An introduction to Transmission Electron Microscopy

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Outline

- Optics
- Contrast
- Spectroscopy

Optics:

- Why electrons
- Electron sources
- Electron Lenses
- Aberration correctors

Abbe' s theory of image formation

- **Spatial resolution of whichever instrument using lenses for obtaining a magnified image of an object is limited by diffraction effects to a minimum value essentially determined by the wavelength.**

$R=0.61\lambda/n \sin\alpha$ (E.Abbe 1873)

- **Visible light has wavelength in the fraction of μm range, while interatomic distances are about three order of magnitude smaller.**

Ernst Abbe
(1840-1905)

Electron can behave as waves and their wavelength depends on the kinetic energy according to the De Broglie equation.

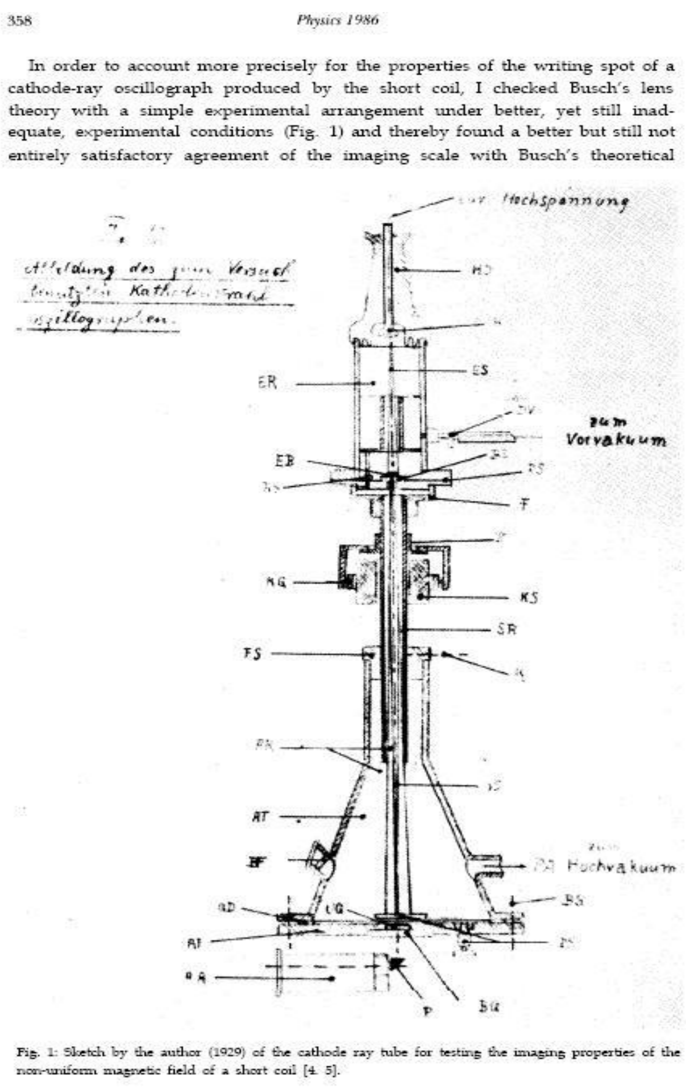
$\lambda=1.22/E^{1/2}$

(De Broglie 1926)

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

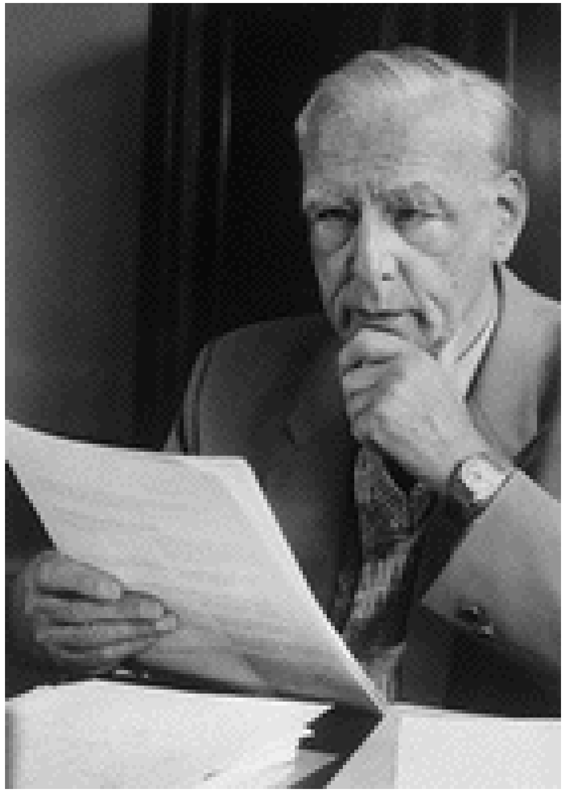
The electron microscope


358 *Physics 1986*

In order to account more precisely for the properties of the writing spot of a cathode-ray oscillograph produced by the short coil, I checked Busch's lens theory with a simple experimental arrangement under better, yet still inadequate, experimental conditions (Fig. 1) and thereby found a better but still not entirely satisfactory agreement of the imaging scale with Busch's theoretical

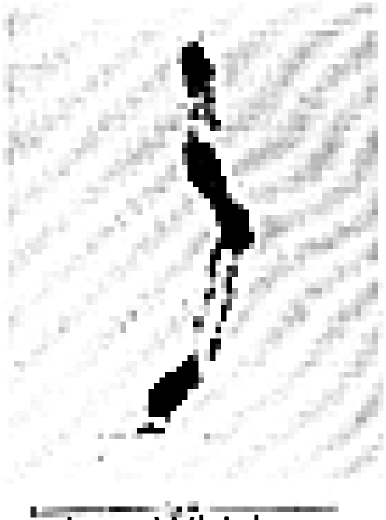
Fig. 1: Sketch by the author (1929) of the cathode ray tube for testing the imaging properties of the non-uniform magnetic field of a short coil [4, 5].

From Nobel lecture by E. Ruska.



Ernst Ruska, 1908-1988
Nobel laureate 1986

Diatoms "Amphipleura pellucida". U=53 kV, Mel =3500



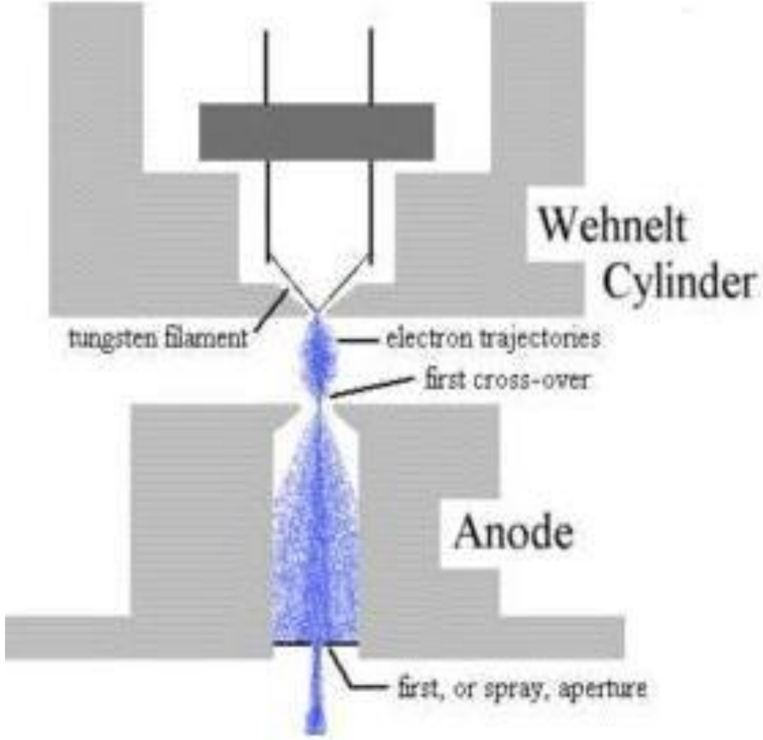
Iron Whisker
(U=79 kV, Mel =3100)

ELECTRON SOURCES

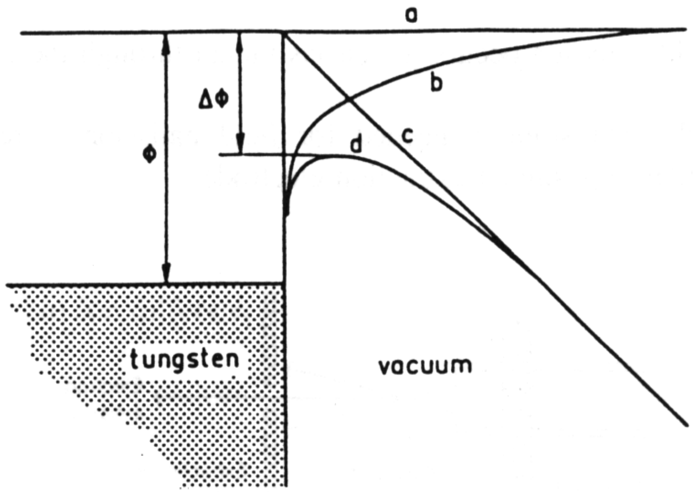
Thermoionic electron gun

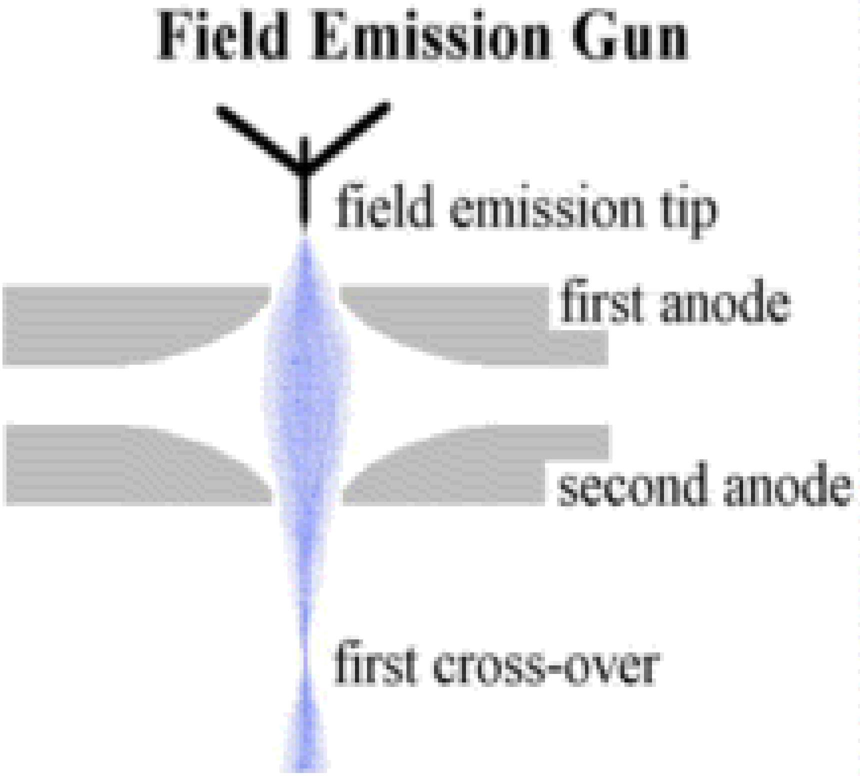
$$J = AT^2 e^{-\Phi/KT}$$

$$\beta = JeV/KT\pi$$

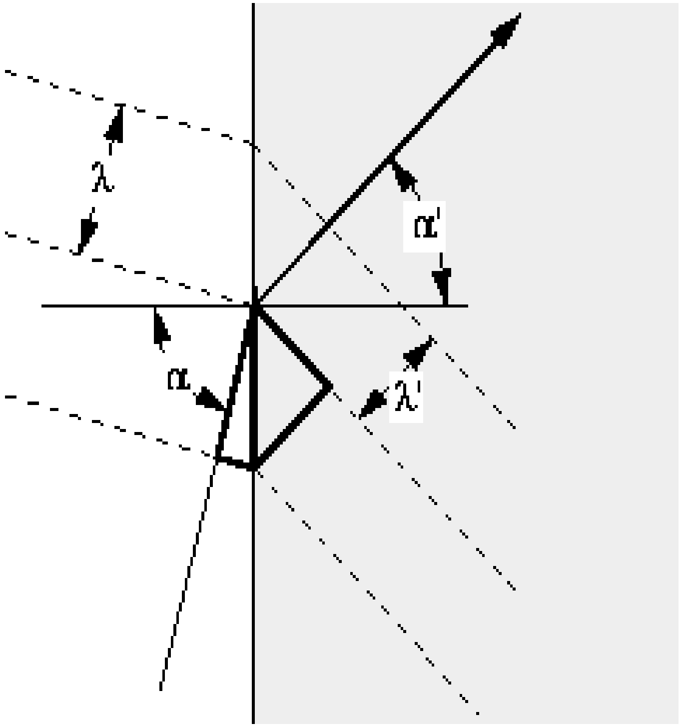


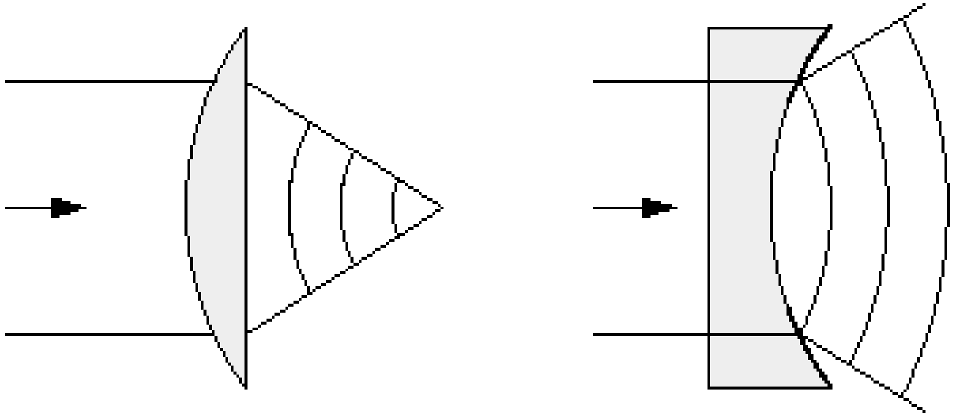
Field emission



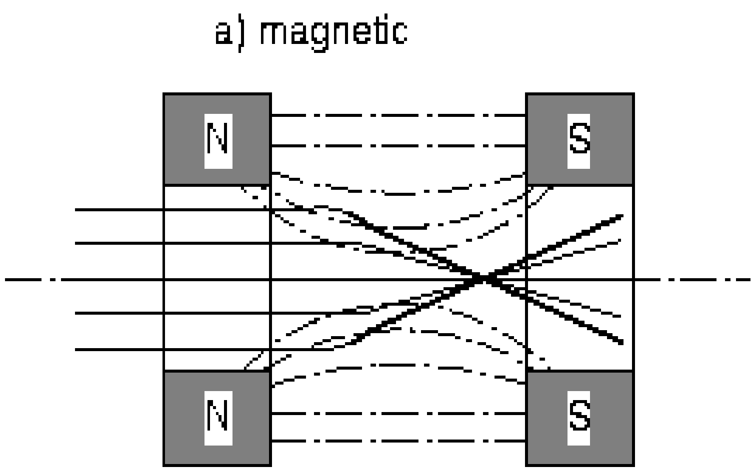


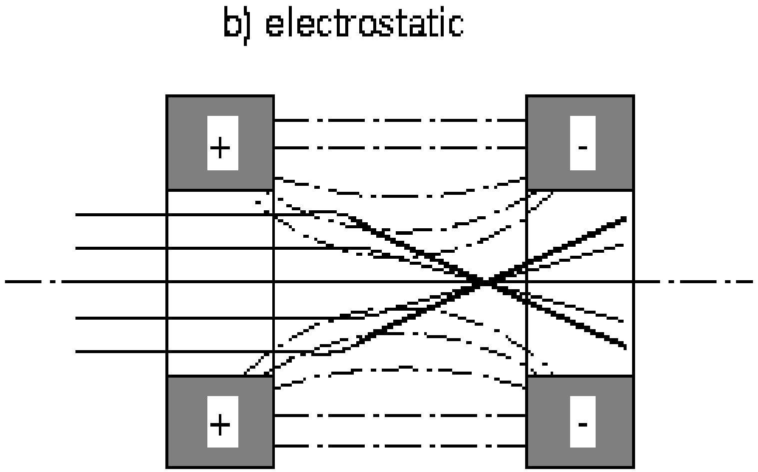
	Thermoionic W	Thermoionic LaB_6	Thermal FEG ZrO-W	Cold FEG
β (200 kV) A/cm^2 ster	$\sim 5\ 10^5$	$\sim 5\ 10^6$	$\sim 5\ 10^8$	$\sim 5\ 10^8$
Source size (μm)	50	10	0.1-1	0.01-0.1
Energy spread (eV)	2.3	1.5	0.6-0.8	0.3-0.5
Operating Pressure (Pa)	10^{-3}	10^{-5}	10^{-7}	10^{-8}
Operating Temp. (K)	2800	1800	1800	300
Beam current (μA)	100	20	100	20



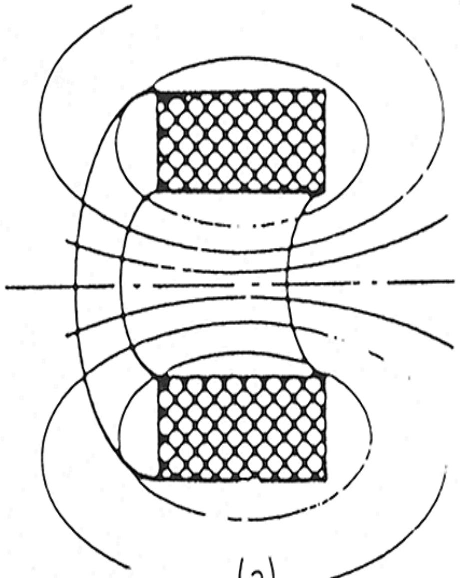


LIGHT:The focusing action is described by the Snell low based on the difference in the refraction index between air and glass.

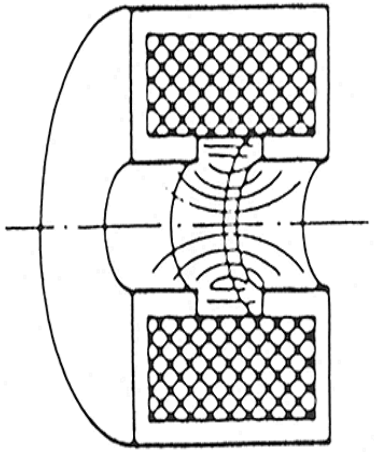




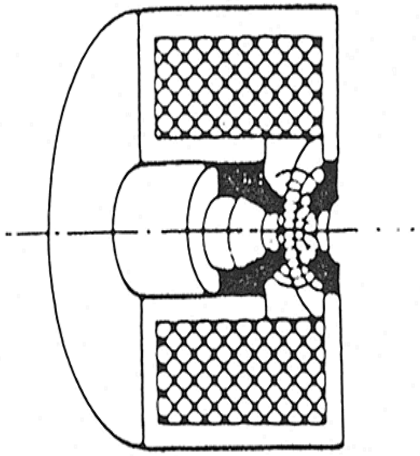
ELECTRONS: the focusing action is operated by a magnetostatic or electrostatic field, which influences the electron trajectories according to the Lorenz force.



(a)



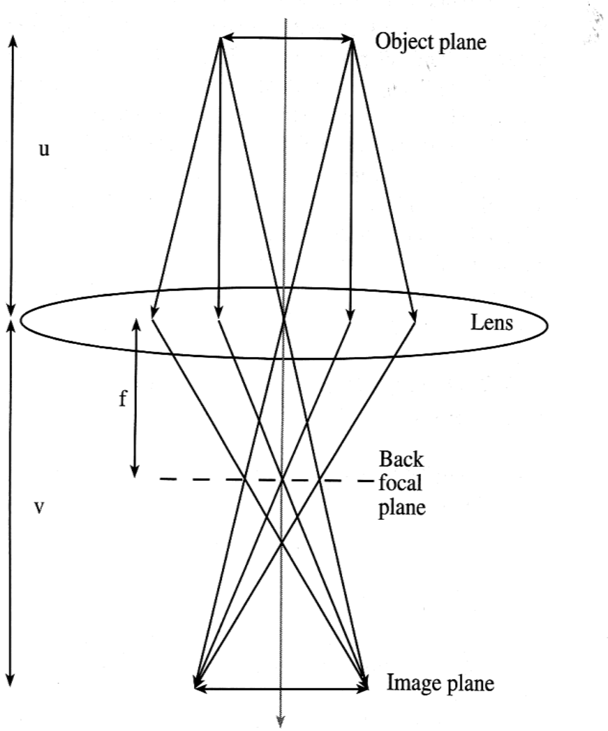
(b)



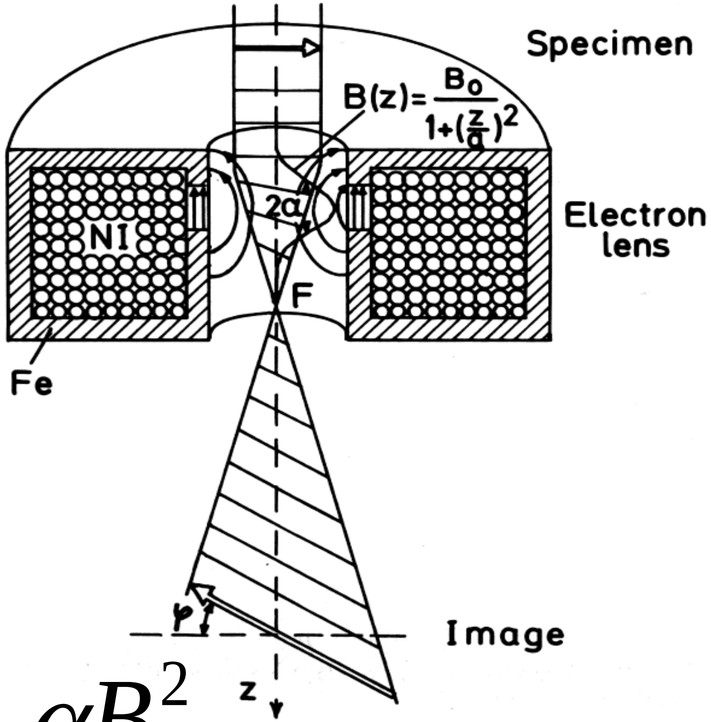
(c)

LENSES

f depends on the radius of curvature of the glass lens



f depends on the z-component of the magnetic field



$$\frac{1}{f}=\frac{\alpha B_z^2}{E_{kin}}$$

The focal length depends on the electron energy and inversely on the square of the axial component of the magnetic field.

The weight of the magnet and the need of keeping the whole column under vacuum imposes fixed distances between lenses.

Optical microscope.

Light lenses with fixed focal length.

Optical column managed by changing the distance between lenses.

Electron microscope.

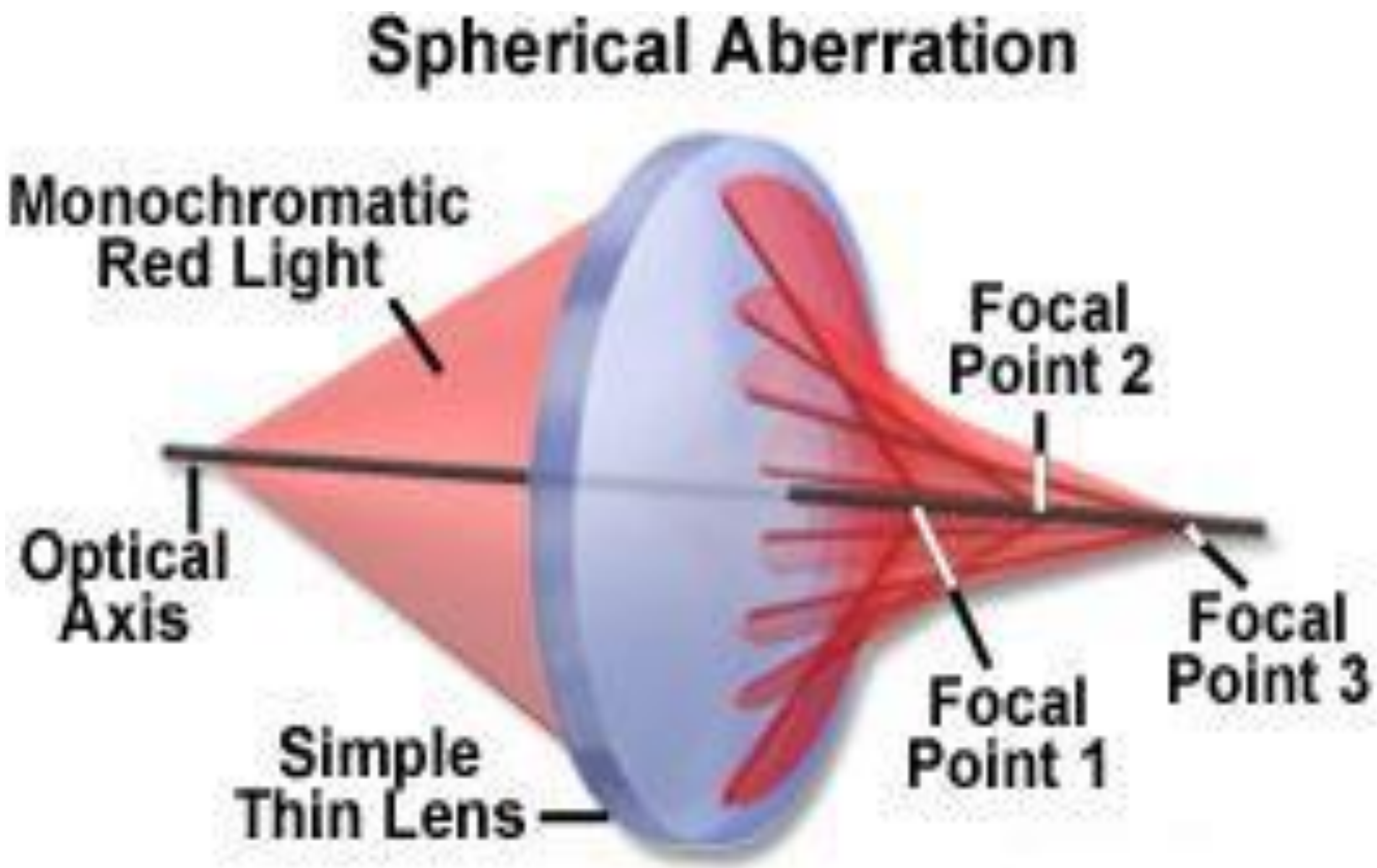
Heavy lenses with variable focal length.

Optical column managed by changing the current in the magnet coils.

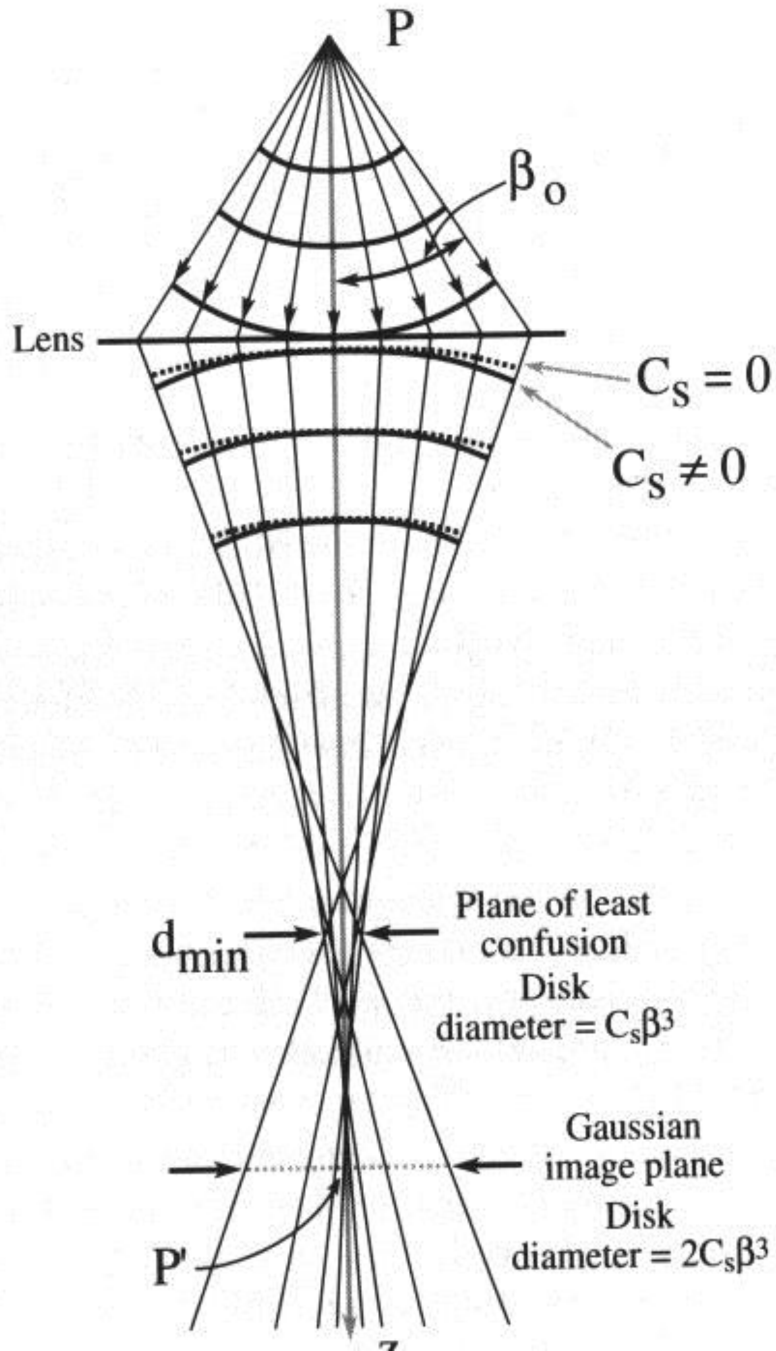
Electron lenses have strong abberrations.

Main aberrations present also on the optical axis are:

- Spherical aberration
- Chromatic aberration
- Astigmatism


Spherical Aberration
Monochromatic
Red Light
Focal
Point 2
Optical
Axis
Focal
Point 3
Focal
Point 1
Simple
Thin Lens

Effect of spherical aberration



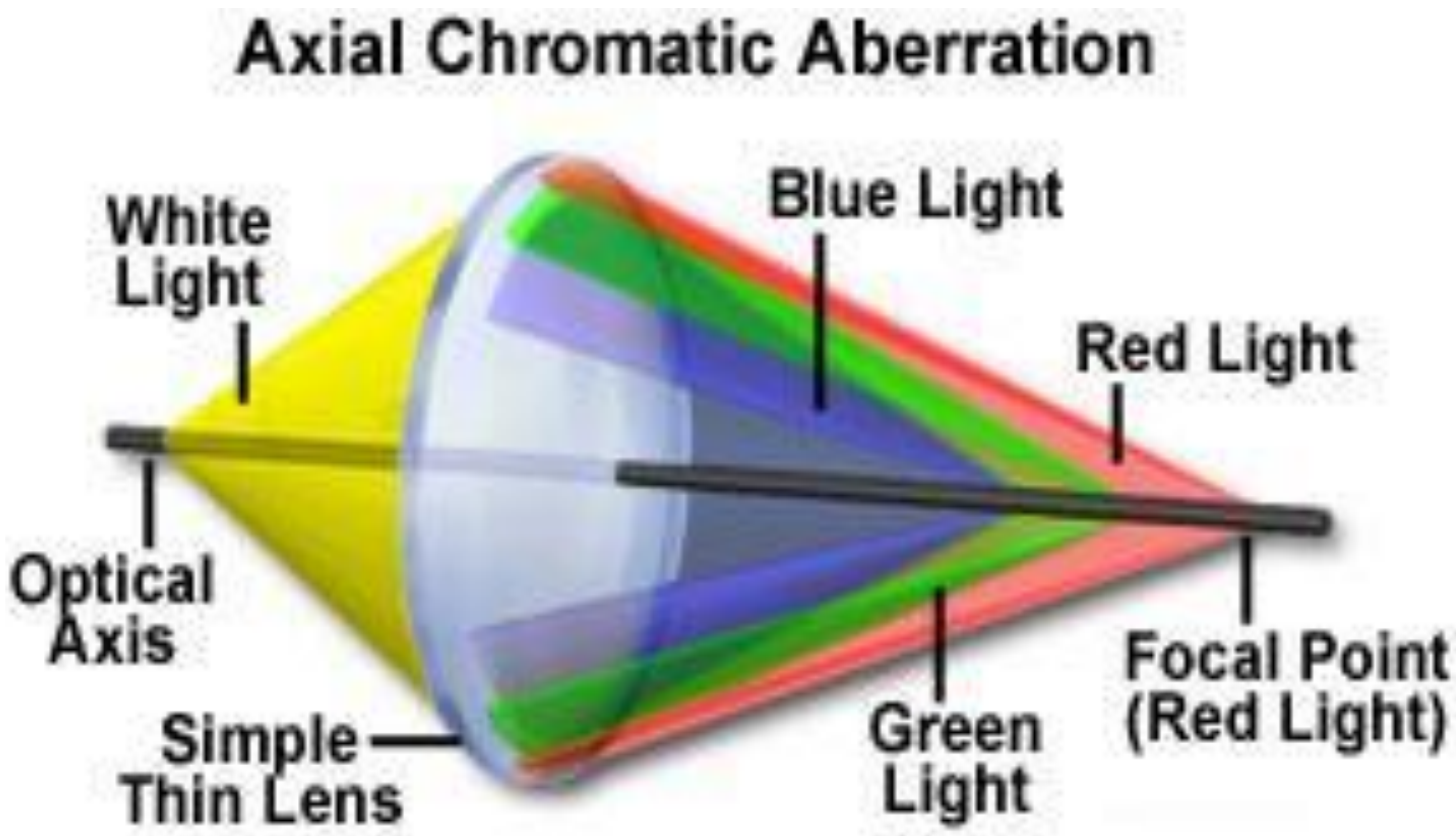
Spherical aberration

- Similar to the aberration of a glass sphere when used as a lens for visible light.

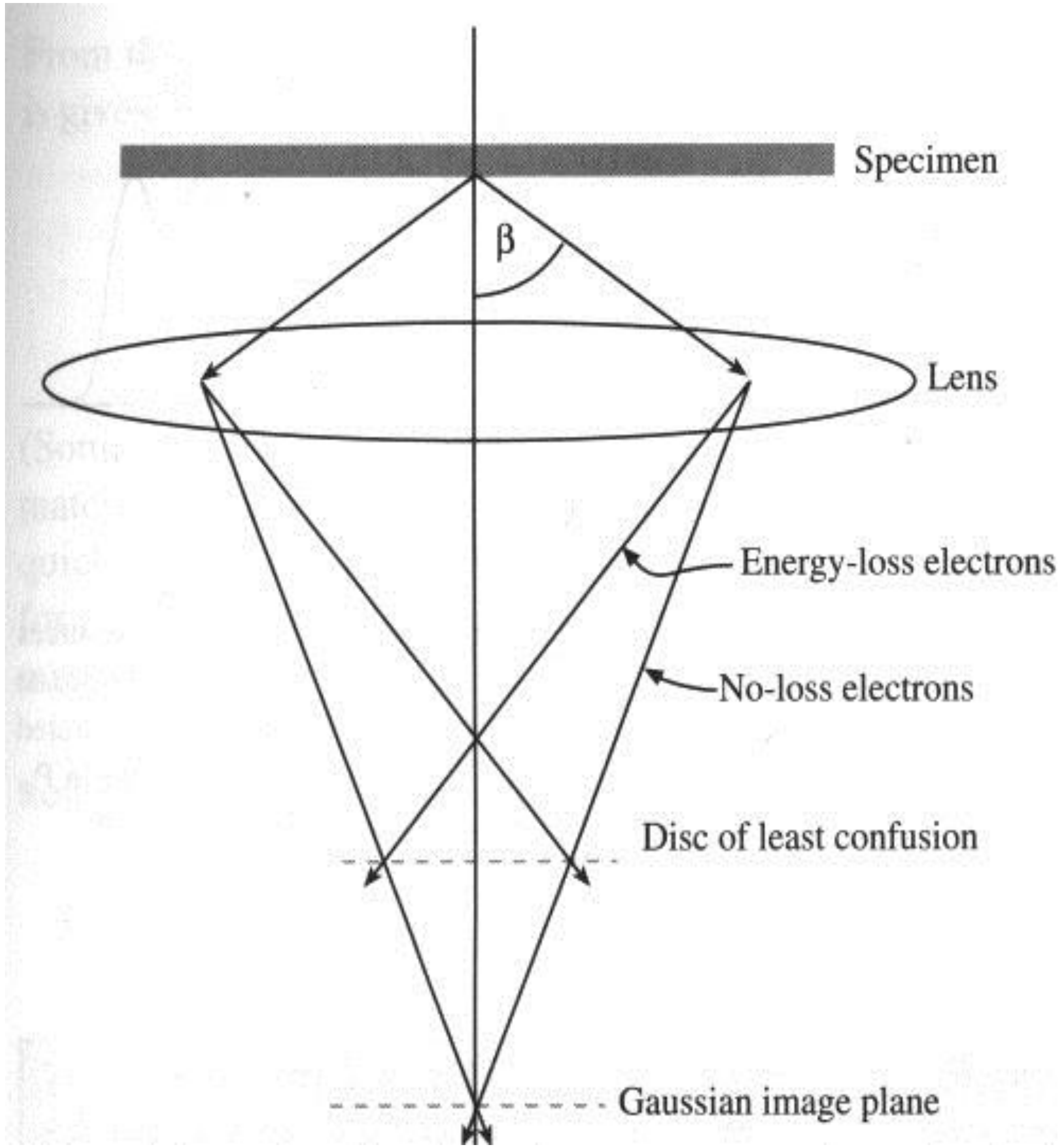
- The effect is to bent the “rays” entering the lens at high angle more than required for a perfect imaging.

- As a consequence a point in the object plane is imaged as a confusion circle with a size (reported at the object level) given by:

$$\Delta r = C_s \alpha^3$$


Axial Chromatic Aberration
White Light
Blue Light
Red Light
Optical Axis
Simple Thin Lens
Green Light
Focal Point (Red Light)

Effect of chromatic aberration



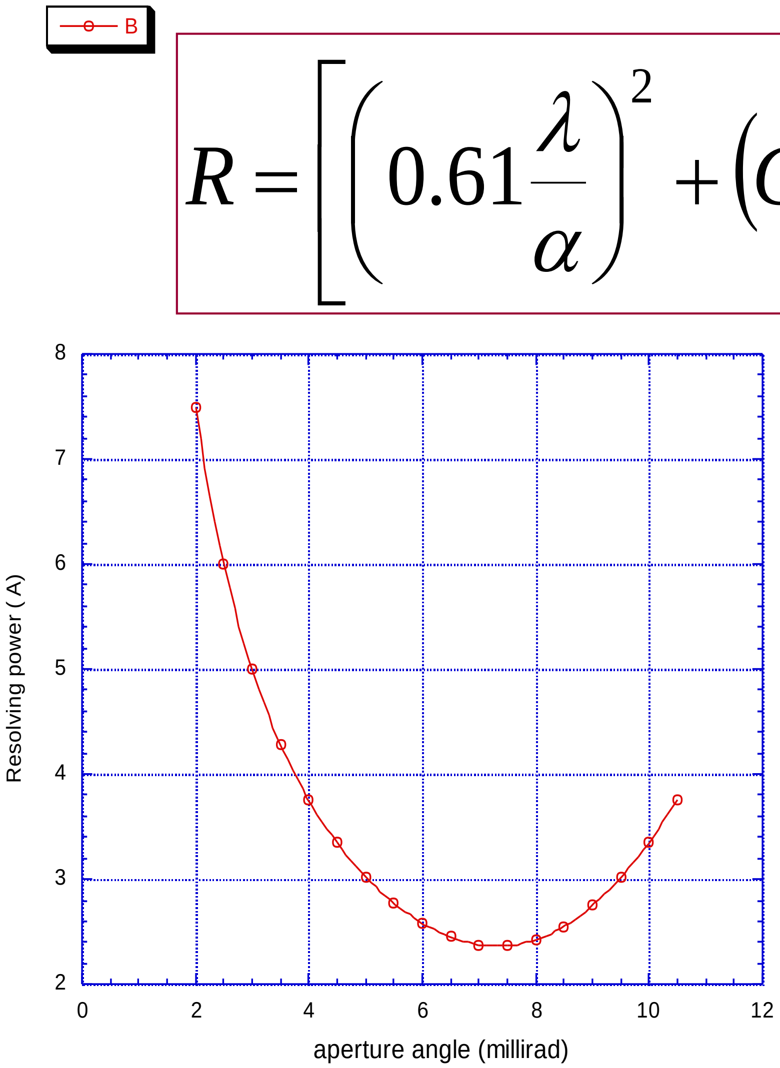
Chromatic aberration

- Is related to the Lorentz force focusing the electrons in a magnetic lens. F=ev x B
- The difference in kinetic energy between two electrons affects the deflection angle imparted by the lens, so that again an image point is transformed in a circle of confusion given by:

$$\Delta r = \alpha C_c \frac{\Delta E}{E}$$

For an ideal specimen which does not induce any energy spread in the electron beam the actual lens resolution is imposed by the combined action of the spherical aberration and of the diffraction limit.

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



C_s=1 mm
λ=0.025Å

SCHERZER THEOREM,
OTTO SCHERZER, 1936

- A strong positive value of the spherical aberration coefficient in electrostatic or magnetostatic lenses with axial symmetry can not be avoided.
- Optical devices, having axial symmetry and DC powering, with a negative spherical aberration coefficient can not be manufactured.
- Spatial resolution of a conventional electron microscope is about 100 times larger than the electron wavelength

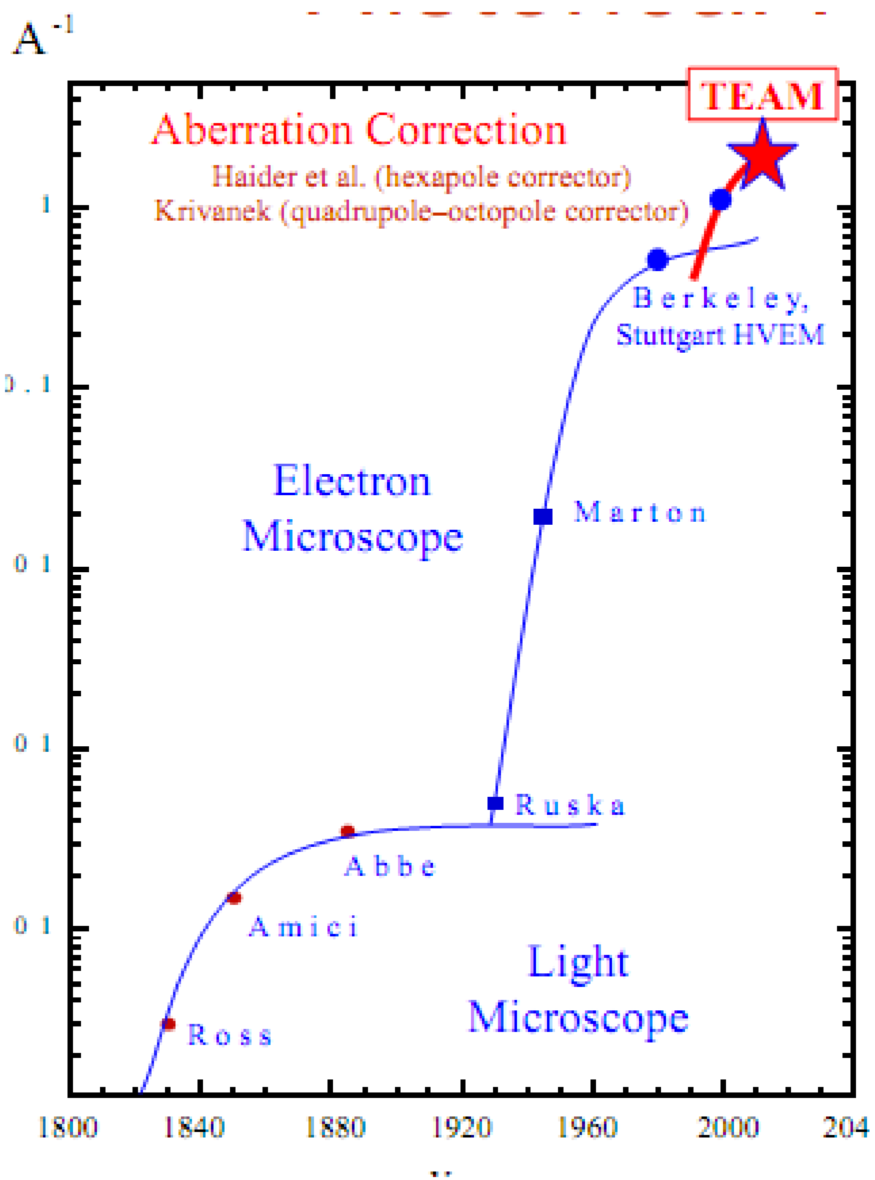
Plenty of Room at the Bottom

Richard P. Feynman

December 1959

Discussing how to write the whole Encyclopaedia Britannica on a nidle tip………

If I have written in a code, with 5 times 5 times 5 atoms to a bit, the question is: How could I read it today? The electron microscope is not quite good enough, with the greatest care and effort, it can only resolve about 10 angstroms. I would like to try and impress upon you while I am talking about all of these things on a small scale, the importance of improving the electron microscope by a hundred times. It is not impossible; it is not against the laws of diffraction of the electron. The wave length of the electron in such a microscope is only 1/20 of an angstrom. So it should be possible to see the individual atoms. What good would it be to see individual atoms distinctly?

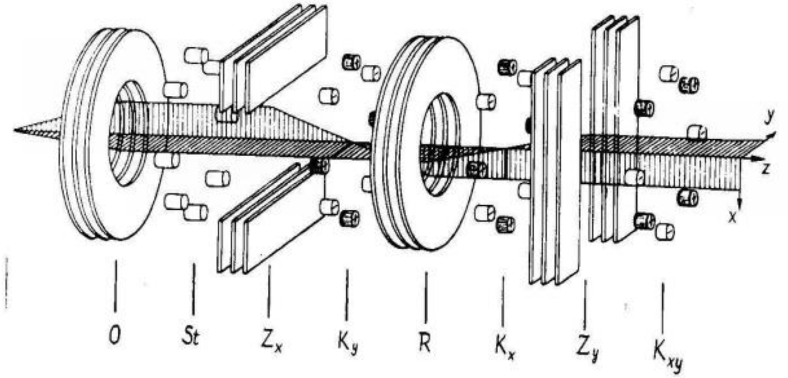


- By improving mechanical stability
- By improving powering stability
- By improving lens design
- By increasing the accelerating voltage.
- By introducing aberration correctors

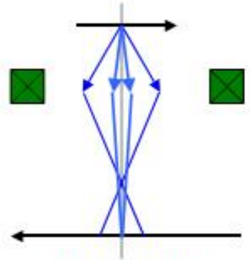
Harald Rose, Technical University Darmstadt, Germany

Spherical aberration correctors

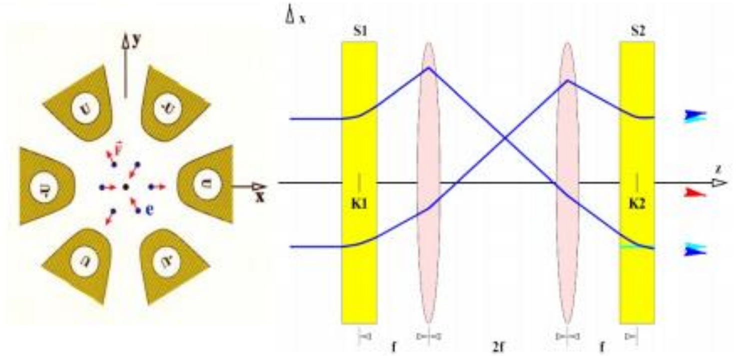
Scherzer-Seeliger Corrector



Correction of Spherical Aberration



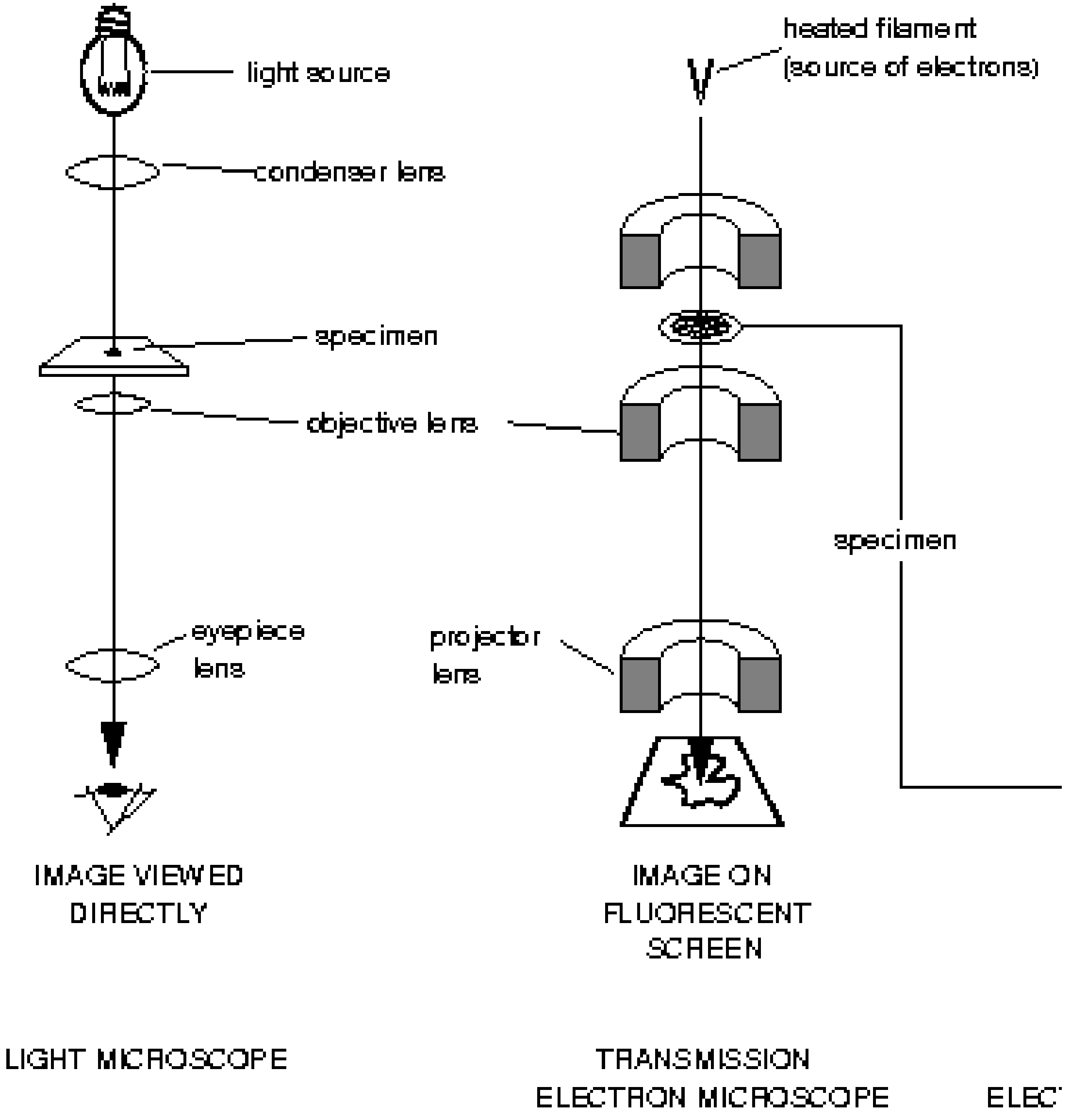
$_{point} \approx 0.6\ \lambda^{3/4}\ C_s^{1/4}$



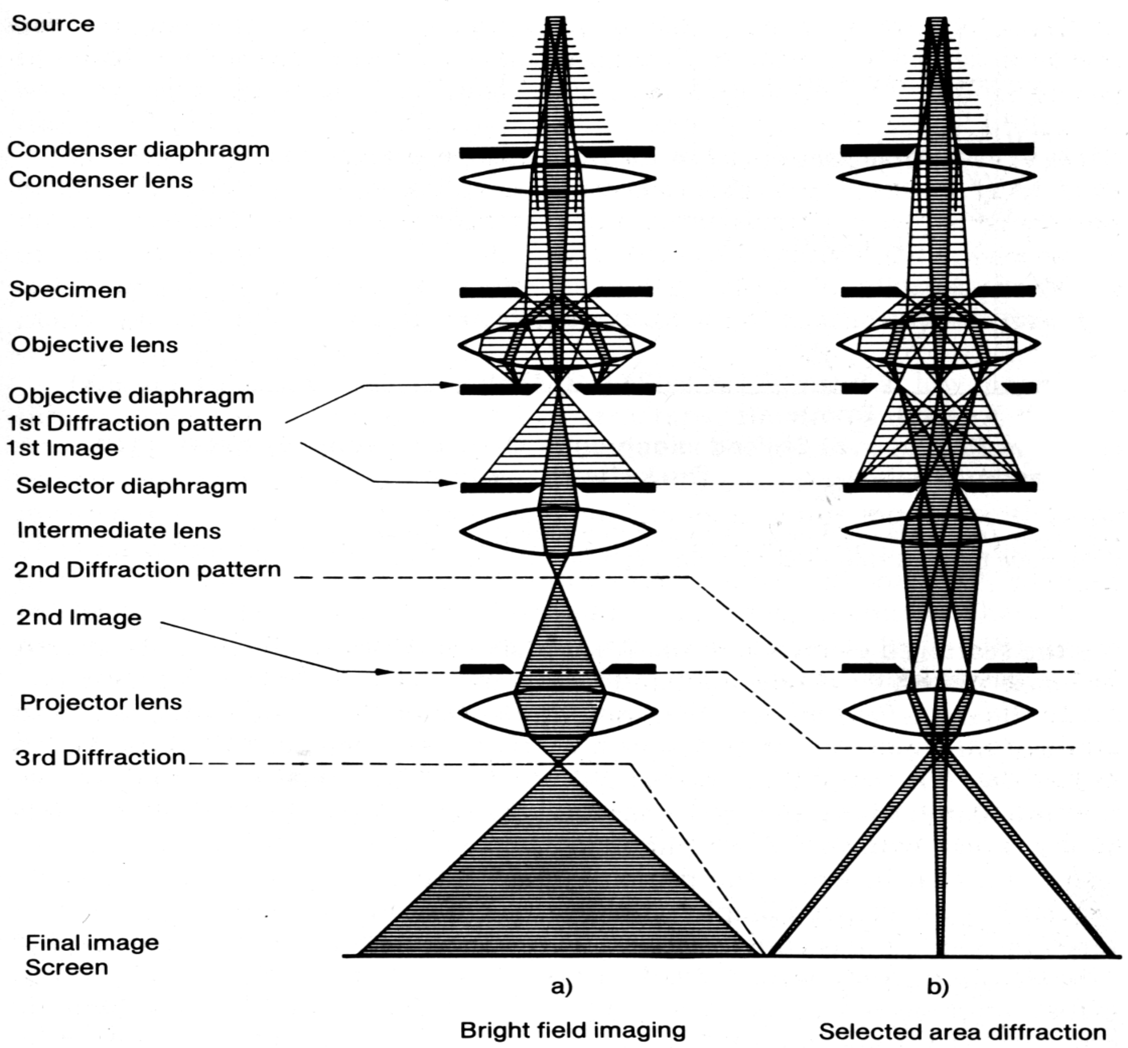
C_s corrector using hexapoles and a round-lens transfer doublet (Rose Optik 1990, Haider et al. Nature 1998)

Contrast:

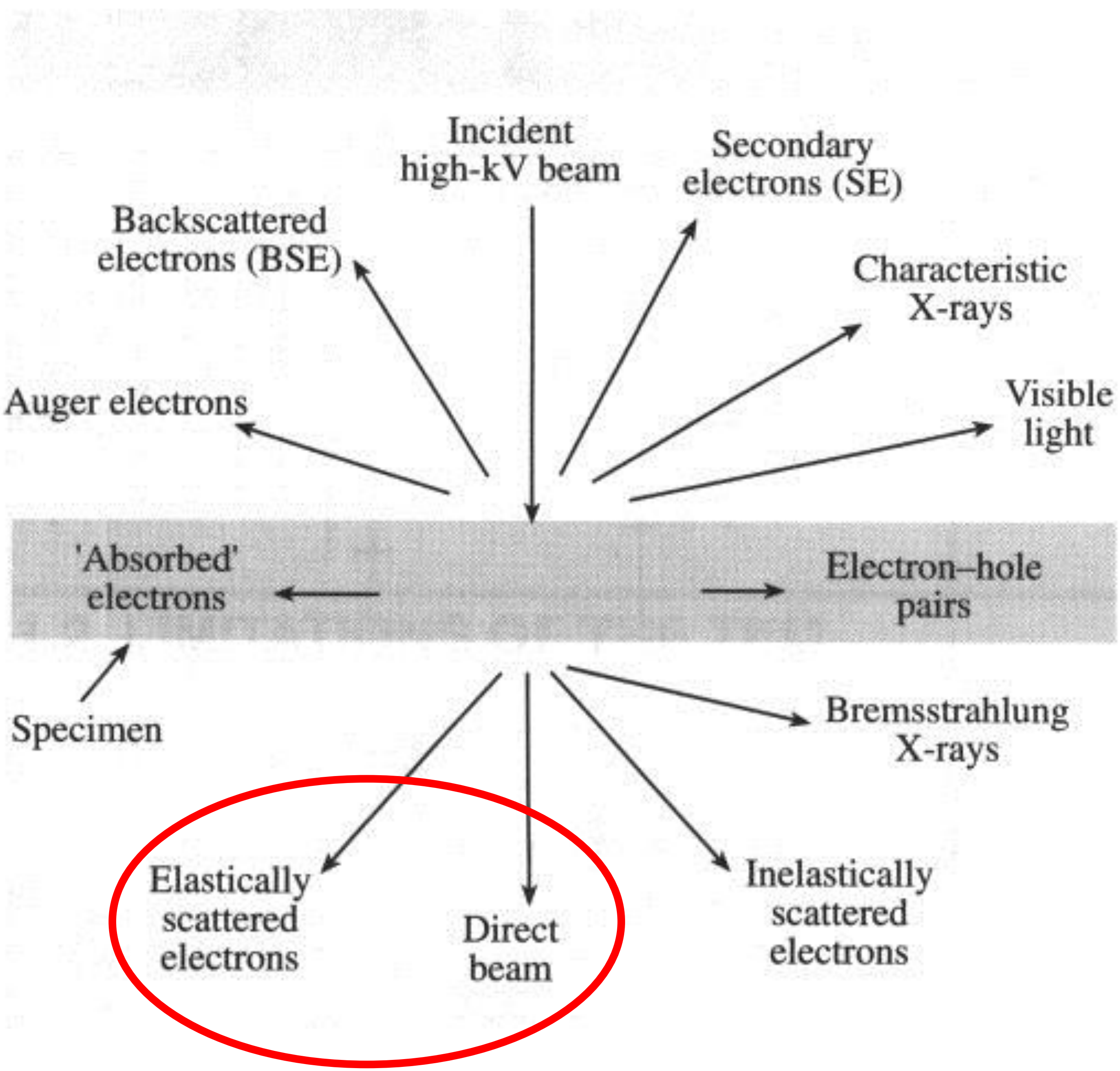
- Contrast mechanisms
- Diffraction contrast
- Phase contrast
- Scattering contrast

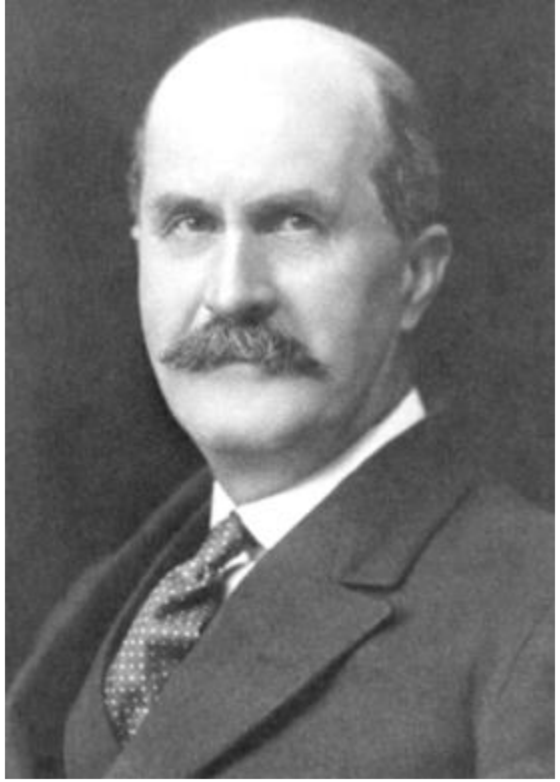

light source
condenser lens
specimen
objective lens
eyepiece lens
IMAGE VIEWED DIRECTLY
heated filament (source of electrons)
specimen
projector lens
IMAGE ON FLUORESCENT SCREEN
LIGHT MICROSCOPE
TRANSMISSION ELECTRON MICROSCOPE
ELEC

TEM IMAGING SYSTEM WITH PARALLEL BEAM

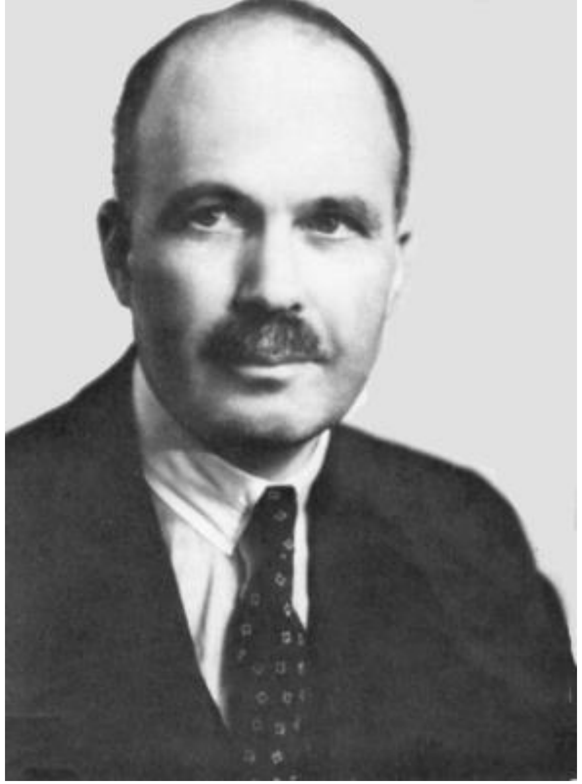


Ray diagram for a transmission electron microscope in (**a**) the bright-field mode and (**b**) selected-area electron diffraction (SAED) mode


Incident
high-kV beam
Secondary
electrons (SE)
Backscattered
electrons (BSE)
Characteristic
X-rays
Auger electrons
Visible
light
'Absorbed'
electrons
Electron–hole
pairs
Specimen
Bremsstrahlung
X-rays
Elastically
scattered
electrons
Direct
beam
Inelastically
scattered
electrons



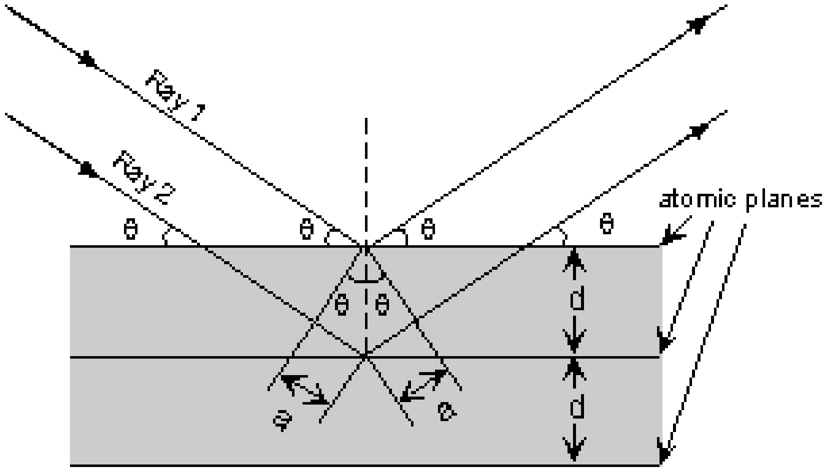
William Henry Bragg

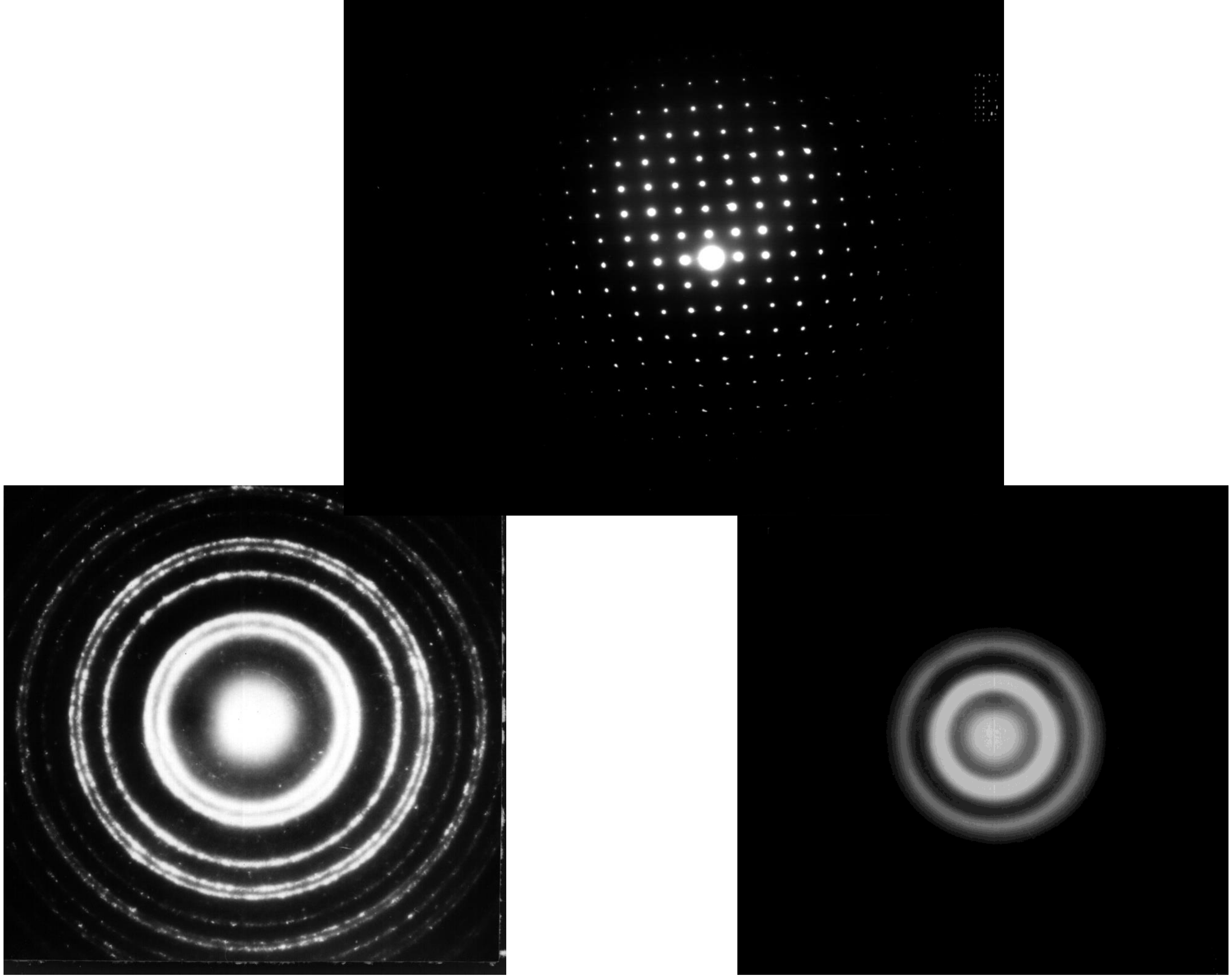


Williams Lawrence Bragg

Nobel Laureates in Physics 1915

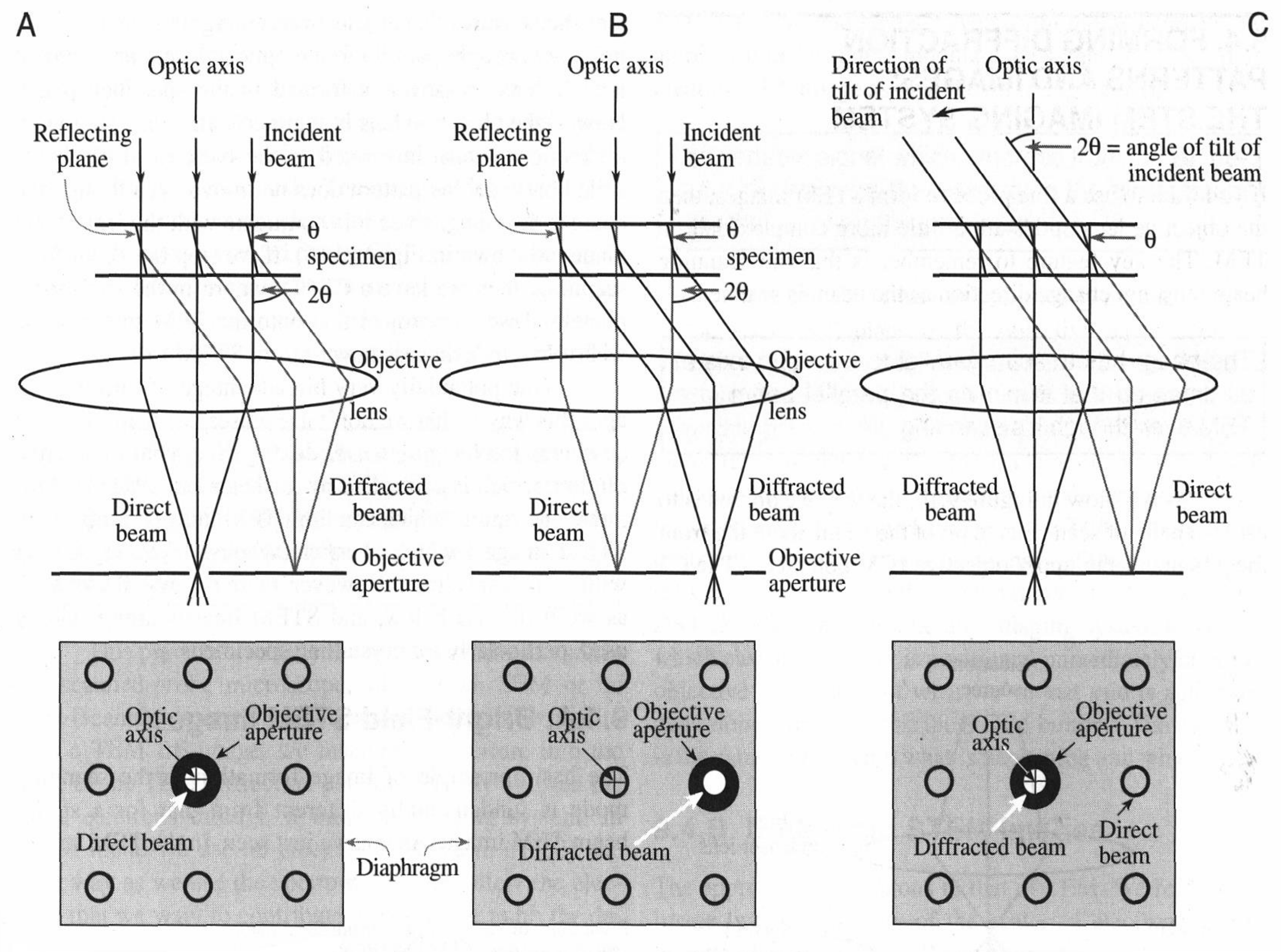
$$n\lambda=2d\ \text{sen}\theta$$





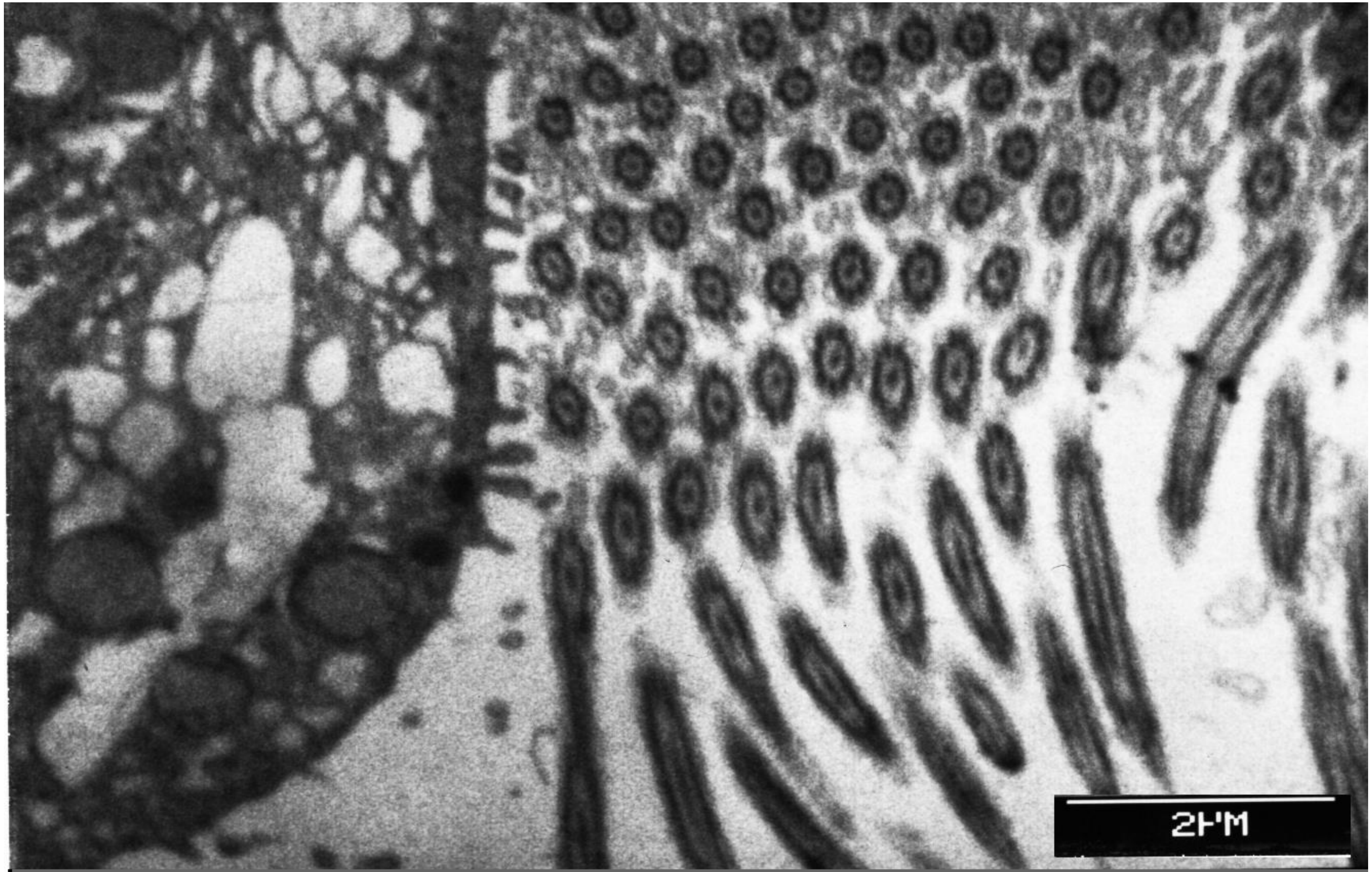
BRIGHT-FIELD AND DARK-FIELD IMAGING

The diffraction contrast is given by selecting one scattered beam by an aperture placed in the back focal plane of the objective lens.



- Contrast in the image is related to the electron wave amplitude scattered by different points of the object in the direction and within the angular range defined by the position and the size of the objective lens aperture.

- For amorphous specimen the contrast in bright field is dominated by the total local elastic scattering cross section so that areas with heavy elements apperar darker owing to their capability of scattering electrons out of the angle defined by the objective aperture.

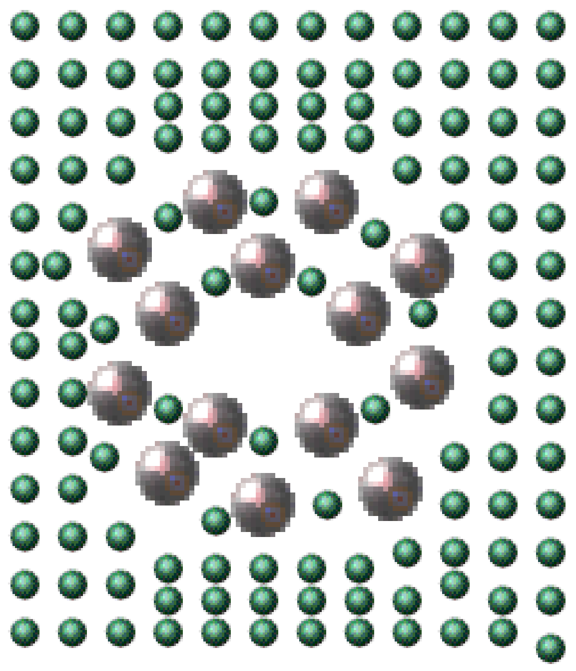

2μM

For crystalline sample it is possible to observe all the features affecting the diffracted intensity and in particular:

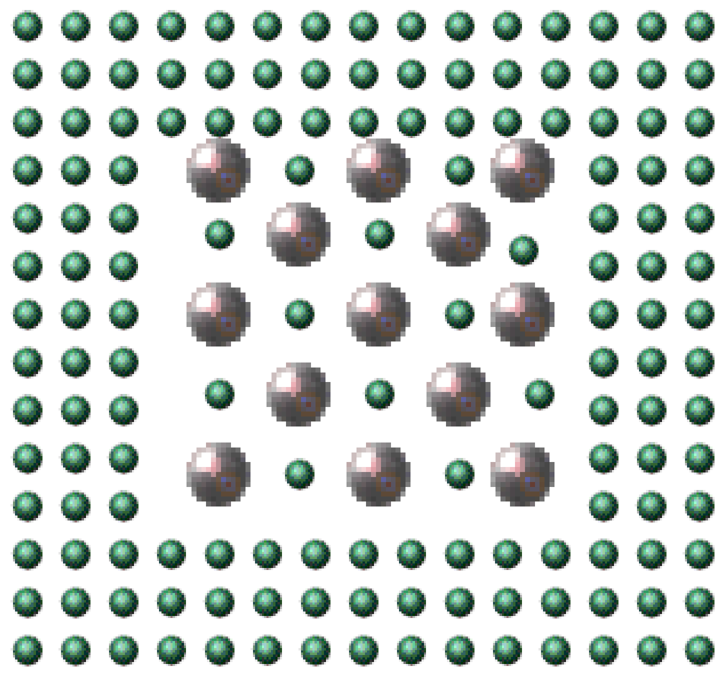
- Thickness variation
- Crystal defects locally affecting the periodicity of the crystal lattice.
- Change in the lattice orientation i.e. at a grain boundary.
- Change in the local crystal structure i.e. precipitation

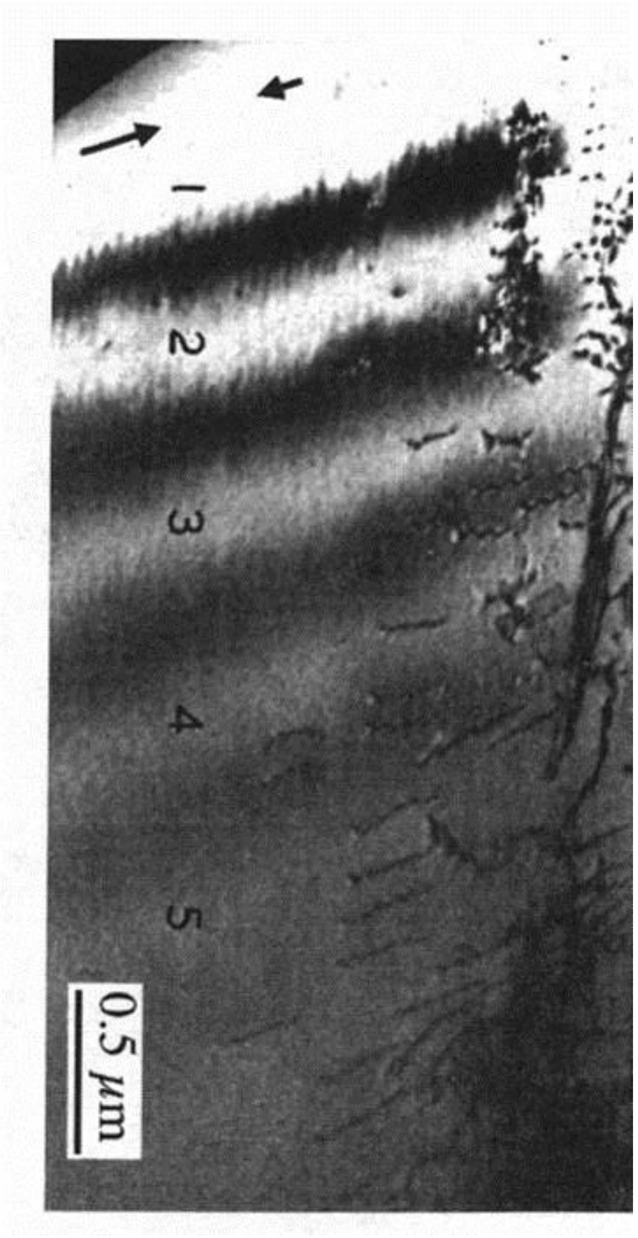
Types of Precipitates

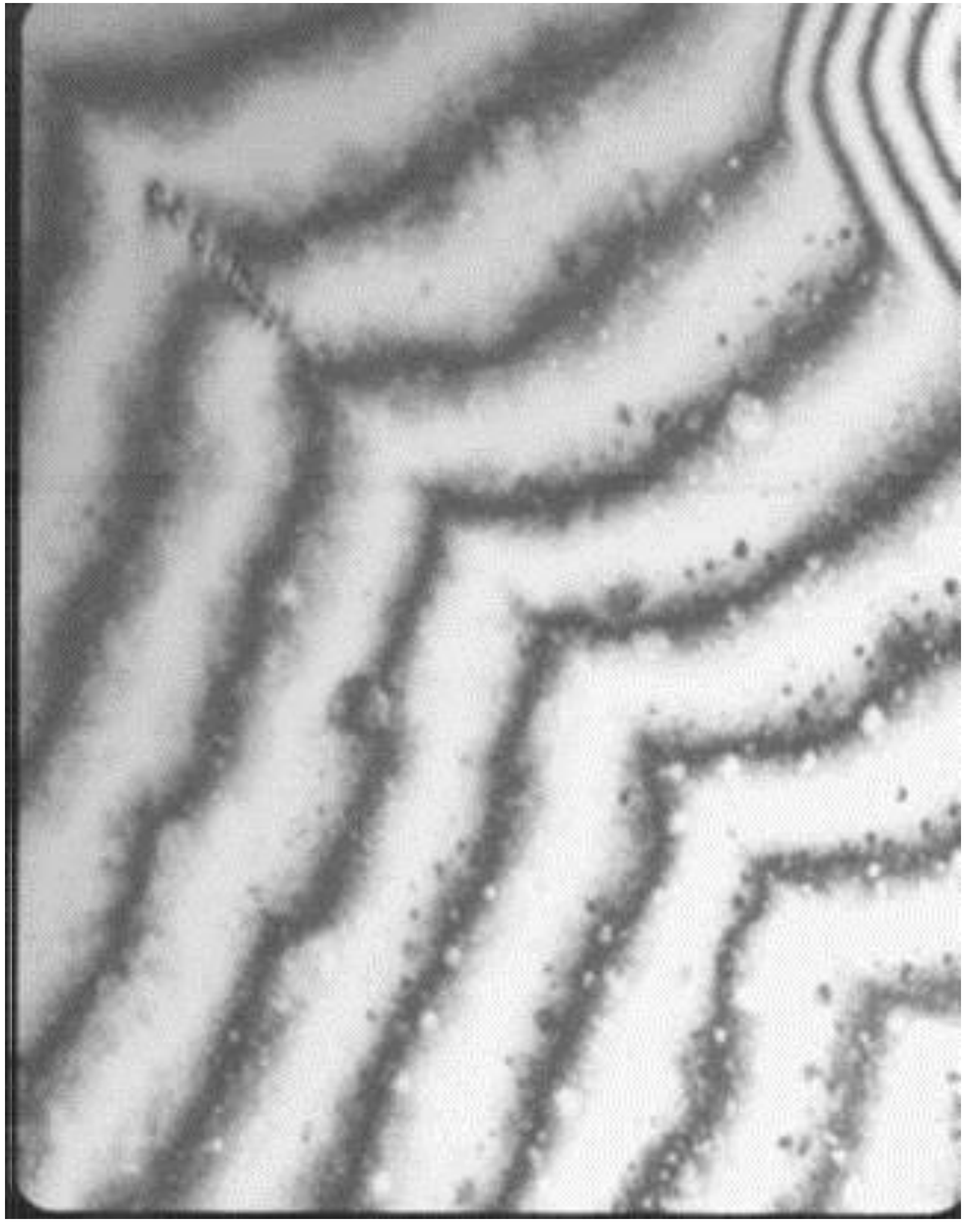
Coherent

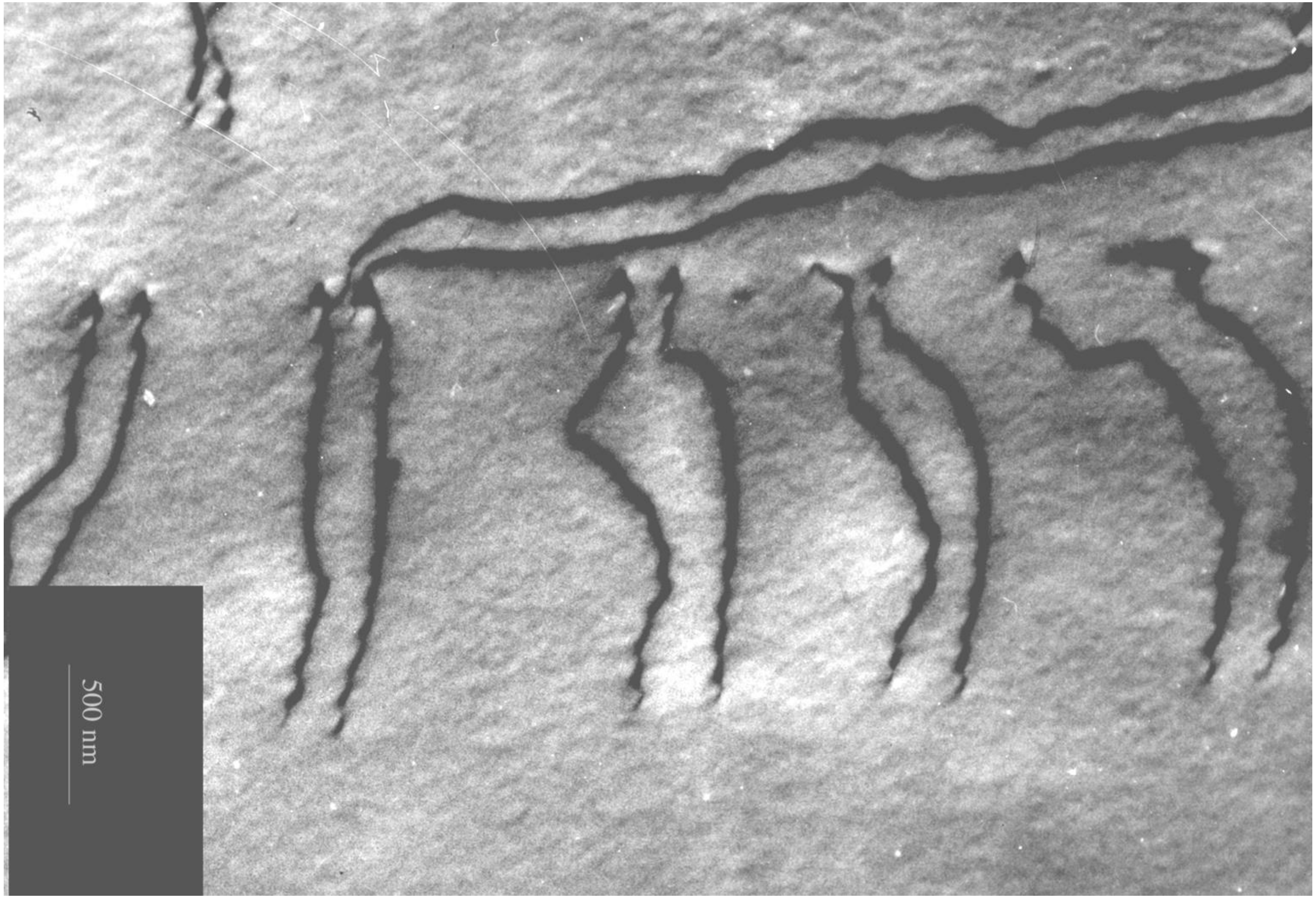


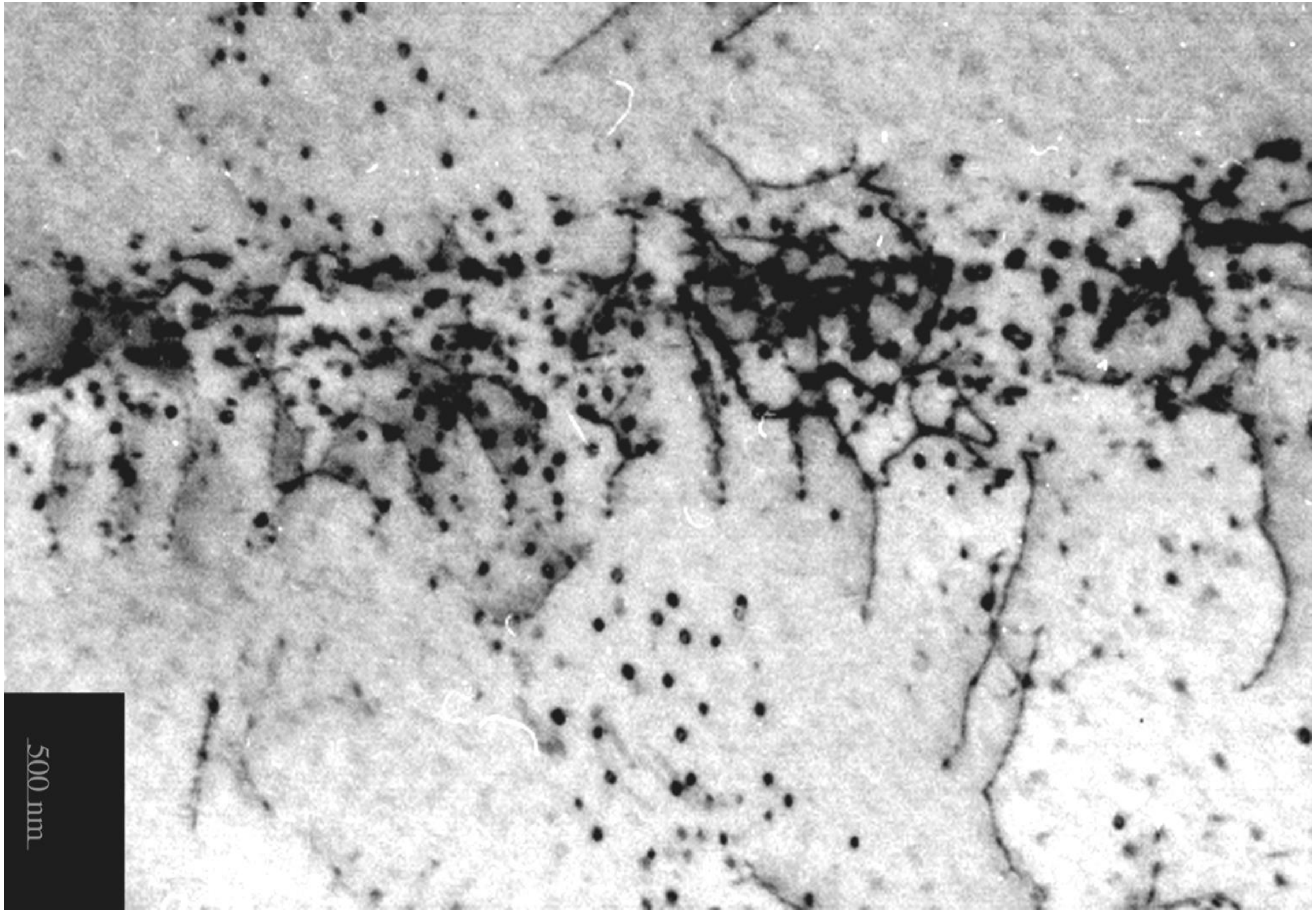
Non Coherent

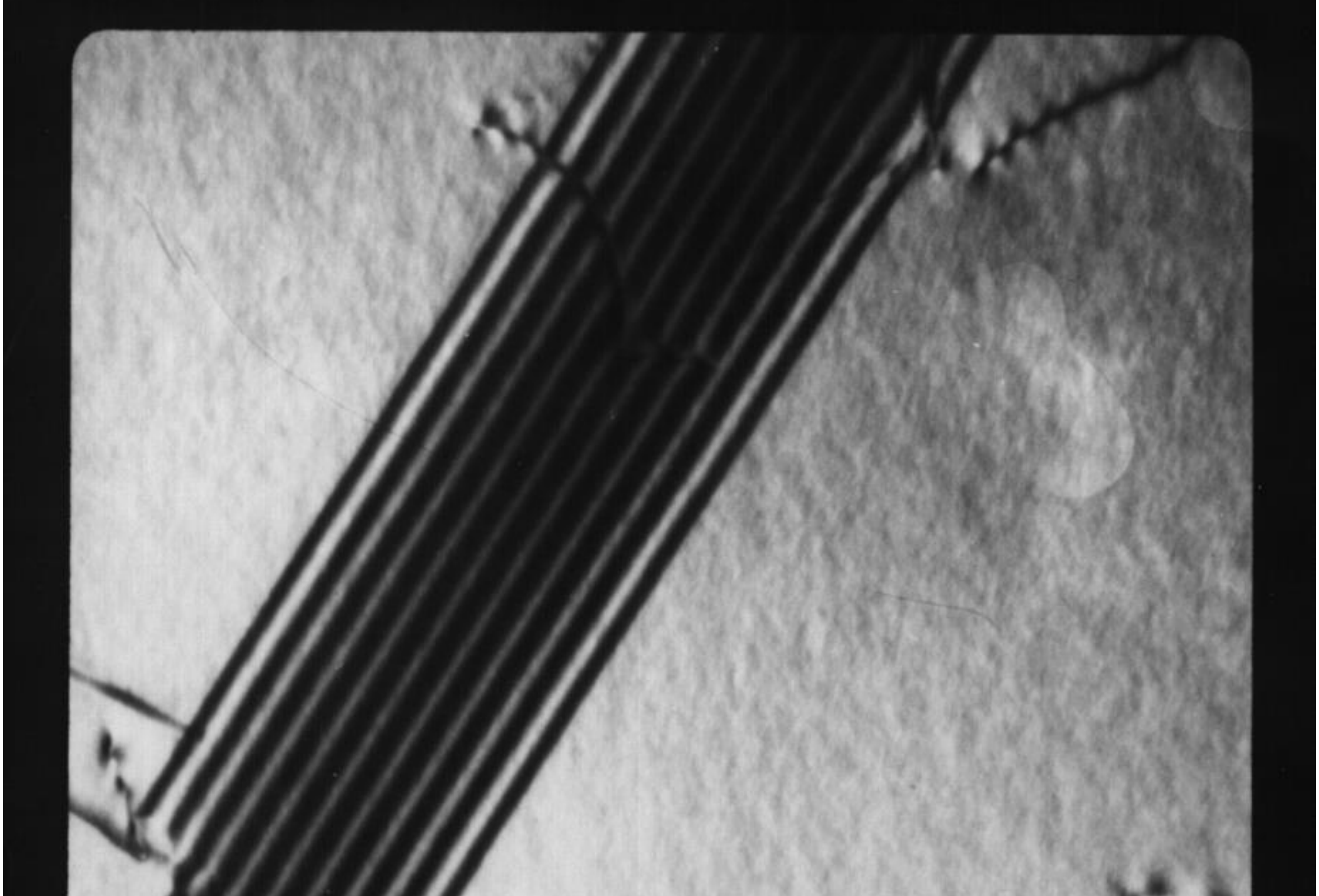


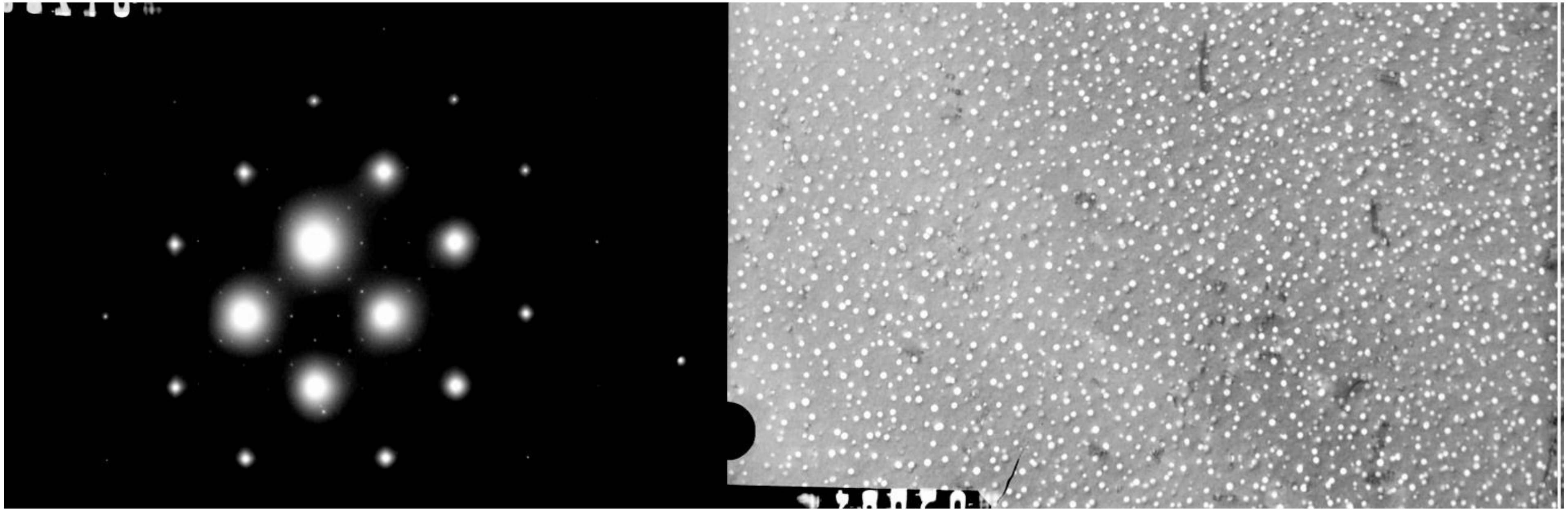

1
2
3
4
5
0.5 μm



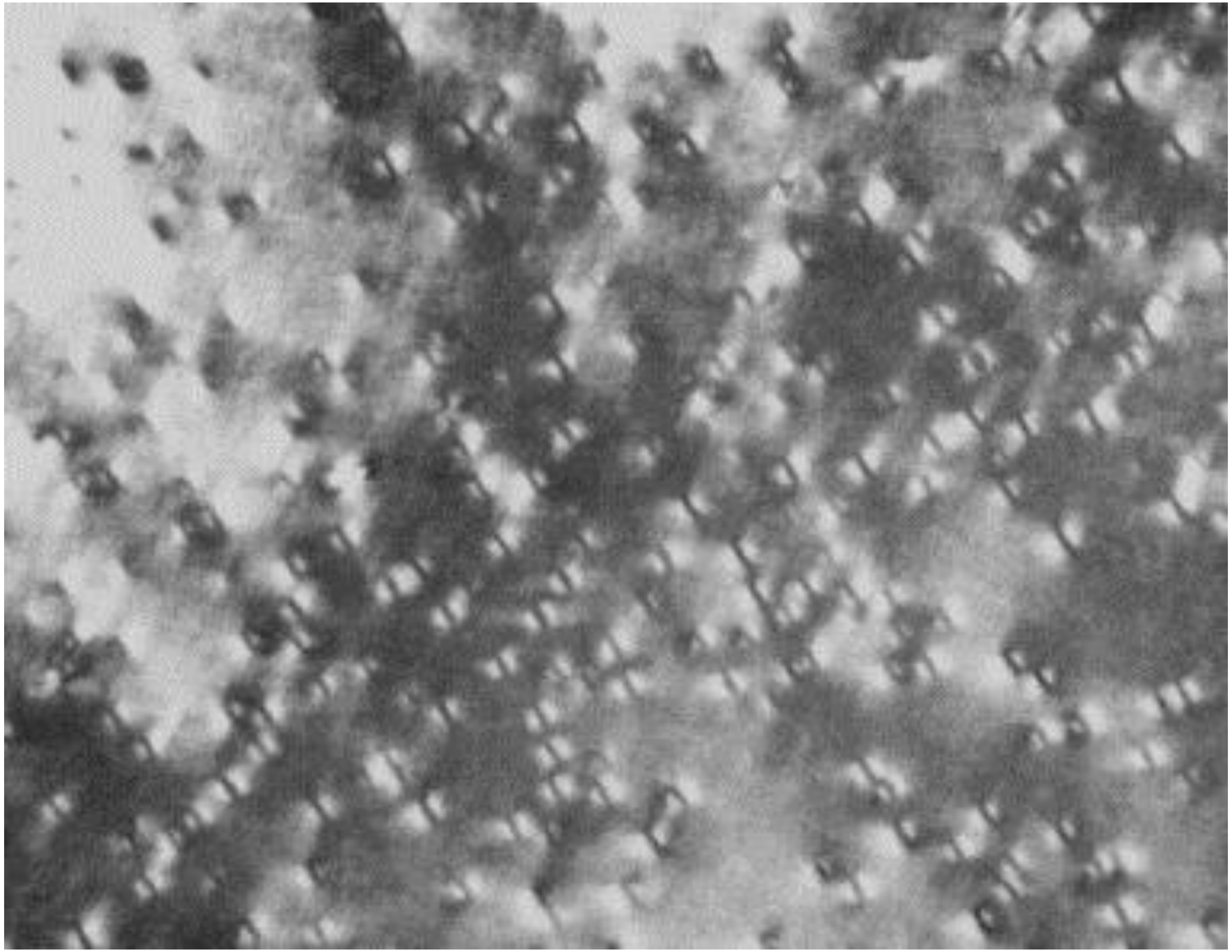

500 nm

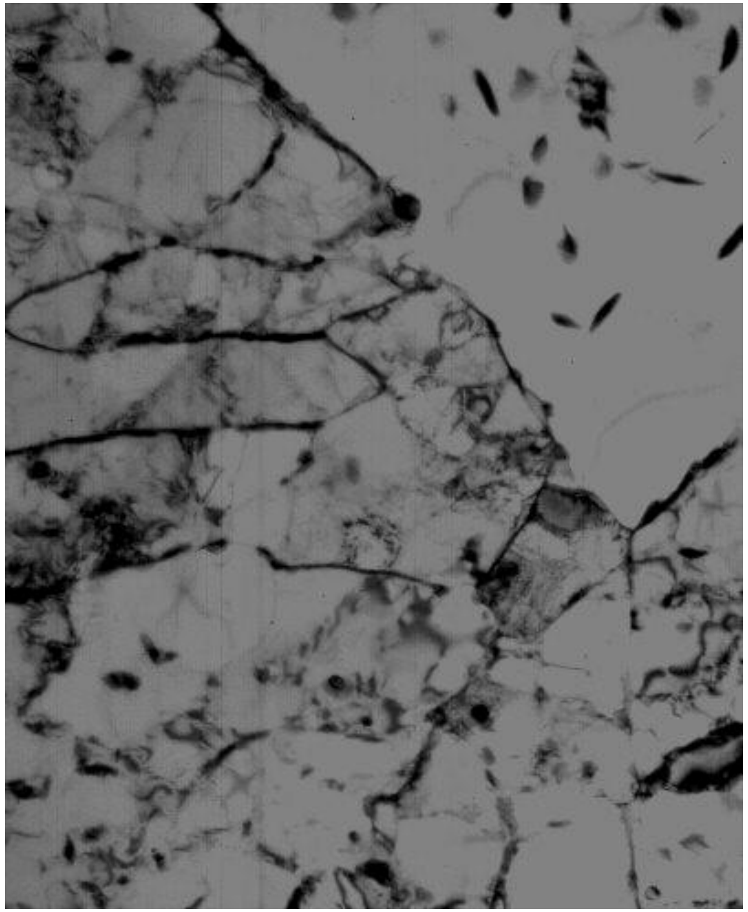

500 nm





Electron diffraction and D.F. image of a γ/γ’ iron base alloy.





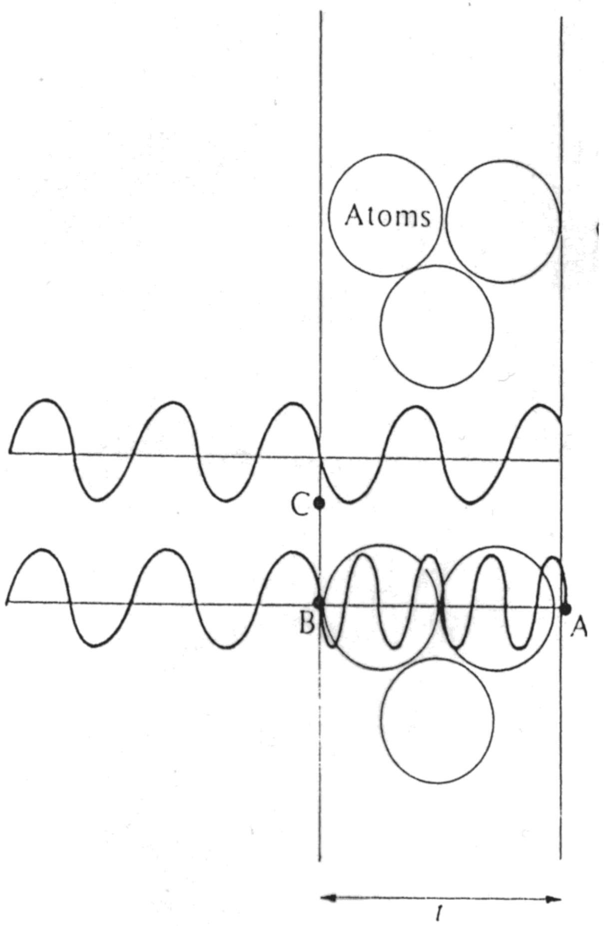
ATOMIC RESOLUTION

- In diffraction contrast mode, the spatial resolution is limited by the aperture inserted in the back focal plane of the objective lens
- In order to improve spatial resolution down to the inter-atomic distances, the aperture has to be removed
- In this condition the main effect of the sample is to aaffect the electron phase
- Only projected images are provided, so that atomic columns have to be aligned with the electron beam.
- The only method to obtain direct lattice information from the specimen bulk in direct space.

WEAK PHASE OBJECT APPROXIMATION

$\psi = \exp(-ikr)$

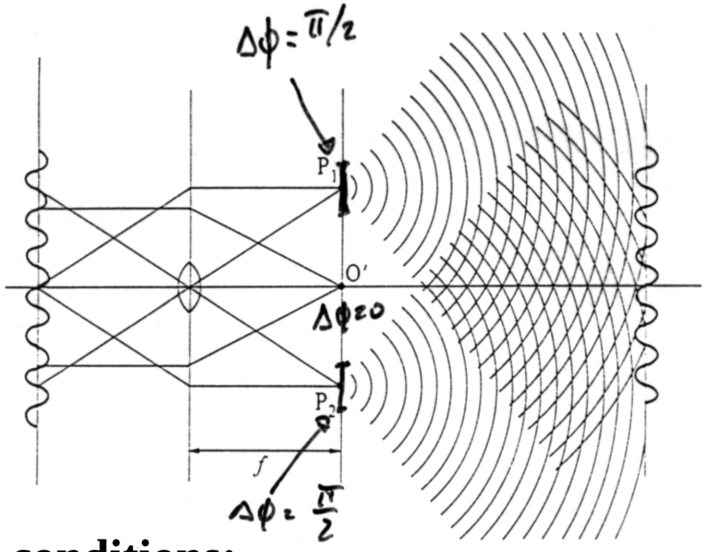
$\lambda = 1.22/E^{1/2}$



$\psi = \exp(i\Phi(x)) \Rightarrow |\psi|^2 = 1$

$\Phi(x) \ll 1$

$\psi = \exp(i\Phi(x)) \approx 1 + i\Phi(x)$



$\psi = \exp(i\Phi(x)) \approx 1 + i\,\Phi(x)$

If we can setup the following conditions:

$\Delta\Phi=\pi/2$ out of the optical axis (diffracted beams carrying the spacing information)

$\Delta\Phi=0$ on the optical axis (reference unscattered beam)

In the image plane the wavefunction is:

$\psi = 1+i\,\Phi(x)\,\exp i\,\pi/2 = 1+i\,i\,\Phi(x) = 1-\Phi(x)$

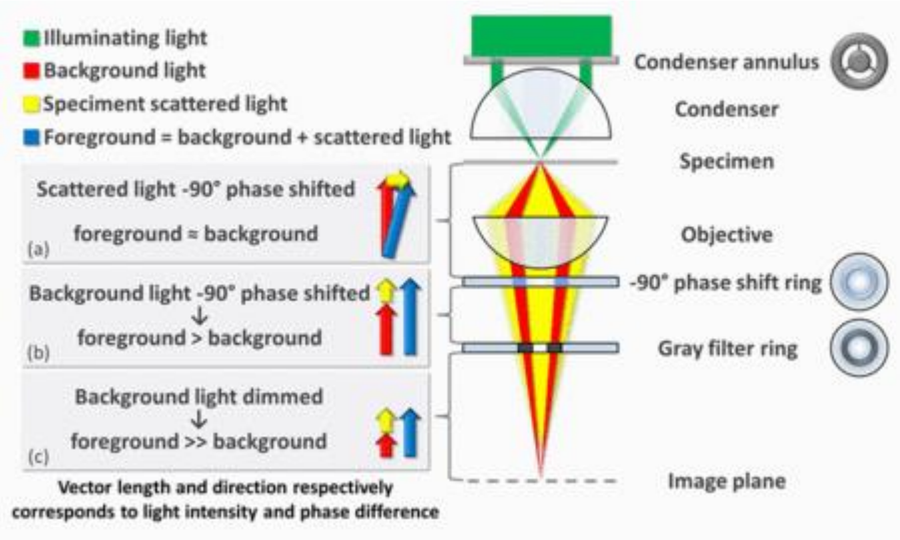
And the beam intensity (square of the wavefunction) neglecting the square of the phase shift (WPOA) is:

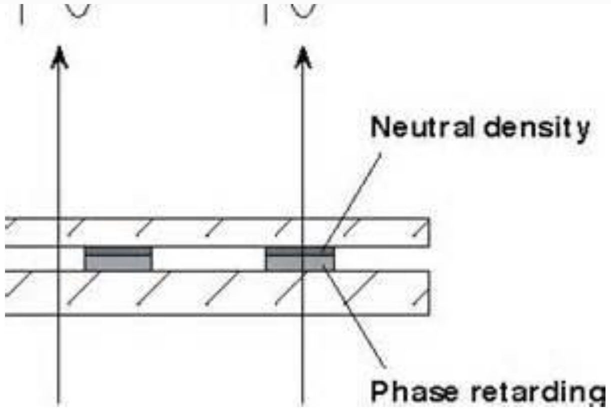
$|\psi|^2 \approx 1-\Phi(x)$

The phase shift results in this way in an intensity modulation which can be detected and recorded.

A similar problem is present also in optical microscopy.
A solution has been found by Frits Zernike, Nobel laureate in Physics in 1953, and is based on the insertion of a glass ring in the back focal plane of the objective lens in order to affect in the proper way the phase of the scattered radiation.

Frits Zernike
(1888-1966)





How can we induce a phase shift in the diffracted beams without affecting the transmitted beam?

In optical microscopy this is acheived by inserting in the back focal plane of the lens a physical device constituted by a glass plate with a defined thickness and a central hole for the direct beam, shifting the phase of the diffracted beams by a quarter of the wavelength.

In electron microscopy such a device can not be realized and we use the phase shift induced by the lens abberrations.

In fact, in terms of physical optics the deflecting effect caused by the spherical aberration corresponds to a phase shift given by:

$\Delta\phi = 1/2\ \pi\ C_s \lambda^3 \alpha^4$

A further phase shift can be governed by the defocus:

$\Delta\phi = \pi\ \Delta f\ \lambda\ \alpha^2$

Considering that the phase shift due to defocus shows a different angular behaviour and can be either positive or negative and the absolute value depends on the amount of overfocus or underfocus (controlled by the objective lens setting), we can try to use the combined action of the two kind of aberration to induce a phase shift of $\pi/2$ in the diffracted beams, leaving unaffected the transmitted beam.

The optimum condition for balancing the two aberrations at the desired phase shift has been derived by Scherzer in 1949.

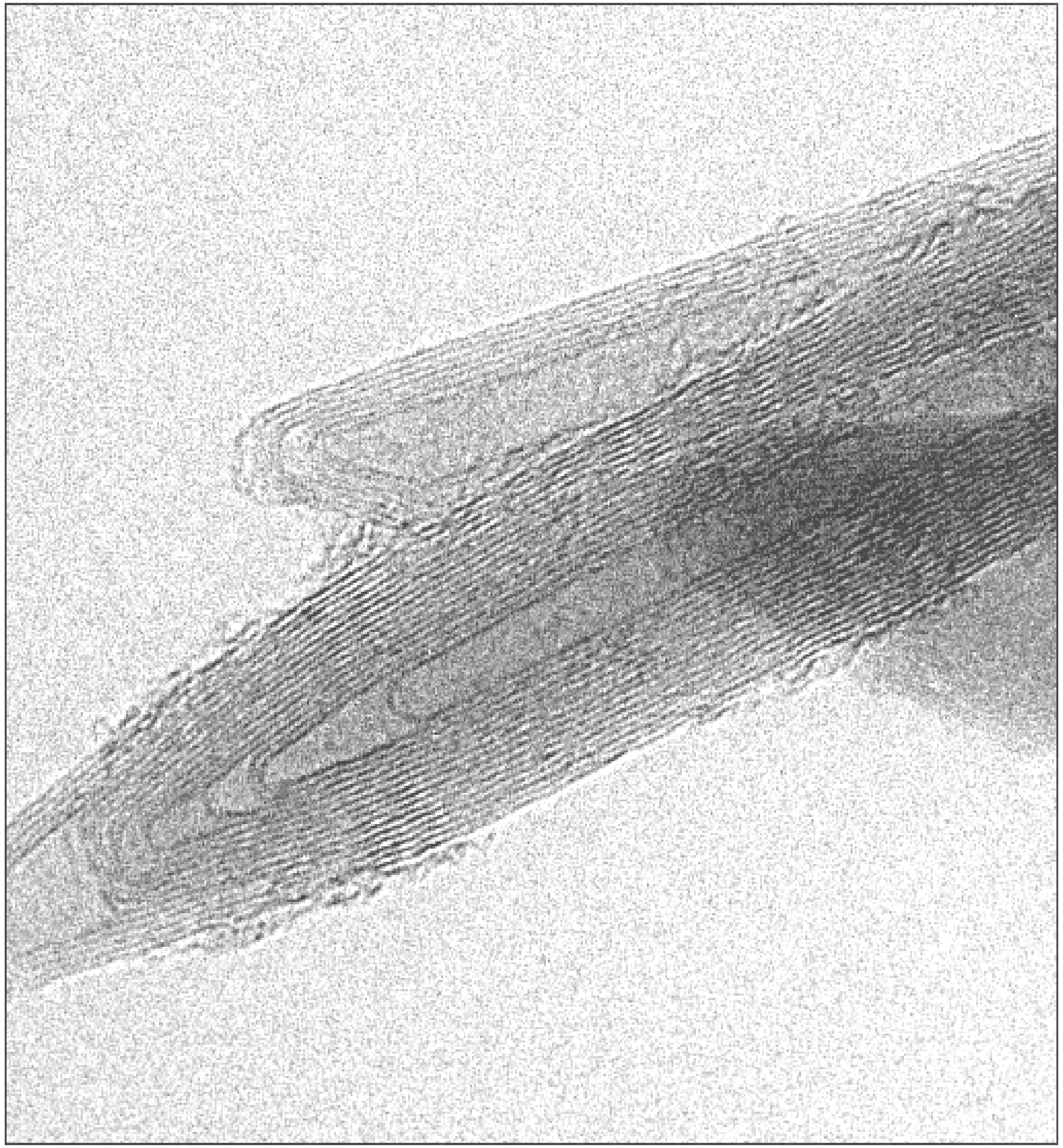
$$\Delta f = -1.2 (C_s \lambda)^{1/2}$$

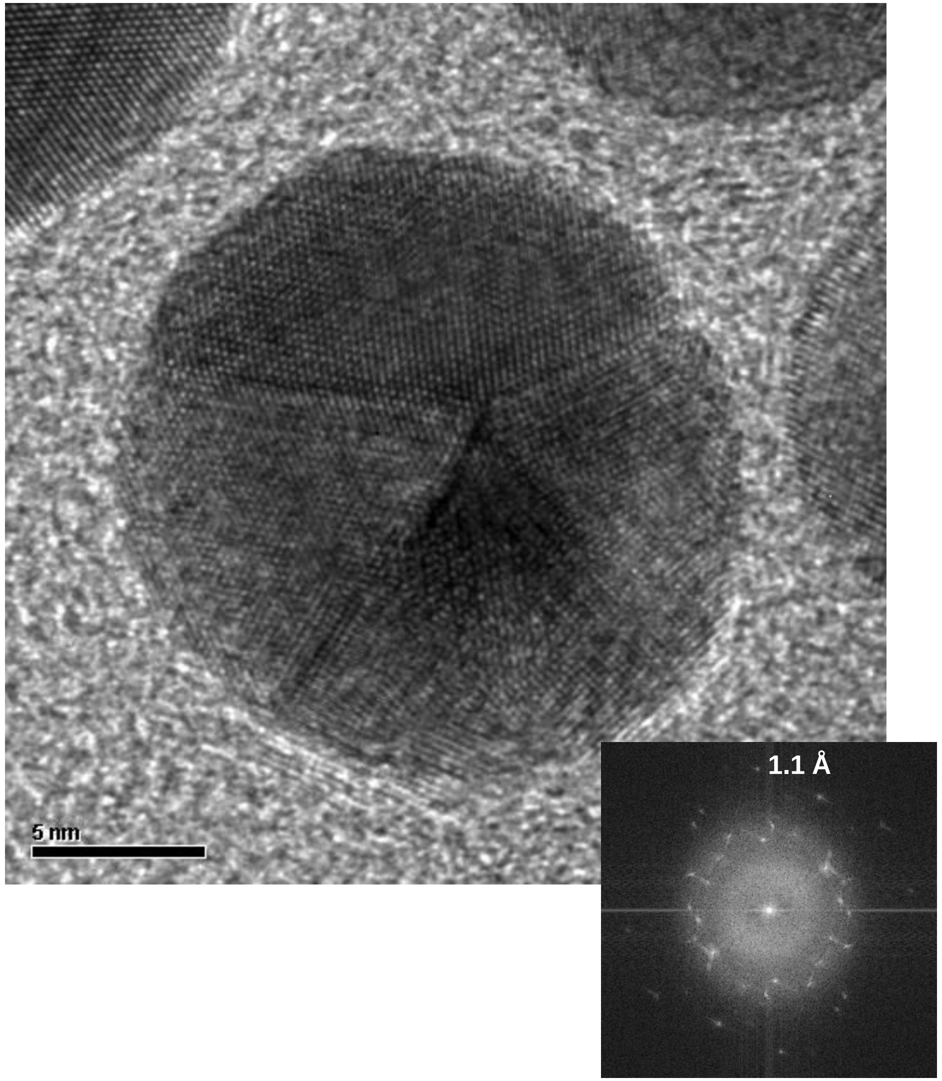
The resolution in this condition results

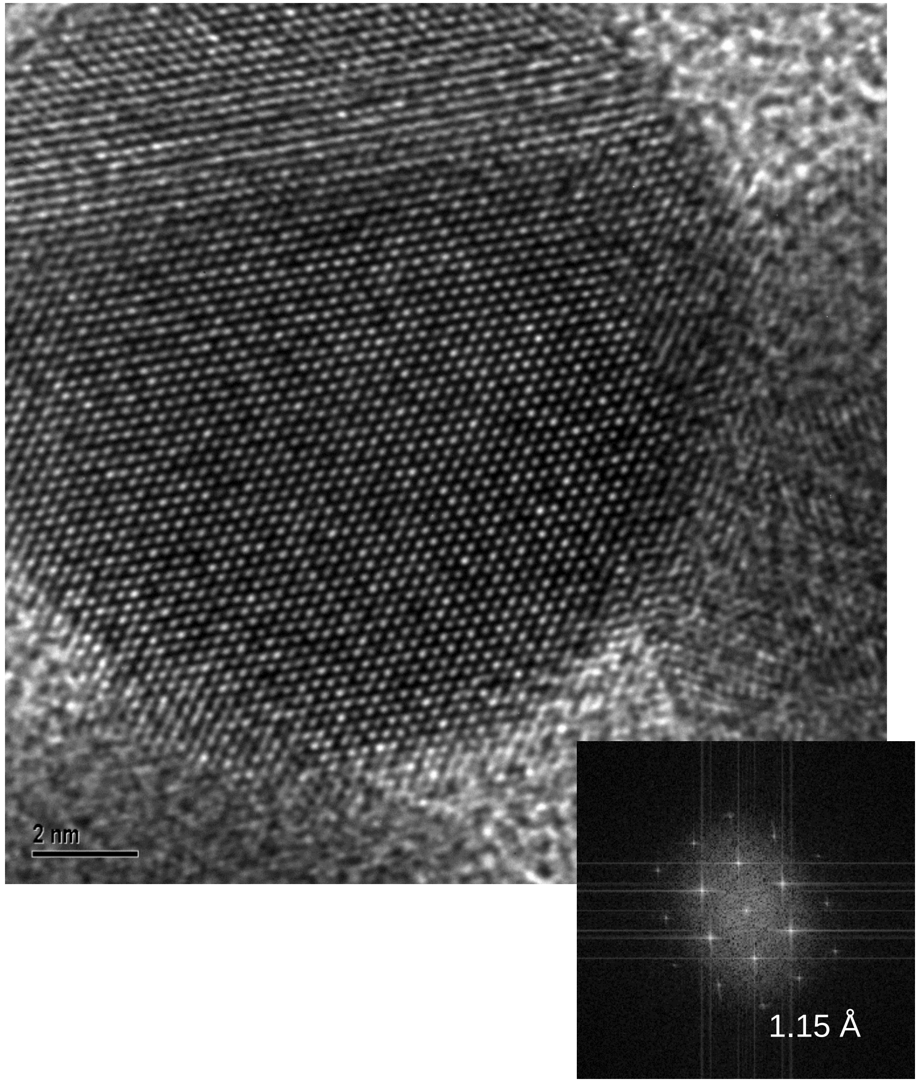
$$R_{Sc} = 0.66\, C_s^{1/4} \lambda^{3/4}$$

Similar to what obtained by the combined analysis of the relative confusion circles.


235TBUFFER




5 nm
1.1 Å


2 nm
1.15 Å

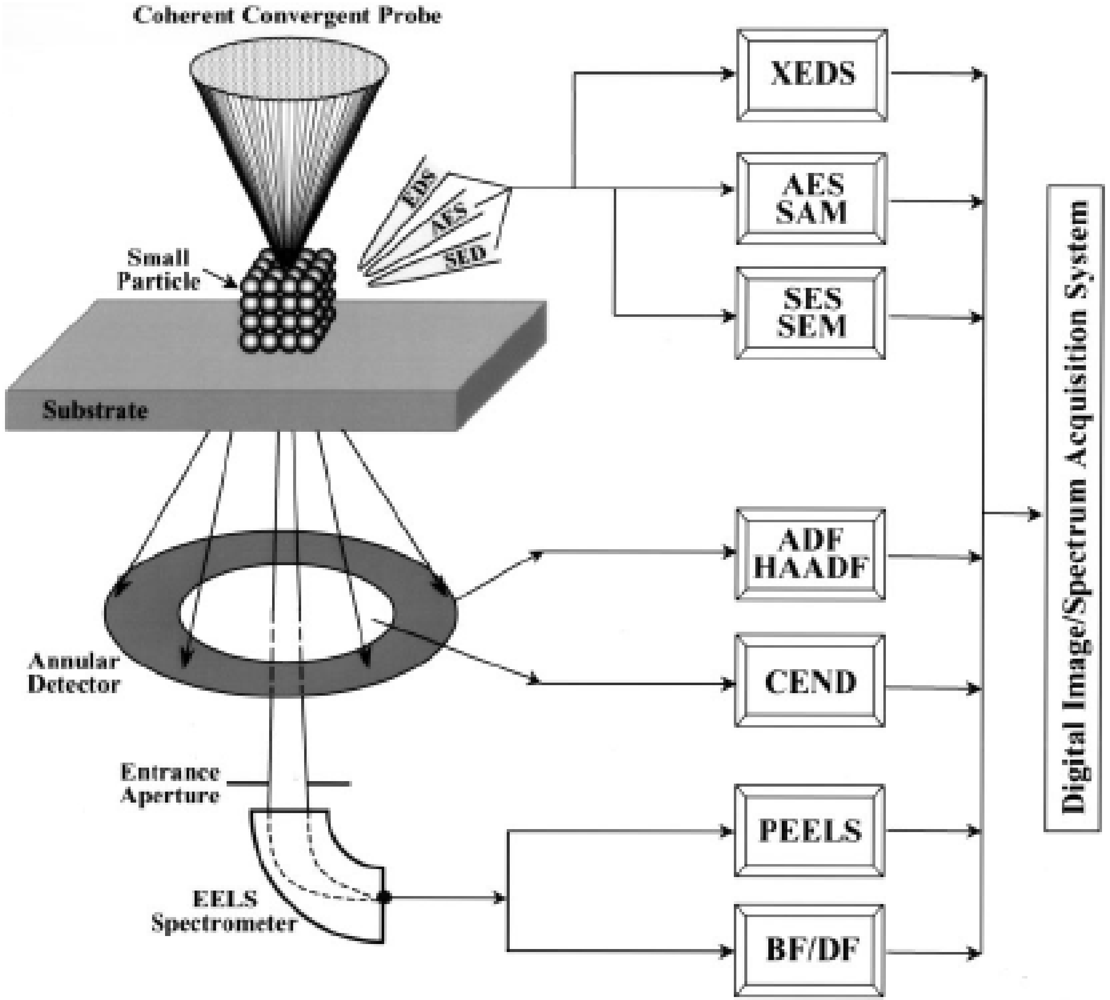
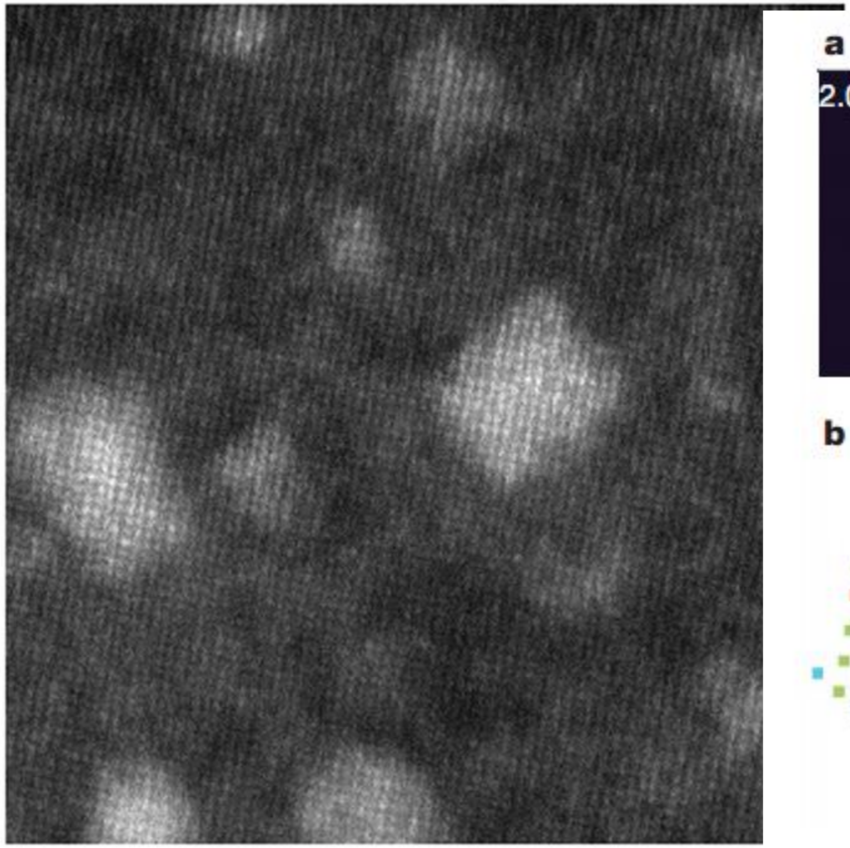
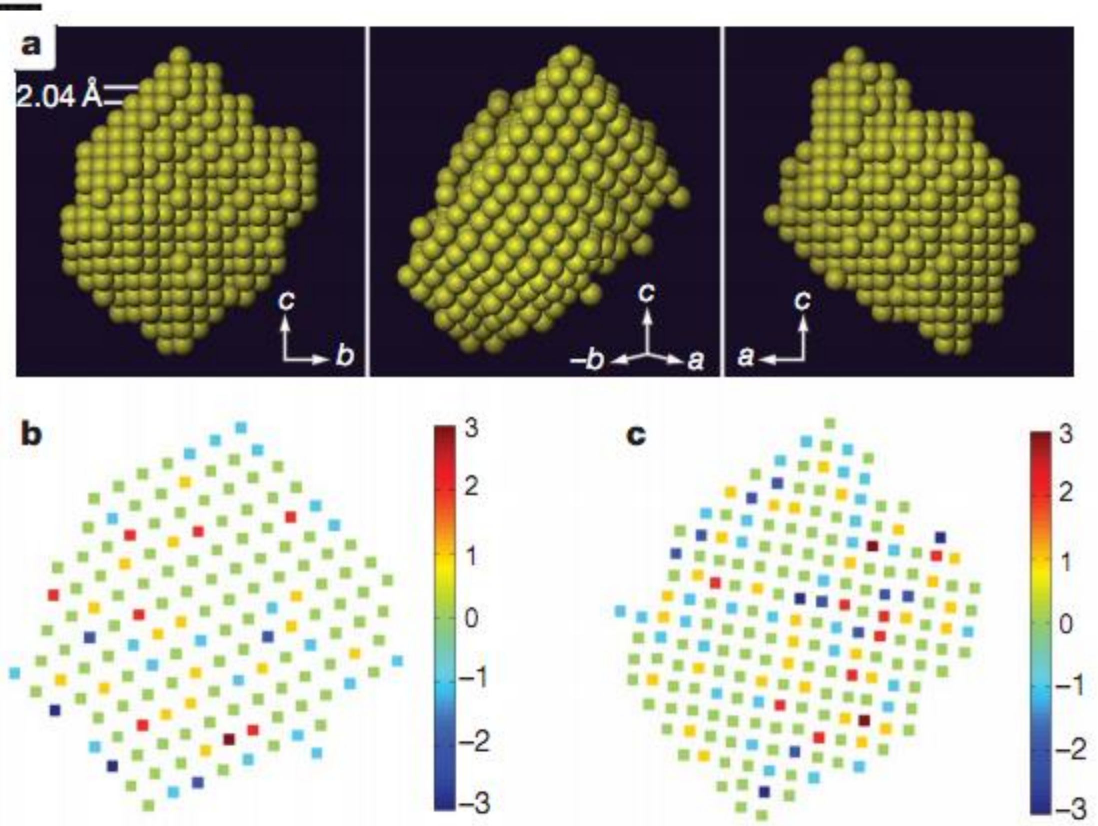


Fig. 1 Schematic diagram illustrates the various signals generated inside a scanning transmission electron microscope that can be used to form high resolution images, nanodiffraction patterns or spectra of the region of interest. X ray energy dispersive spectroscopy (XEDS); Auger electron spectroscopy (AES) and scanning Auger microscopy (SAM); secondary electron spectroscopy (SES) and secondary electron microscopy (SEM); annular dark field (ADF) and high angle annular dark field (HAADF); coherent electron nano diffraction (CEND); parallel electron energy loss spectroscopy (PEELS); bright field (BF) and dark field (DF).

In this case, the contrast is not governed by interference phenomena but it is only related to the nature of the atoms in the explored volume. The contrast is not dependent on the sample orientation and the STEM configuration is particularly suitable for tomography.

3D reconstruction of Ag precipitates in Al





Sandra Van Aert, Kees J. Batenburg, Marta D. Rossell, Rolf Erni& Gustaaf Van Tendeloo
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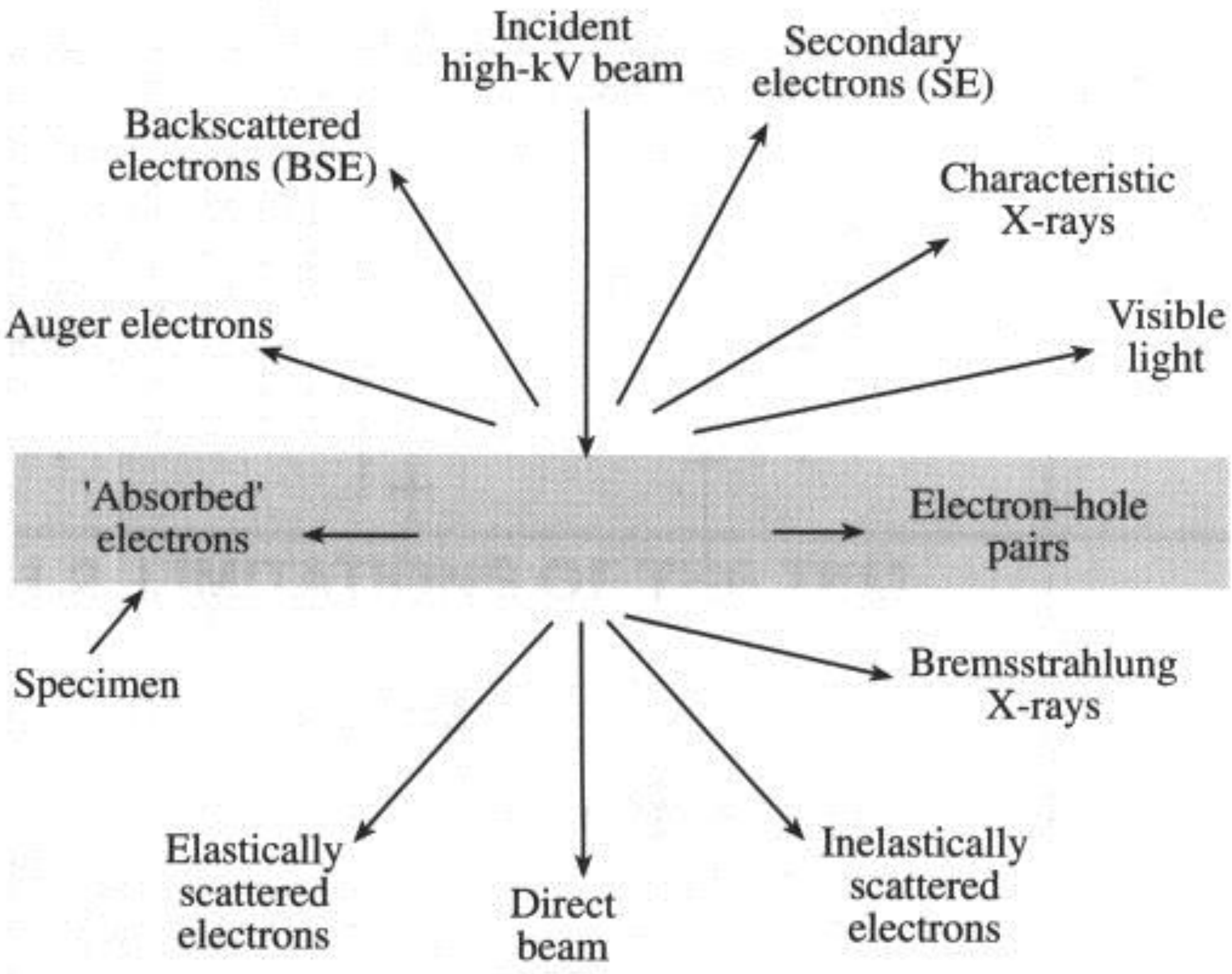
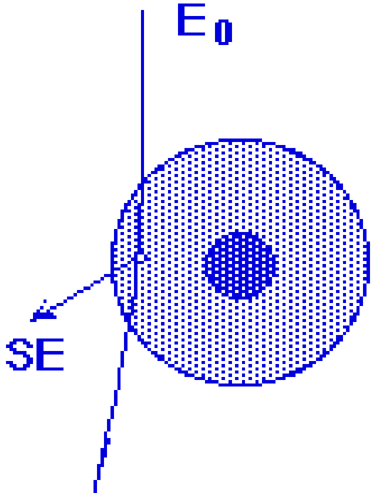
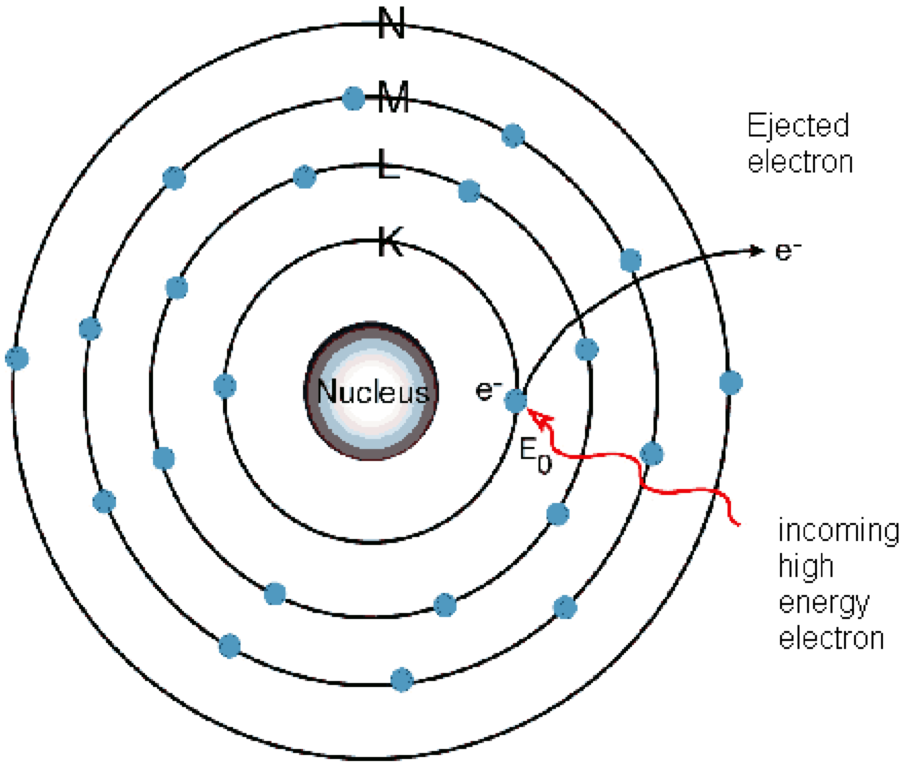
Spectroscopy:

- X-Ray spectroscopy
- Electron energy loss spectroscopy


Incident
high-kV beam
Secondary
electrons (SE)
Backscattered
electrons (BSE)
Characteristic
X-rays
Auger electrons
Visible
light
'Absorbed'
electrons
Electron–hole
pairs
Specimen
Bremsstrahlung
X-rays
Elastically
scattered
electrons
Direct
beam
Inelastically
scattered
electrons

Ionization by an incident electron

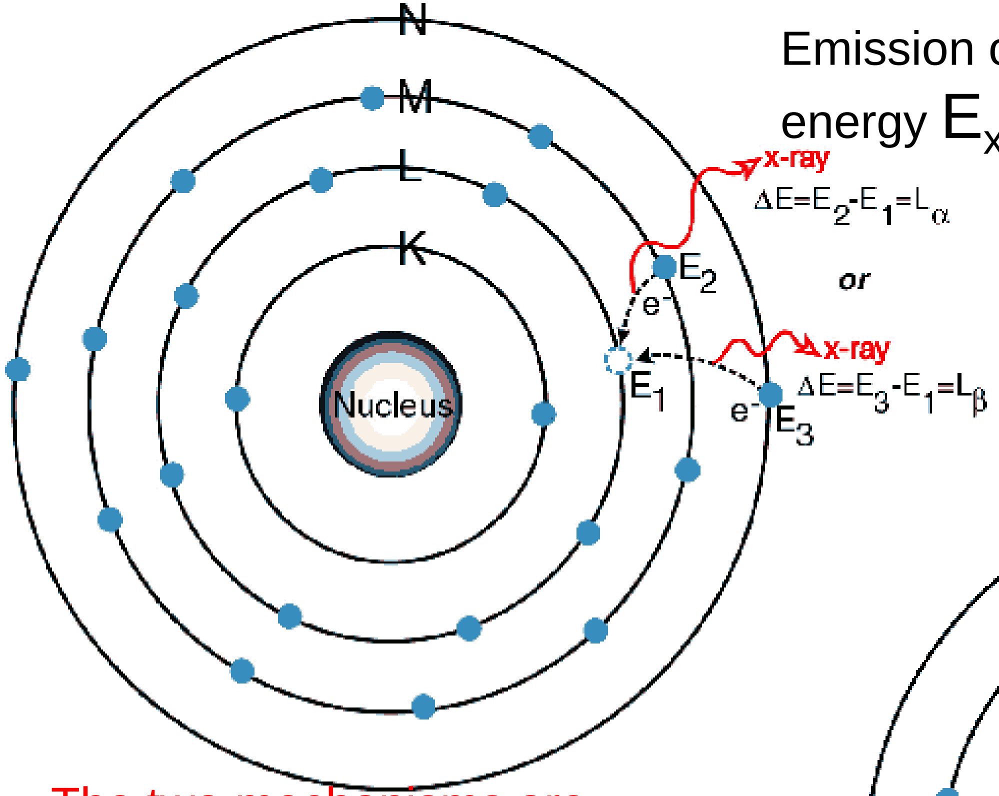




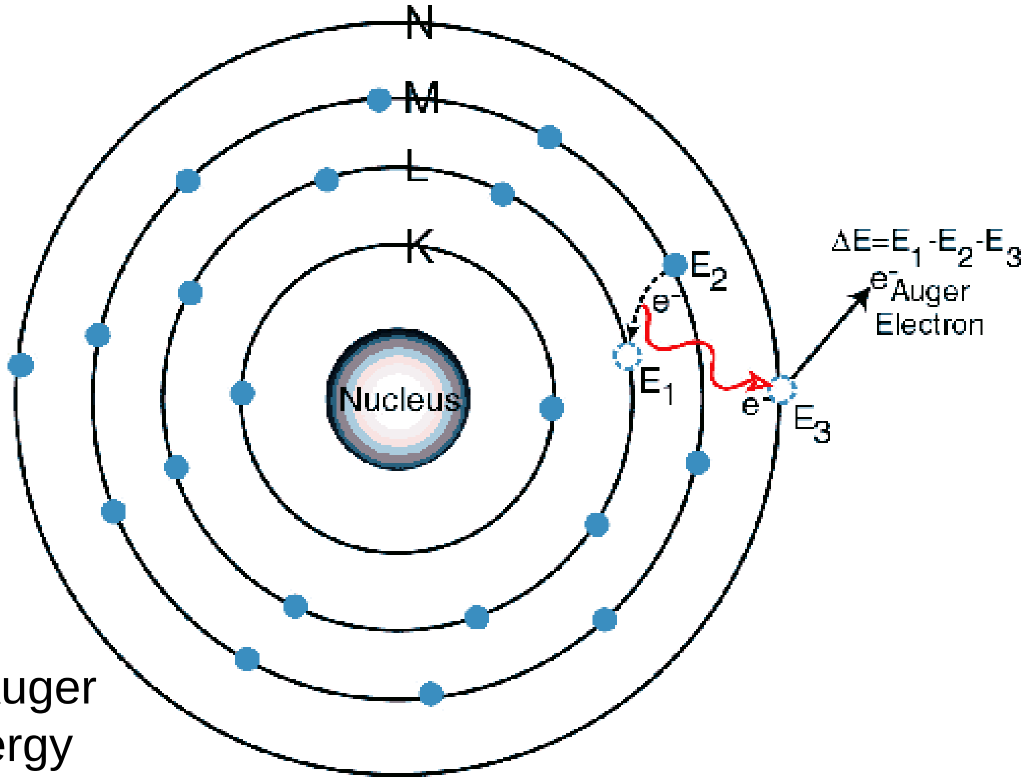
The interaction of a primary electron with an atomic electron can give rise to the ejection of an inner level electron which leaves the atom in a ionized state with an electron vacancy.

Mechanisms of atomic relaxation

Emission of a X-Ray photon with energy $E_x = E_i - E_f$

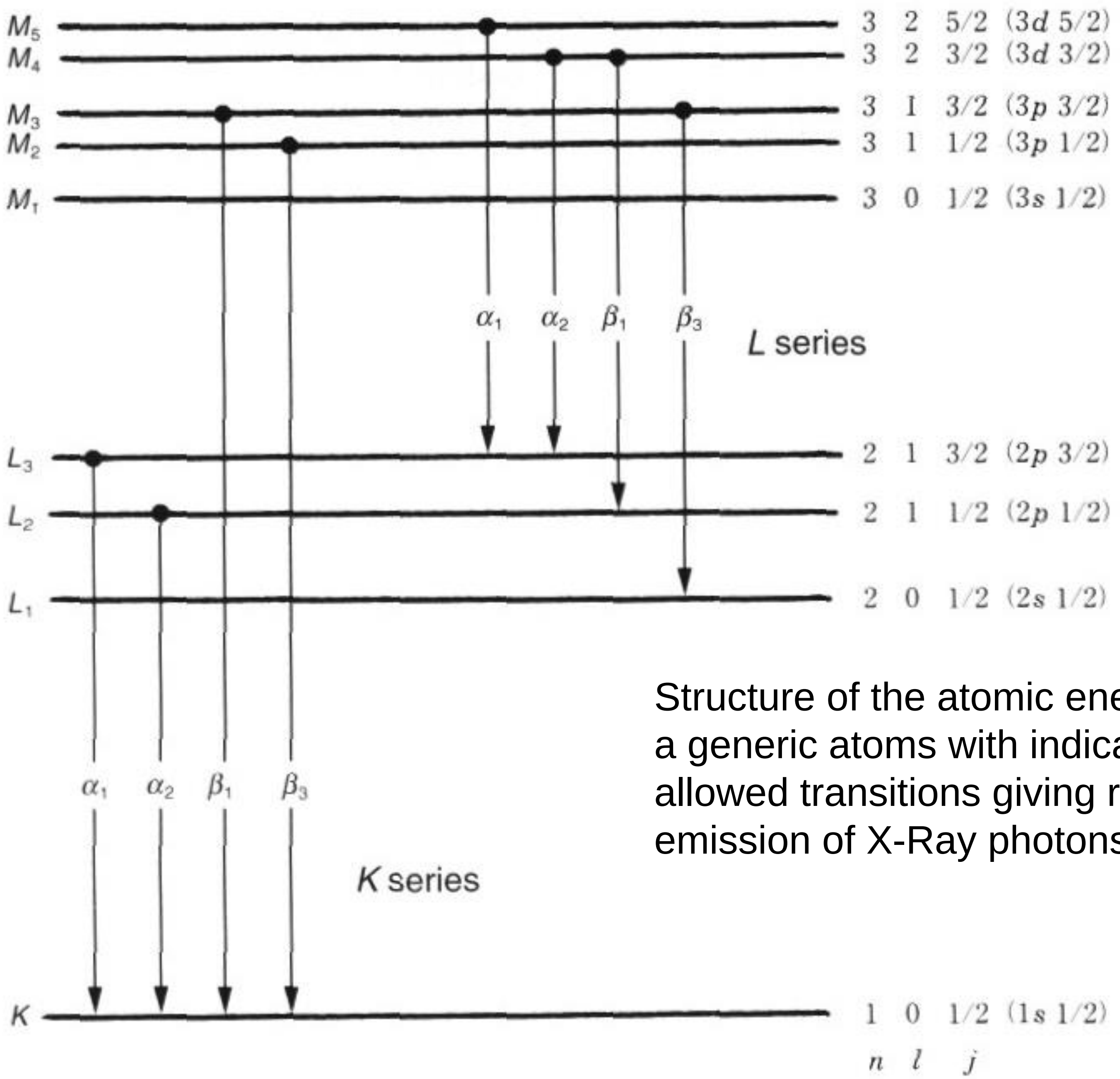


The two mechanisms are alternative and occur with a probability which depends on the atomic number, with the Auger emission dominating the relaxation of light



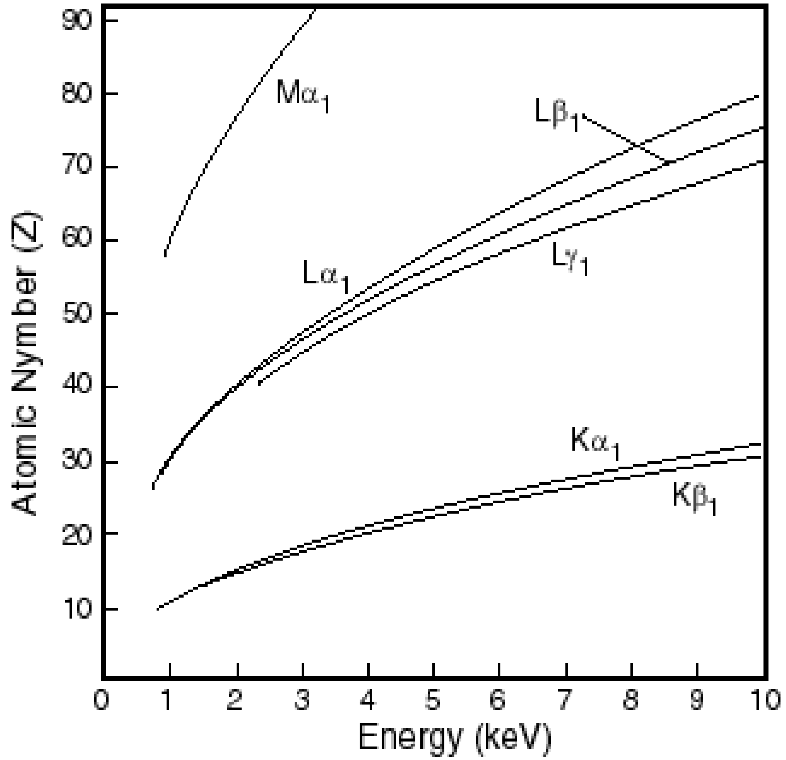
Emission of an Auger electron with energy

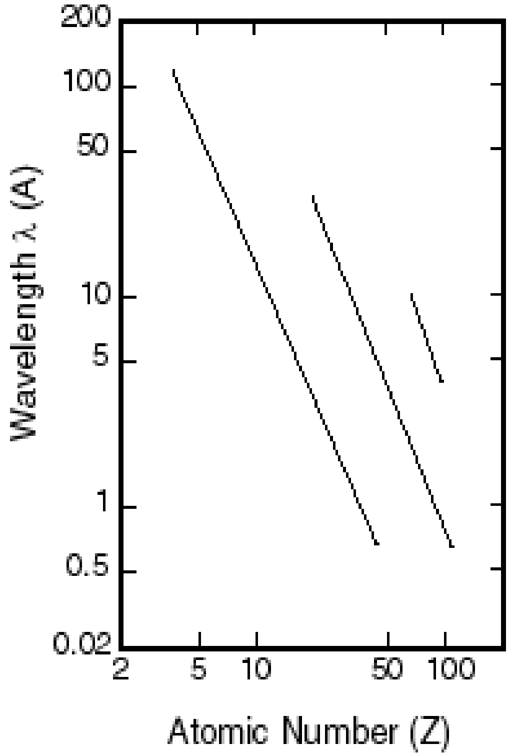
$E_A = E_i - E_f - E_e$



Structure of the atomic energy level of a generic atoms with indication of the allowed transitions giving rise to the emission of X-Ray photons.

Not all the transitions are allowed by the “selection rules”





$$E\ (\text{kev}) = K\,(Z - 1)^2$$

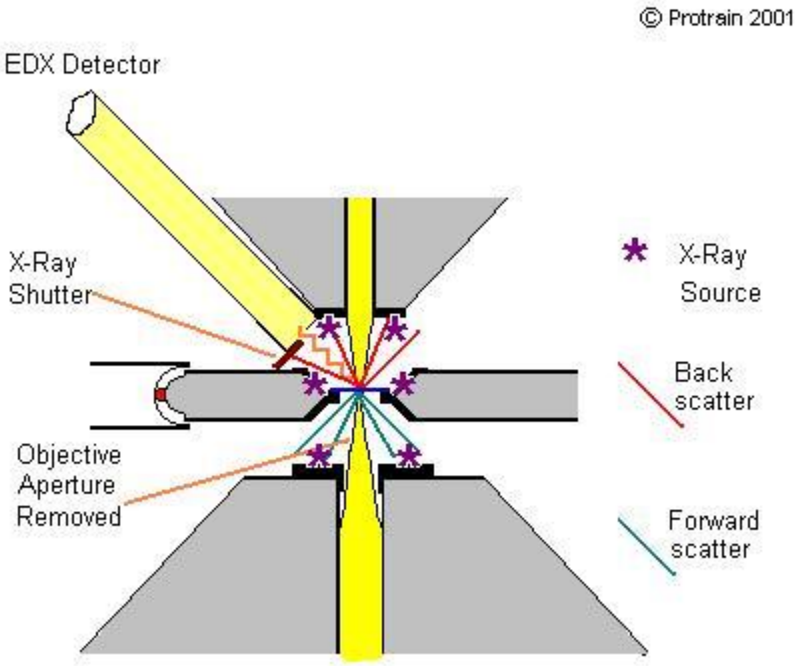
where Z = atomic number and
$K = 1.042 \times 10^{-2}$ for the K-shell
$K = 1.494 \times 10^{-3}$ for the L-shell
and $K = 3.446 \times 10^{-4}$ for the M-shell.

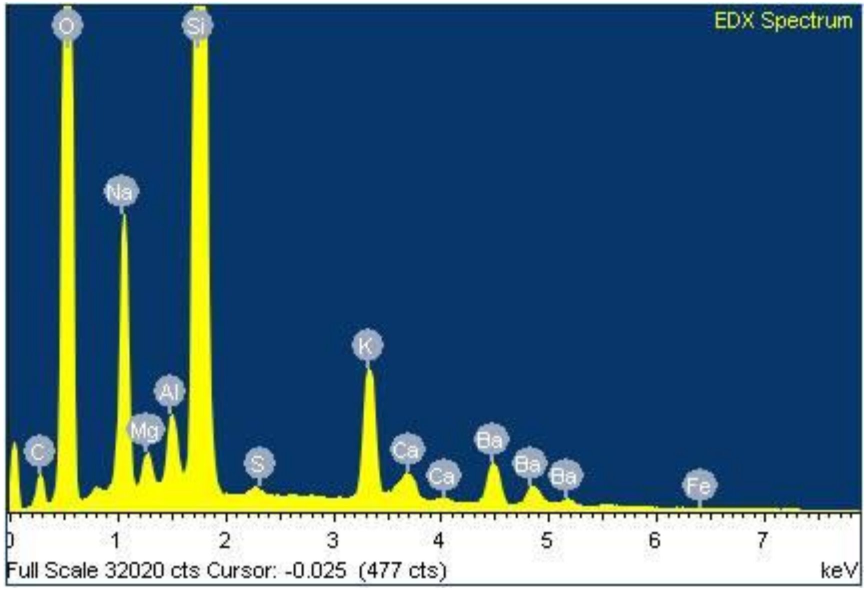
$$E = hc/\lambda$$

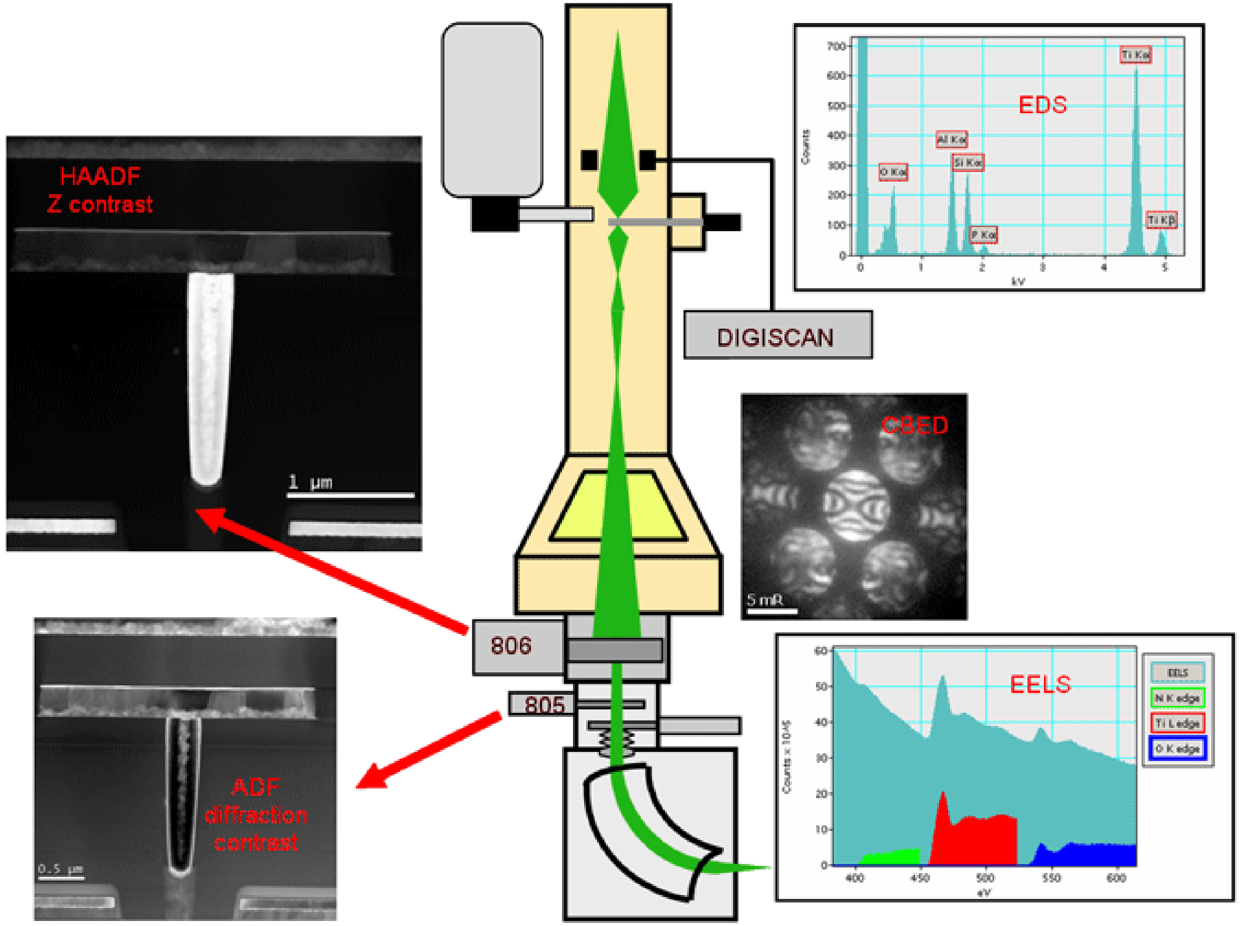
$$\lambda = \frac{K}{(Z - \sigma)^2},$$

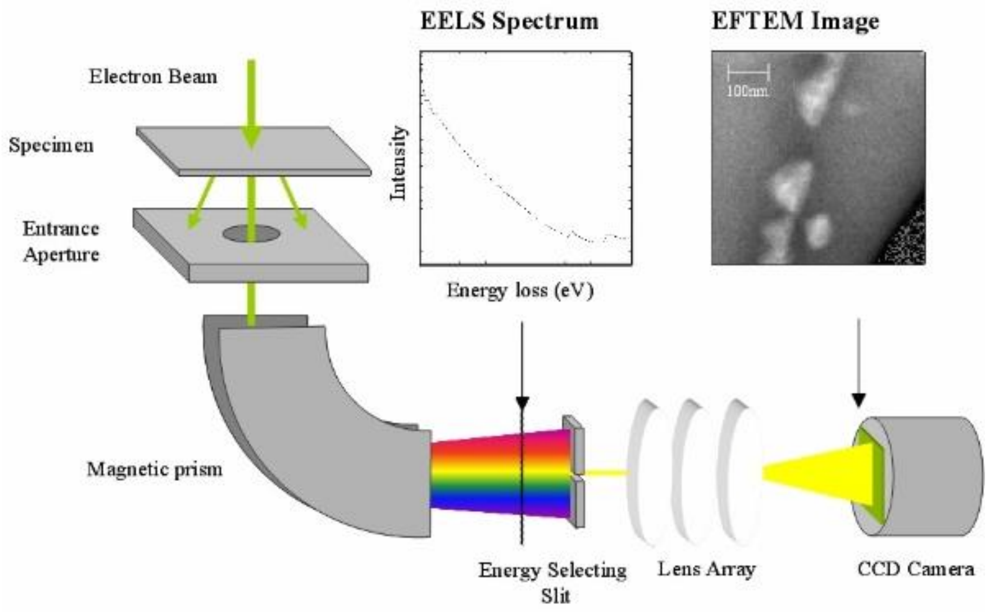
where K and σ are constants for a given spectral line.

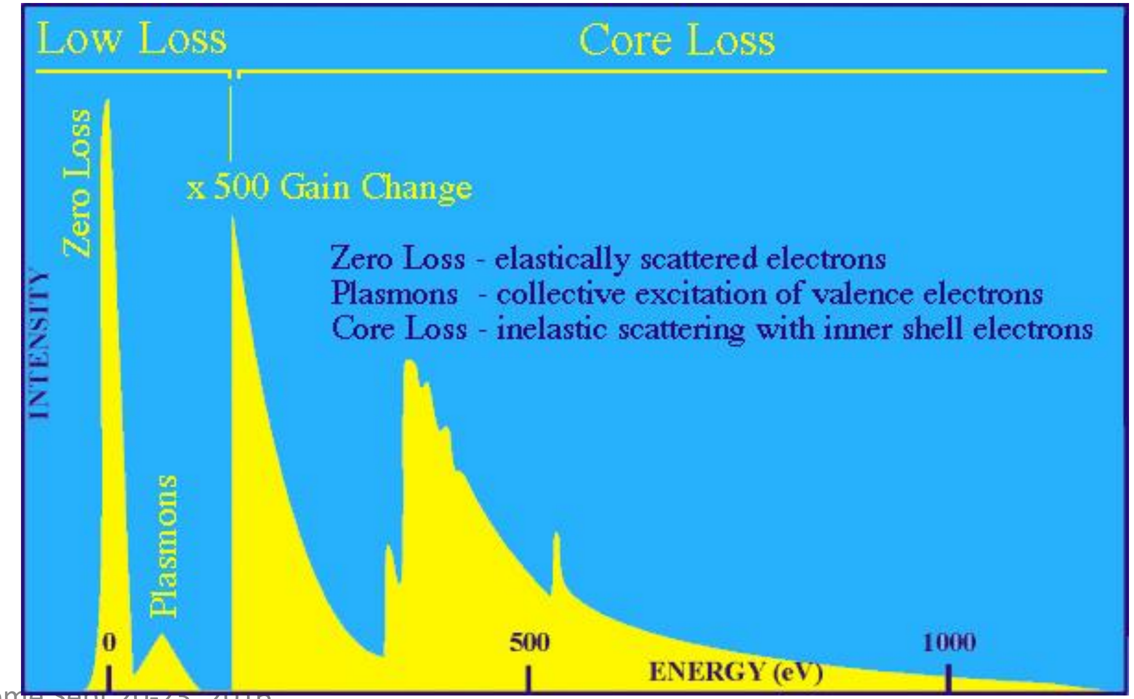
Element	K_{abs}	$K\alpha_1$	$K\beta_1$	$L\text{-}III_{abs}$	$L\alpha_1$	$L\beta_1$	$M\text{-}V_{abs}$	$M\alpha_1$	$M\beta$
9 F	0.687	0.677							
11 Na	1.072	1.041	1.067						
12 Mg	1.305	1.253	1.295						
13 Al	1.559	1.486	1.553						
14 Si	1.838	1.740	1.829						
15 P	2.142	2.013	2.136						
16 S	2.472	2.307	2.464						
17 Cl	2.822	2.622							
18 Ar	3.202	2.957	3.190						
19 K	3.607	3.313	3.589						
20 Ca	4.038	3.691	4.012	0.346	0.341	0.345			
21 Sc	4.496	4.090	4.460	0.403	0.395	0.400			
22 Ti	4.965	4.510	4.931	0.454	0.452	0.458			
23 V	5.465	4.951	5.426	0.513	0.511	0.519			

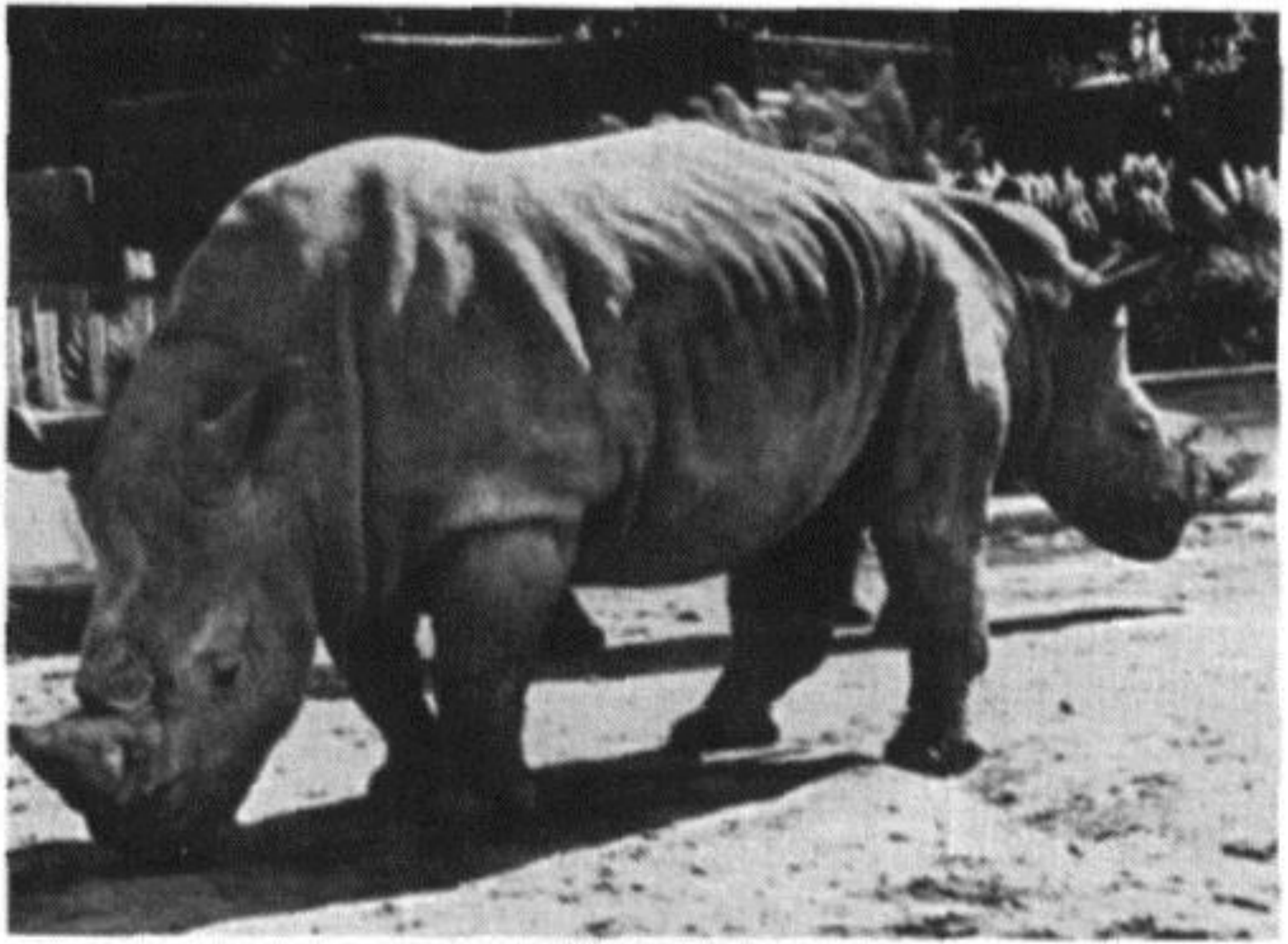

© Protrain 2001
EDX Detector
X-Ray Shutter
Objective Aperture Removed
X-Ray Source
Back scatter
Forward scatter


EDX Spectrum
O
Si
Na
Al
Mg
C
S
K
Ca
Ca
Ba
Ba
Ba
Fe
1
2
3
4
5
6
7
Full Scale 32020 cts Cursor: -0.025 (477 cts)
keV


HAADF
Z contrast
1 μm
ADF
diffraction
contrast
0.5 μm
806
805
DIGISCAN
EDS
O Kα
Al Kα
Si Kα
P Kα
Ti Kα
Ti Kβ
Counts
kV
CBED
5 mR
EELS
N K edge
Ti L edge
O K edge
eV


Electron Beam
Specimen
Entrance
Aperture
Magnetic prism
EELS Spectrum
Intensity
Energy loss (eV)
EFTEM Image
100nm
Energy Selecting
Slit
Lens Array
CCD Camera


Low Loss
Core Loss
Zero Loss
x 500 Gain Change
Zero Loss - elastically scattered electrons
Plasmons - collective excitation of valence electrons
Core Loss - inelastic scattering with inner shell electrons
INTENSITY
Plasmons
0
500
1000
ENERGY (eV)



Thank you

for your attention!