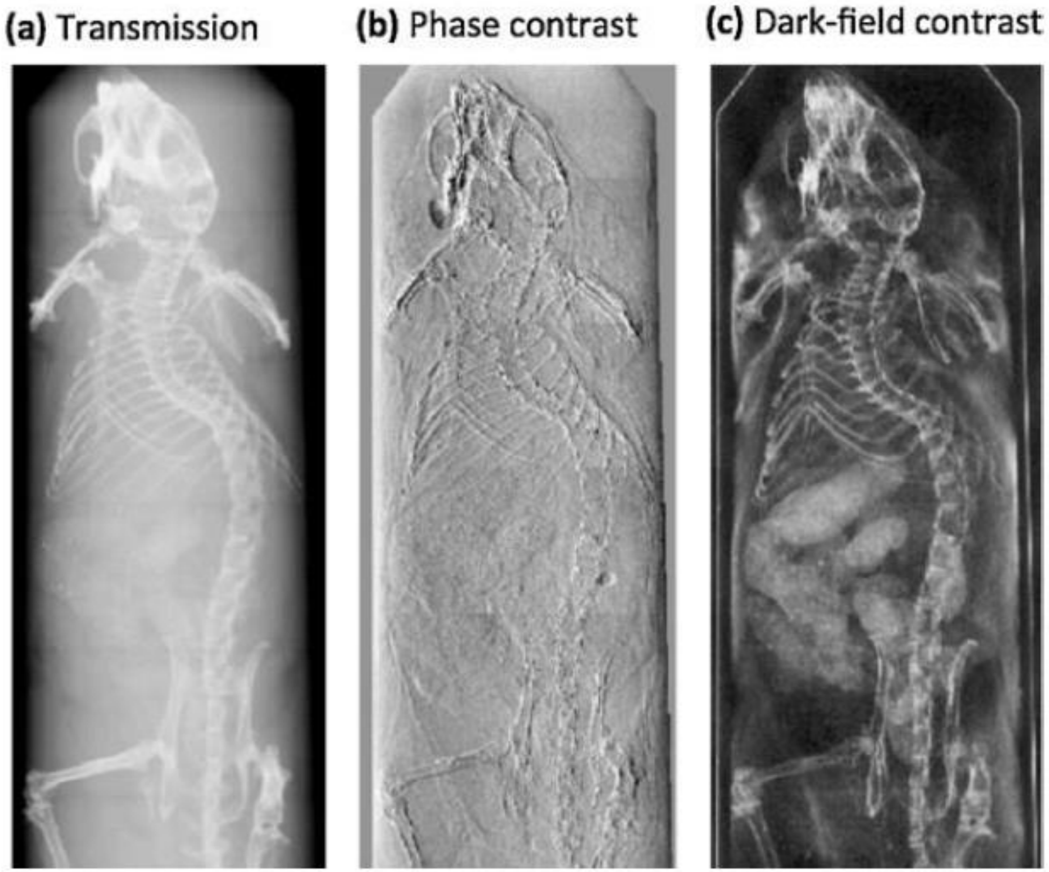


1. F. Pfeiffer et al., Zeitschrift fur Medizinische Physik,23,176-185(2013)
2. B. L. Henke et al., Atomic Data and Nuclear DATA Tables, 54, 181-342 (1993)
3. U. Bonse and M. Hart, APL, 6, 155-156 (1965)
4. V. N. Ingal and E.A. Beliaevskaya, Joutnal of physics D: applied physics, 54, 181-342 (1993)
5. K. A. Nugent et al., PRL, 77, 2961 (1996)

Modalities – dark field imaging







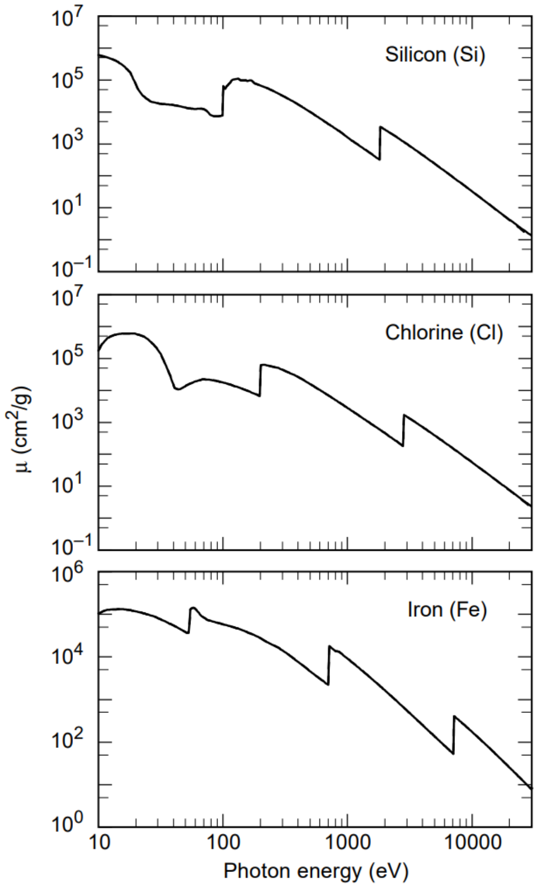
Extract details smaller than the voxel size

Information exist – just need to separate them ☺

1. F. Pfeiffer et al., Zeitschrift fur Medizinische Physik,23,176-185(2013)
2. S. Berujon et al., PRA, 92, 013837 (2015)

Modalities – Spectral imaging

An approach to obtain chemical contrast



Absorption vs photon energy

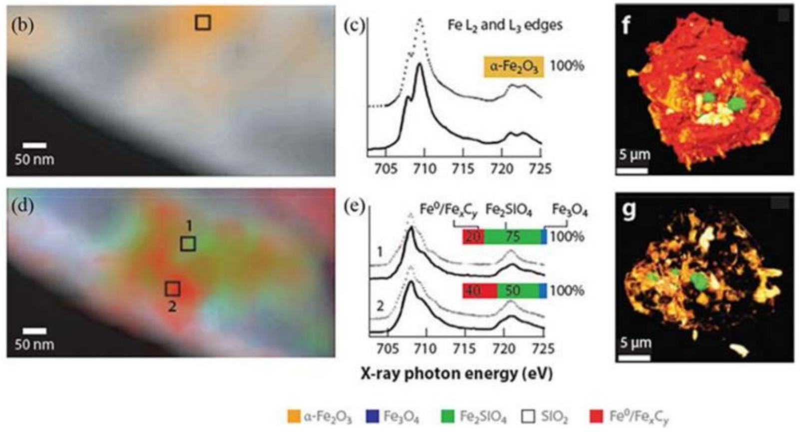
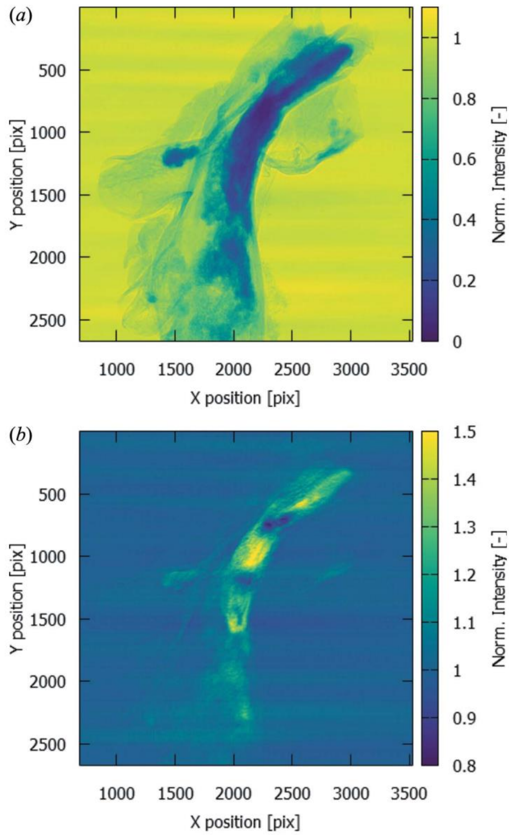
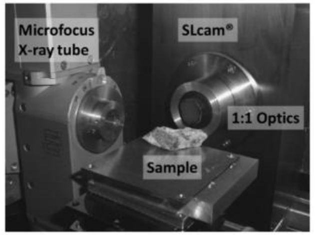


Image a sample with different absorption edges



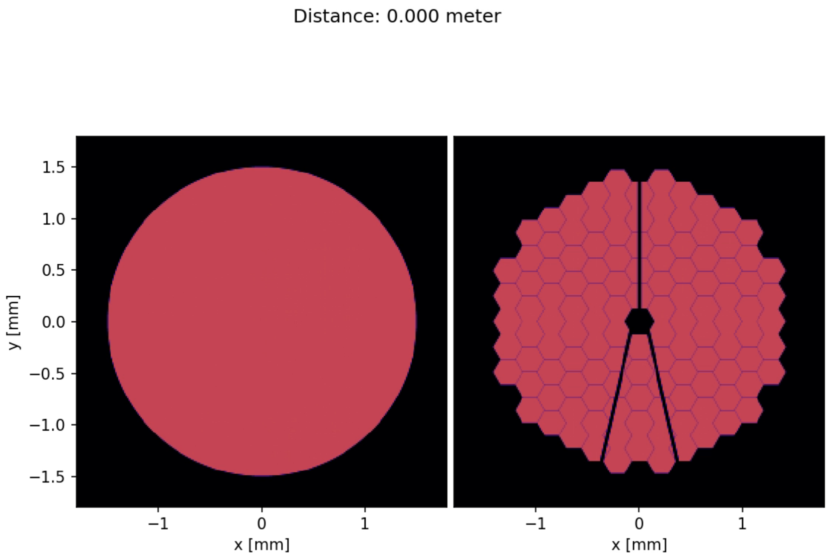


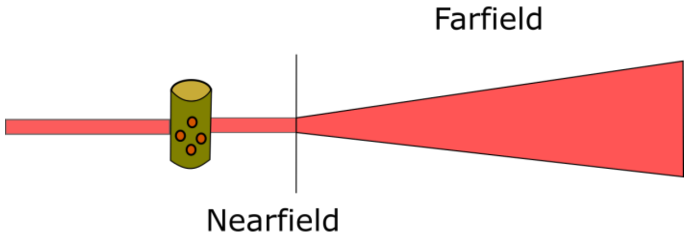
Spectral/hyperspectral imaging -Detector level, create bins to separate interacted photons

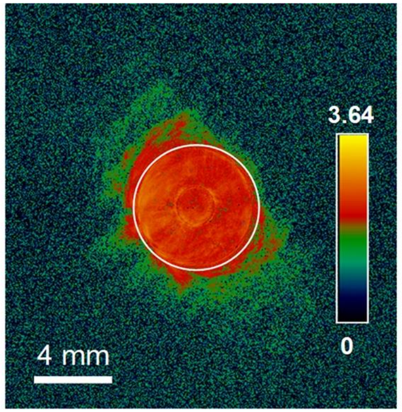
1. M. Gogate, Chemical Engineering Communications. 204,1-27(2017)
2. B. L. Henke et al., Atomic Data and Nuclear DATA Tables, 54, 181-342 (1993)
3. M. N. Boone et al., Nuclear Instruments and Methods in PhysicsResearch Section !: Accelarators, Spectrometers and Detector and Associated Equipments, 735, 644-648 (2014)
4. M. Boone et al., JSR, 27, 110–118 (2020)
5. A. Bjeoumikhov et al., Journal of Instruments, 7 (2012)
6. K. Desjardins et al., JSR, 27, 1577-1589 (2020)

Modalities – X-ray diffraction imaging

Coherent diffraction imaging → Near field diffraction and far-field diffraction (resolution less than 10 nm)

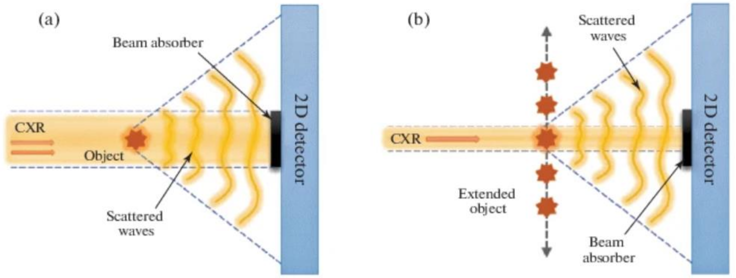






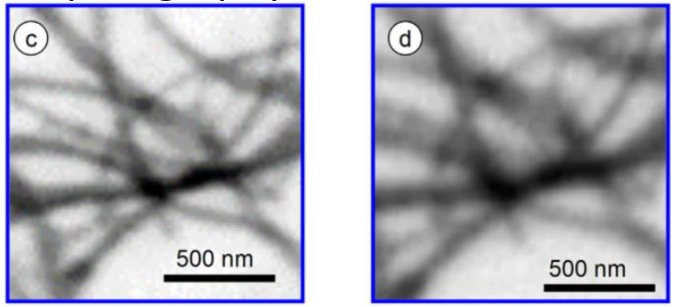
Coherent diffraction imaging Ptychography

Fig. 1.



More the beam scatters → king of magnification!

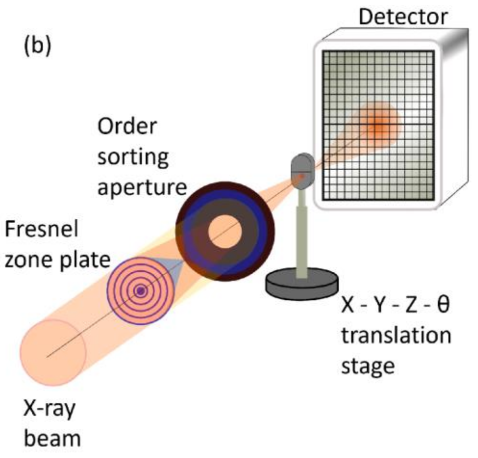
Ptychography STXM

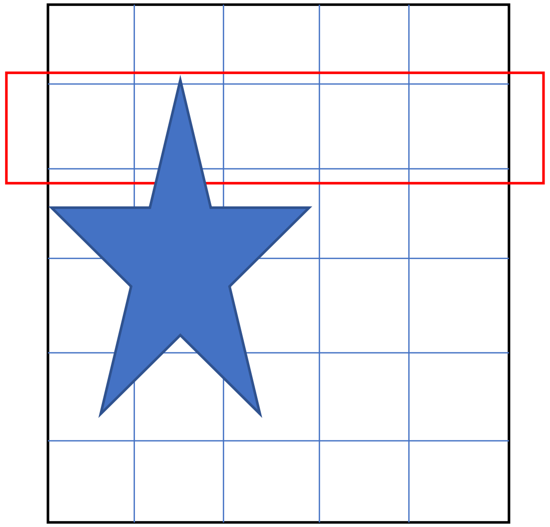


P. A. Prosekov et al., Methods of Coherent X-ray Diffraction Imaging, 66, 867-882 (2021)
N. Mille et al., Communications materials, 3,8 (2022)

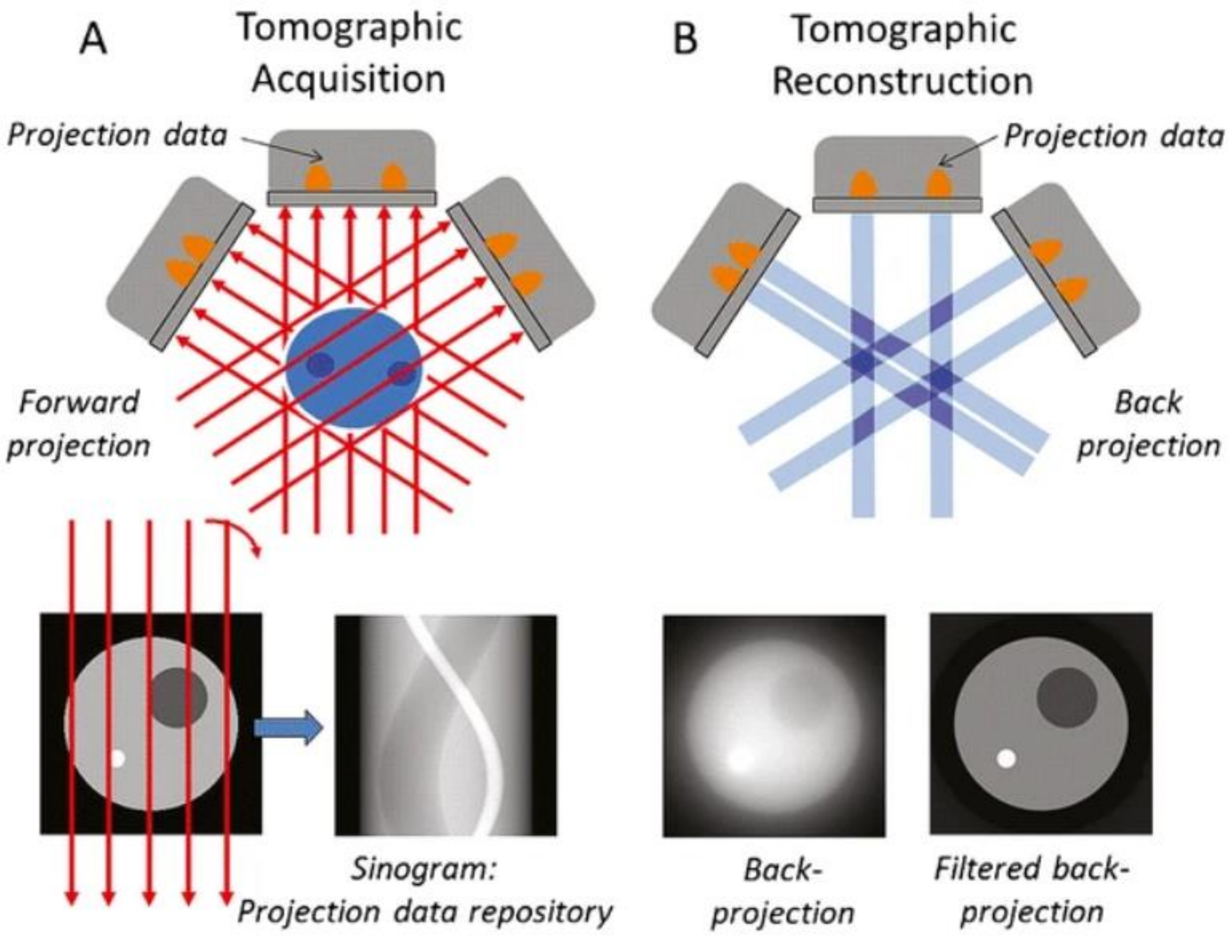
[Near and far-field diffraction — HCIPy 0.5.2.dev325+g3cdb57f documentation]

Modalities – Computed Tomography





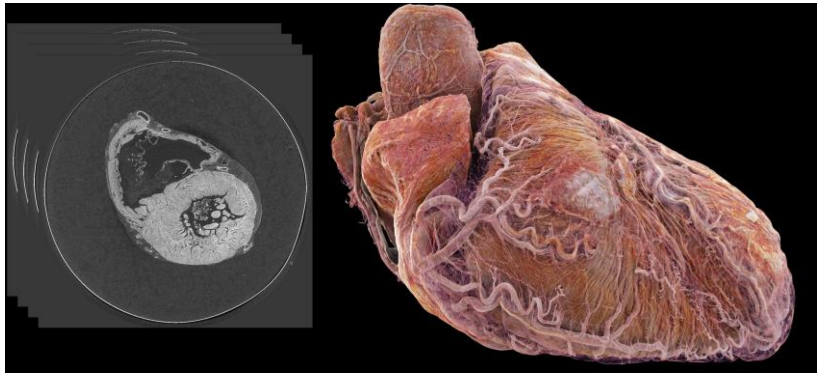
Detector pixels



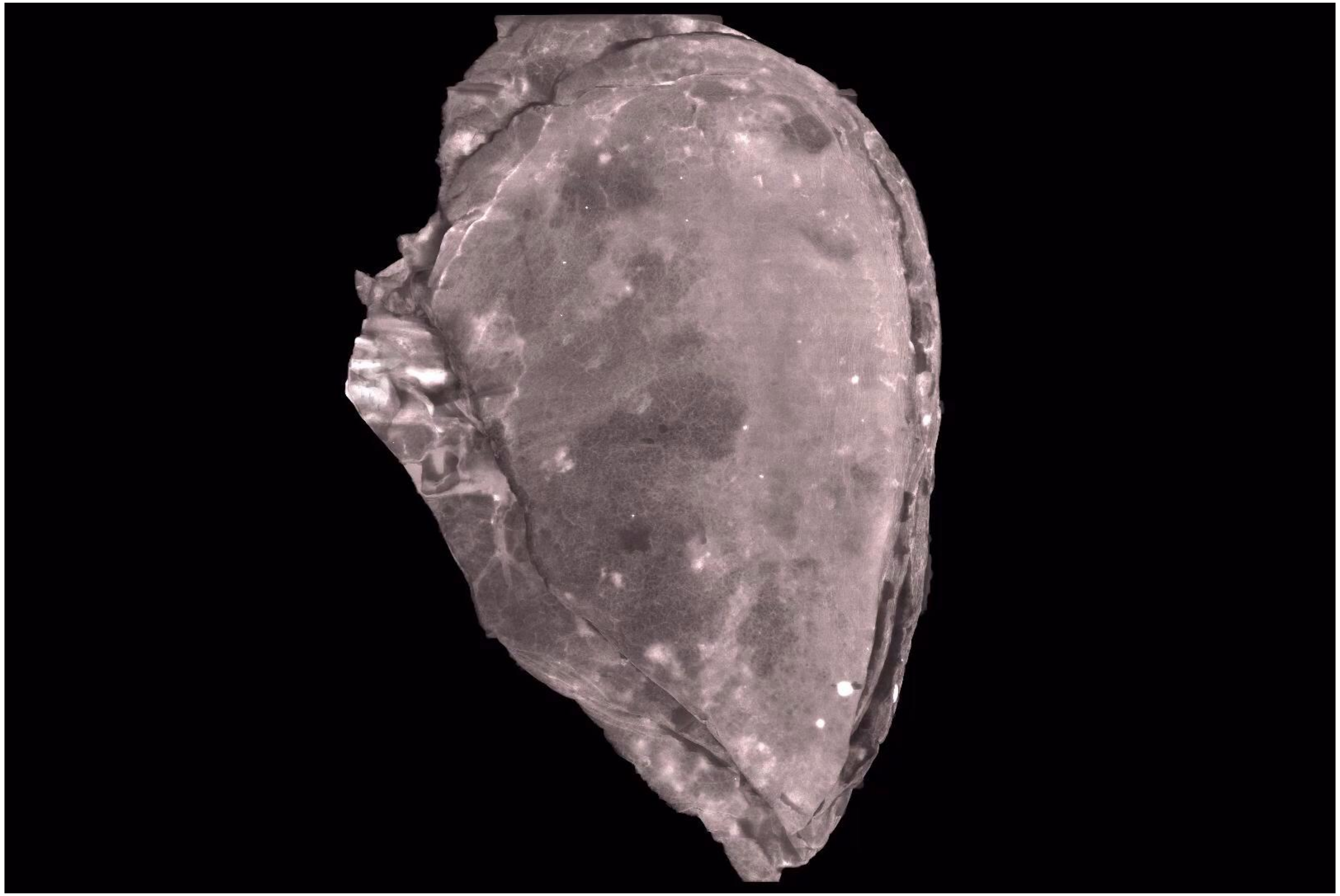
J. A. Seibert, Pediatr. Ridiol 44 (Suppl 3), 431-439 (2014)

[Human Health Campus - 3D image reconstruction (iaea.org)]

Modalities – microtomography



Courtesy - J. Brunet (BM18 - ESRF/UCL) - human heart



Human Lung

[Human Organ Atlas (esrf.eu)](https://www.esrf.eu)

C. L. Walsh et al., Nature Methods, 18, 1532-1541 (2021)

J. Vijayakumar et al., Pharmaceuticals, 16(5), (2023)

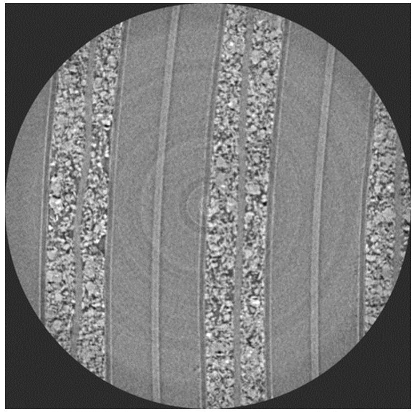
Modalities – microtomography

Lobster - Courtesy – K. Dollman (BM18)

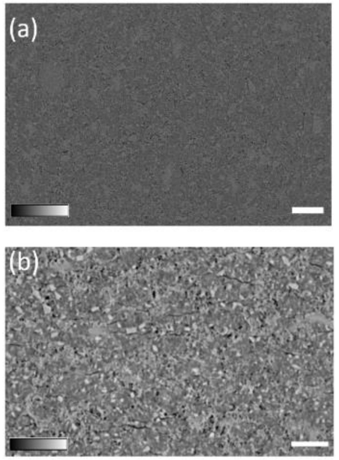
Shrimp - Courtesy – P. Tafforeau (BM18)

Dinosaurs and birds - Courtesy – V. Beyrand (BM18)

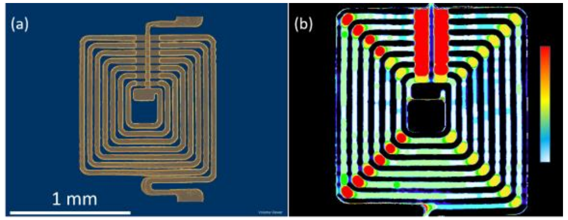
Nanotomography



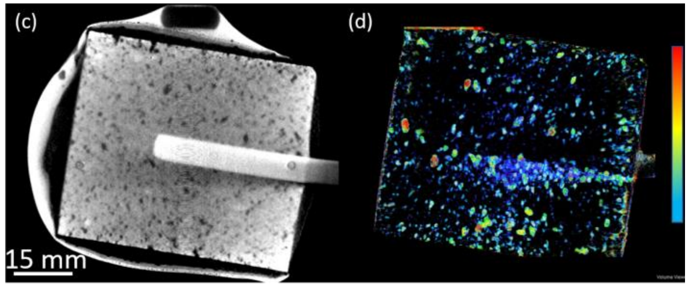
Battery



Tablet



Inductor



Capacitor

Modalities – Diffraction contrast tomography

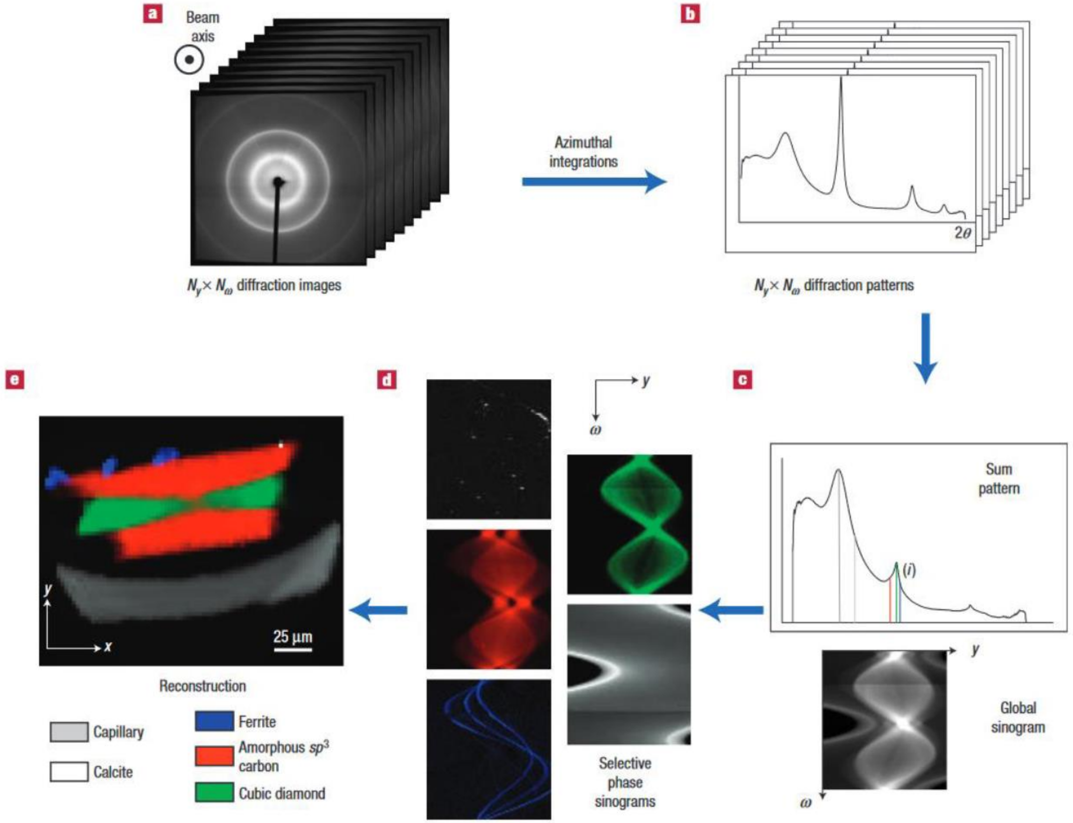
To measure local crystallinity

There are two types – DCT/XDT

Computationally intense

Resolution depends on beam size

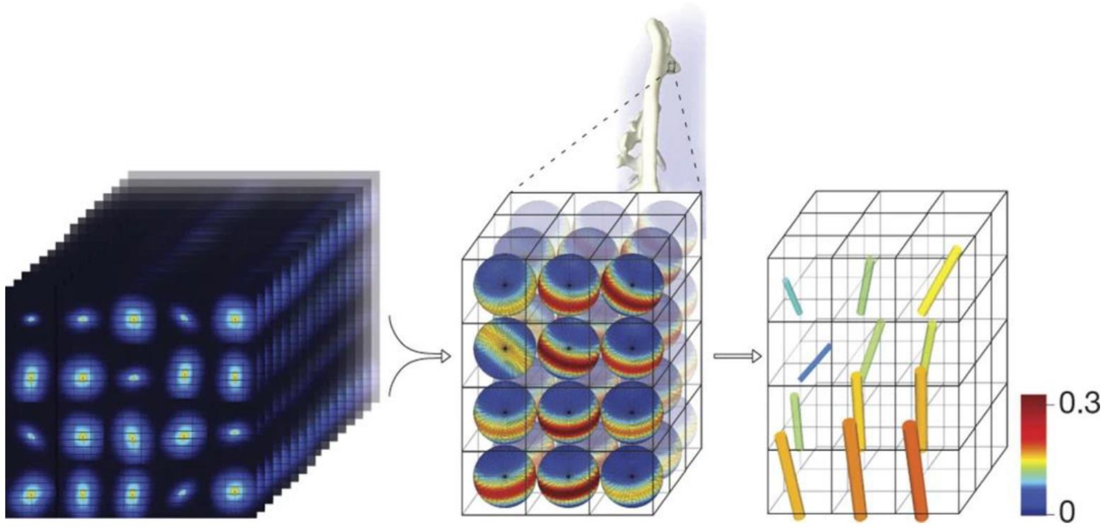
Lab based sources – not implemented

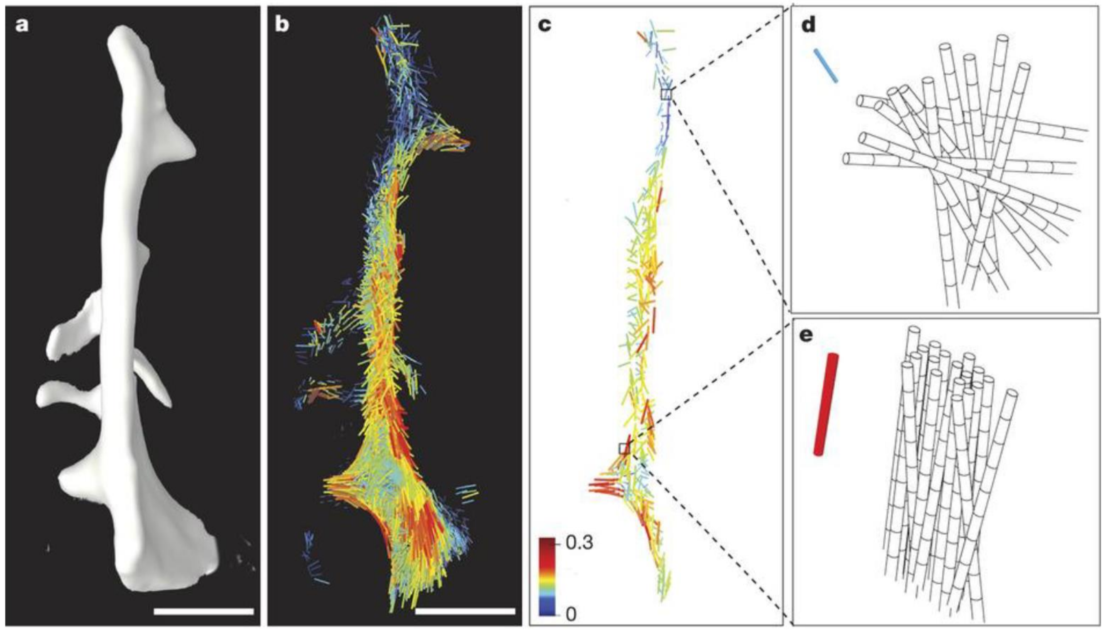


1. Henning Friis Poulsen, Three-dimensional X-ray diffraction microscopy: mapping polycrystals and their dynamics, Vol. 205 (Springer Science & Business Media, 2004).
2. P. Bleuet, et al., “Probing the structure of heterogeneous diluted materials by diffraction tomography,” Nature materials7,468–472 (2008)
3. Wolfgang Ludwig et al., “High-resolution three-dimensional mapping of individual grains in polycrystals by topotomography,” Journal of Applied Crystallography40, 905–911 (2007).
4. Peter Reischig et al., “Advances in X-ray diffraction contrast tomography: flexibility in the setup geometry and application to multiphase materials,” Journal of Applied Crystallography46, 297–311 (2013).
5. W. Ludwig et al., “X-ray diffraction contrast tomography: a novel technique for three-dimensional grain mapping of polycrystals. i. direct beam case,” Journal of Applied Crystallography41, 302–309 (2008).
6. G. Johnson et al., “X-ray diffraction contrast tomography: a novel technique for three-dimensional grain mapping of polycrystals. ii. The combined case,” Journal of Applied Crystallography41, 310–318 (2008).
7. W. Ludwig et al., “Three-dimensional grain mapping by x-ray diffraction contrast tomography and the use of Friedel pairs in diffraction data analysis,” Review of scientific instruments80,033905 (2009)

Modalities – SAXS Tensor tomography

- Absorption – scalar quantity
- 3D X-ray scattering vector – tensor quantity





1. G. Gullberg et al., “Tensor tomography,” IEEETransactionson Nuclear Science46, 991–1000 (1999).
2. Marianne Liebi et al., “Nanostructure surveys of macroscopic specimens by small-angle scattering tensor tomography,” Nature527, 349–352 (2015).
3. Marianne Liebi et al., “Small-angle x-ray scattering tensor tomography: model of the three-dimensional reciprocal-spacemap, reconstruction algorithm and angular sampling requirements,” Acta CrystallographicaSectionA: Foundations and Advances74, 12–24 (2018)

Thank you!

Jaianth.Vijayakumar@esrf.fr