Introduction to Electron Microscopy and Spectroscopy

March 21, 2019

Outlines

Electron microscopy and spectroscopy

Basic electron optics

Interaction of high energy (~kV) electrons with sample materials

Image contrast mechanism

Instruments and techniques

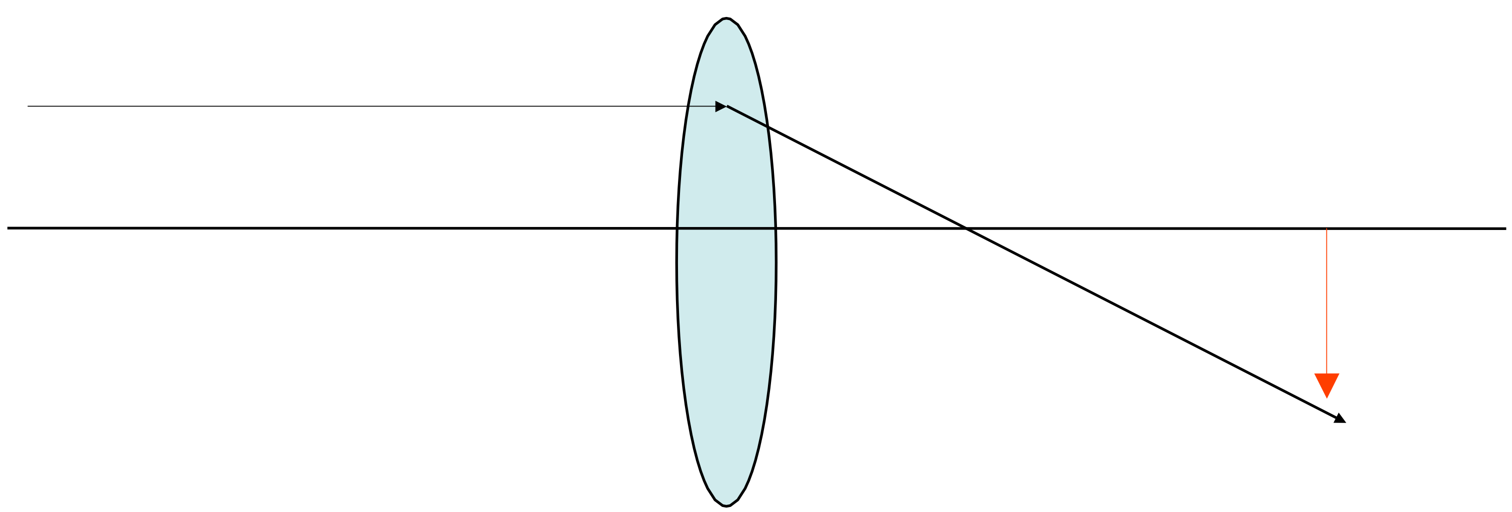
Electron microscopy and microanalysis: aims and means

Microscopies: morphologies in small scales (micrometer or nanometer)
Optical microscopy, Electron microscopy, Ion microscopy, Scanning probe microscopy….., offer images only.

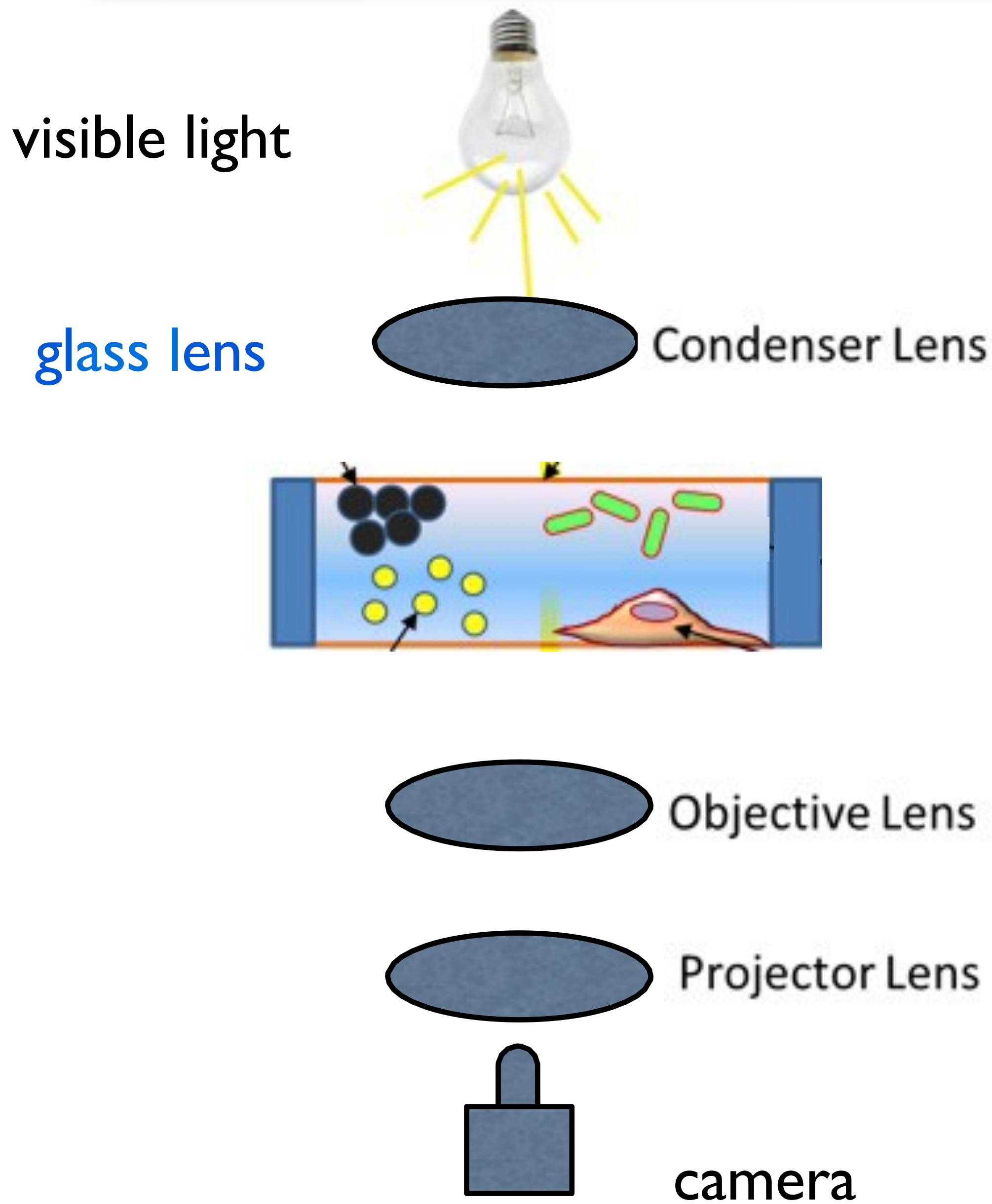
Microanalyses: composition and/or structures in small scales (micrometer or nanometer)
Energy Dispersive Spectroscopy, Wave-length Dispersive Spectroscopy, Electron Energy Loss Spectroscopy, Auger Electron Spectroscopy, Convergent Beam Electron Diffraction, Select Area Diffraction….., offer spectra and/or diffraction patterns

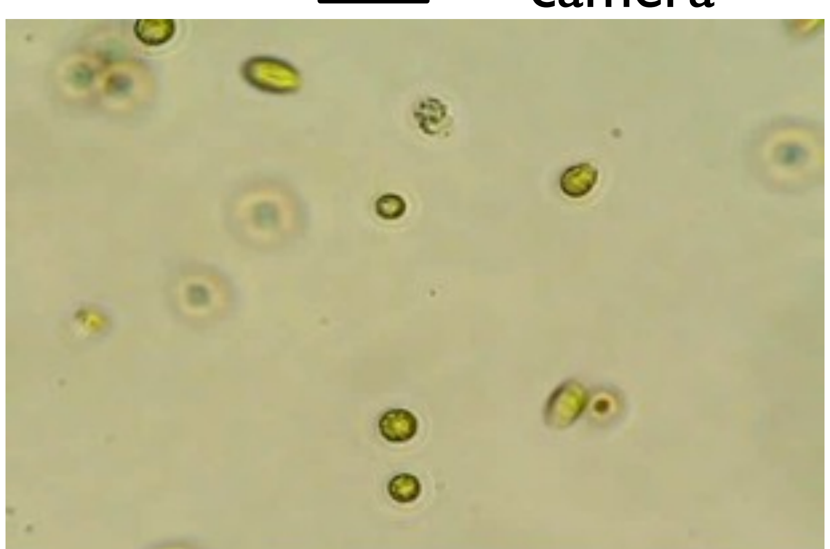
1.1 Why Electron Microscope?

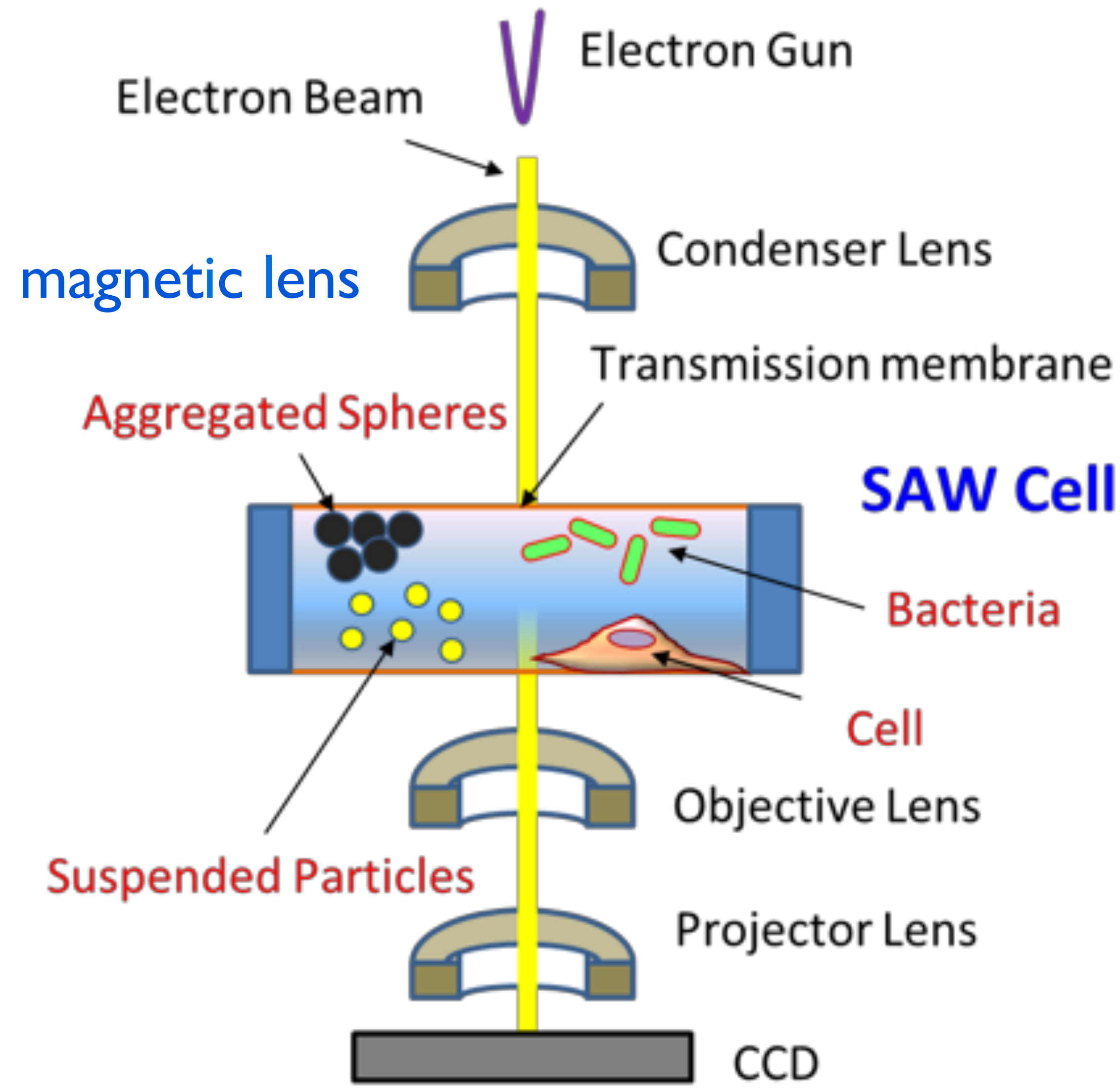
	Optical Microscope	Electron Microscope
source	visible light	electron
lens	glass	electro-magnetic lens



Optical Microscope Electron Microscope

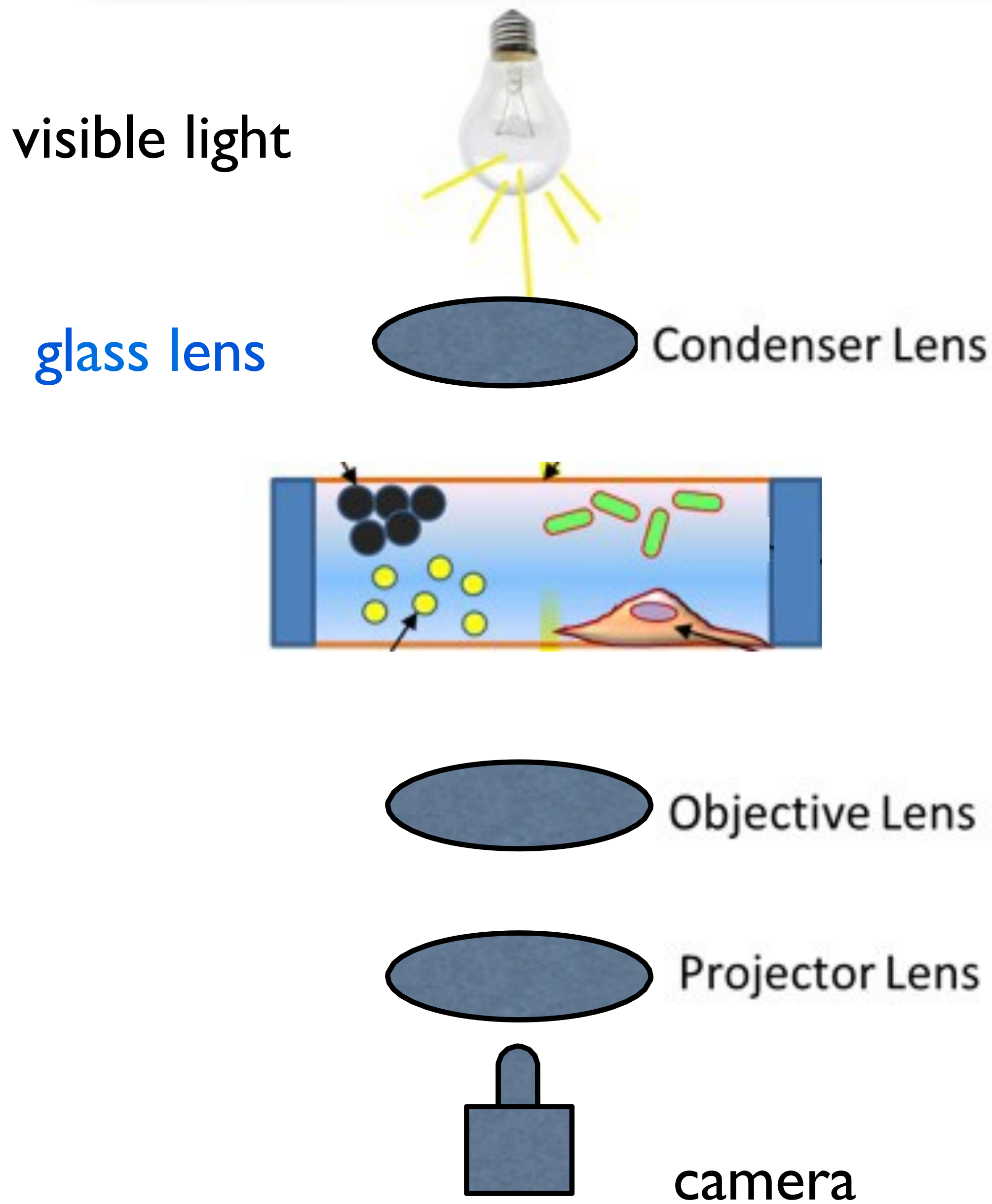






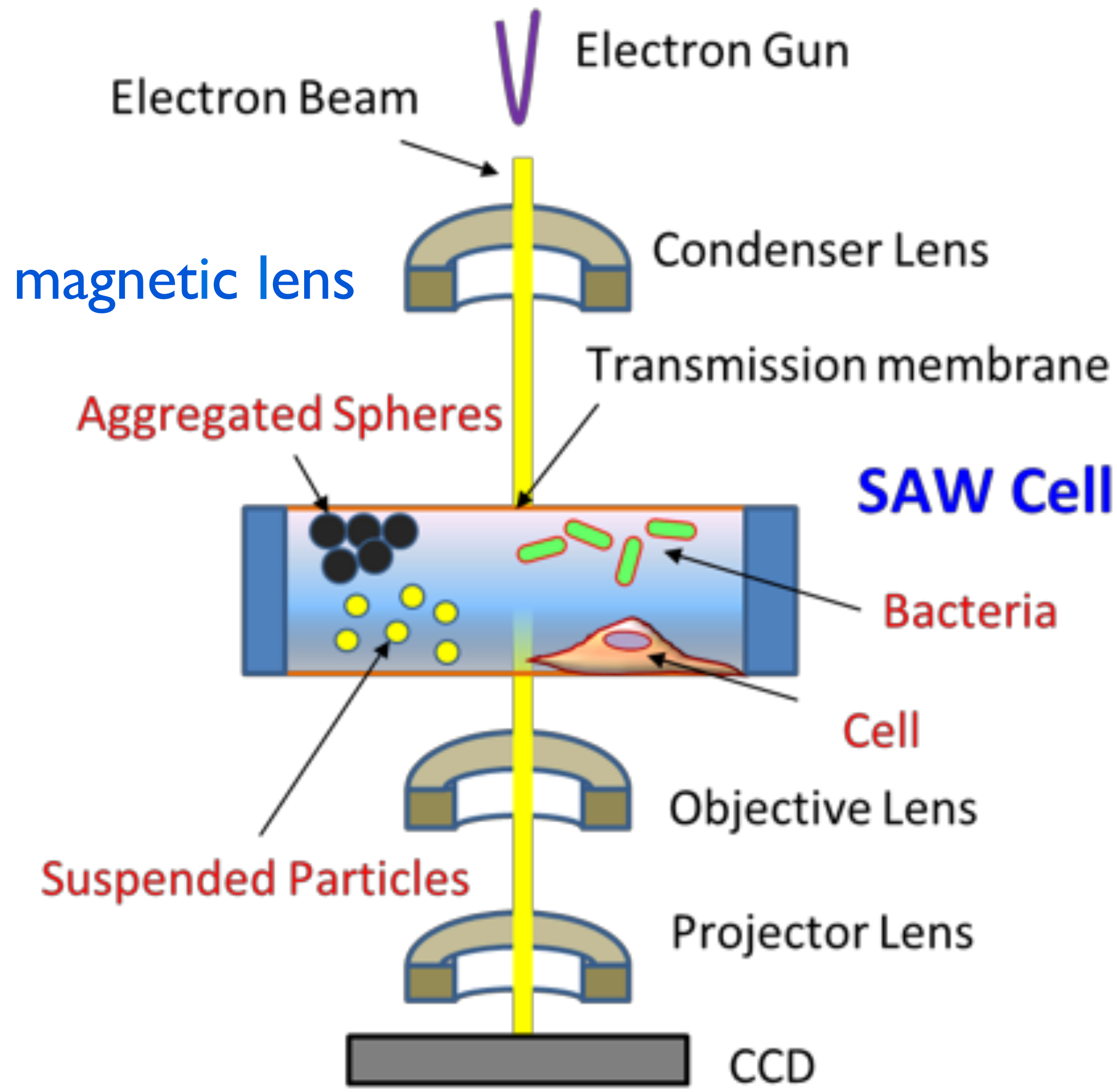
Liquid TEM image

Optical Microscope Electron Microscope



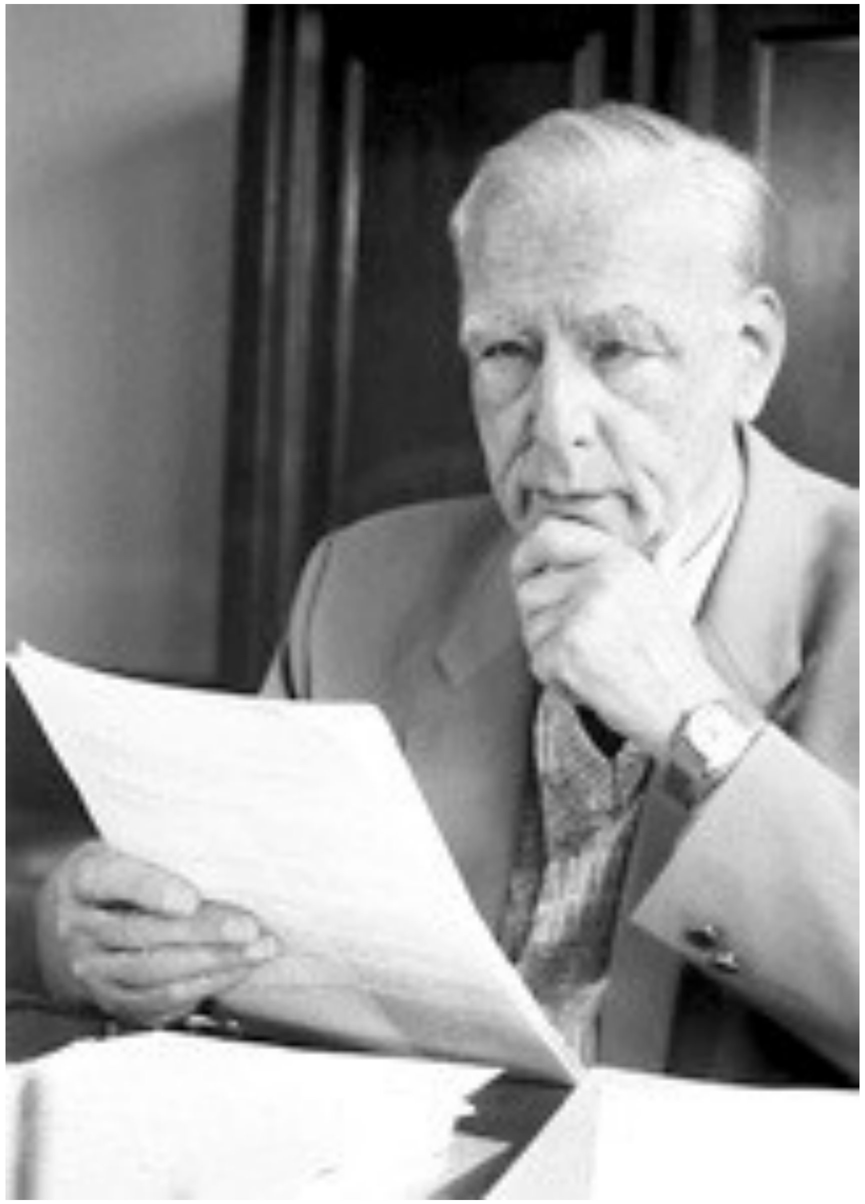
衣藻

Chlamydomonas Reinhardtii



Liquid TEM image

Ernst Ruska



Nobel Prize in Physics 1986

Better Electron Microscope

.... If we can see where the atoms are，then the analysis of chemical substance become very easy ......

The most difficulty is the power of electron microscope must be improved 100 times..

-Richard F. Feynman
-12.29.1959 American Physics Society, CIT

A. Resolution

A the resolution of an optical microscope is limited by the wavelength 。

- visible light : λ~ 6000Å

Rayleigh resolution

$$\delta = \frac{0.61\lambda}{\mu \mathrm{Sin}\beta} \qquad (1.1)$$

δ:Rayleigh resolution

λ: wavelength

u: refractive index

β: semi-angle of lens

Numerical Aperature~1

- resolution of optical microscope is about half of wavelength ~3000Å(1000 atoms)

Particle/ Wave Duality

Louis de Broglie

Nobel laureate in 1929

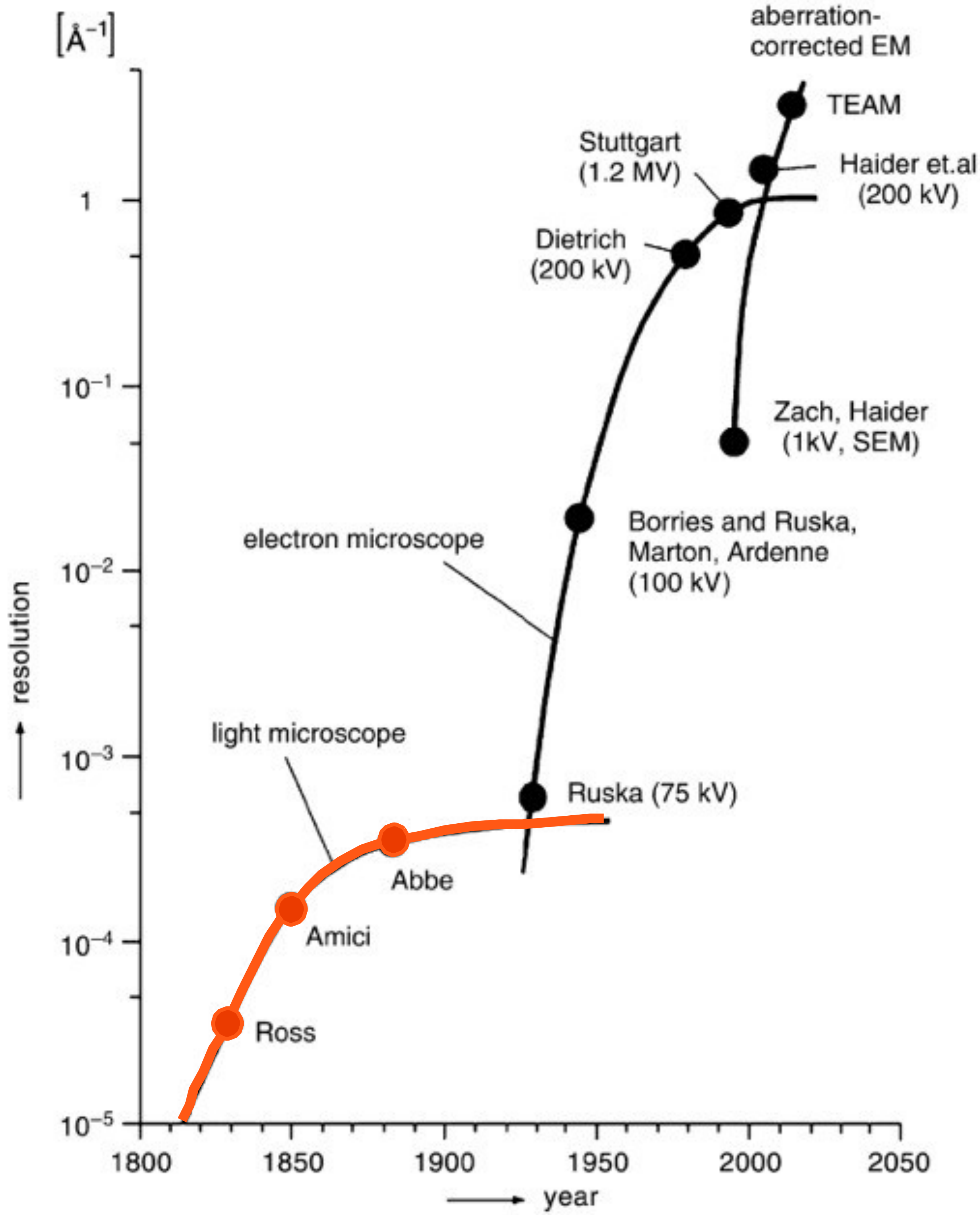
- de Broglie's matter/ wave theory

$$\lambda \sim \frac{1.22}{E^{1/2}}$$

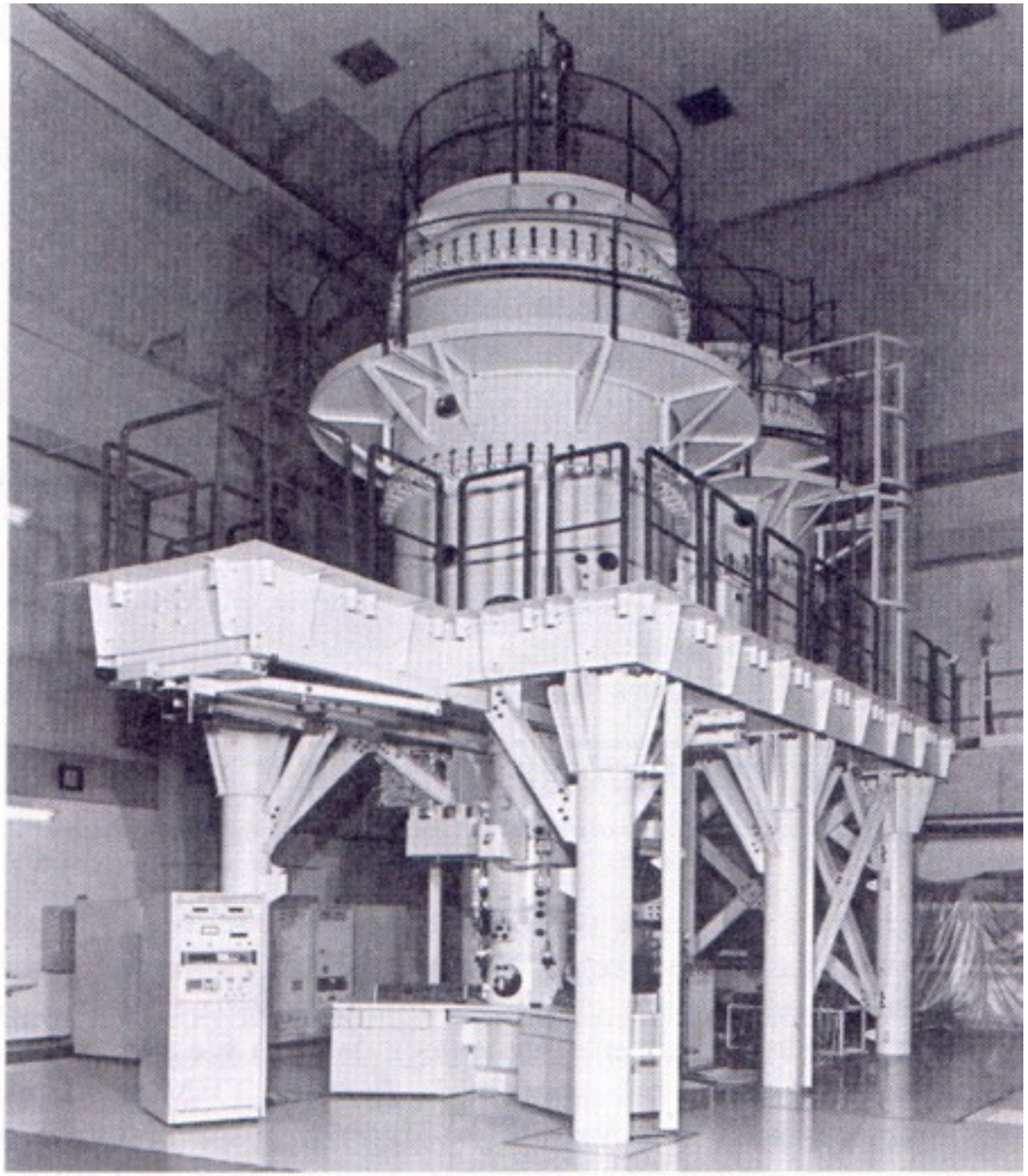
$$E = 1/2\ m_o \upsilon^2$$

E (kev)	λ(Å)
100	0.037
200	0.025
300	0.0196
400	0.0169

Evolution of resolution in EM



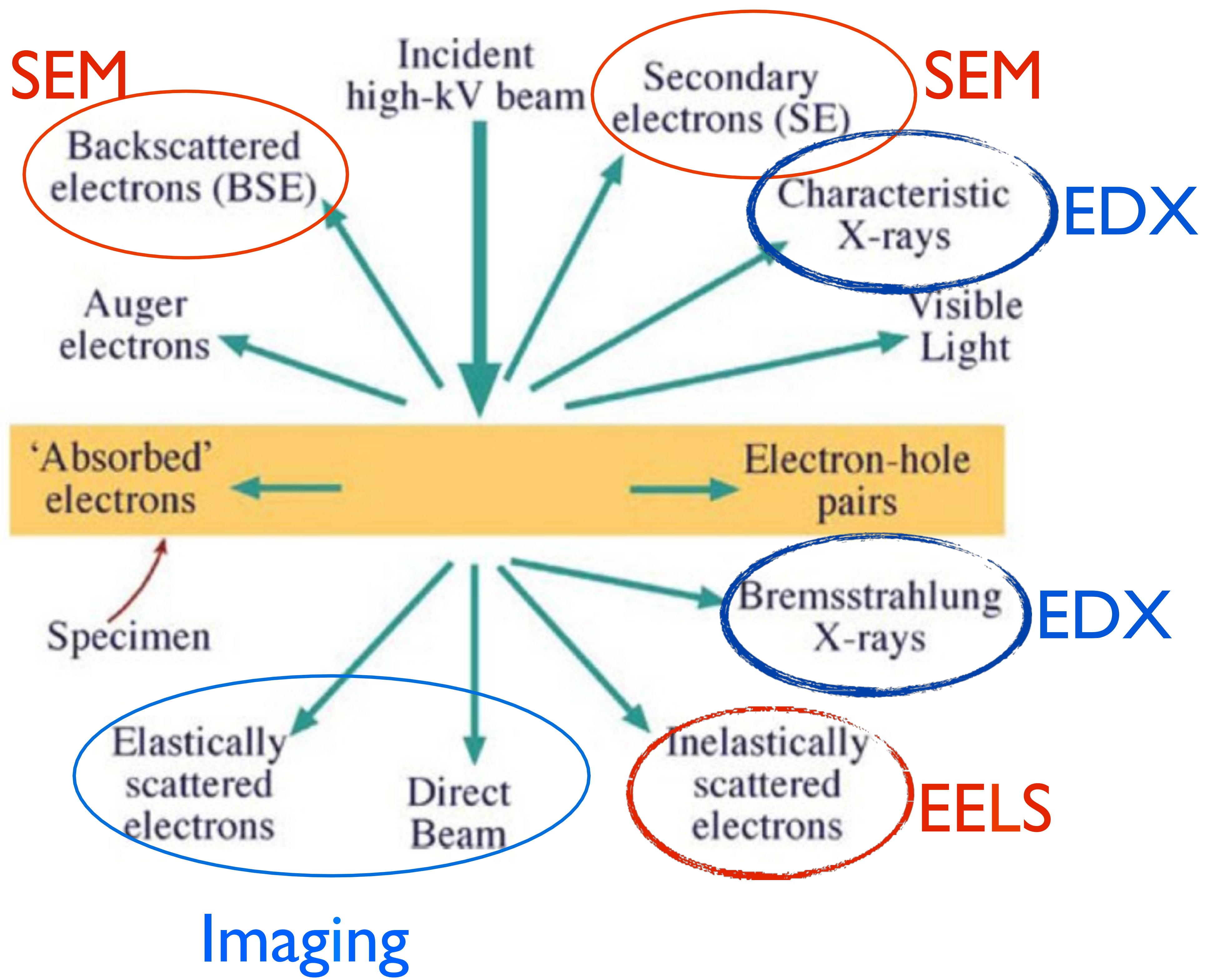
High and Median Voltage TEM



200keV

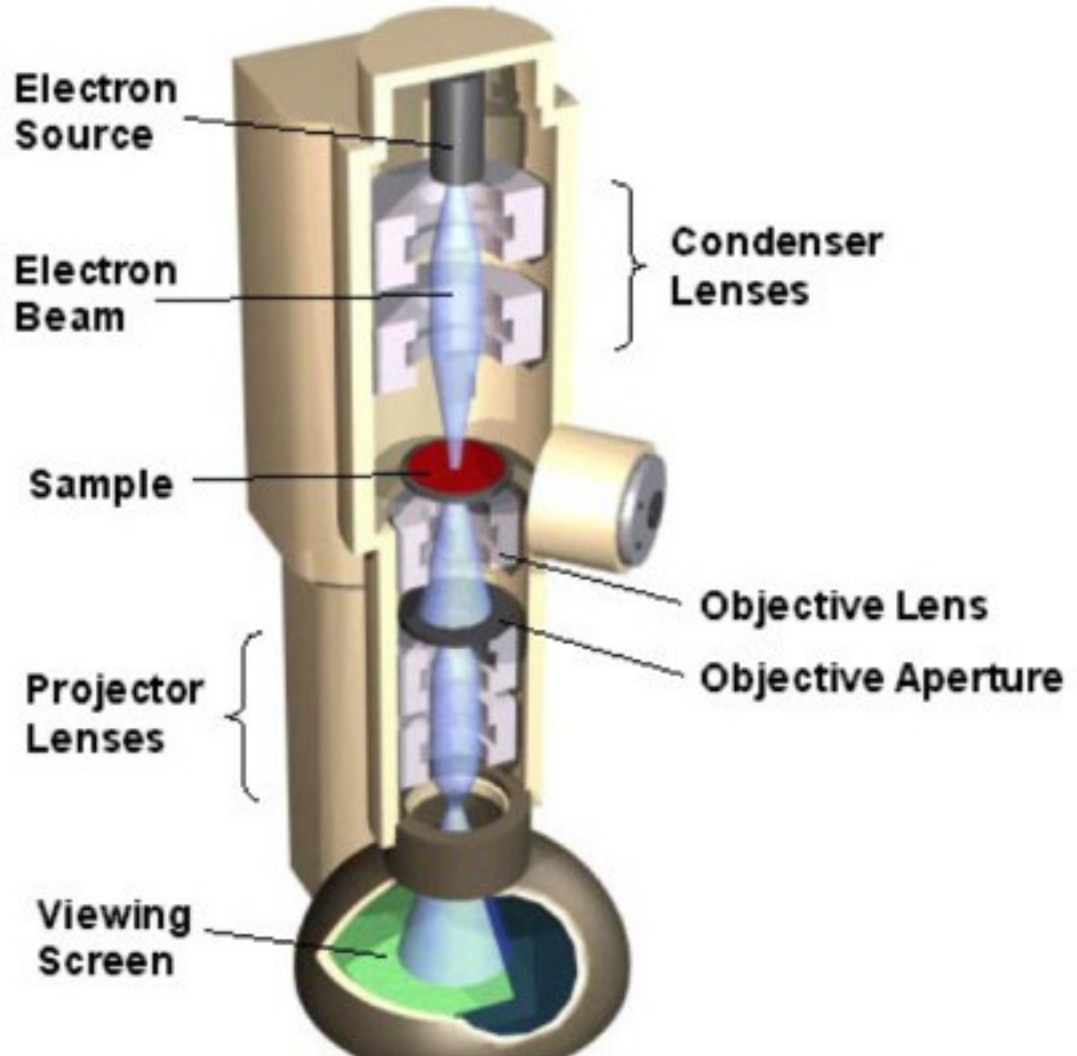
1MeV

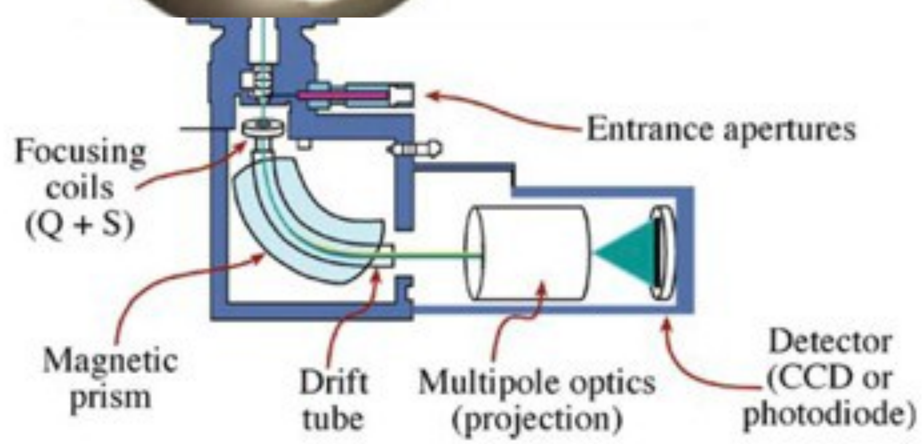
B. MIcroanalysis，(X-Ray)，(EELS)

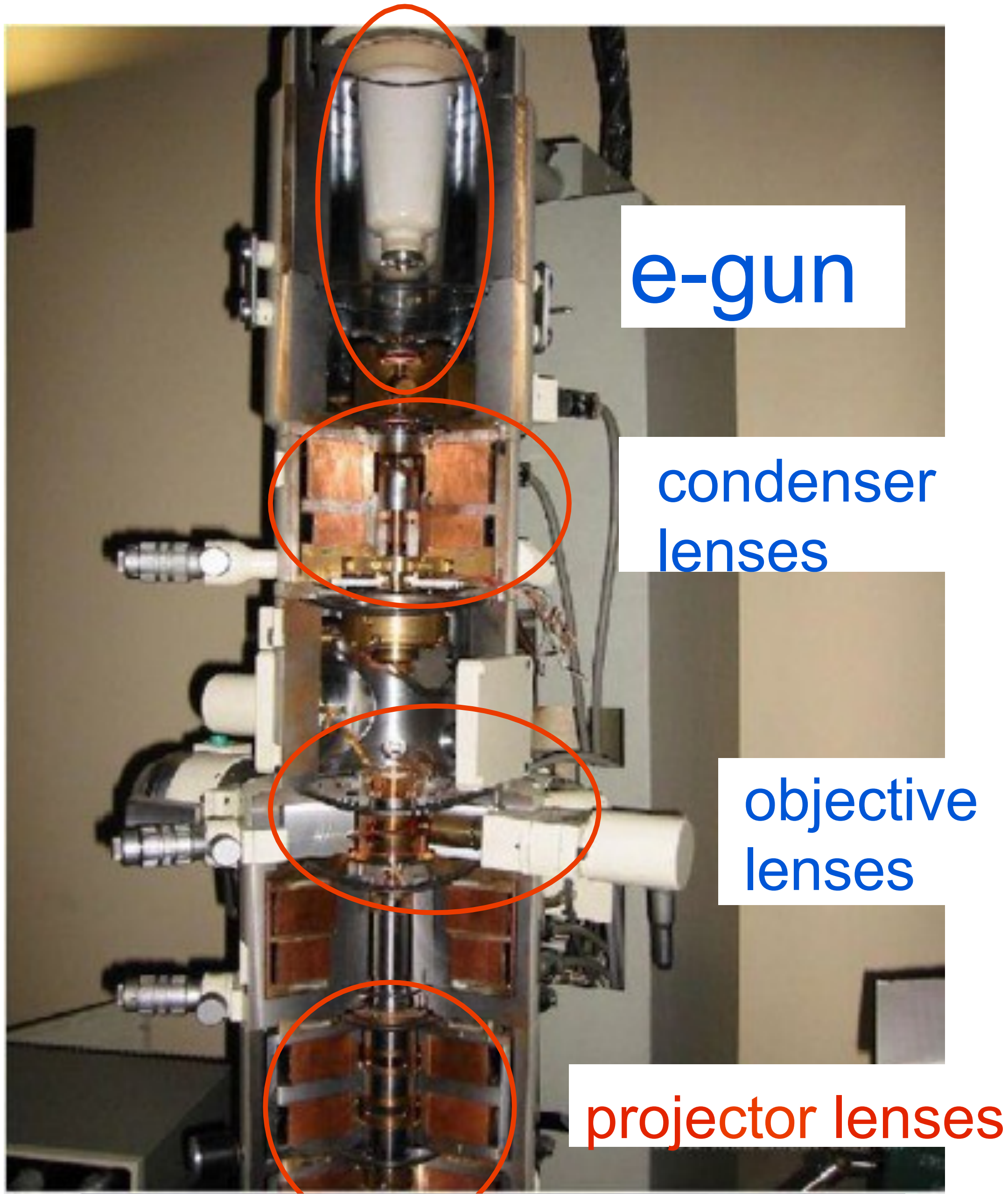


Typical TEM+EDX+EELS

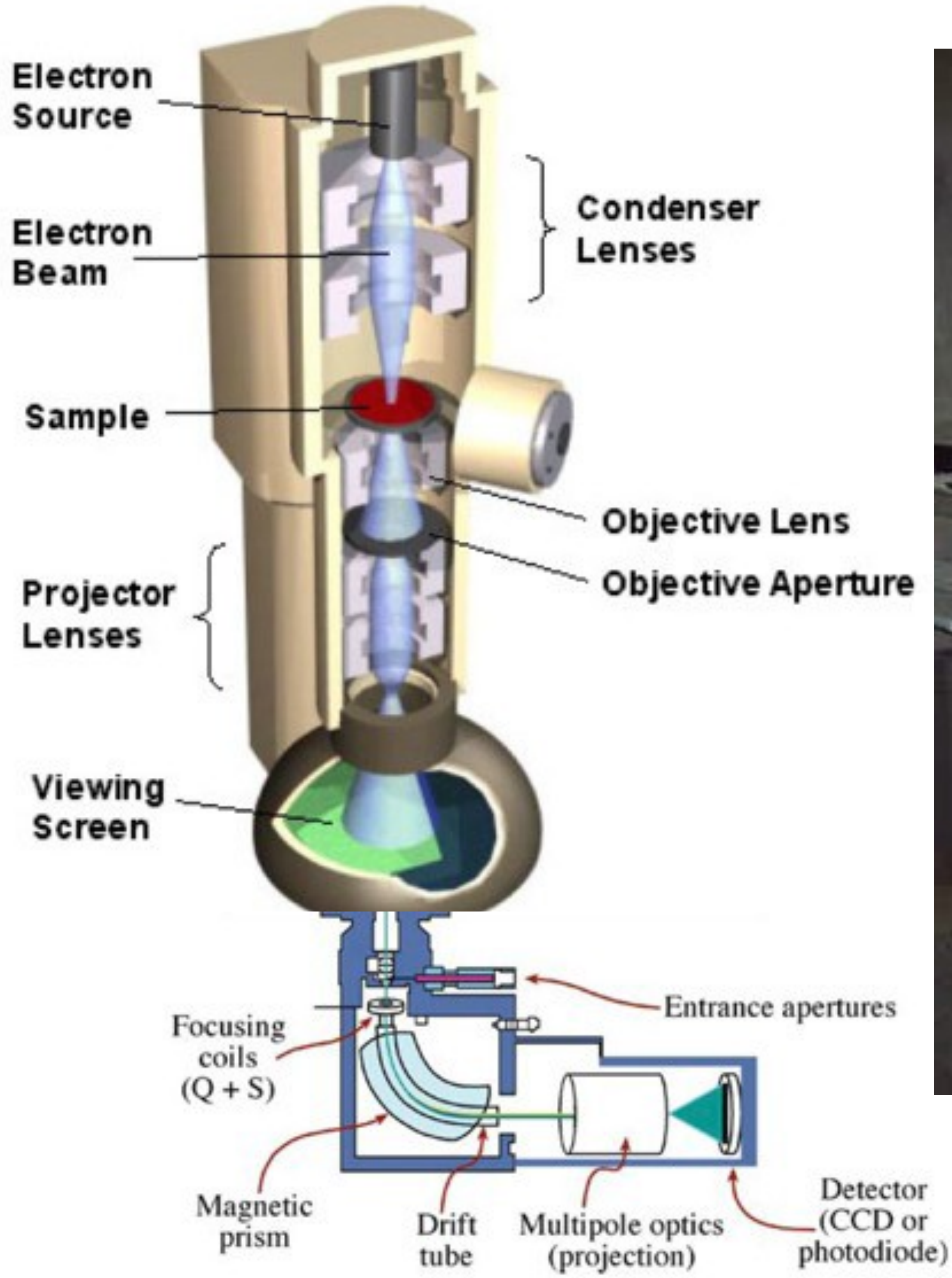
Typical TEM+EDX+EELS



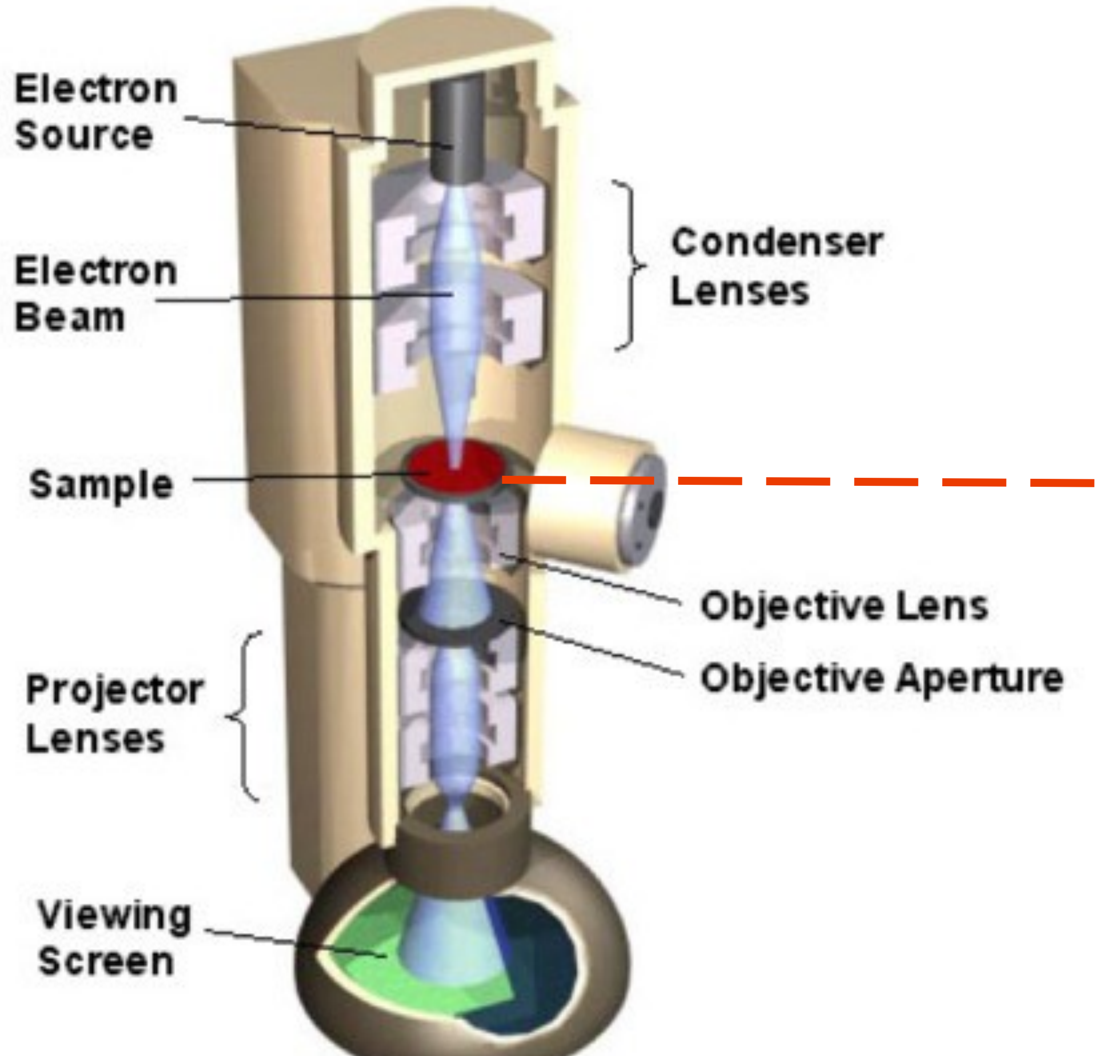


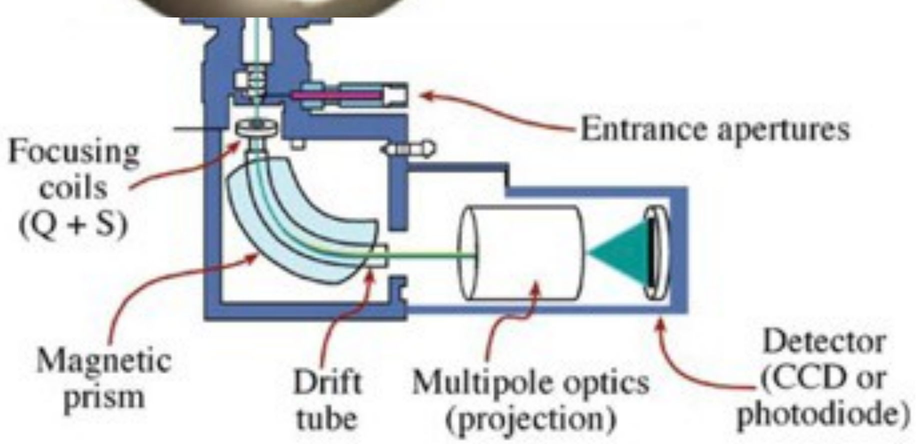


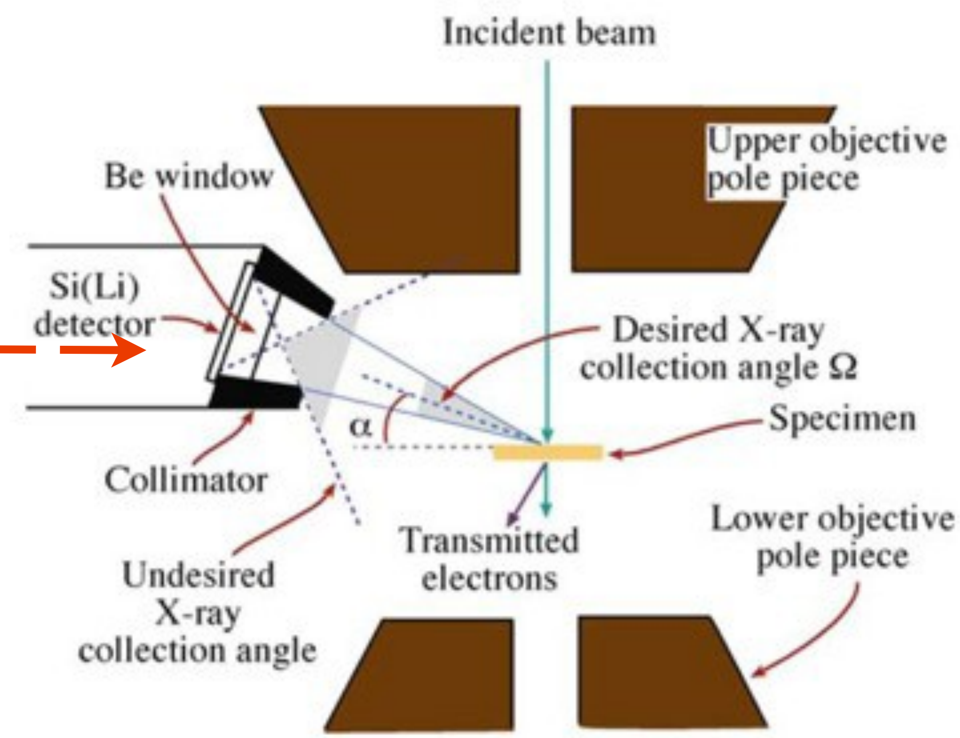
Typical TEM+EDX+EELS

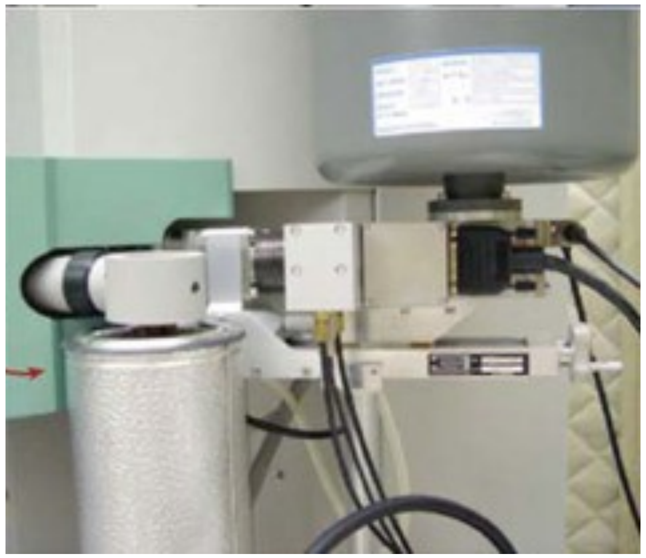


Typical TEM+EDX+EELS





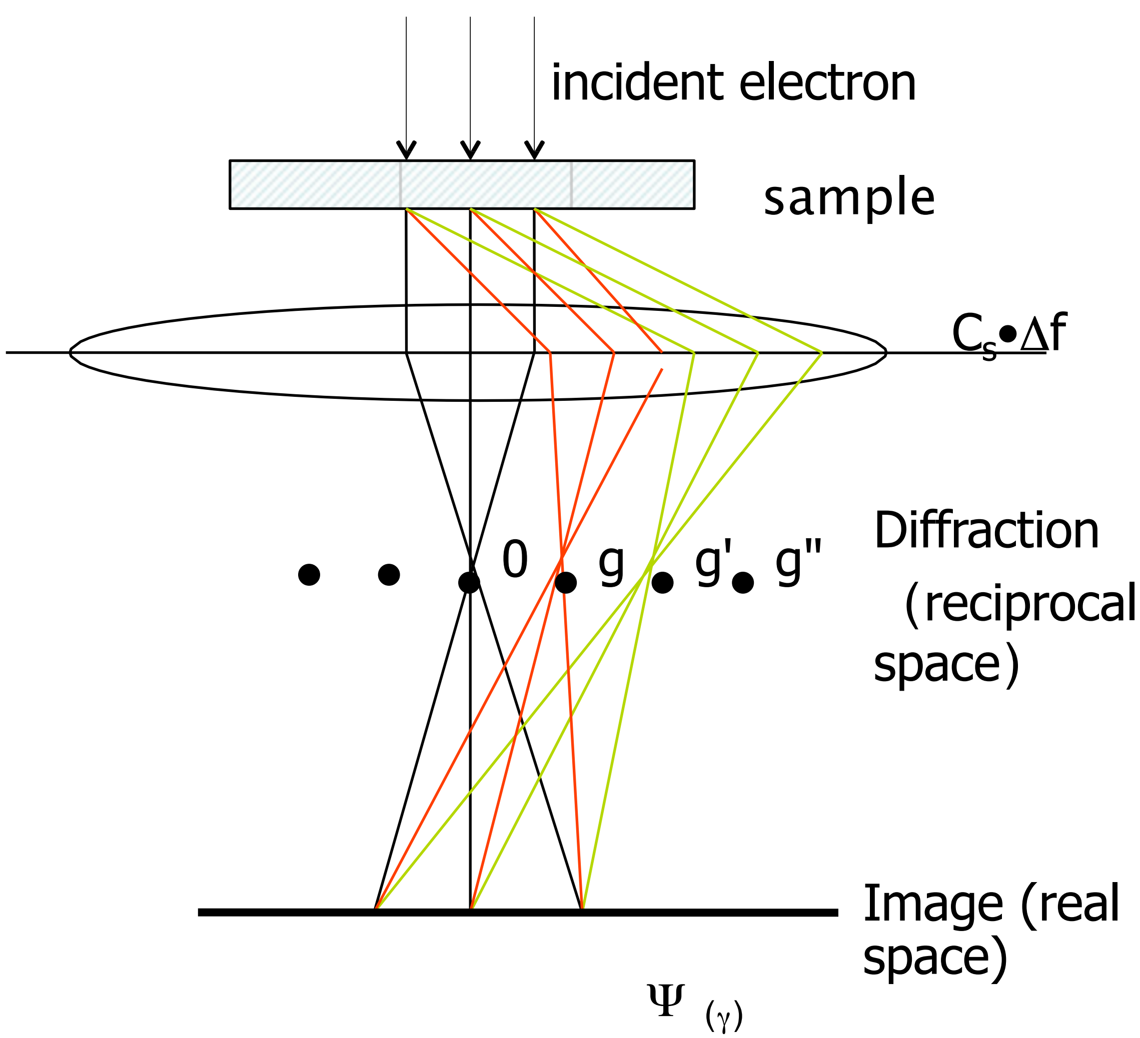


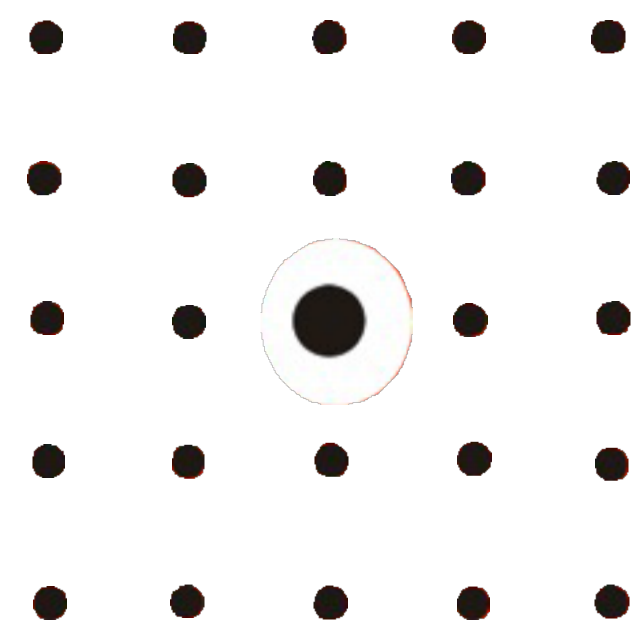


C. Diffraction

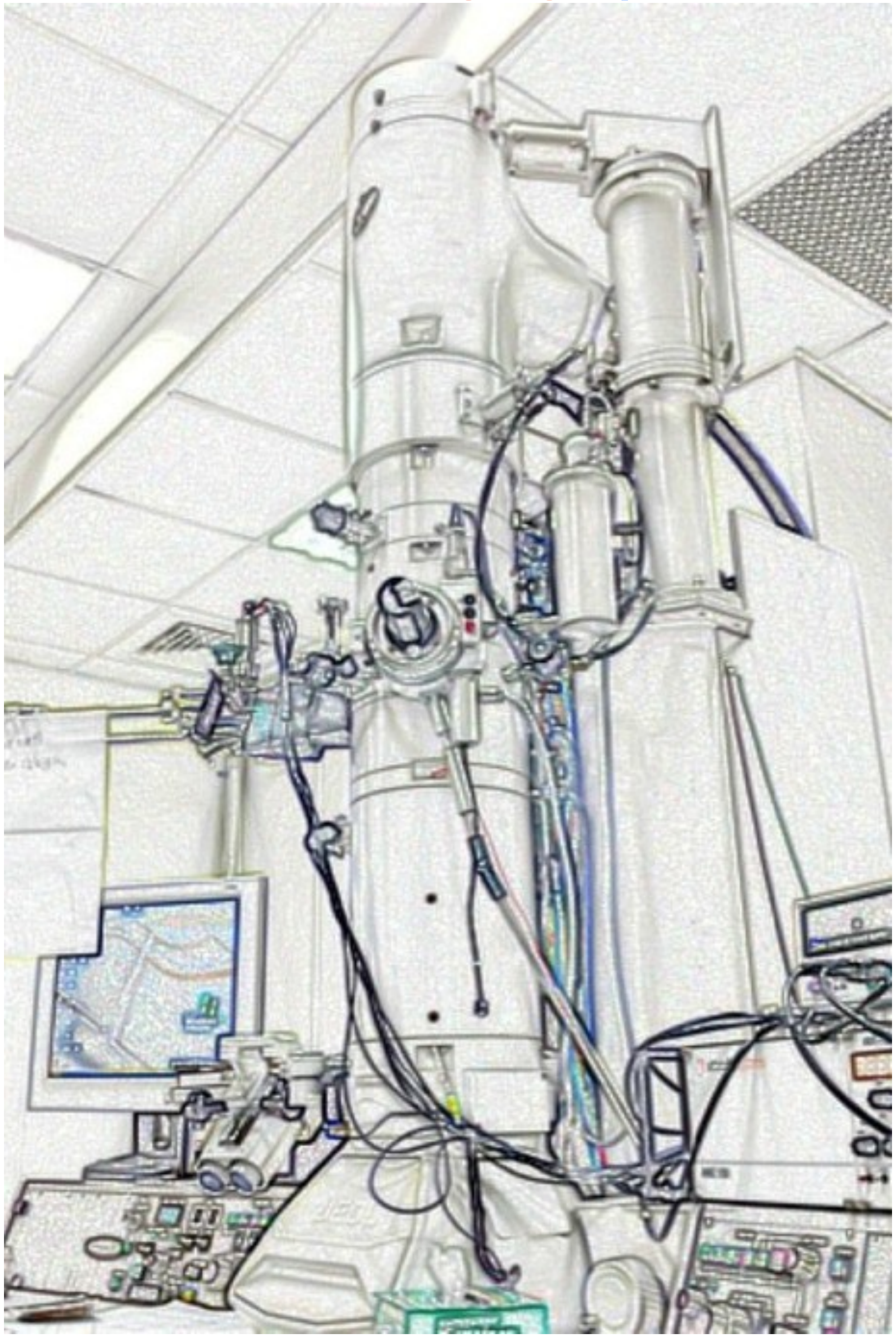
No lens for X-ray: Only diffraction can be seen

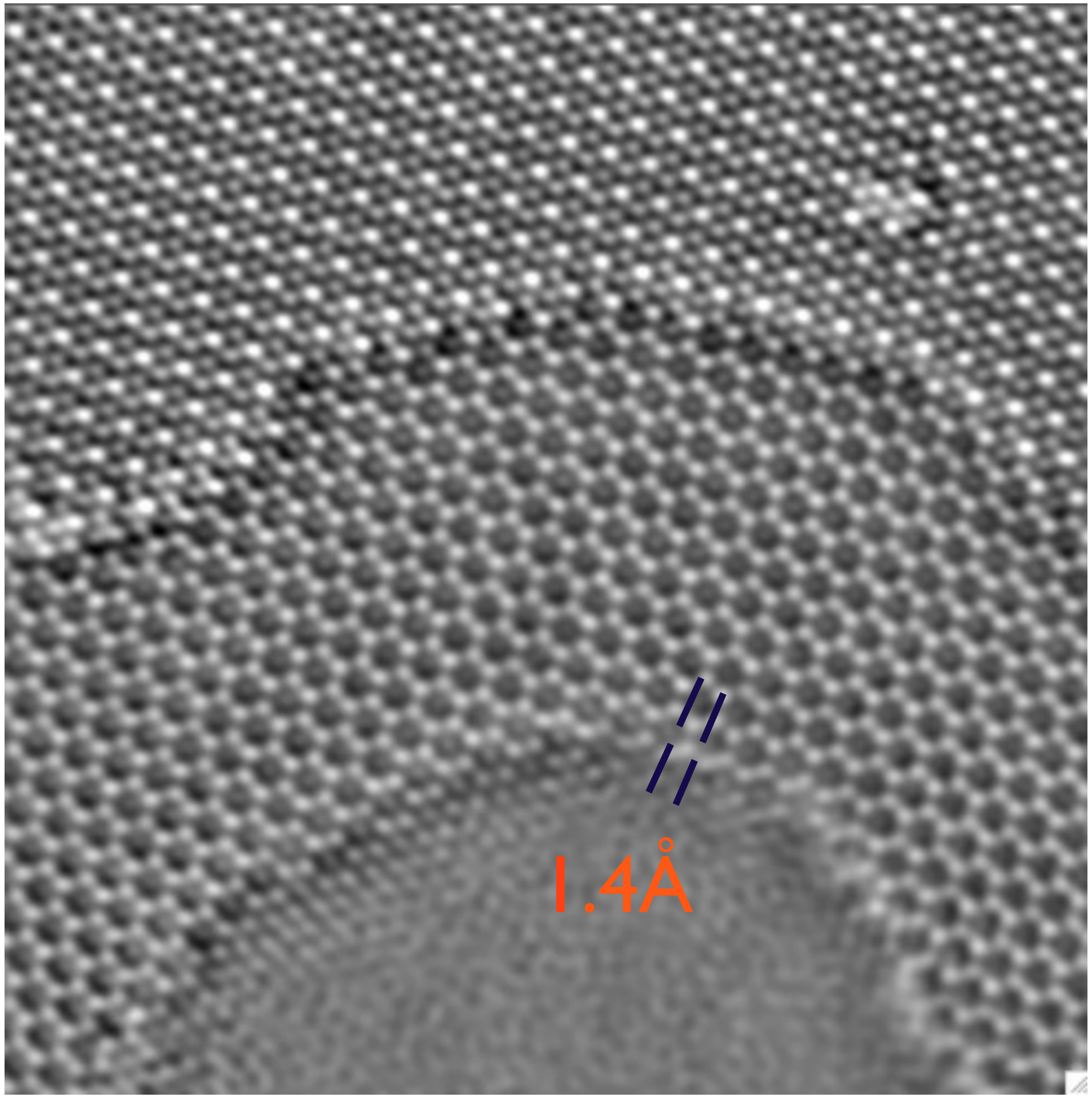
Electron microscope utilizes magnetic lens to see both image and difffraction





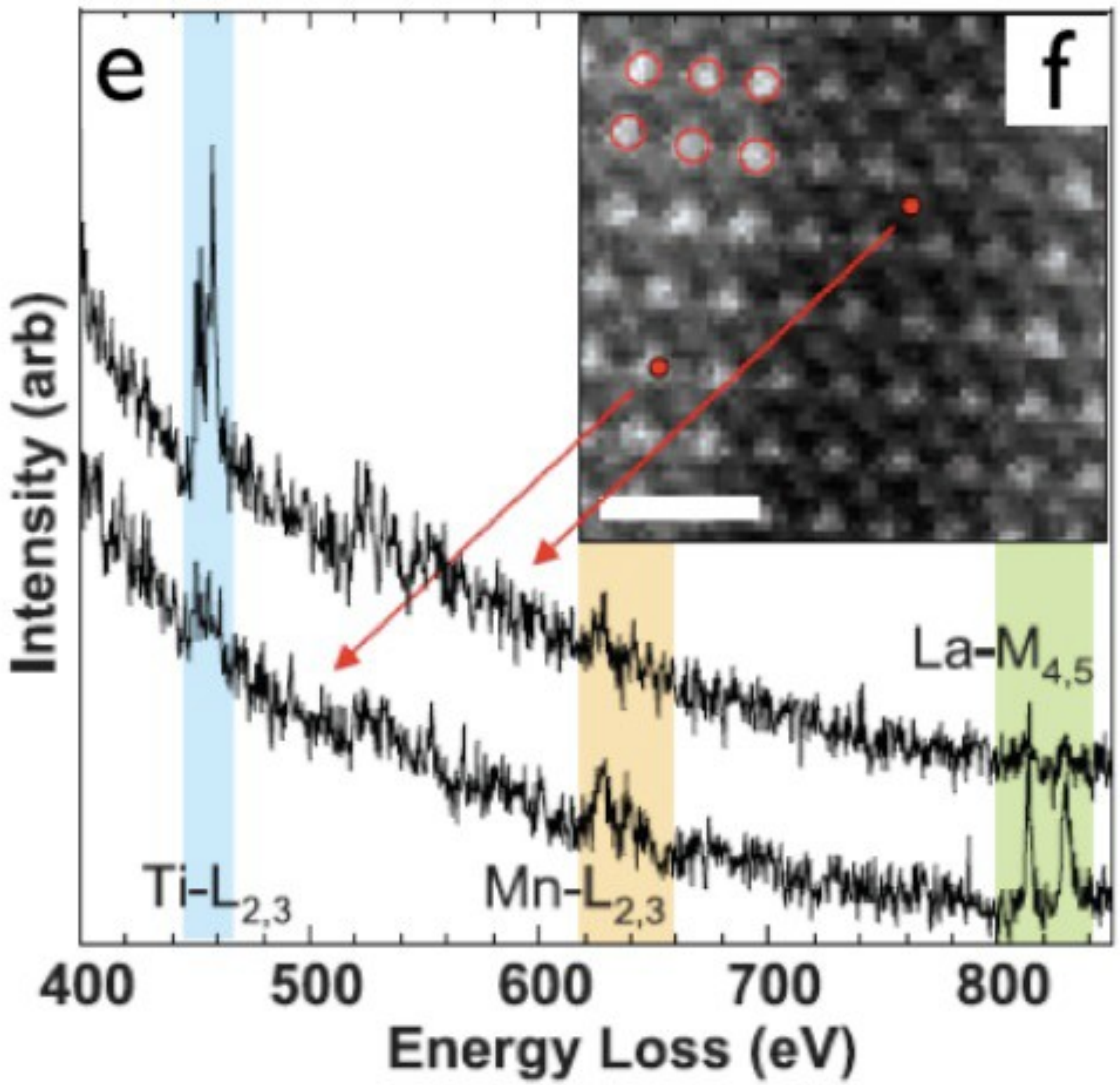
Imaging, Spectroscopy and Diffraction





Atomic Resolution

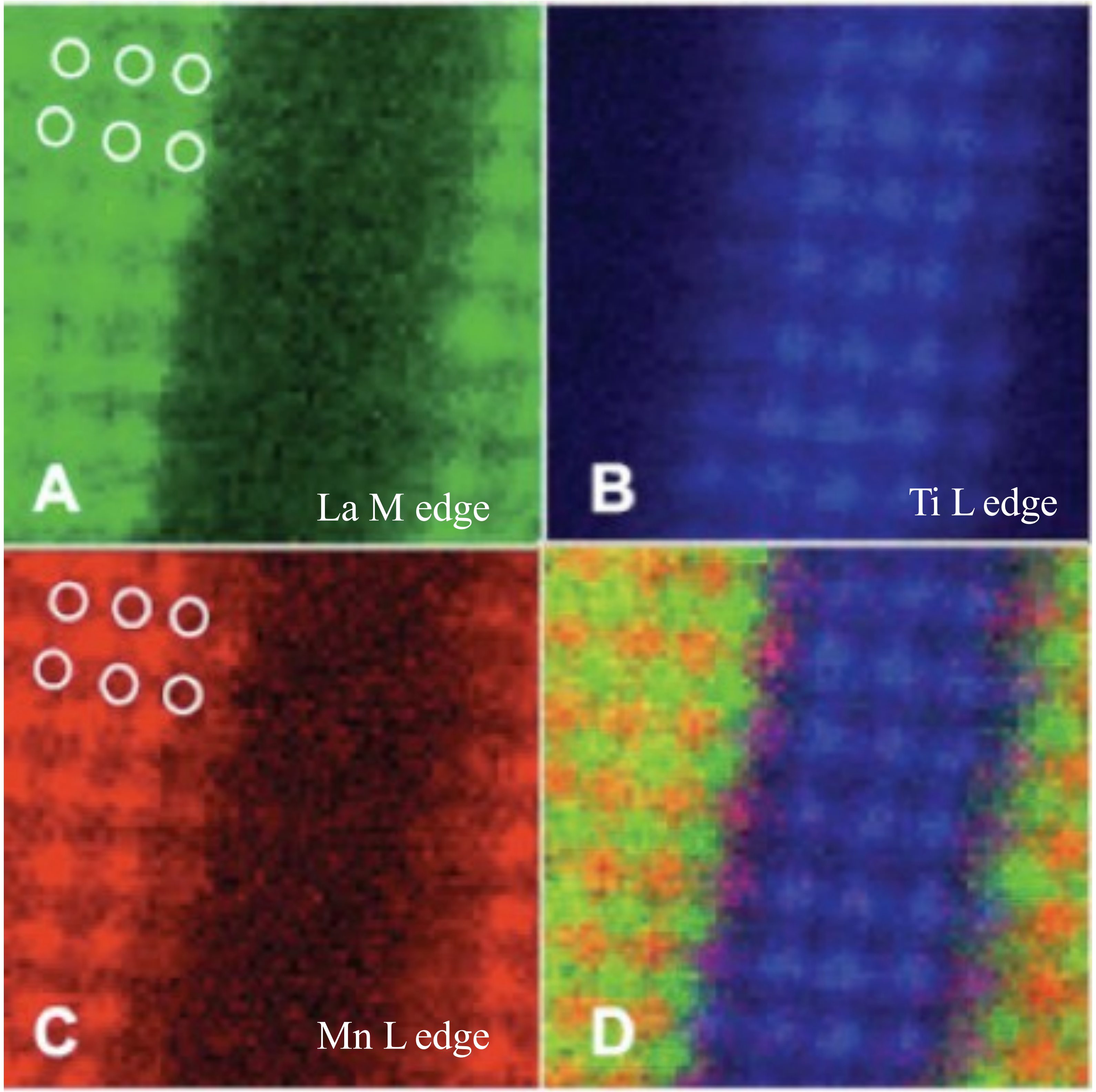
Imaging, Spectroscopy and Diffraction



Atomic resolution of EELS of $La_{0.7}Sr_{0.3}MnO_3/SrTiO_3$ multilayer

- D. A. Muller, et al, SCIENCE **319** 1073-1075 (2008)

Imaging, Spectroscopy and Diffraction



Atomic resolution compositional and bonding maps

1.2 limitation of TEM

- A 2D projection: Averaged from the thickn sample

  --------> tomography
- B Radiation Damage——polymer, bio-sample and ceramics。
- C “thin” sample is very difficult to be made

  t ＜50nm ～ 100nm
- D only dry sample can be observed
- E . NO time resolved capability

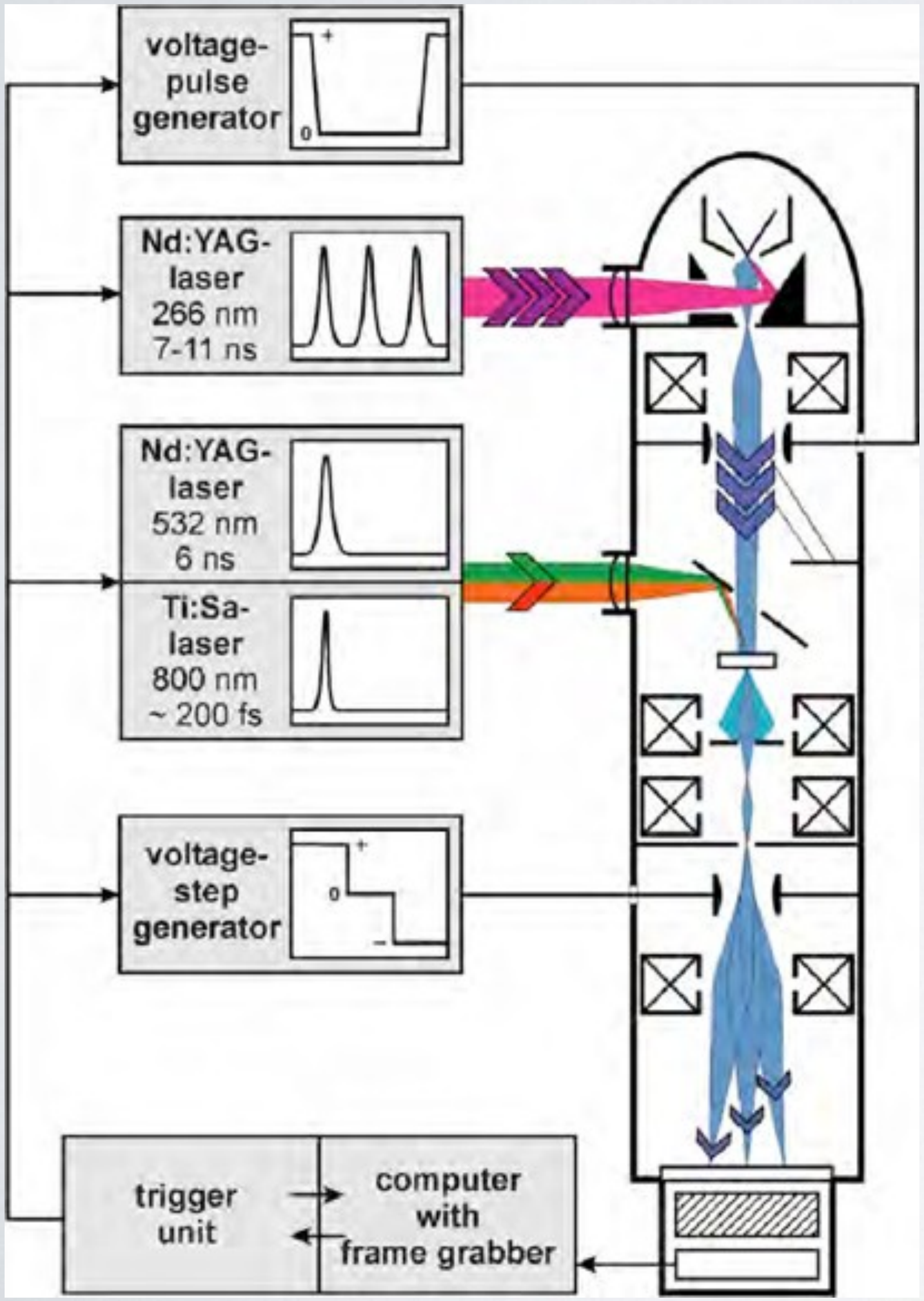
The Future of TEM: 3D atomic resolution in wet environment with time resolved

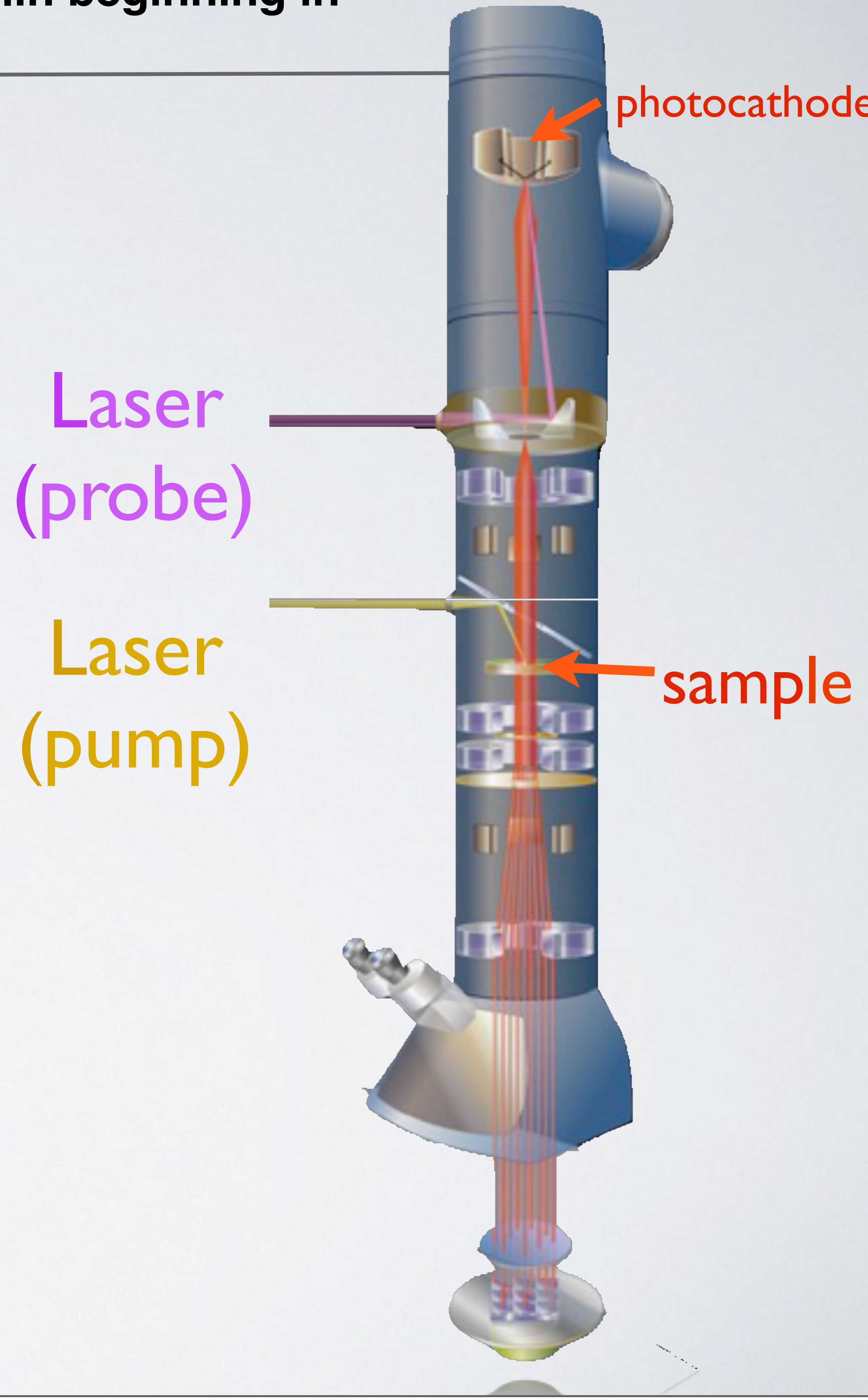
Why 3D ?

Time-resolved TEM was developed at TU-Berlin beginning in the late 1970's

Oleg Bostanjoglo

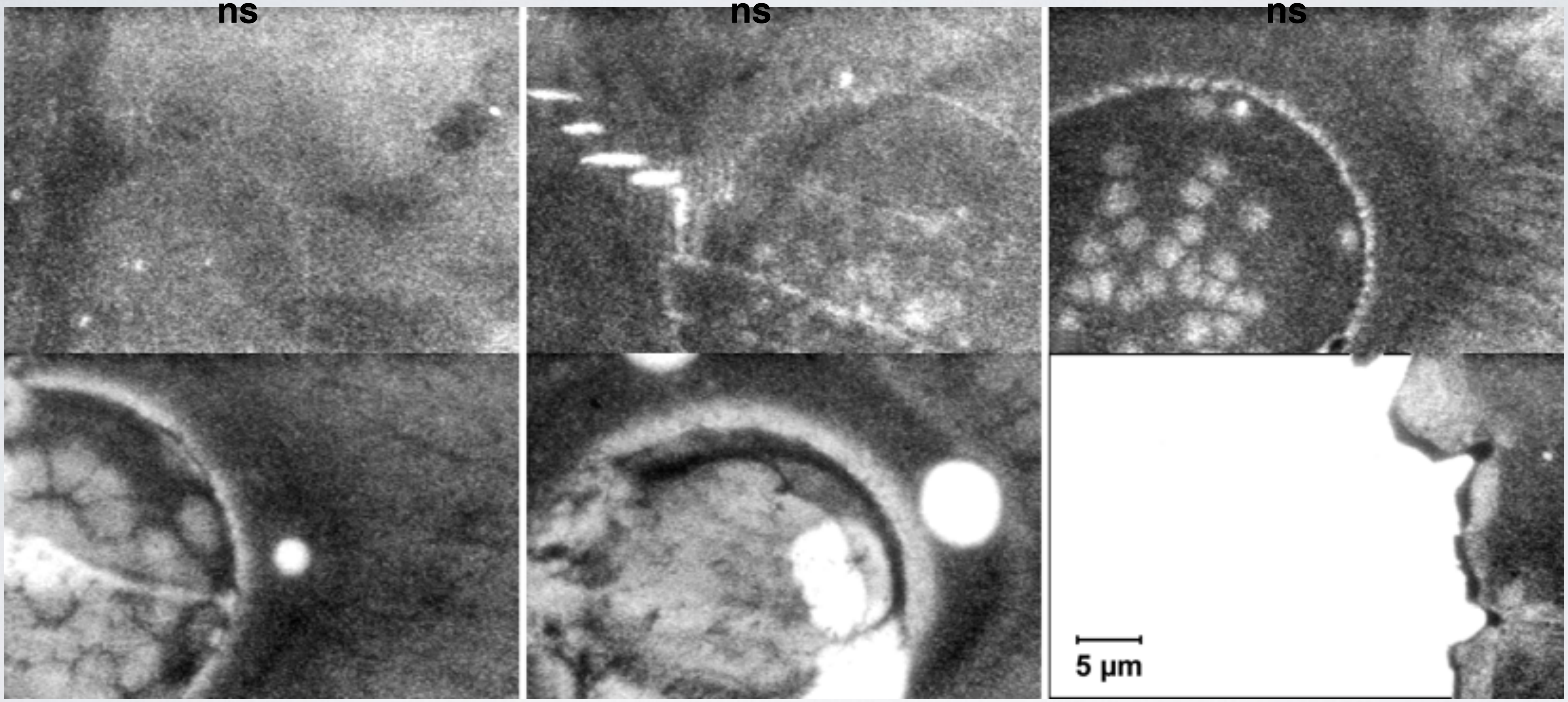
H. Dömer and O. Bostanjoglo, Rev. Sci. Inst. 74, 4369 (2003)





Time-resolved TEM

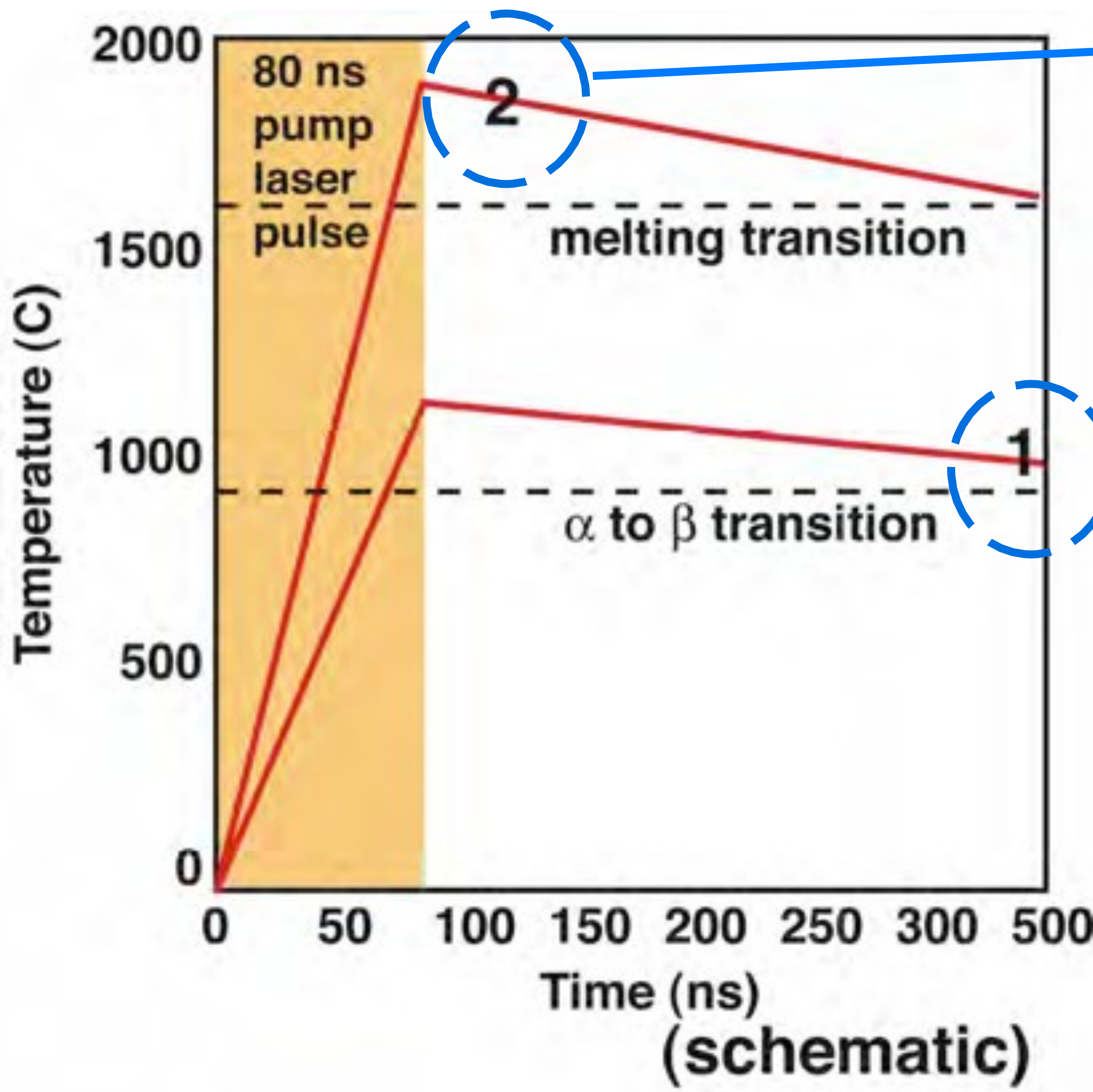
ablation of Ni film by ultrashort laser pulse



H. Domer and O. Bostanjoglo, Journal of Applied Physics **91,** 5462-5467 (2002).

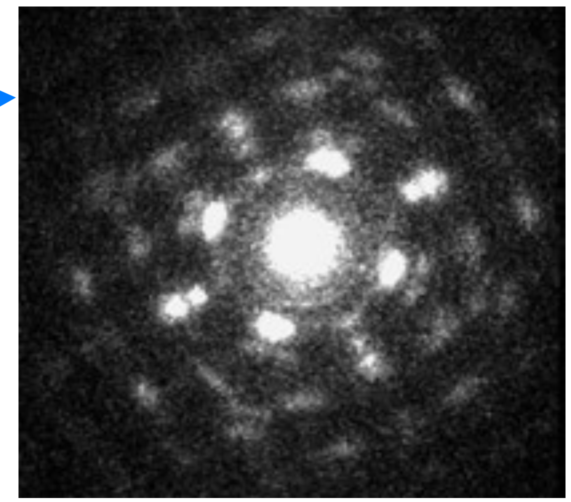
Time-resolved electron diffraction

α-Ti (hcp)---> β-Ti(bcc) martensiic transformation

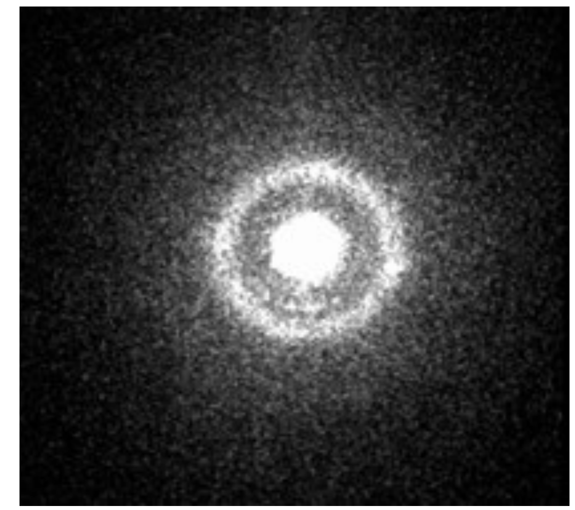


Bryan W. Reed, NLLB, UC, 2008

Melting transition

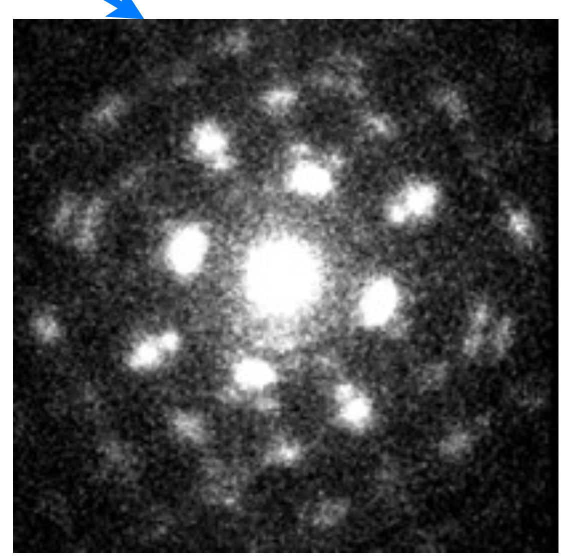


hcp Ti, α-phase

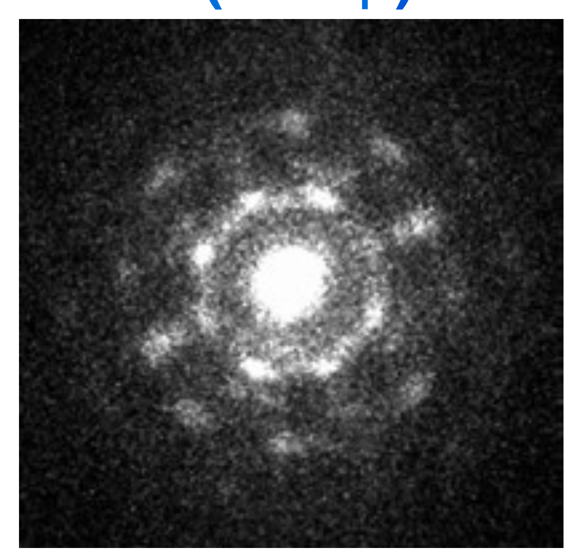


Melt structure

Phase transformation (α to β)



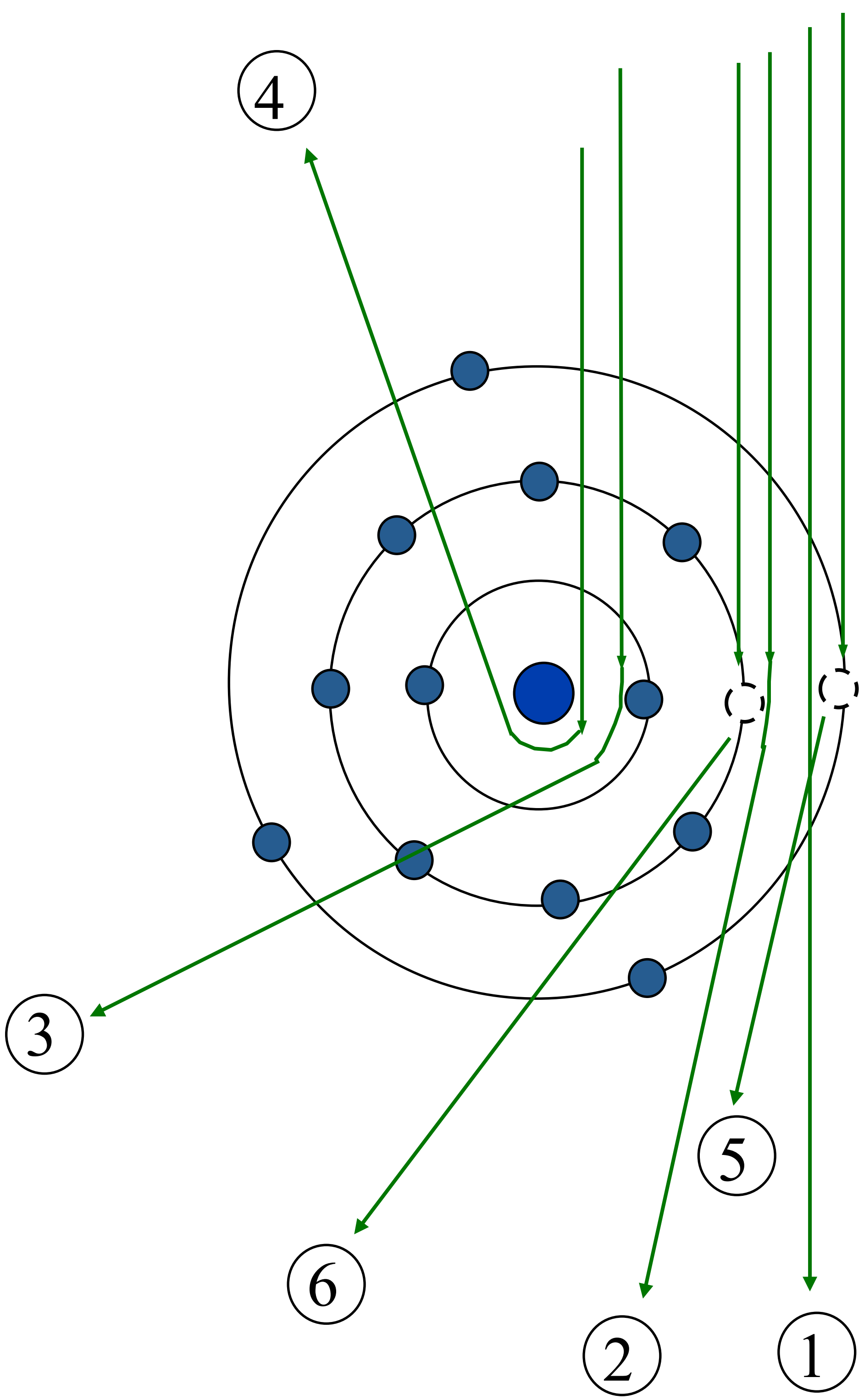
hcp Ti, α-phase



bcc Ti, β-phase

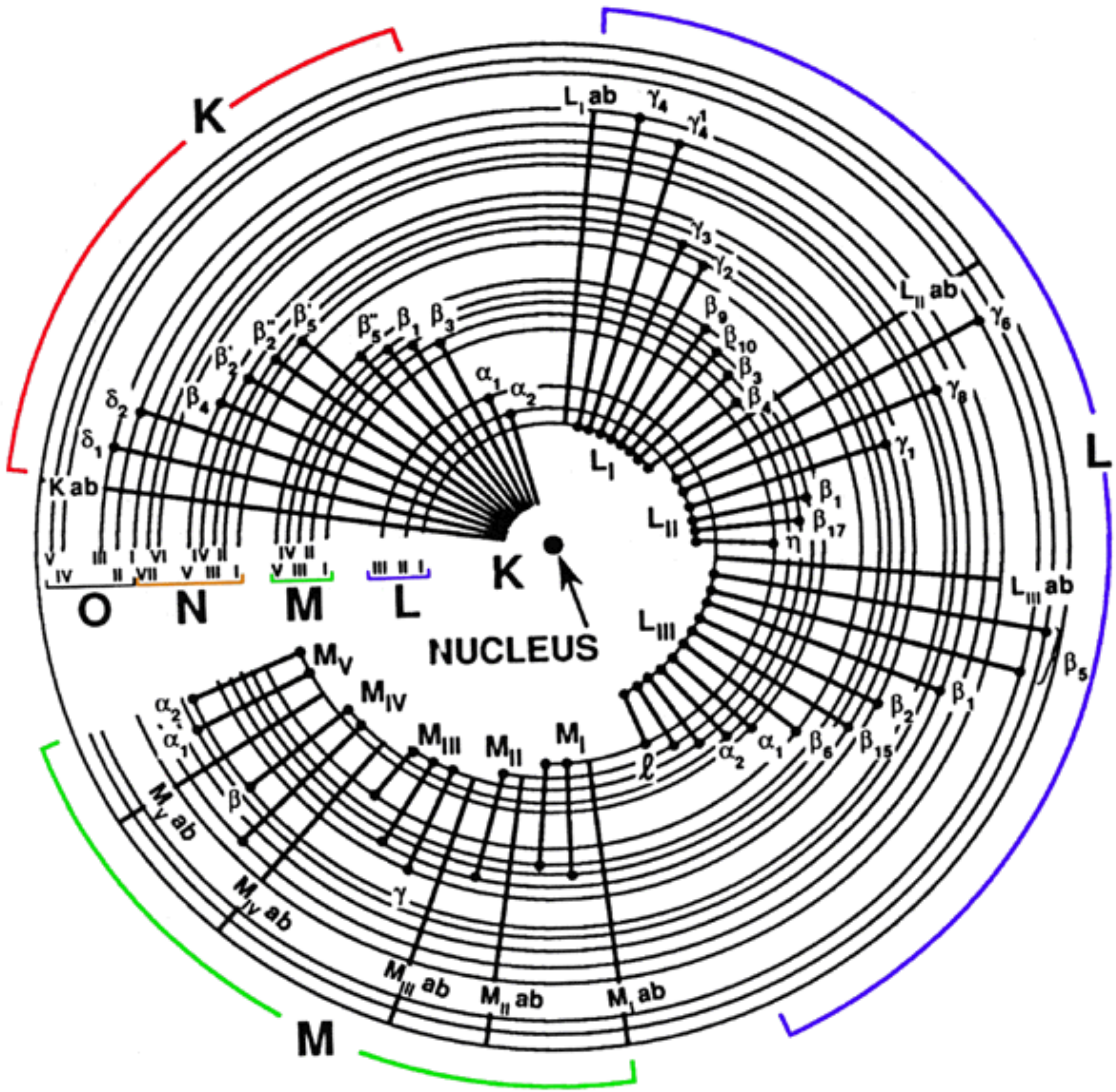
Interaction of high energy (~kV) electrons with (solid) materials-I

Interaction with an Atom



1. Unscattered
2. Low angle elastically scattered
3. High angle elastically scattered
4. Back scattered
5. Outer shell inelastically scattered
6. Inner shell inelastically scattered

Interaction of high energy (~kV) electrons with (solid) materials-I, cont.



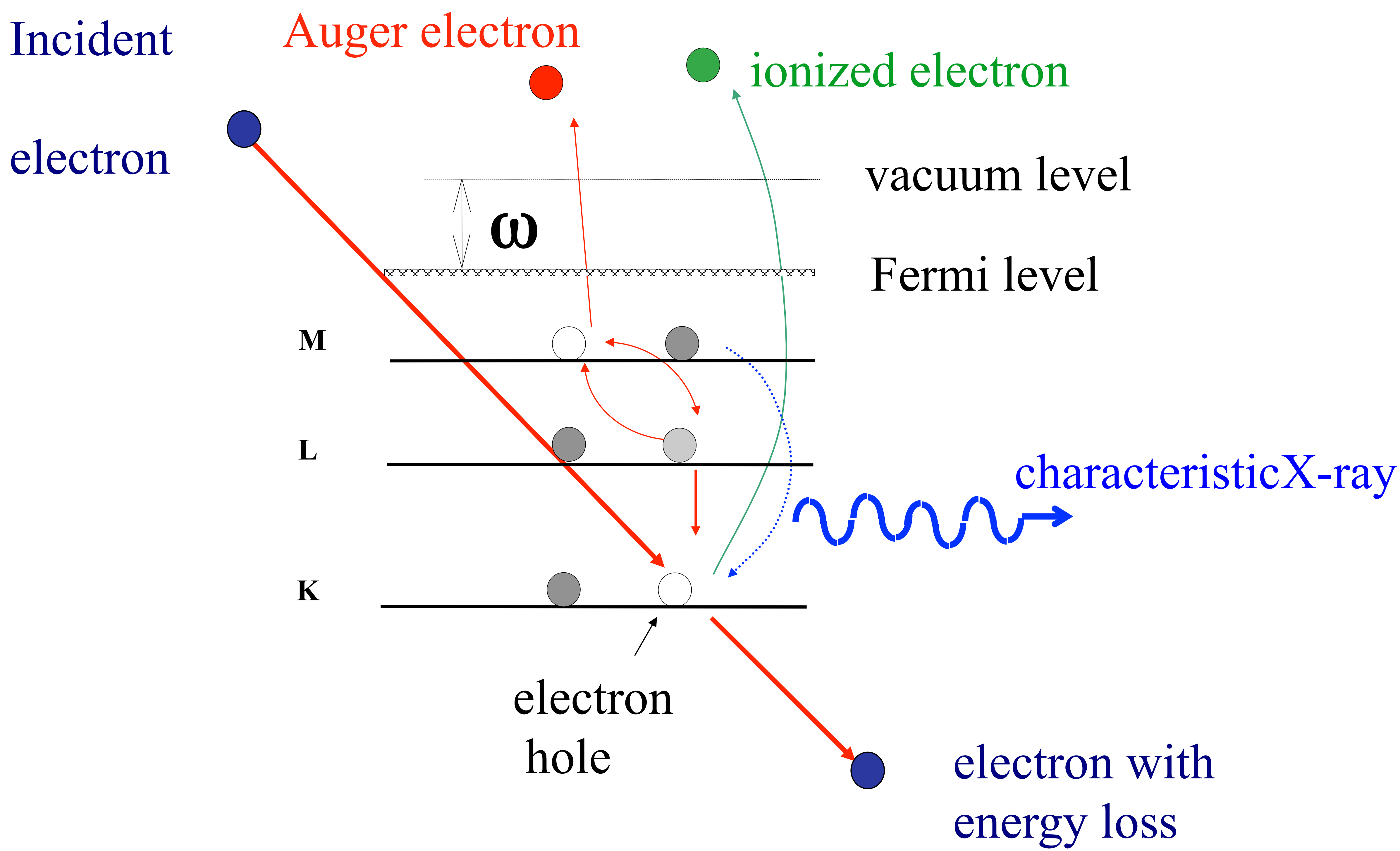
K lines

Kα, L → K
Kβ, M → K

L lines

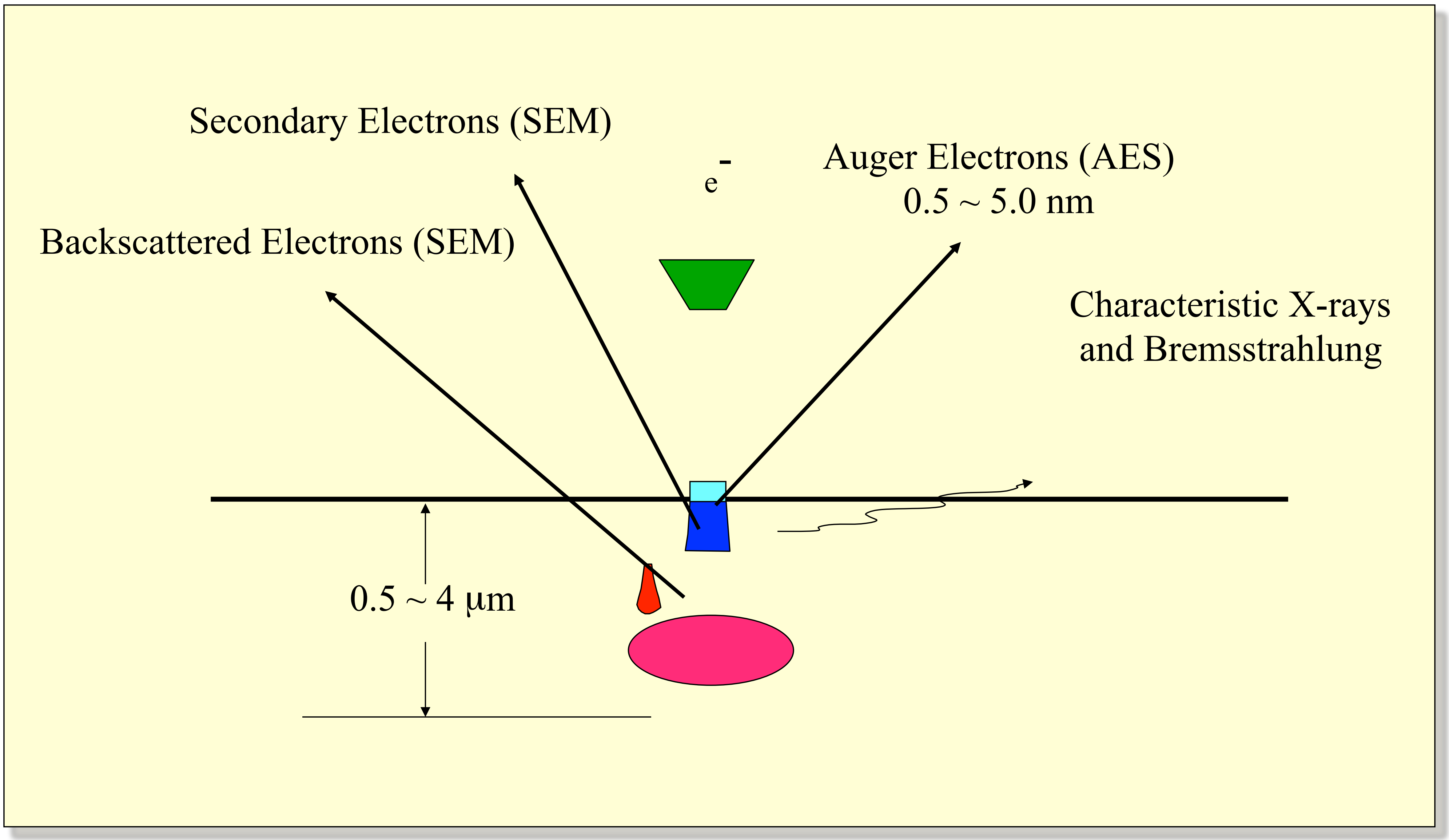
Lα, M→L,
Lβ, N →L,
Lγ, O →L

Interaction of high energy (~kV) electrons with (solid) materials-I, cont.

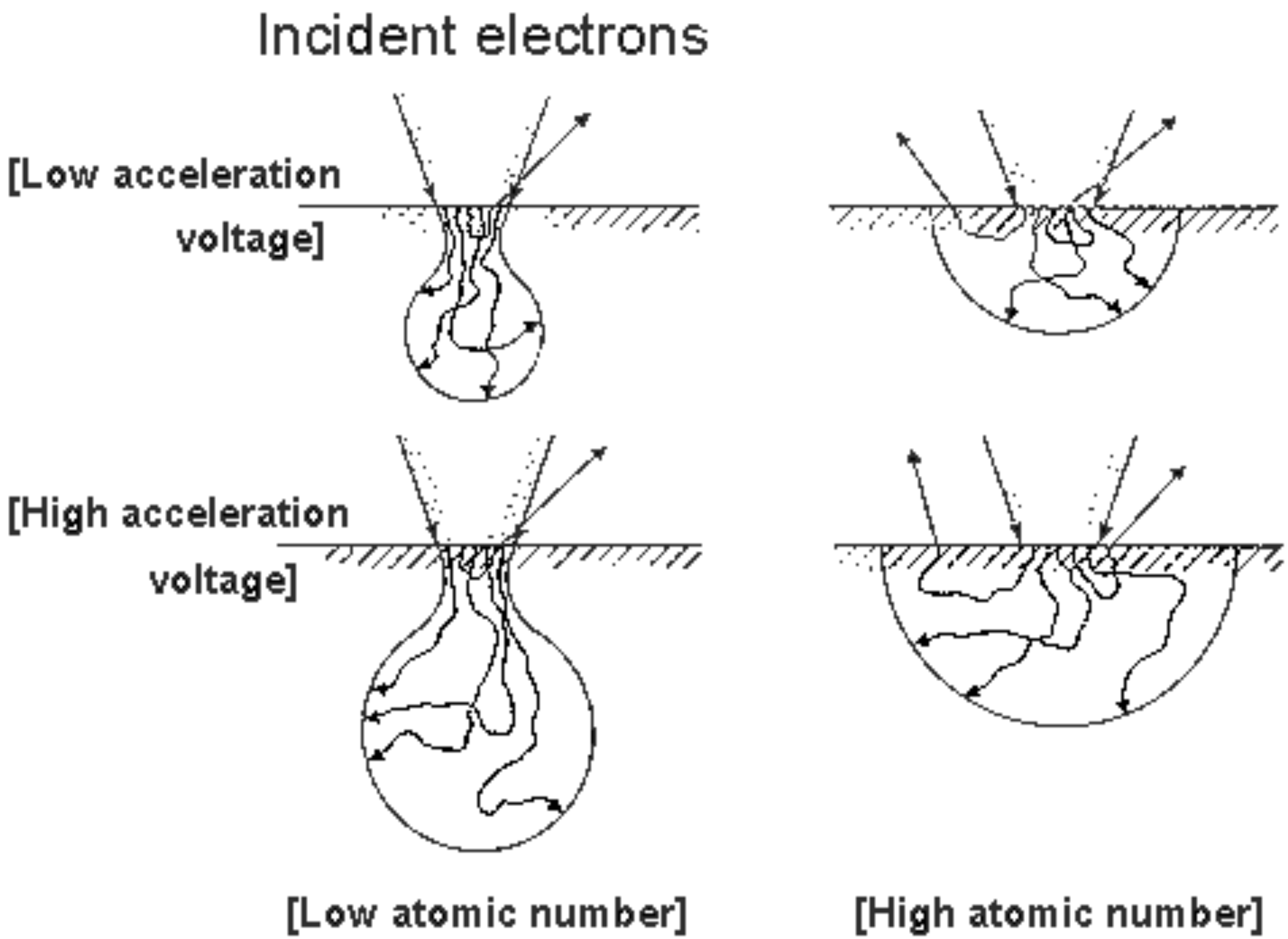


Interaction of high energy (~kV) electrons with (solid) materials-III

Interaction with a thick specimen (SEM)



Penetration power of e-beam



Basic electron optics

Electrons and ions are charged particles, they can be accelerated in a **E** field.
The trajectory of an accelerated charged particle can be deflected by **E** and/or **B** field.
According to *de Broglie*, the accelerated (high-energy) particles also behave like waves.

Electron Optical Elements and Attachments

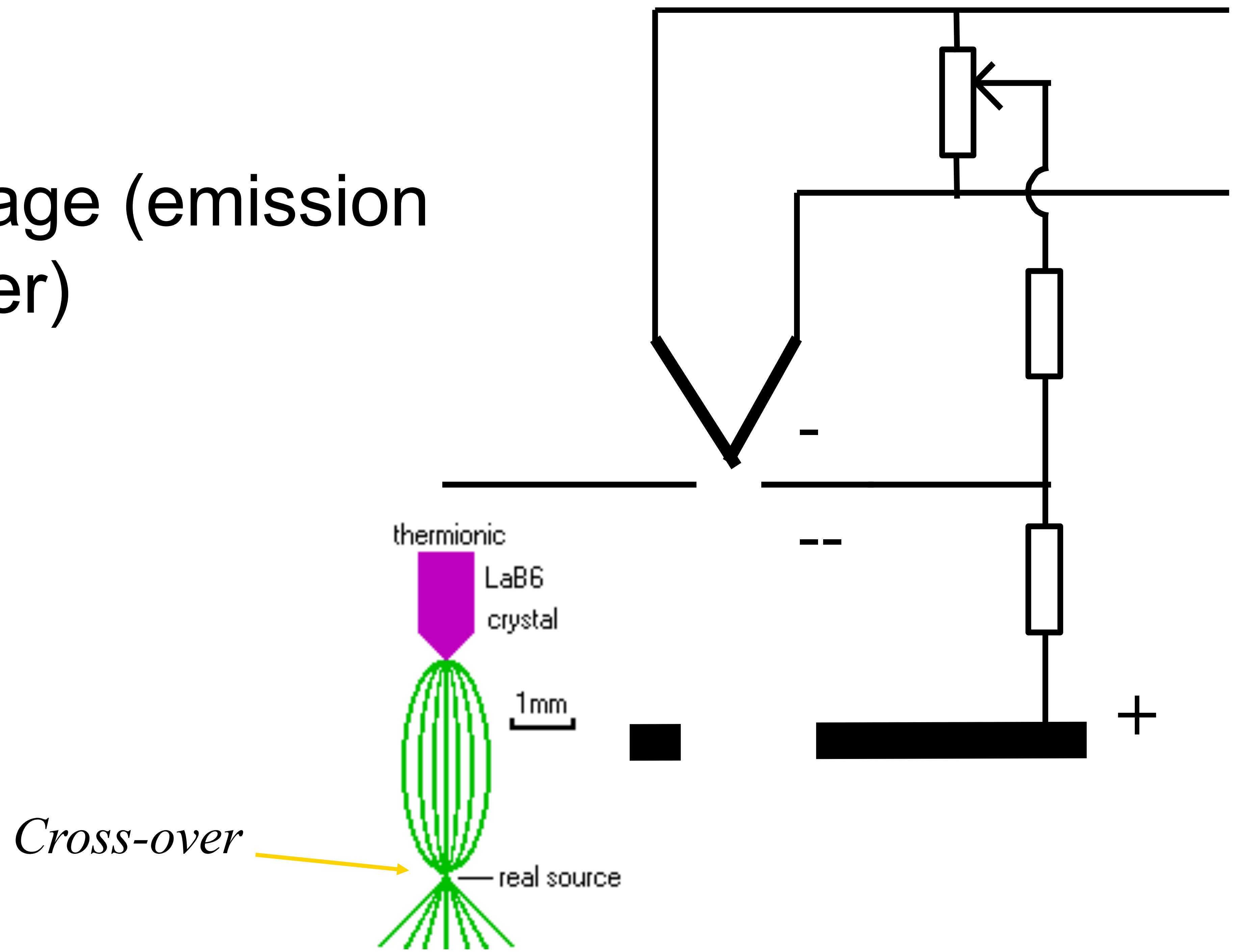
Electron Sources
Lenses
Deflection Coils
Stigmators
Electron Detectors
Attachments for photons or X-rays

Electron Source

- Thermionic Gun
  - triode or self-biasing gun
  - W, LaB_6, CeB_6
- Field Emission Gun
  - single crystal W

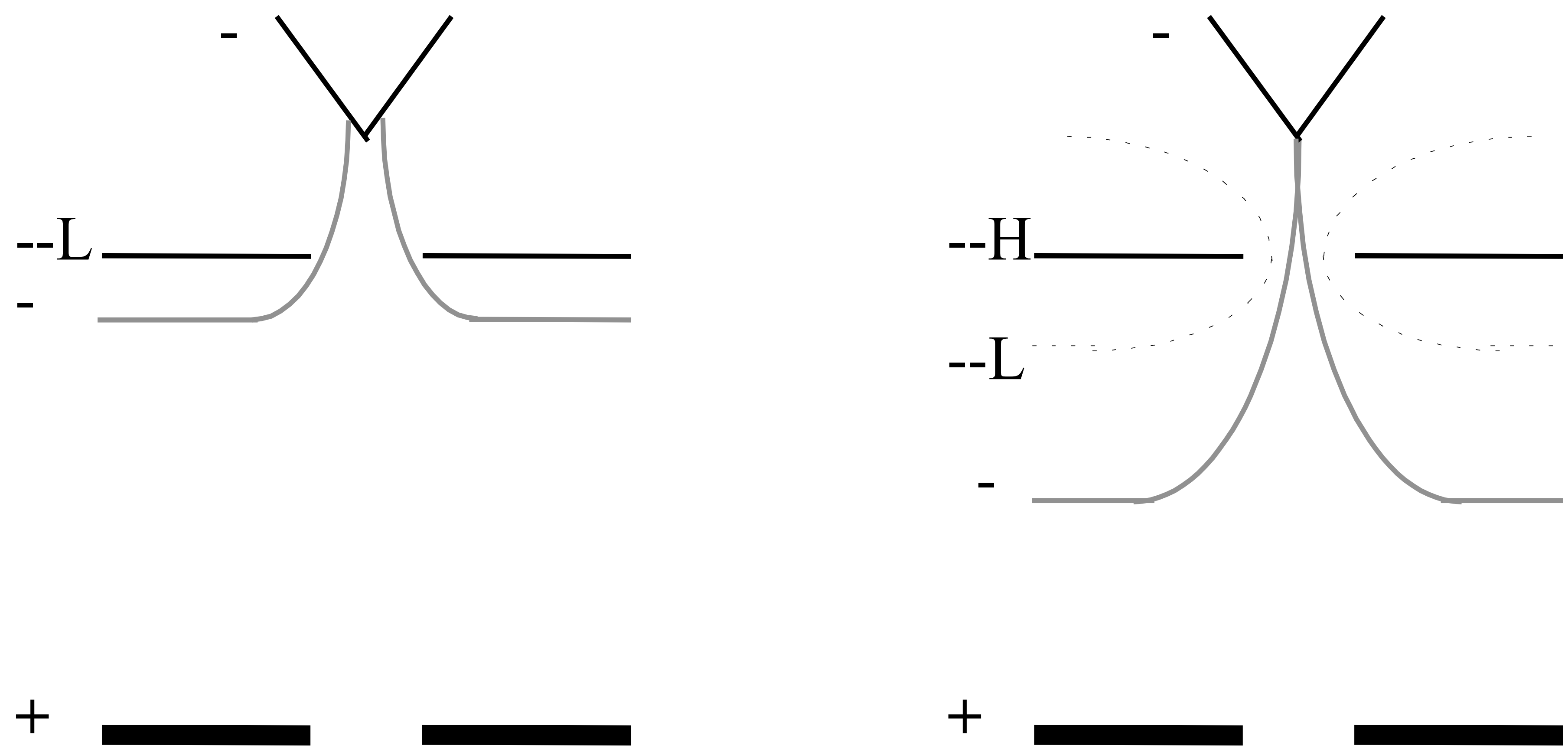
Electron Source
Thermionic Gun

- Filament
- Wehnelt
  - bias voltage (emission parameter)
- Anode



Electron Source
Thermionic Gun

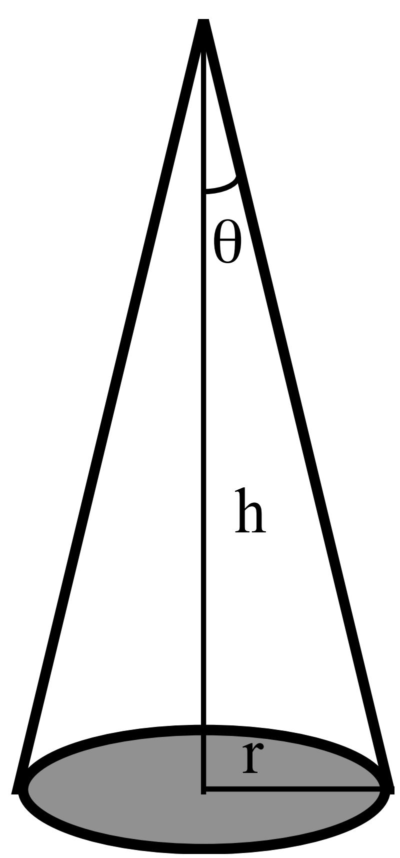
- Increasing bias voltage restricts emission, thereby reducing the total emitted current



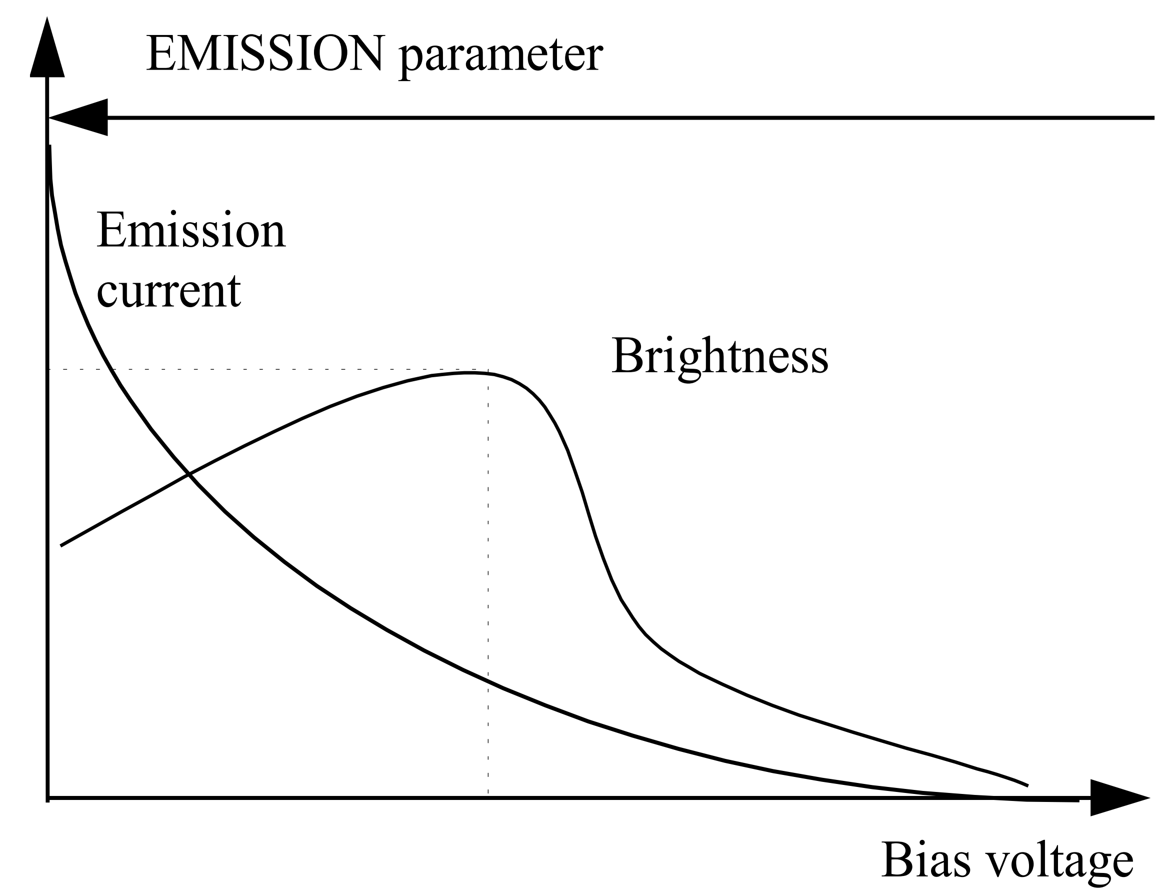
Electron Source
Thermionic Gun

- Brightness = electron current by a source with unit area and unit solid angle

$$\beta = I / (A\Omega)$$



$$\Omega = \pi\theta^2$$

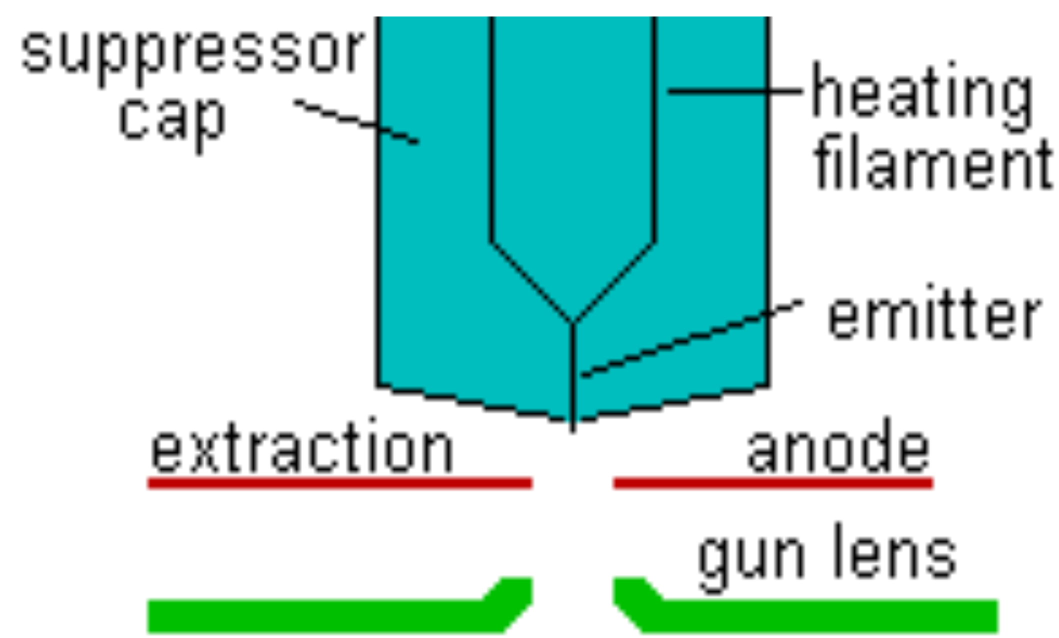


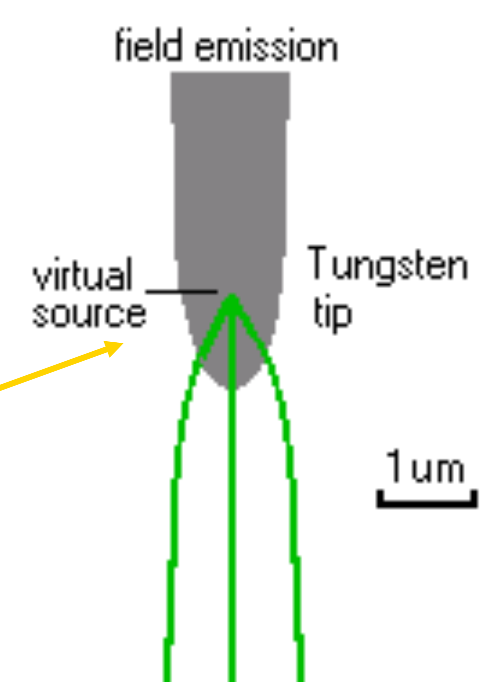
Electron Source
Thermionic Gun

- Energy Spread
  - imperfections of filament
  - instability of high tension
  - surface temperature
  - Boersch effect (mutual interaction)
- Source Spotsize
  - 30 μm for W
  - 5 μm for LaB_6

Electron Source
Field Emission Gun (FEG)

- Heating Filament
- Single Crystal Emitter
- Suppressor Cap
- Extraction Anode
- Electrostatic lens

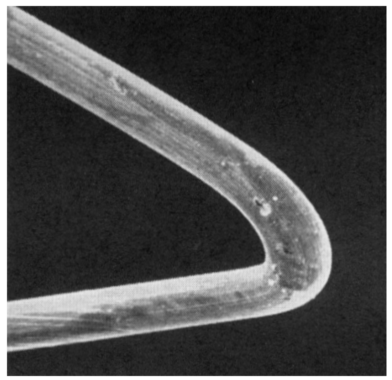


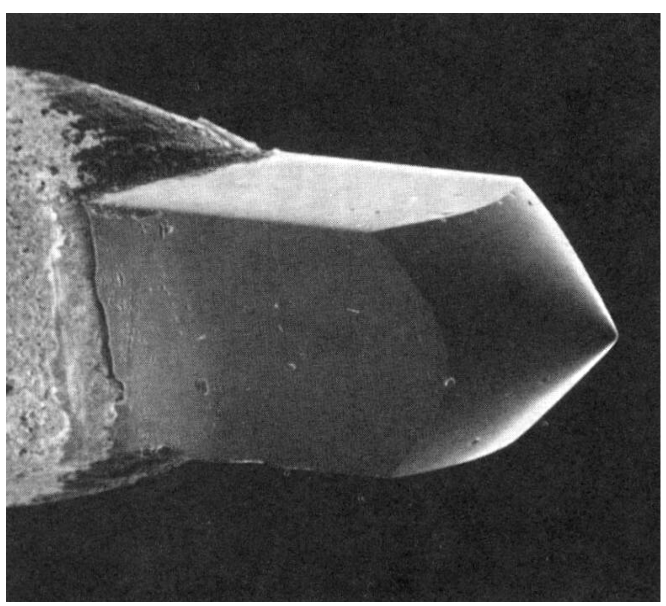


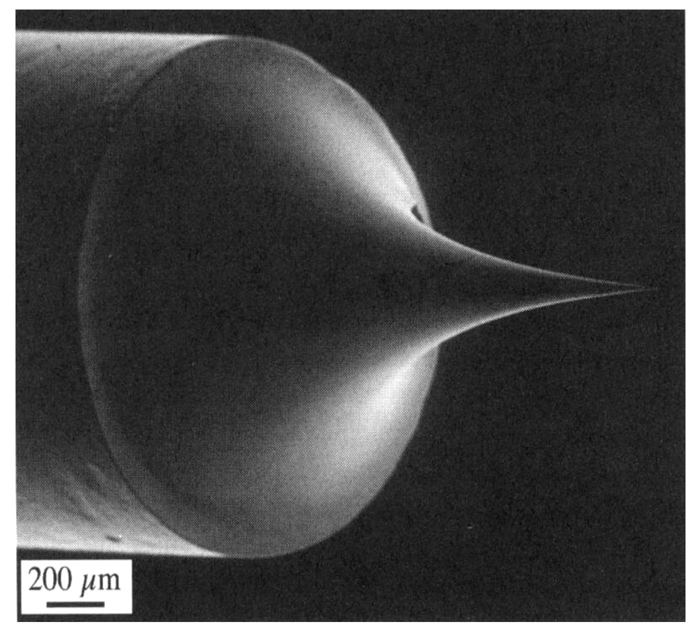
Electron seemingly originating from tip itself

Comparison of Electron Sources

	W	LaB_6	FEG (Schottky)
Maximum Current (nA)	1000	500	300
Normalised Brightness (-)	1	10-30	2500
Energy spread (eV)	3-4	1.5-3	0.6-1.2
Source spotsize	30-100 μm	5-50 μm	15-30 nm
Required Vacuum (Pa)	10^{-3}	10^{-5}	10^{-7}
Temperature (K)	2700	2000	1800
Life time (hr)	60-200	1000	>2000
Normalised Price (-)	1	10	100



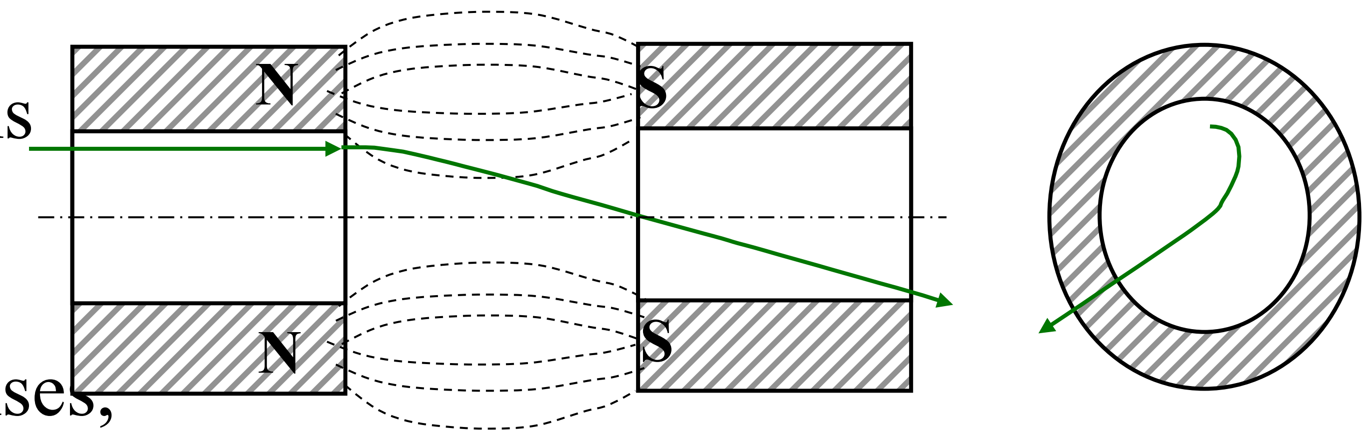



200 μm

Lenses

- Provide means to (de)focus the electron beam on the specimen, to focus the image, to change the magnification, and to switch between image and diffraction

Round Lenses



Magnetic lenses

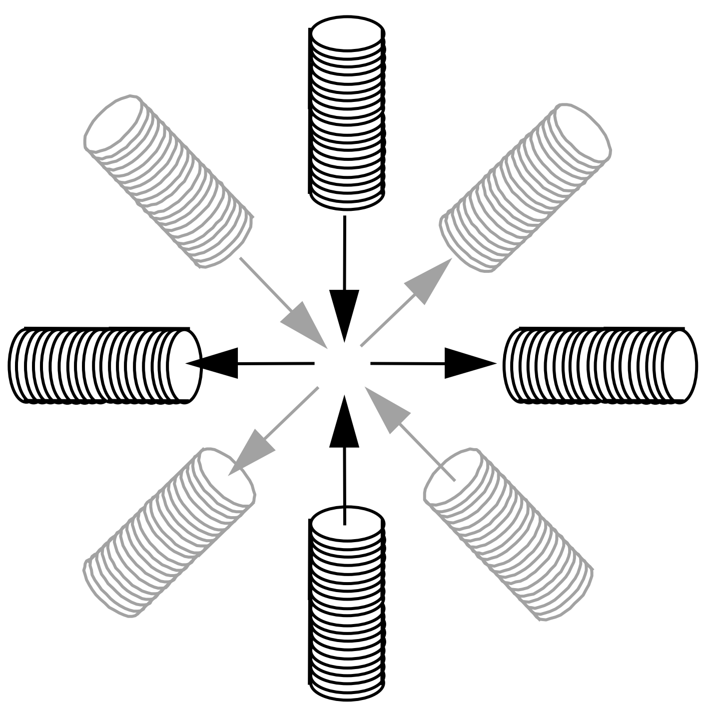
- change the direction of electrons
- magnifying (diverging)
- diminishing (converging)
- condenser lenses, objective lenses,
- intermediate lenses, projection lenses

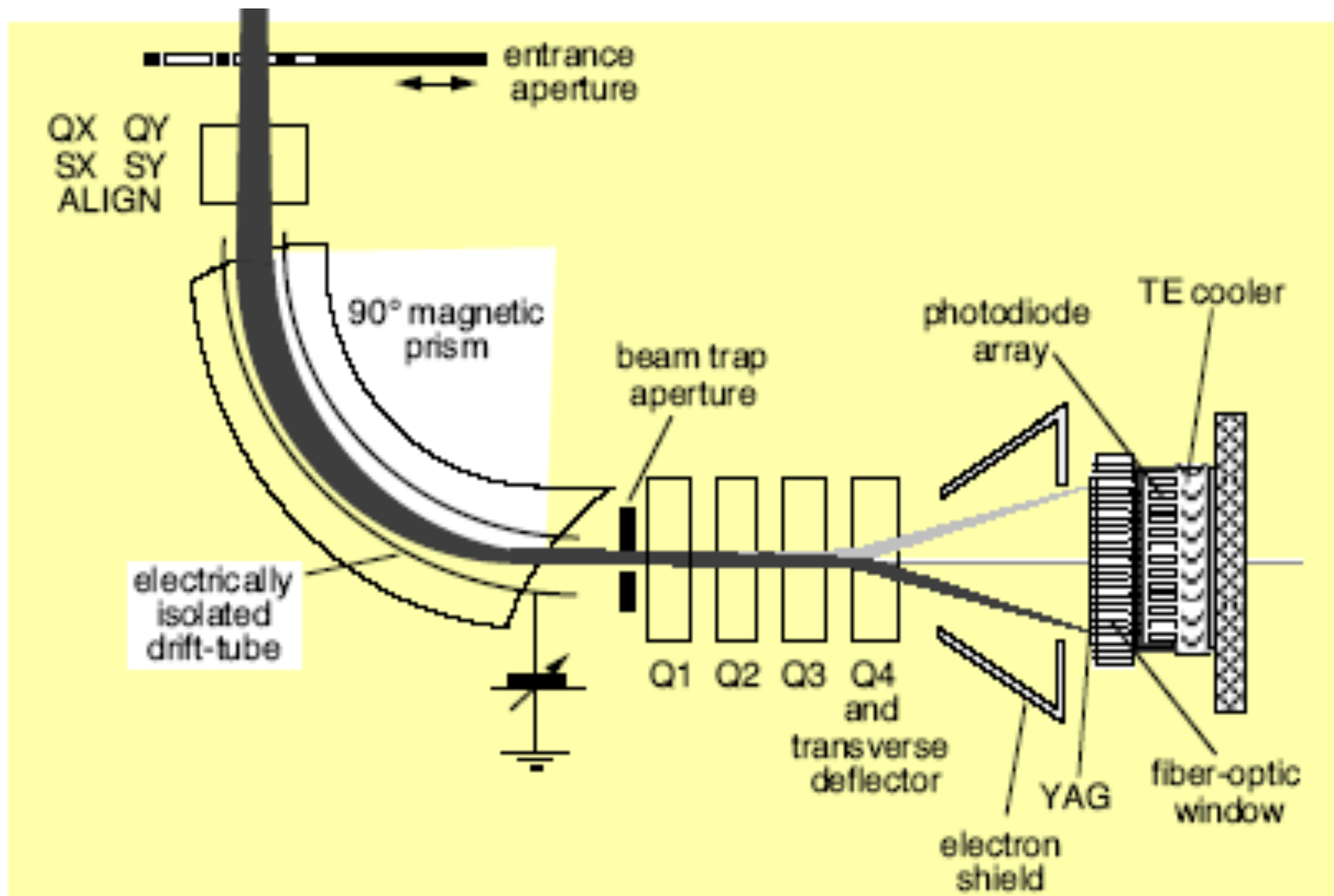
Electrostatic lenses: the Wehnelt cap

- Advantage
  - rotation free
- Disadvantage
  - high precision in construction
  - high precision in alignment
  - extreme cleanliness

Pole Lenses

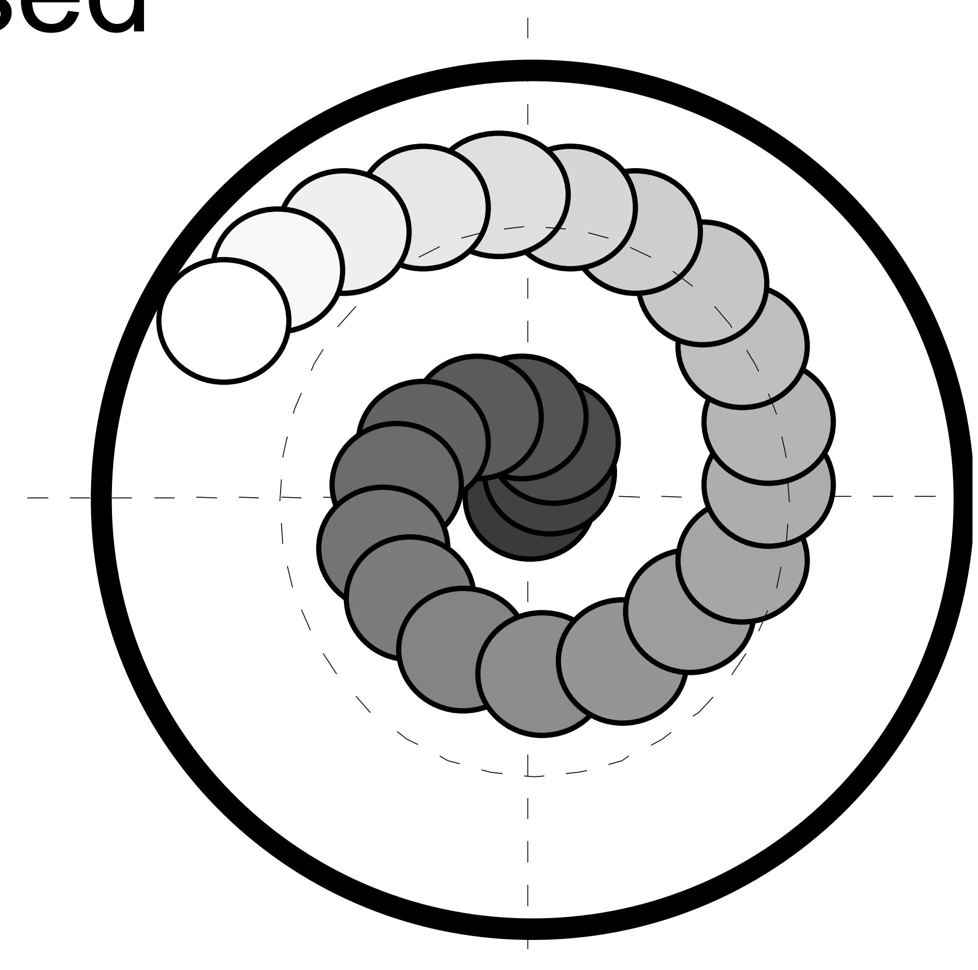
- Pole lenses are all electromagnetic, no electrostatic
- Different magnifying power in X, Y direction is possible
- The construction is just like the stigmators
- Usually seen in Cs correctors and EELS
- Qudrapole, Hexapole, Octupole lenses are common.





Lenses

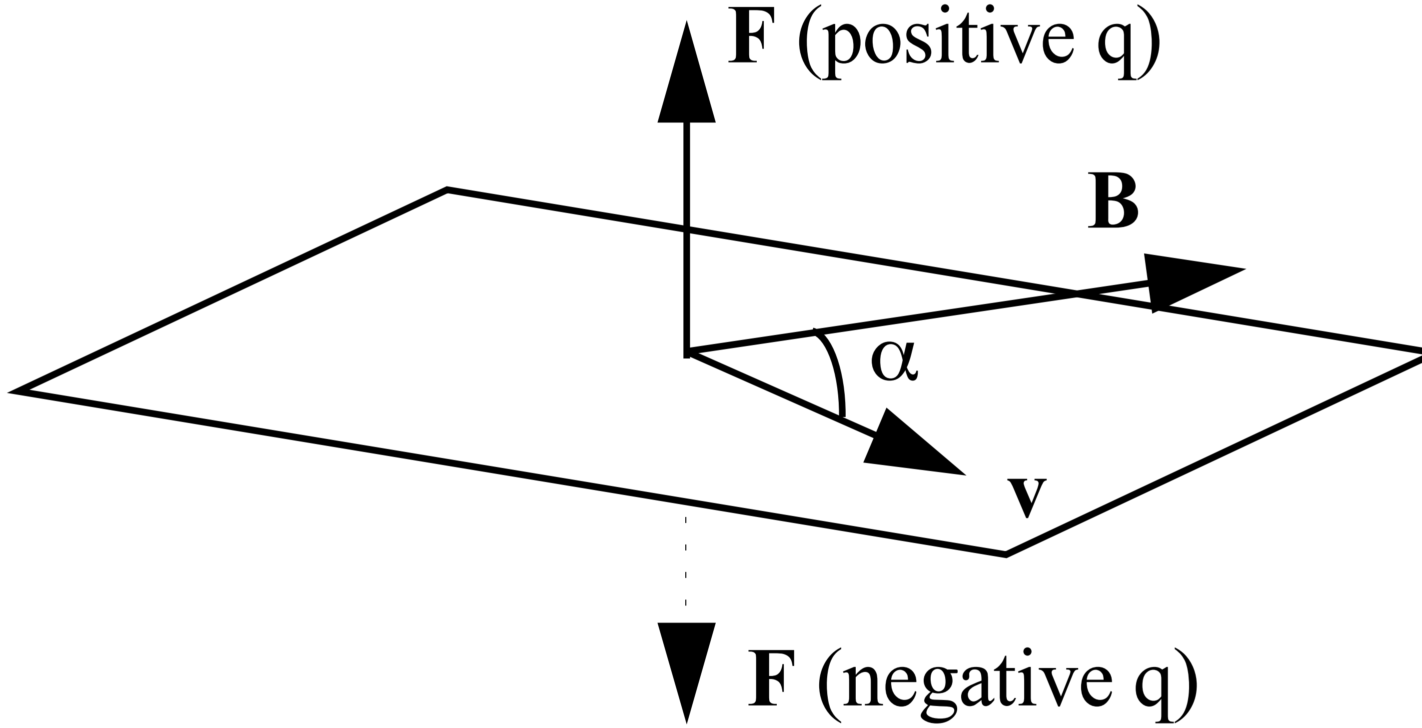
- Electromagnetic lenses are based on the fact the moving electrons are forced into a spiral trajectory, i.e. focused into one point



Lenses

- Working Principle: Lorenz Force
  - electrons are only *deflected* by magnetic fields

$$\mathbf{F} = q(\mathbf{v} \times \mathbf{B})$$



Lenses

- the focal length is given by:

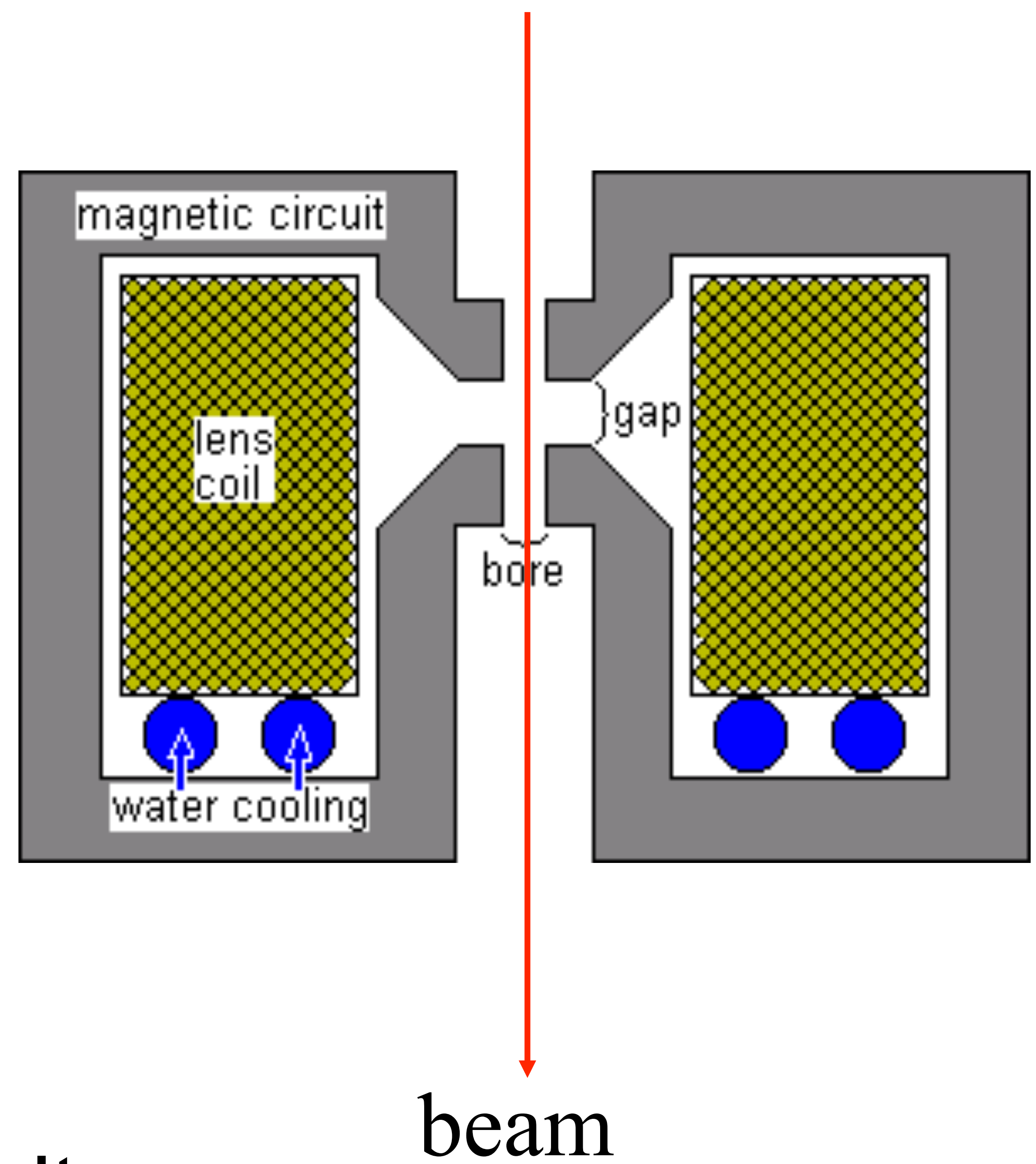
$$f = \frac{K \cdot U}{(N \cdot I)^2}$$

K : constant

U : accelerating voltage

N : windings

I : lens current



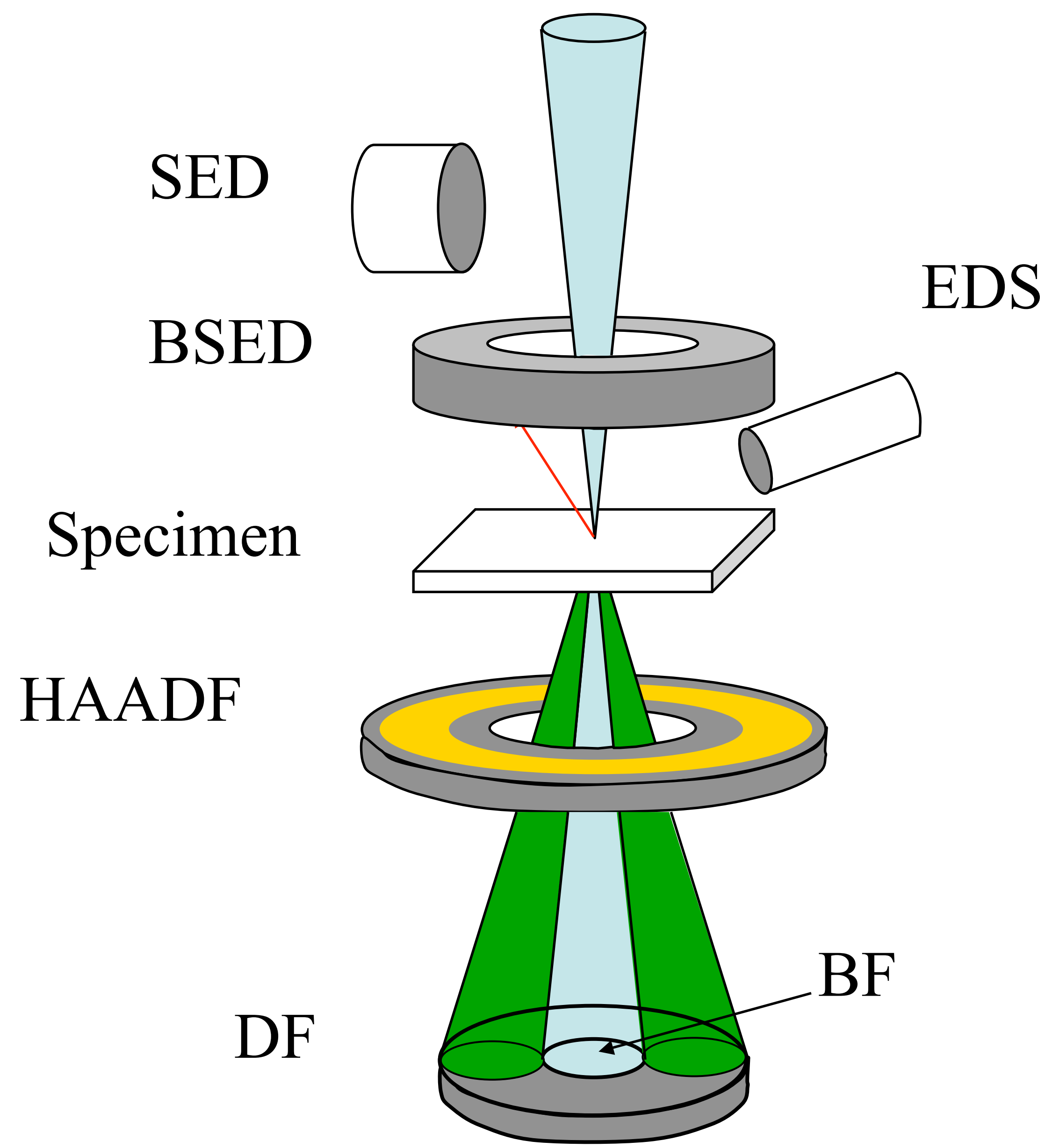
Electromagnetic Lenses for Electrons

- Focus
- Magnification and demagnification
- Electron trajectory changed by magnetic field
- **F** = - e **v** x **B**
- F = evB sinθ
- If **v** // **B**, F = 0

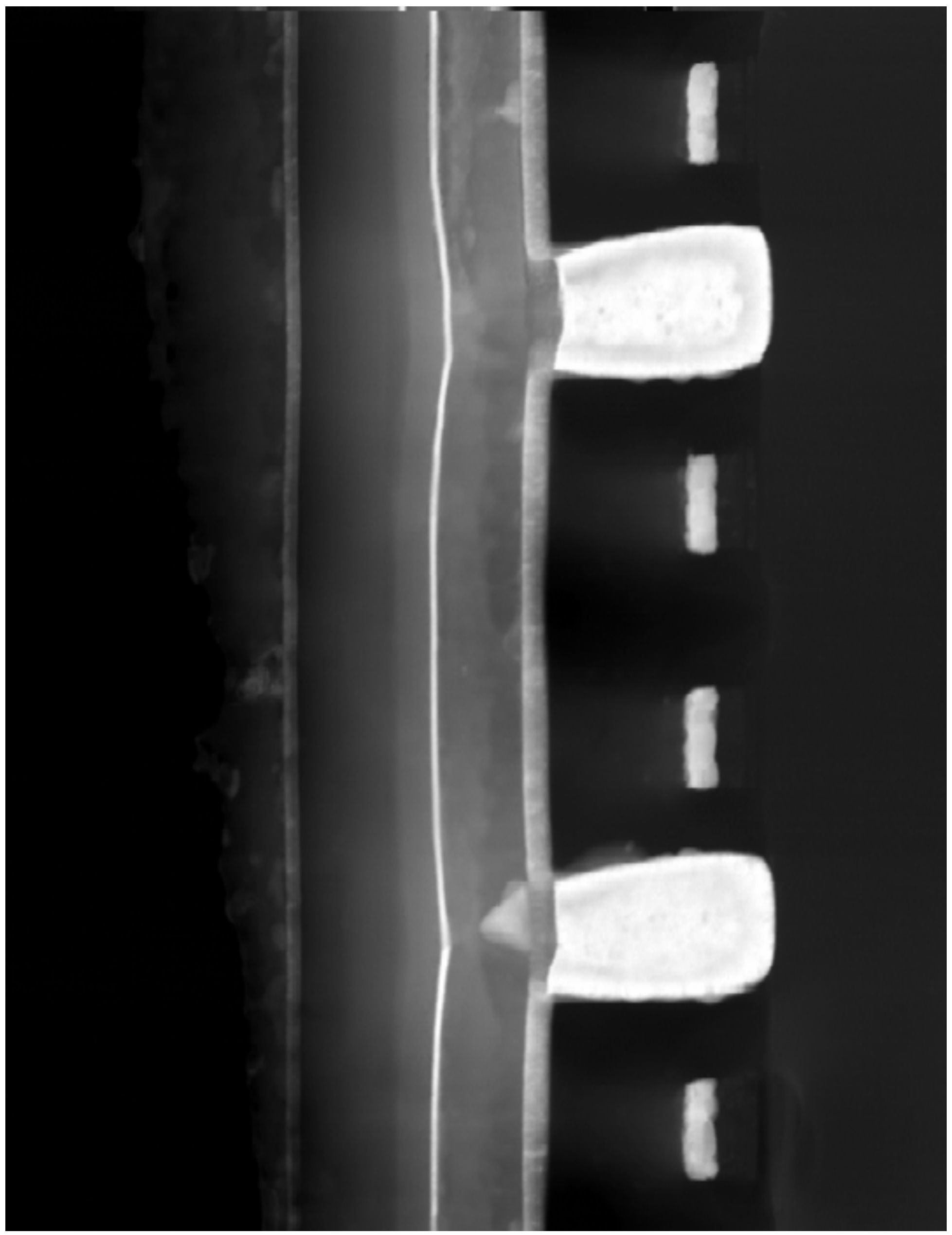
$$R = \frac{m_0 v}{eB}$$

Fundamentals of STEM

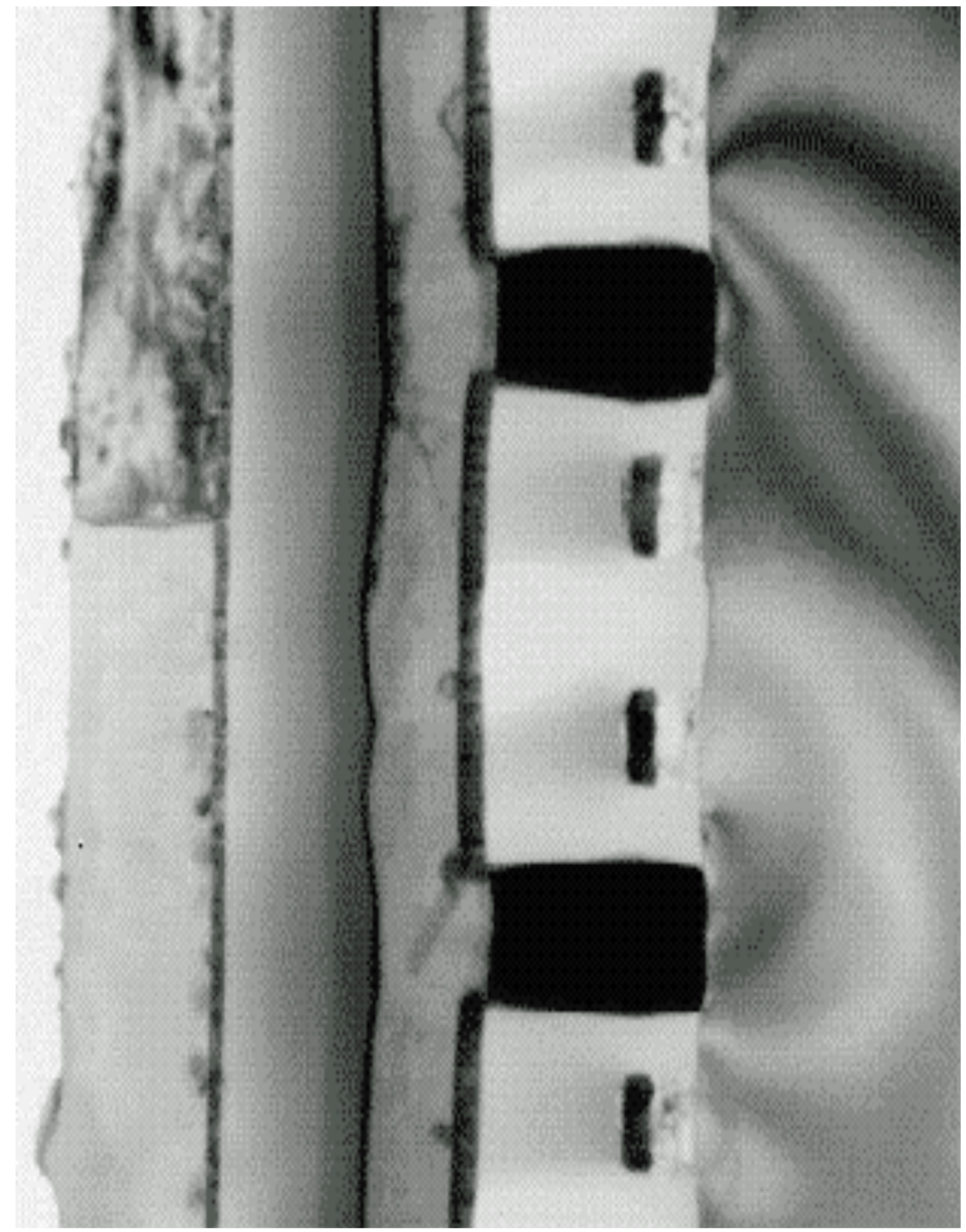
- More detectors than a SEM below the specimen, which collect beam transmitted, or diffracted, from the specimen
- The beam intensity variation contains the useful information about the location where beam is currently situated



STEM BF and ADF images from a semiconductor device

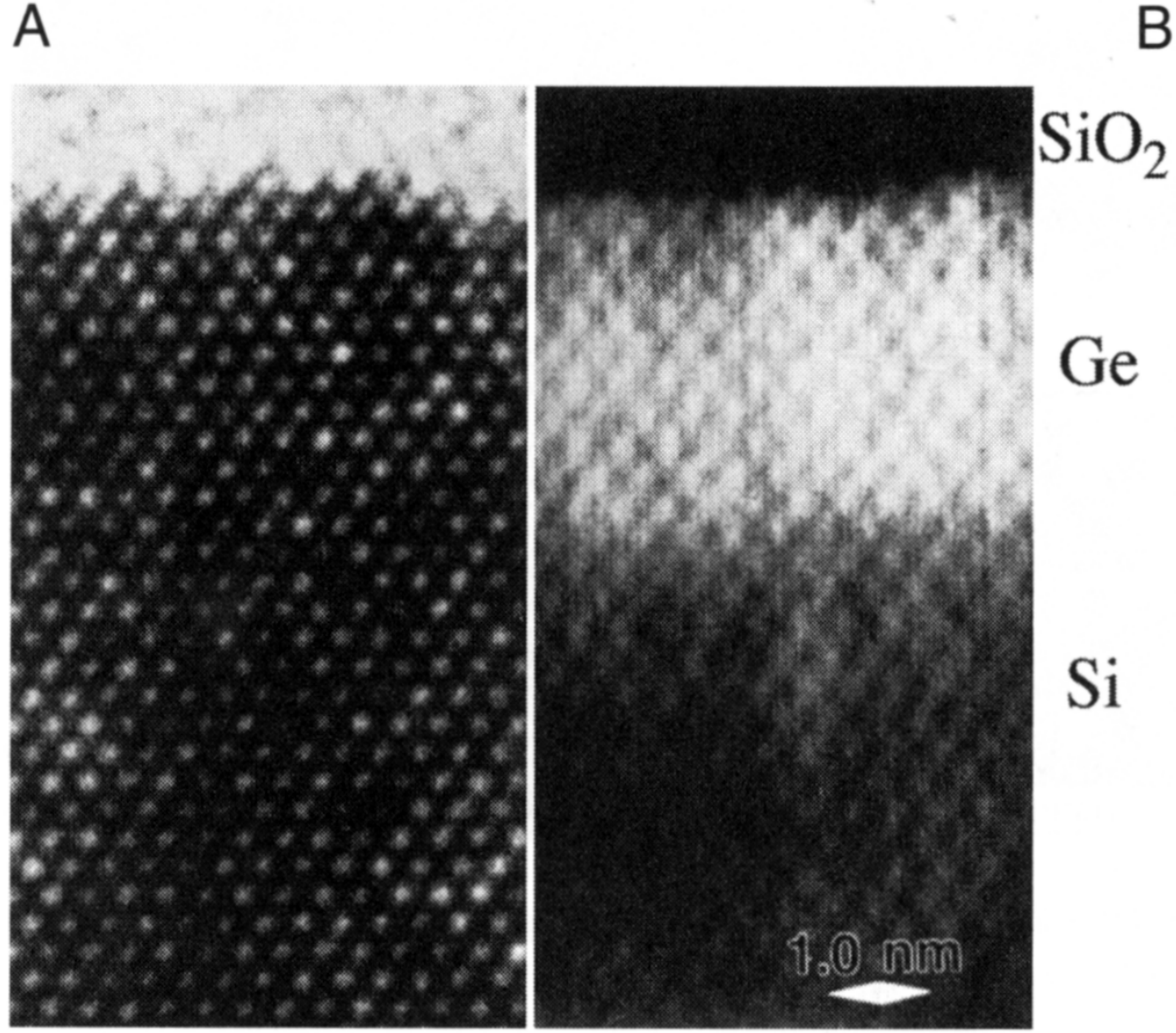


ADF



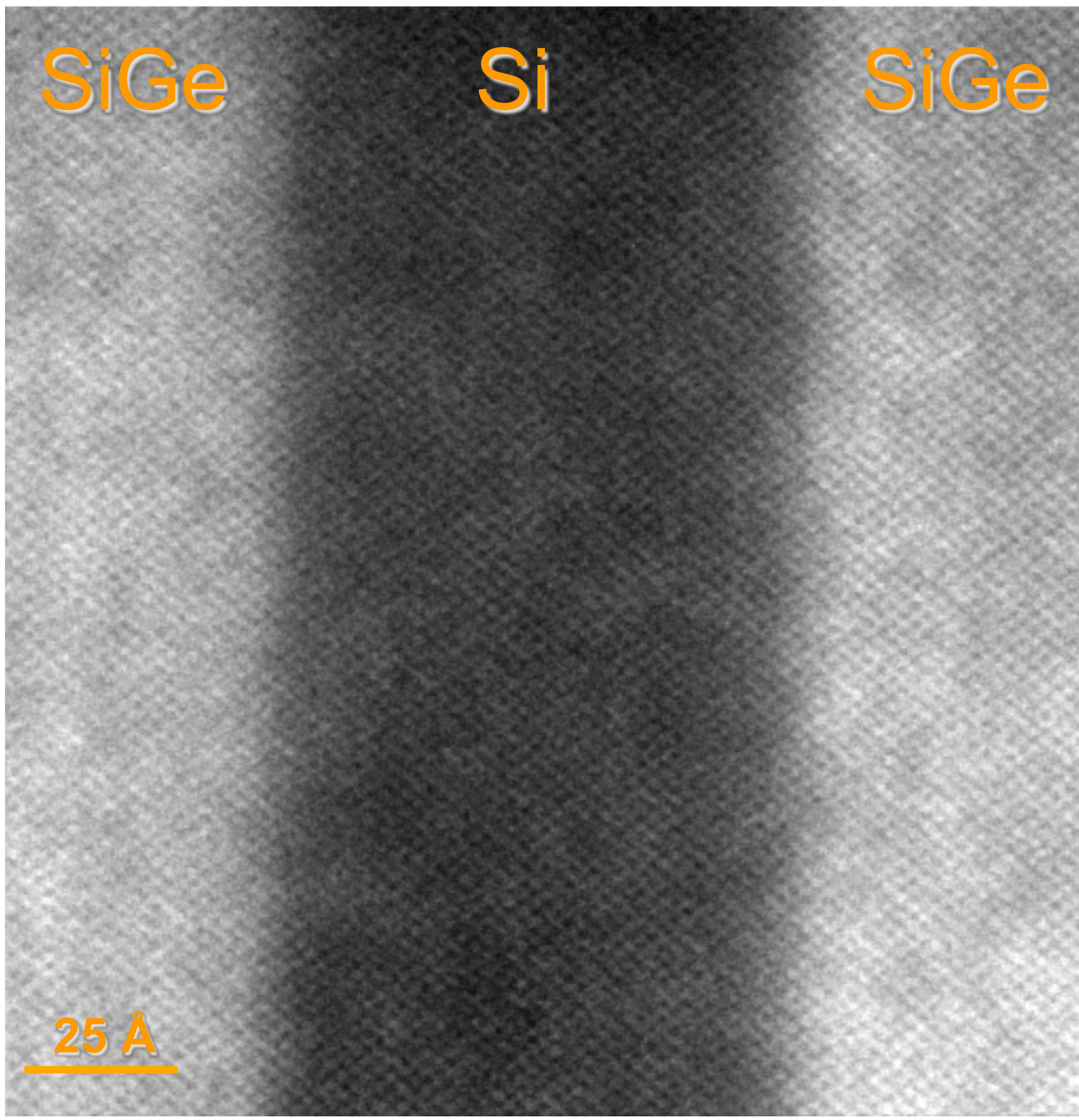
BF

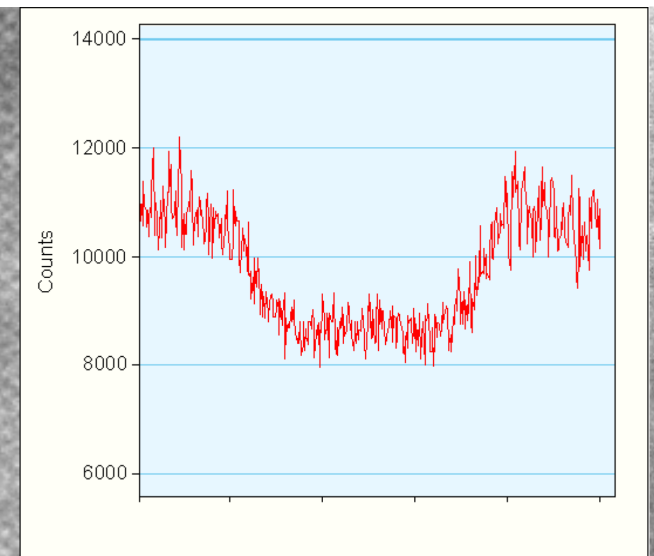
HREM vs. STEM HAADF Image — Interface

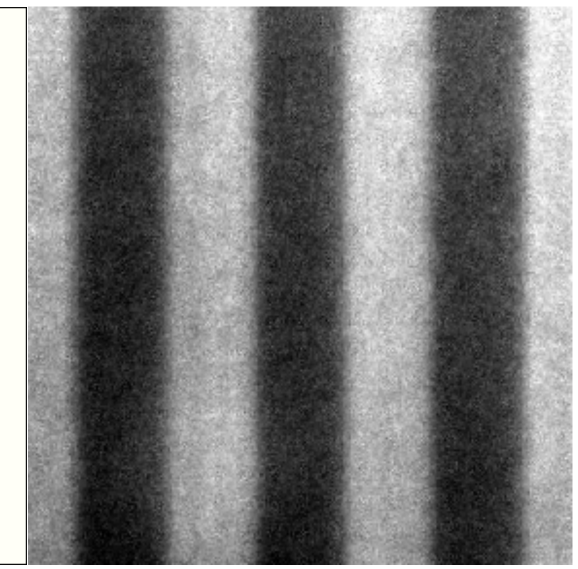


HRTEM STEM HAADF

HAADF image of SiGe alloy layers



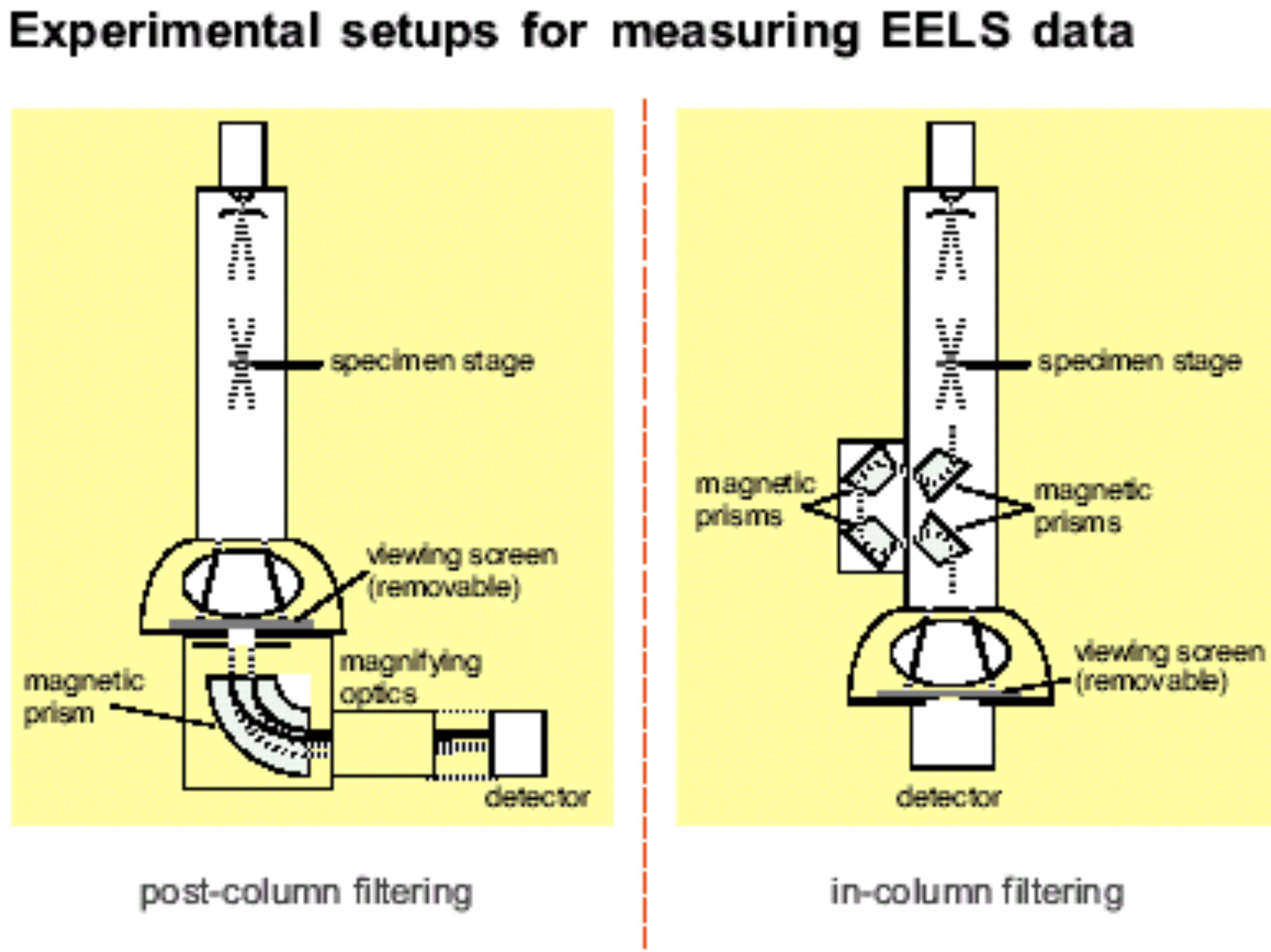




ADF image of $Si_{0.81}Ge_{0.19}$ alloy layers alternating with pure Si layers in a <110> orientation. Due to the Z -dependence of incoherent ADF STEM, the Ge rich layers are brighter than the pure Si. The interface between the layers is not sharp but shows a gradual decay.

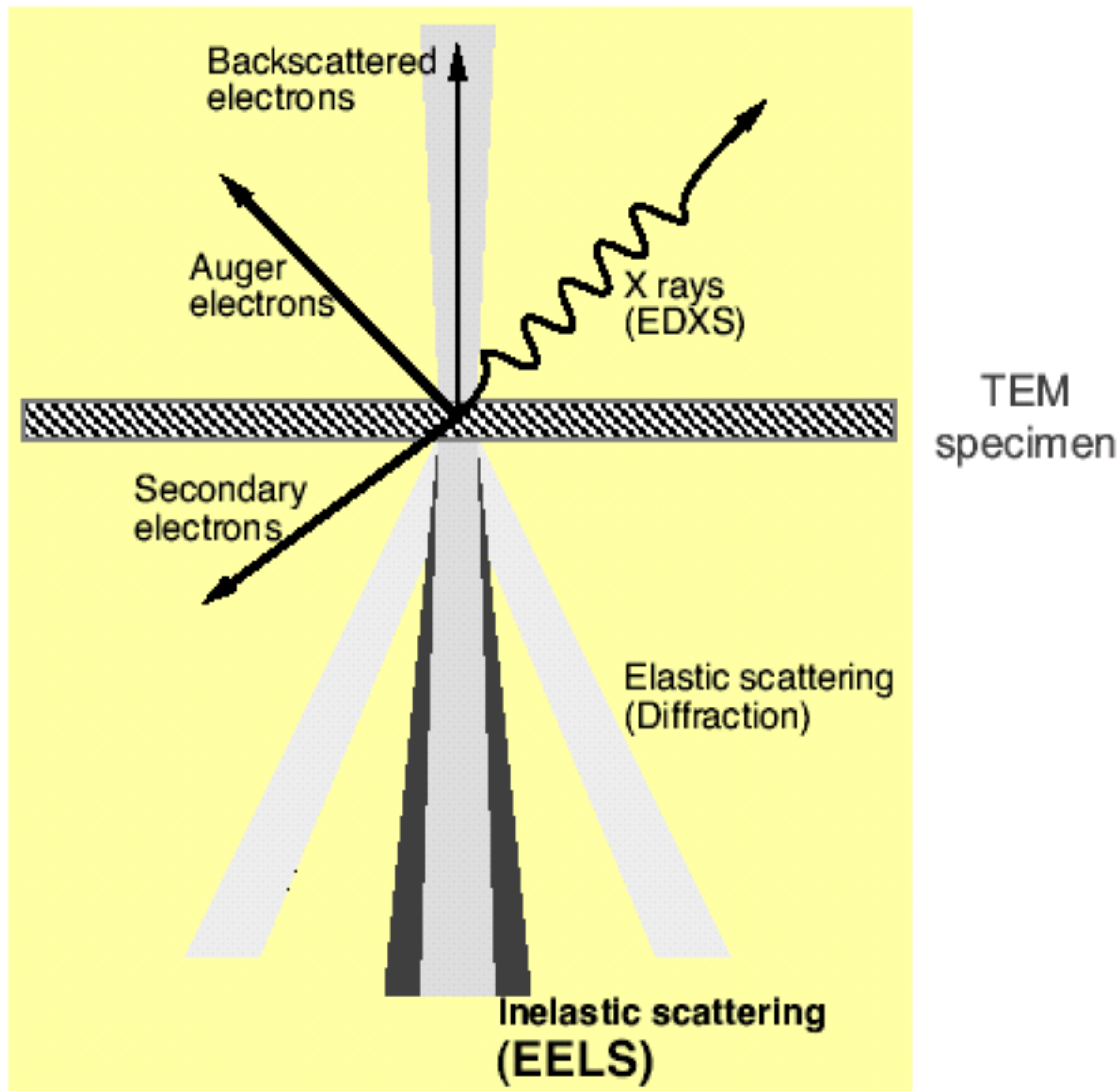
Tecnai F20 S-Twin

EELS configurations in TEM

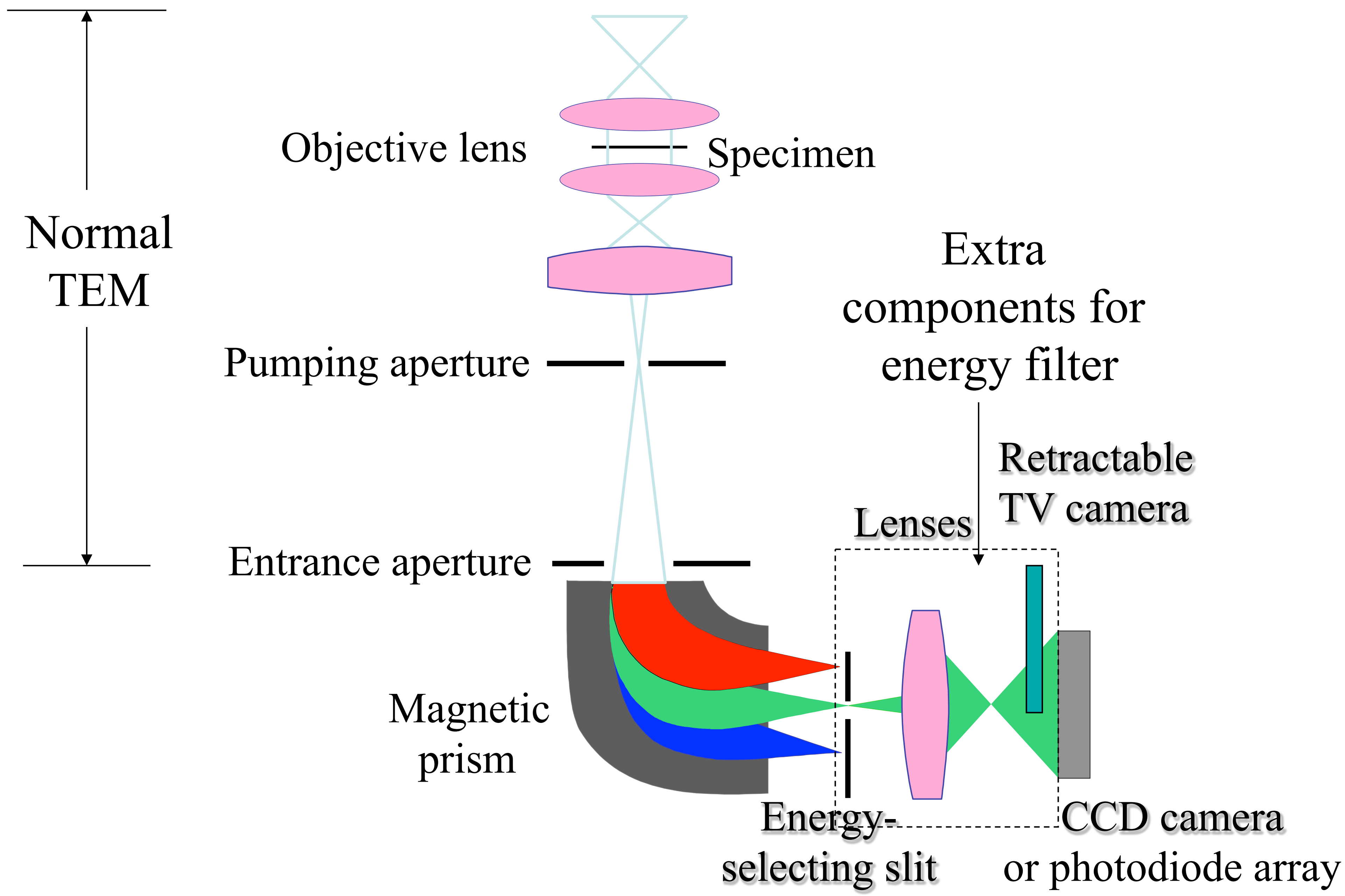


Signals for EELS

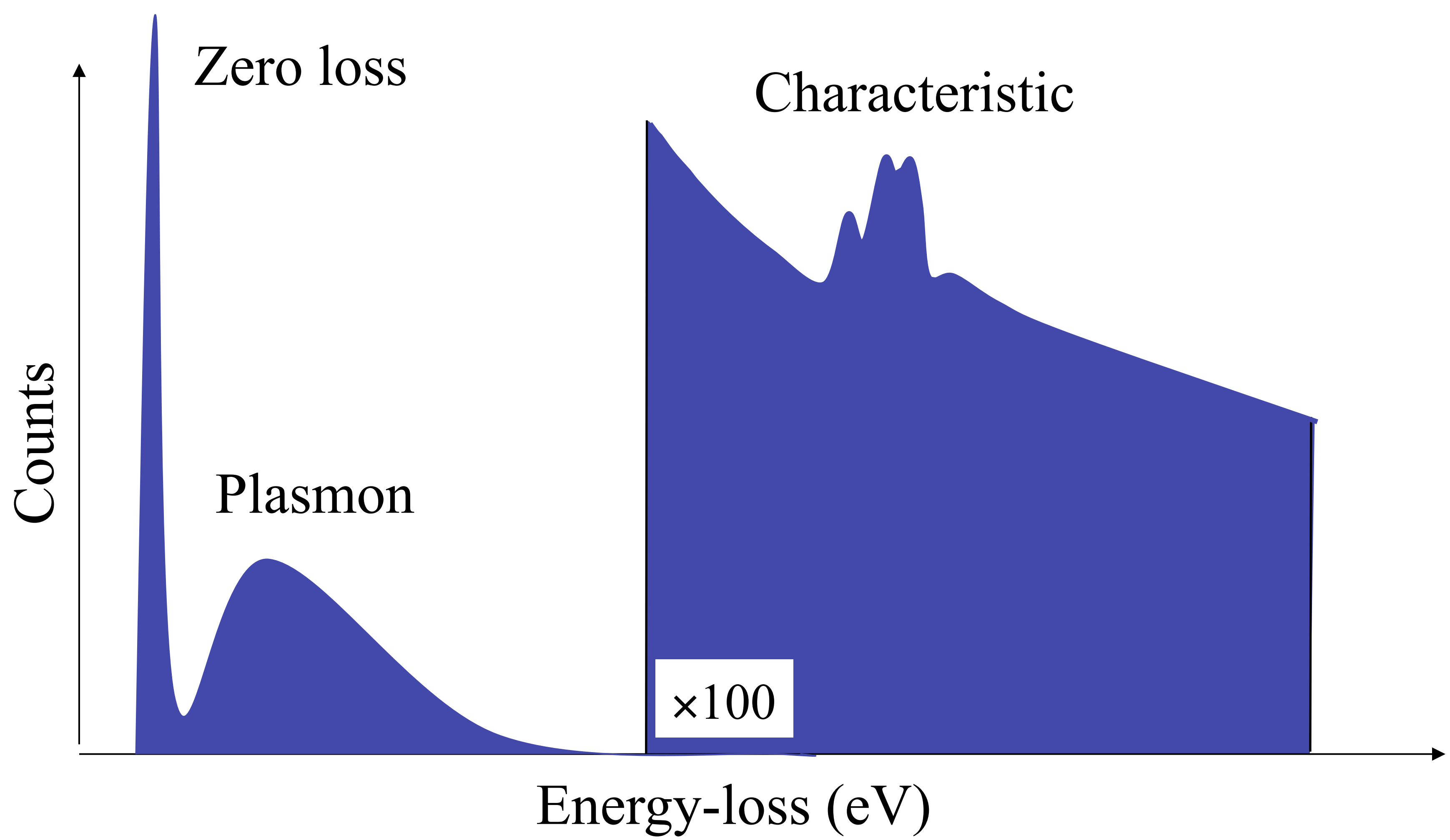
TEM beam-specimen interactions and signals



Post-column EELS

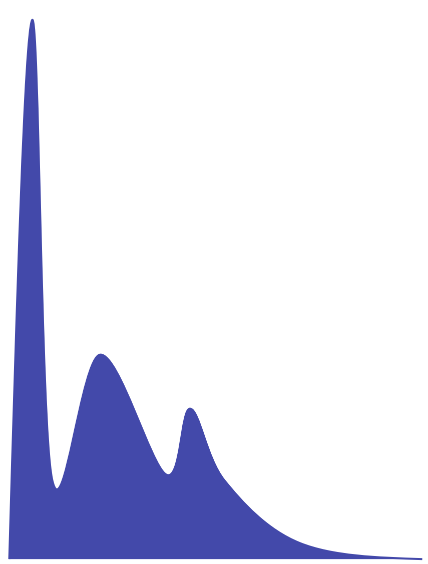


A typical EELS spectrum



Plasmon peak

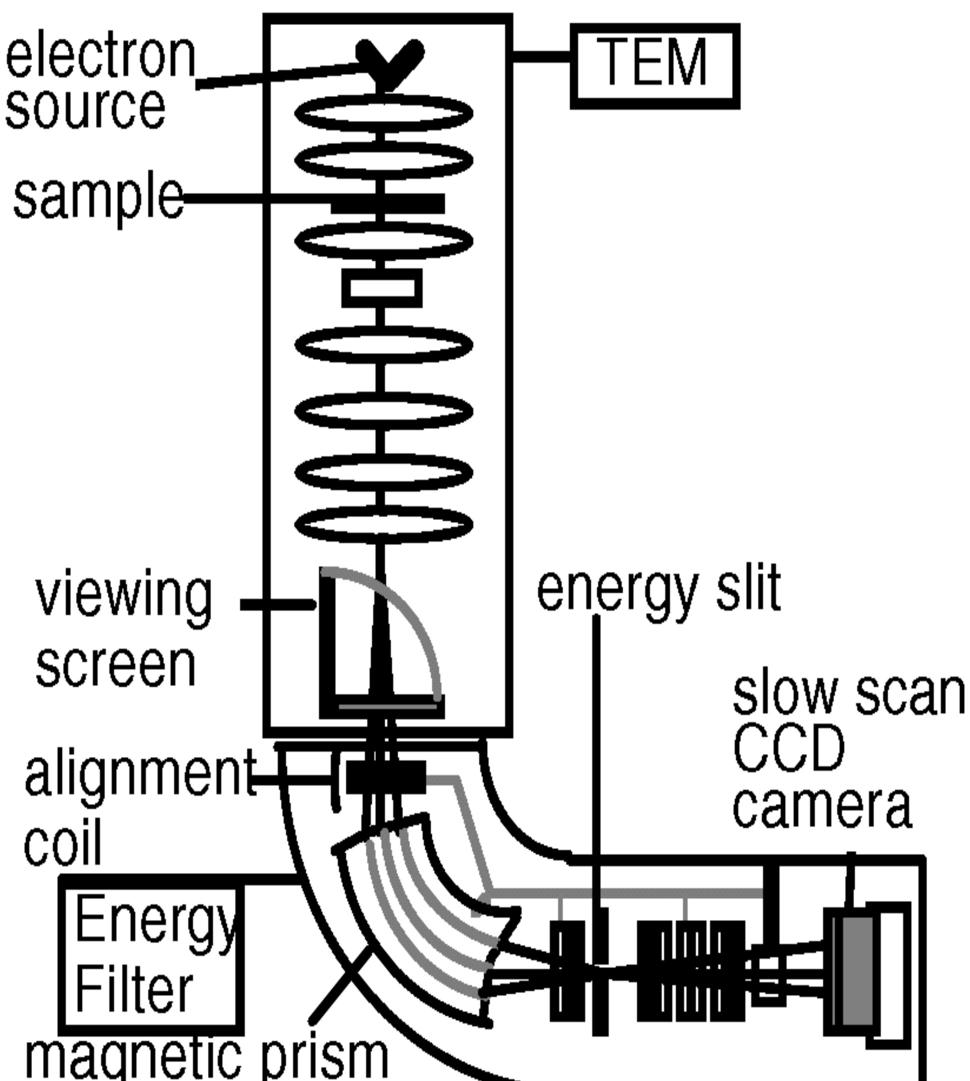
- Caused by the collective response to the incident beam by all the valence electrons
- If the sample is thicker, the plasmon peak is also higher and the second peak may appear
- The ratio of plasmon peak intensity to zero-loss peak intensity may estimate the sample thickness



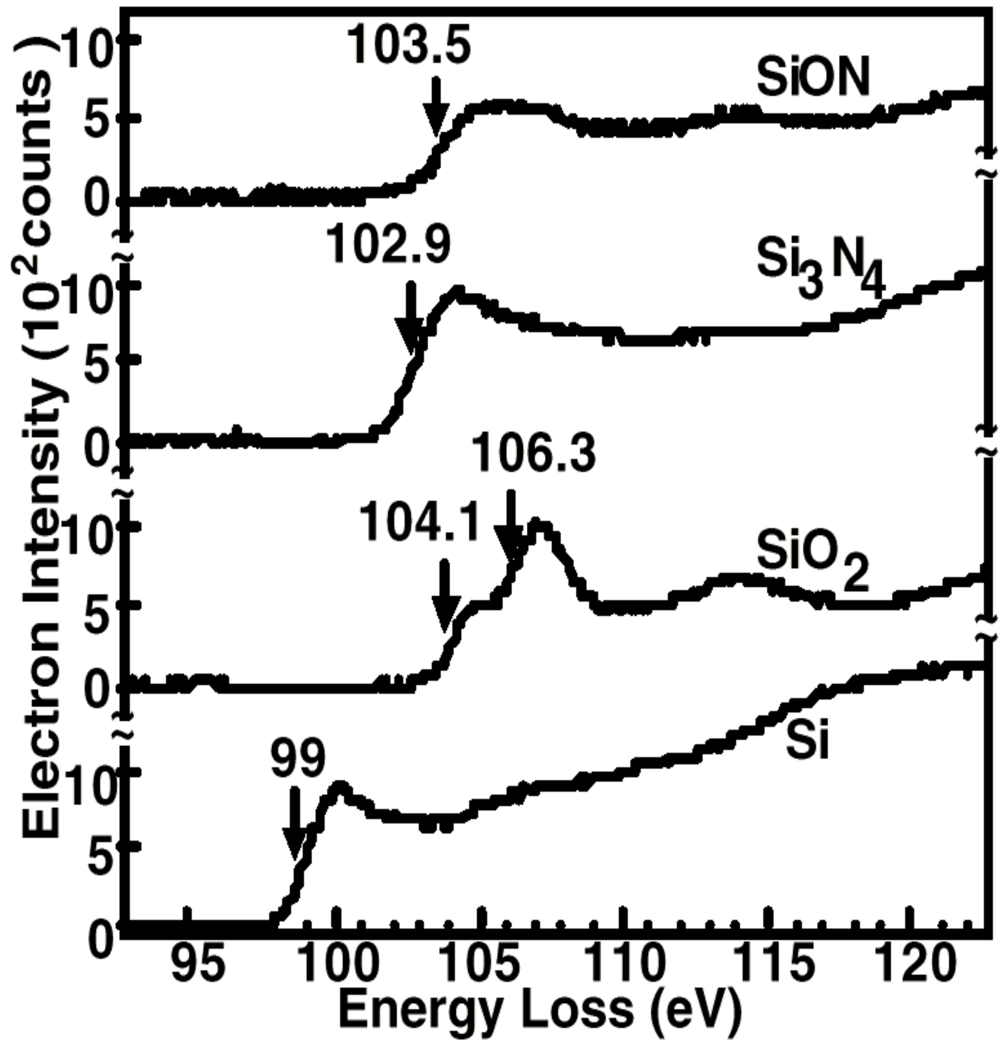
EELS vs. EDS

- More efficient signal collection
  - the first order phenomenon
  - most of the transmitted electrons enter the prism, comparing to 1% X-rays being detected
- Better signal to noise ratio
- Spectrum is electronic structure sensitive, e.g. O peaks in MnO and NiO are different in shape
- Slightly better spatial resolution
- Very high background and worse peak to background ratio, leading to the large error in quantification
- Complex peak structure makes identification difficult, it is worst when there is peak overlap
- Thin sample needed
- Operation and interpretation are more difficult

EELS for light elements



Schematic diagram of AEM-EELS



Chemical shift of core-loss edge energy in EELS spectra of some Si compounds

Y. Mitsui et al., IEDM'98