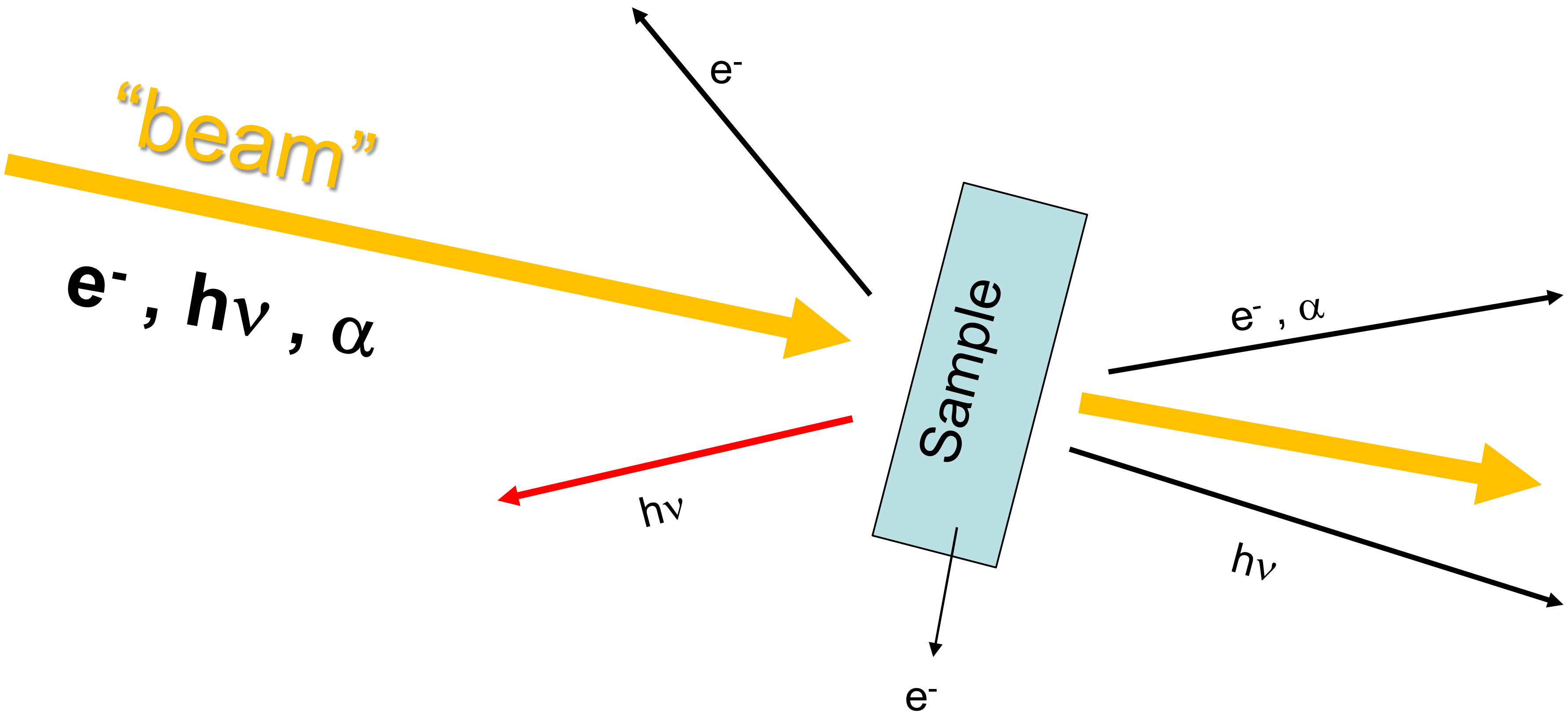
particle beam analysis and spectroscopy (Electron Microscopy, surface analysis, tomography)

An Introduction

"actio = reaction"



methods matrix

Primary particle	Secondary particle			
	hv	electron	ion	atom
hv	ELL, ESR, FTIR, NMR, Raman, XRD, SNOM	XPS UPS, XPD	LAMMA **APT**	
e	EDX	AES, (EELS), LEED, RHEED, SEM		
i +/-	PIXE, GDOES		GDMS SIMS ISS, RBS	
a				FAB He SC

- APT: Atom Probe Tomography
- ELL: Ellipsometry
- ESR: Electron Spin Resonance
- FTIR: Fourier Transform Infrared Spectroscopy
- NMR: Nuclear Magnetic Resonance
- Raman Spectroscopy
- XRD: X-ray Diffraction
- XPS: X-ray Photoelectron Spectroscopy
- UPS: Ultraviolet Spectroscopy
- XPD: X-ray Photoelectron Diffraction
- LAMMA: Laser Microprobe Mass Analysis
- EL: Electron Microprobe
- AES: Auger Electron Spectroscopy
- EELS: Electron Energy Loss Spectroscopy
- LEED: Low Energy Electron Diffraction
- RHEED: Reflection High Energy Electron Diffraction
- SEM: Scanning Electron Microscopy
- PIXE: Proton Induced X-ray Emission
- GDOES: Glow discharge Optical Emission Spectr.
- GDMS: Glow Discharge Mass spectroscopy
- SIMS: Secondary Ion Mass Spectroscopy
- ISS Ion Scattering Spectroscopy
- RBS: Rutherford Back Scattering
- FAB: Fast Atom Bombardment
- He Sc: Helium Scattering

Outline

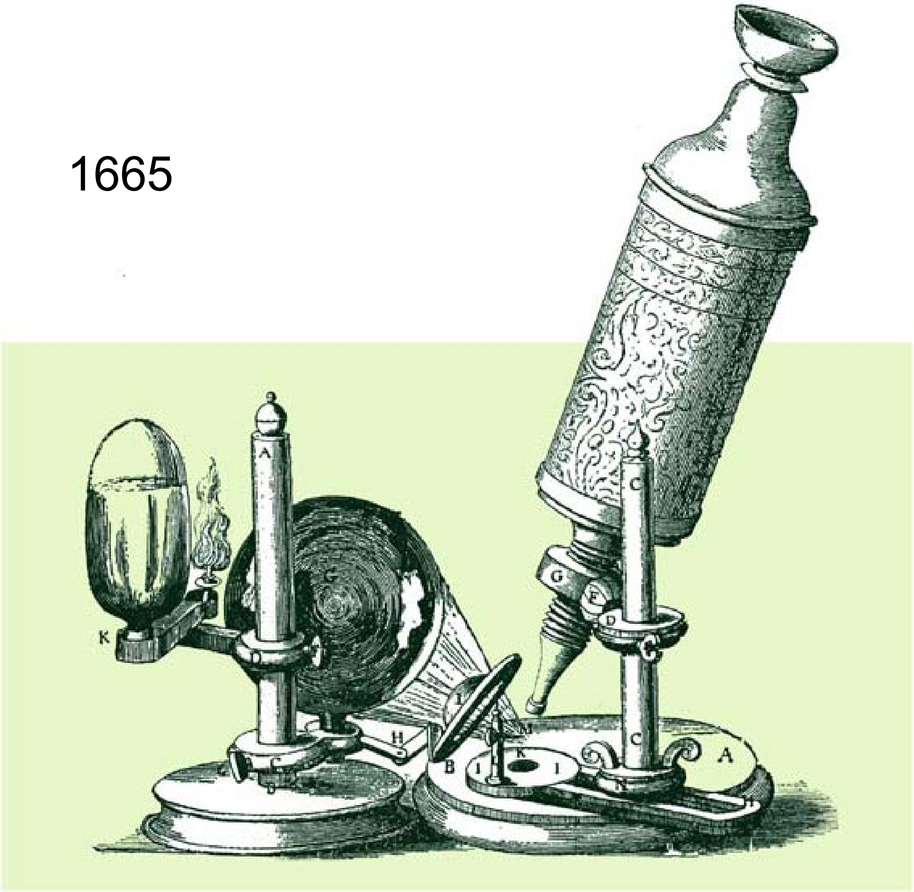
1. Introduction to (electron) microscopy
2. SEM, Imaging and EDX Analysis
3. Surface Analysis (XPS,RBS,AES)
4. Electron diffraction and TEM
5. STEM and Analytical TEM
6. 3D: FIB-NT, Tomography, APT

1. Introduction to electron microscopy

- Historical introduction, overview
- Components of an electron microscope (sources, lenses, detectors)

Microscopes…

1665

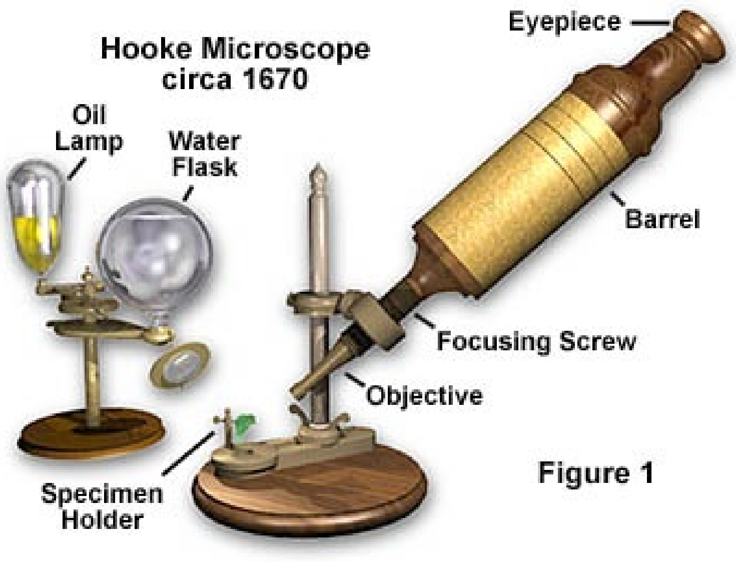


http://www.ucmp.berkeley.edu/history/hooke.html

today

Optical Microscopy

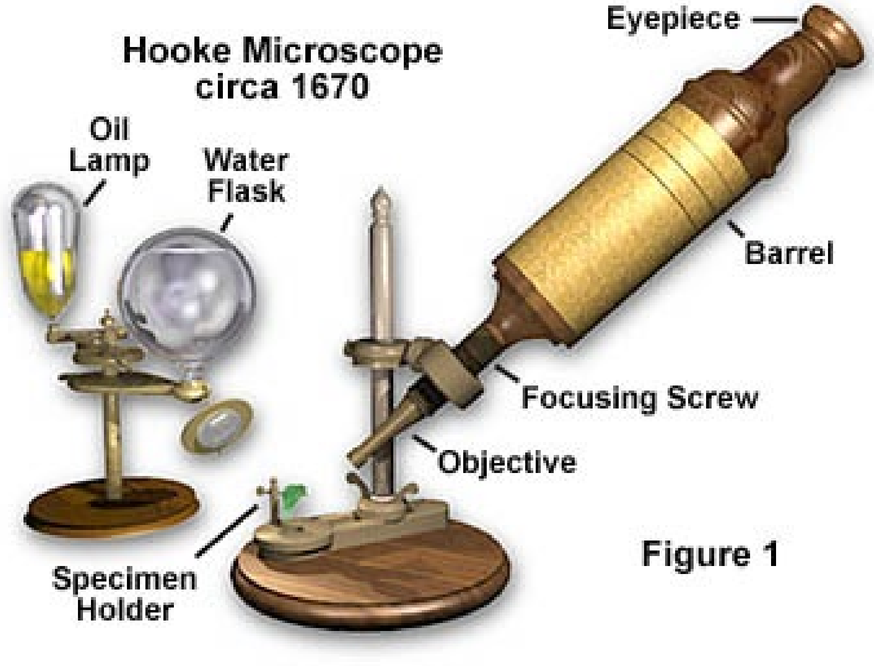
- *Antique: first convex lenses*
- *XII-XIII century:* magnifying power of convex lenses: magnifying glass, looking glasses
- *1590:* Janssen, first compound microscope
- *1609* Galilei: Occhiolino
- *1665* Hooke: first image of biological cell
- *1801* Young: wave character of light
- *1872 (~)* Abbe: resolution limit linked to the wave length of the illuminating wave



a microscope is…

- a light source
- an illumination system
- a sample
- a magnifying lens system
- a detector (eye, photographic plate, camera…)

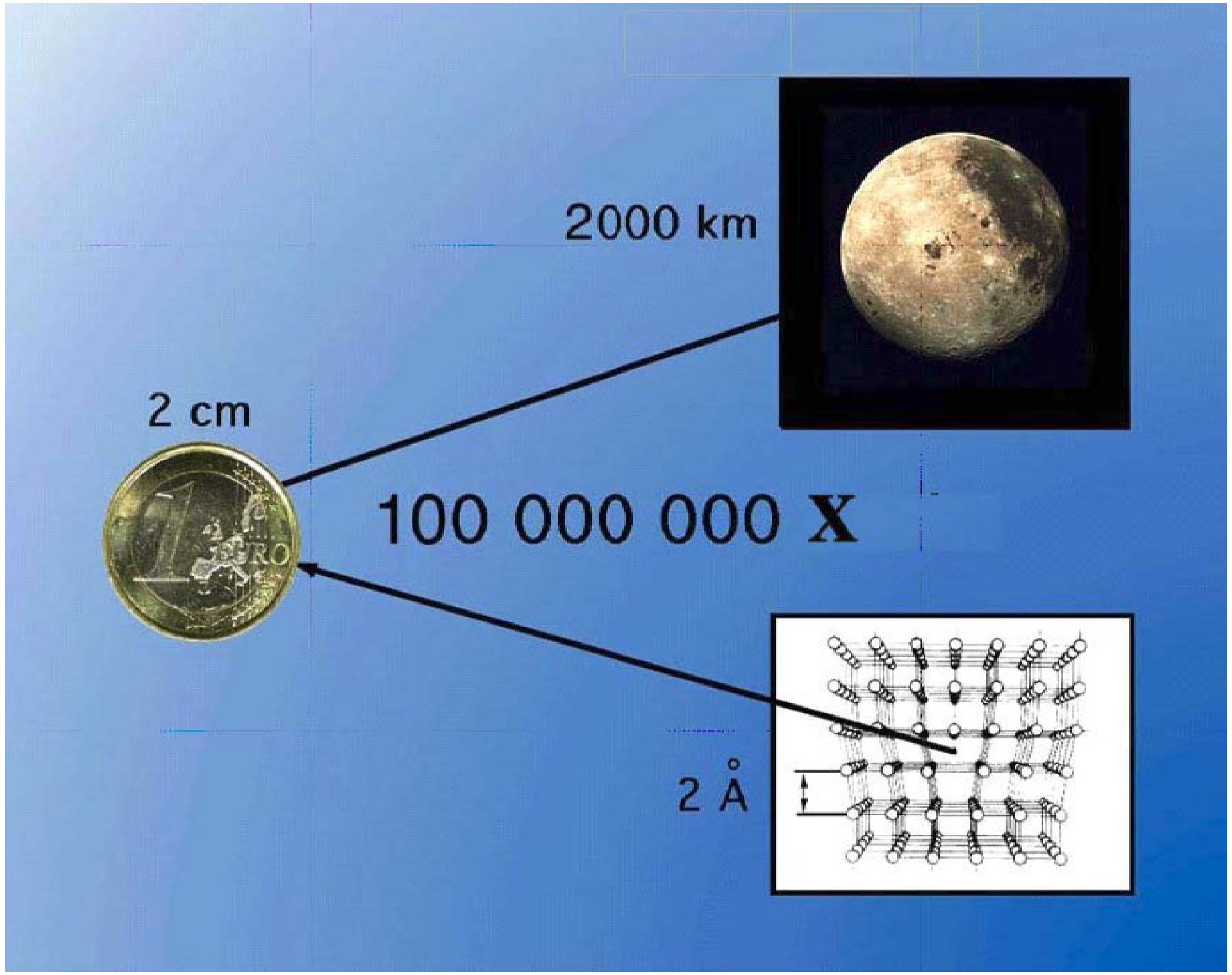
in principle…



http://micro.magnet.fsu.edu/primer/index.html

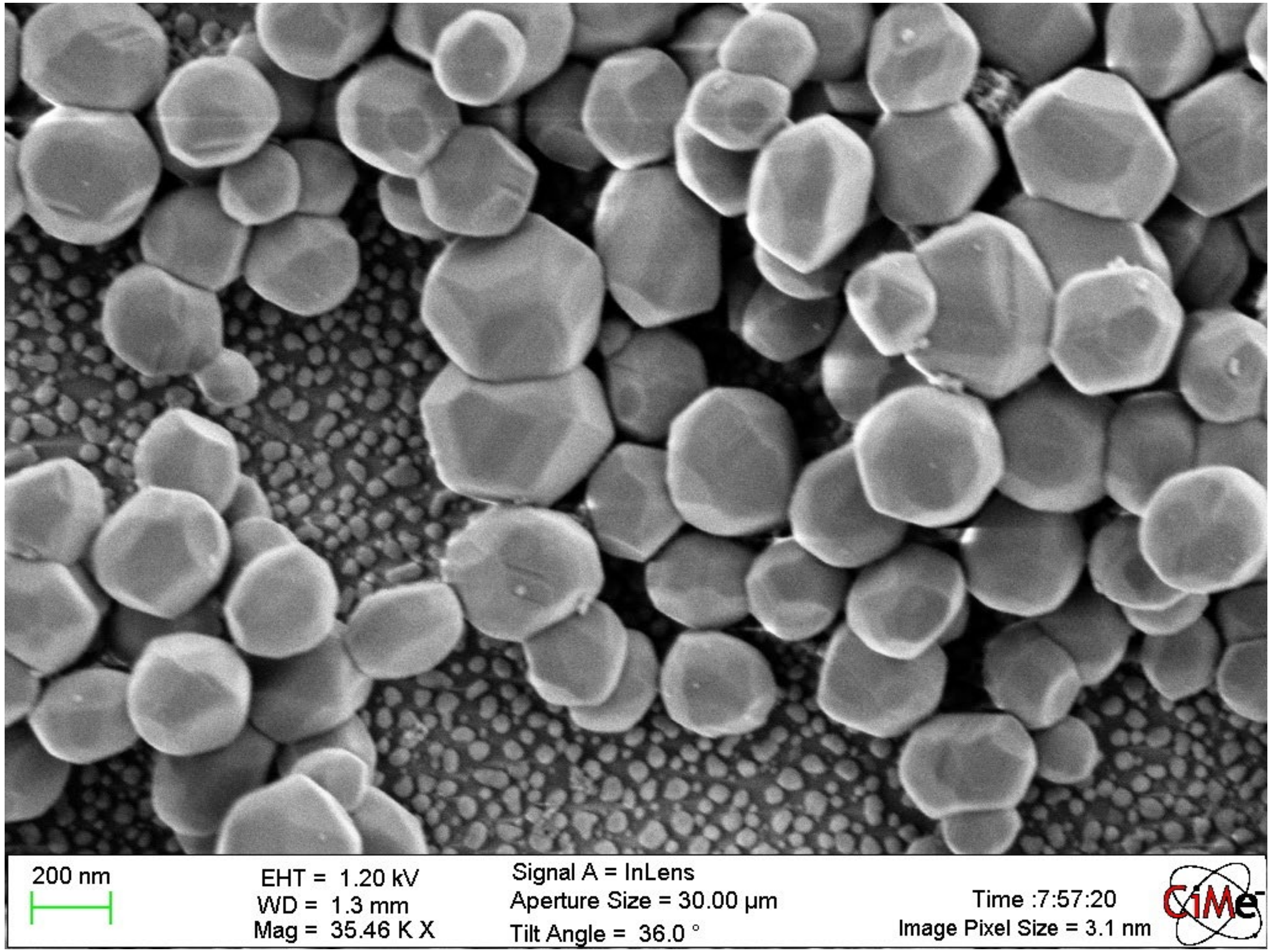
magnification

- Ratio between
  -the apparent size of an object on the image (paper, screen, eye)
  and
  -the real size of the object



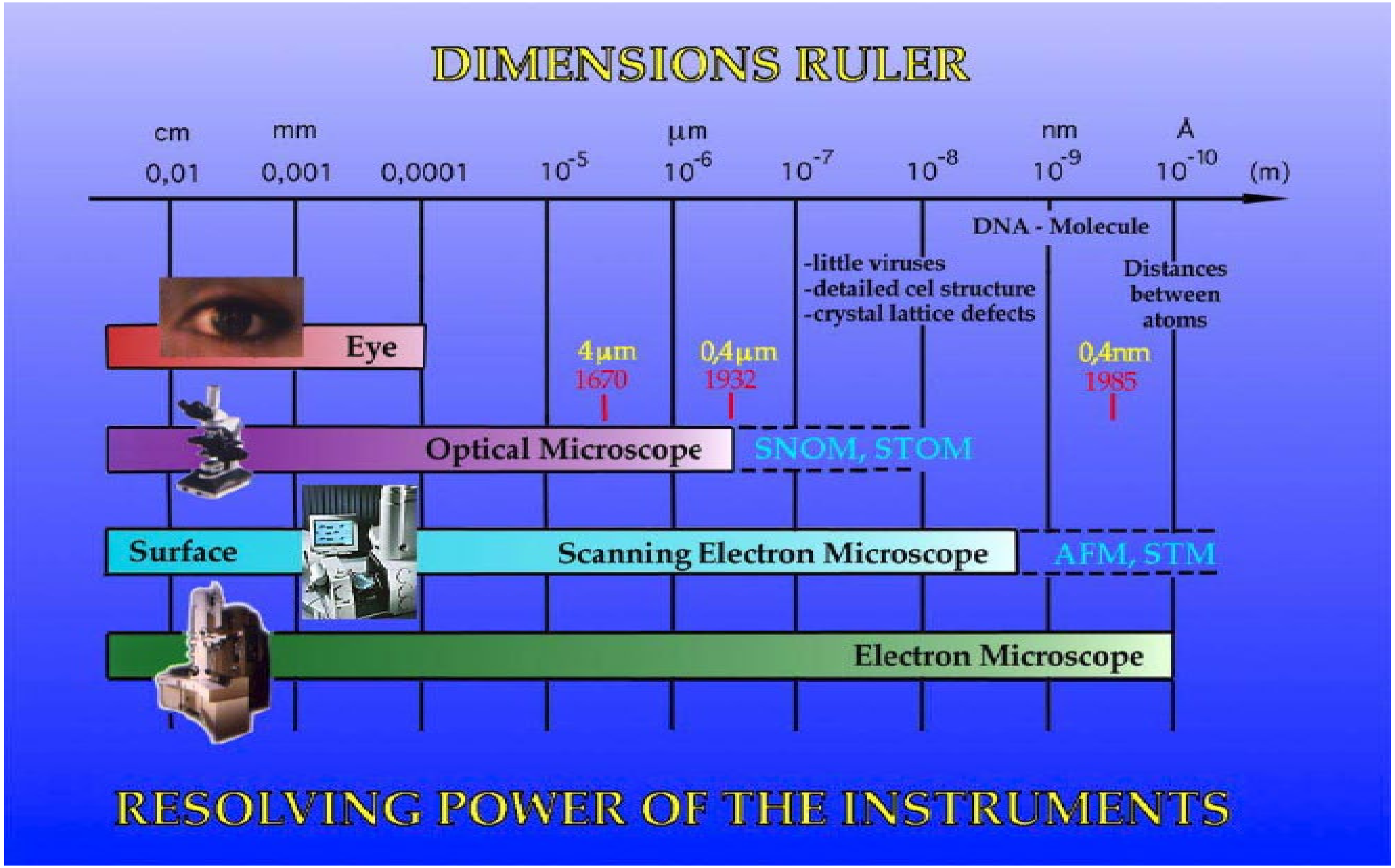
Which magnification…?

Measure the size of a grain on the screen and calculate the magnification !



Who is lying ? The scale bar or the indicted magnification……?

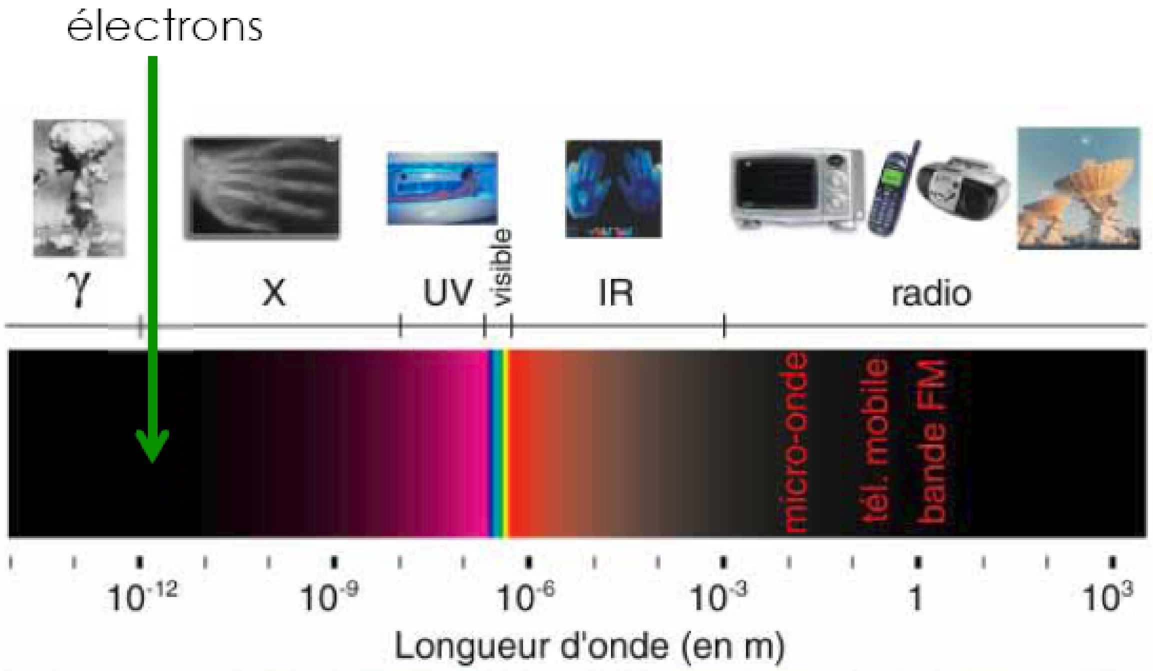
resolution


DIMENSIONS RULER
cm
mm
µm
nm
Å
0,01
0,001
0,0001
10-5
10-6
10-7
10-8
10-9
10-10
(m)
DNA - Molecule
-little viruses
-detailed cel structure
-crystal lattice defects
Distances between atoms
Eye
4 µm
1670
0,4 µm
1932
0,4nm
1985
Optical Microscope
SNOM, STOM
Surface
Scanning Electron Microscope
AFM, STM
Electron Microscope
RESOLVING POWER OF THE INSTRUMENTS

Why electrons…?

Table 1.2. Electron Properties as a Function of Accelerating Voltage

Accelerating voltage (kV)	Nonrelativistic wavelength (nm)	Relativistic wavelength (nm)	Mass ($\times m_0$)	Velocity ($\times 10^8$ m/s)
100	0.00386	0.00370	1.196	1.644
120	0.00352	0.00335	1.235	1.759
200	0.00273	0.00251	1.391	2.086
300	0.00223	0.00197	1.587	2.330
400	0.00193	0.00164	1.783	2.484
1000	0.00122	0.00087	2.957	2.823



$$\lambda = \frac{h}{\left[2m_0 eV\left(1 + \frac{eV}{2m_0c^2}\right)\right]^{1/2}}$$

electrons

Table 1.1. Fundamental Constants and Definitions

Charge (e)	(−) 1.602×10^{-19} C
1 eV	1.602×10^{-19} J
Rest mass (m_0)	9.109×10^{-31} kg
Rest energy (m_0c^2)	511 keV
Kinetic energy (charge × voltage)	1.602×10^{-19} N m (for 1 volt potential)
Planck's constant (h)	6.626×10^{-34} N m s
1 ampere	1 C/sec
Speed of light in vacuum (c)	2.998×10^{8} m/sec

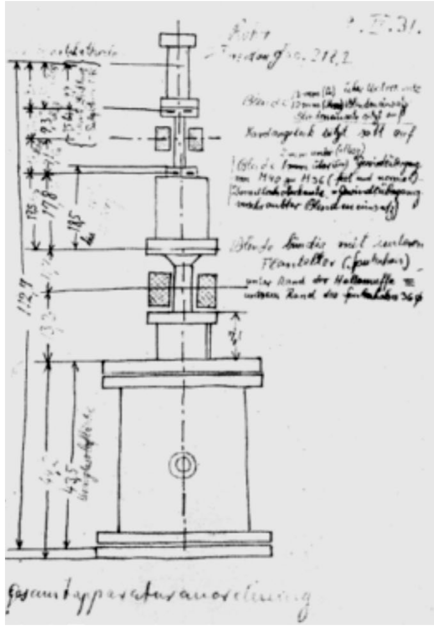
History

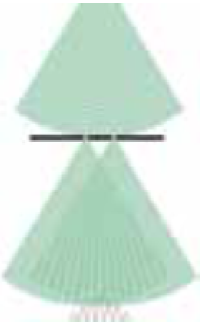
1897: J.J. Thomson: predicts the existence of electrons

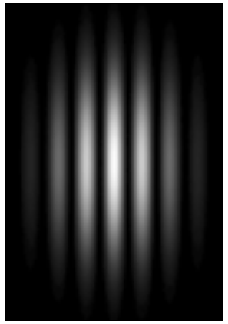
1923: De Broglie: concept of wavelength associated to particles, confirmation by Young's experiment

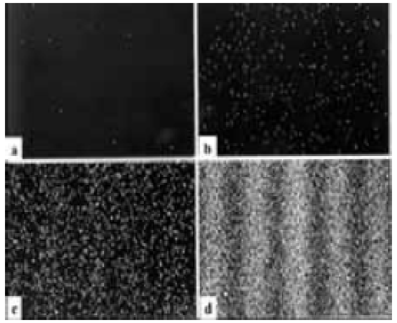
1927: Busch: focalisation law for magnetic fields, Davisson, Gremer, Thomson: electron diffraction

1931: Ruska, Knoll: first images by electron microscope

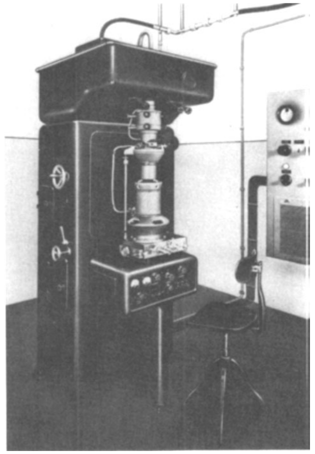






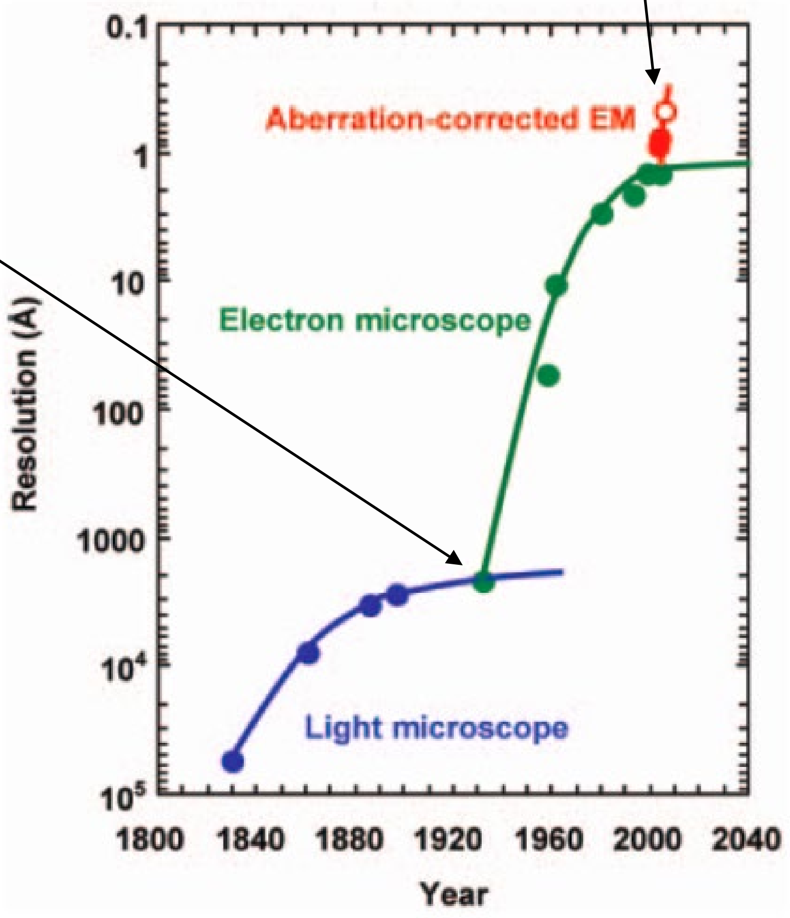


History



Siemens 1939

FEI Titan 2009

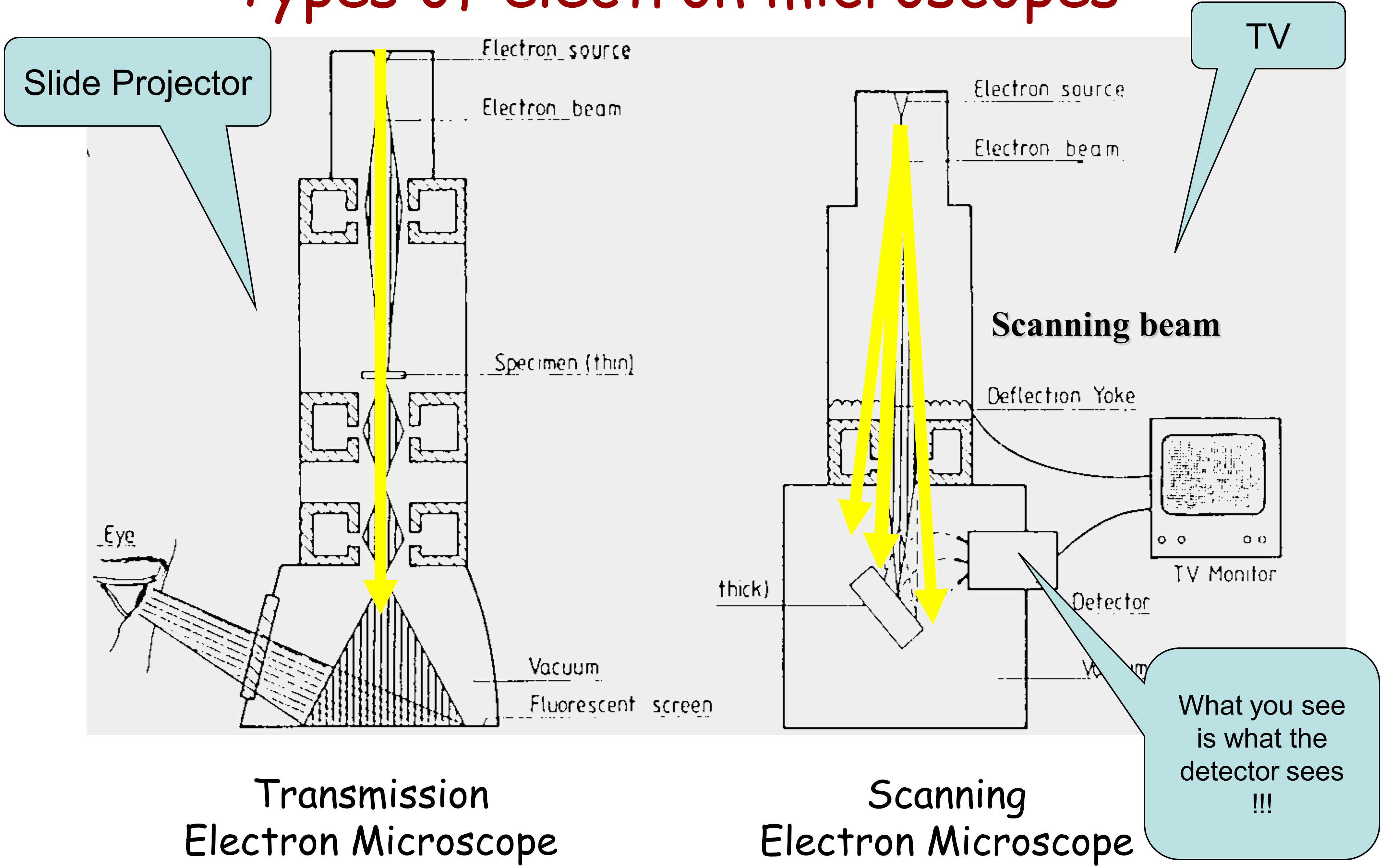


1936 Scherzer: main electromagnetic lens aberrations cannot be avoided
1938 Von Ardenne: first microprobe scanning electron microscope
1939 Siemens: first industrial electron microscopes
1948 Gabor: holography invention
1951 Castaing: first X-ray micro-analyser
1960 XX: first MV microscope, competition for resolution
1965 Crewe: first scanning transmission electron microscope
1982 Binnig et Rohrer: scanning tunnelling microscope
1986 Ruska, Binnig et Rohrer: Prix Nobel Physiqcs
1990 Rose: proposes the Cs corrector principle
1995 Haider: first realisation of the Cs corrector

Comparison of different microscopes

Microscope	Resolution	Depth of focus	Interaction / matter	Environment	pixels/image
"optical" (photons)	~ 0.3-1 μm	low <1μm@10^3x	light (electro-magn. wave), insulating vs conductor	transparent: air, gas, vacuum, liquids, ≠ temperatures	4'000 x 3'000
SEM Scanning Electron Microscope	1-10 nm	large ~ 1mm@10^3x	charged particle / electric (+magn) field, secondary +backscattered e-, interband trans.+ionisation	vacuum (+recently: gas p<50mbar)	1000 x 1000 (4000 x 3000)
TEM Transmission Electron Microscope	0.2nm	Large, but thin sample...	charged particle / electric (+magn) field, e- scattering and diffraction	vacuum (gas or liquid in "environmental cells"	4000 x 3000 (1000 x 1000)
STM Scanning Tunneling Microscope	lateral 0.2nm, depth 0.01nm	very low 0.01nm	e- tunnelling, density of empty electronic state	dielectric media: vacuum, air, gas and liquid	512 x 512
AFM Atomic Force Microscope	lateral ~ 1nm, depth 0.1nm	very low 0.1nm	atomic forces (van der Waals, covalent, ionic), friction, electrostatic +magnetic forces	vacuum, air, gas and liquid	512 x 512

Types of electron microscopes



Transmission Electron Microscope

Scanning Electron Microscope

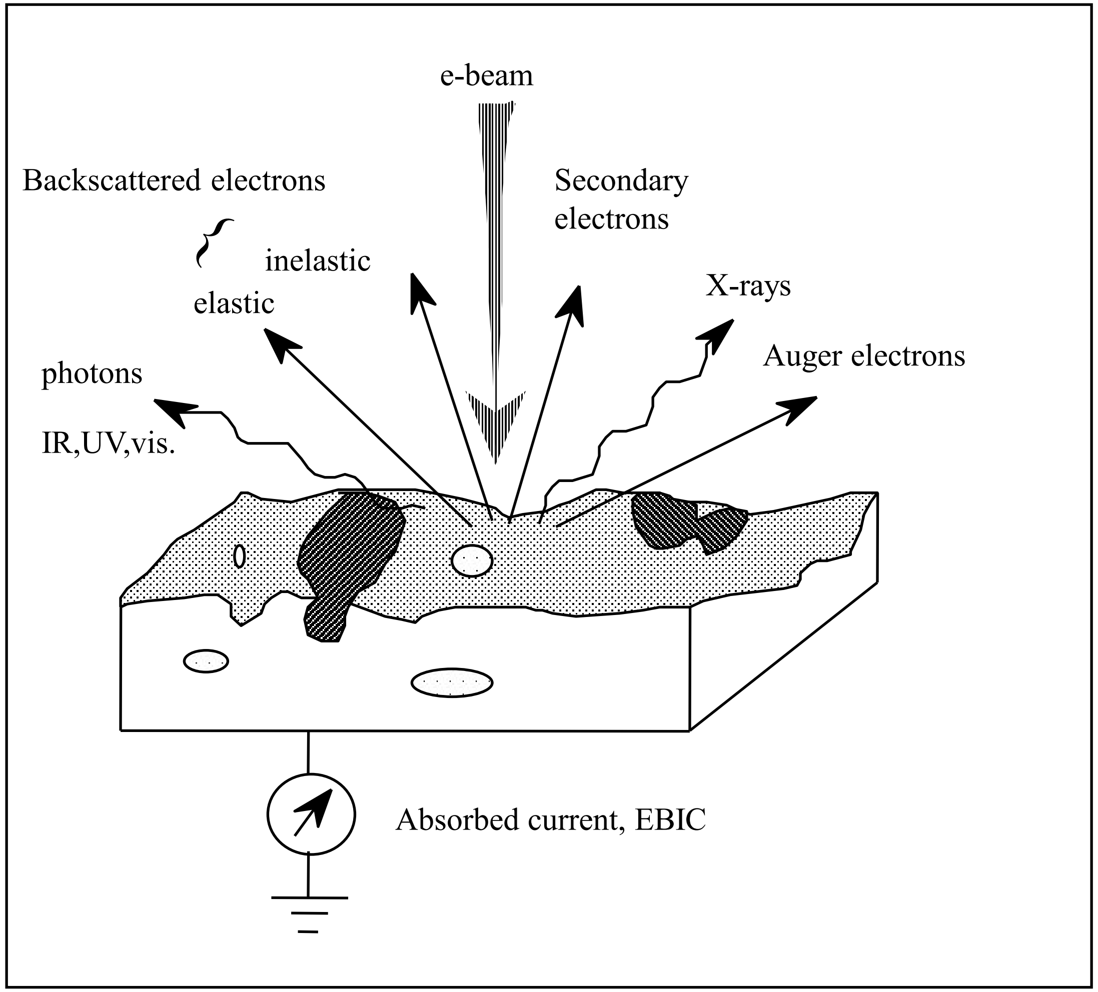
Types of microscopes

SEM

- 0.3-30keV
- Inelastic scattering: surface information: **topography**
- Chemical **composition** (EDX)
- Pseudo-elastic (back-) scattering: mass density information, (diffraction, EBSD)

- TEM
- 60-300keV (up to 3 MeV)
- "Projection" of the THIN sample
- Elastic scattering dominant: Diffraction and diffraction contrast
- Interference between transmitted and diffracted beams: high-resolution (atomic resolution)
- Inelastic scattering: chemical composition (EDX)

SEM, signals



- Secondary electrons (~0-30eV), **SE**
- Backscattered electrons (~eVo), **BSE**
- Auger electrons
- Photons: visible, UV, IR, **X-rays**
- Phonons, Heating
- Absorbtion of incident electrons (EBIC-Current)

Some typical SEM

At CIME:
Zeiss NVision40
(FIB)

Zeiss Ultra
HR-SEM

Resolution

1.0 nm @ 15 kV, 1.7 nm @ 1 kV, 4.0 nm @ 0.1 kV

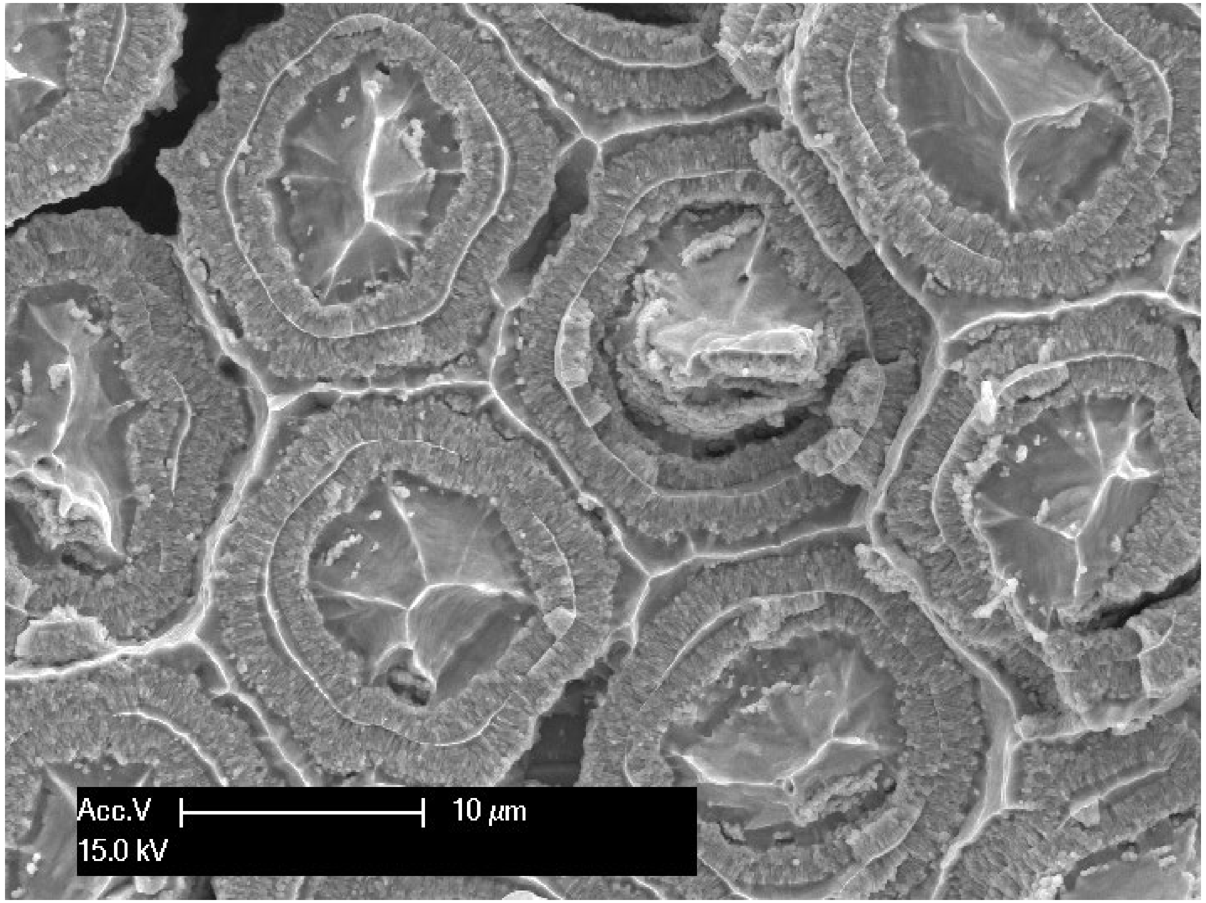
Magnification12 - 900,000x in SE mode
Acceleration Voltage0.02 - 30 kV

Probe Current4 pA - 10 nA

Standard Detectors:
EsB Detector with filtering grid
High efficiency In-lens SE Detector
Everhart-Thornley Secondary Electron Detector

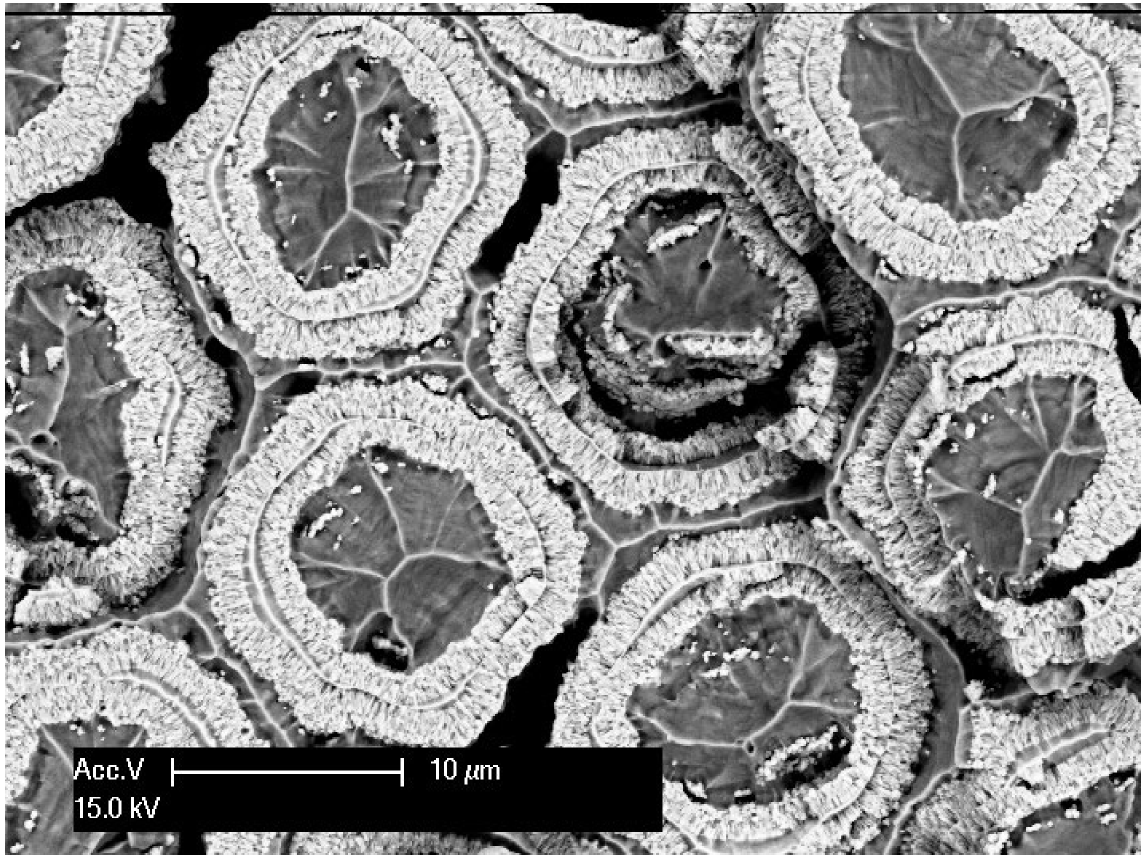
SEM, secondary electrons

- Electrons with low energy (0-50eV) leaving the sample surface
- Intensity depends on inclination of the surface
- topography



SEM, backscattered electrons

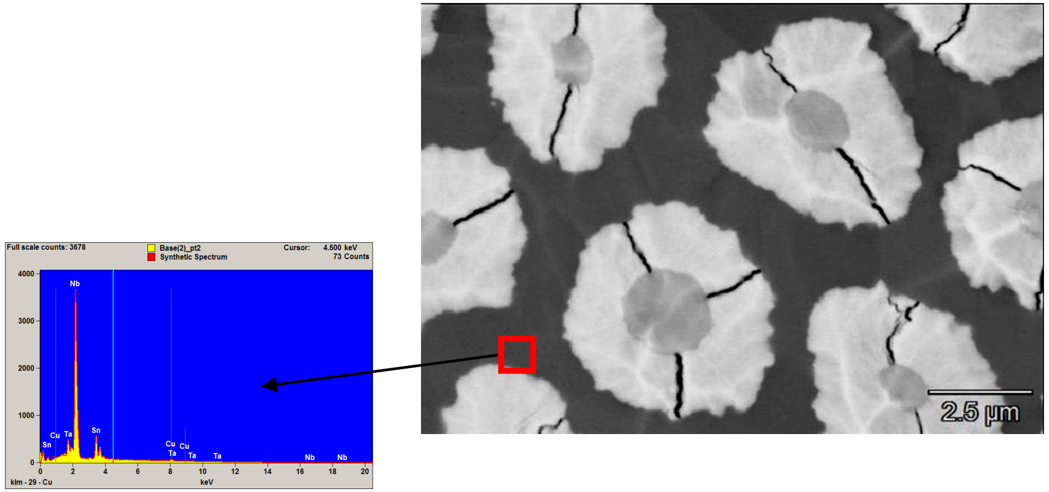
- Electrons with high energy (~Eo) backscattered from below the surface
- Intensity depends on density (atomic weight)
- ~composition



Nb_3Sn in Cu matrix

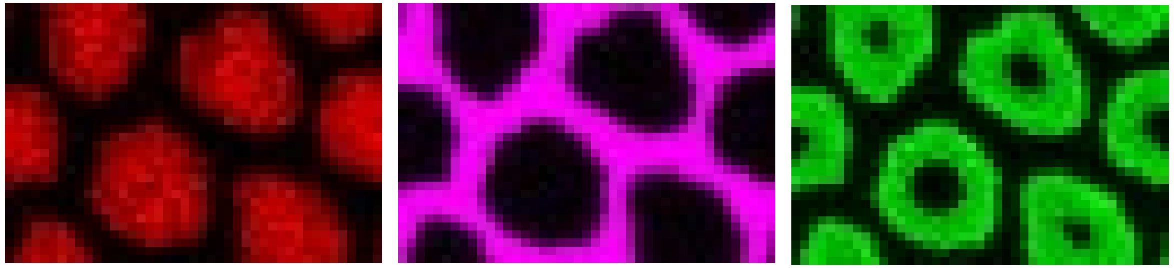
SEM, characteristic X-rays

- Incident electrons ionize atoms in the sample: Emission of characteristic X-rays when atoms relax
- Detection and analysis of X-ray allows determination of chemical composition

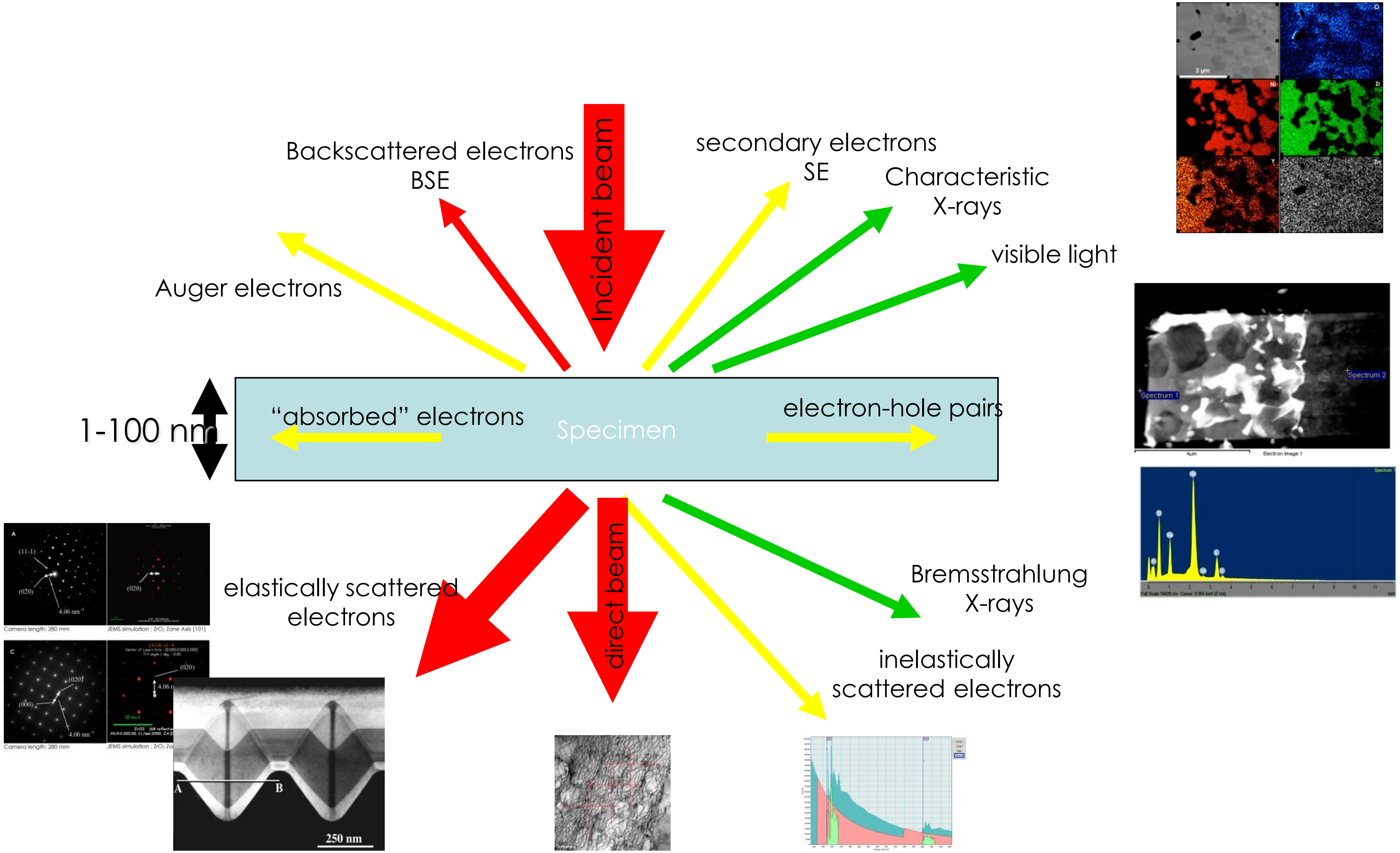


Acquire X-ray spectrum in each image point

Extraction of element maps



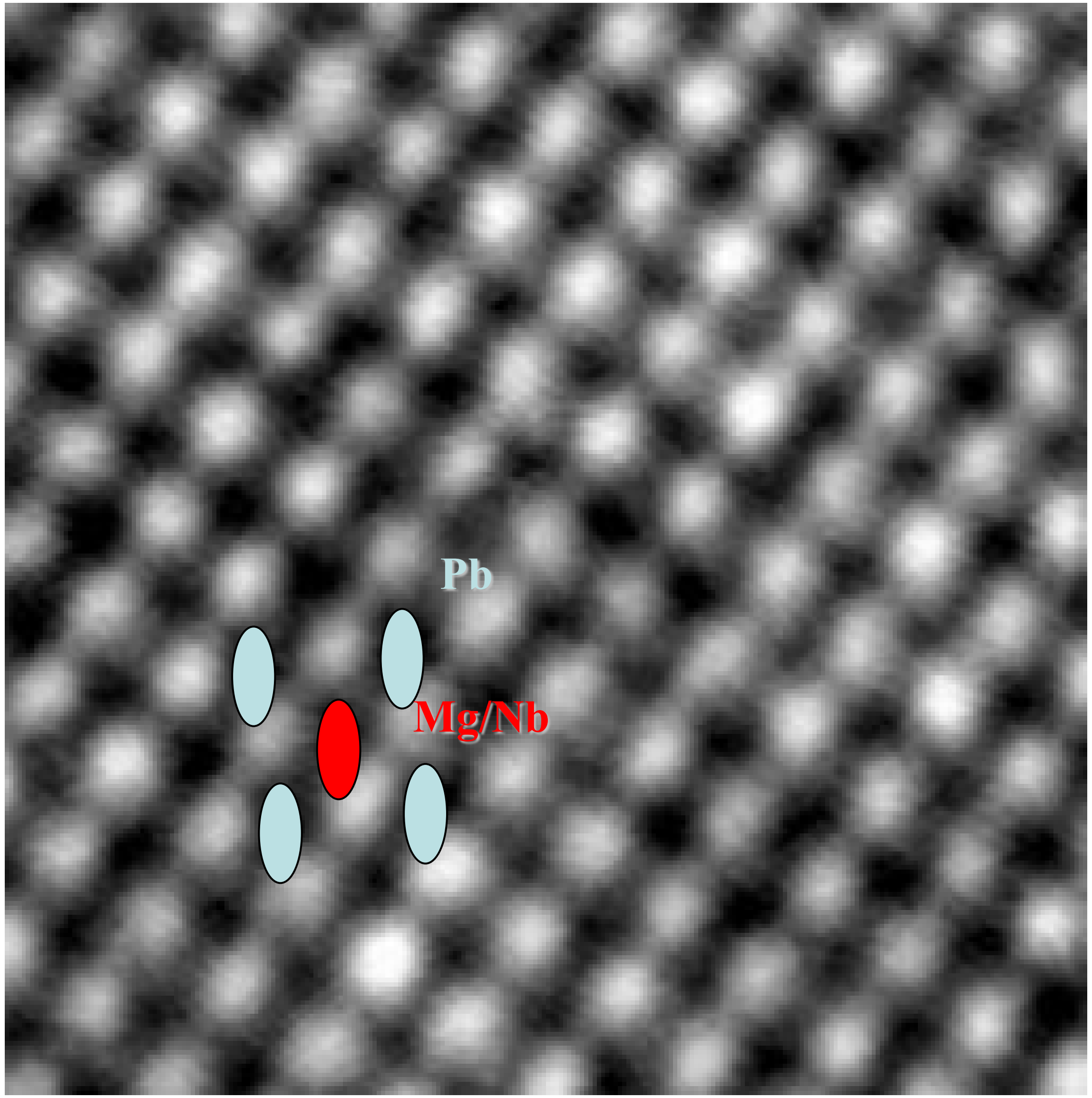
Signals from a thin sample



Some "typical" TEMs

Some "numbers"

- "Speed"of a 300keV electron: 76% of light-speed
- Wavelength of a 300'000V beam: 0.0197 Å ~2pm (0.002nm)
- Image resolution: ~1.7Å
- Typical scattering angles: 10^{-3} rad



TEM as a "multipurpose" machine

- **Atomic structure**
  - Diffraction (lattice parameters, orientations) **~1pm**
  - High resolution TEM **~0.2nm**
- **Microstructure & Defects**
  - Bright field image
  - Dark field imaging **~ μm - nm**

Elastic scattering

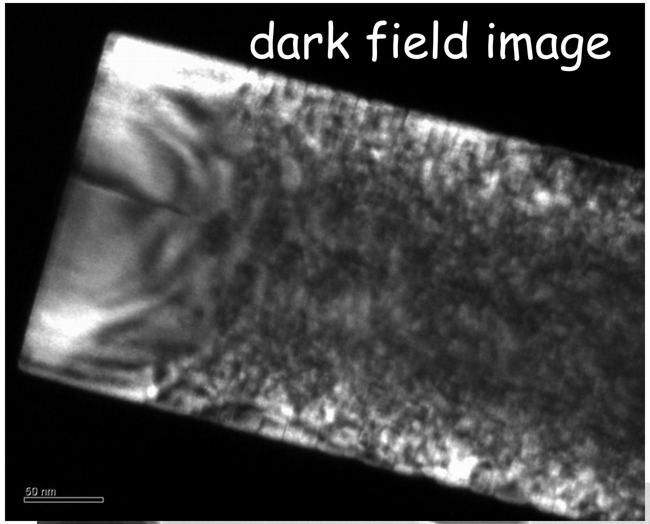
- **Chemistry**
  - Energy dispersive X-ray microanalysis EDX, **~1nm**
  - Electron Energy Loss Spectroscopy EELS

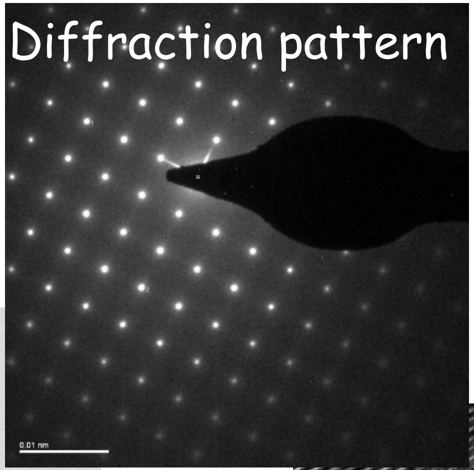
Inelastic scattering

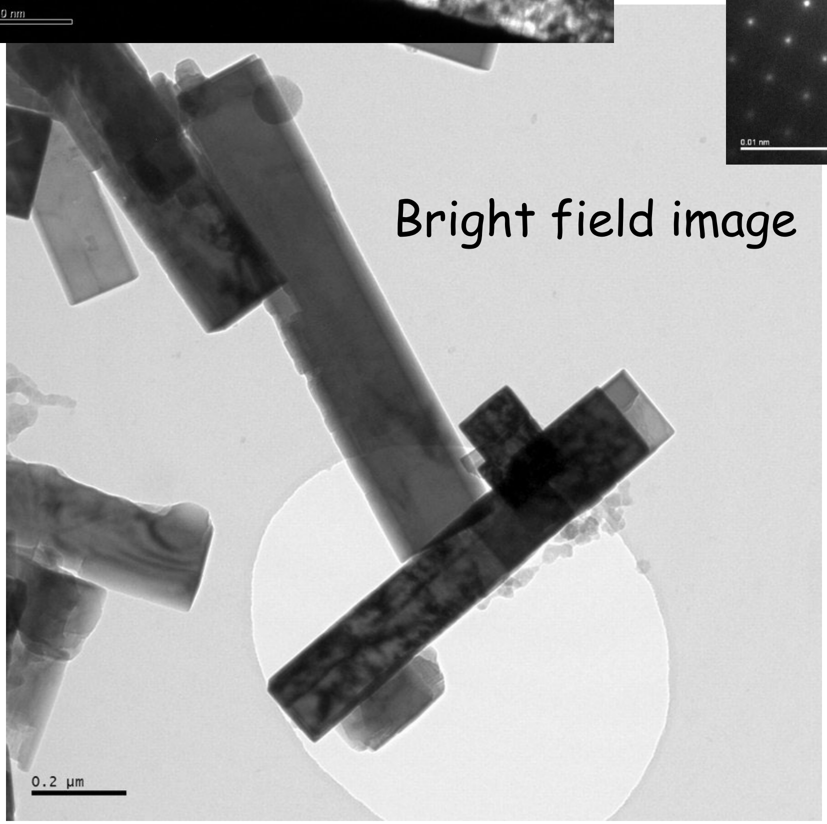
In-situ Cooling/Heating

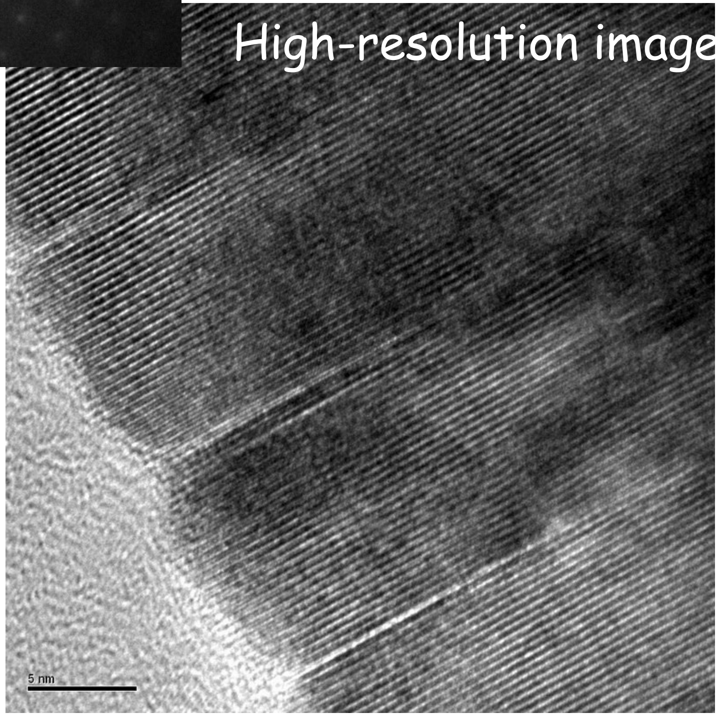
$(K,Nb)O_3$ Nano-rods

Shape, lattice parameters, defects, lattice planes



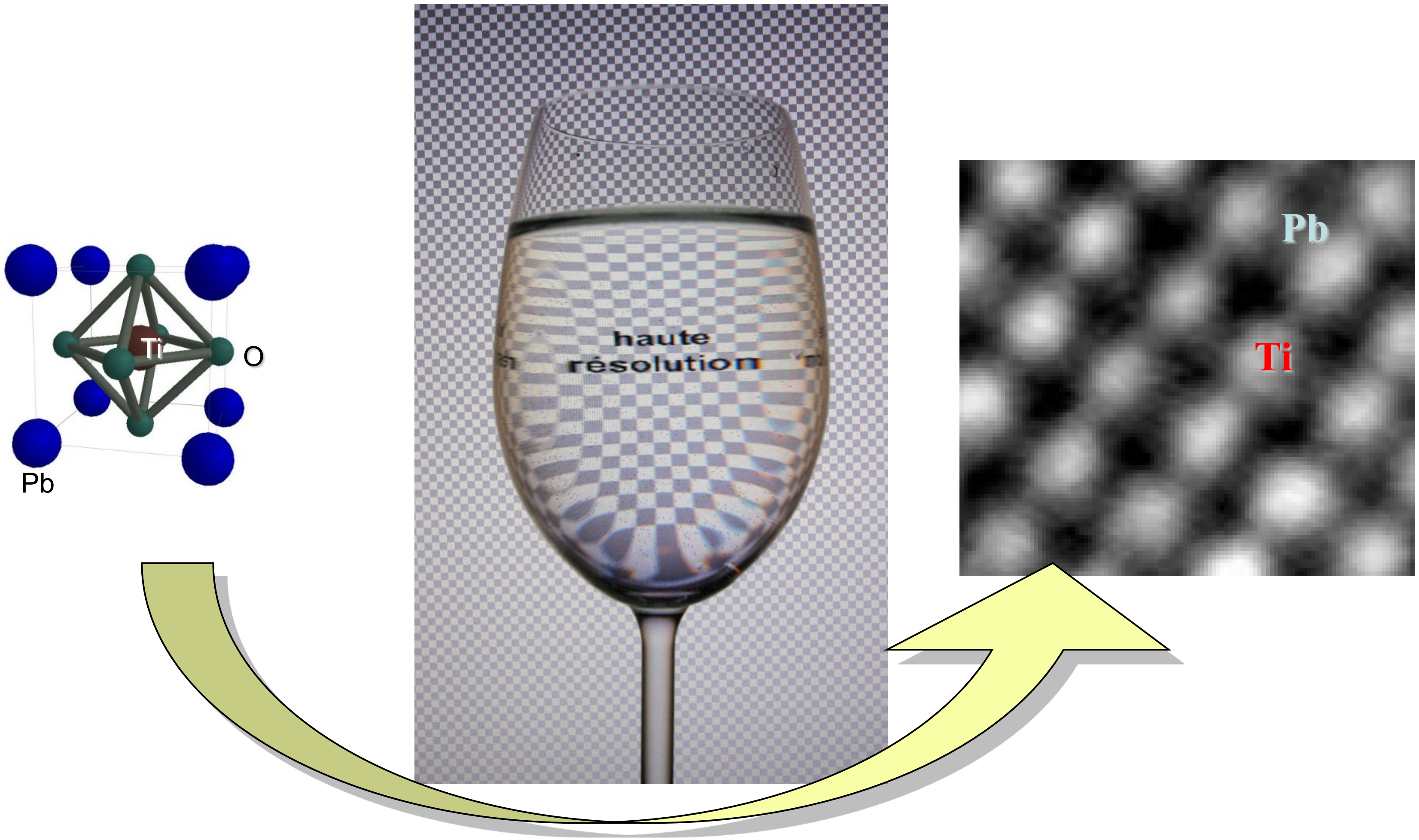






High-resolution TEM

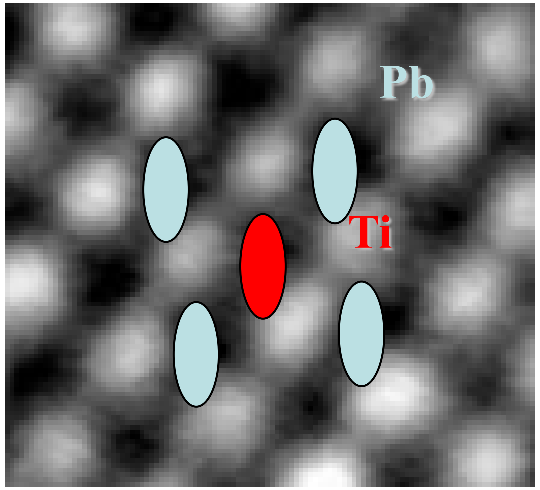
- K. Ishizuka (1980) ***"Contrast Transfer of Crystal Images in TEM"***, Ultramicroscopy **5**,pages 55-65.
- L. Reimer (1993) ***"Transmission Electron Microscopy"***, Springer Verlag, Berlin.
- J.C.H. Spence (1988), ***"Experimental High Resolution Electron Microscopy"***, Oxford University Press, New York.



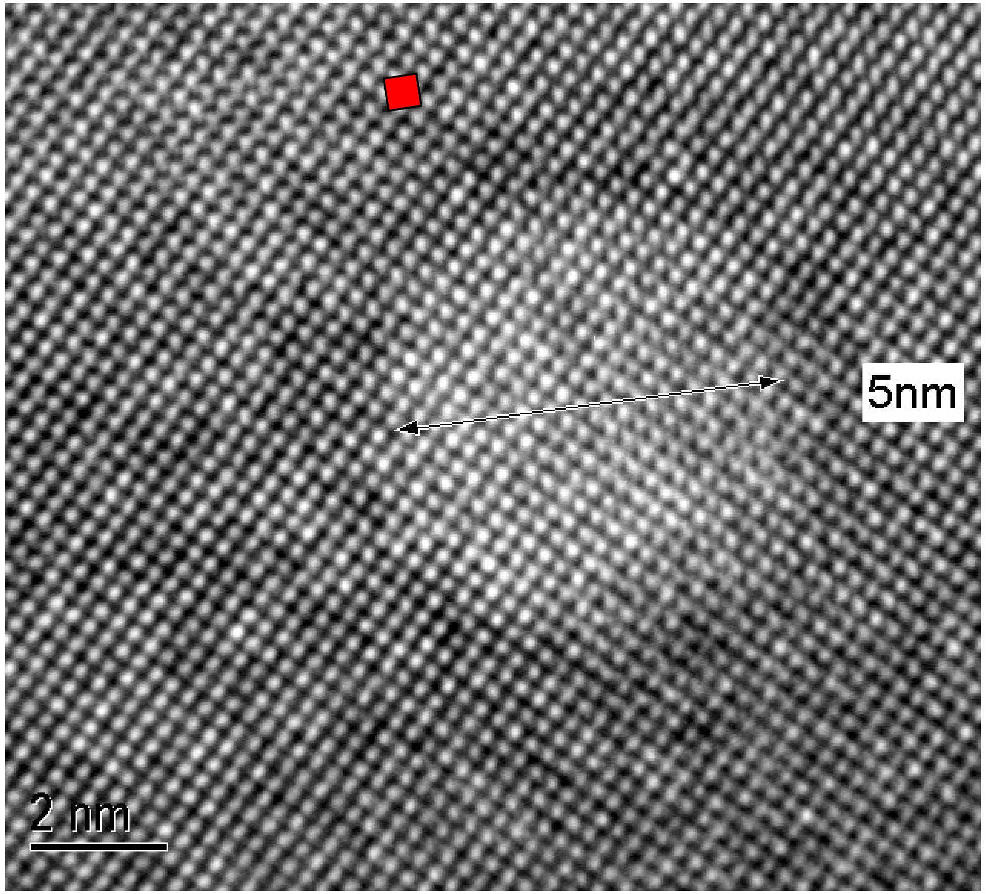
High resolution….?!

electron at 300keV:

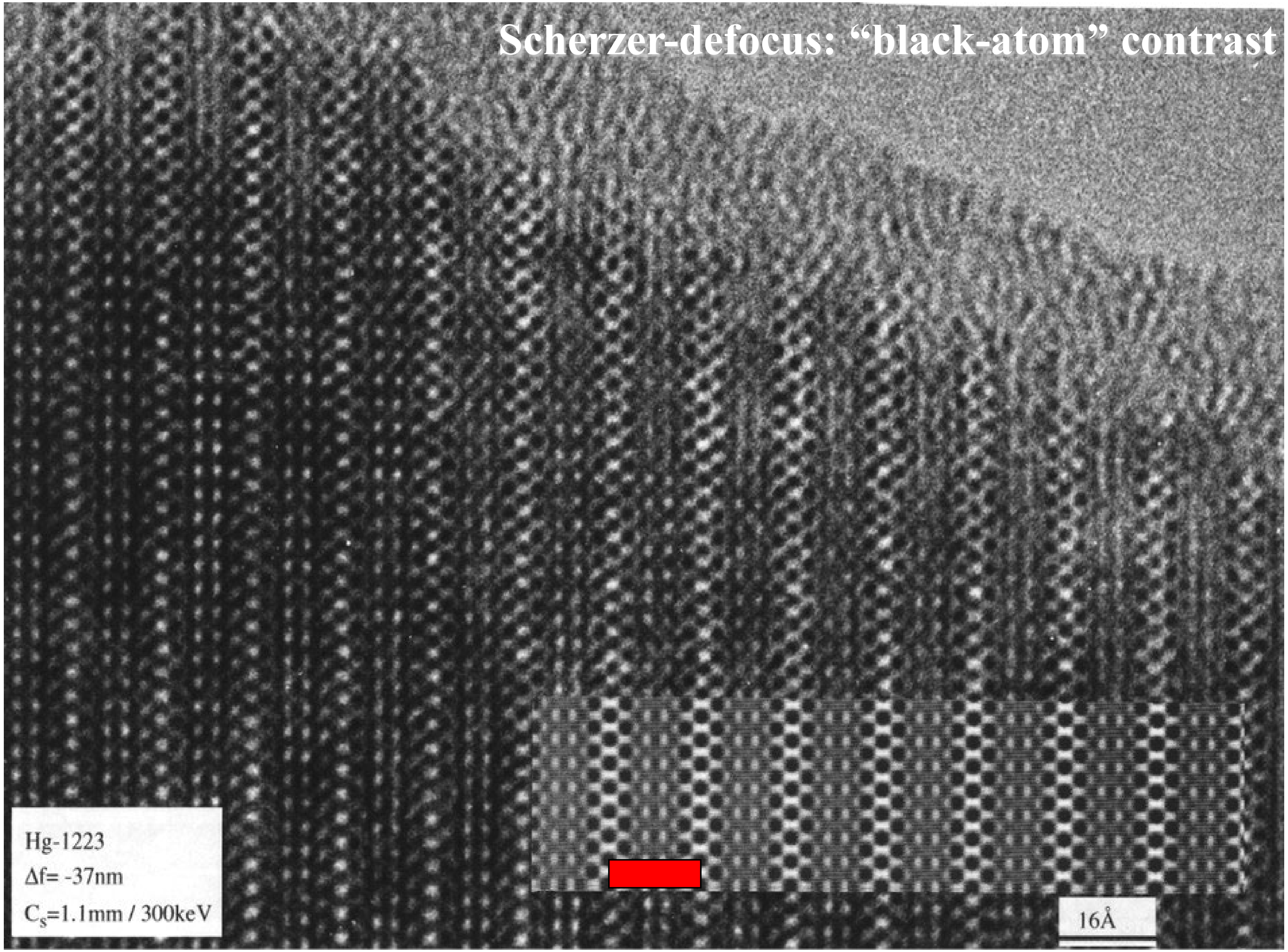
- v = 2.33 10^{-8} m/s (0.78 c)
- λ = 0.00197nm
- θ_{diff} = 10^{-3} rad

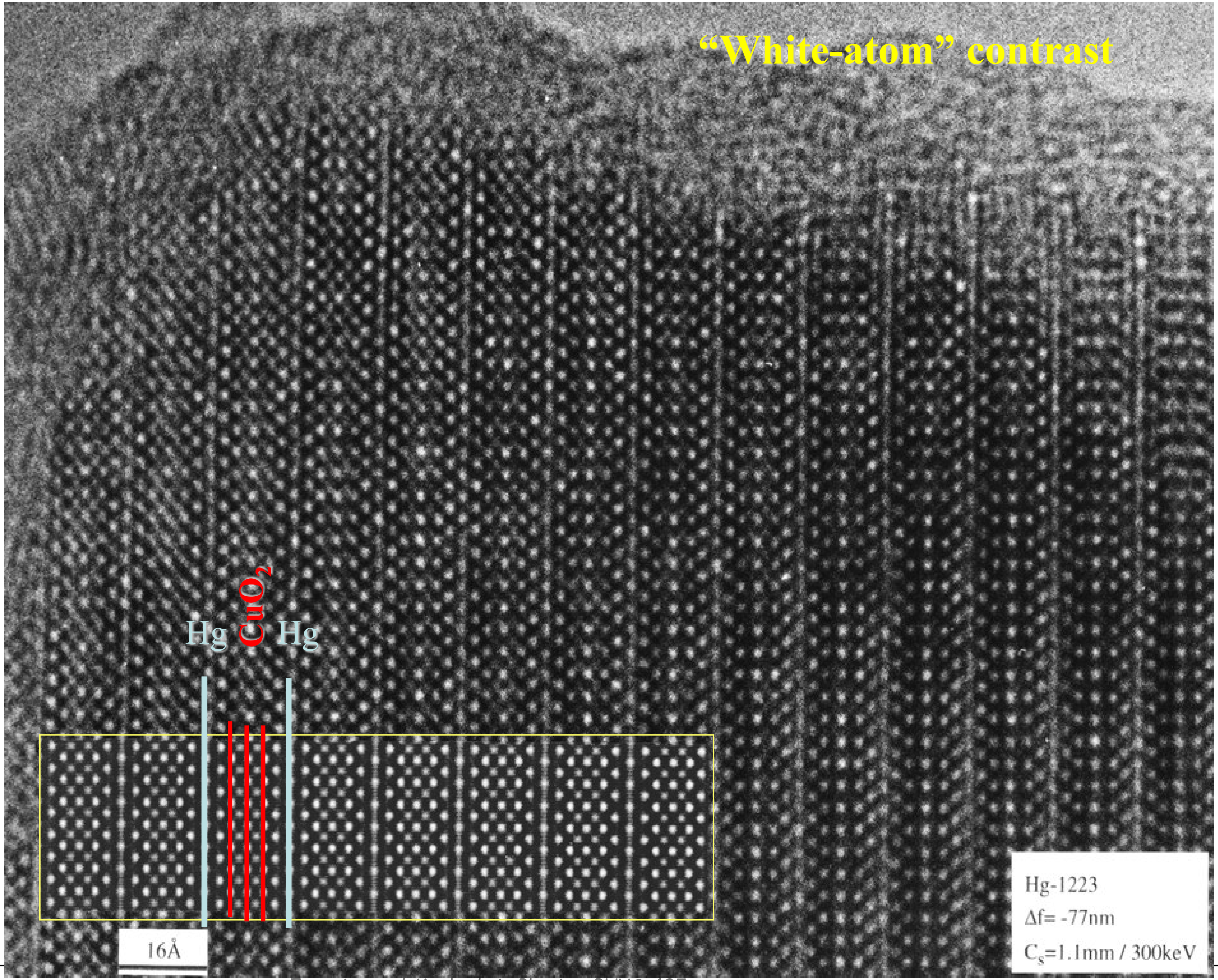


Atomic resolution…?



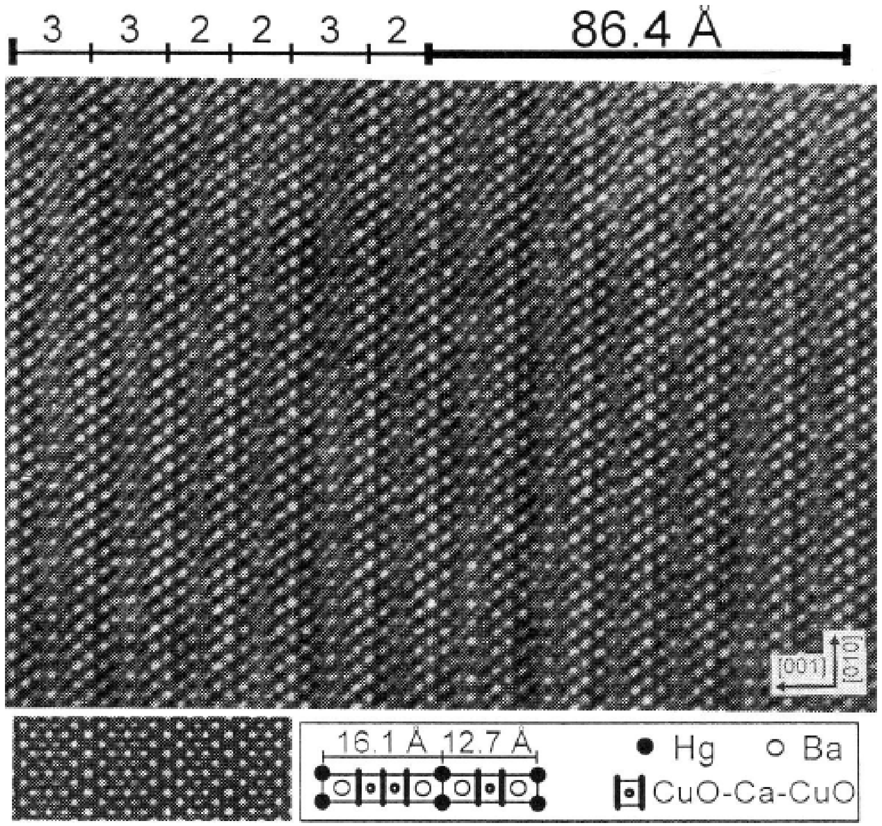
Precipitate in a $PbTiO_3$ ceramic

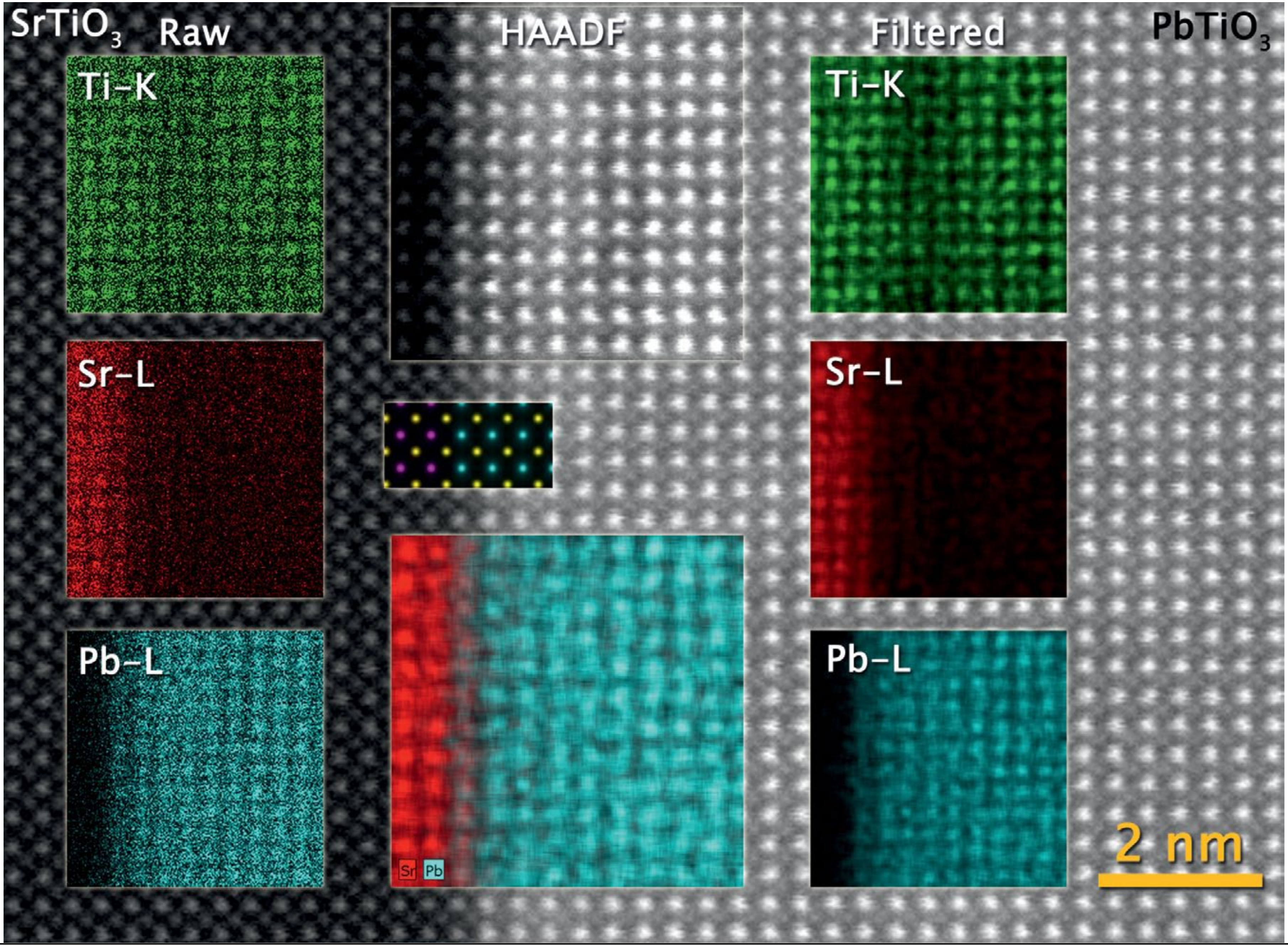

Scherzer-defocus: “black-atom” contrast
Hg-1223
Δf= -37nm
Cs=1.1mm / 300keV
16Å


"White-atom" contrast
CuO_2
Hg
Hg
16Å
Hg-1223
Δf= -77nm
C_s=1.1mm / 300keV

High resolution

- The image should resemble the atomic structure of the sample !
- Atoms…?
  Thin samples: atom columns: sample orientation (beam // atom columns)
- Contrast varies with sample thickness and defocus…!
- Comparison with simulation necessary !




SrTiO3
Raw
HAADF
Filtered
PbTiO3
Ti–K
Sr–L
Pb–L
Ti–K
Sr–L
Pb–L
Sr
Pb
2 nm

Electron microscopy

- A very powerful and versatile tool
- "nice images"
- High resolution
- Different information (signals) from the same sample
- Understanding the image contrast requires understanding of the interaction of electrons with matter and electron optics (especially in TEM).

Bibliography, SEM

SEM

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Bibliography, TEM

TEM

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Multi-techniques

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E. Fuchs, H. Oppolzer, H. Rehme, *Particle Beam Microanalysis*, VCH Verlag 1990)

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M. Amou éd., *Microcaractérisation des Solides*, Ecole CRAM (CRAM 1989, 541 p)

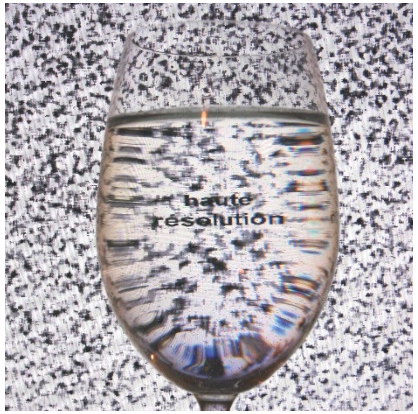
D.K. Bowen, C.R. Hall, *Microscopy of Materials*, McMillan (1975)

Components of an electron microscope

Electron propagation is only possible through vacuum. The vacuum level varies in the different areas of an electron microscope. The highest vacuum level (<10^{-7} Pa or 10^{-9}mBar) is required in the gun where electrons are emitted through field emission. Also the specimen area requires a high vacuum level especially for chemical analysis when the electron beam is resting for a longer time in the same area. Hydrocarbon build up (contamination) on the observed area is often the result of a low system vacuum level. Turbomolecular and oil-diffusion pumps for high vaccum cannot work against atmospheric pressure and need a mechanical prevaccum pump in order to function.

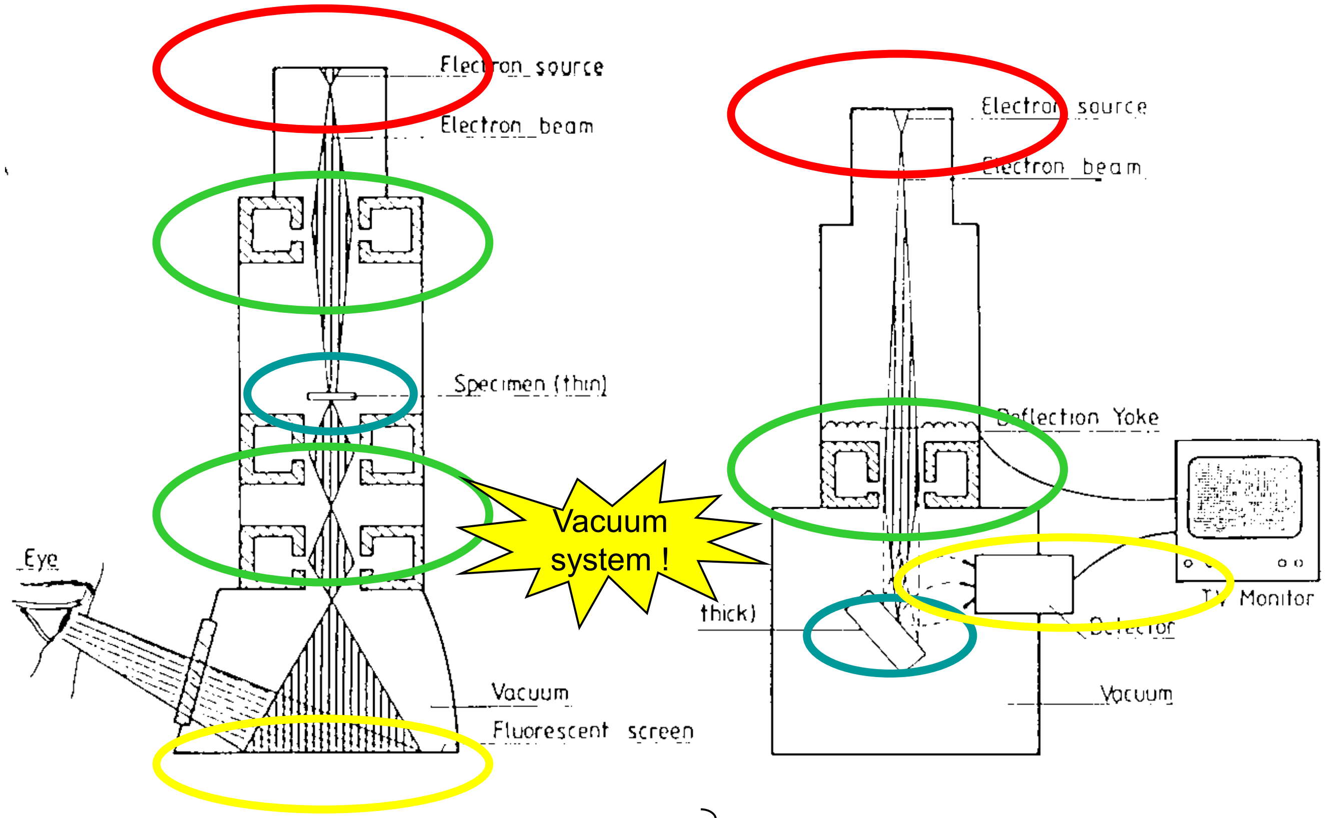
Electron beams can either be generated by thermal emission (thermionic sources, cheap) or field emission. Only field emission sources can provide the necessary low energy spread and coherence for modern high resolution electron microscopy and electron spectroscopy.

Electrons are focused by simple round magnetic lenses which properties resemble the optical properties of a wine glass…. Unlike in light optics the wavelength (2pm for 300kV) is not the resolution limiting factor. However lens aberrations and instabilities of the electronics (lens currents etc.) limit the resolution of even the best and most expensive transmission electron microscopes to about 50pm.



Recording an image means detecting electrons. Depending on their energy electrons can be detected by different detectors. A high detector efficiency and a high signal to noise ratio allows faster recording and reduces the exposure (beam damage) of the sample to the electron beam. A high linearity and high dynamic range permits to quantify images and to record high and low intensities in one image (important for diffraction experiments).

Components of an electron microscope

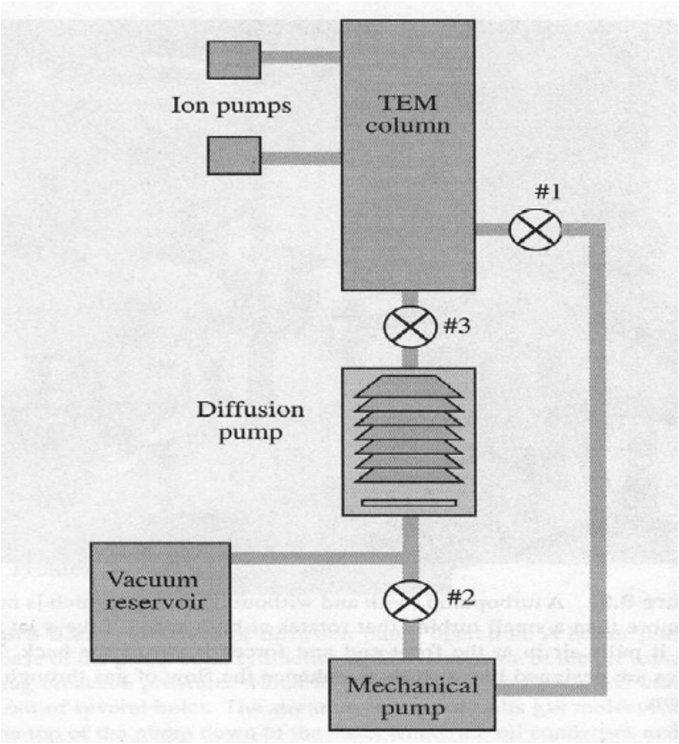


- Source: electron gun
- Lenses and apertures
- Sample holder (stage)
- Detector(s)

common SEM and TEM

Specific for each technique

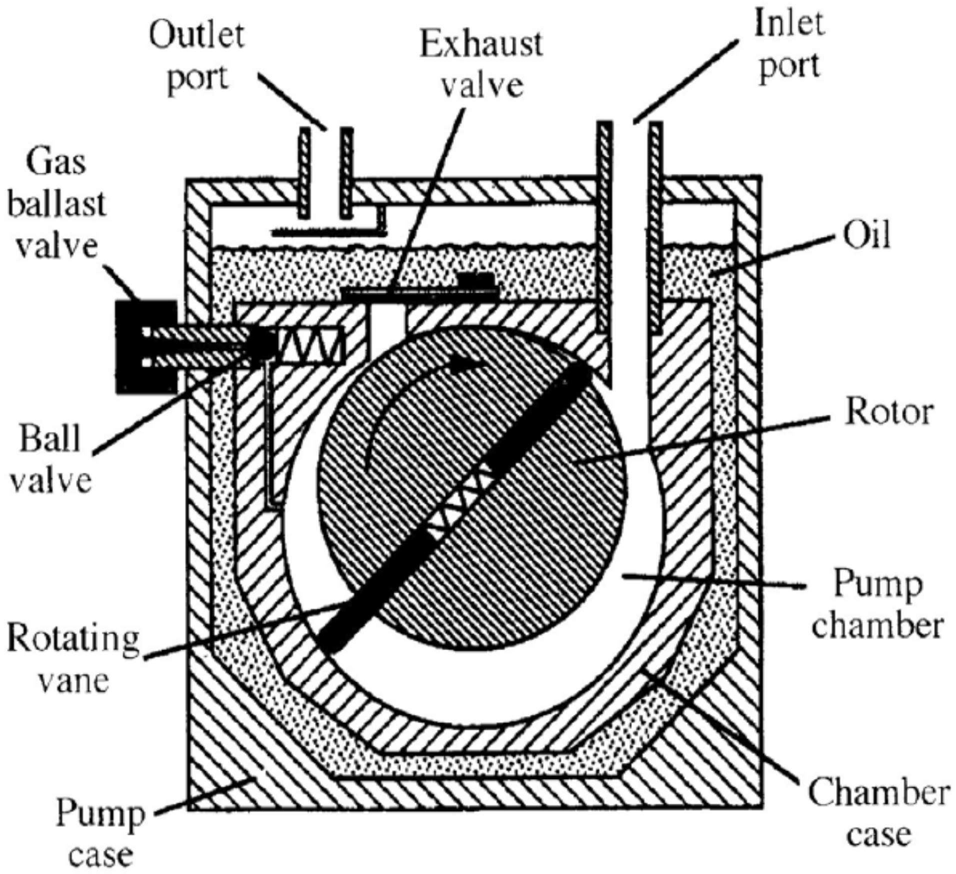
Pumping system



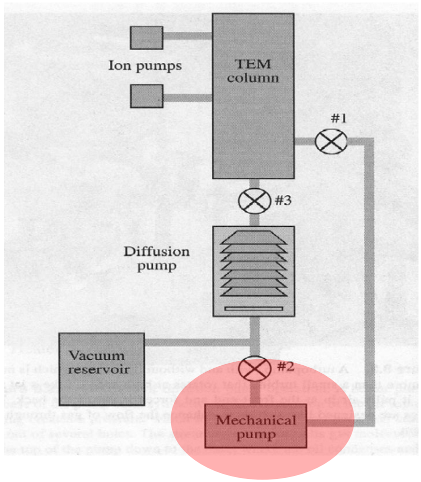
- **Primary vacuum (>0.1 Pa)**
  - Mechanical pump
- **Secondary vacuum (<10^{-4} Pa)**
  - Oil diffusion pump
  - Turbomolecular pump
- **High and ultra-high vacuum**
  Gun & specimen area (<10^{-6} Pa)
  - Ion getter pump
  - Cold trap

Vaccum level in space:
1 Pa at 100km
above earth surface

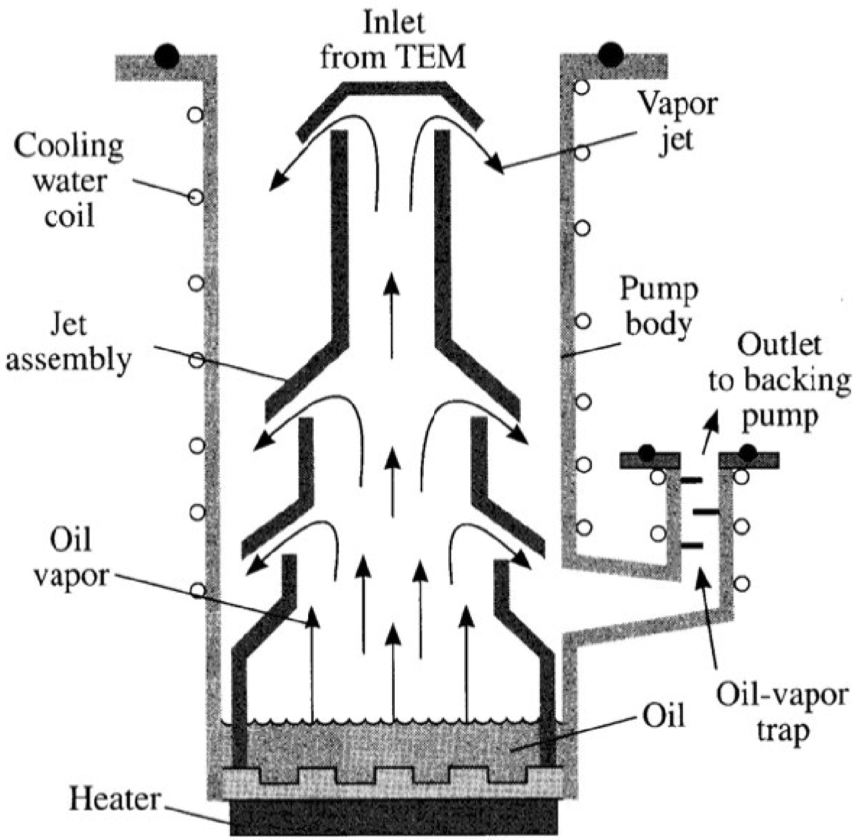
Primary vacuum



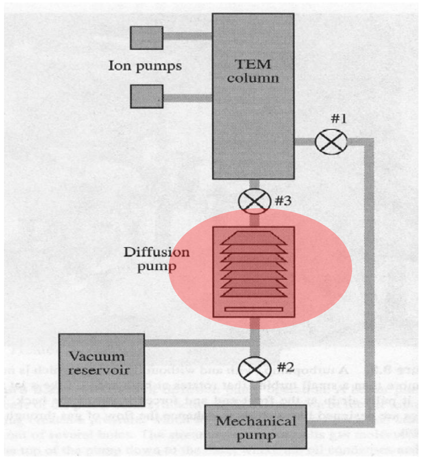
- Rotary vane pump
  - Uses oil
  - noisy



Secondary vacuum

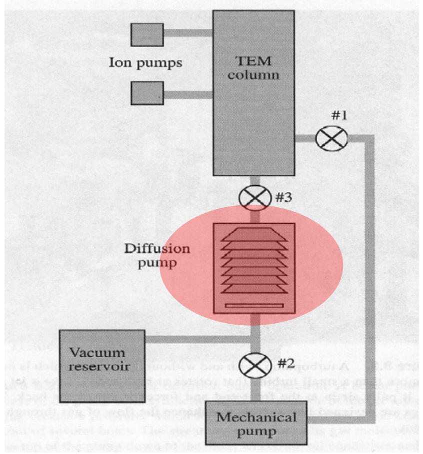


- **Oil diffusion pump**
  - Vibration free
  - Contamination possible oil vapor
  - High pumping capacity (>500 l/s)
  - Best with cold trap

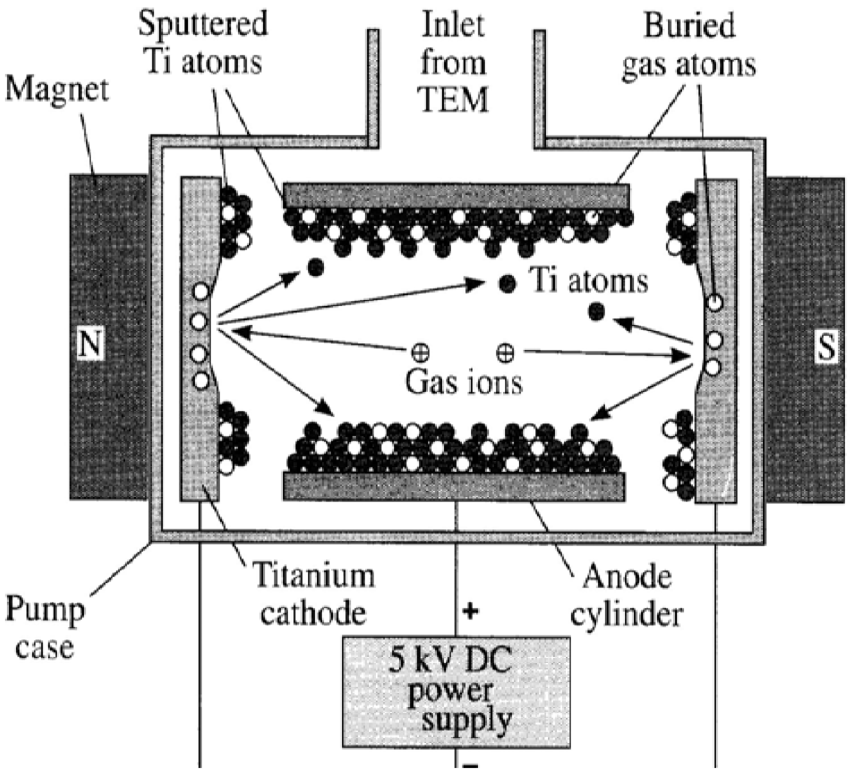


Secondary vacuum

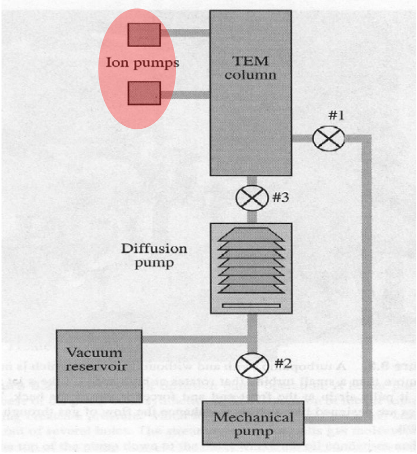
- Turbomolecular pump
  - Rotation speed 20-50'000 rpm
  - Magnetic bearings
  - Pumping volumes 50-500 l/s



High / Ultra-high vacuum



- Ion getter pump
  - no vibrations
  - No exit: improves vacuum !

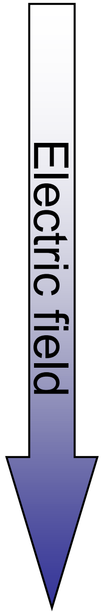


SOURCES (gun)

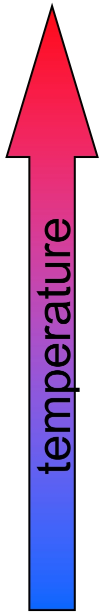
LaB_6 Cathode

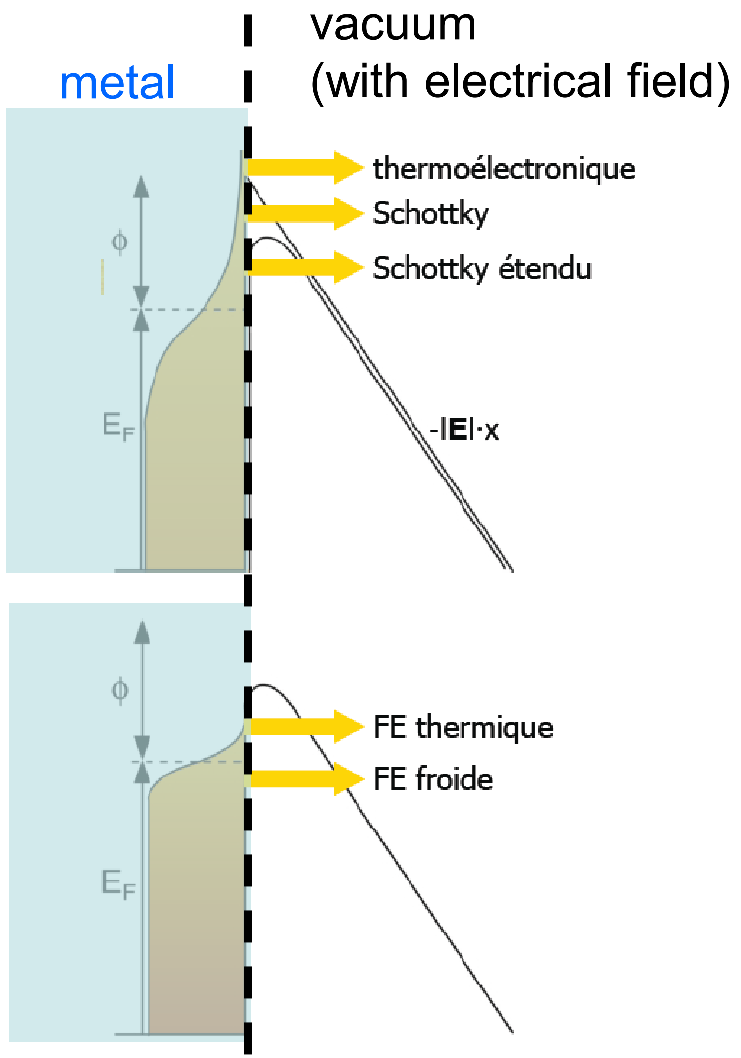
http://www.feibeamtech.com

Emission of electrons

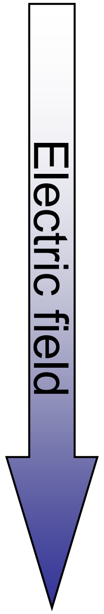


- Thermionic emission
- Shottky emission
  field-enhanced
  thermionic emission
  (10^8V/m)
- Extended Shottky
  emission
  thermally assisted
  field emission
- Cold field emission
  tunnel effect
  (quantum tunnelling)





Emission of electrons



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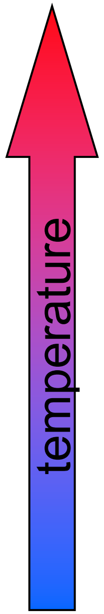
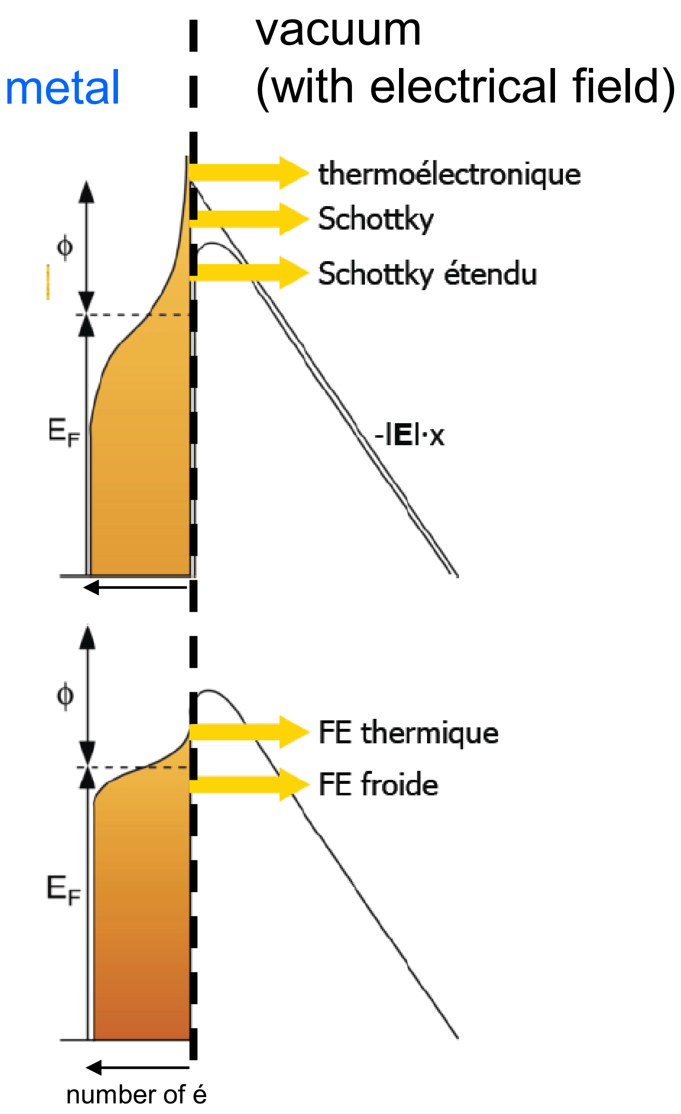
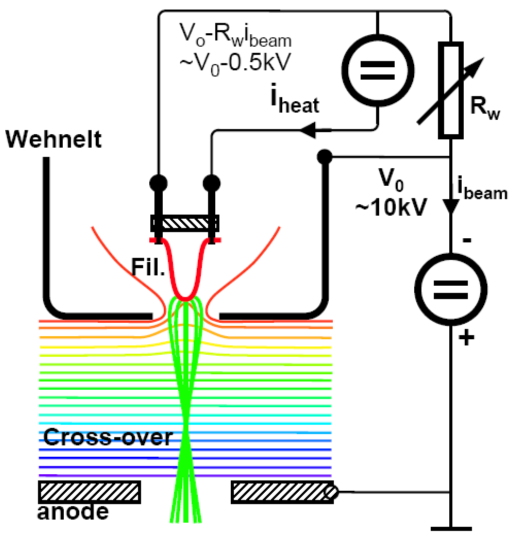


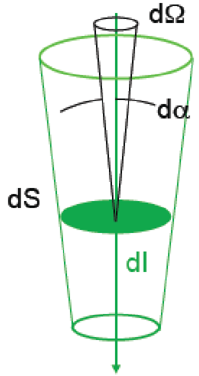


Electron gun

Important parameters

- Emitted current, energy
- Energy dispersion
- Brightness
  current per surface unit and solid angle
- Coupling to the column
- the gun incorporates often a first lens (Wehnelt, gun lens)

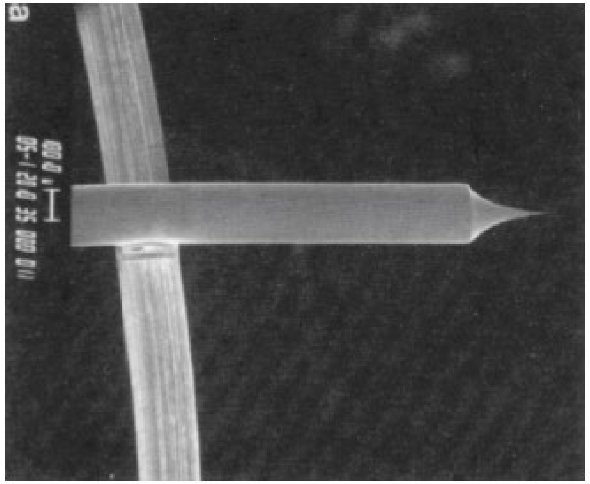


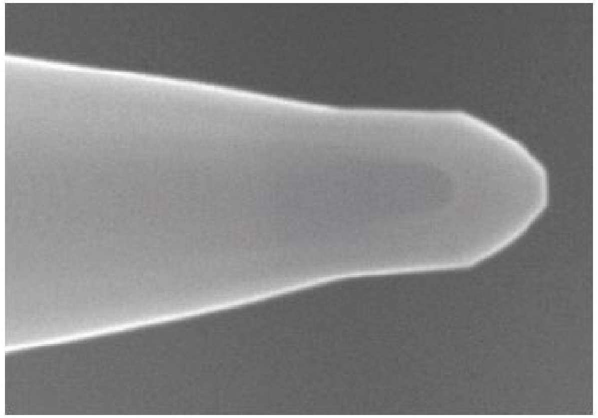


Field emission guns

Cathods

- Cold field emission ($E \approx 10^9$V/m)
  W monocristal with sharp tip
  tip radius ~100nm
- Thermally assisted emission:
  Shottky effect
  W/Zr tip at 1700-1800K





- Advantages
  Small energy dispersion (<0.4eV)
  high coherence, high brightness
  -> higher resolution at lower energies
- Disadvantages
  expensive
  high vacuum necessary
  cold emission needs flushing (cleaning)
  after 8 hrs

Field emission guns

First anode (extractor)

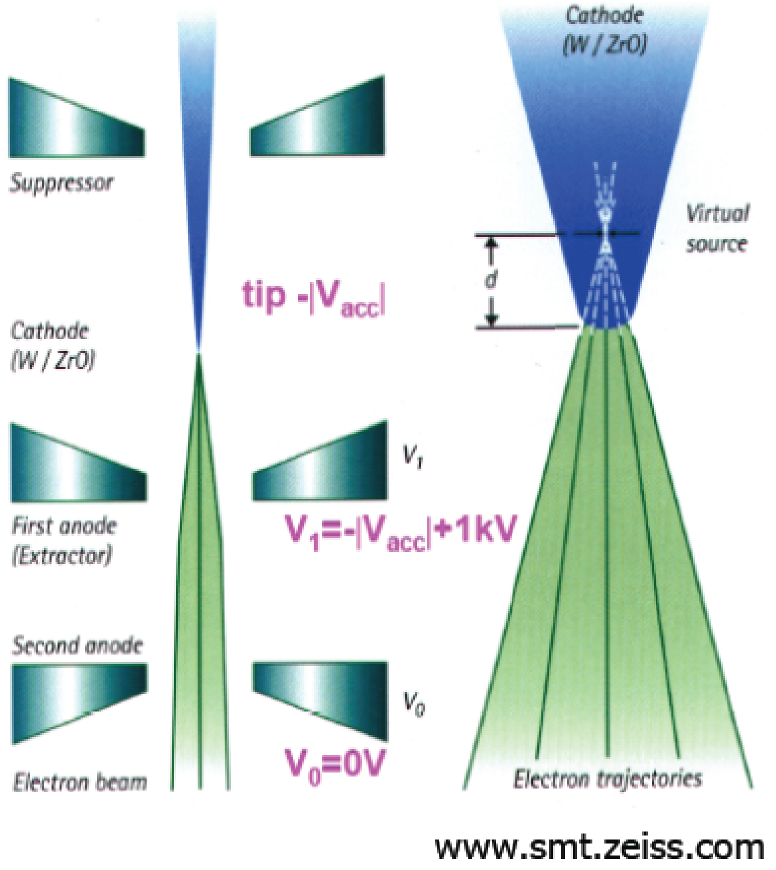
- Some kV
- 5.10^9 V/m

Second anode

- Final acceleration
- Grounded

Characteristics

- Tip and anodes form an electrostatic condensor
- Cross-over (source) is virtual
  Ø~5nm



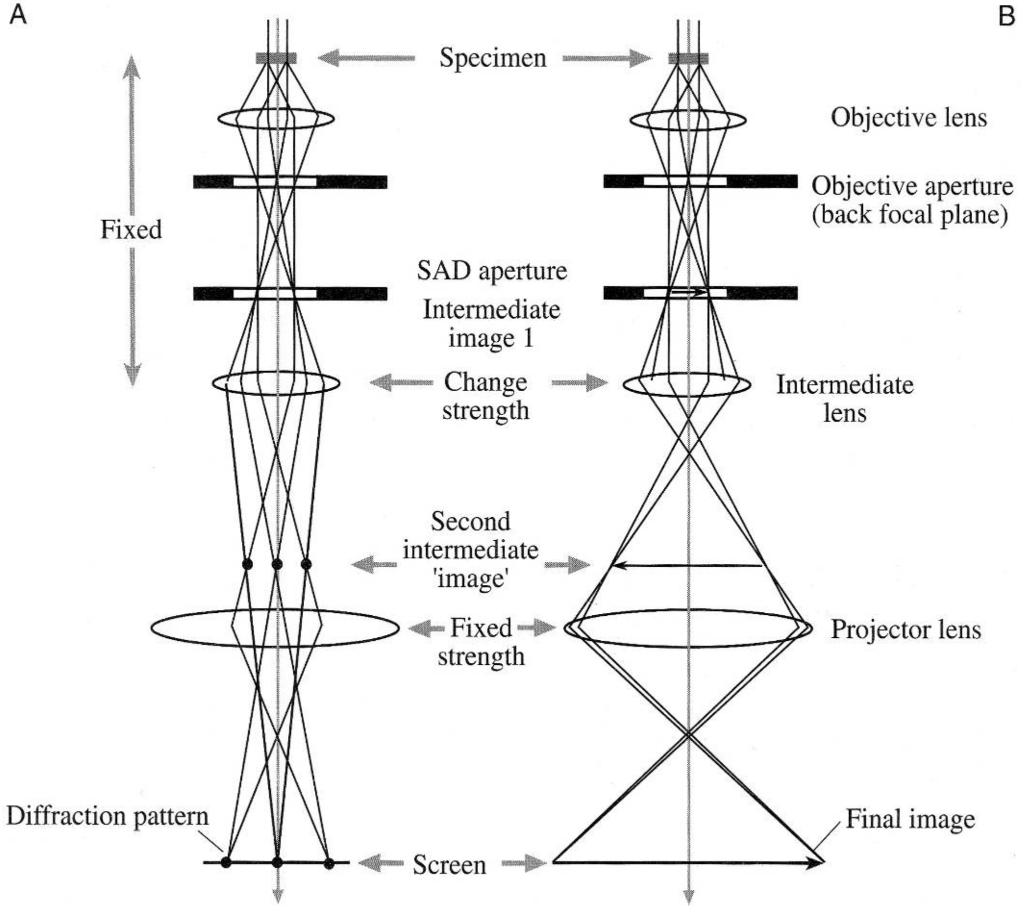
Some data for electron guns (≈20kV)

Gun type	Thermoionic		Field emission	
Emitter	W filament	LaB6 cathode	W cold cathode	Schottky ZrO/W cathode
Temperature (K)	2'800	1'900	300	1'800
Cathode diameter (μm)	100	20	<0.1	<1
Source diameter	30 (μm)	10 (μm)	5 (nm)*	30 (nm)*
Current density at emitter(A/cm^2)	3	30	2 10^4	5 10^3
Emitted current (total) (μA)	200	80	5	200
Brightness β @20 kV(A/cm^{2}sr)	1 10^4	1 10^5	1 10^8	1 10^7
Max. beam current (nA)	1000	1000	0.5	30
Energy spread at emitter (eV)	0.6	0.4	0.3	0.3
Energy spread at the gun exit (eV)	1.5-2.5	1.3-2.5	0.3-0.7	0.4-0.7
Probe current noise (%)	1	1	5-10	1
Probe current drift (%/h)	<1	<1	>5	<1
Operating gun pressure (mbar)	< 1.10^{-5}	< 5.10^{-6}	< 1.10^{-10}	<1.10^{-9}
Emitter lifetime (h)	10 - 100	1000	2000	2000
cathode conditioning	not necessary	not necessary	flash≈6 h	not necessary
gun annealing	not necessary	not necessary	≈ 6 months.	cathode exchange
sensitivity to external magnetic field and vibrations	faible	faible	fort	faible

(adapted from LEO 1500/1997)

* virtual cross-over

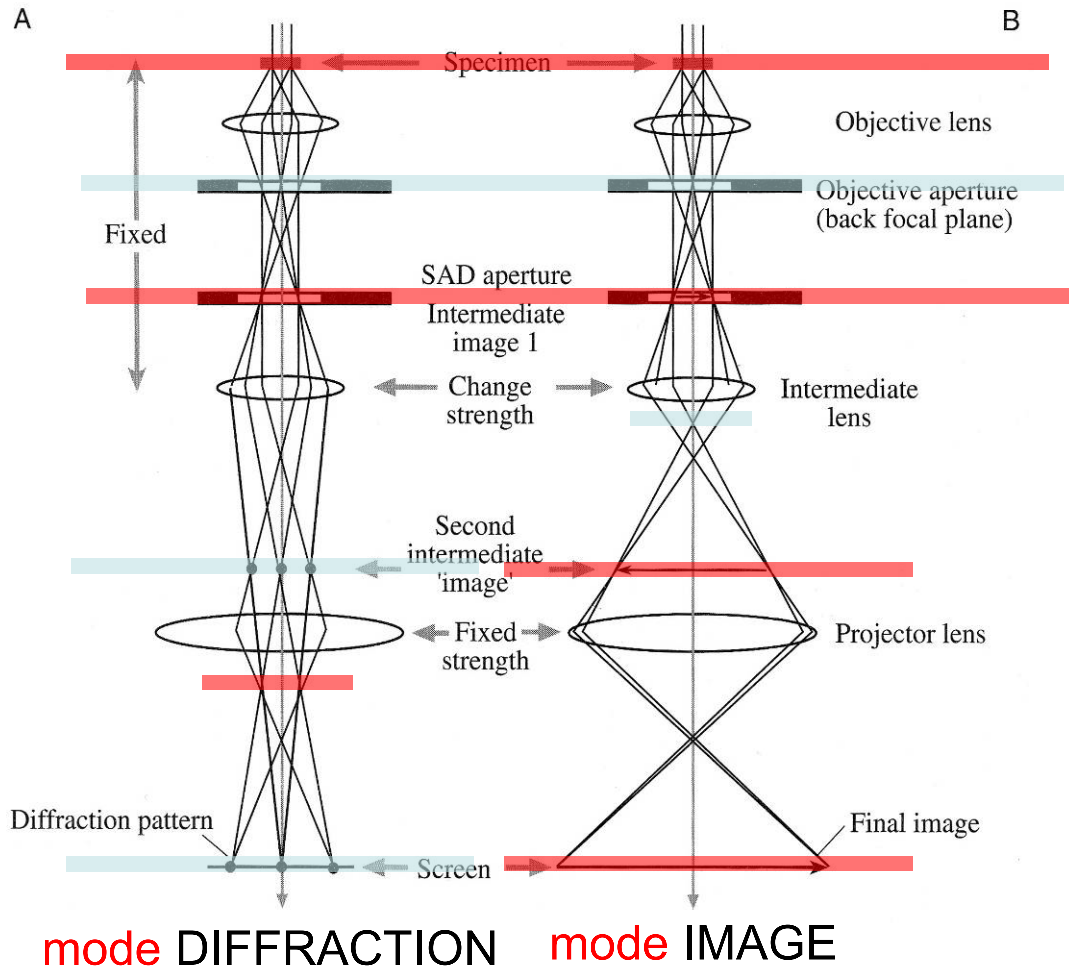
Projector lens system, TEM



mode DIFFRACTION mode IMAGE

- TEM:
- Intermediate and projector lenses
  - Projection of the back focal plane to the screen **"diffraction" mode**
  - Projection of the image plane to the screen **"image" mode** (haute resolution)

Projector lens system, TEM



- TEM:
- Intermediate and projector lenses
  - Projection of the back focal plane to the screen **"diffraction" mode**
  - Projection of the image plane to the screen **"image" mode** (haute resolution)

Lenses for electrons

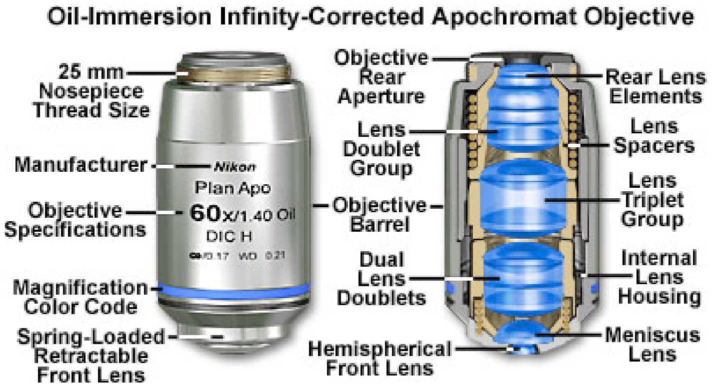
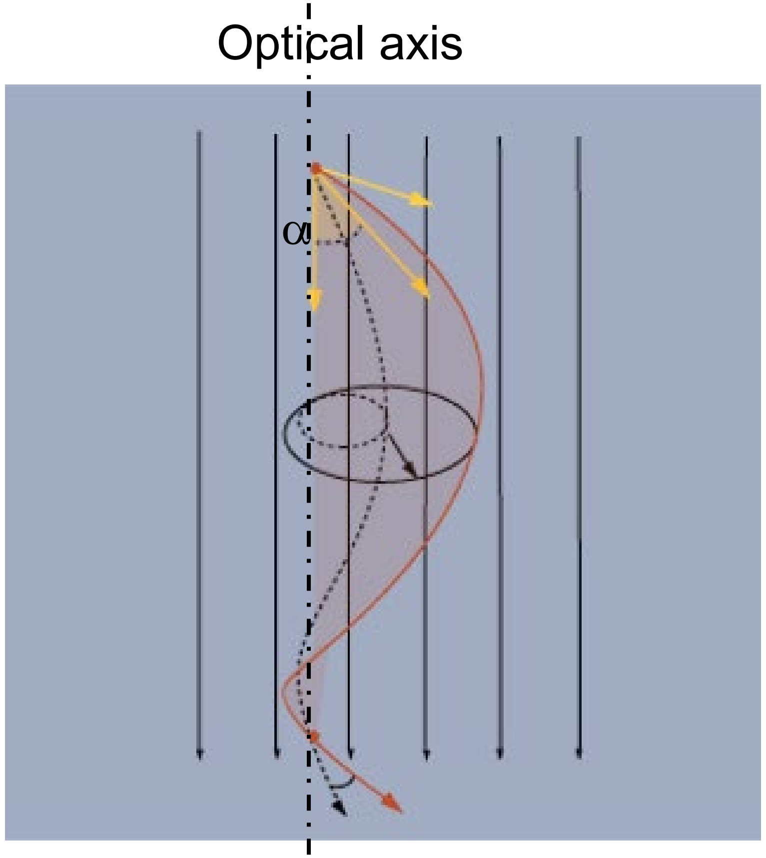


Figure 1

- Light: glass lenses
  deflection of light through changing refraction index
- Charged particles
  Lorentz Force!
  Electrostatic lenses
  Magnetic lenses
- Particularity:
  Variable focus
  Tunable correctors (astigmatisme)

Electrons in a magnetic field



www.x-raymicroanalysis.com

- Homogeneus field, α small
- Component of **v** // **B** almost unchanged
- Component of **v** ⊥ **B**: v_r << |**v**|
- Spiral with radius r = m vr/eB
- All electrons crossing the axis in one point are focused into the same point, α, v_r
- Focal length depends on B increasing B lowers f

Magnetic lens

- Field with rotational symmetry
- Lorenz Force : **F** = -e **v** ^ **B**
  e on optical axis: **F** = 0
  e not on optical axis : deviated
  optical axis: symmetry axis

- Scherzer 1936:
- Magnetic lens with rotational symmetry:

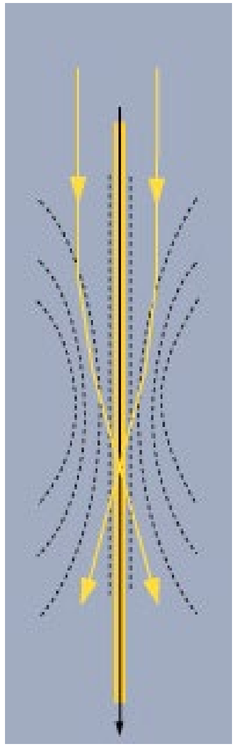
  Aberration coefficients:

  C_s: spherical

$$C_s = \frac{1}{16}\int_{z_o}^{z_i}\left\{b^4h^4 + 2\left(hb' + h'b\right)^2 h^2 + 2b^2h^2h'^2\right\}dz$$

$$C_c = \frac{1}{4}\int_{z_o}^{z_i} b^2h^2\,dz$$

Resolution limit: $D_{res} = 0.66\lambda^{3/4}C_s^{\ 1/4}$

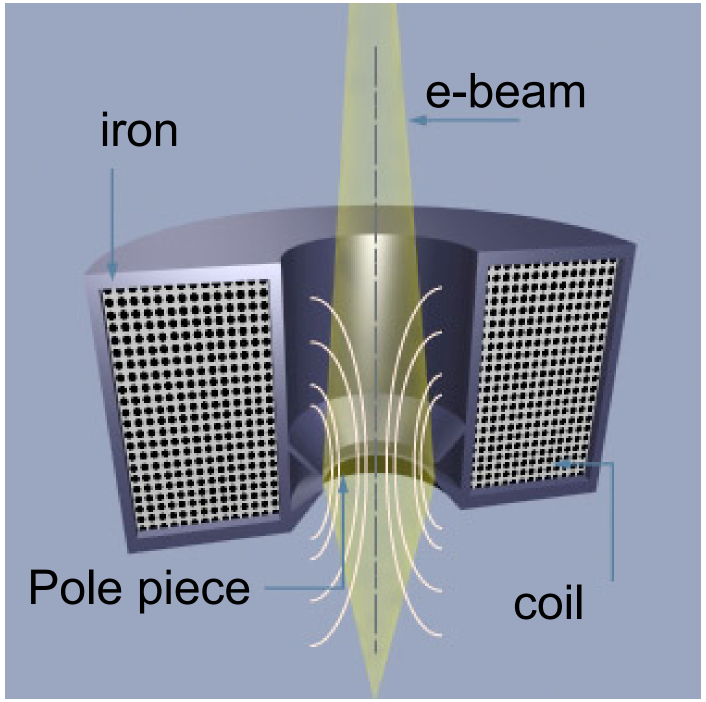


Example:

λ= 0.00197nm, C_s = 1 mm

D_{res} = 1.8 10^{-10} = 1.8Å

Magnetic lens

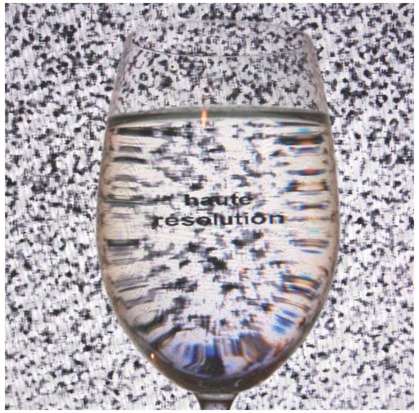


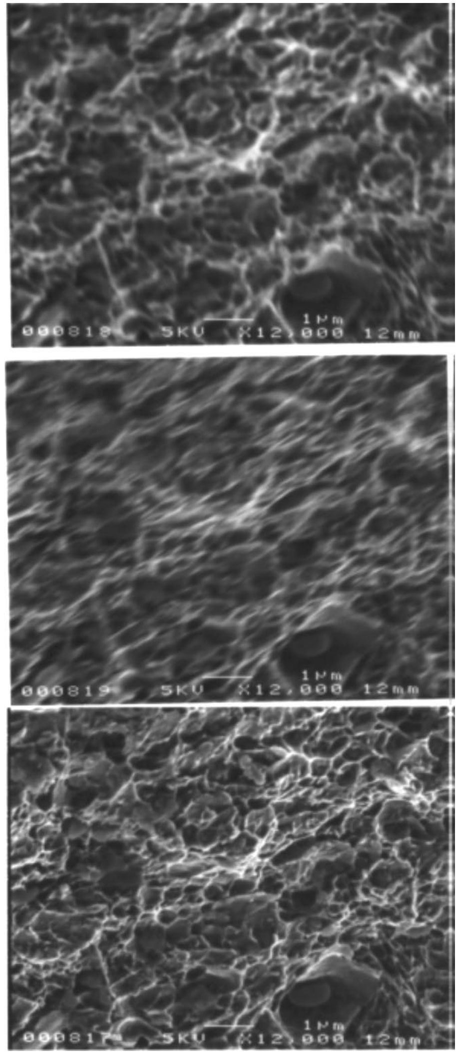
www.x-raymicroanalysis.com

- Electron optics: no sharp interface at lens « surface »
- No divergent lens !
- Electron beam diverges by itself
  - Electrostatic repulsion
- "multi-poles" lenses
  - Correction of aberrations
- "Pole piece"
  metal cone that confines the magnetic field
- Image rotation !

Aberrations:

- Lens aberrations
  - sperical and chromatical aberrations
  - Astigmatism
  - Can be corrected or minimised
- Physical limits
  - Diffraction effect

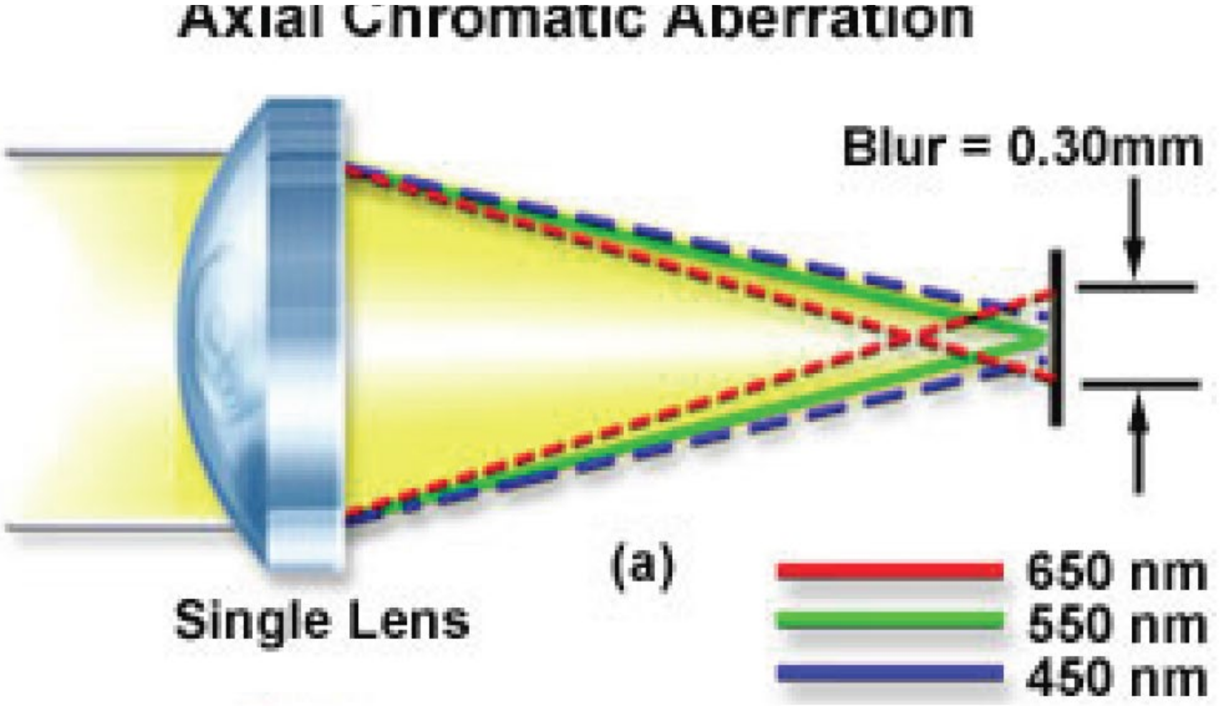




Clichés: P.-A. Buffat

chromatical aberration

Focal length varies with energy
critical for non-monochromatic beams (advantage for FE guns)

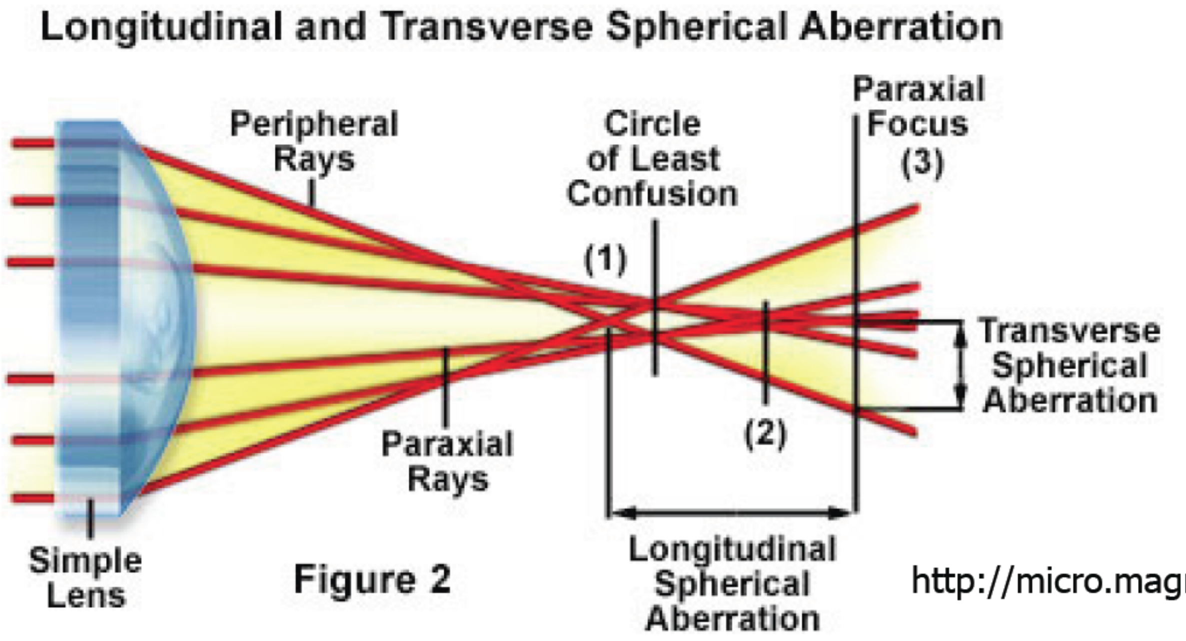


Spherical aberration

Focal length depends on the distance from optical axis

Image of the object is dispersed along the optical axis

Circle of least confusion $d_s = \frac{1}{2} C_s \lambda^3$



http://micro.magnet.fsu.edu/

Aberrations: astigmatism

Astigmatism: focal length varies in different planes.

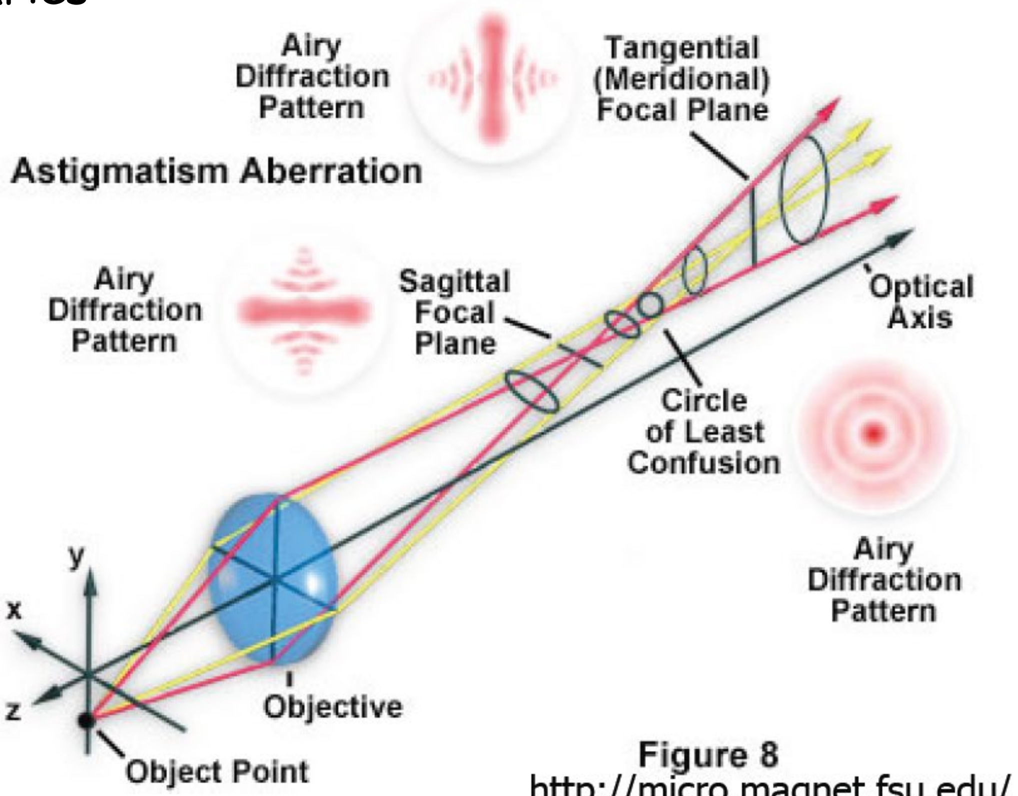


Figure 8
http://micro.magnet.fsu.edu/

correctors

Astigmatism:

Light optics: correction with cylindrical lenses

Electron optics:

Correction with quadrupole lenses:
2 quadrupole lenses under 45 degree allow to control strenght and direction of correction

Spherical Aberration:

Light optics: correction with combination of convergent and divergent lenses

Electron optics:

Correction with hexapole or quadrupole and octopole lenses

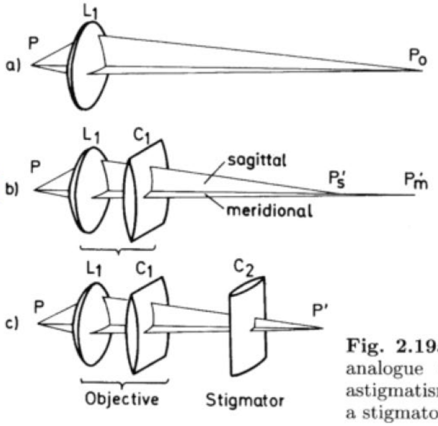


Fig. 2.19a–c. Light-optical analogue of the action of astigmatism (L1 + C1) and a stigmator C2 [2.30]

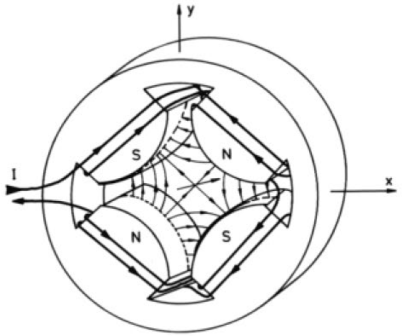
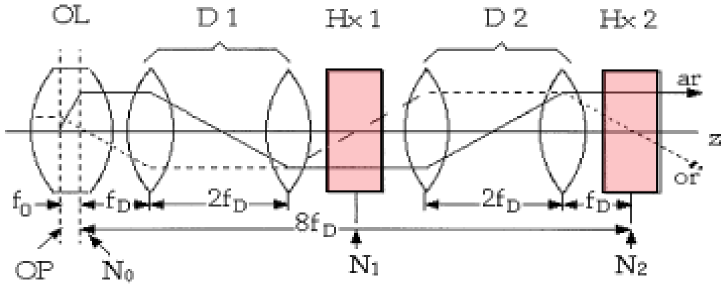
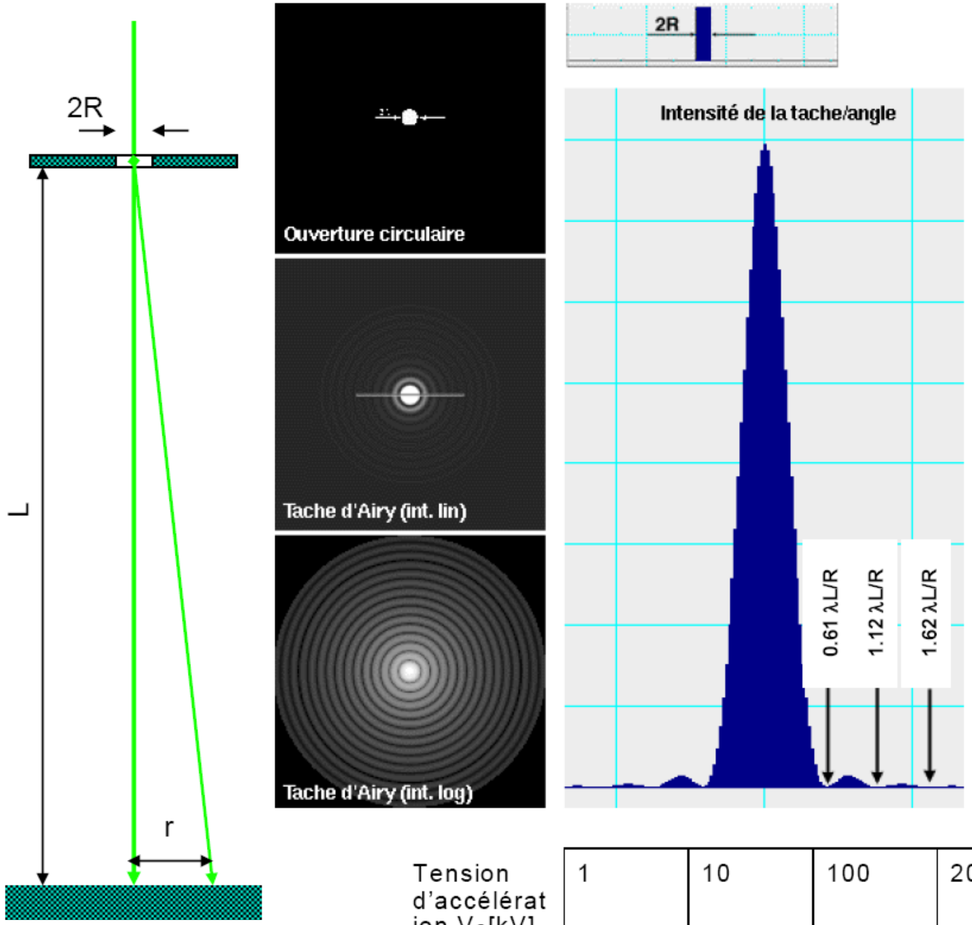


Fig. 2.12. Construction of a quadrupole lens



- Cs-corrector

Aberrations: diffraction



Diffraction, tache d'Airy

Une onde cohérente tombe sur une ouverture circulaire pratiquée dans un écran opaque. La tache dite d'Airy produite sur un écran situé à une grande distance L (par rapport à la longueur d'onde λ et au diamètre 2R de l'ouverture) s'étend autour de l'axe à une distance radiale r avec une intensité donnée par $I(x) \approx [J_1(x)/x]^2$ où $J_1(x)$ est le fonction de Bessel de premier ordre et $x=2\pi Rr/\lambda L$

La largeur à mi-hauteur de la tache centrale est

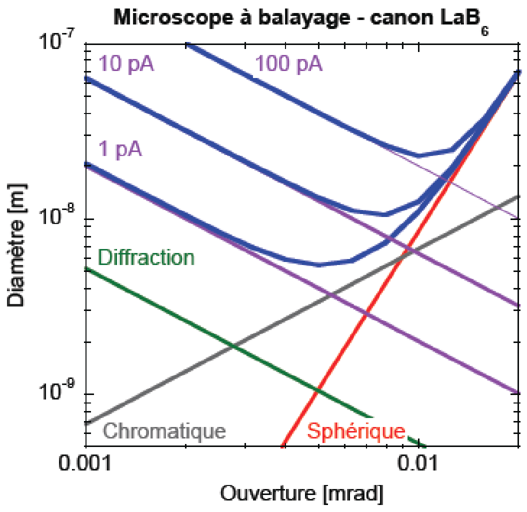
$d_d=0.61\ \lambda/\alpha \approx 1/\alpha$ $\alpha \cong R/L$

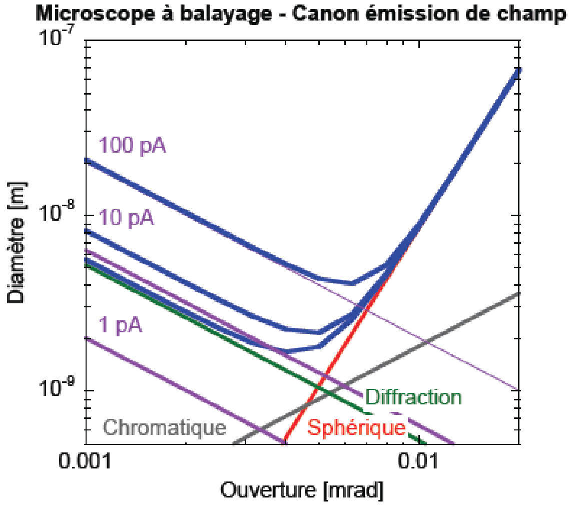
Tension d'accélération V_0[kV]	1	10	100	200	300	400	1'000	3'500
longueur d'onde λ [pm]	38.8	12.2	3.70	2.51	1.97	1.64	0.87	0.31

Résolution SEM

- Limite SEM modèrne

  - Conservation de la brillance
    - $d_g = (1/\alpha)\cdot(4I/\pi^2\beta)^{0.5}$
  - Diamètre minimal de la sonde
    - "Résolution"
    - $D = (d_s^2 + d_c^2 + d_g^2 + d_d^2)^{0.5}$ →
    - Astigmatisme parfaitement corrigé
  - Résolution
    - Canon thermoélectronique: faible brillance limite la résolution
    - Canon à émission de champ: abbérations du canon et des lentilles





$C_s = 17$ mm; $C_c = 9$ mm