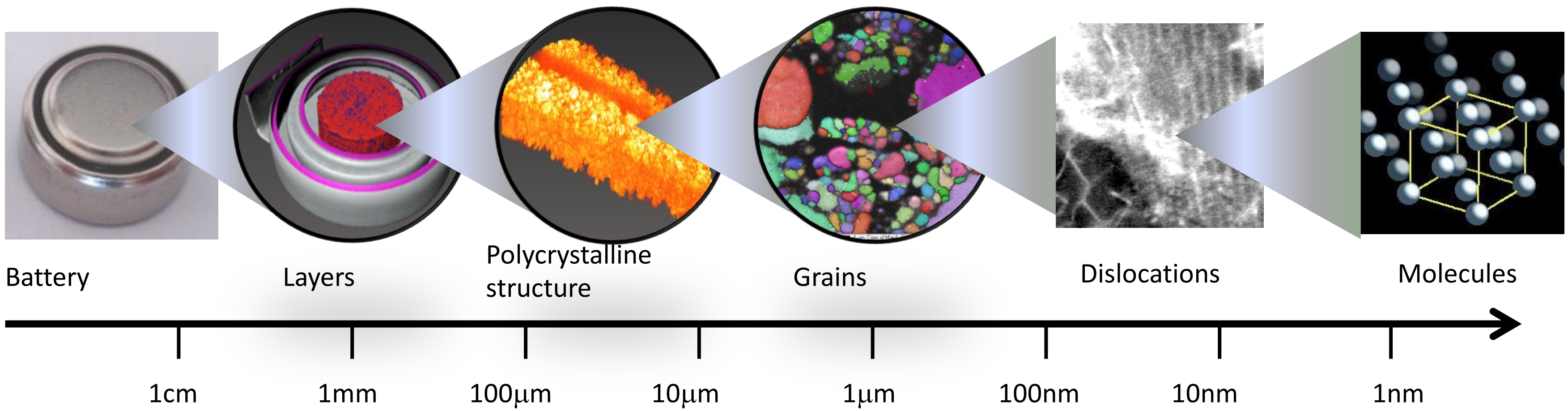
Imaging: diffraction and microscopy

Callister&Retwitsch version 10: 3.16 and 4.9-4.11

Henning Friis Poulsen, hfpo@dtu.dk



- Many types of contrast
- Imaging in 2D, 3D and 4D (as function of time)

Imaging of materials

Methods

Diffraction | Microscopy | Tomography | Cantilever arm
Atoms in a crystal | 2D image | 3D image | Surface studies

Sources

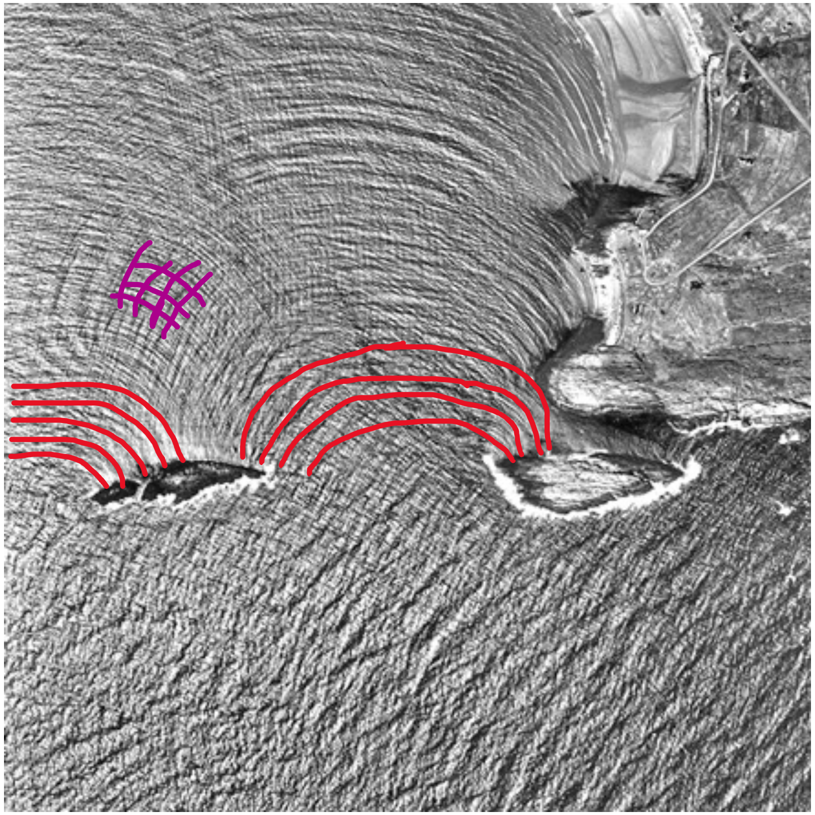
Visual light

Electrons

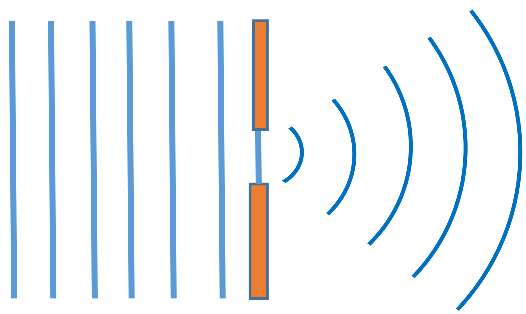
X-rays

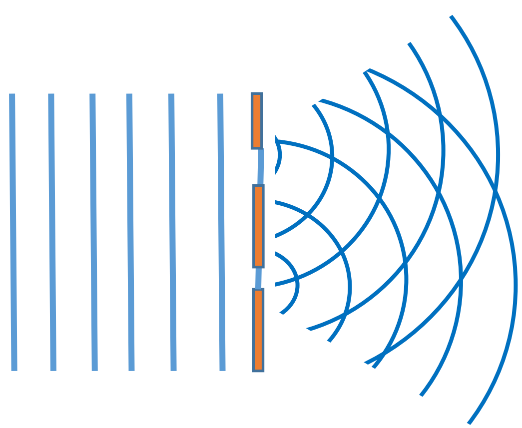
Scanning probe

Diffraction in 2D: interference of waves

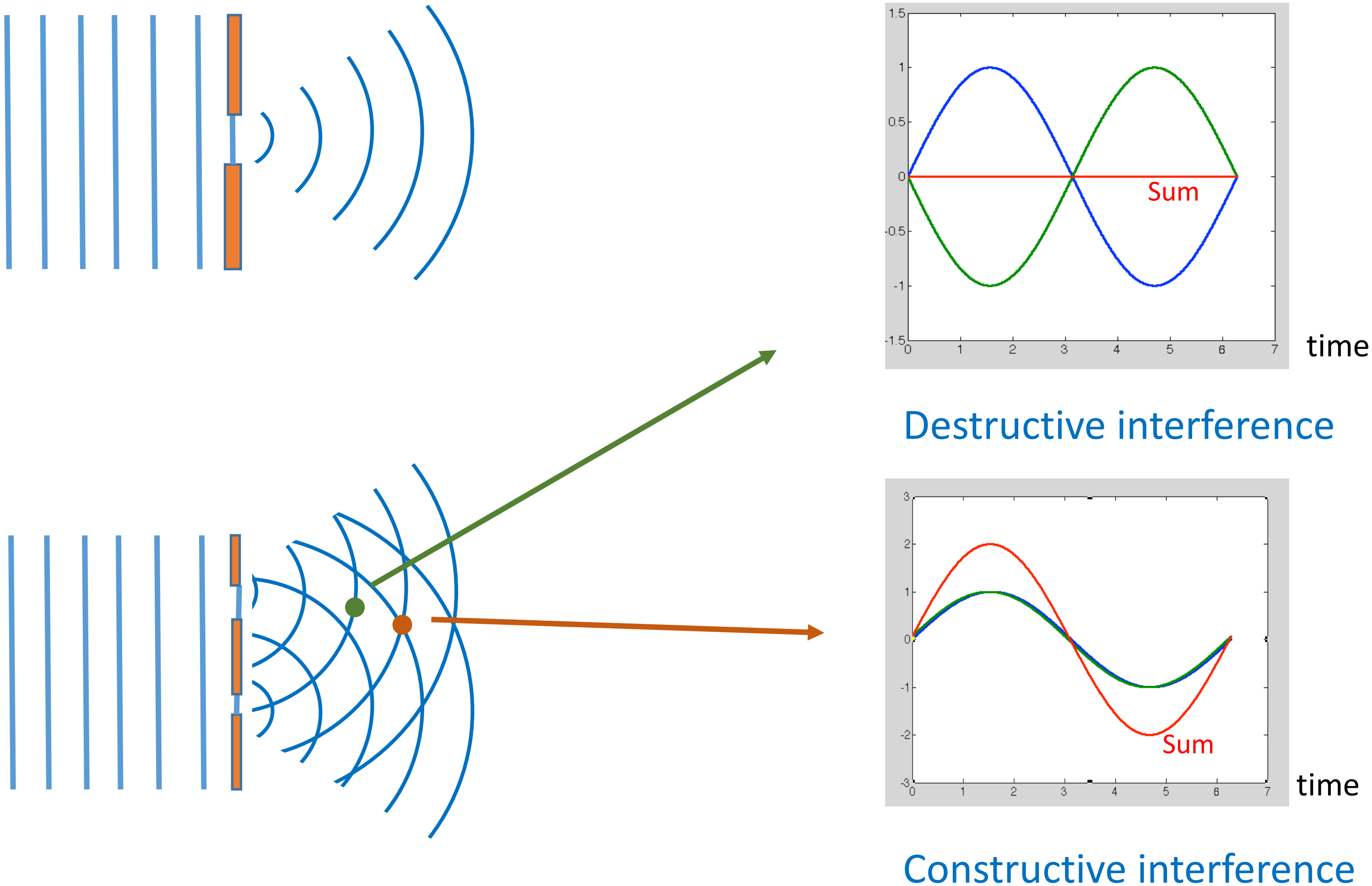


Diffraction in 2D: interference of waves

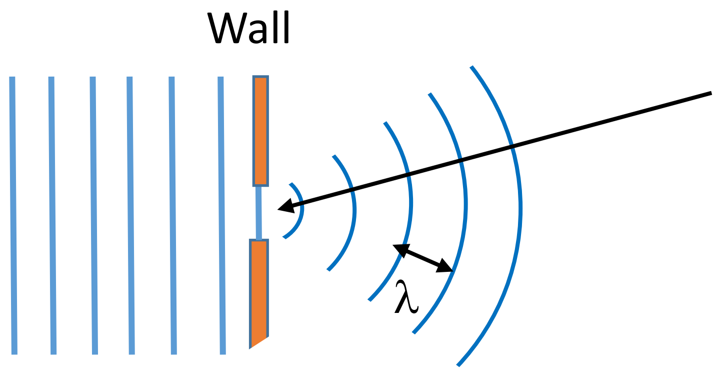




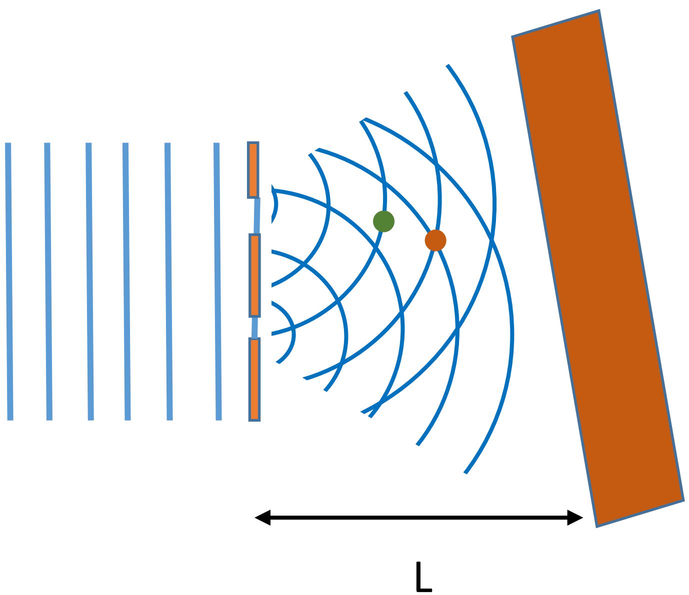
Diffraction in 2D: interference of waves



Diffraction in 3D: interference of waves



Aperture: a cylindrical hole, size Δ



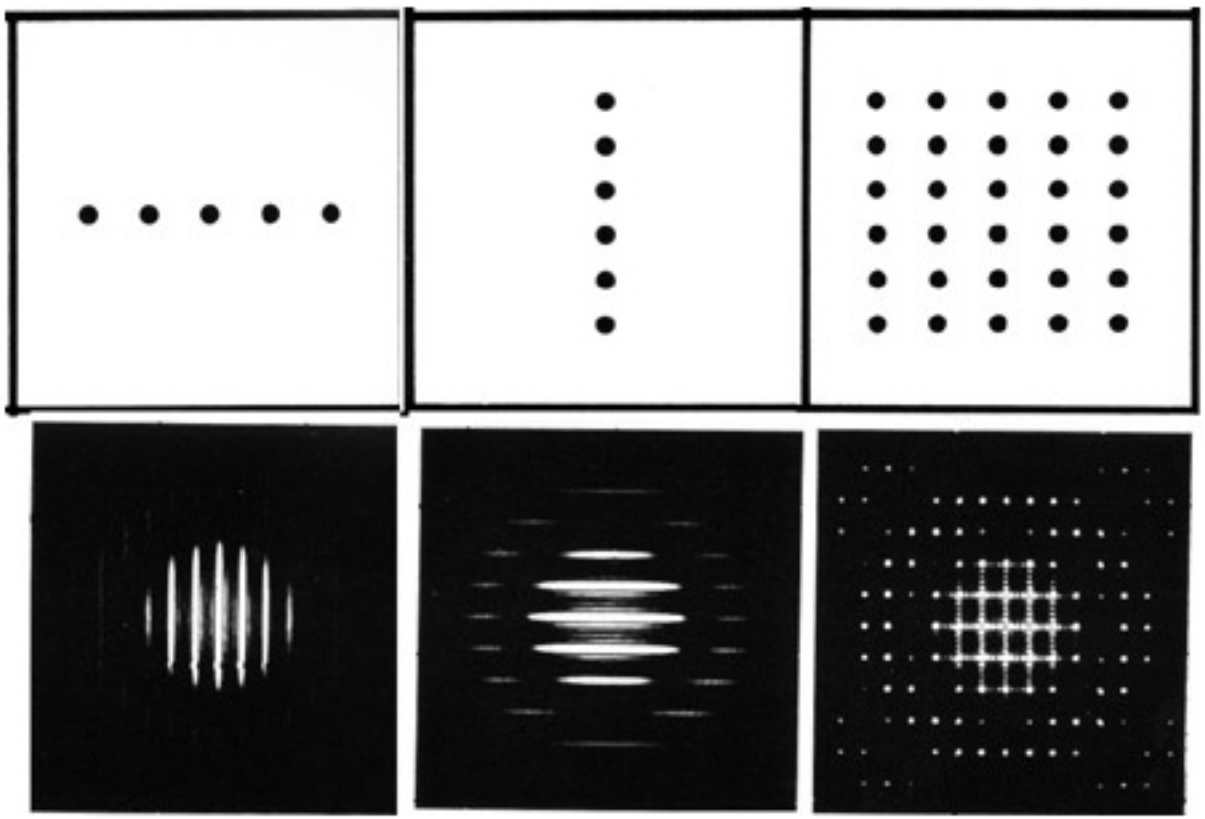
2D detector

Assume: $\Delta^2/(\lambda L) << 1$
(Frauenhofer limit)

Diffraction: interference of waves

Aperture

Image on detector



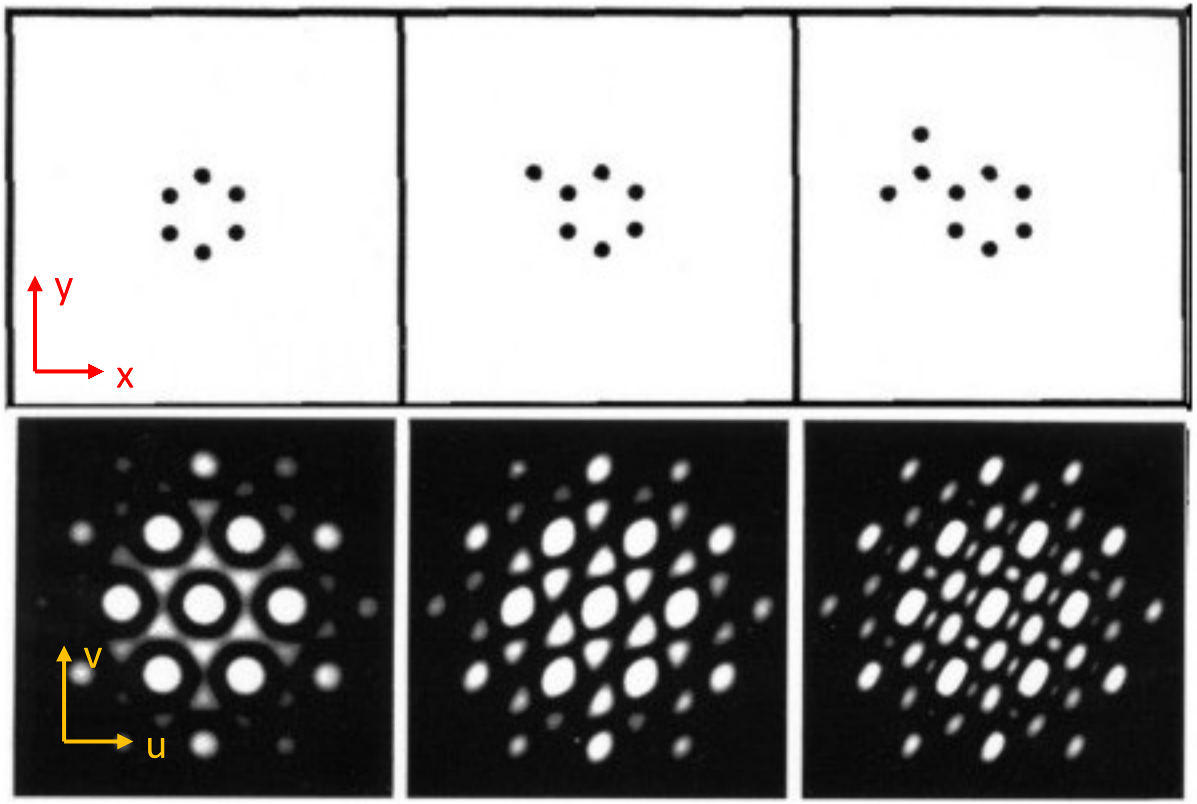
More holes
=> Sharper pattern

Diffraction: interference of waves

Fourier Transform

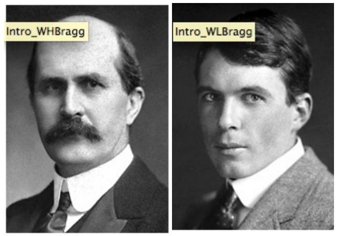
f(x,y)

F(u,v)

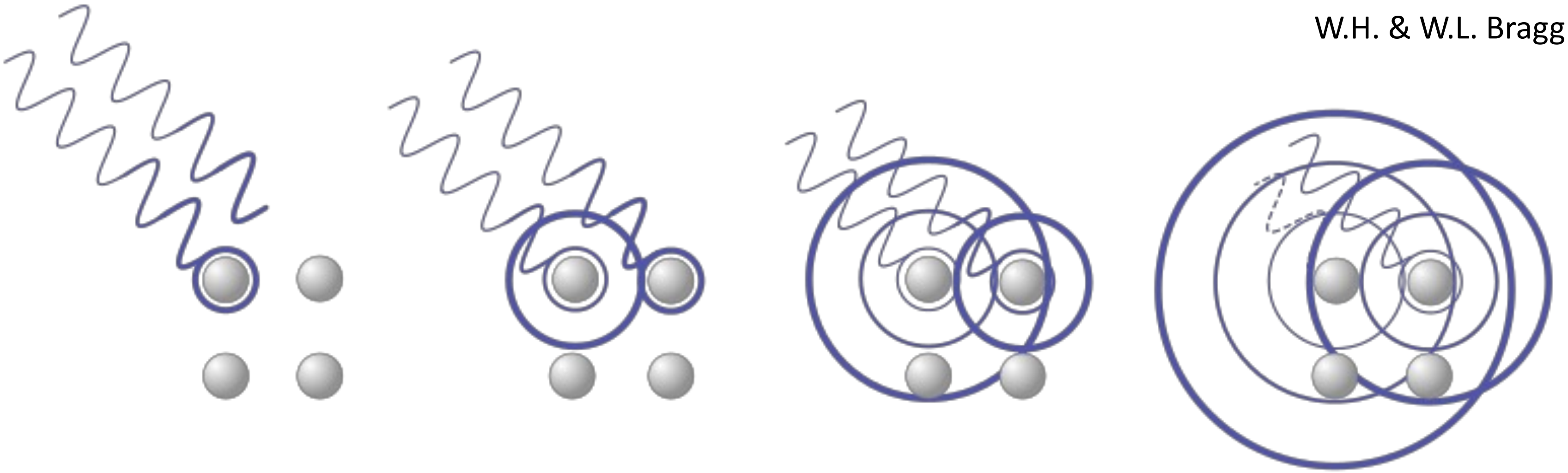


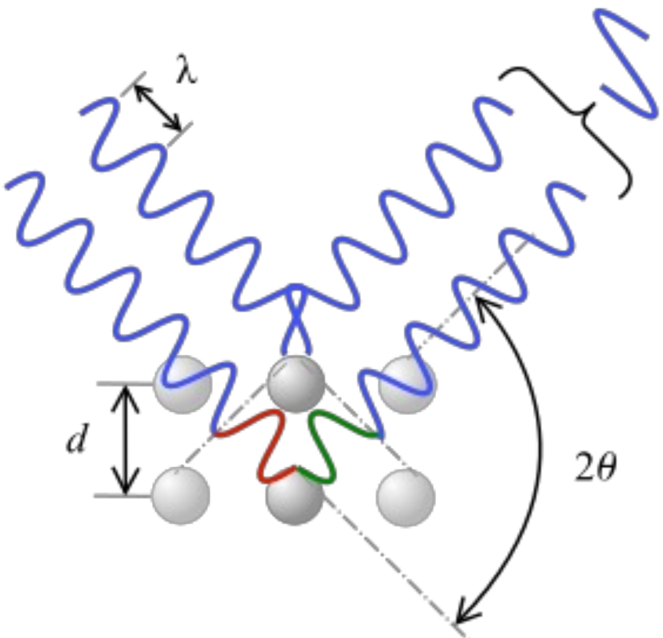
From diffraction pattern we can determine the hole pattern

Bragg Diffraction



W.H. & W.L. Bragg 2013





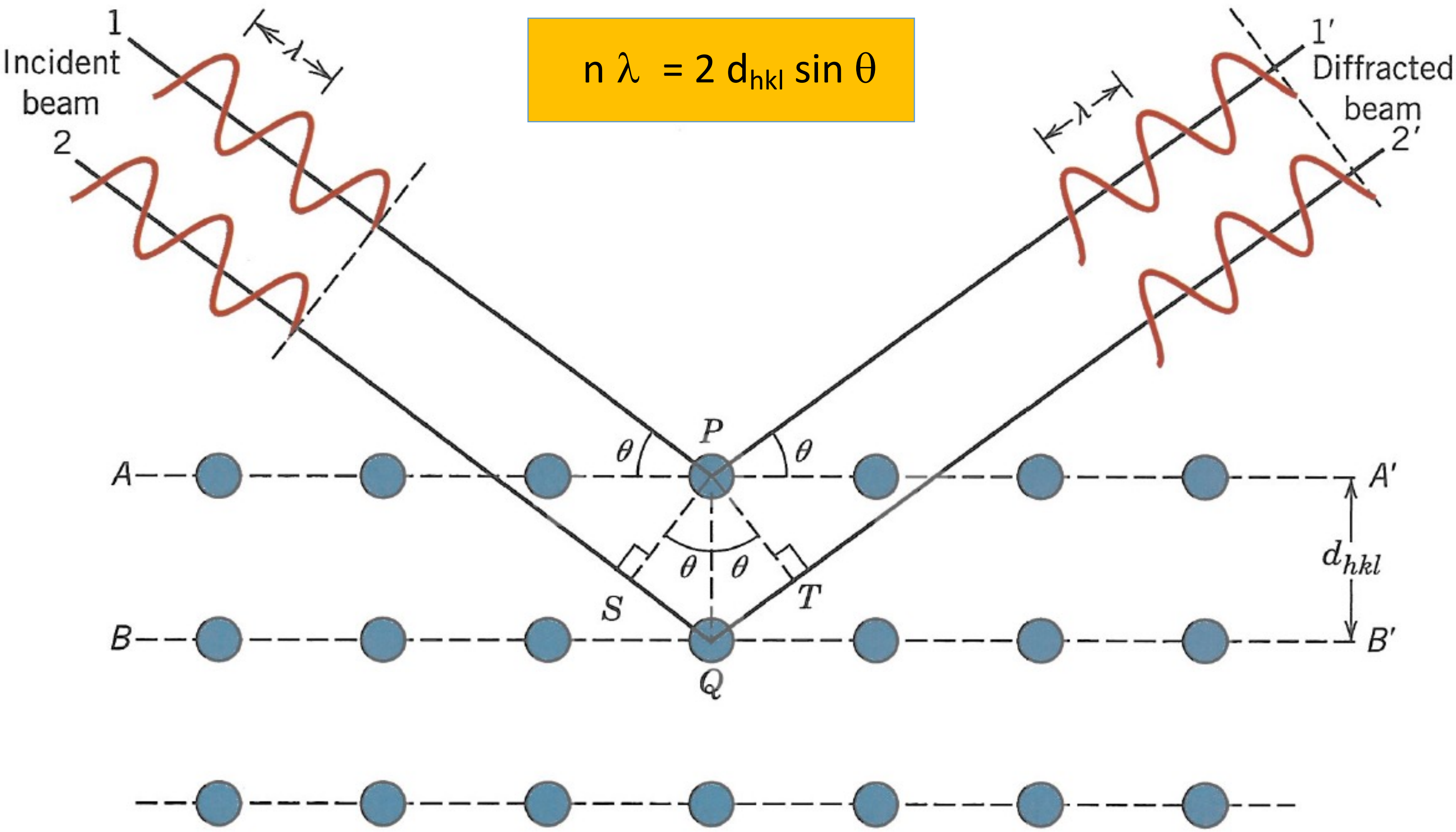
Source: wikipedia

Bragg Diffraction

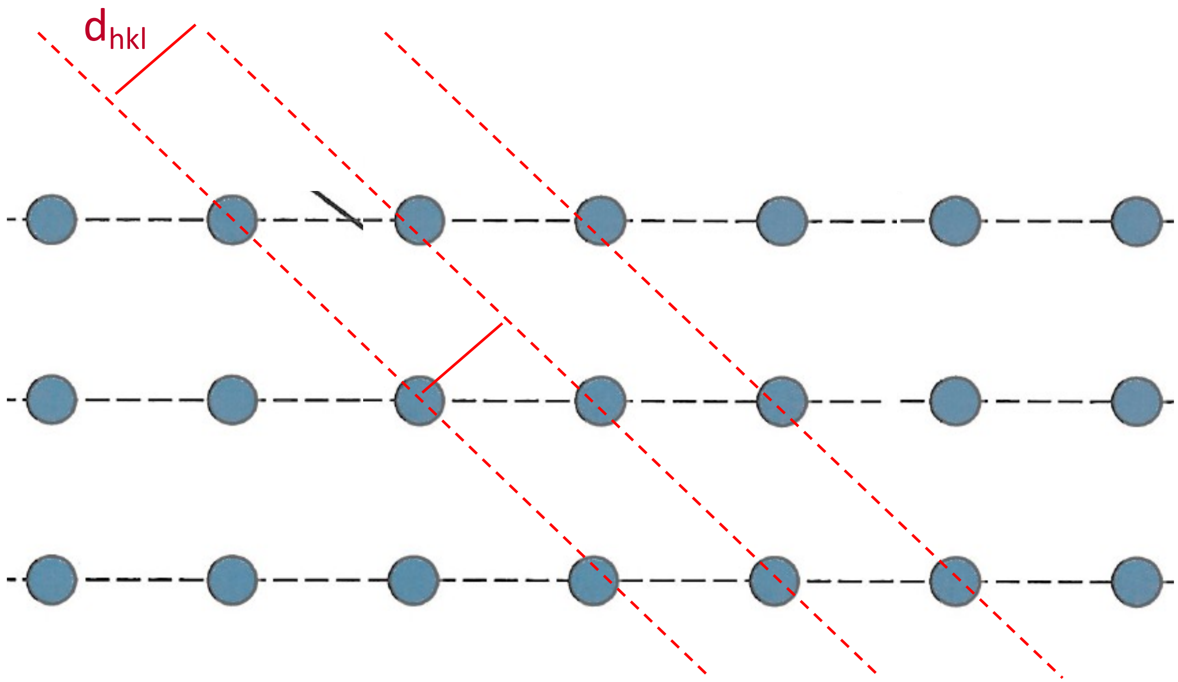
$n\lambda = |SQ| + |QT|$

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

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

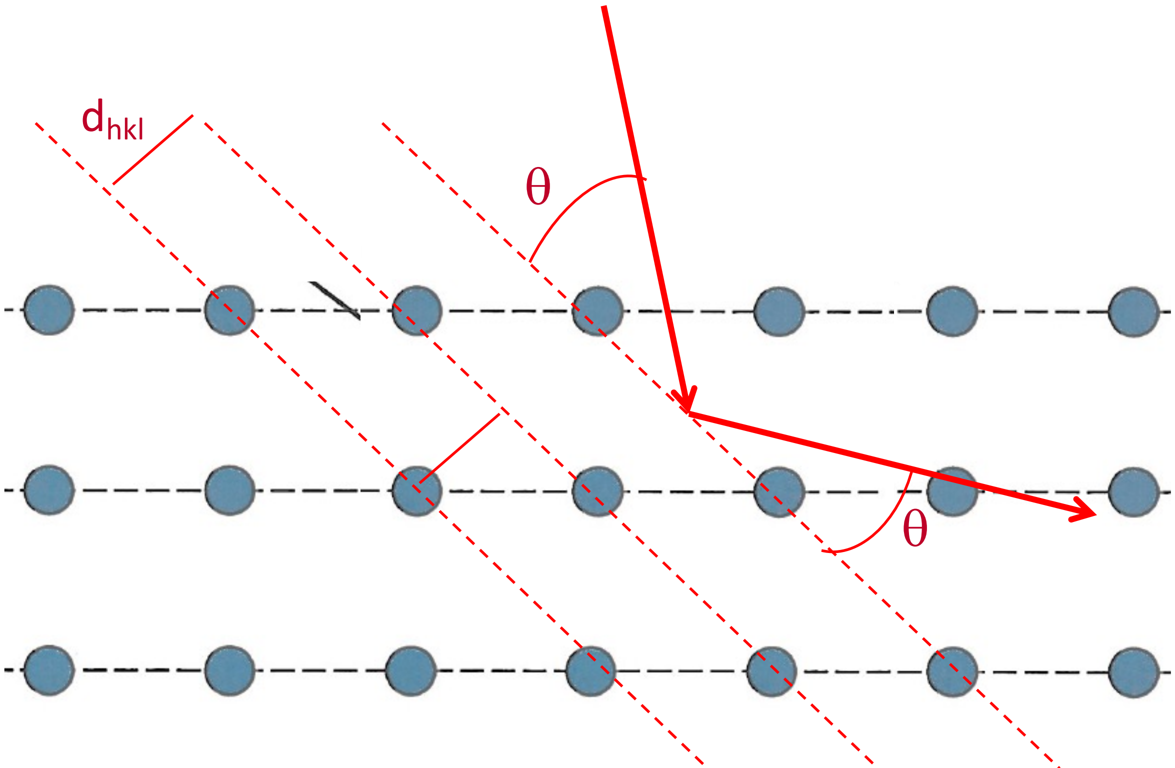


Bragg Diffraction



Bragg Diffraction

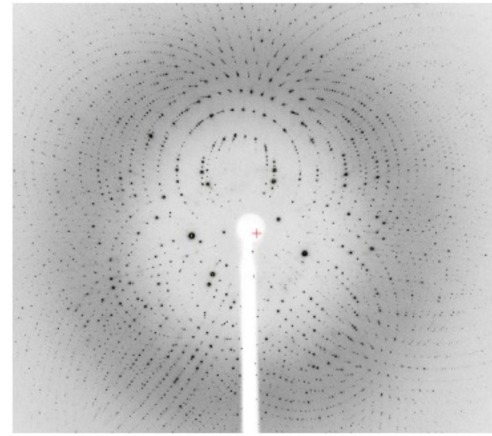
$n\lambda = 2\, d_{hkl} \sin\theta$

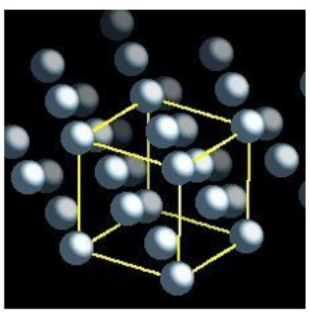


For every d we can measure a scattering angle, 2θ

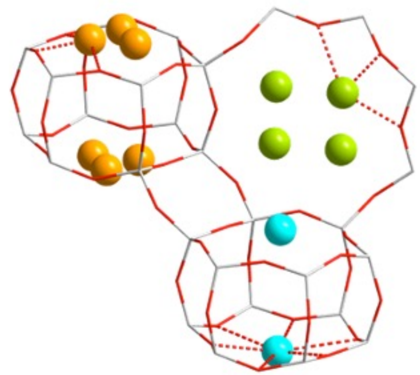
Crystallography

$N_A = 6\ 10^{23}$ => Very narrow spots on detector
with extreme enhanced intensity

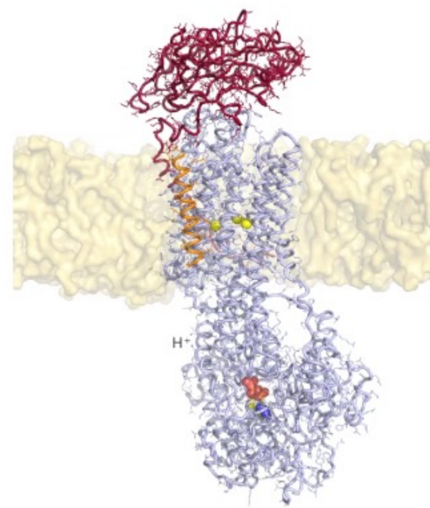




Al



Catalysis molecule



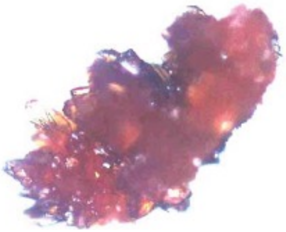
Membrane protein

Crystallography

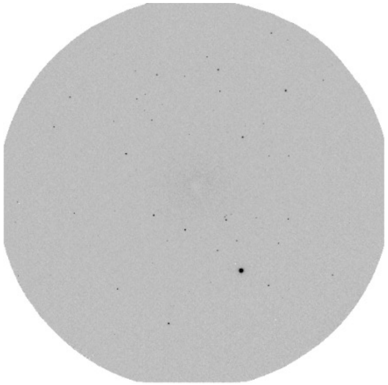
Single Crystal

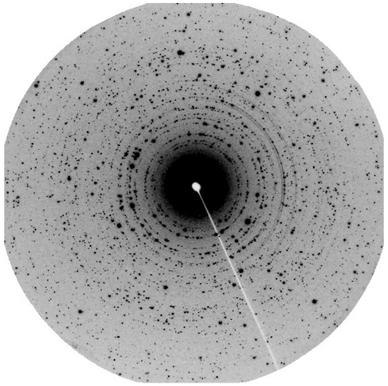
More crystals

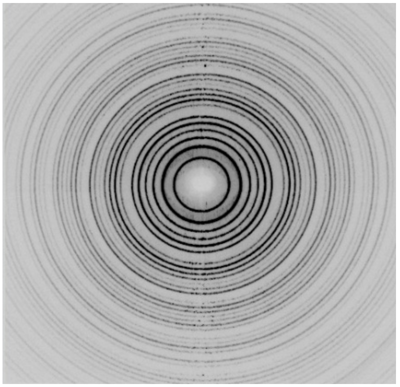
Powder



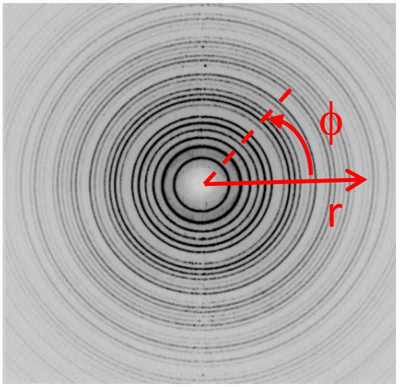
X-ray diffraction:







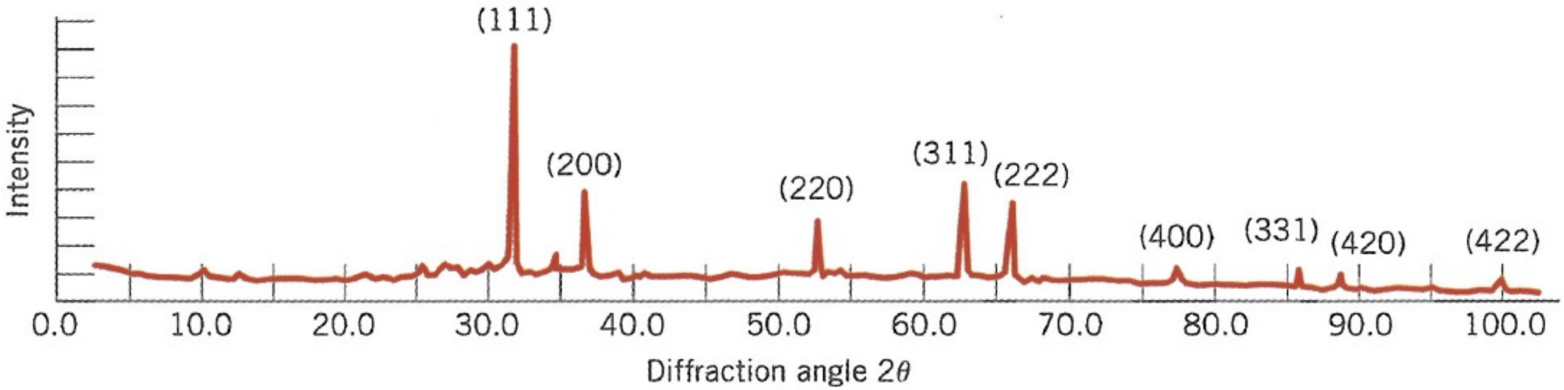
Powder diffraction



Radial variation: Intensity as fct of r

Intensity as fct of 2θ

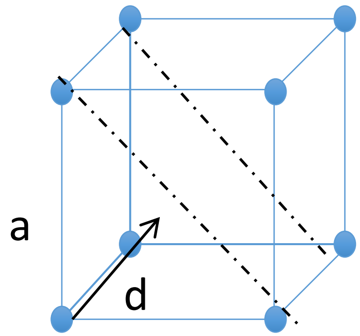
X-ray data on Pb (FCC)



Cubic symmetry

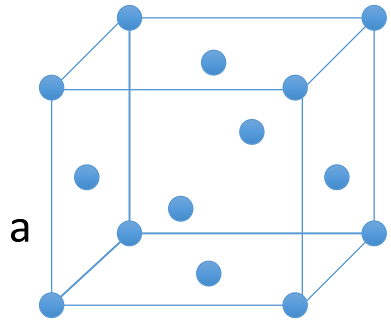
$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

without basis

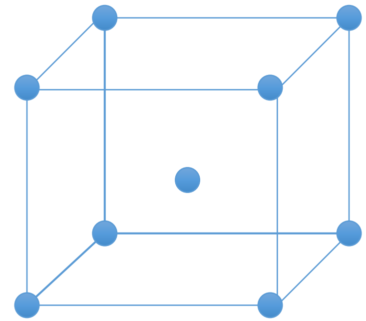


with a basis:

FCC



BCC



Crystal structure	**Reflections present**	**First 6 indices**
Simple	All	100,110,111, 200,210,211
FCC	h and k and l either all even or all odd	111,200,220, 311,222,400
BCC	(h+k+l) even	110,200,211, 220,310,222

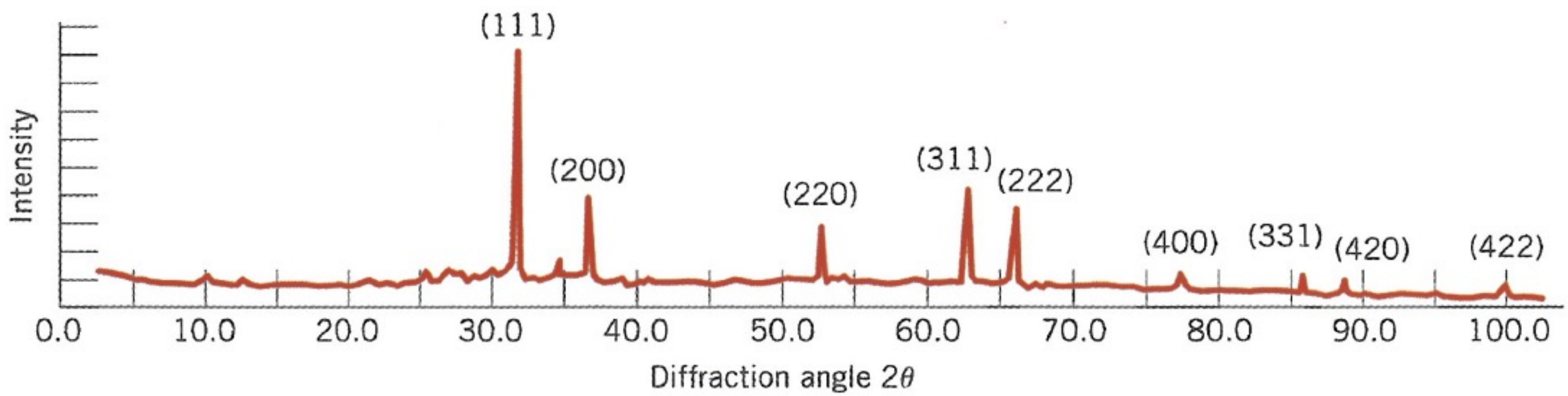
Determine peak position

Study Pb. λ = 0.1542 nm, FCC; a = 0.495

$$d_{hkl} = \frac{a}{\sqrt{h^2+k^2+l^2}};\ \sin\theta = \frac{\lambda}{2d_{hkl}}$$

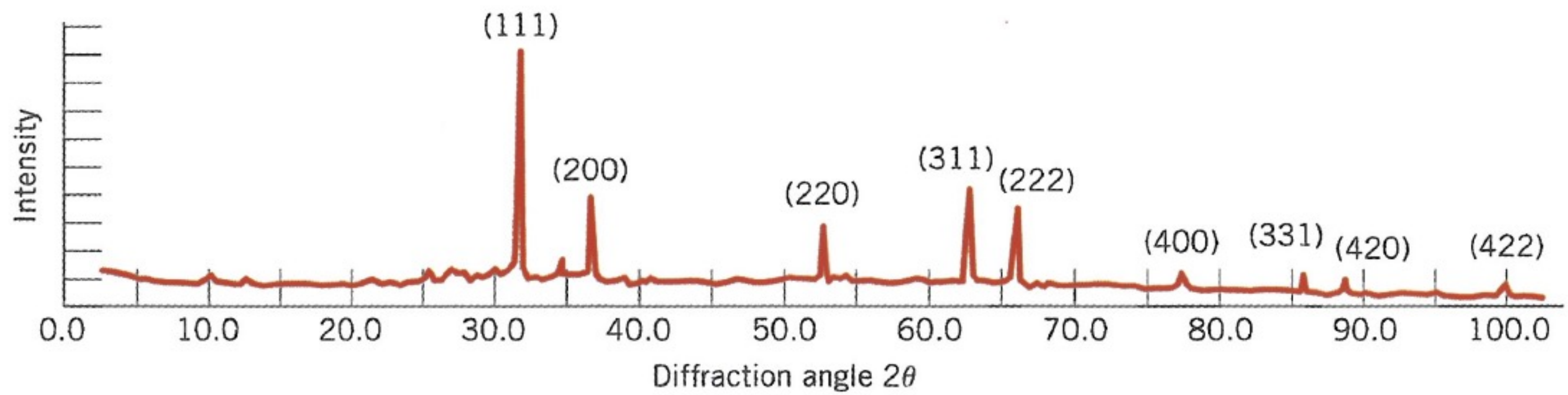
Peak index	d_{hkl} (nm)	2θ (deg)
111	0.285	31.3
200	0.245	36.6
220	0.174	52.6
311	0.149	62.5
222	0.143	65.5

Compare x-ray data

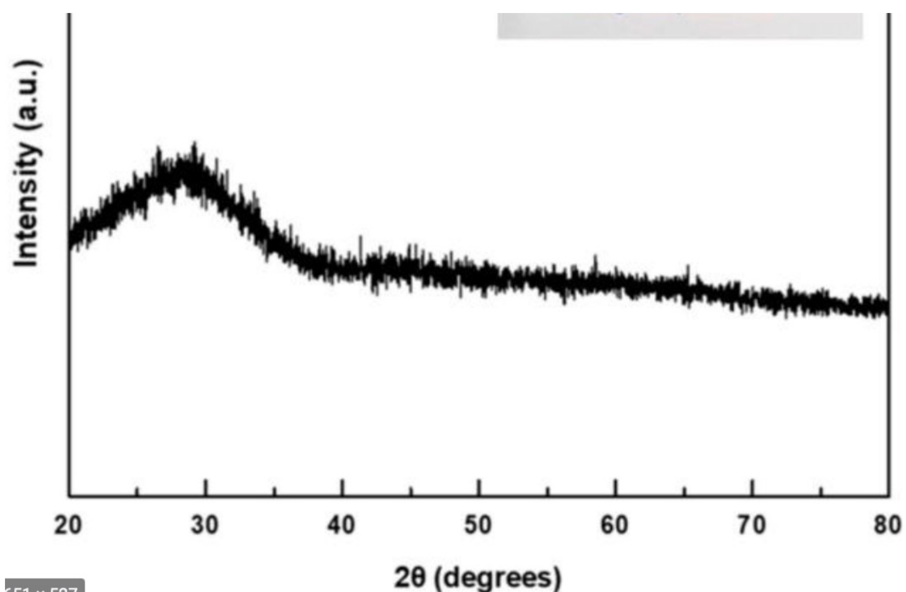


One question for the break

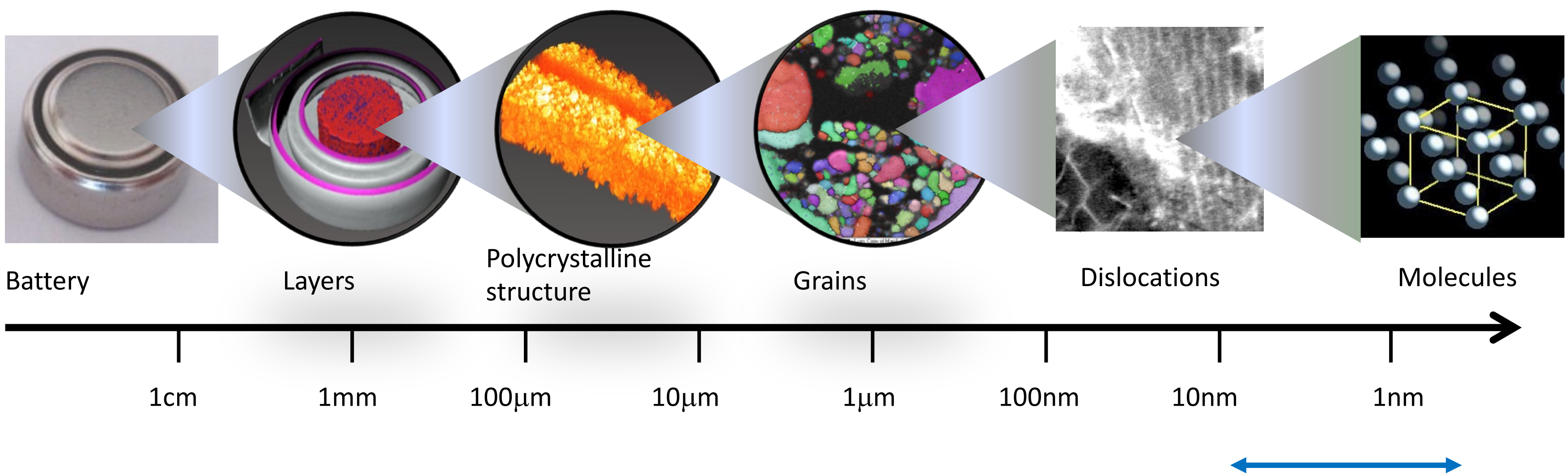
X-ray data on a crystal



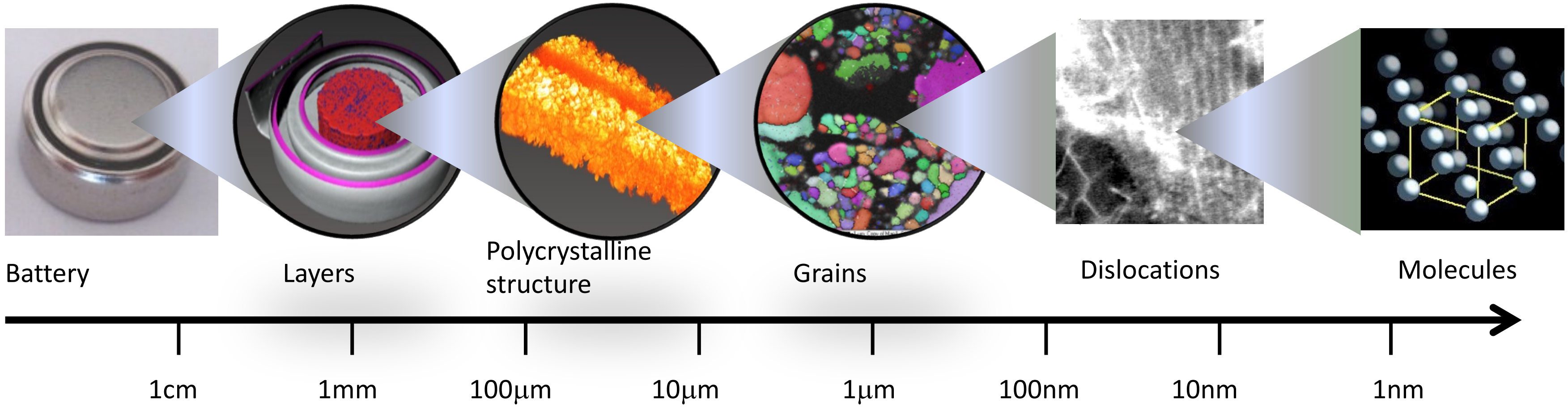
X-ray data on amorphous material



Imaging: diffraction and microscopy



Imaging: diffraction and microscopy



Which length scale do we probe by diffraction?

Microscopy

Key specifications ?

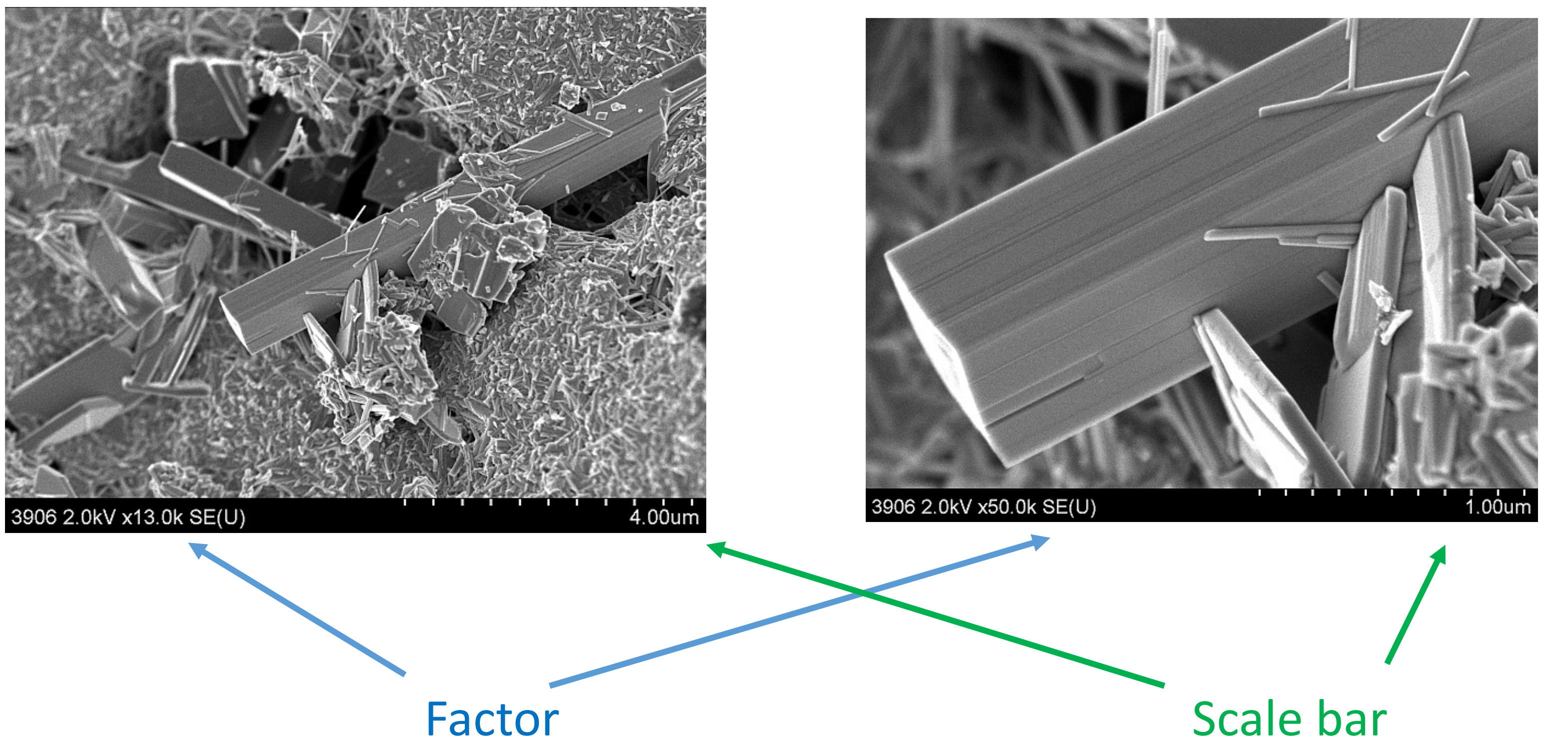
Consider for 2.5 minutes…

Microscopy

Key specifications ?

- Magnification
- Spatial resolution
- Field of view
- Time resolution
- Contrast
- Sample preparation
- Sample environment

Magnification



Magnification and spatial resolution

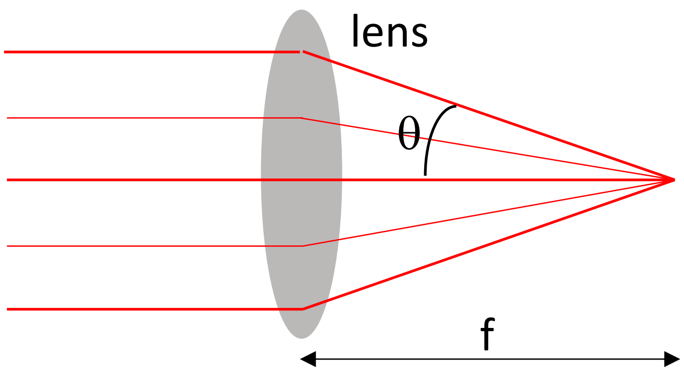
Hubble telescope:

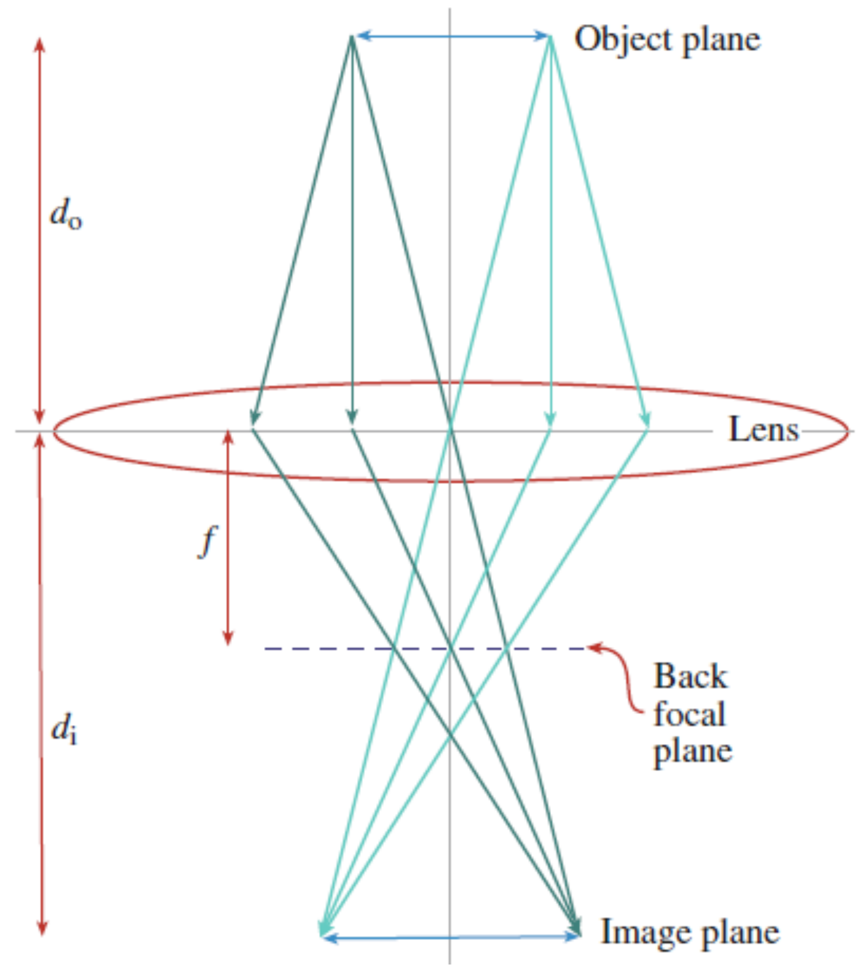
Wide Field Planetary Camera 1

Wide Field Planetary Camera 2

Lens equation and magnification

Focal length: f



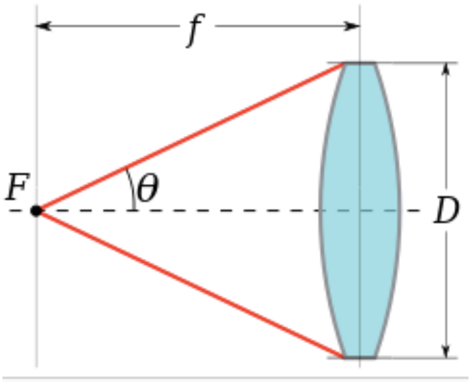


$$1/d_i+1/d_0=1/f$$

$$M=d_i/d_0$$

Spatial resolution

Numerical aperture:



$$NA = n\sin\theta \approx n\frac{D/2}{f};$$

D is the diameter of the lens
n: refractive index
For vacuum/air: $n = 1$.

Diffraction limit: Rayleigh criterion:

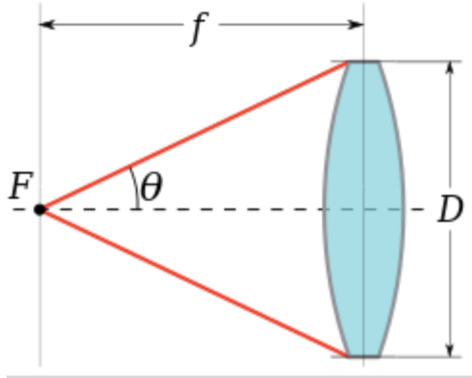
$$Res \geq 0.6\frac{\lambda}{NA} = 0.6\frac{\lambda}{n\sin(\theta)}$$

Spatial resolution

$$NA = n \sin\theta \approx n\frac{D/2}{f}; \quad Res = 0.6\frac{\lambda}{n\,\mathrm{si\,n}(\theta)}$$

	VISUAL LIGHT	ELECTRONS	X-RAYS
WAVELENGTH	500 nm	0.03 nm	0.1 nm
NA	Up to 1	0.01	Towards 0.01
RESOLUTION	500 nm	0.3 nm	Towards 10 nm

Spatial resolution



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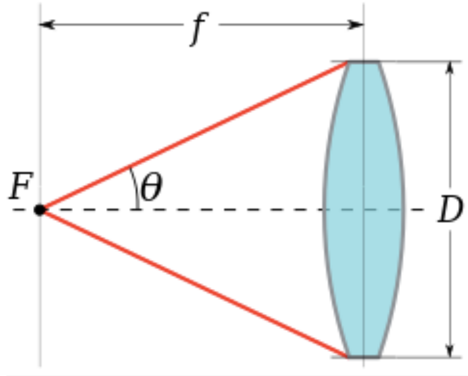
NA	Up to 1	0.01	Towards 0.01
RESOLUTION	500 nm	0.3 nm	Towards 10 nm
	Grains	Atoms	Domains

Spatial resolution



$$NA = n\sin\theta \approx n\frac{D/2}{f};\; Res = 0.6\frac{\lambda}{n\sin(\theta)}$$

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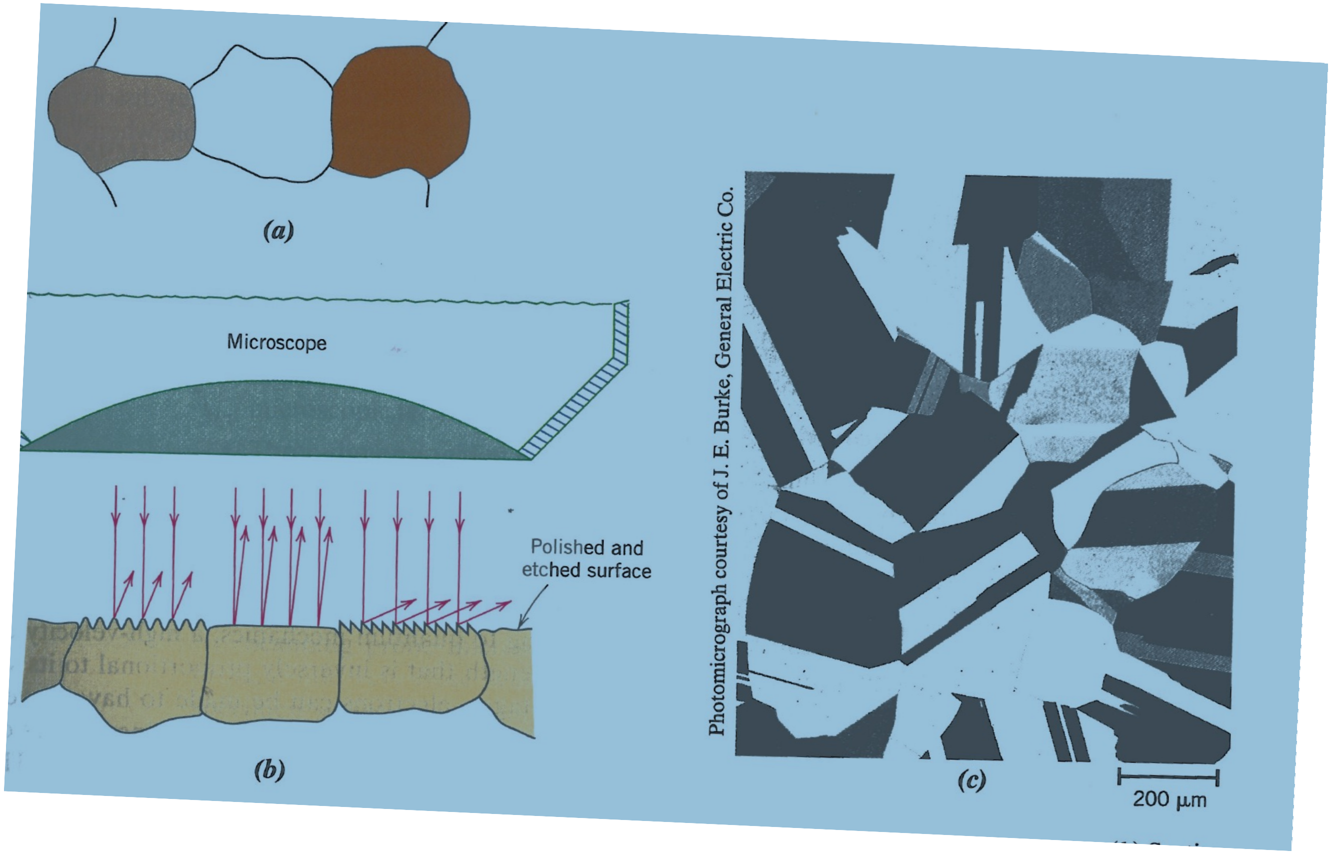
NA	Up to 1	0.01	Towards 0.01
RESOLUTION	500 nm	0.3 nm	Towards 10 nm

Grains | Atoms | Towards domains

Constrast: Light microscope

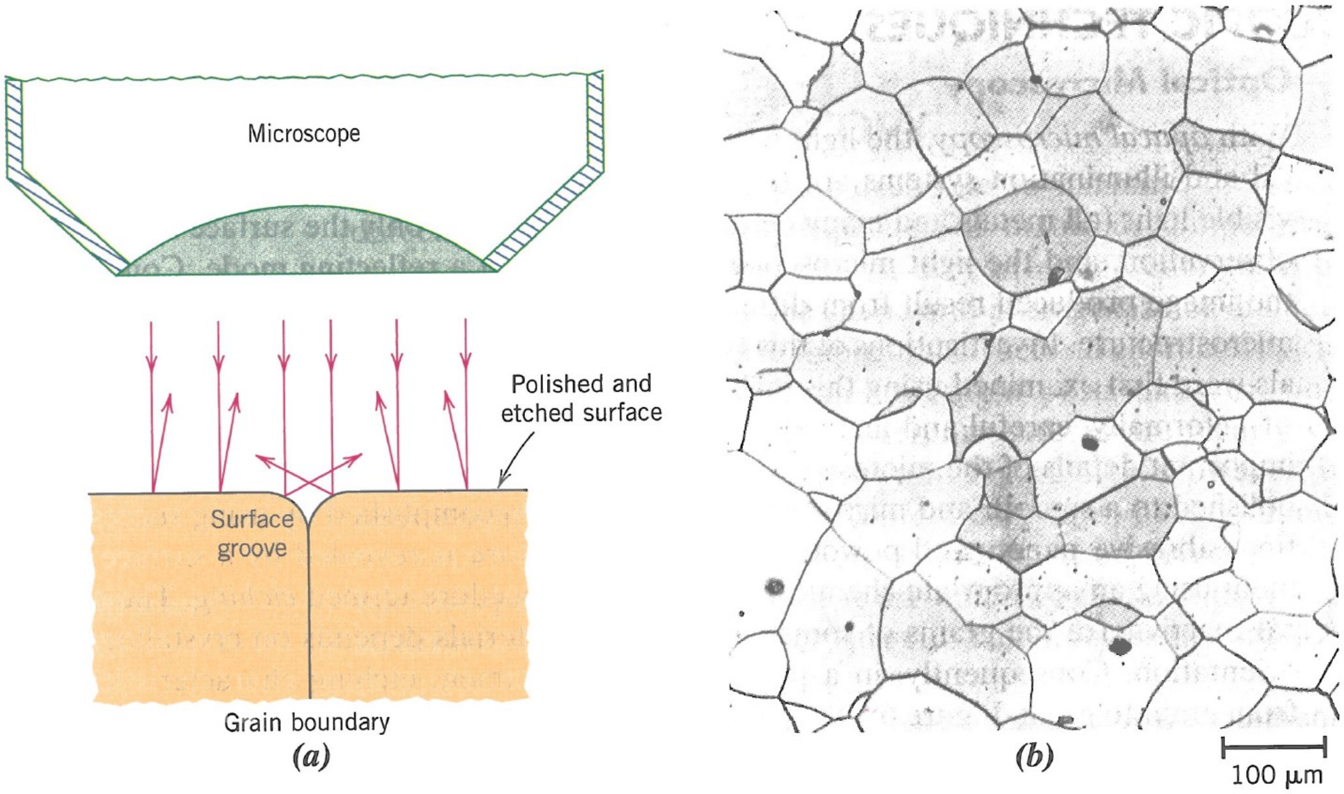
Polish + etch:



See: difference in orientation

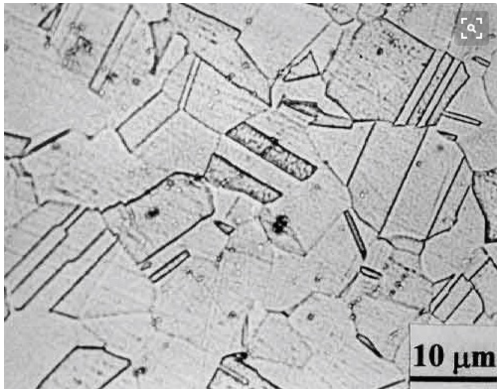
Constrast: Light microscope

Polish + etch:

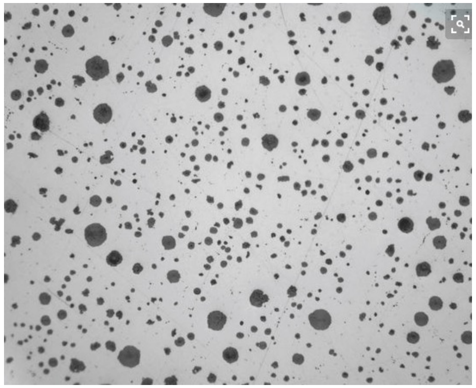


See: grain boundaries

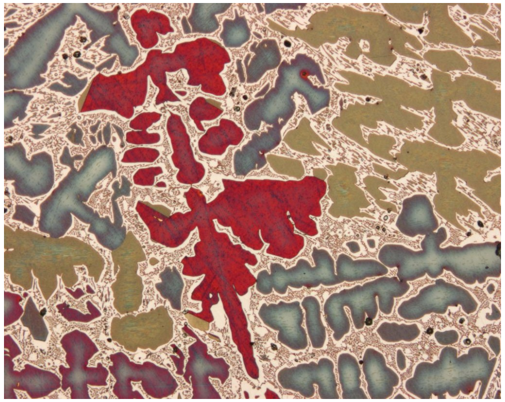
Microstructures….



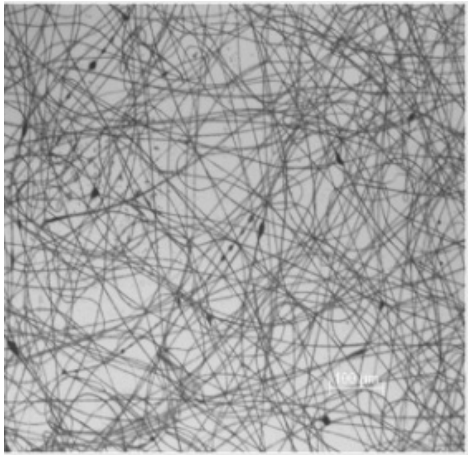
Type 304 stainless steel



Cast steel

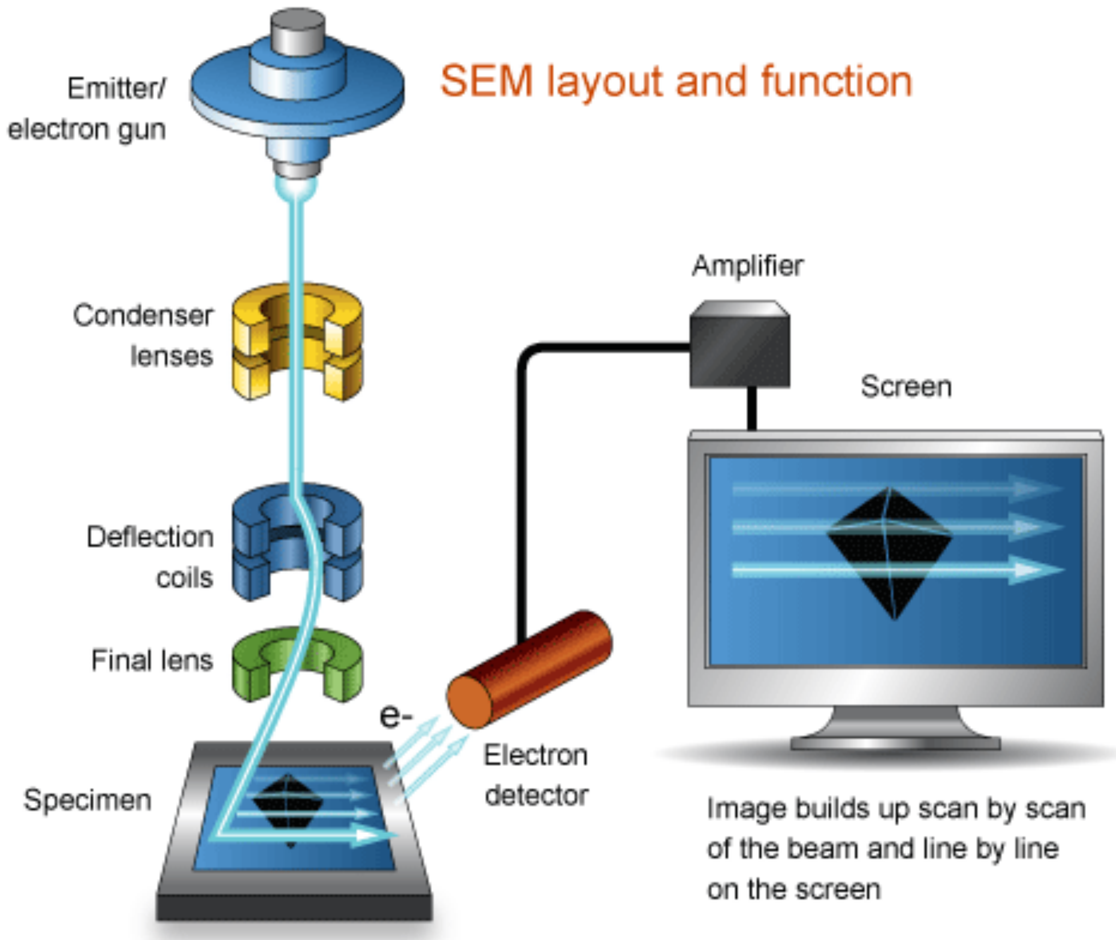


Cast bronze



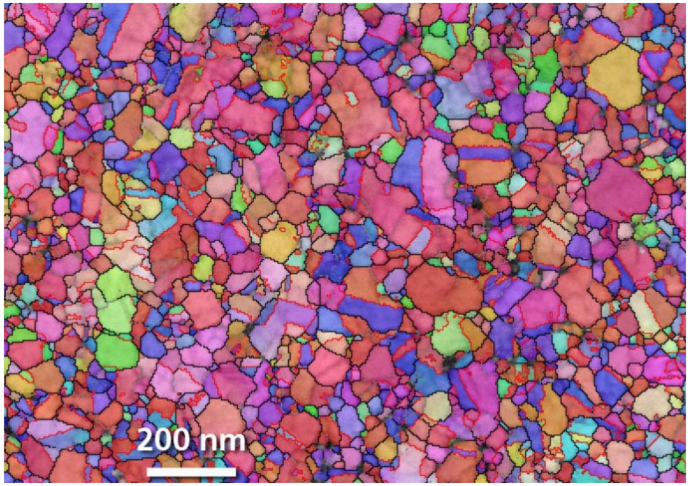
Polymer strings

Scanning Electron Microscopy (SEM)

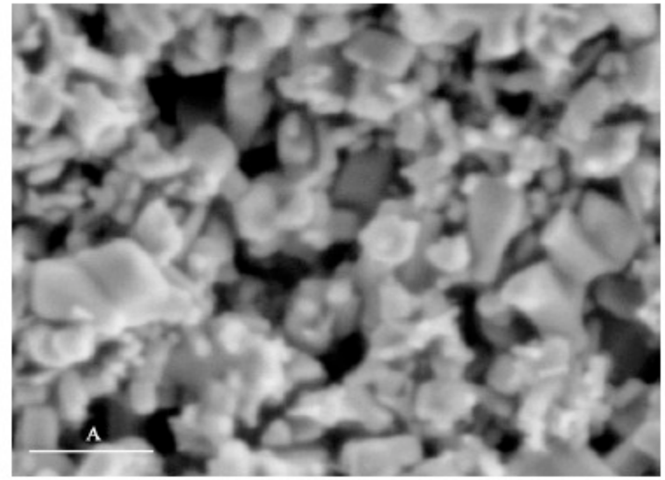


Typically in vacuum

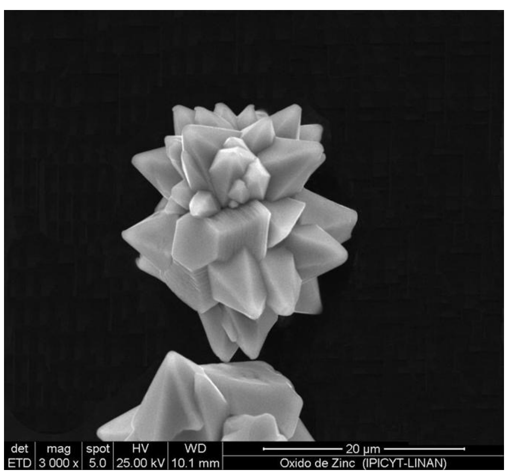
Contrast, SEM



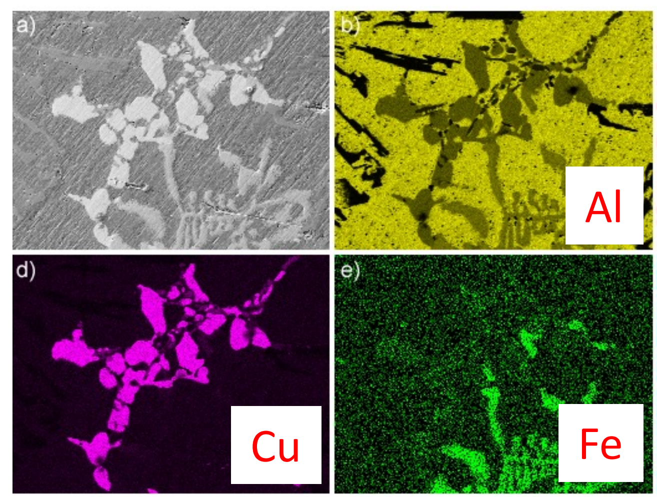
Nano-size grains



Porous structures



Small stuff on surface

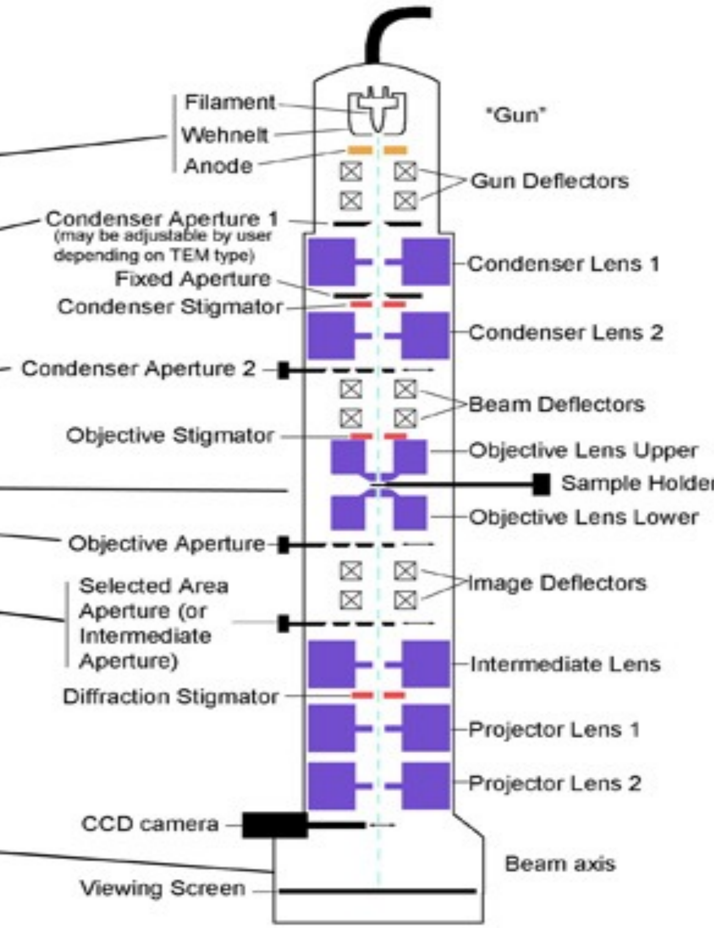


Chemical map of Al alloy

Transmission electron microscope (TEM)

One of many types of TEMs

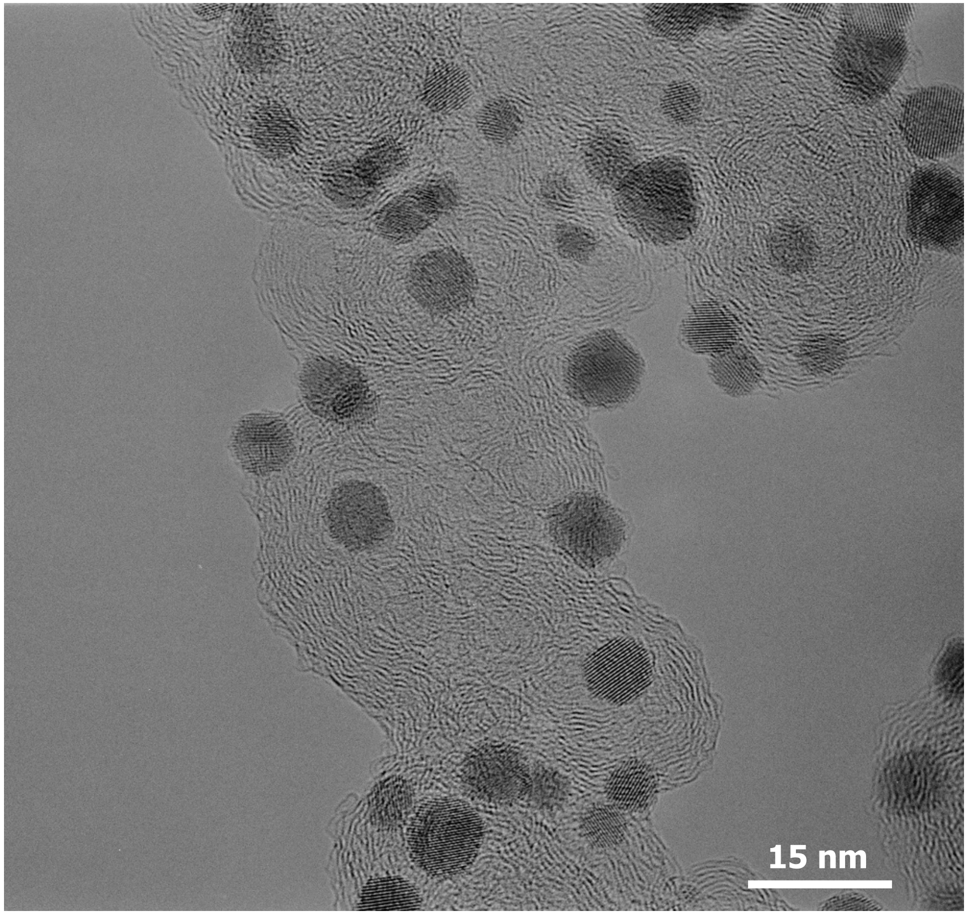
Example TEM Schematic

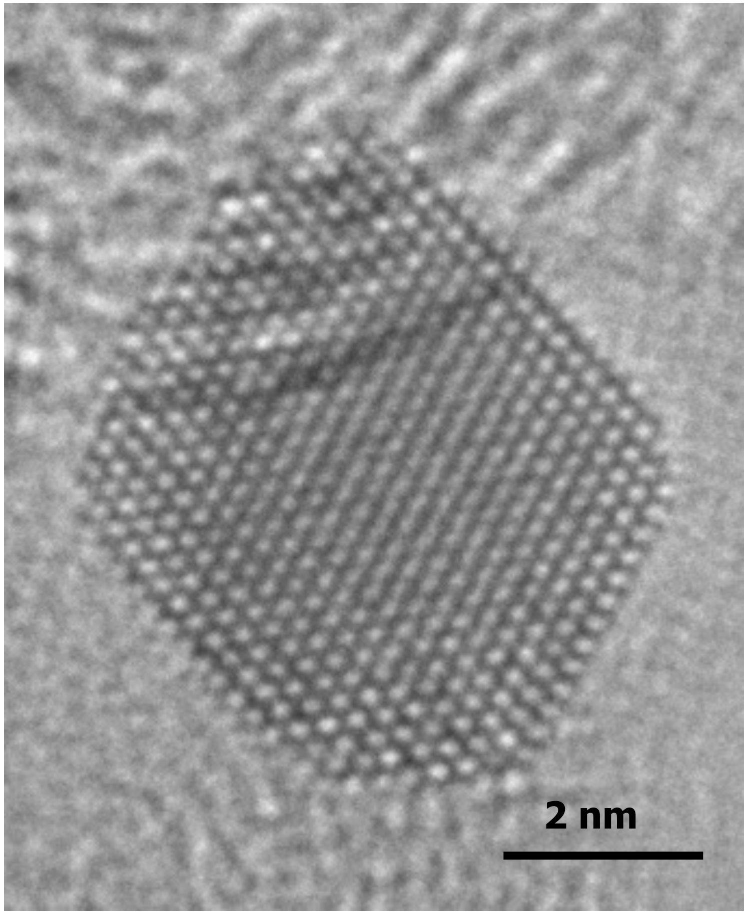


Always in vacuum

Contrast,TEM

Data from DTU Nanolab:





Why x-rays ?

MAX IV, Lund :

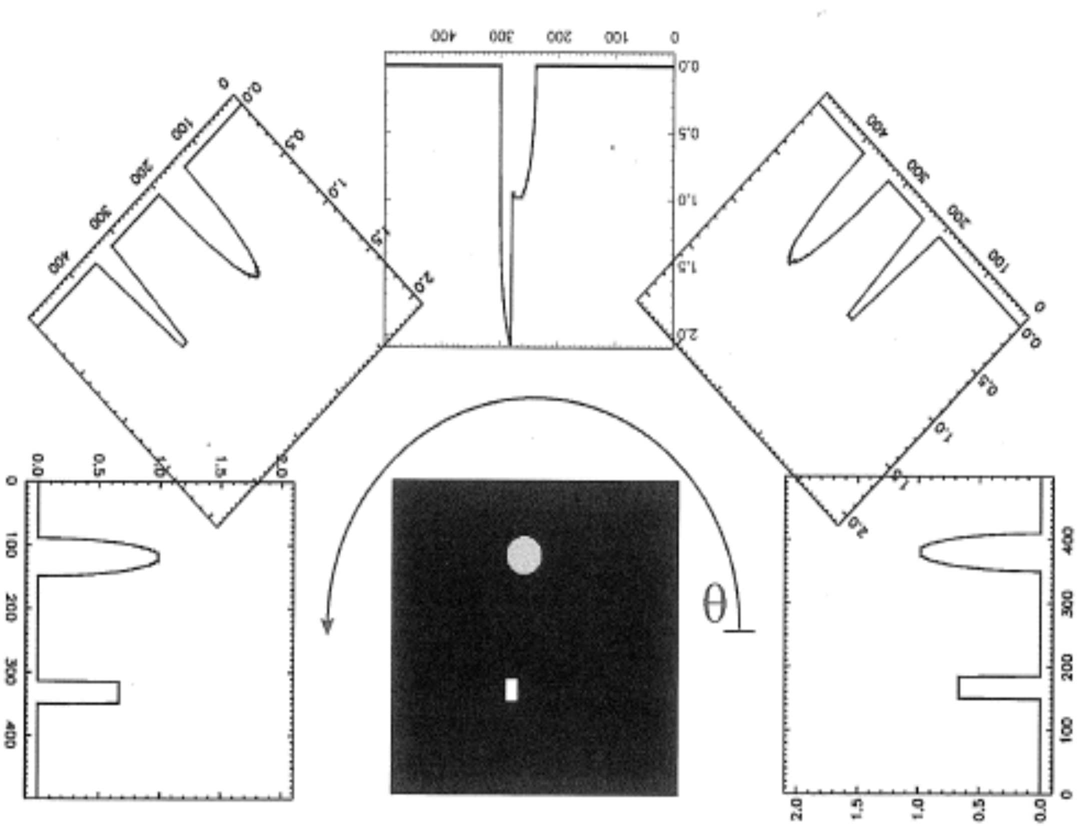
X-ray radiography

Wilhelm Conrad Röntgen discovered X-ray November 8 in 1895

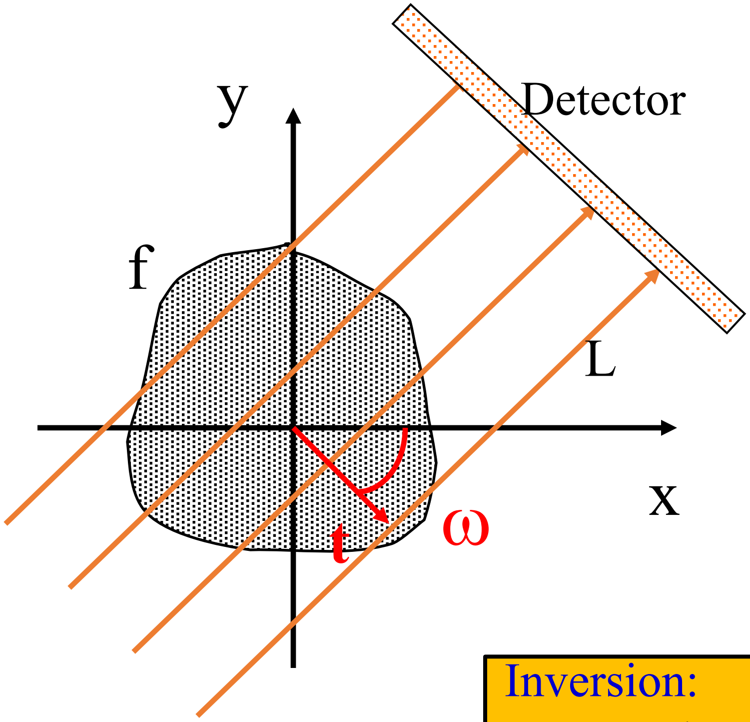
Wilhelm Röntgen

Frau Röntgen´s left hand

Tomography: the principle



Is there a solution?



$R(f) = \int_L f(x,y)\, ds$

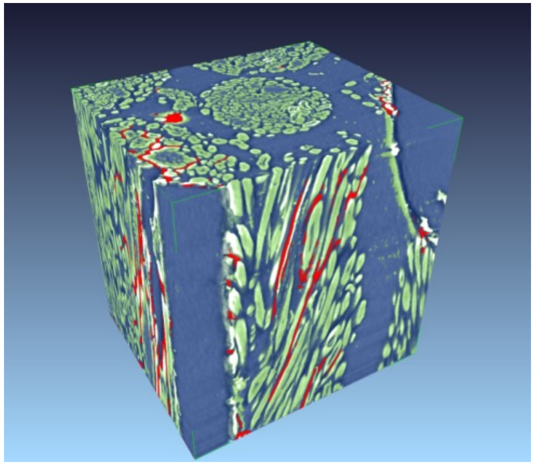
Radon Transform: $(x,y) \rightarrow (t, \omega)$

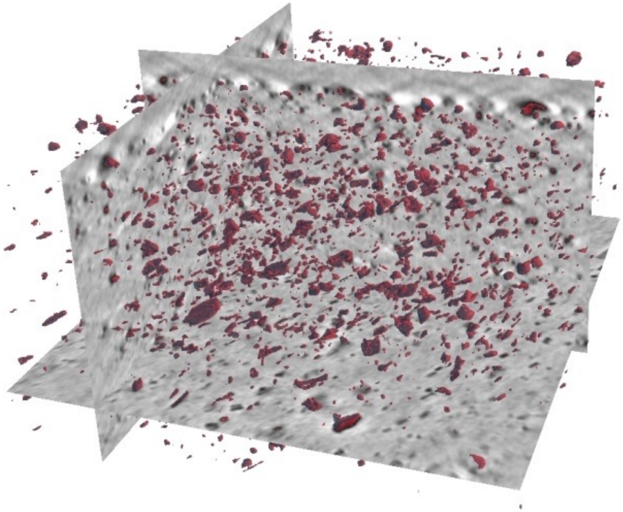
Inverse transform $(t, \omega) \rightarrow (x,y)$

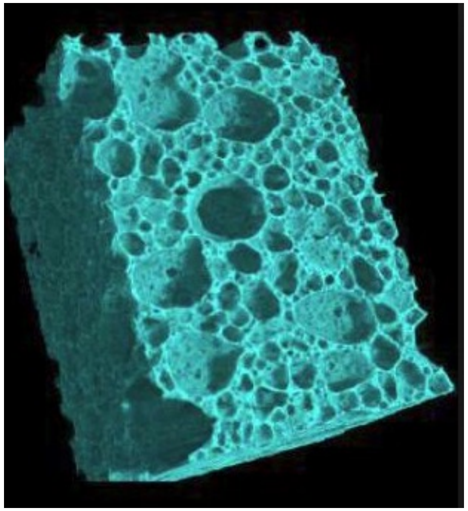
Inversion:
1917: Johann Radon: *On the determination of functions from their integrals along certain manifolds.*

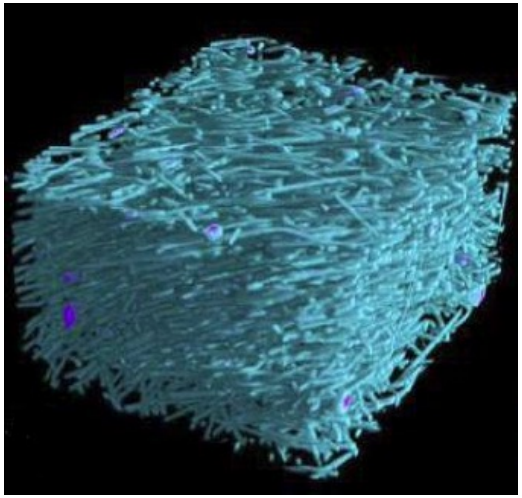
Computer–aided Tomography (CAT):
1979: Nobel price to A. Cormarck and G. Hounsfield

3D structures

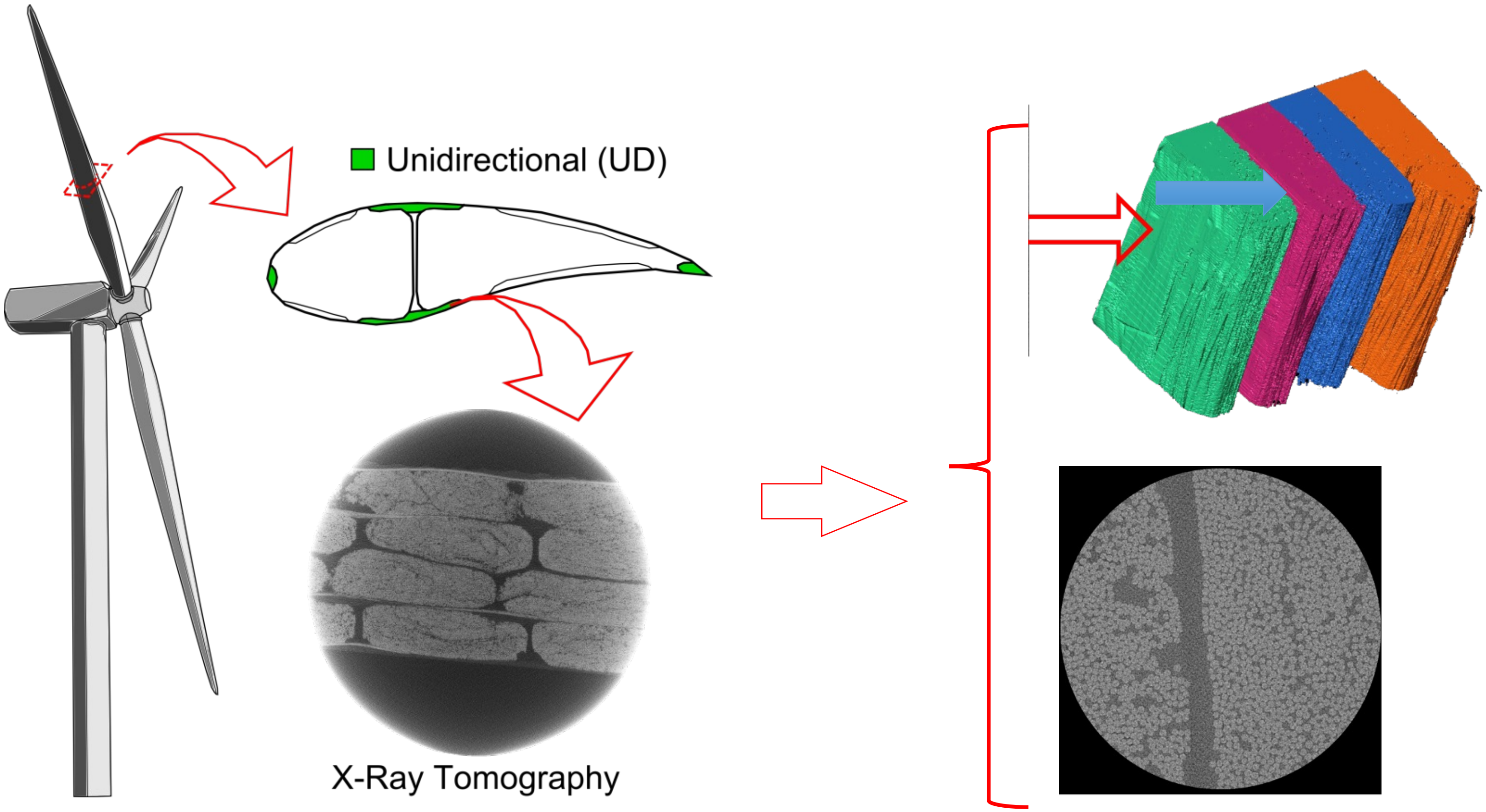






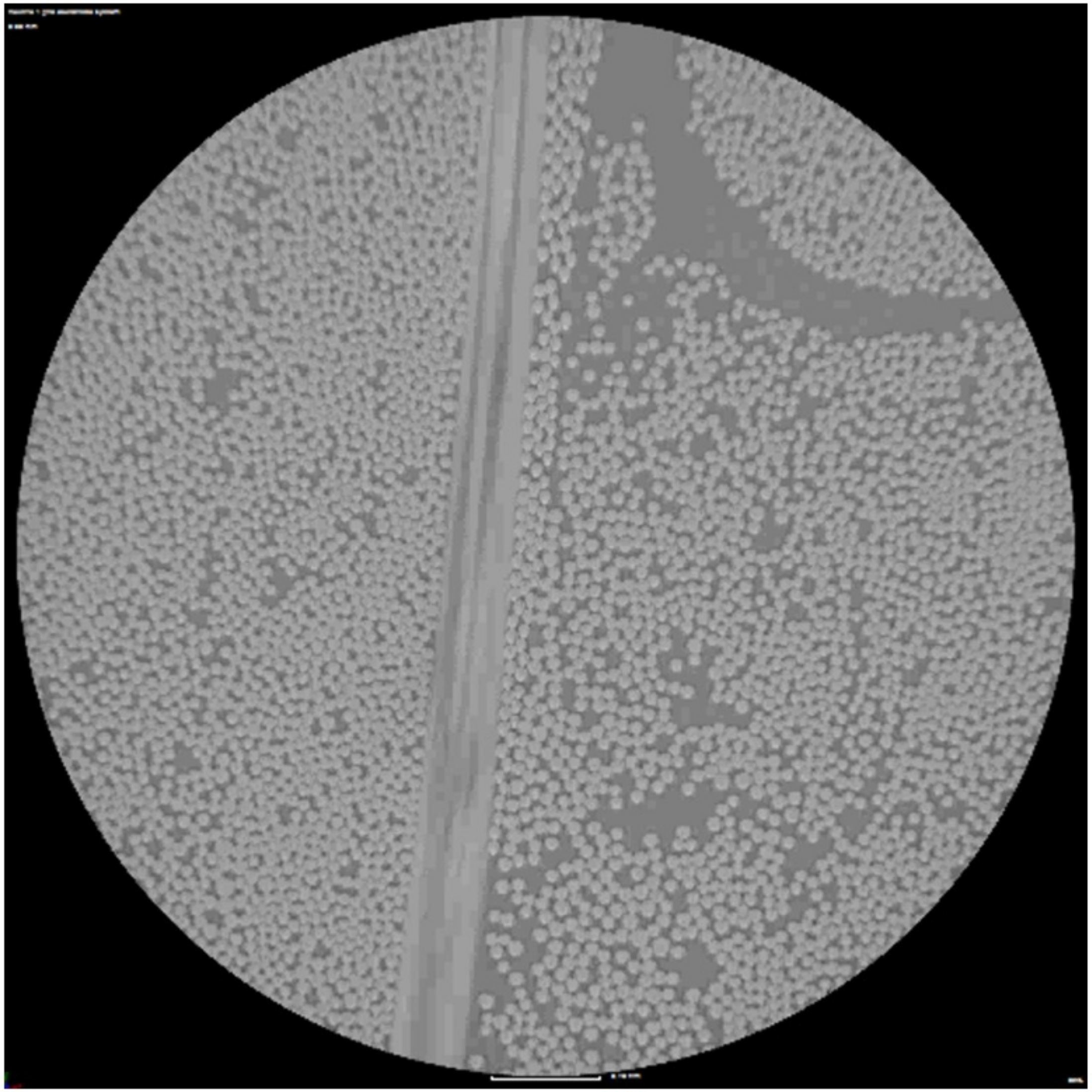


Crack evolution in fibre composites for wind turbine blades



In situ study of fiber failure

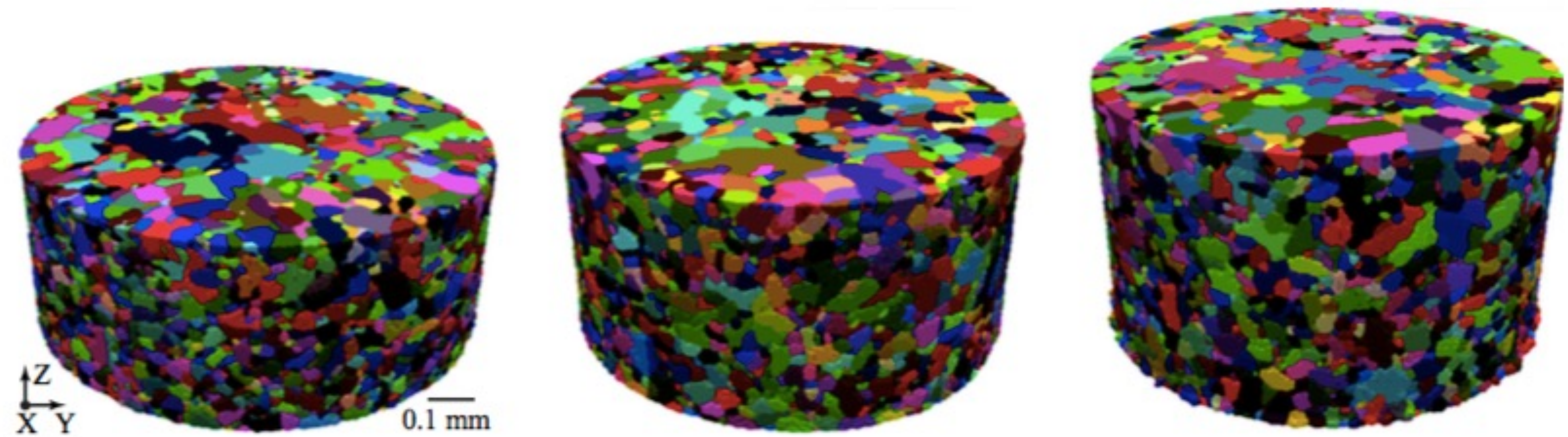
- Sample: 3mm x3mm x20mm
- Resolution ~1 micron



Slide from Lars Pilgaard Mikkelsen, DTU Vind

3D mapping of grains

Cu specimen with 20,000 grains during tensile deformation



0% 6% 12% deformation

Resolution: 1-2 μm

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R. Pokharel *et al. Internat. J. Plasticity* **67,** 217–234 (2015)

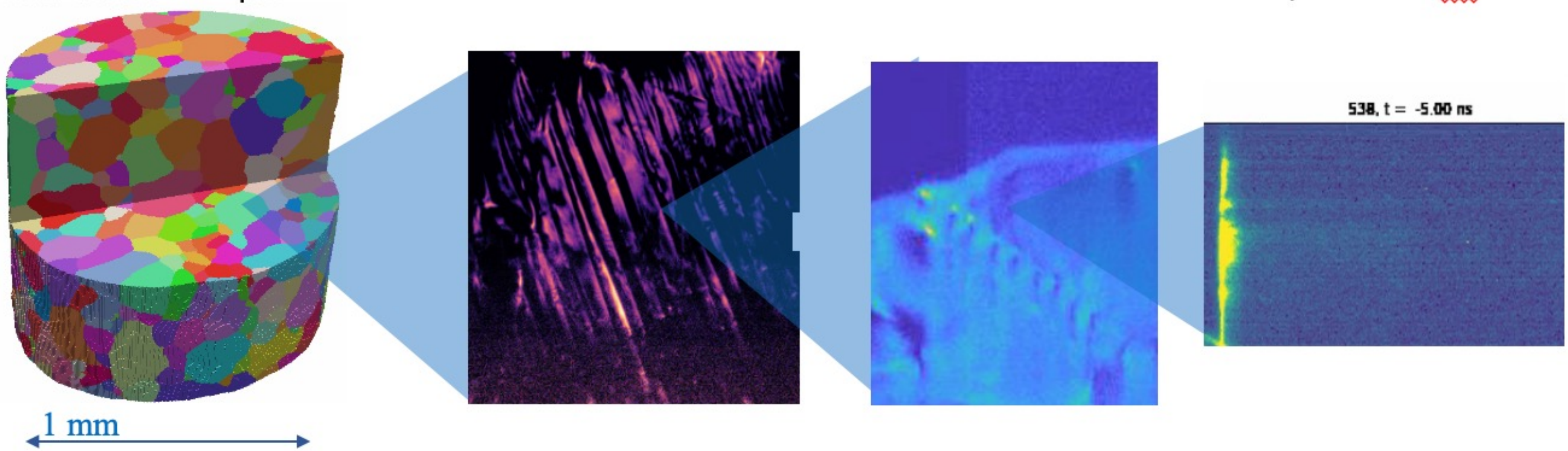
Understanding metals using a multiscale 3D x-ray microscope

3D Grain movie
Resolution: 2 µm

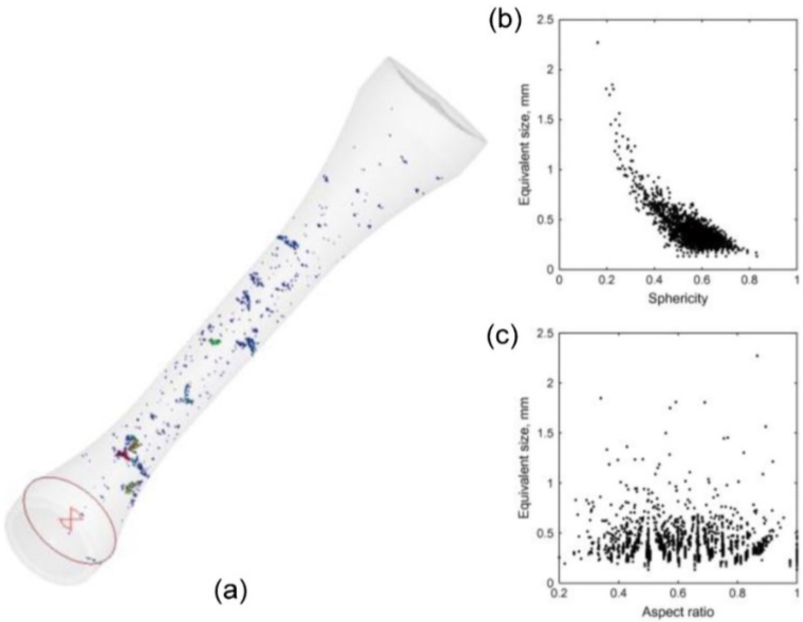
3D domain movie
100 nm

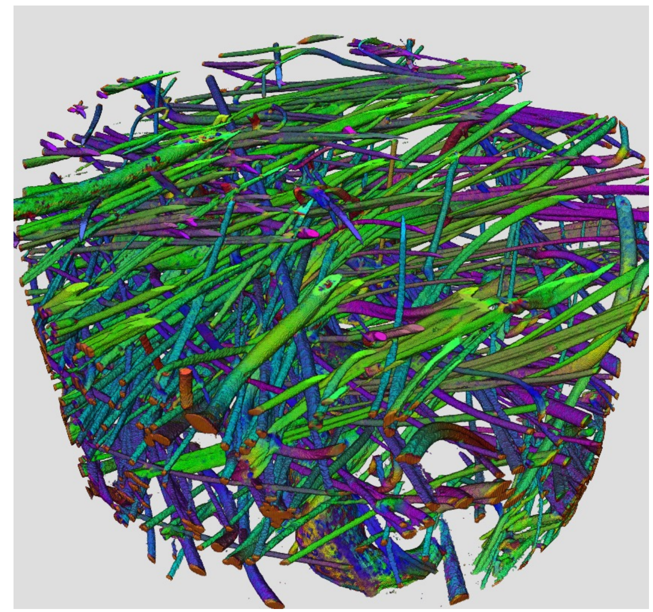
3D defect movie
so far 30 nm

Phonons in 3D
1 µm but 10 ps

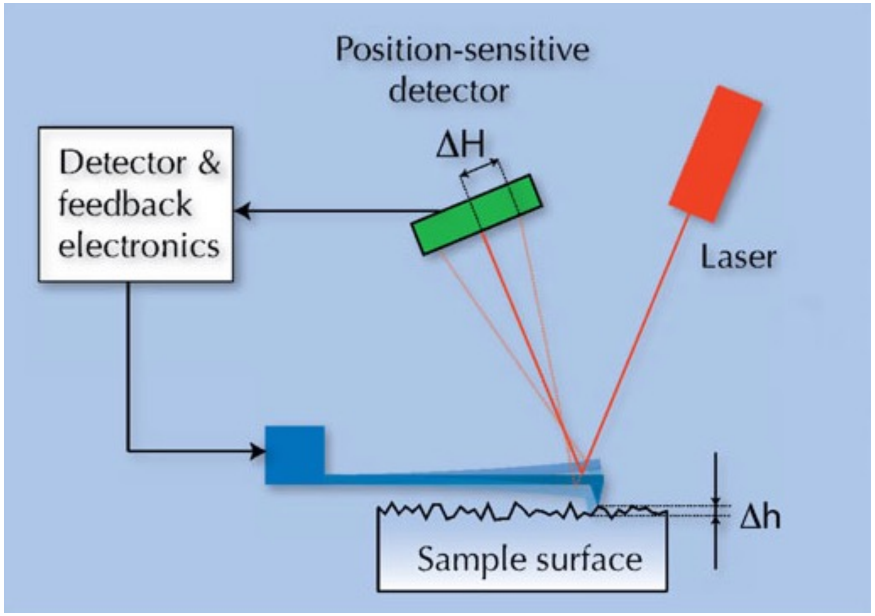


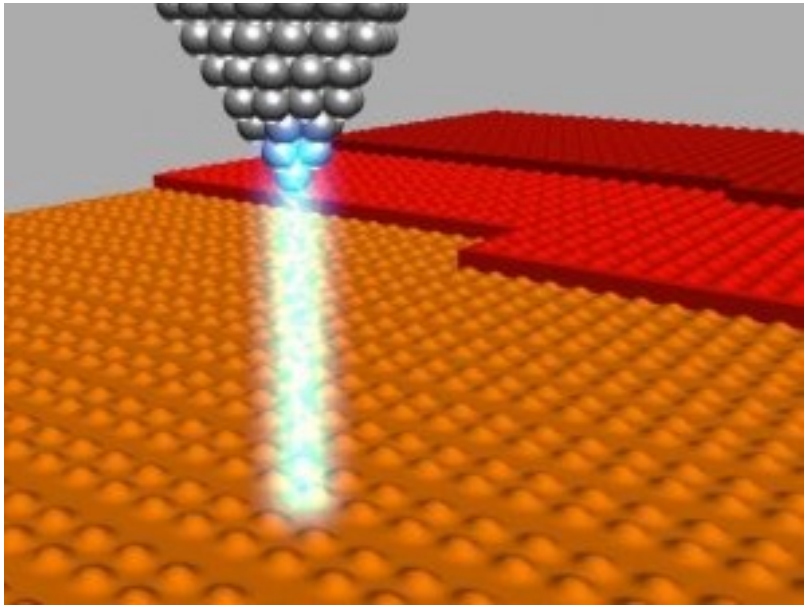
Quantification





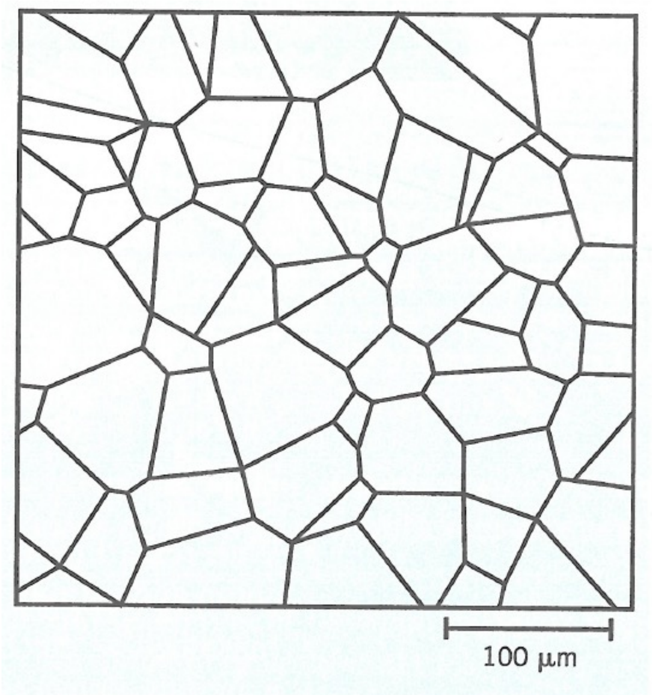
Scanning probe microscopy





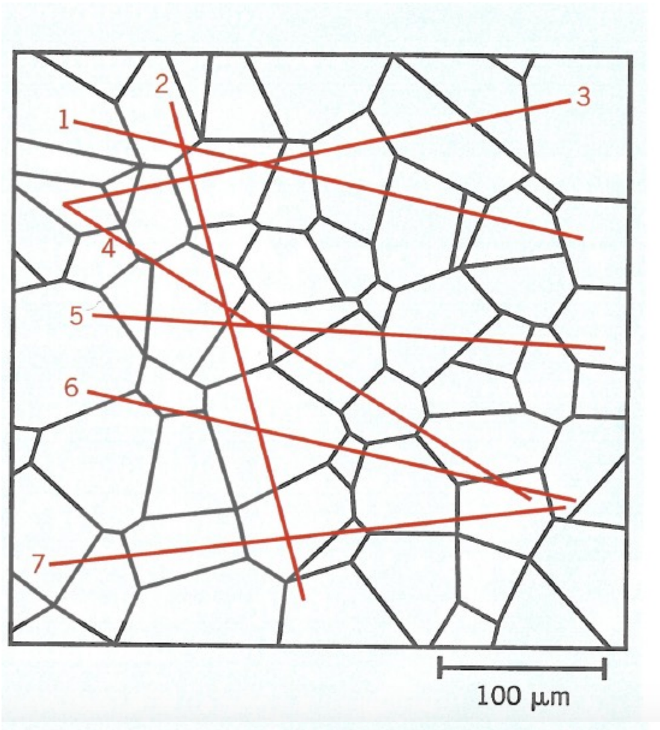
Mapping the surface: atom positions and forces
NOT diffraction limited

Determining grain sizes



How to do this?

Determining grain sizes



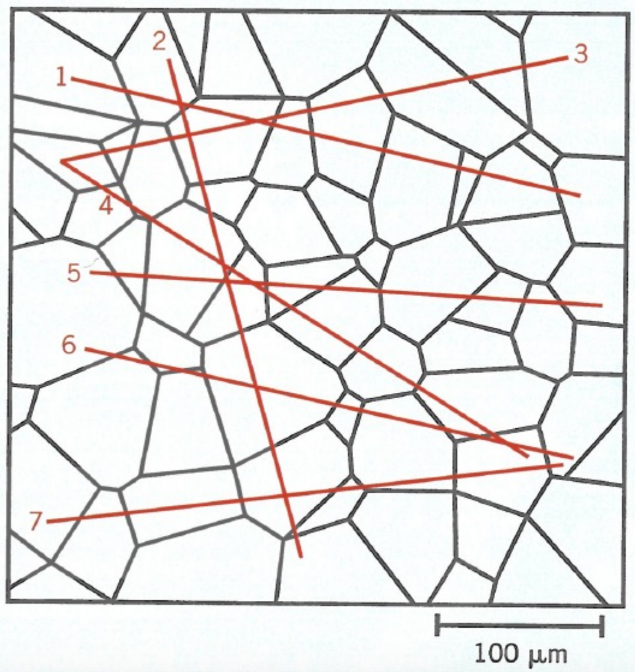
How to do this?

Mean intercept length, $\bar{l}$:

- Draw N random lines
- Determine total length L_T
- Determine total #intercepts, P
- Determine magnification, M

$$\bar{l} = \frac{L_T}{P\ M}$$

Determining grain sizes



M =   160;

L_T = 7* 50 mm/line = 350 mm

Line Number	*Number of Grain-Boundary Intersections*
1	8
2	8
3	8
4	9
5	9
6	9
7	7
Total	58

$$\bar{l} = \frac{L_T}{P\,M} = \frac{350\ mm}{58*160} = 0.0377\ \text{mm}$$

Imaging of materials

Methods

Diffraction	Microscopy	Tomography	Scanning Probe
Crystal	-------------------No need for crystal--------------------------		
Braggs law	Diffraction limit Lens equation	Radon Transform	Cantilever

	LIGHT	ELECTRONS	X-RAYS	SCANNING PROBE
RESOLUTION	500 nm	0.3 nm	10-100 nm	0.1 nm
DEPTH	None	100 nm	1 mm	None
FIELD OF VIEW	Large	Small	Medium	Small
DIFFACTION	-	+	+	-
ENVIRONMENT	All	Vacuum	All	All
ACCESS	Lab	Uni center	Uni center or Synchrotron	Lab