Electron Microscopy

NanoDefine Workshop
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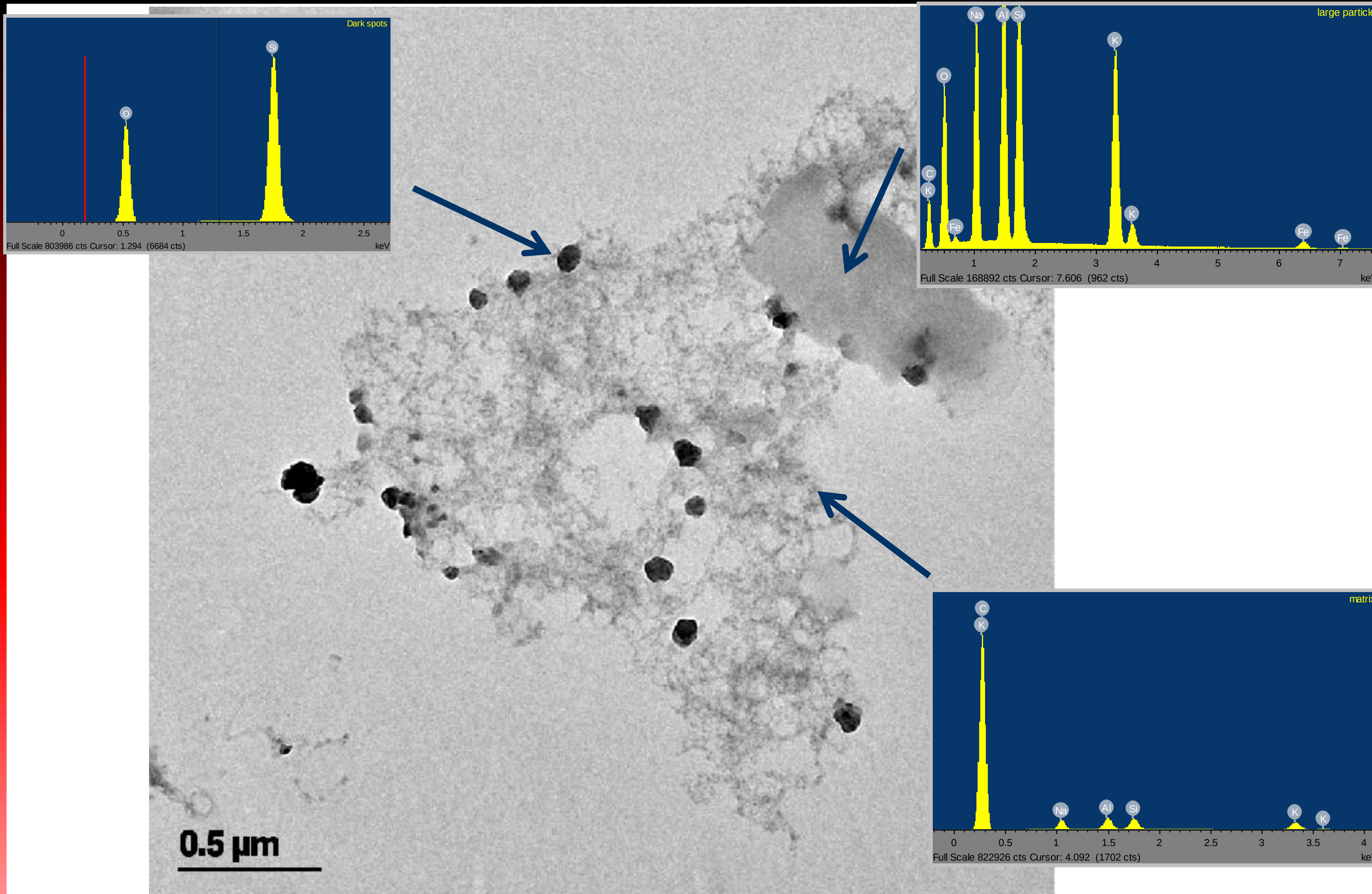
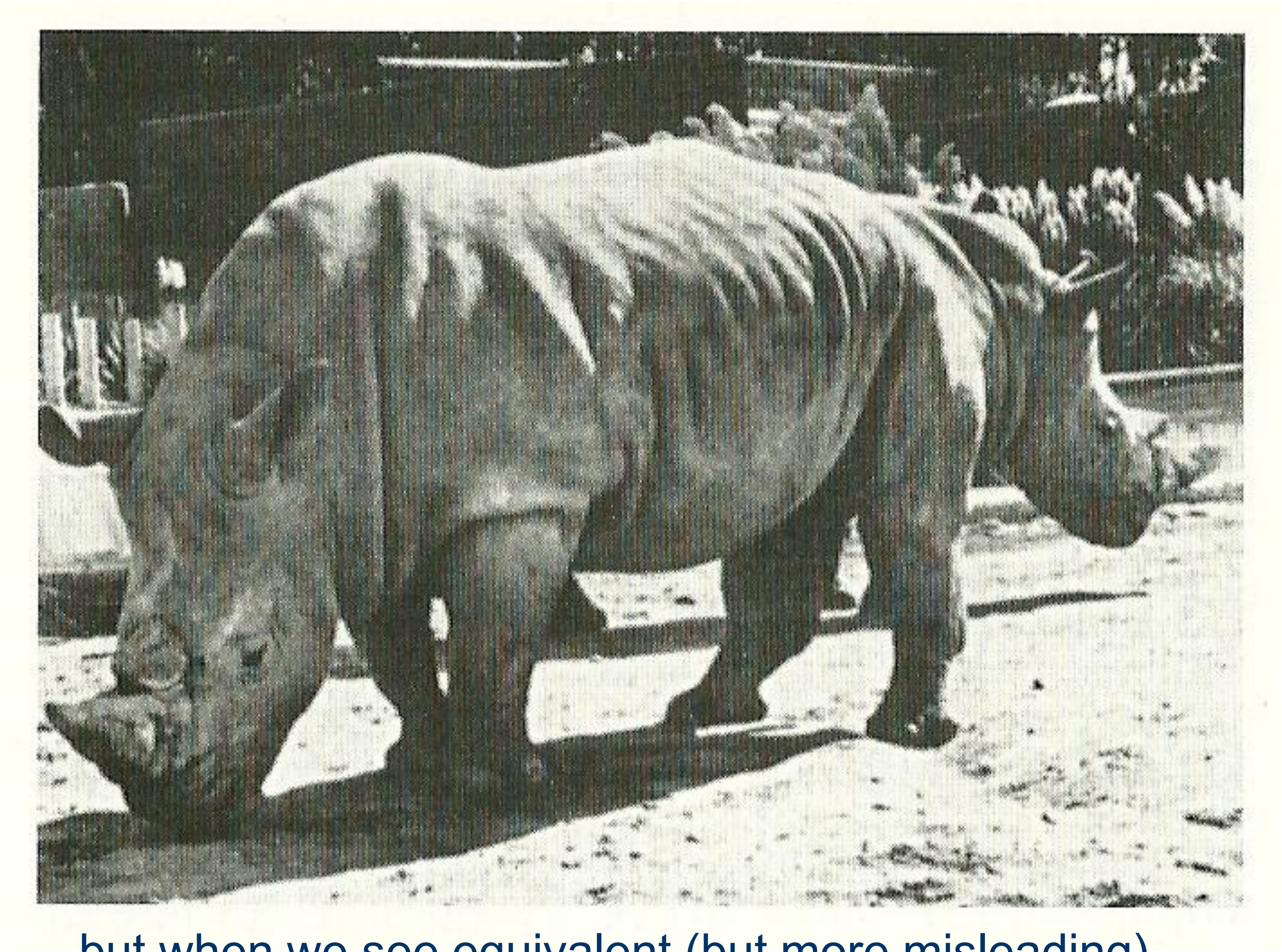


Electron microscopy can resolve structures, spatial arrangements from the **mm** down to the **nano**-scale

However, as stated by Hayes (in Williams and Carter, 1996): When we this image we laugh (because we understand its true nature in 3D)…

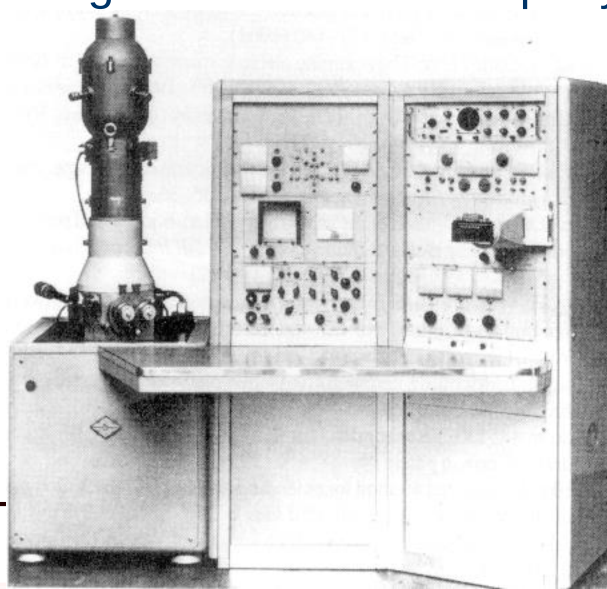


… but when we see equivalent (but more misleading) images in the TEM, we publish!

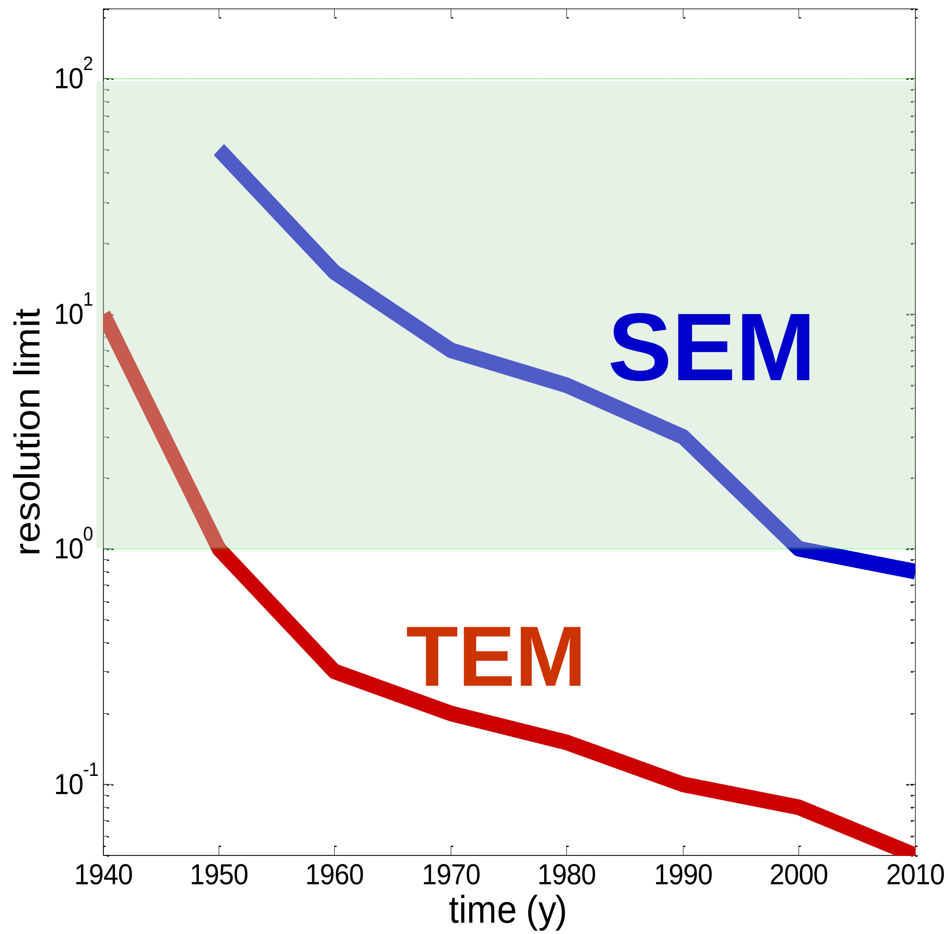
Current resolution limits

first electron microscope: Knoll and Ruska (1932)

Prototype of the first Stereoscan (Cambridge Instruments Company).



The increase in resolution



Resolution limit

*The **Rayleigh criterion** is the generally accepted criterion for the minimum resolvable detail - the imaging process is said to be diffraction-limited when the first diffraction minimum of the image of one source point coincides with the maximum of another (Figure 3) (http://hyperphysics.phy-astr.gsu.edu/Hbase/phyopt/Raylei.html).*

$$d = 0.61 \cdot \frac{\lambda}{n \cdot \sin\alpha}$$

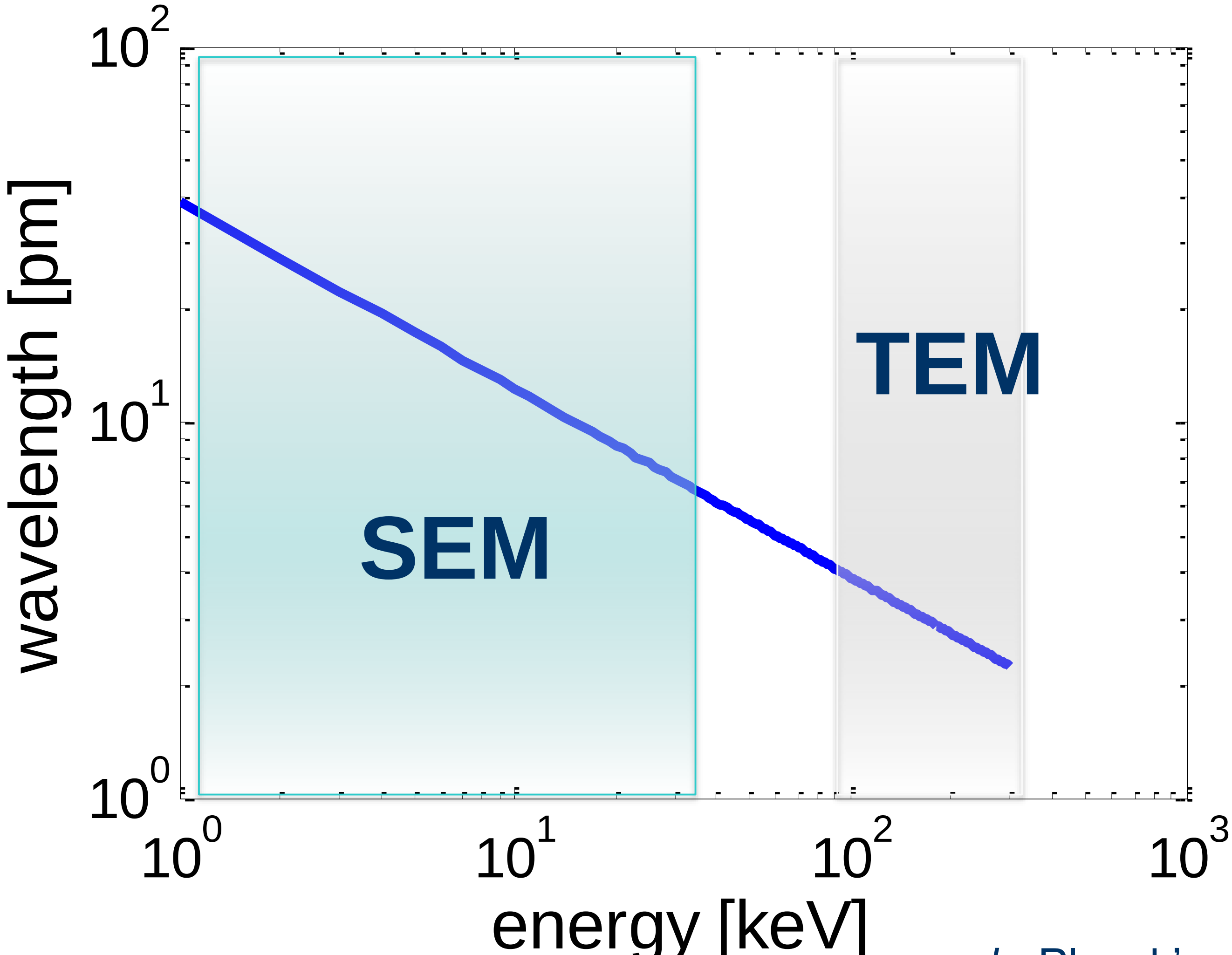
$n \times \sin\alpha$: numerical aperture.
d= resolution, λ = wavelength,
n=refractive index, α=angular aperture.

Typical EM: α 10mrad

Limits of resolution of different, optical systems'

Eye	**0.1 mm**
Light microscope (white light)	**0.2 µm**
UV-microscope	**0.1 µm**
SEM	**1 nm**
TEM	**0.1 nm**

Wavelength of electrons (λ)

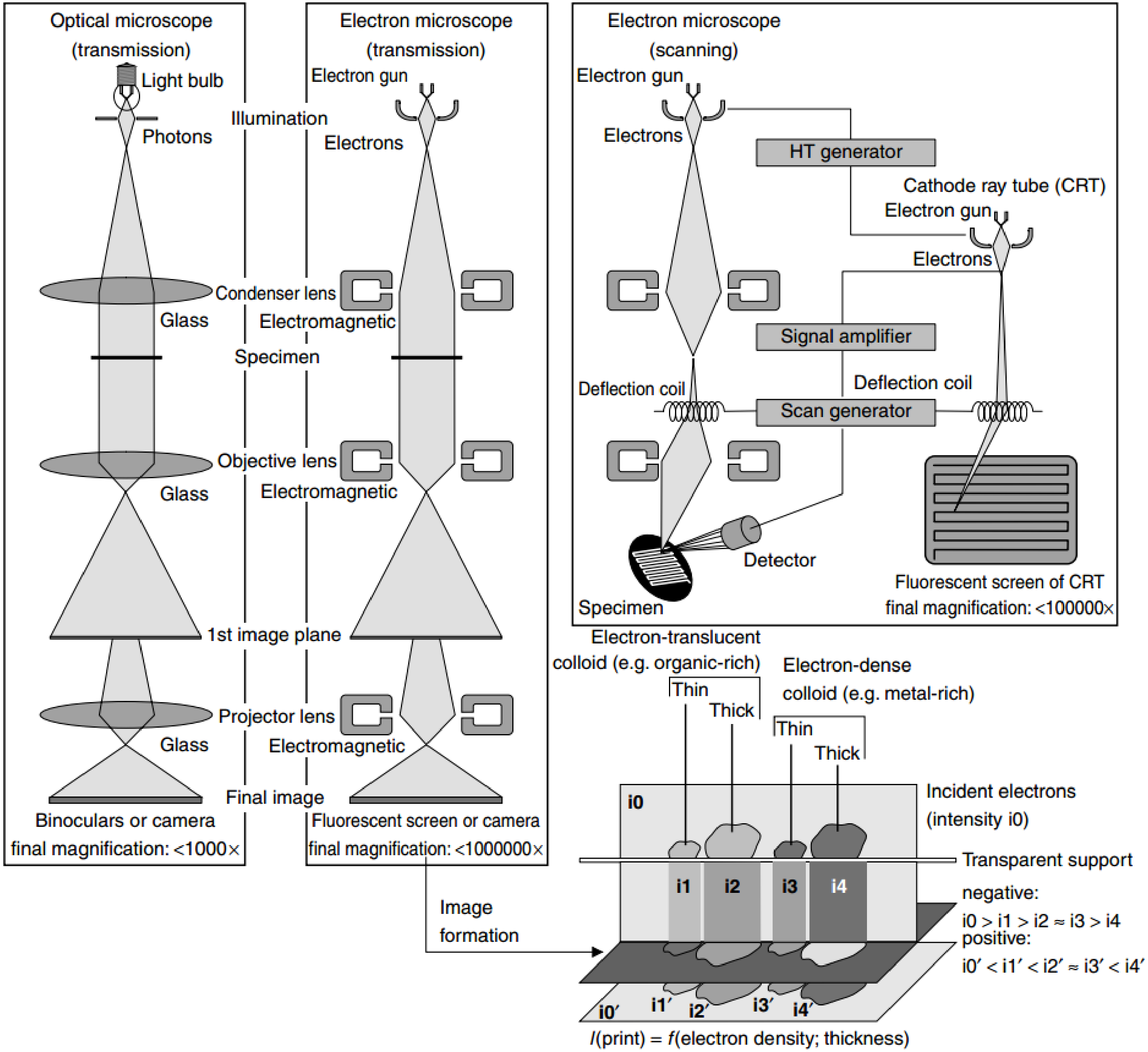


$$\lambda = \frac{h}{\sqrt{2 \cdot m_0 \cdot e \cdot V}} \qquad \lambda \approx \frac{1.22}{\sqrt{V}}$$

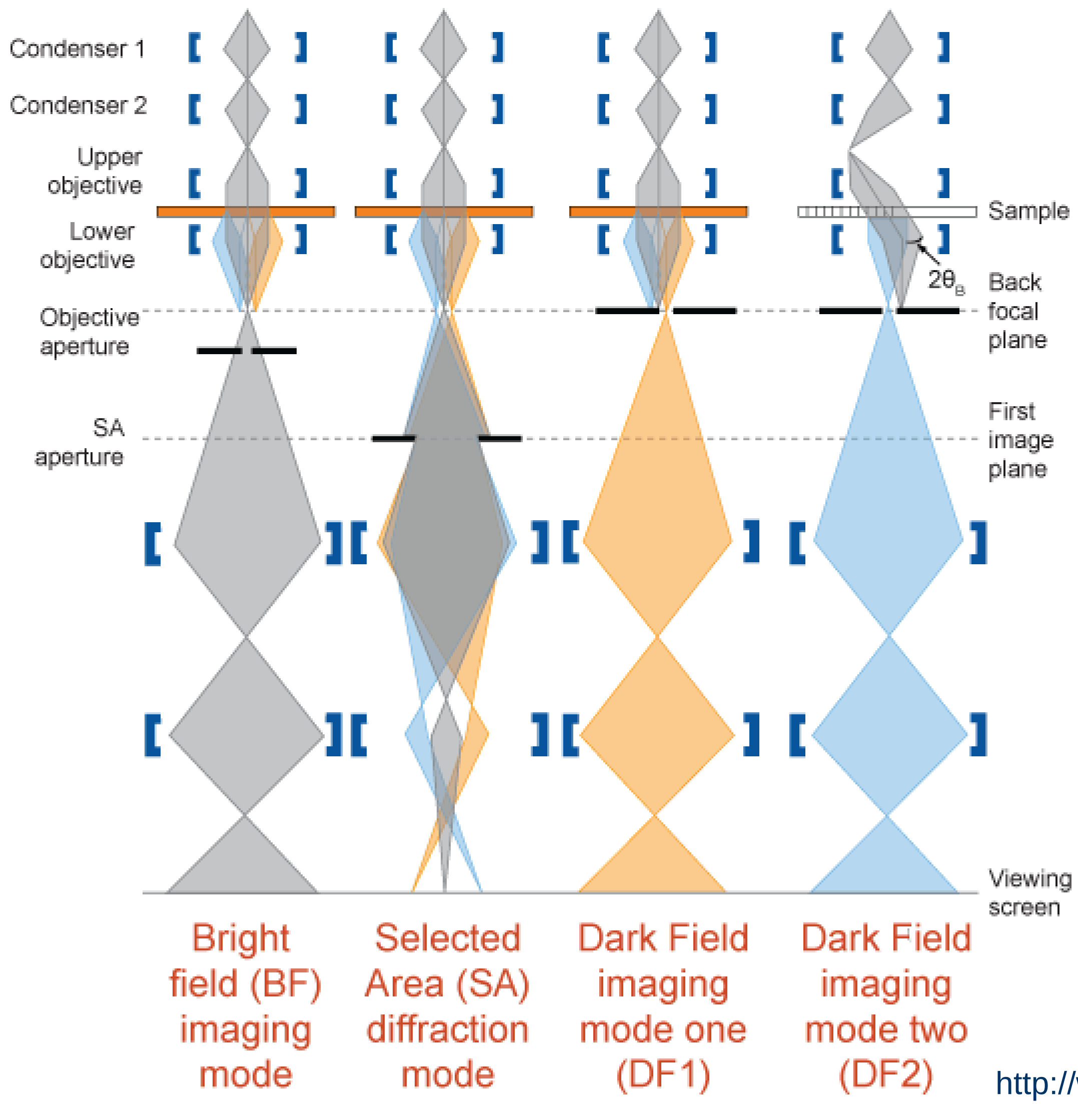
h: Planck's constant
m_0: Rest mass of e^-
E: elementary charge
V: acceleration voltage

The Instruments

Light vs. electron microscope

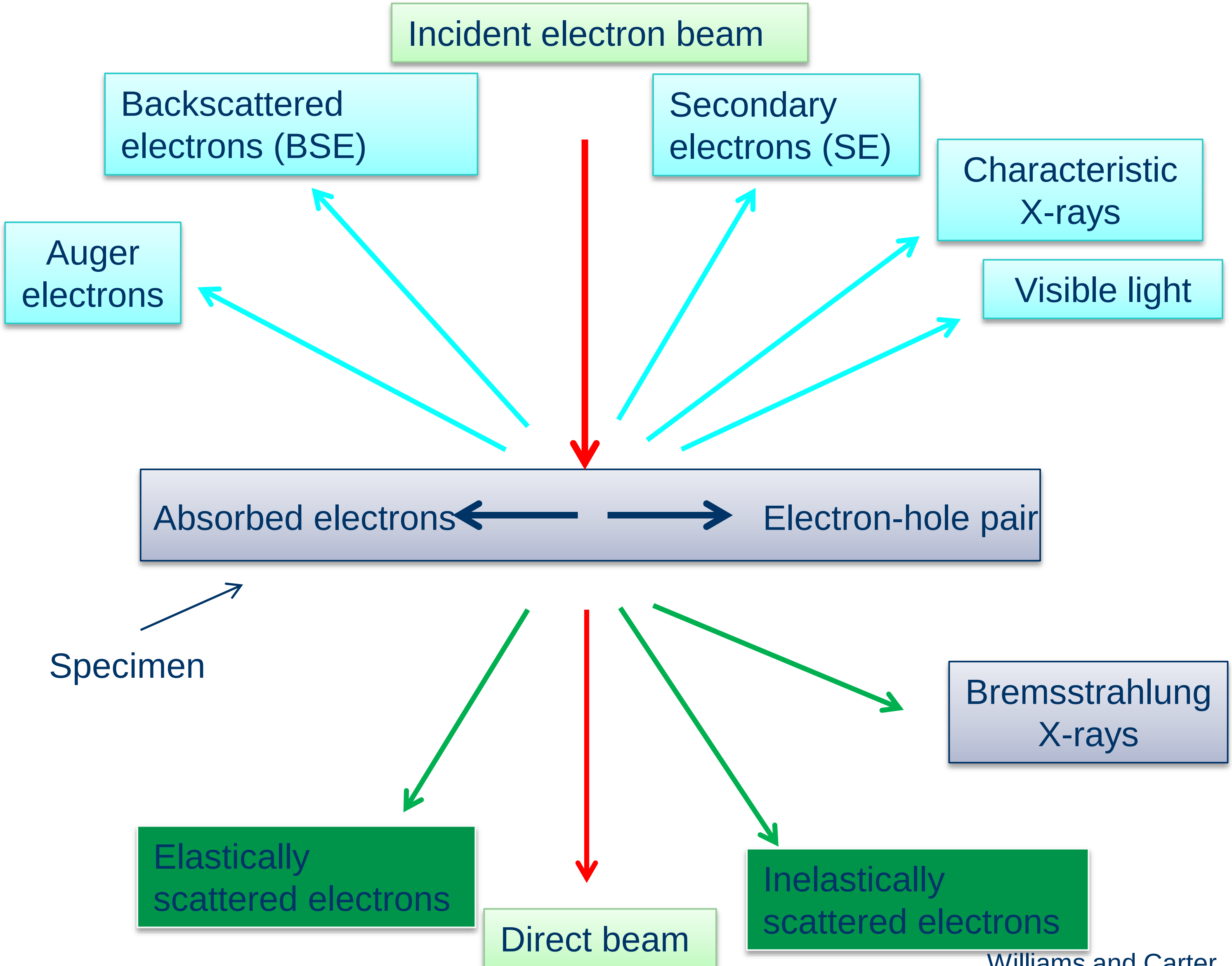


Different imaging modes in the TEM

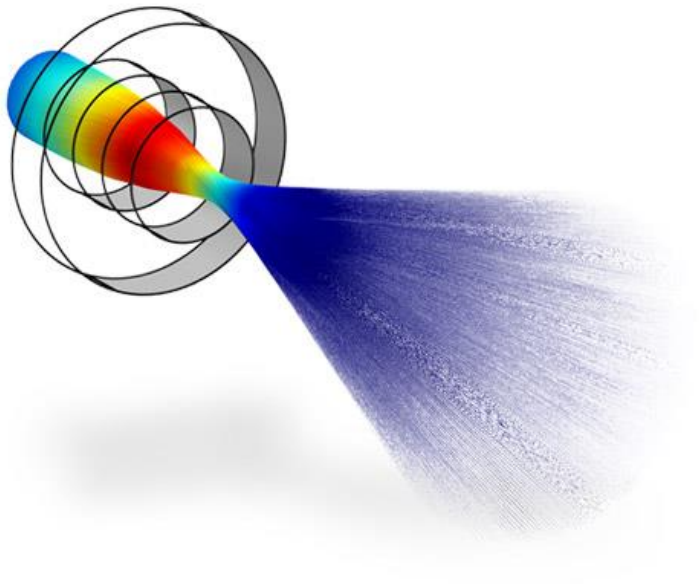


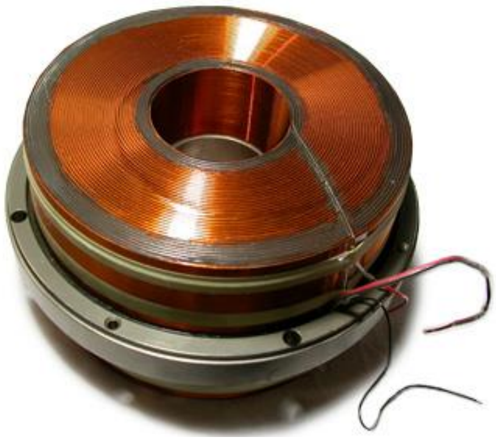
http://www.ammrf.org.au/myscope/

Interaction of the electron beam with the specimen (focused beam, SEM (STEM))

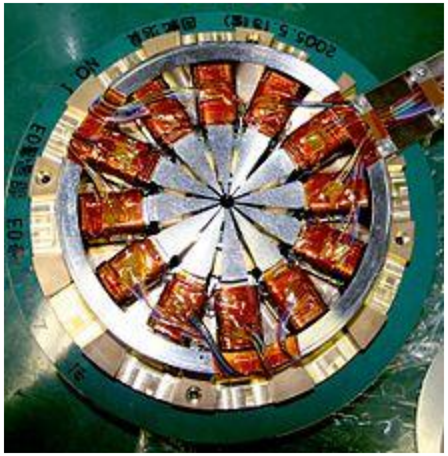


Williams and Carter, 1996

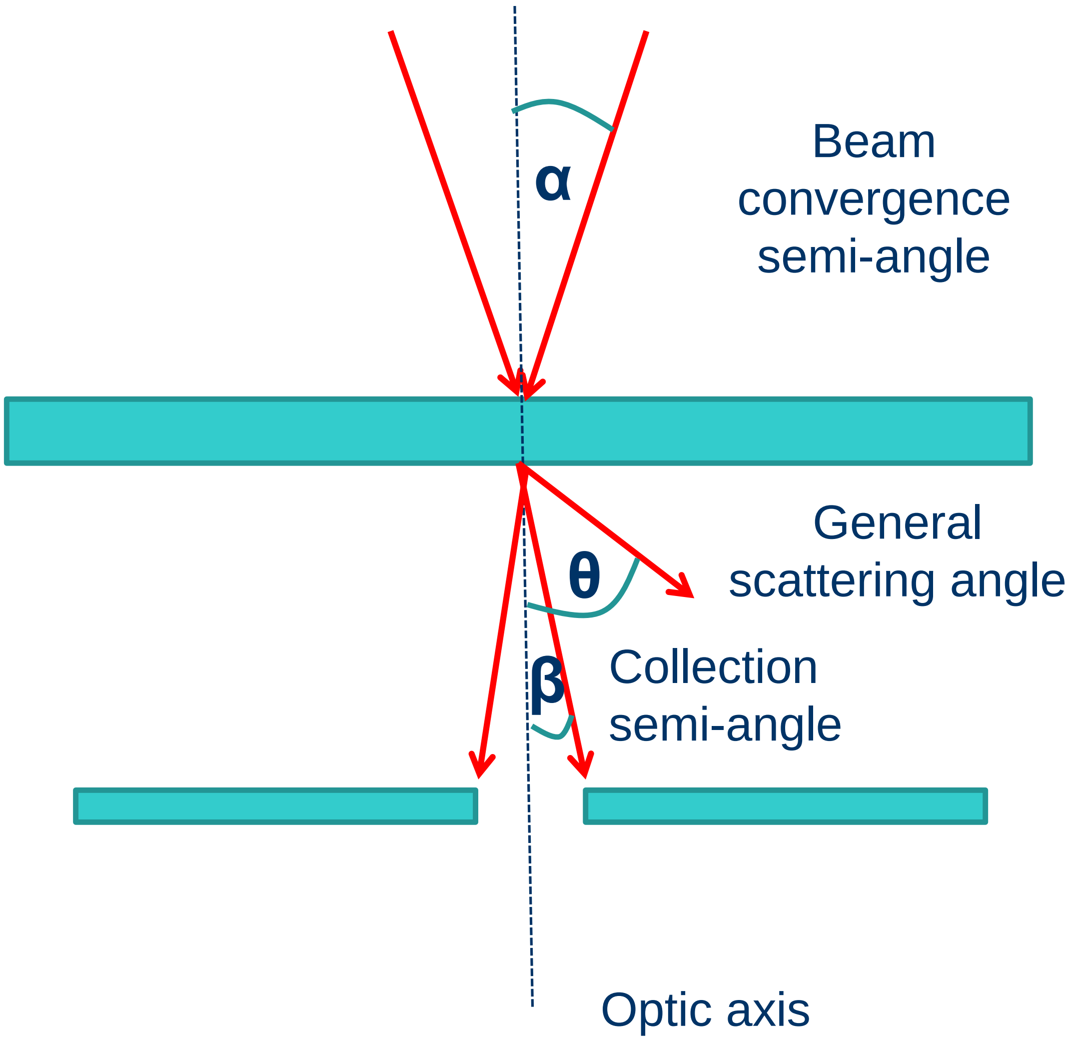




Lenses



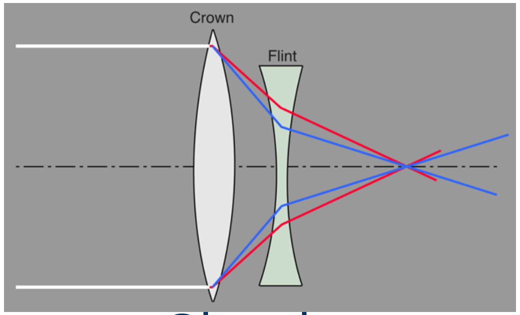
General definition



Lens imperfections -> Aberrations

In optics, a ***circle of confusion***, (also known as ***disk of confusion, circle of indistinctness, blur circle***, etc.), is an optical spot caused by a cone of light rays from a lens not coming to a perfect focus when imaging a point source (http://en.wikipedia.org/wiki/Circle_of_confusion).

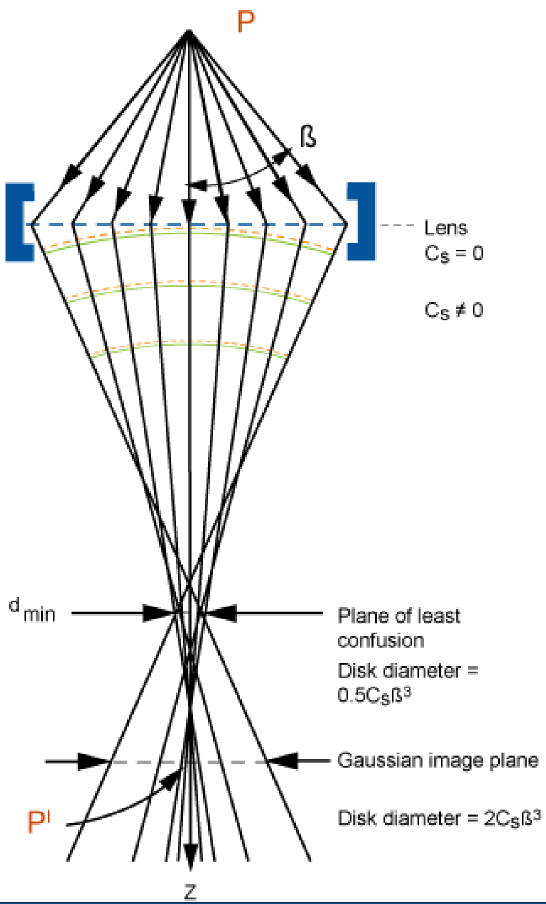
A point will not be transformed into a point but into a disk of finite size. The size / diameter of the disk determines the practical resolution of the EM.



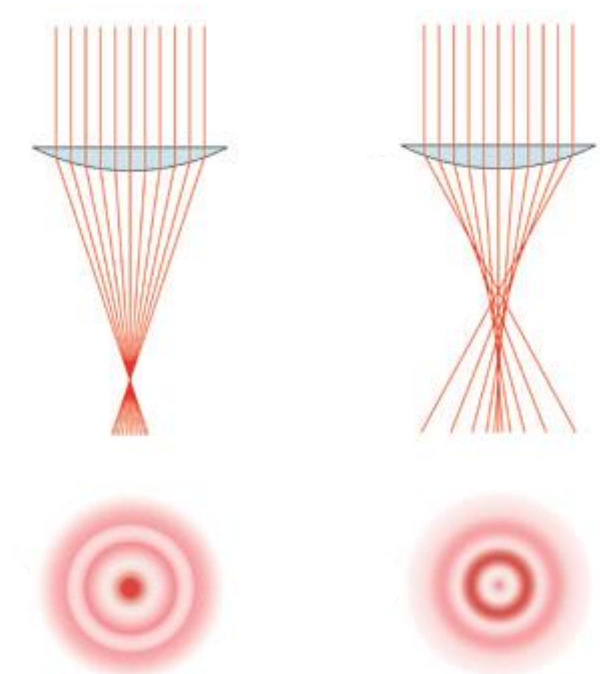
Glass lens

Impossible solution for as EM lenses 'can' only focus

Spherical aberration (C_s)



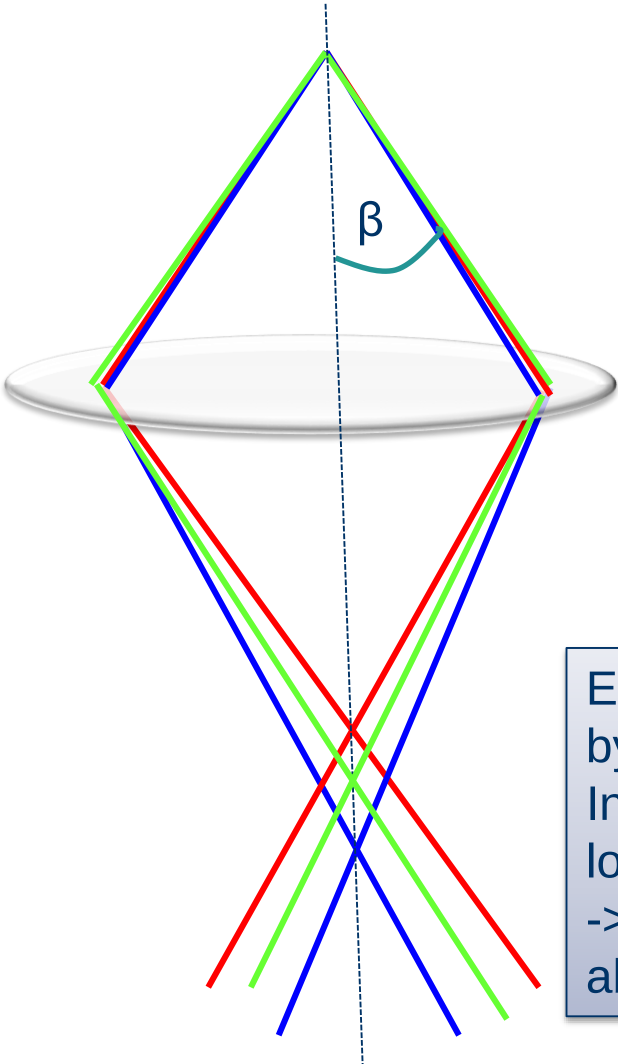
$$r_{sph} = C_s \cdot \beta^3$$



With increasing β, the electron a more strongly focused resulting in a decreased focal length of the lens.
C_s is always positive for cylindrical, symmetric EM lenses

Blurring of Airy disc cause by spherical aberration

Chromatic aberration (C_c)



$$r_{chr} = C_c \cdot \frac{\Delta E}{E} \beta$$

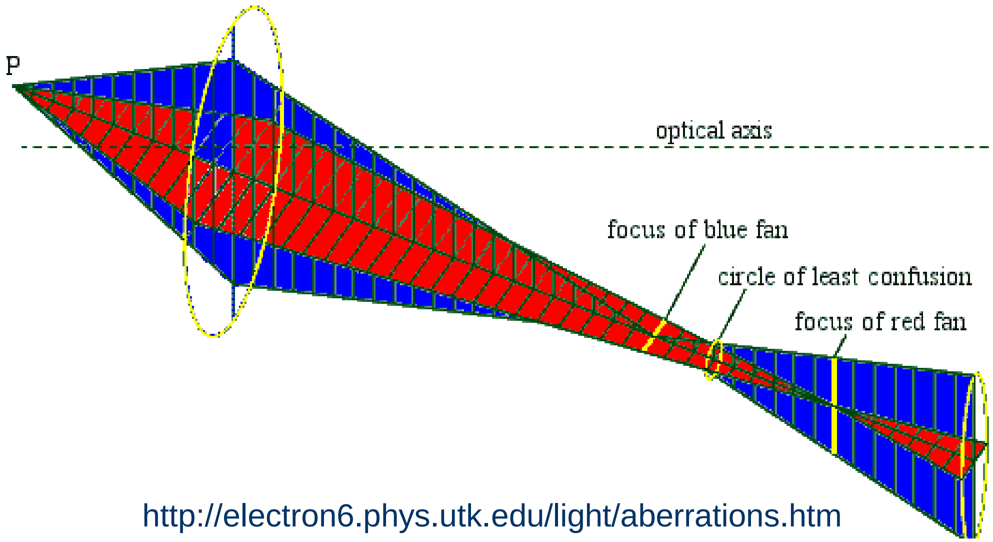
Electron with lower energies are stronger scattered by the lens -> focal length decreases.
Inelastic scattering processes result in an energy loss of the incoming e^-.
-> **samples thickness** impacts the chromatic aberration of the transmitted e-beam

Astigmatism

Astigmatism is when an optical system has different foci for rays (electrons) that propagate in two perpendicular planes

$$r_{ast} = \beta \cdot \Delta f$$

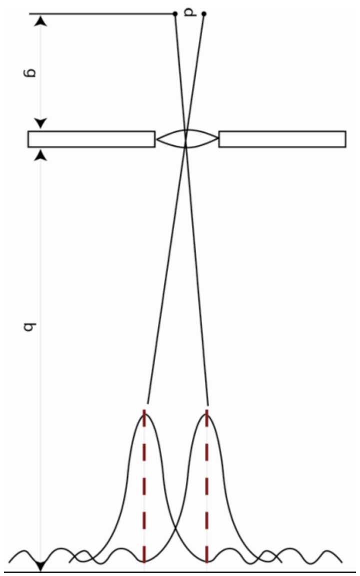
Δf = difference in focus caused by the astigmatism.



http://electron6.phys.utk.edu/light/aberrations.htm

Inhomogeneous magnetic field along the length of the lens and imperfect apertures cause astigmatism.
Imperfections are compensated through additional octapole lenses.

Theoretical resolution (diffraction limitation)



$$r_{th} = 0.61 \cdot \frac{\lambda}{\beta}$$

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Practical resolution (Cs limited)

After correcting of the astigmatism and with a thin sample the practical resolution is determined by the diffraction and the spherical aberration.

$$r = \left(d_{th}^{2} + d_{sph}^{2}\right)^{\frac{1}{2}}$$

$$r(\beta) = \left[\left(0.61 \cdot \frac{\lambda}{\beta}\right)^{2} + \left(C_s \cdot \beta^{3}\right)^{2}\right]^{1/2}$$

$$\frac{dr(\beta)}{d\beta} = 0 = -2 \cdot \frac{(0.61 \cdot \lambda)^{2}}{\beta^{3}} + 6 \cdot C_s^{\,2} \cdot \beta^{5}$$

$$\beta_{opt} = 0.77 \cdot \frac{\lambda^{1/4}}{C_s^{\,1/4}}$$

$$r_{\min} = 0.91 \cdot \left(C_s \lambda^{3}\right)^{1/4}$$

$r_{\min} \sim 0.15 – 0.3\,\mathrm{nm}$

Specimen limited resolution (Cc limited)

$$r_{chr} = C_c \cdot \frac{\Delta E}{E} \beta$$

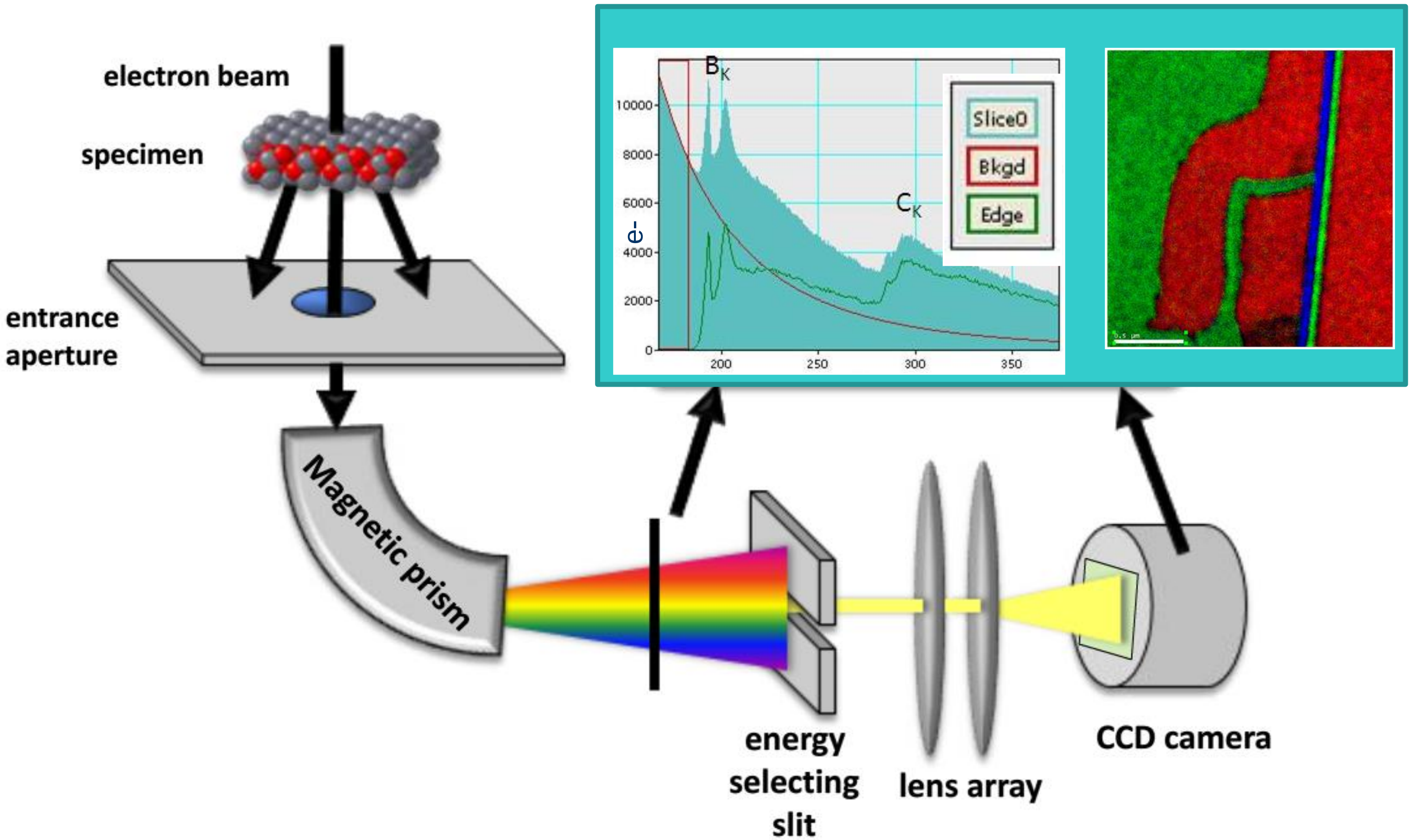
Due to inelastic interactions of the primary electron beam with the specimen, the primary electrons loose a fraction of their energy (typically ~20 eV (Plasmon)).

ΔE = 20 eV, E = 100 keV, β_{opt} = 4.5 mrads → **r_{chr} = 2.5 nm**

→ r_{chr} is about 10 x larger than r_{sph}.
Thick specimen will result in poor resolution!

Elemental analysis in the EM

Electron energy loss spectroscopy (EELS) Energy filtered TEM (TEM)



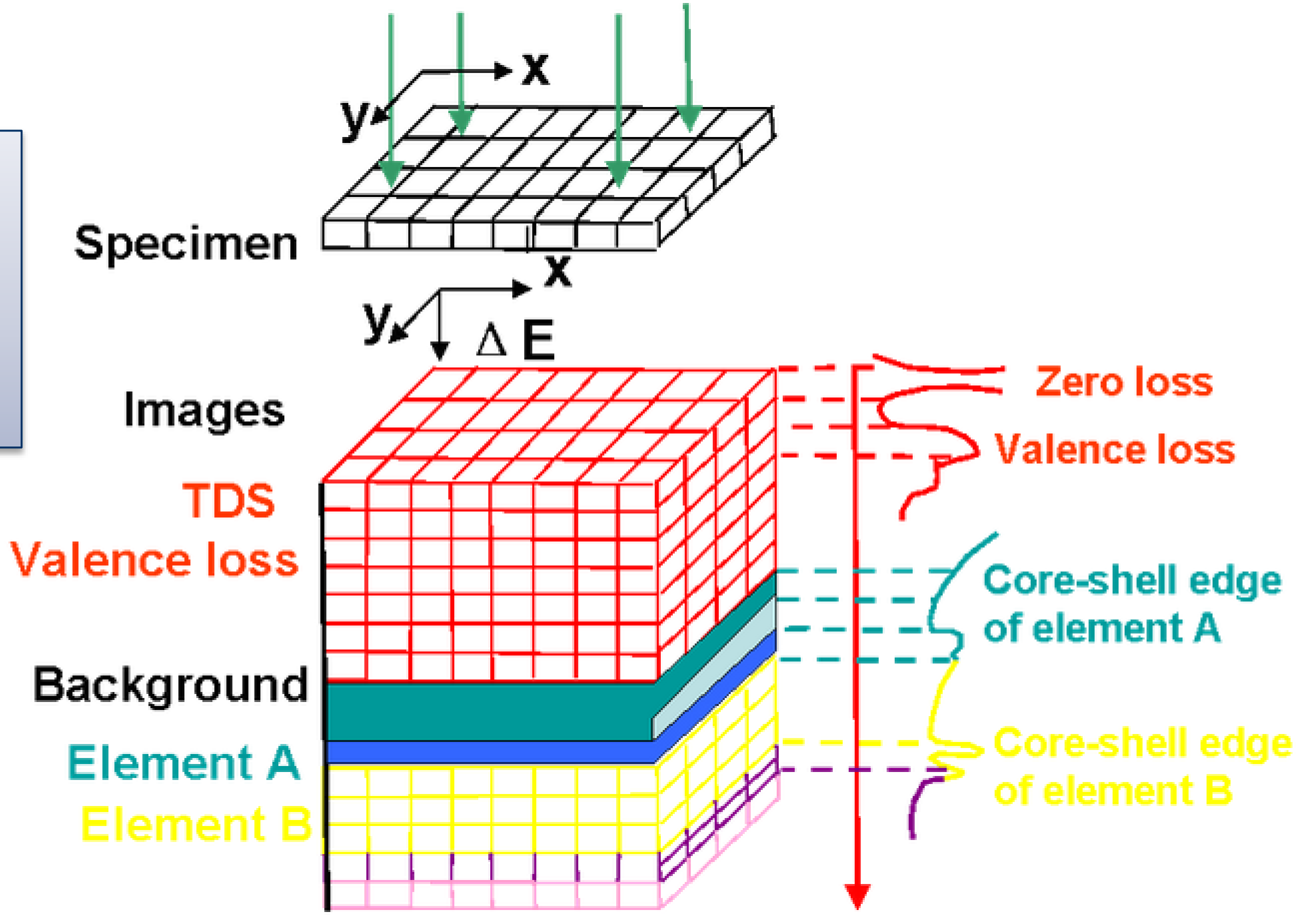
http://www-hrem.msm.cam.ac.uk/research/EFTEM/EFTEM.html

http://www.pdx.edu/cemn/tem-gallery

STEM spectral imaging vs. EFTEM spectral imaging

STEM SI
- Parallel **spectrum** acquisition with scanned probe (x,y)
- Serial image acquisition

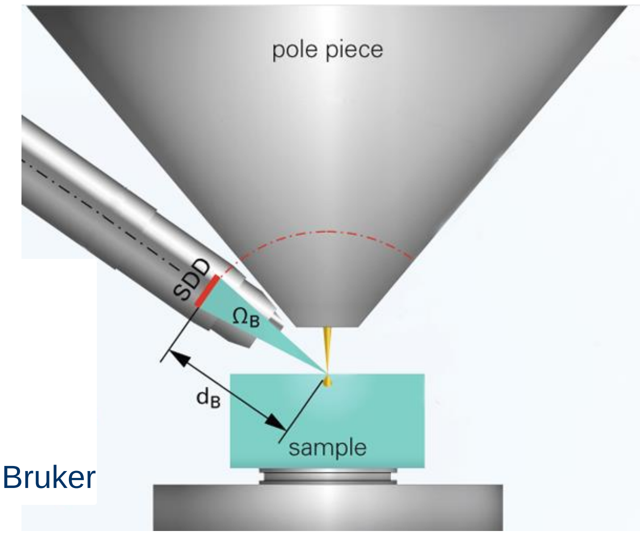
EFTEM SI
- Parallel **image** acquisition with ΔE spacing.
- Serial spectral acquisition (ΔE)



http://www.globalsino.com/EM/page4619.html

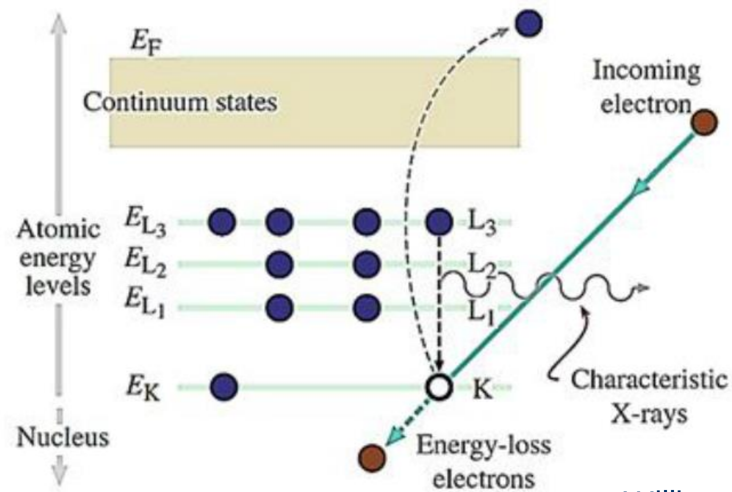
Spatial resolution ~ 1nm
Detection limit ~ 2 – 5 % local atomic concentration

Energy dispersive x-ray (EDX) analysis



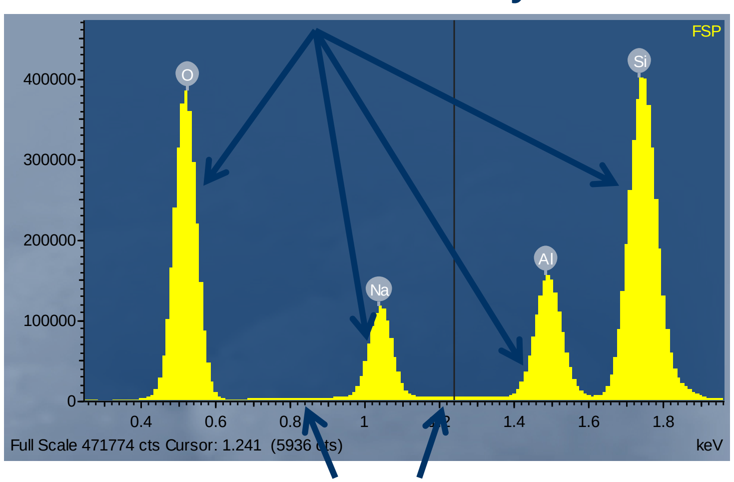
Bruker

Generation of x-rays



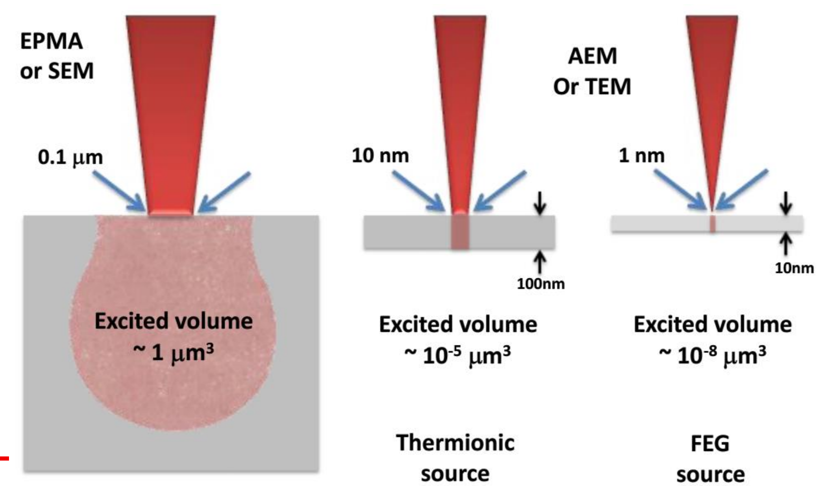
Williams and Carter, 1996

Characteristic x-rays



Bremsstrahlung

Spatial resolution



References and some useful links…

Books:

- Barry C. Carter and David B. Williams, Transmission Electron Microscopy, A Textbook for Materials Science, 2009 (http://www.matter.org.uk/tem/)
- Michael J.R. Goldstein et al., Scanning Electron Microscopy and X-ray Microanalysis, 2003
- Debbie J. Stokes, Principles and Practice of Variable Pressure/Environmental Scanning Electron Microscopy (VP-ESEM), 2008
- Ray F. Egerton, Physical Principles of Electron Microscopy, 2007
- P.F. Schmidt, Praxis der Rasterelektronenmikroskopie und Mikrobereichsanalyse Expert-Verlag, 1994 (Kontakt & Studium ; 444 : Meßtechnik)

Links:

http://www.ammrf.org.au/myscope/
http://www.globalsino.com/EM/
http://tx.technion.ac.il/~mtyaron/TEMcourse_flash/ (Jaron Kauffmann)
http://www.ccmr.cornell.edu/igert/modular/docs/3_Scanning_Electron_Microscopy.pdf
http://nanohub.org/resources/4092

www.NanoDefine.eu

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