A practical introduction to scanning electron microscopy

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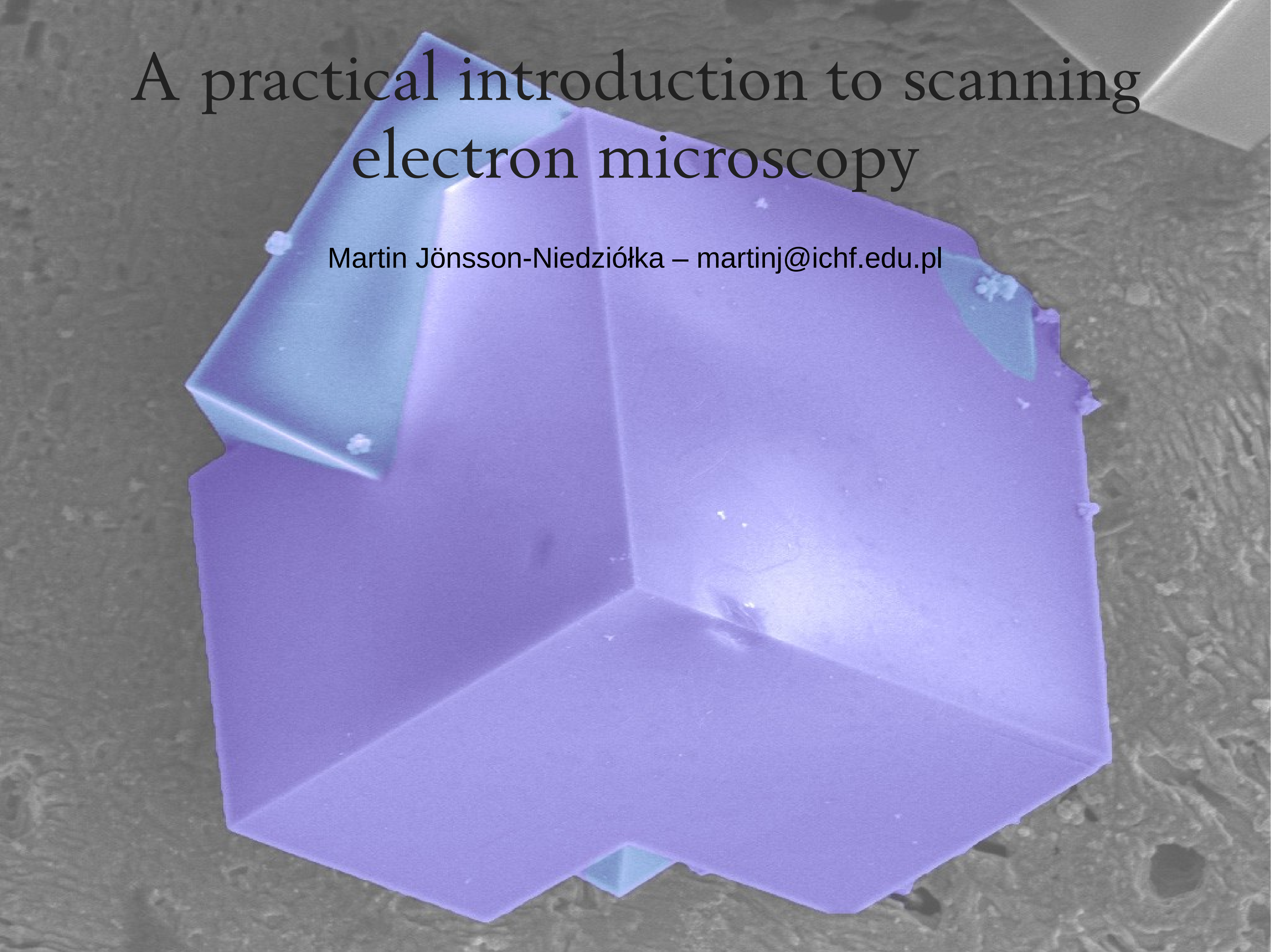


Practical information

4 hours lectures
4 hour practicals (groups of 3)

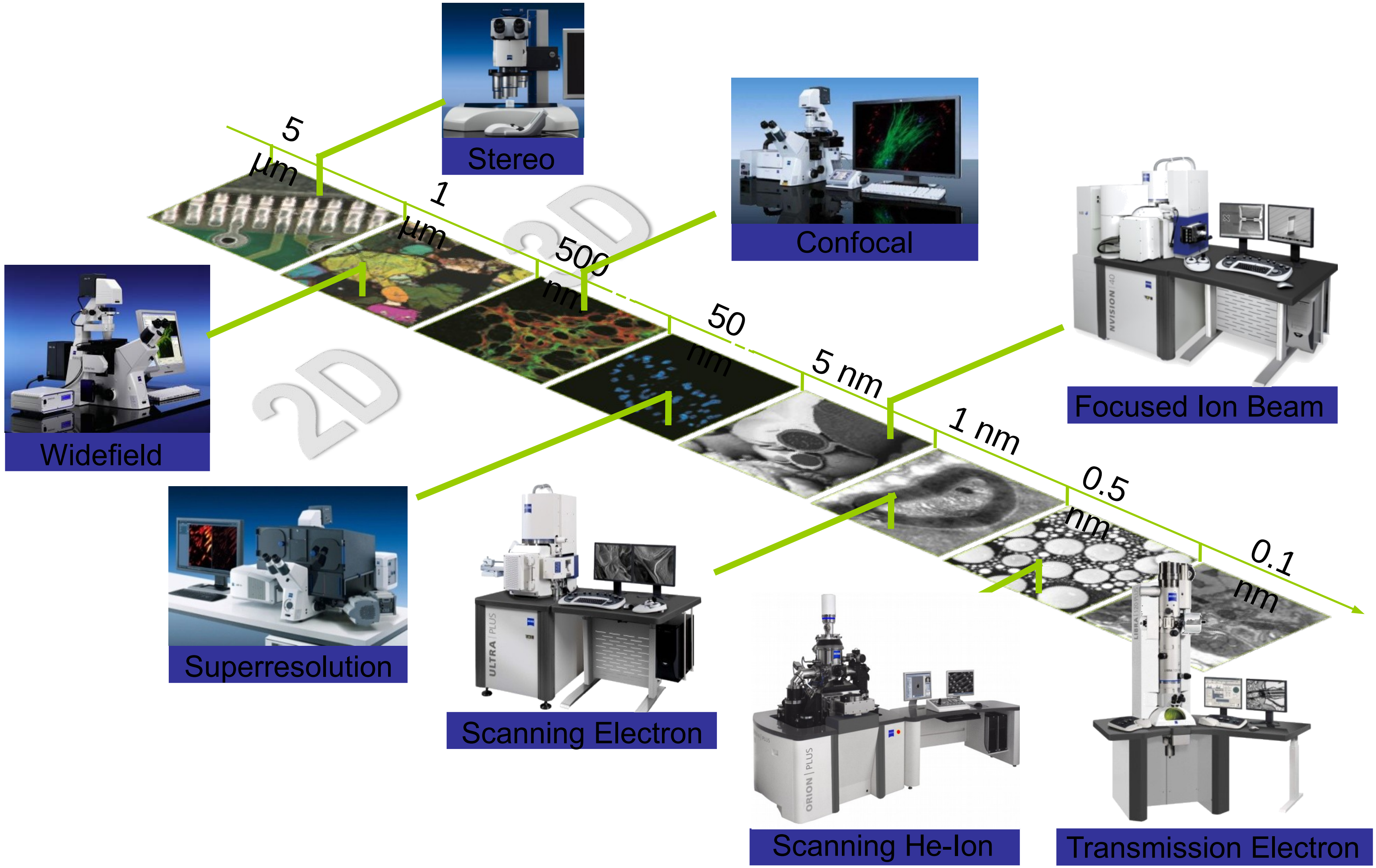
Exam: Measure "mystery sample"
Analyse images (size, composition)
Hand in report

Info on www.charge-transfer.pl/sem-course

Content

- Basic principles
- Electron-matter interaction
- Settings and limitations
- Different detection modes
- Our system
- Sample preparation
- EDX / WDX

Why SEM?

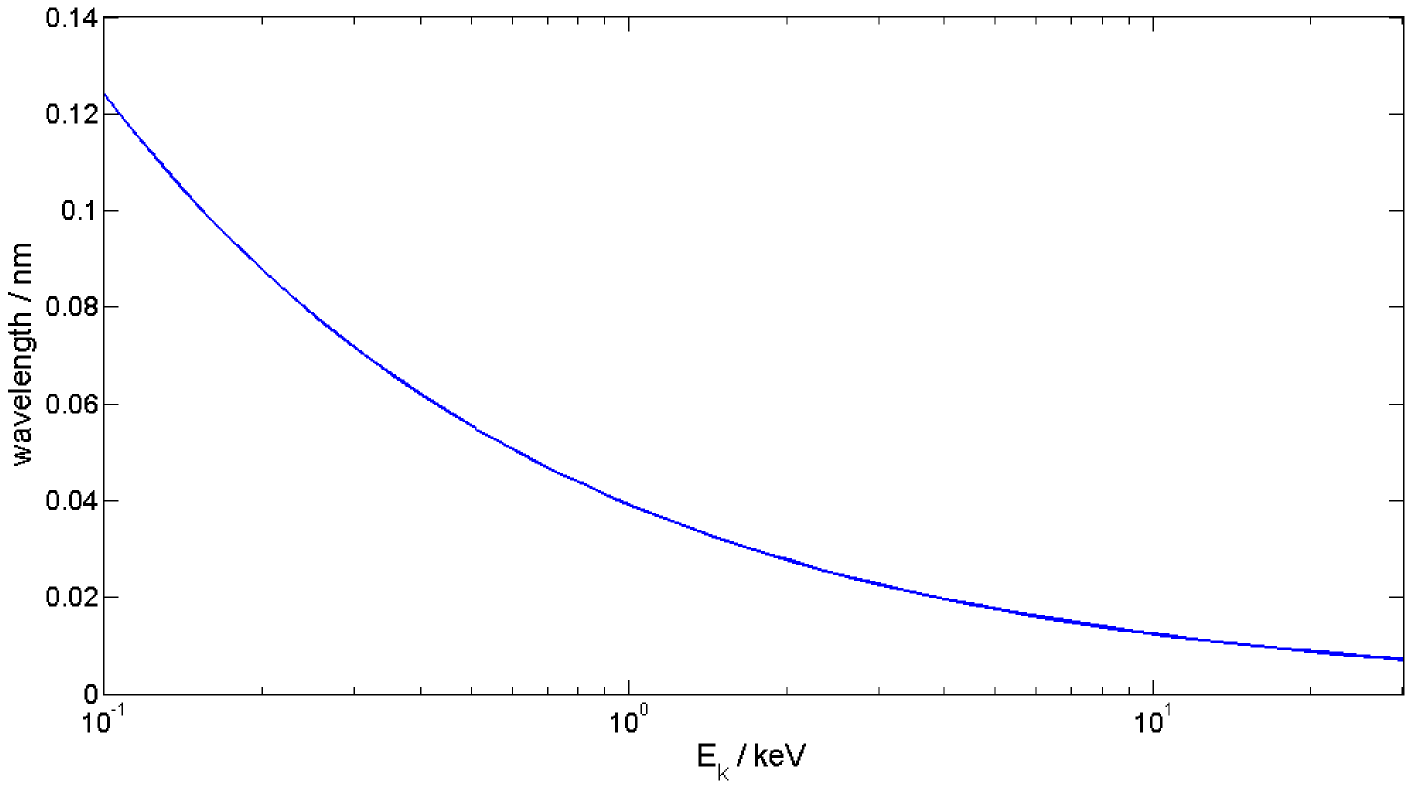


Stereo
Confocal
Focused Ion Beam
Widefield
Superresolution
Scanning Electron
Scanning He-Ion
Transmission Electron
5 µm
1 µm
500 nm
50 nm
5 nm
1 nm
0.5 nm
0.1 nm
2D
3D

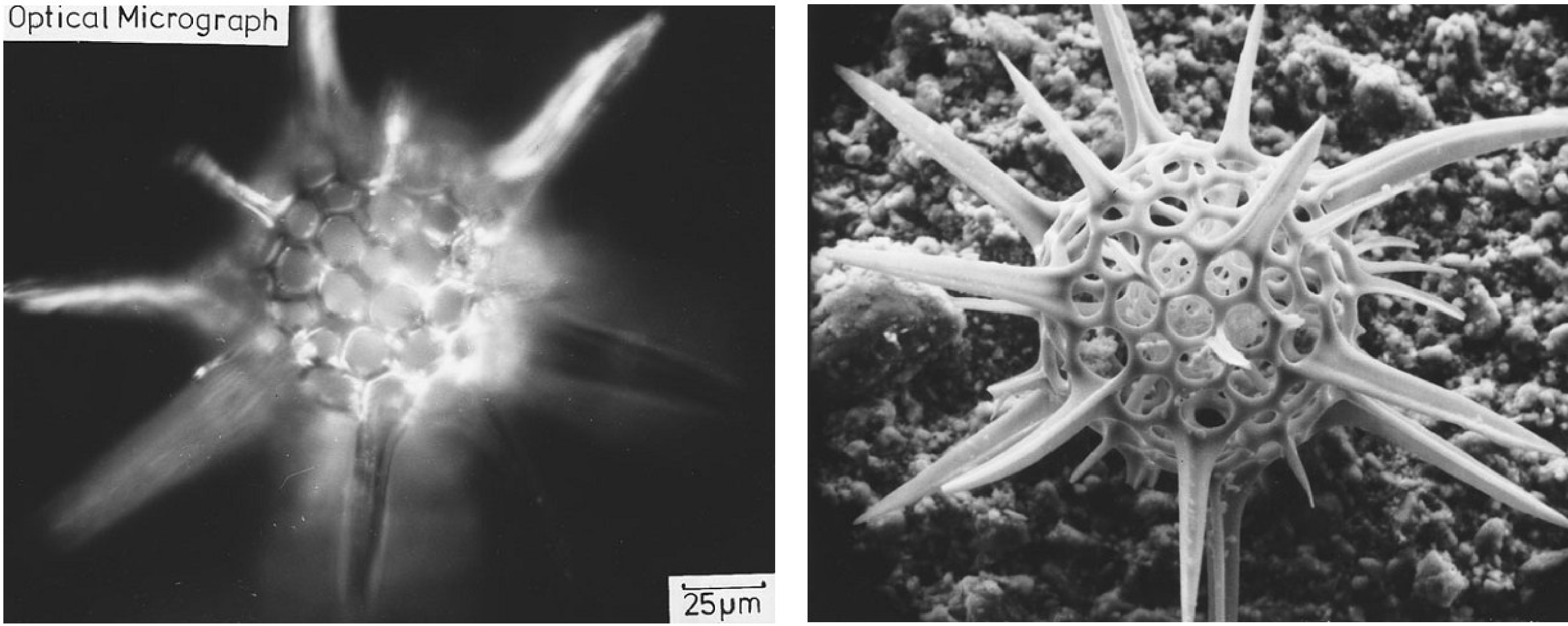
Why SEM?

de Broglie wave-length

$$\lambda = \frac{h}{\sqrt{2mE_{kin}}} \approx \frac{1.23}{\sqrt{E_{kin}}} \text{ nm}$$



Why SEM?

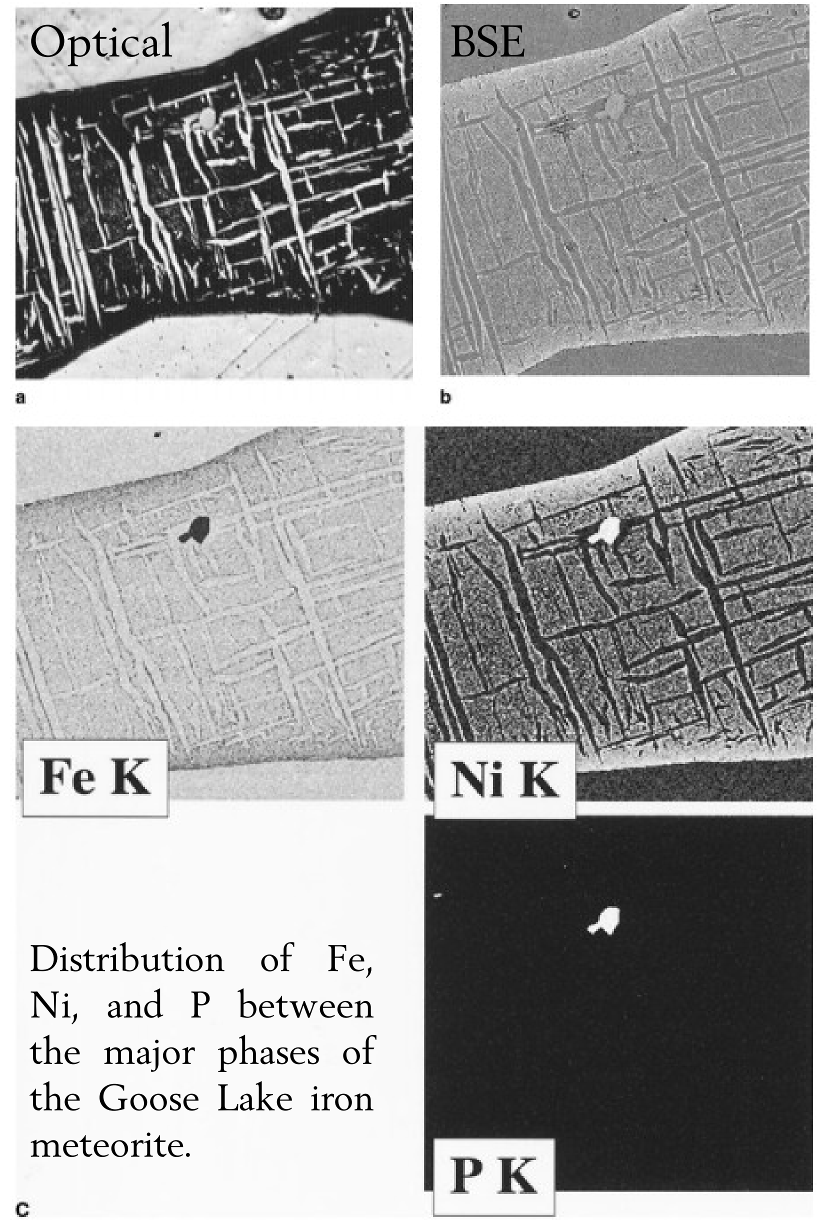


Optical and SEM micrograph of the radiolarian *Trochodiscus longispinus*

Source: Goldstein

Why SEM?

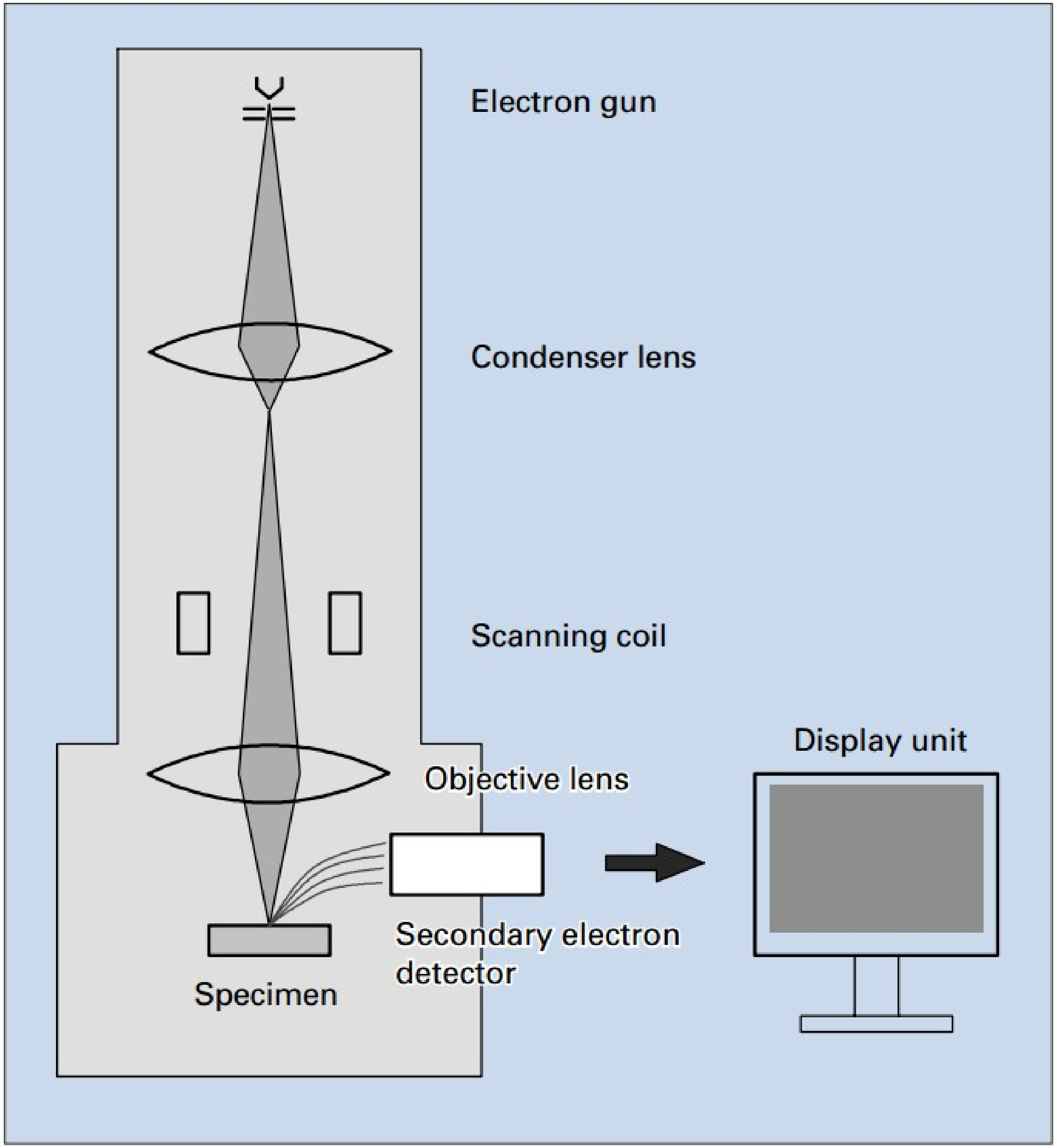
- Versatile
  - Topography
  - Elemental information
  - Crystallinity
- Fast
- Non-descructive (mostly)
- Easy (mostly)

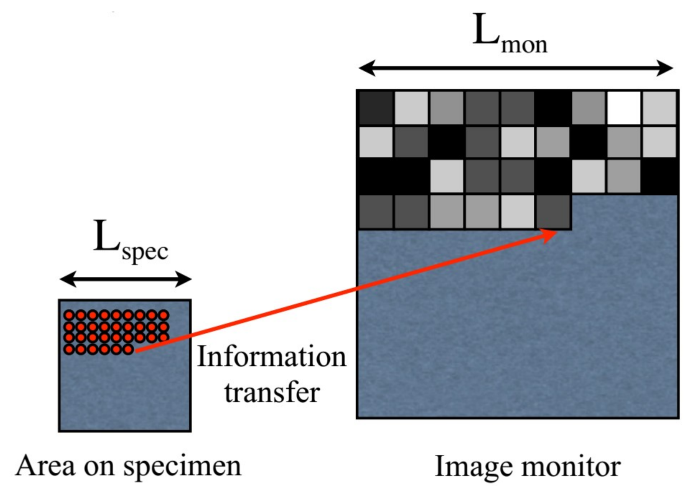


Distribution of Fe, Ni, and P between the major phases of the Goose Lake iron meteorite.

Source: Goldstein

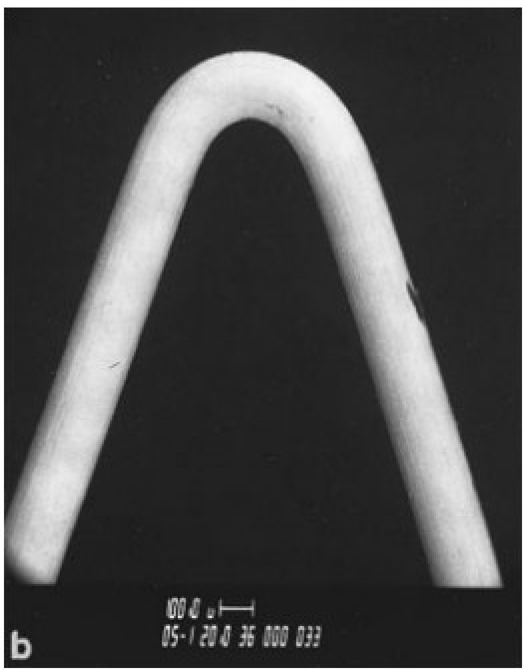
Working principle





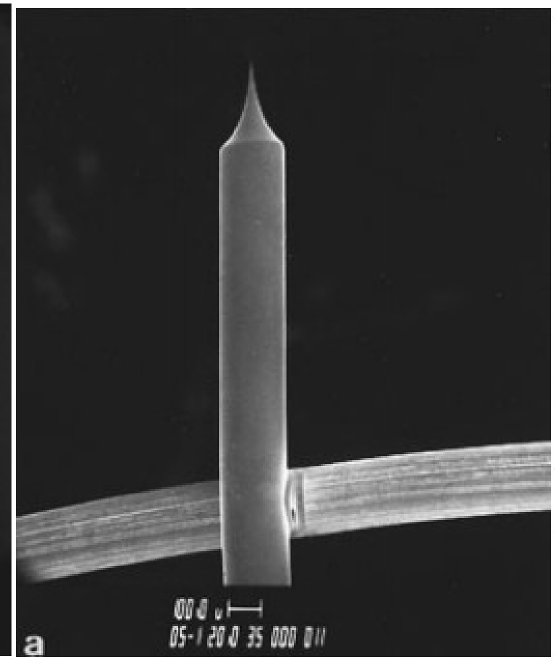
Electron gun

	TE gun		FE gun	SE gun
	Tungsten	LaB_6		
Electron-source size	15 ~ 20 μm	10 μm	5 ~ 10nm	15 ~ 20nm
Brightness (Acm^{-2} rad^{-2})	10^5	10^6	10^8	10^8
Energy spread (eV)	3 ~ 4	2 ~ 3	0.3	0.7 ~ 1
Lifetime	50 h	500 h	Several years	1 to 2 years
Cathode temperature (K)	2800	1900	300	1800
Current fluctuation (per hour)	<1%	<2%	>10%	<1%

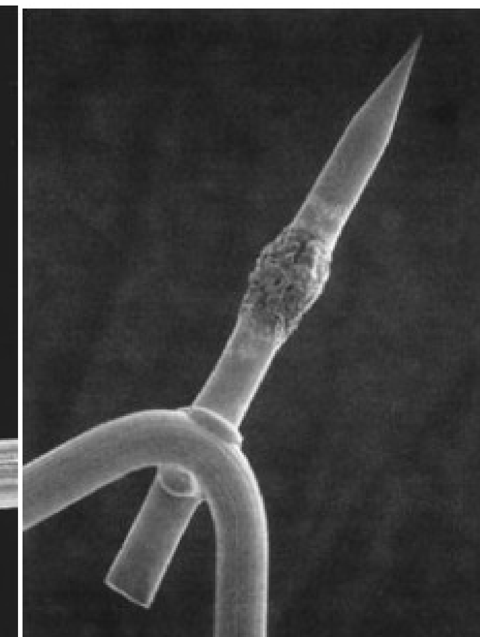


Tungsten wire thermionic emitter

Lanthanum hexaboride thermionic emitter



Tungsten singly-crystal cold field emission tip


Tungsten/ZrO_2 Schottky field emission tip

Source: Goldstein

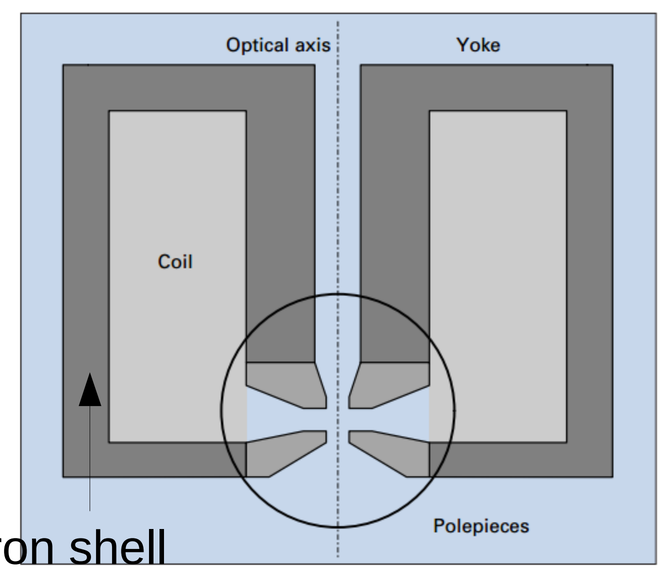
Electron lenses

In SEM magnetic lenses are used.
- Cylindrically symmetric
- “Soft magnetic” shell
- Field leaks out at narrow gap.
- Only the pole pieces need to be very accurately fabricated

Focal length of the lens can be changed

$$f \approx V_0 / (N I)^2$$

Beam is twisted through the lens



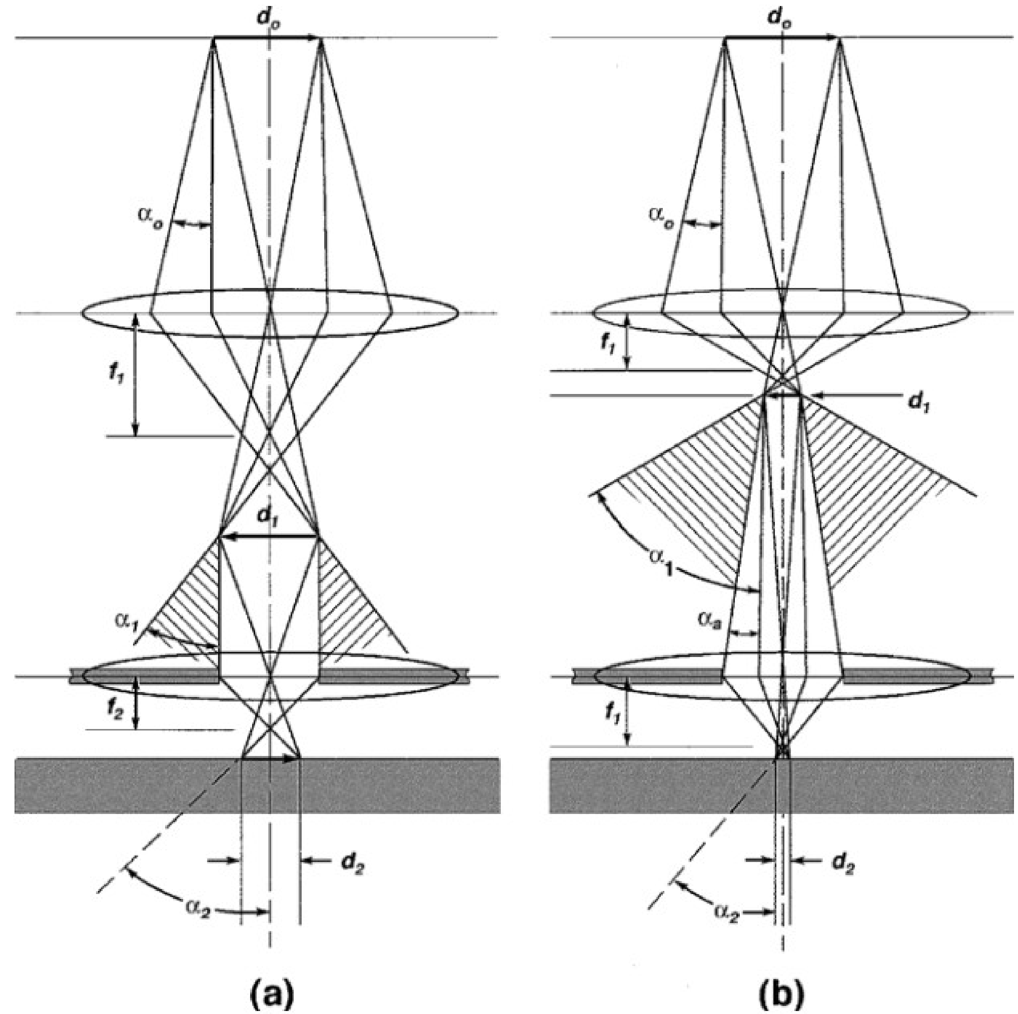


Figure 2.20. Effect of condenser lens strength in a two-lens lens system. (a) Weak condenser lens, (b) strong condenser lens.

Strong lens ('large spot') → Higher current
→ Lower resolution

Objective lens

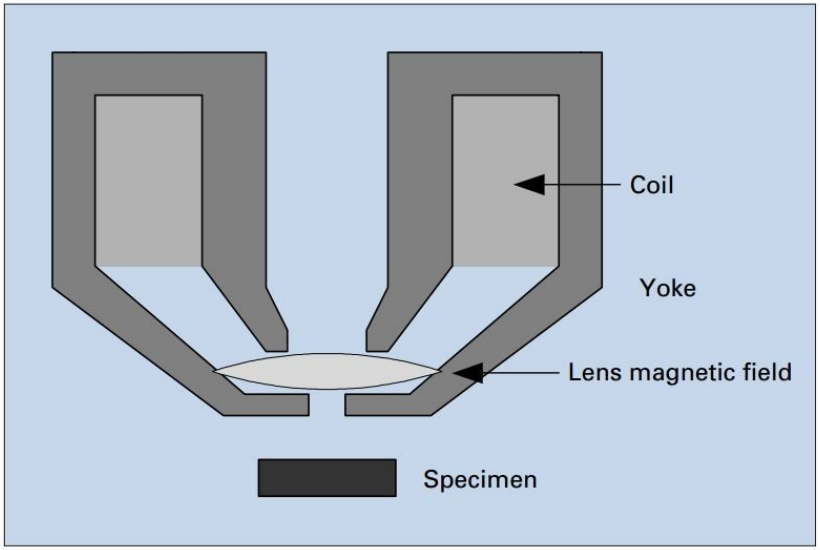


Fig. 34 Construction of the conventional objective lens.

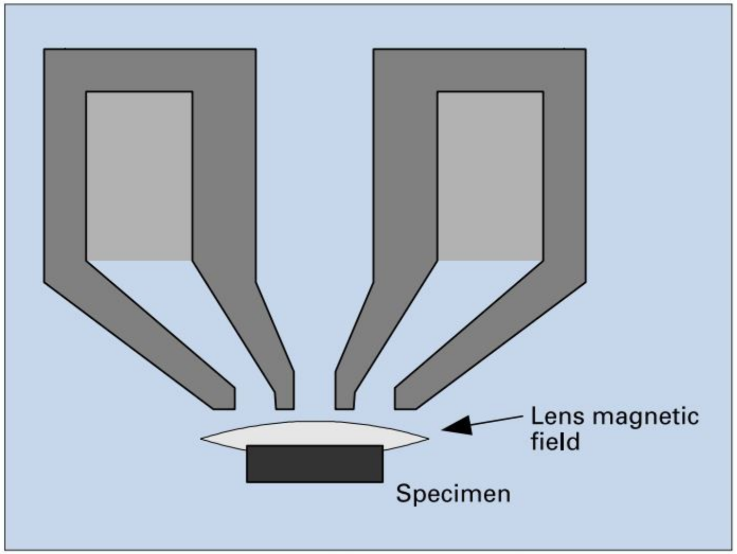


Fig. 36 Construction of the semi-in-lens objective lens.

snorkel
immersion

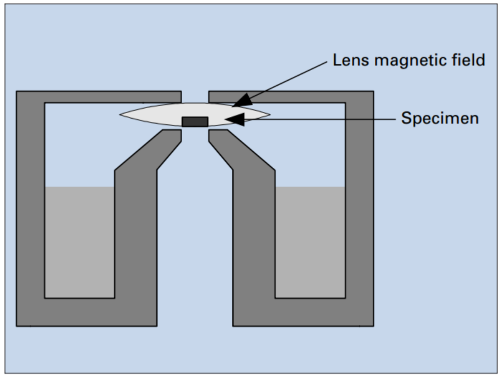


Fig. 35 Construction of the in-lens objective lens.

Snorkel:

- Higher resolution
- Lower max field of view (min ca 1500x)
- Only in-lens detector
- Limit on usable voltages

WD / mm	E_{max} / kV
1	1.2
2	3.5
3	7
4	12
5	18
6	24
7	30

Limits on resolution

Resolution is given by probe diameter (size of electron beam)

- Gaussian beam diameter (no aberration)

$$d_G = \sqrt{\frac{4i_p}{\beta\pi^2\alpha_p^2}}$$

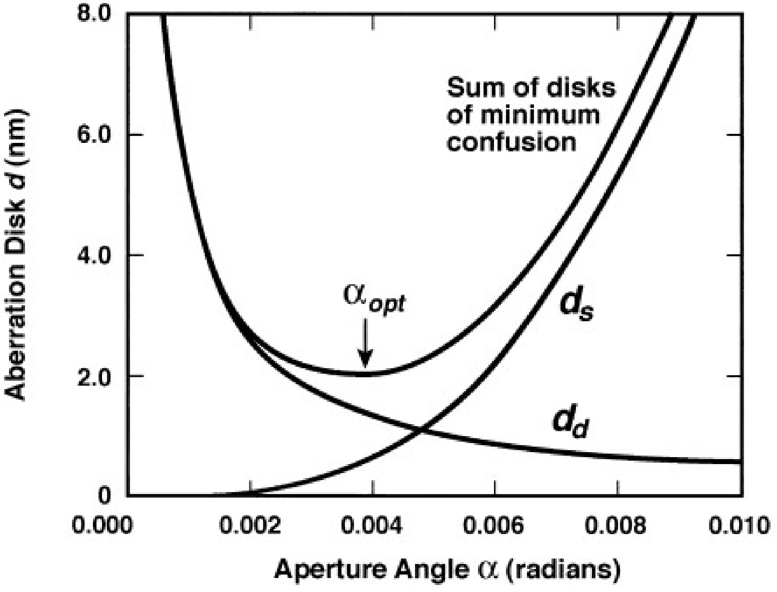
- Diffraction in aperture
- Spherical aberration

$$d_s = \frac{1}{2}C_s\alpha^3,$$

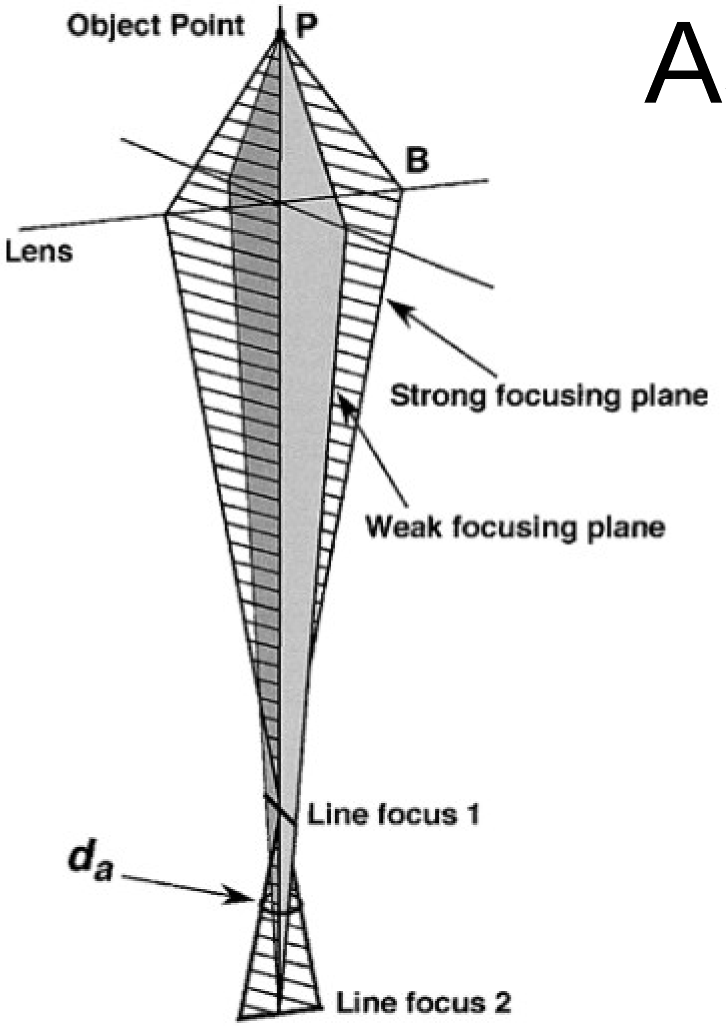
Almost prop to f

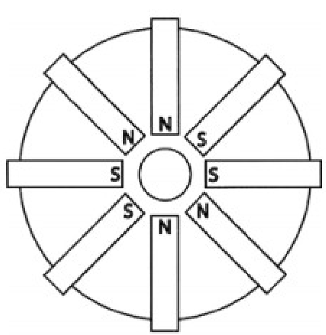
- Chromatic aberration

$$d_c = C_c\,\alpha\left(\frac{\Delta E}{E_0}\right)$$

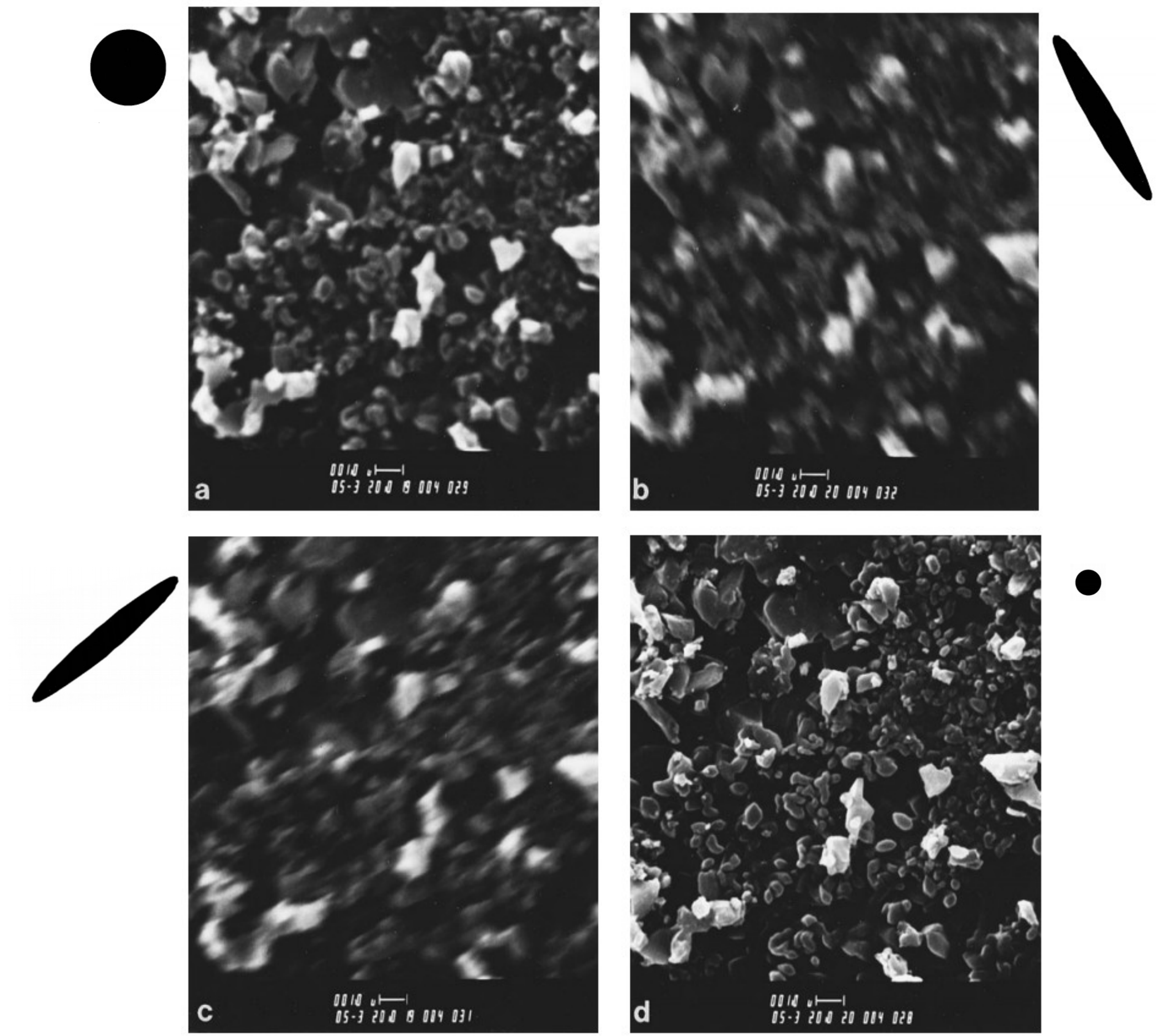


Astigmatism

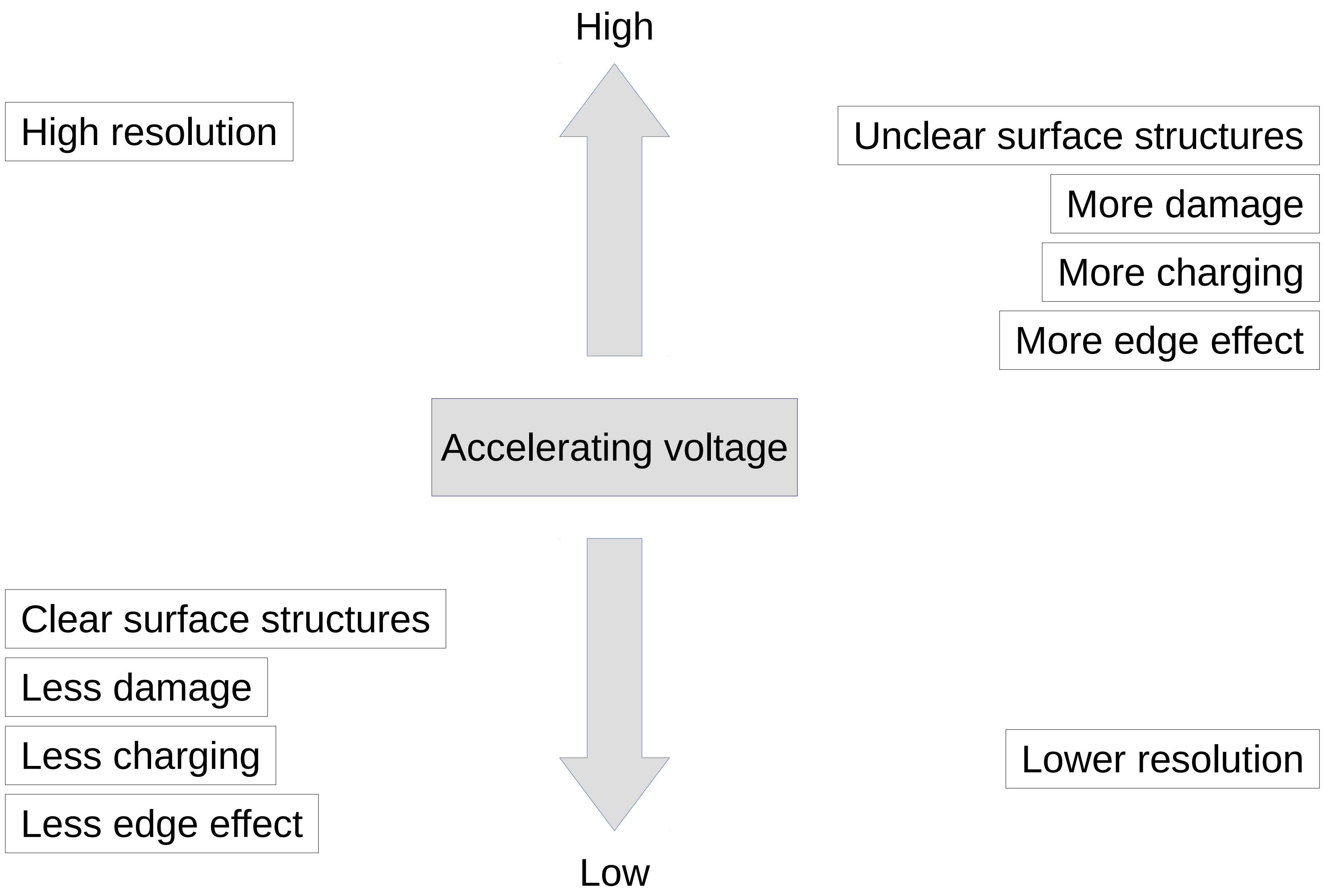




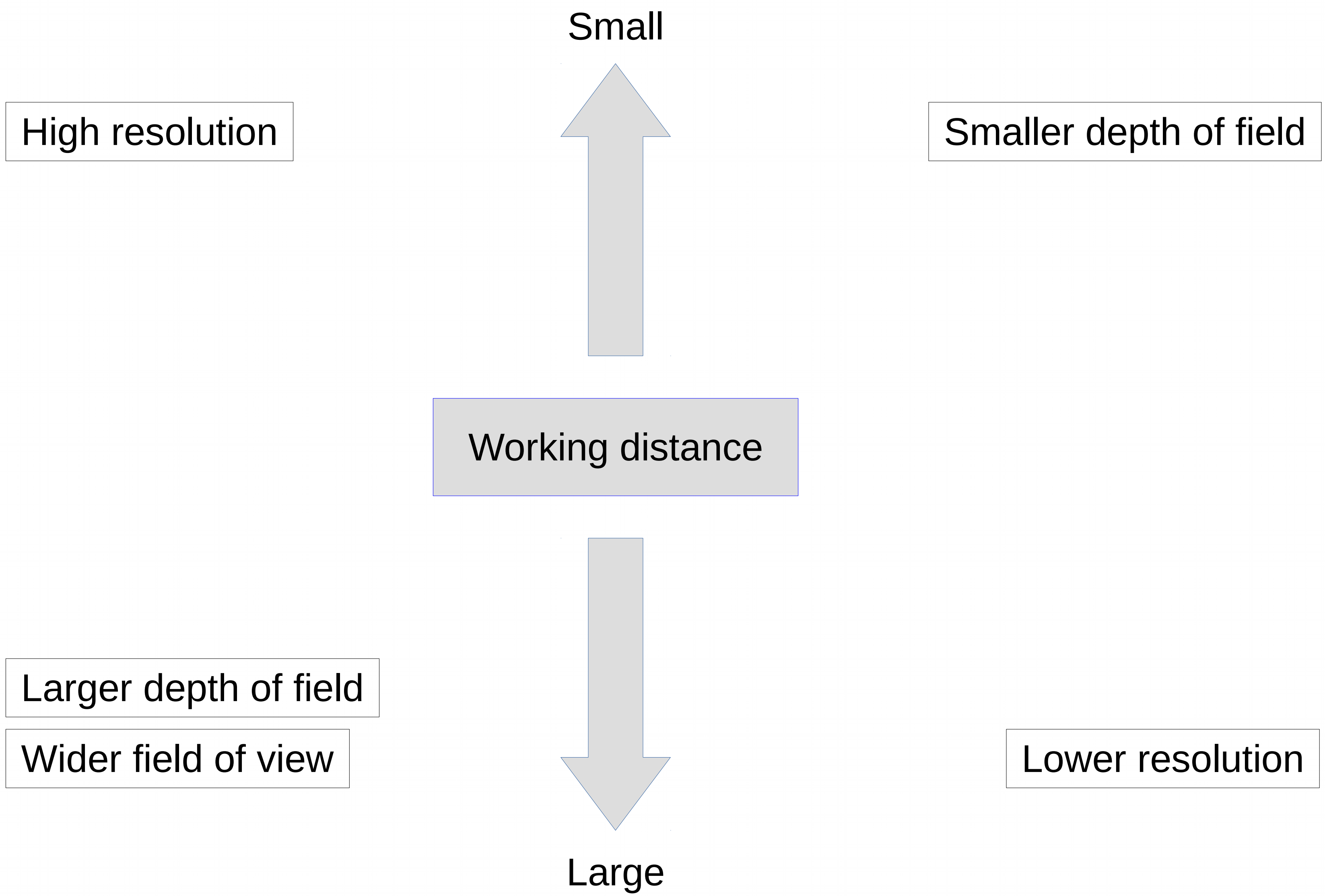
The lens does not focus with the same strength in all directions.



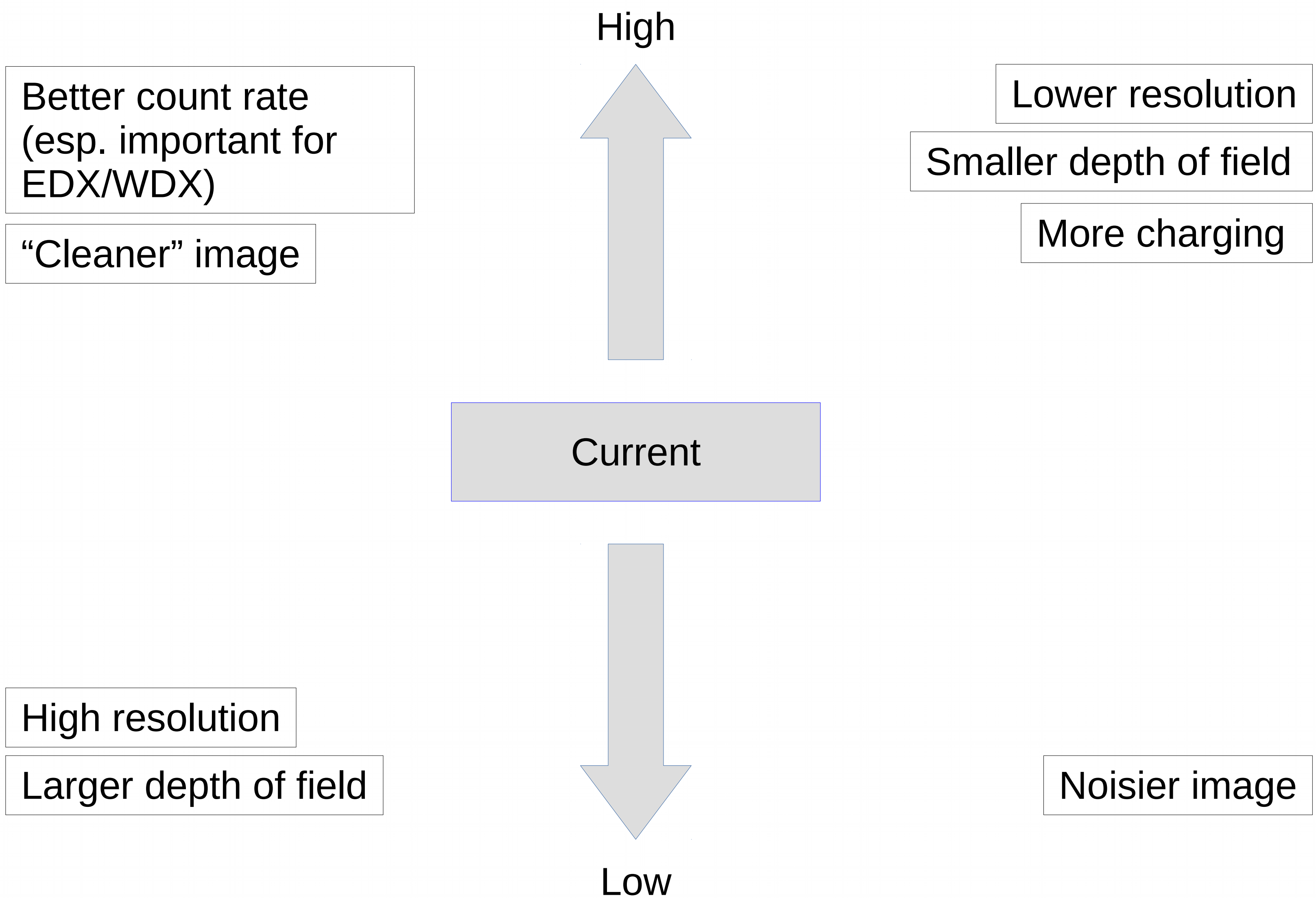
A game of compromises



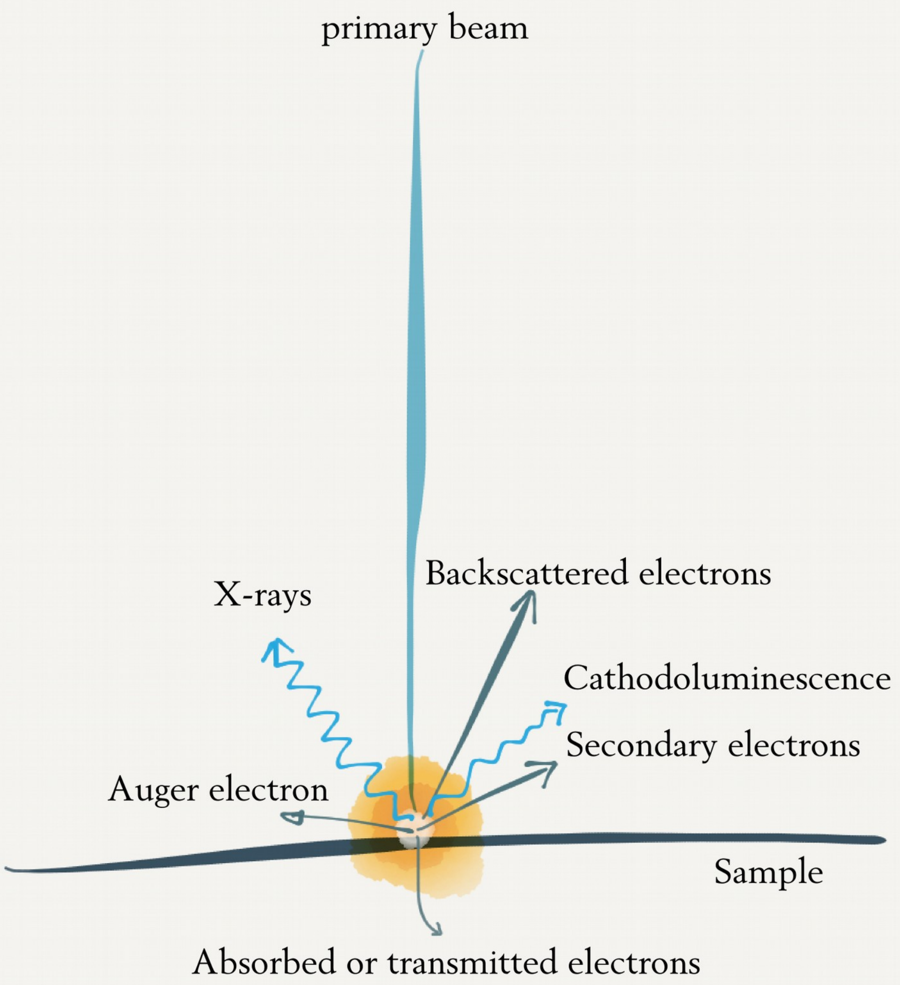
A game of compromises



A game of compromises

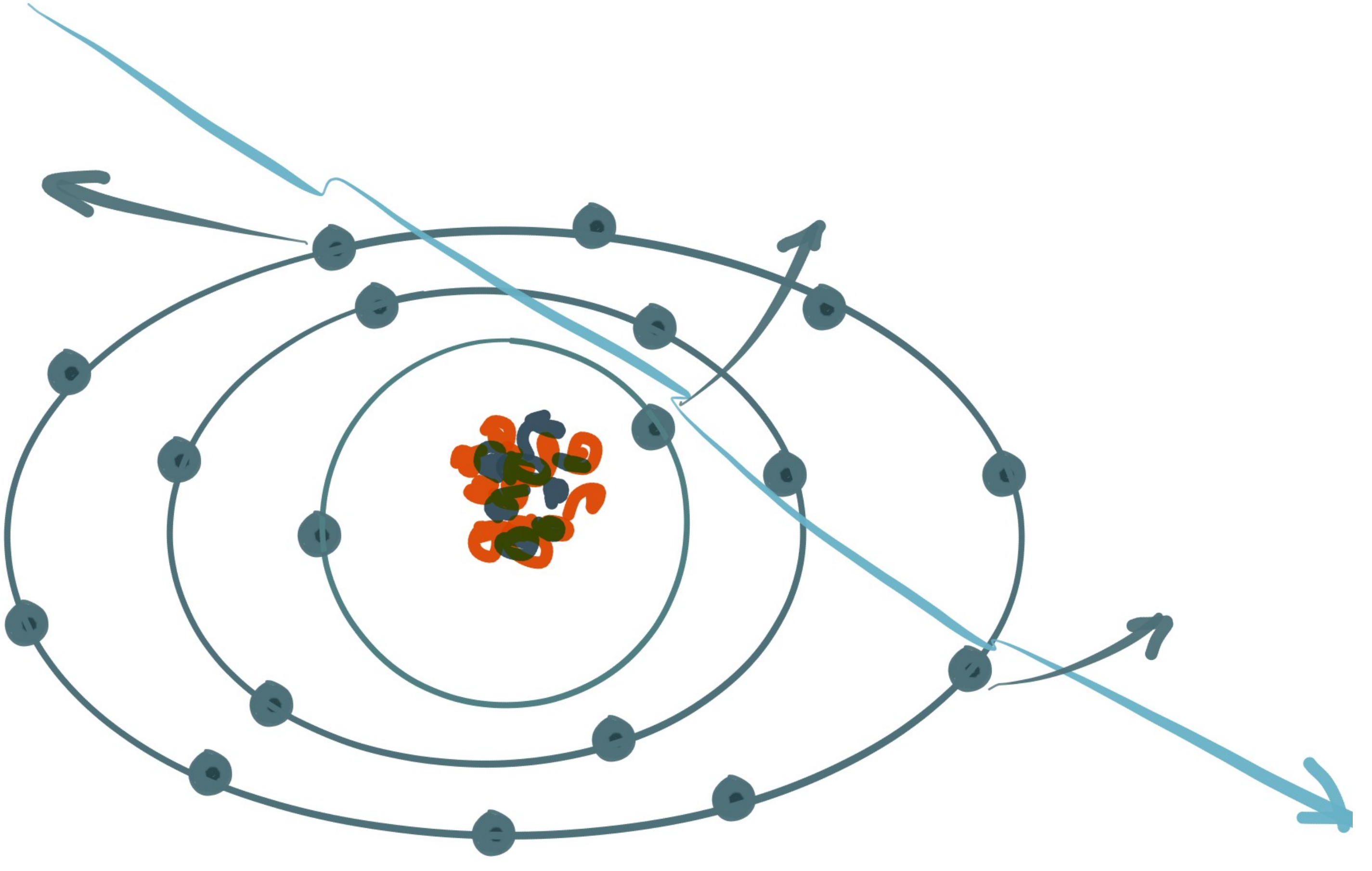


Electron matter interaction



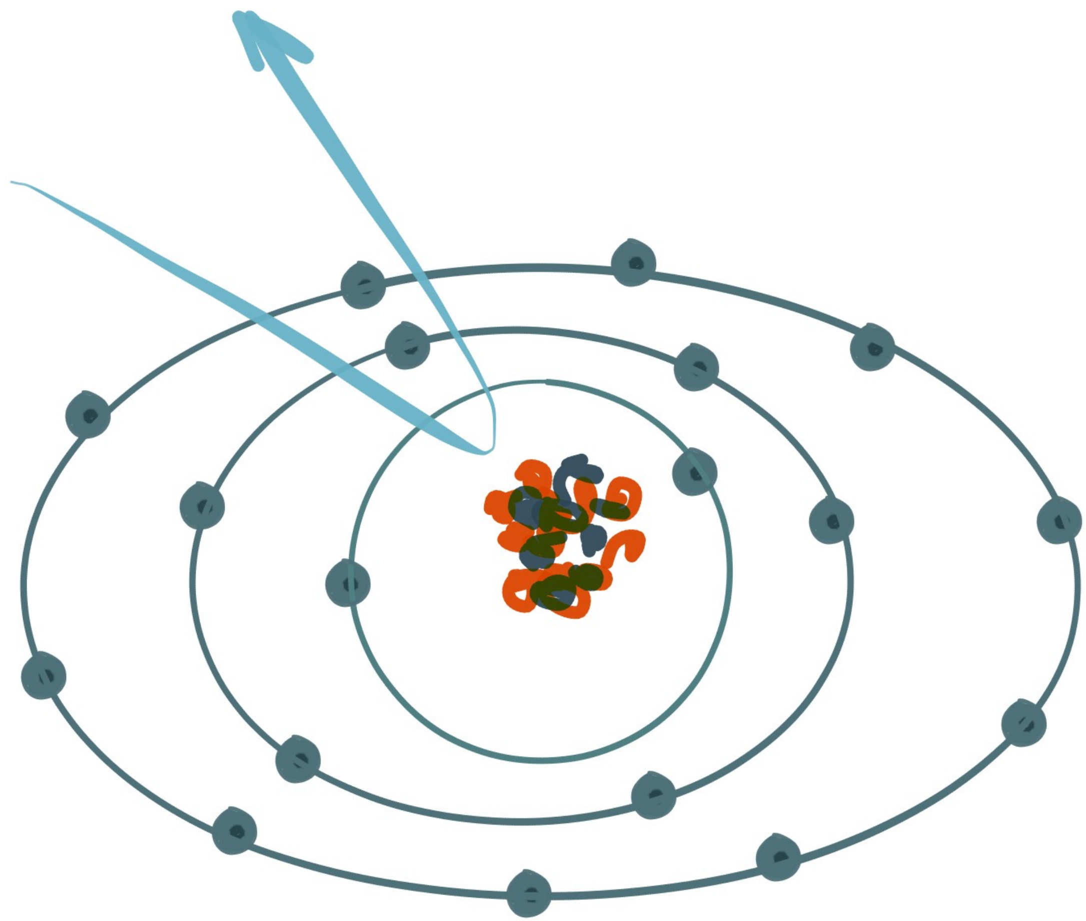
Secondary electrons

- Non-elastic collision
- Electrons come from inside the atoms in the sample.
- Low energy
- Information about surface topography



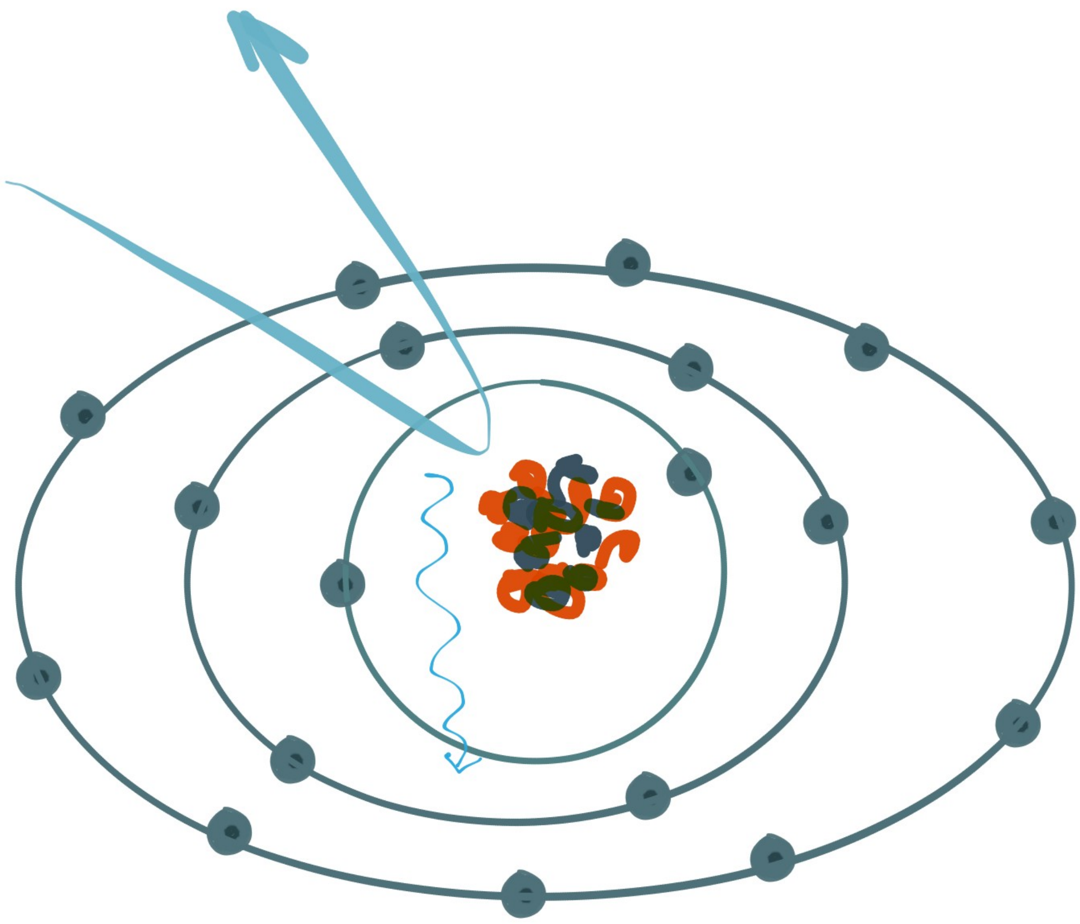
Backscattered electrons

- (Almost) elastic collision
- Electrons come from the primary beam.
- High energy
- Probes deeper into the sample
- Some information about elemental composition



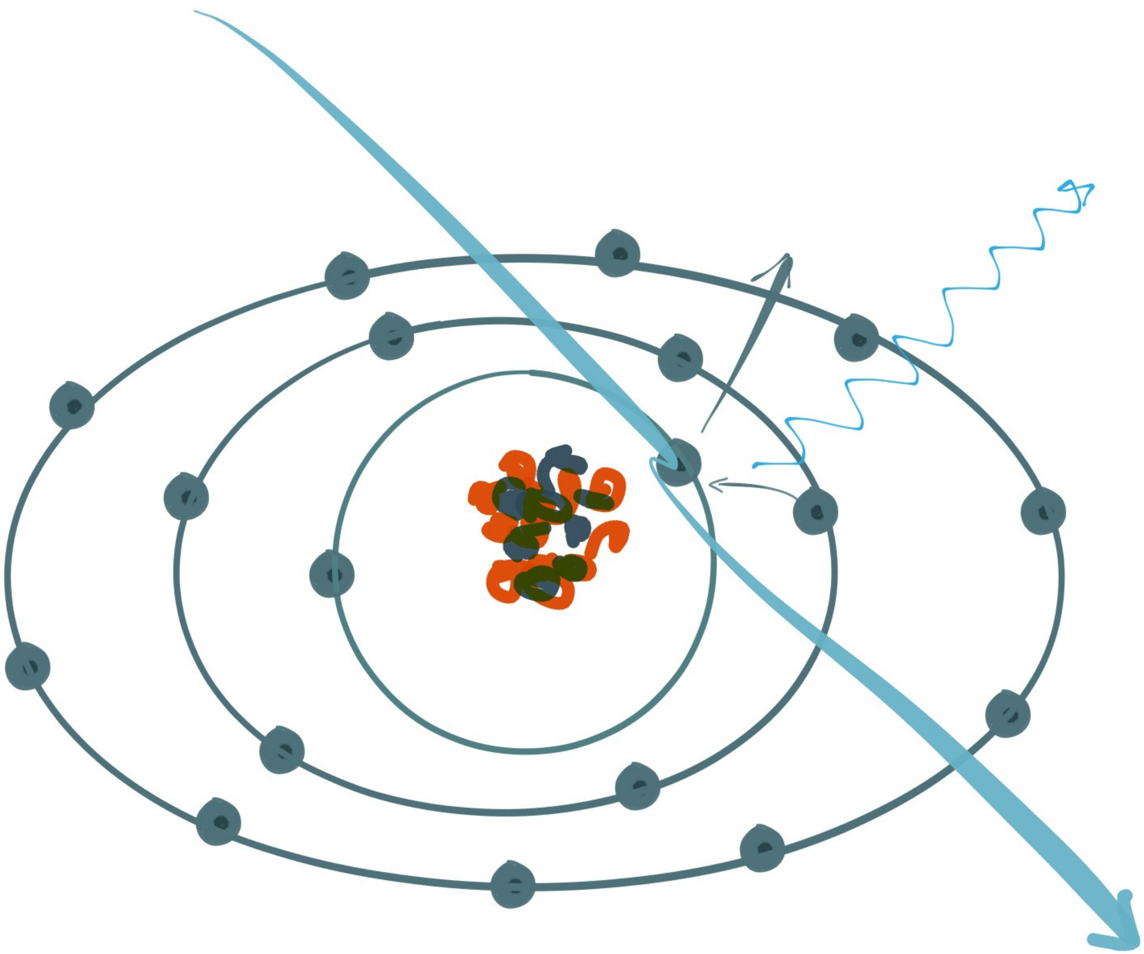
X-ray emission (Bremsstrahlung)

- X-ray emission sent out when an electron is accelerated.
- No elemental information.
- Forms background in EDX.

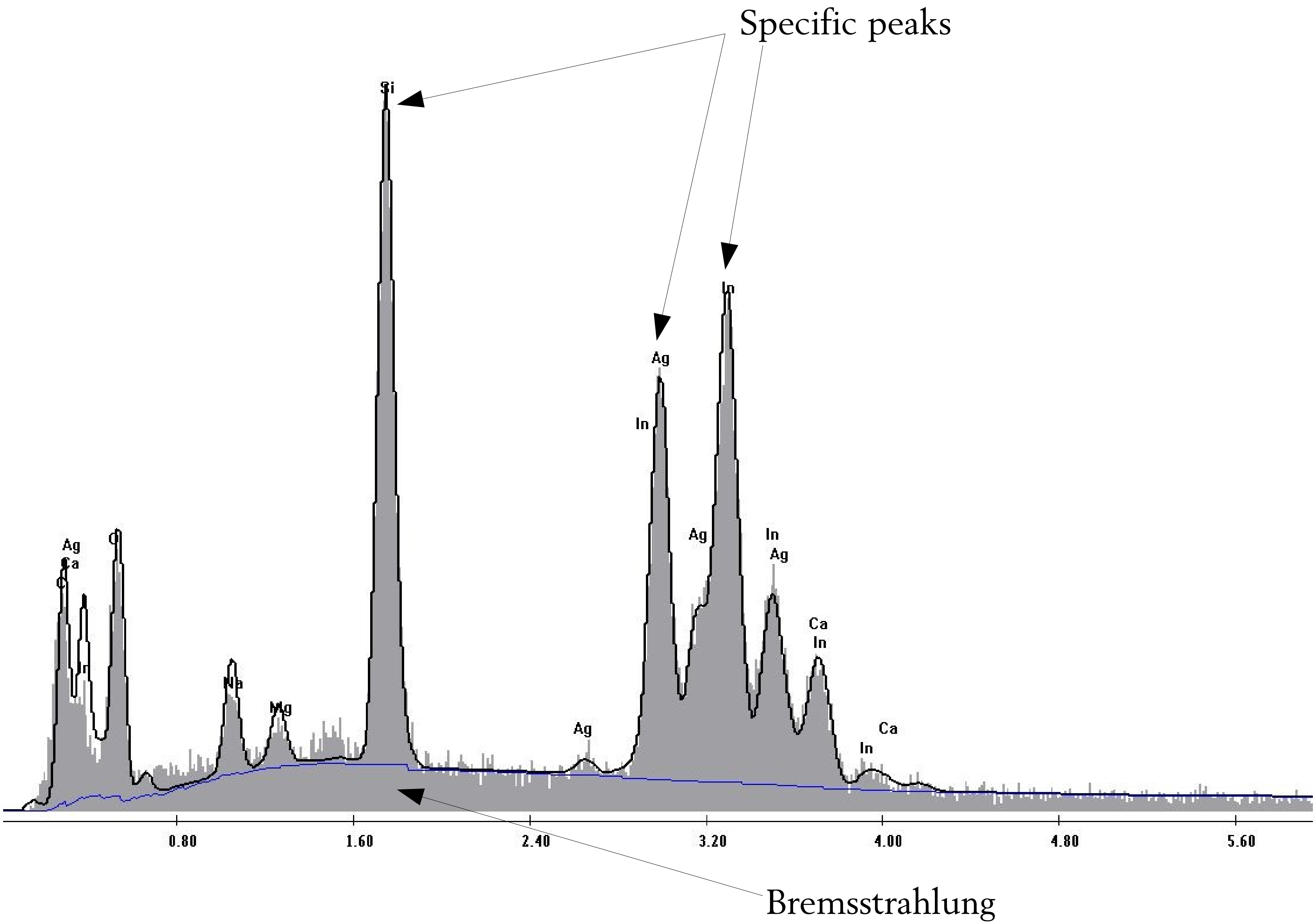


X-ray emission

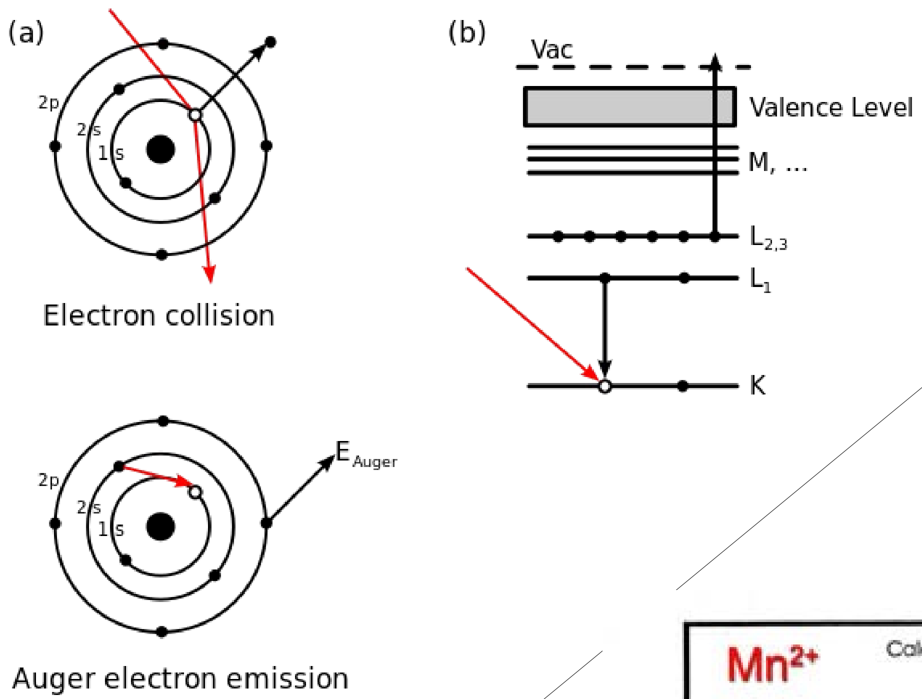
- X-ray emission sent out when an electron emitted from an inner shell and another outer electron fills the hole
- Specific elemental information.
- Basis of EDX.

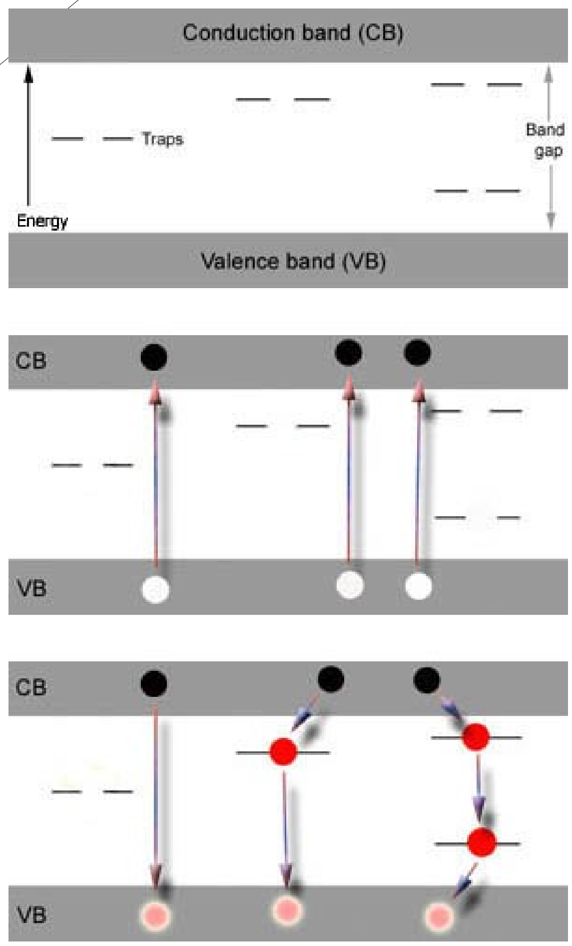


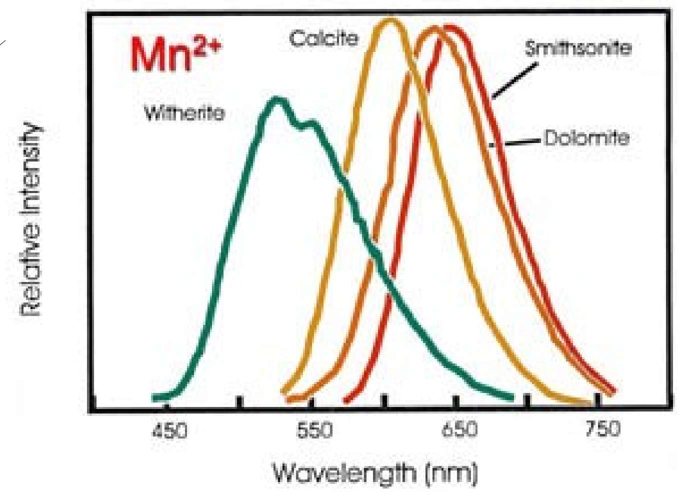
X-ray emission



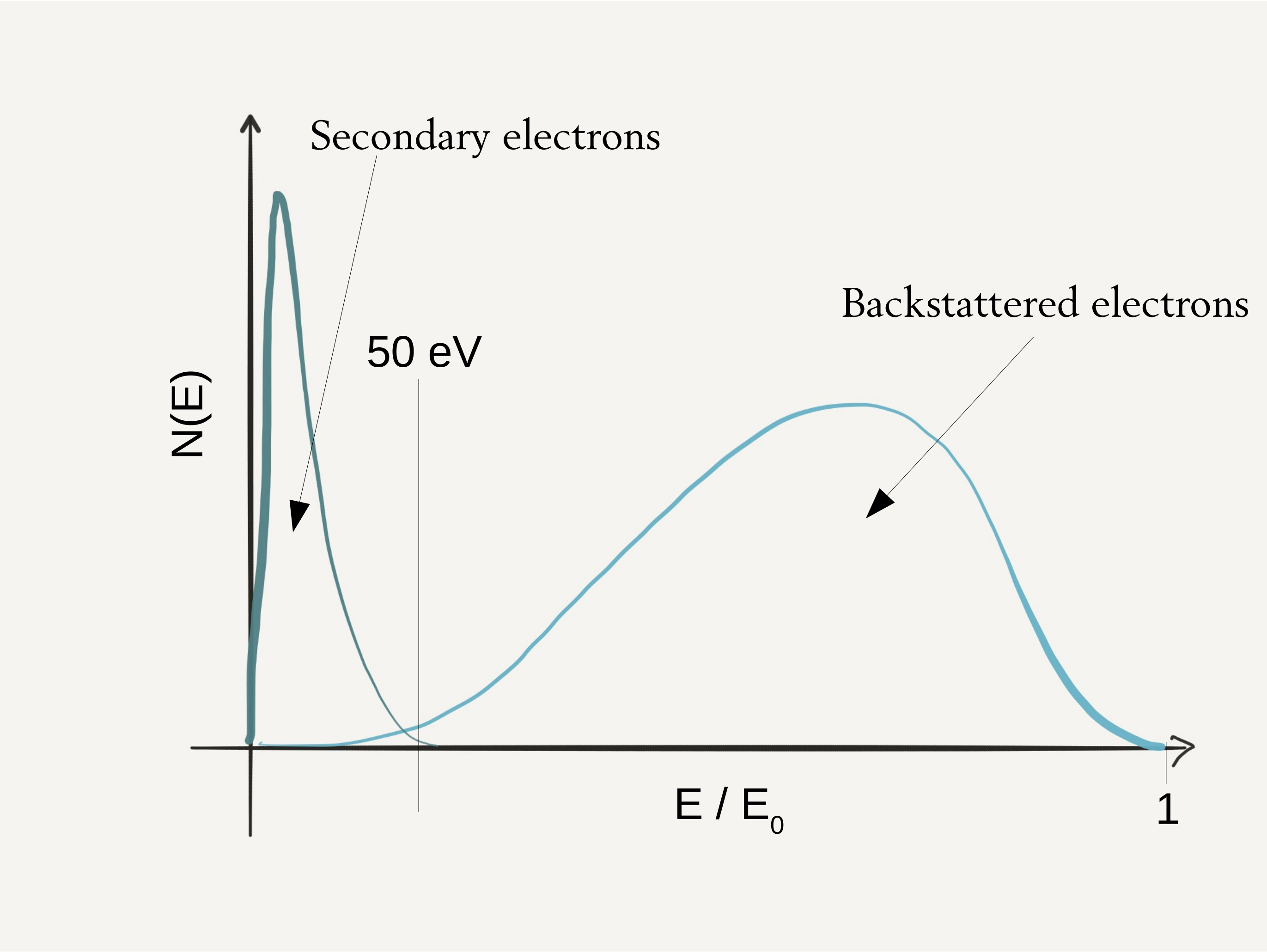
Auger electrons / CL





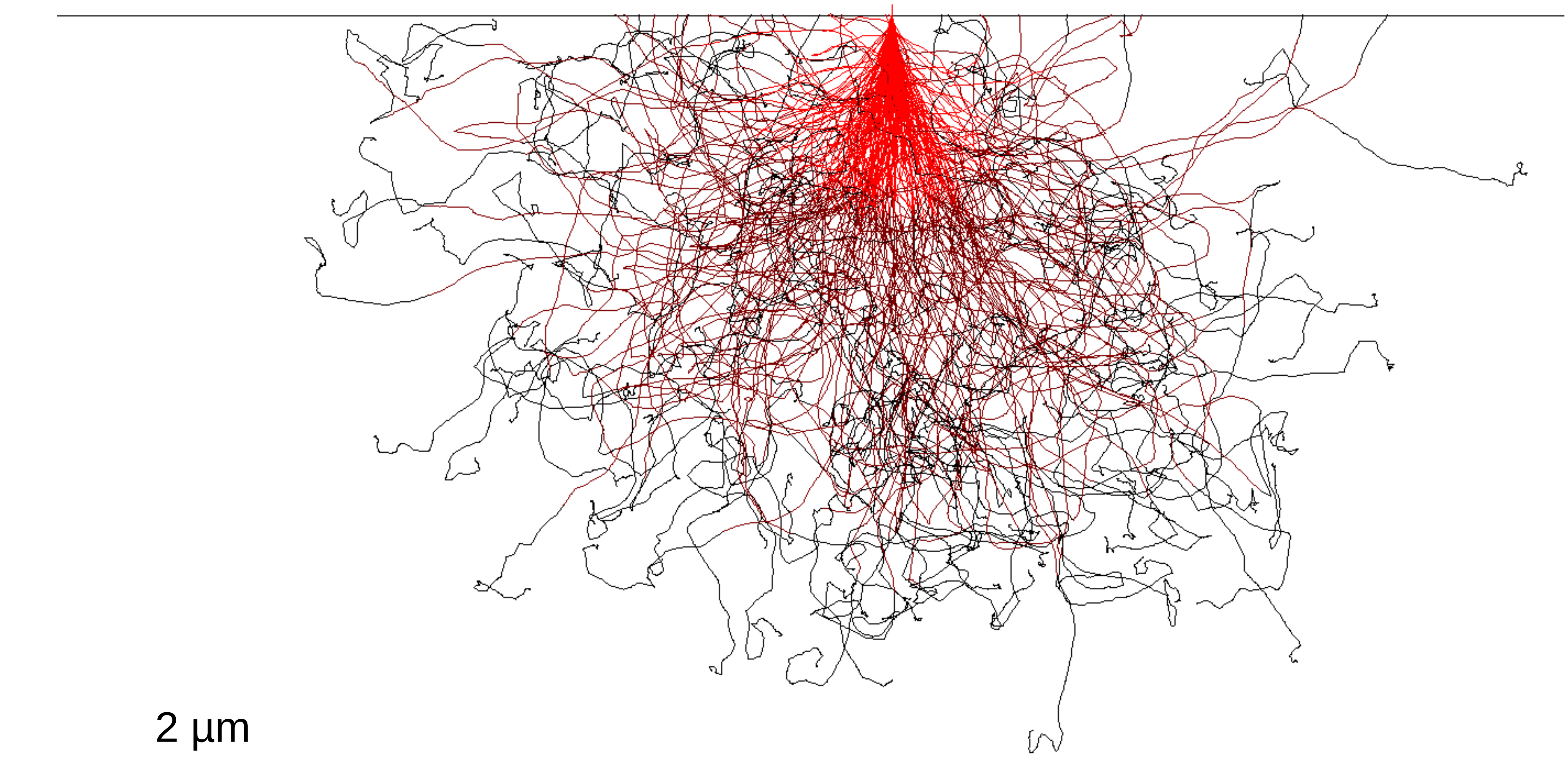


“Types” of electrons

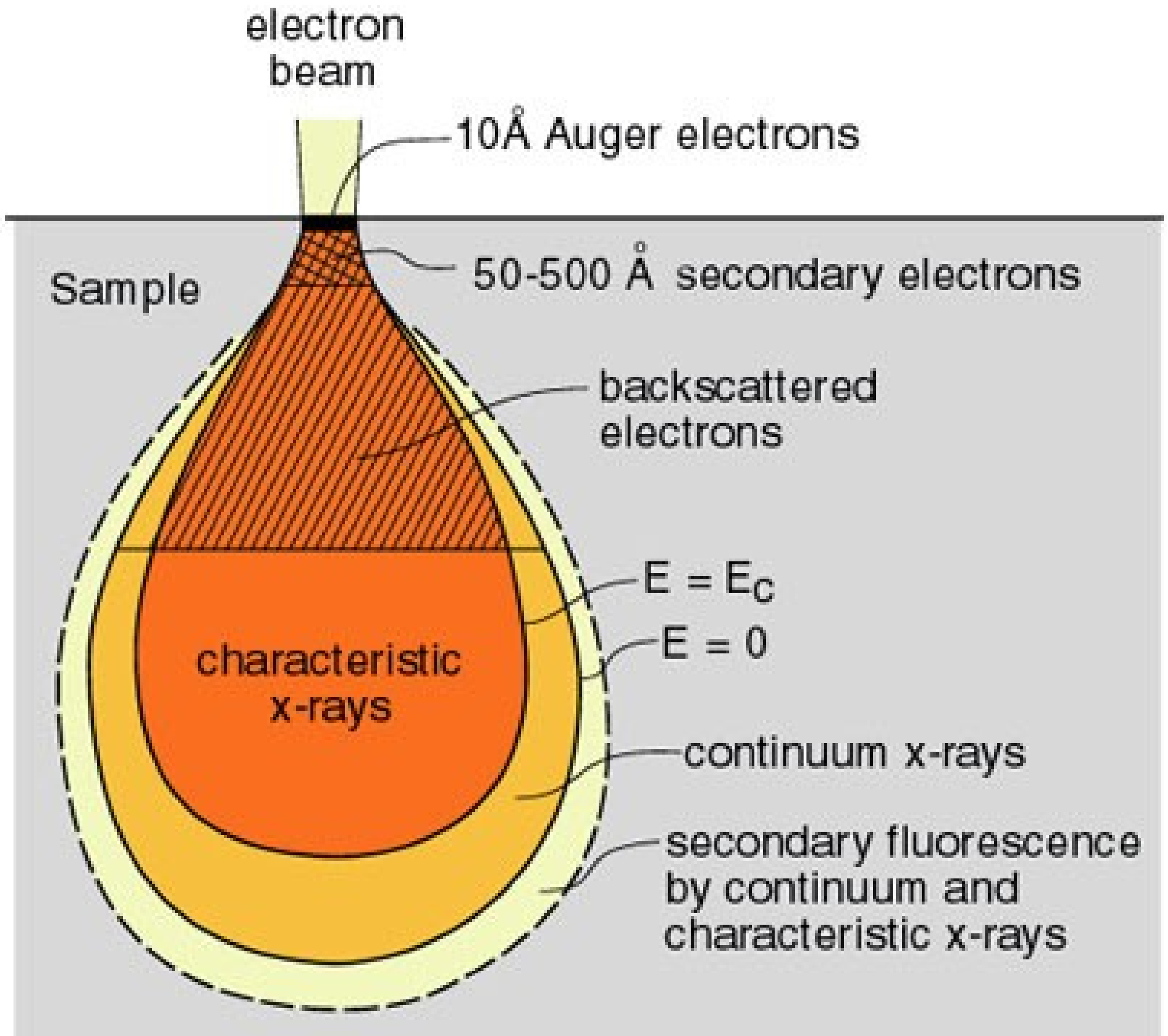


Interaction volume

20 kV beam in Si

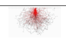


Interaction volume

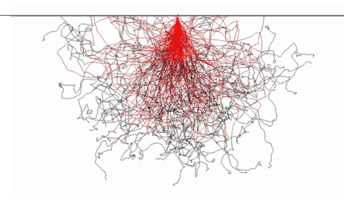


Interaction volume

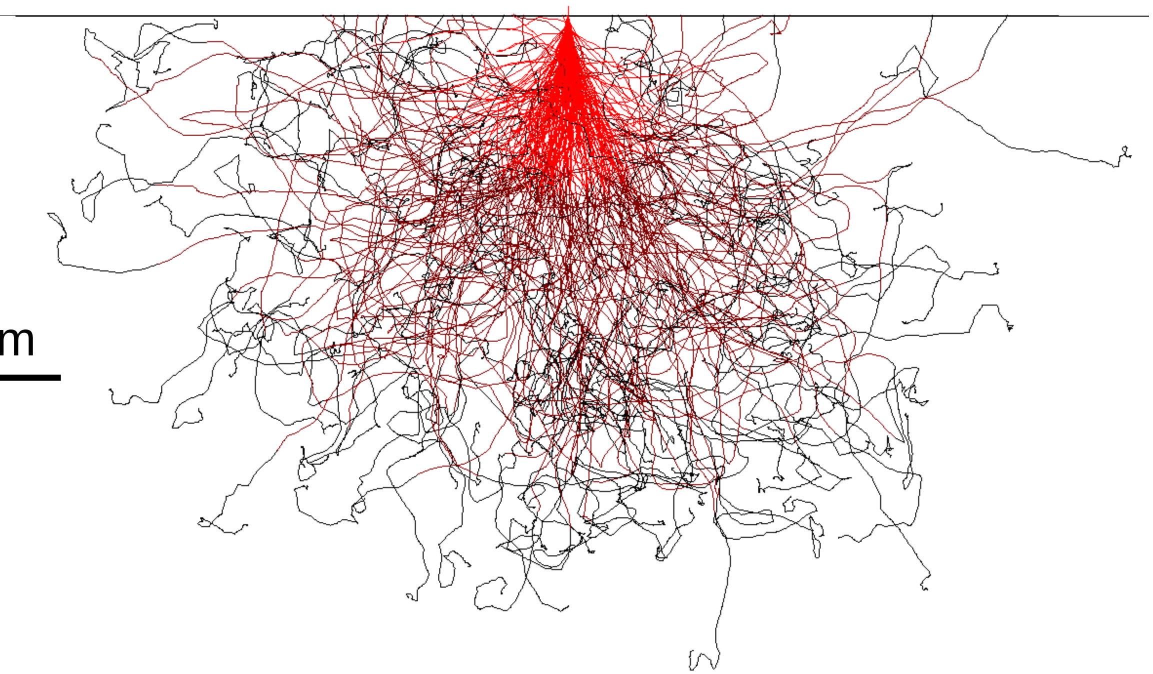
2 kV beam in Si



10 kV beam in Si

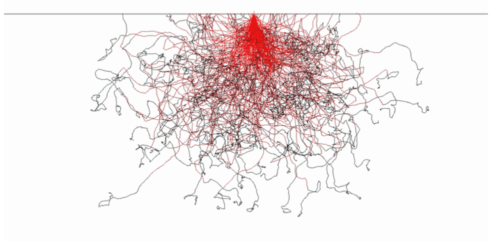


20 kV beam in Si

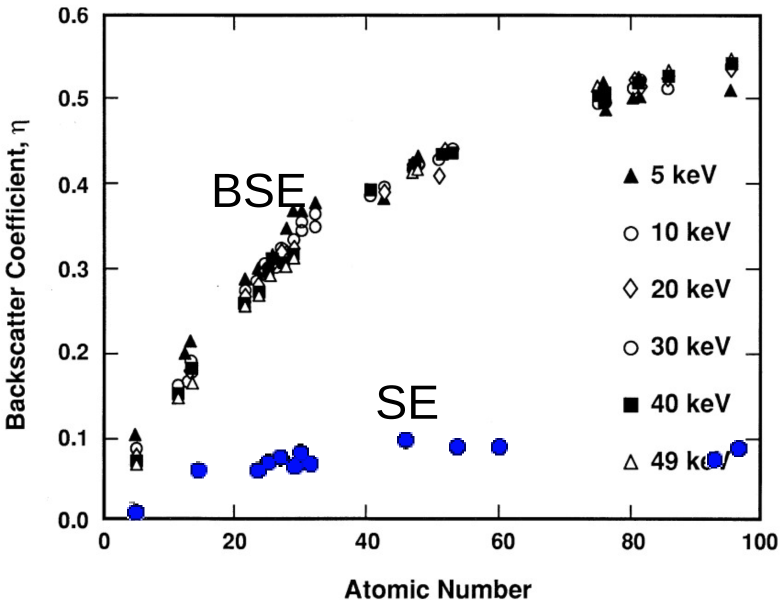


1 µm

20 kV beam in SS

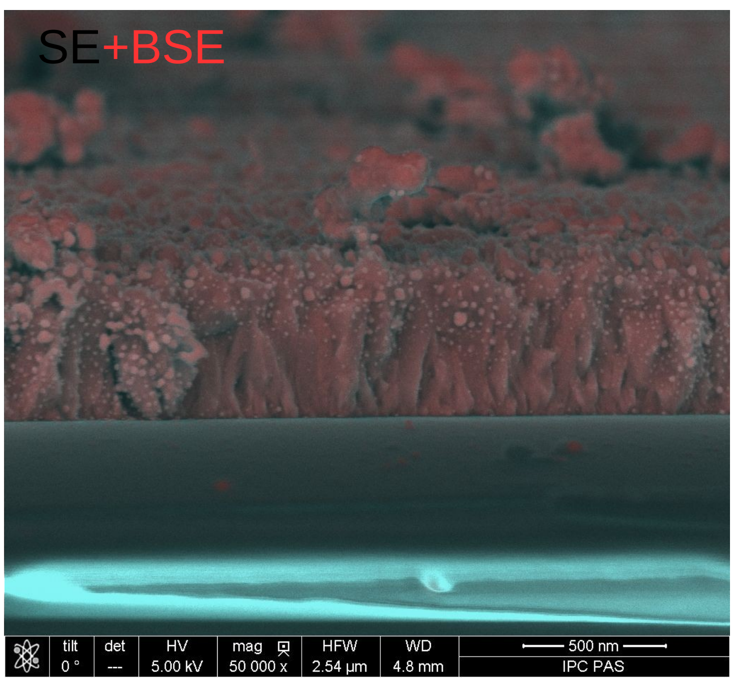


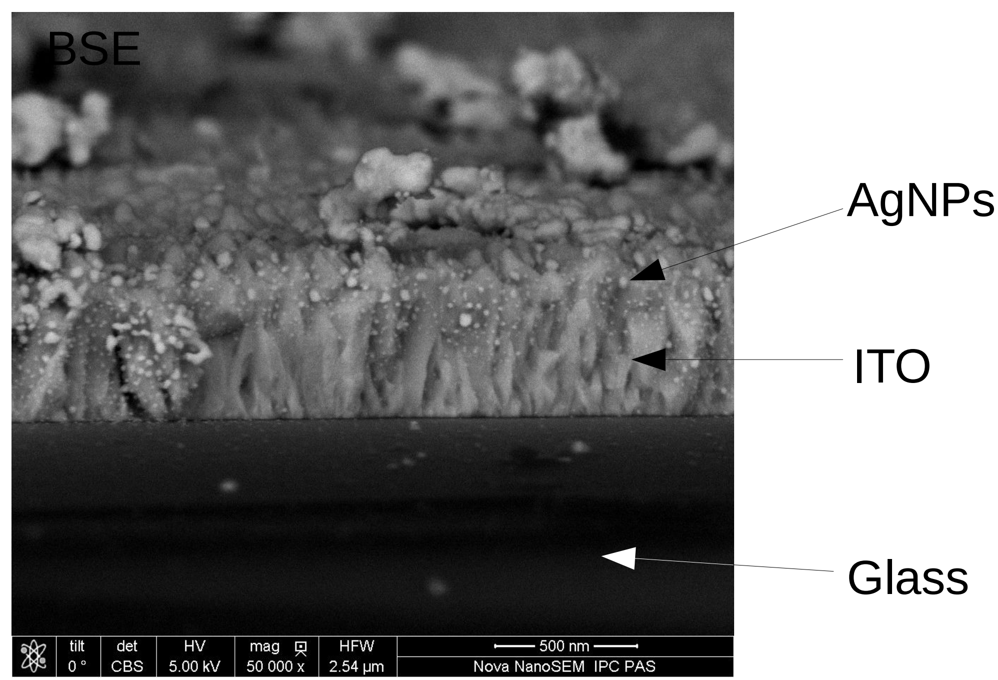
Backscatter efficiency



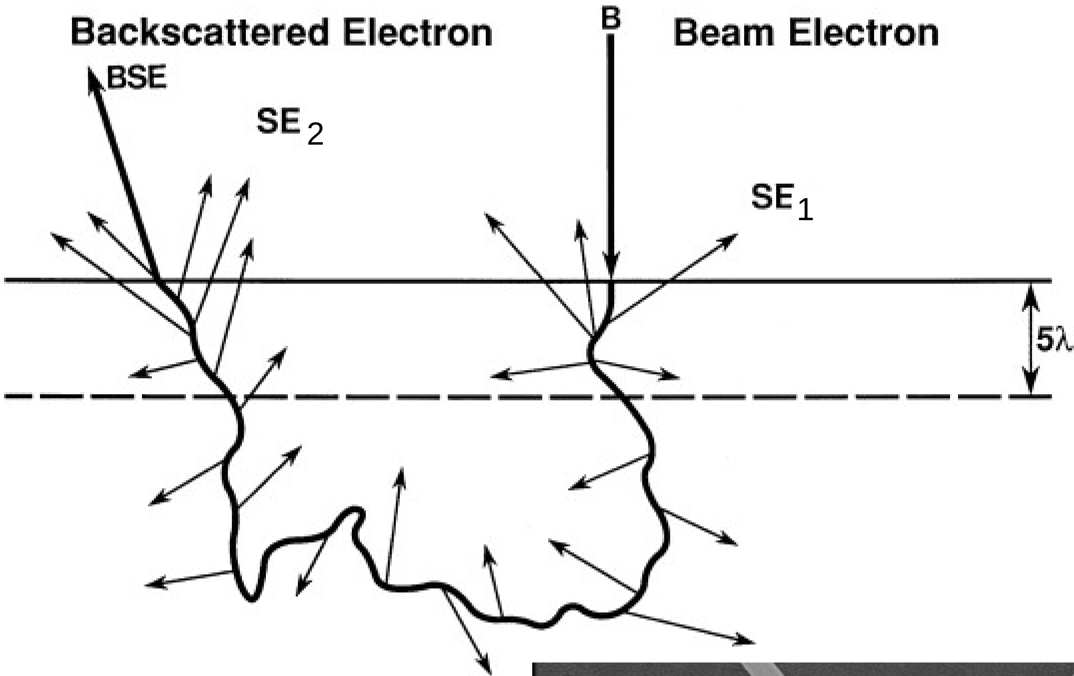
Backscatter efficiency is element dependent, but relatively insensitive to energy.

$$C = \frac{\eta_1 - \eta_2}{\eta_2} \qquad \eta = \sum_i C_i \eta_i,$$





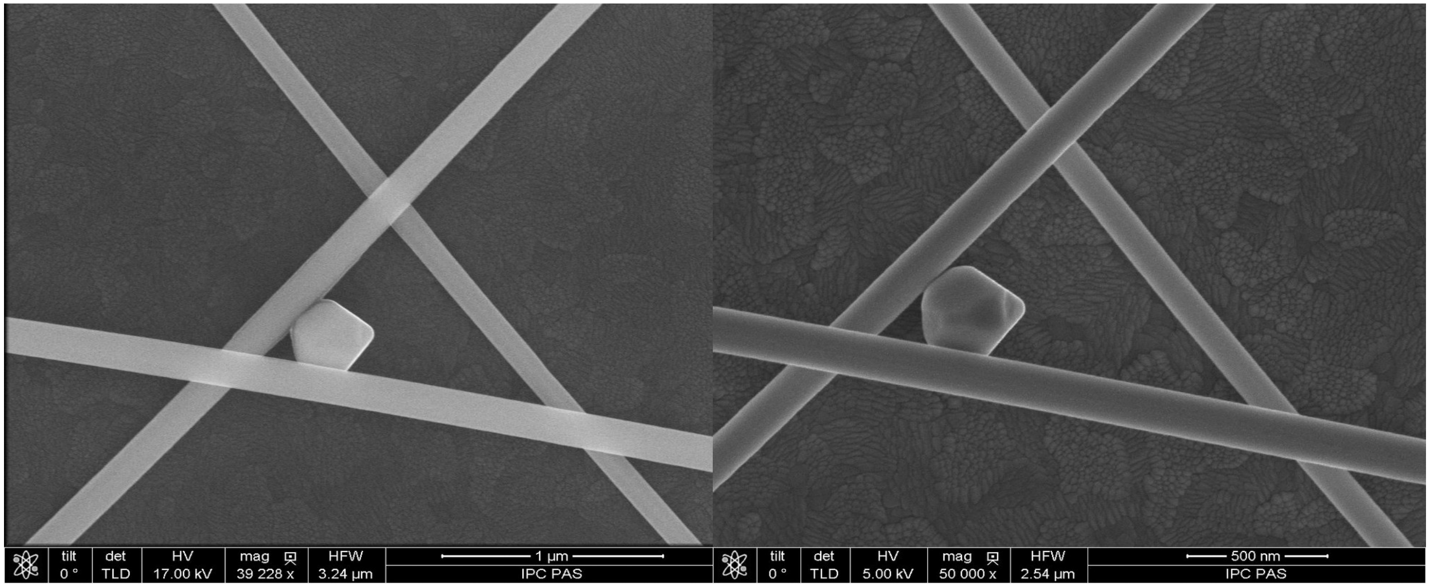
Secondary electons



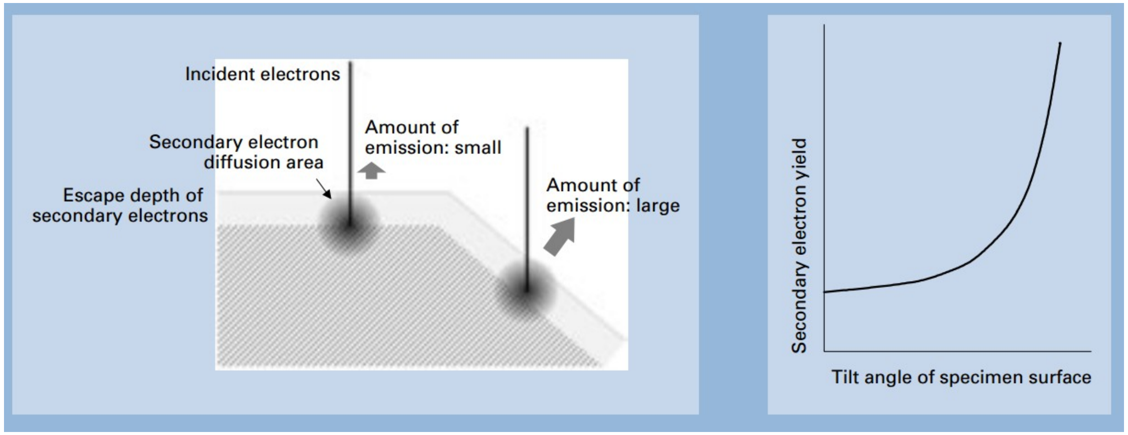
Secondary electrons are created both from the initial beam, and the scattered BSEs. Information about deep structures also in SE.

λ ~ 1 nm for metals
~ 10 nm for insulators

SE images of nanowires with different acceleration voltages.



Secondary electrons



SE yield is strongly angle dependent.
→ bright sides of structures

Edge effect also gives over-saturated edges.

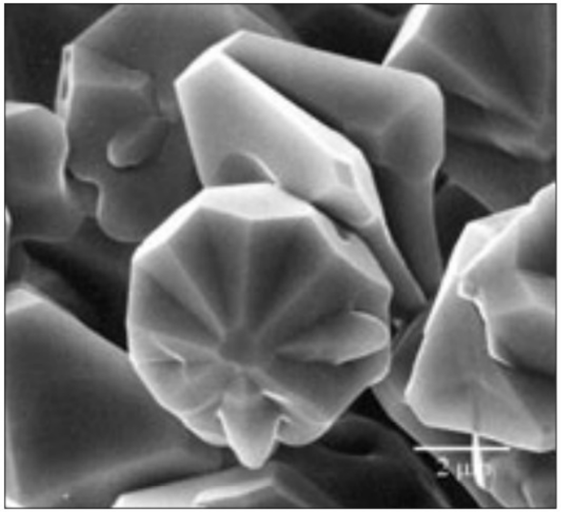
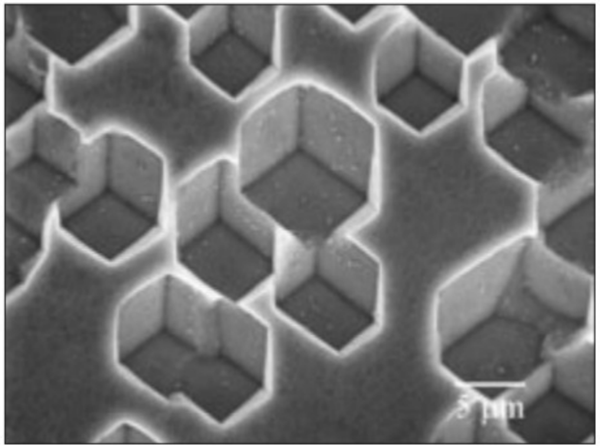
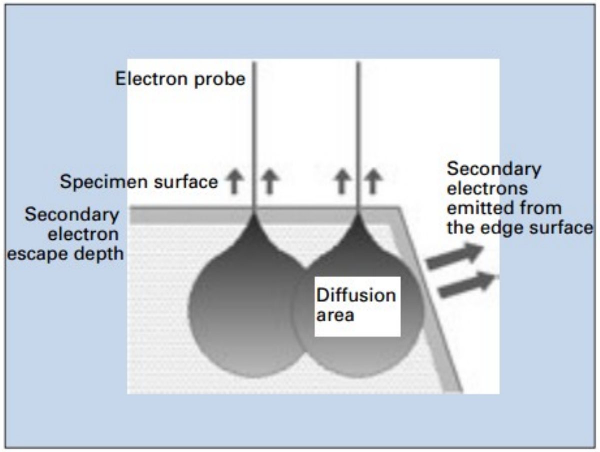


Fig. 15 Secondary electron image of tungsten oxide crystal.





Charging

Charging occurs when the sample, or part of the sample, is not sufficiently conductive.

- → Anomalous contrast
  Too bright, too dark. Often changing over time
- → Distortion

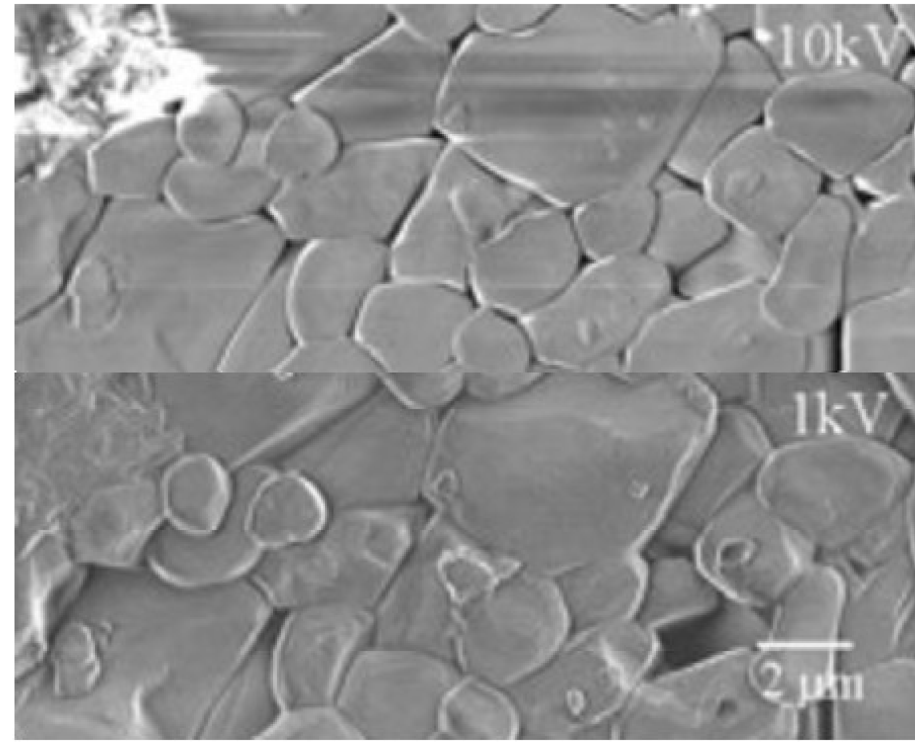


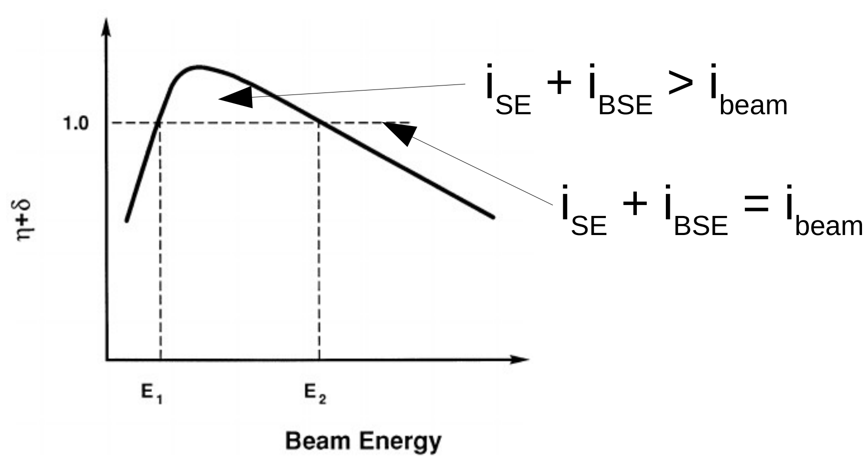
Table 3.5. Secondary Emission as a Function of Energy[a]

Element	5 keV	20 keV	50 keV
Al	0.4	0.1	0.05
Au	0.7	0.2	0.10

[a] After Reimer and Tollkamp (1980).

Table 3.6. Upper Crossover Energy for Various Materials (Normal Beam Incidence)

Material	E_2(keV)	Reference
Kapton	0.4	Joy (unpublished)
Electron resist	0.55–0.70	Joy(1987)
Nylon	1.18	Joy (unpublished)
5% PB7/nylon	1.40	Krause *et al.* (1987)
Acetal	1.65	Vaz (1986)
Polyvinyl chloride	1.65	Vaz (1986)
Teflon	1.82	Vaz and Krause (1986)
Glass passivation	2.0	Joy (1987)
GaAs	2.6	Joy (1987)
Quartz	3.0	Joy (1987)
Alumina	4.2	Joy (unpublished)



How to avoid charging?

- Lower voltage
- Lower current

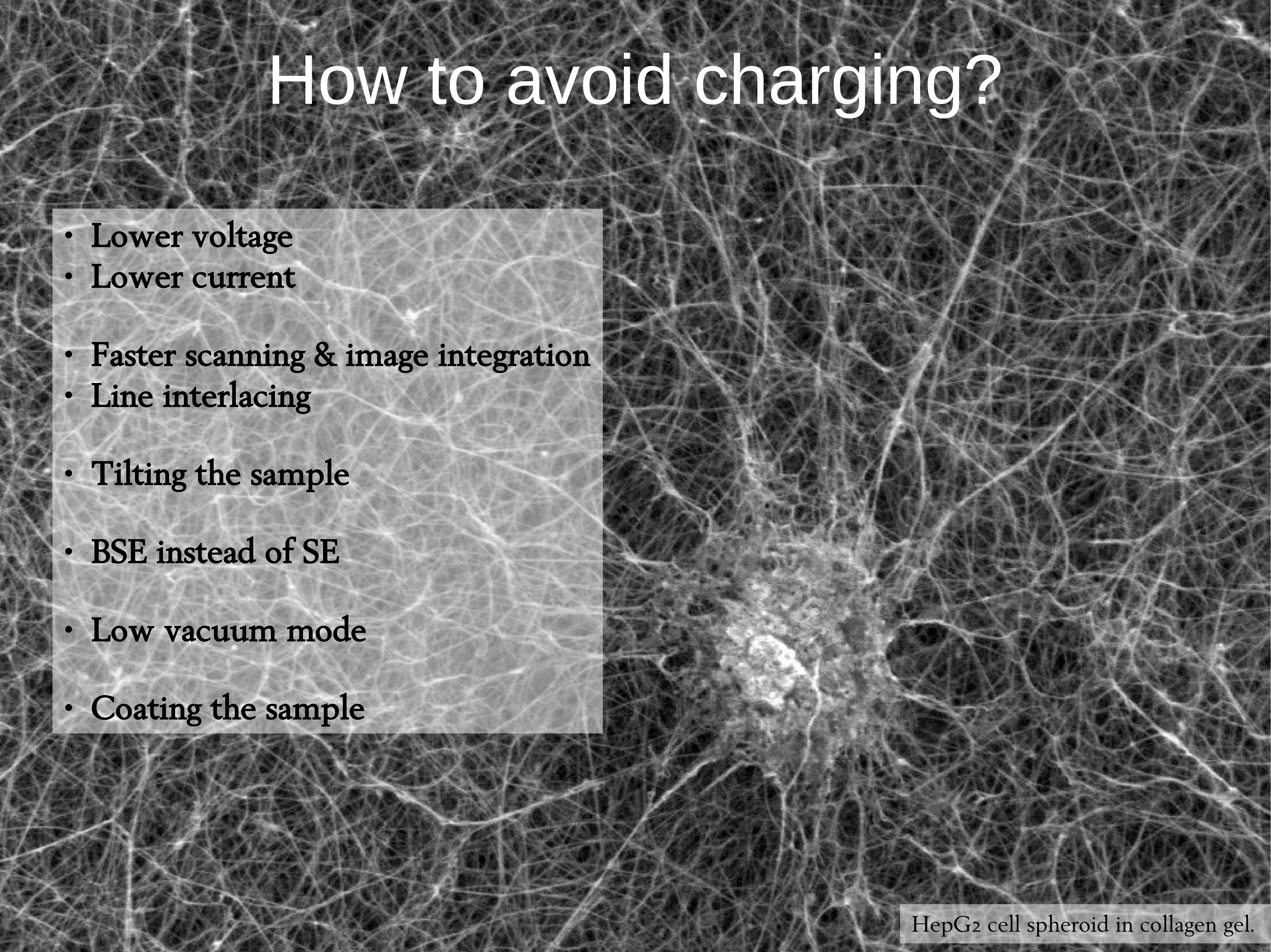
- Faster scanning & image integration
- Line interlacing

- Tilting the sample

- BSE instead of SE

- Low vacuum mode

- Coating the sample



HepG2 cell spheroid in collagen gel.