CURSO INTRODUCCIÓN A NANOCIENCIA

HERRAMIENTAS PARA VER EL NANOMUNDO

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Barranquilla, 19 de octubre de 2010

VIEWING NANOWORLD

- Introduction
- Fundamentals
  - * Radiation – Matter Interaction
  - * Force microscopy
- Scanning Electron Microscopy – SEM
- Scanning Probe Microscopy SPM
- Transmission Electron Microscope -TEM
- High Resolution Electron Microscope HRTEM

MICROSCOPY

- Introduction
  - ❖ History
  - ❖ Why microscopy?
  - ❖ Why to use electrons?
  - ❖ Resolution Power

MICROSCOPY

- Why Microscopy?
  - ❖ It is important to see exactly what it is we are doing
  - ❖ As the things get smaller and smaller we need more powerful microscopy
  - ❖ Eventually this will necessitate advances in the physics
  - ❖ material science, physics, semiconductor science, metallurgy, electrochemistry, molecular biology, nanotechnology

The Nobel Prize in Physiology or Medicine 2010
Robert G. Edwards

"for the development of in vitro fertilization"

http://nobelprize.org/nobel_prizes/medicine/laureates/2010/edwards.html

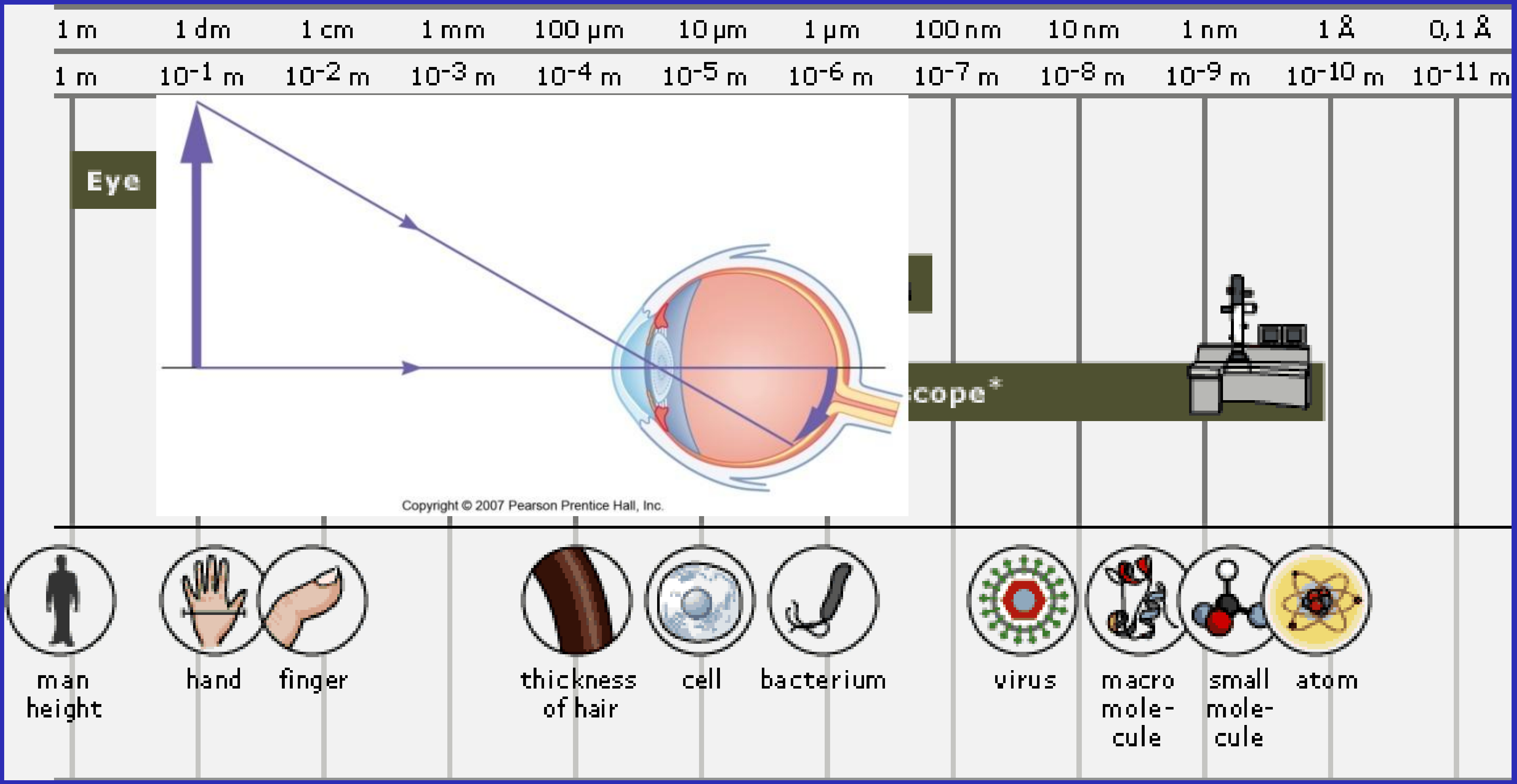
MICROSCOPY HISTORY: PROSPECTS AND PROJECTIONS

The Nobel Prize in Physics 2010: Andre Geim, Konstantin Novoselov

“for groundbreaking experiments regarding the two-dimensional material graphene“

MLA style: "Acrobat Reader". Nobelprize.org. 15 Oct 2010 http://nobelprize.org/tech_support/acrobat.html
http://nobelprize.org/nobel_prizes/physics/laureates/2010/info.html

MICROSCOPY


1 m
1 dm
1 cm
1 mm
100 µm
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10-1 m
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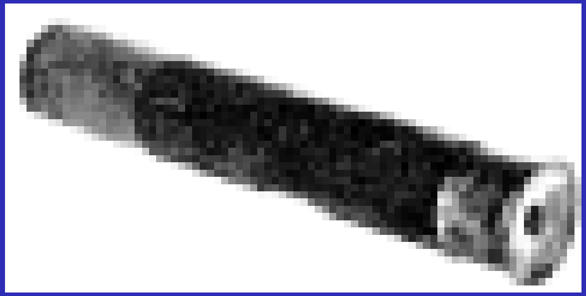
10-3 m
10-4 m
10-5 m
10-6 m
10-7 m
10-8 m
10-9 m
10-10 m
10-11 m
Eye
cope*
Copyright © 2007 Pearson Prentice Hall, Inc.
man height
hand
finger
thickness of hair
cell
bacterium
virus
macro mole-cule
small mole-cule
atom

MICROSCOPY HISTORY

- 17th Century Microscopy:
  - Simple microscopes
  - Compound Microscopes

- 18th Century Microscopy:
  - spermatazoa - red blood cells – bacteria



Janssen's microscope was a tube with two lenses

MICROSCOPY HISTORY

- 19th Century Microscopy:
  - Advances due to improved lenses
  - Cell: basic unit of life

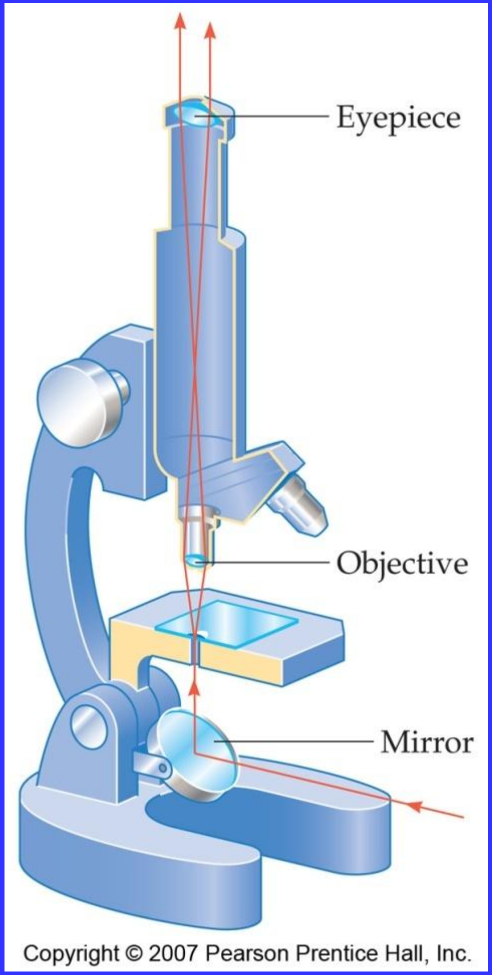
Both plants and animals are composed of cells.

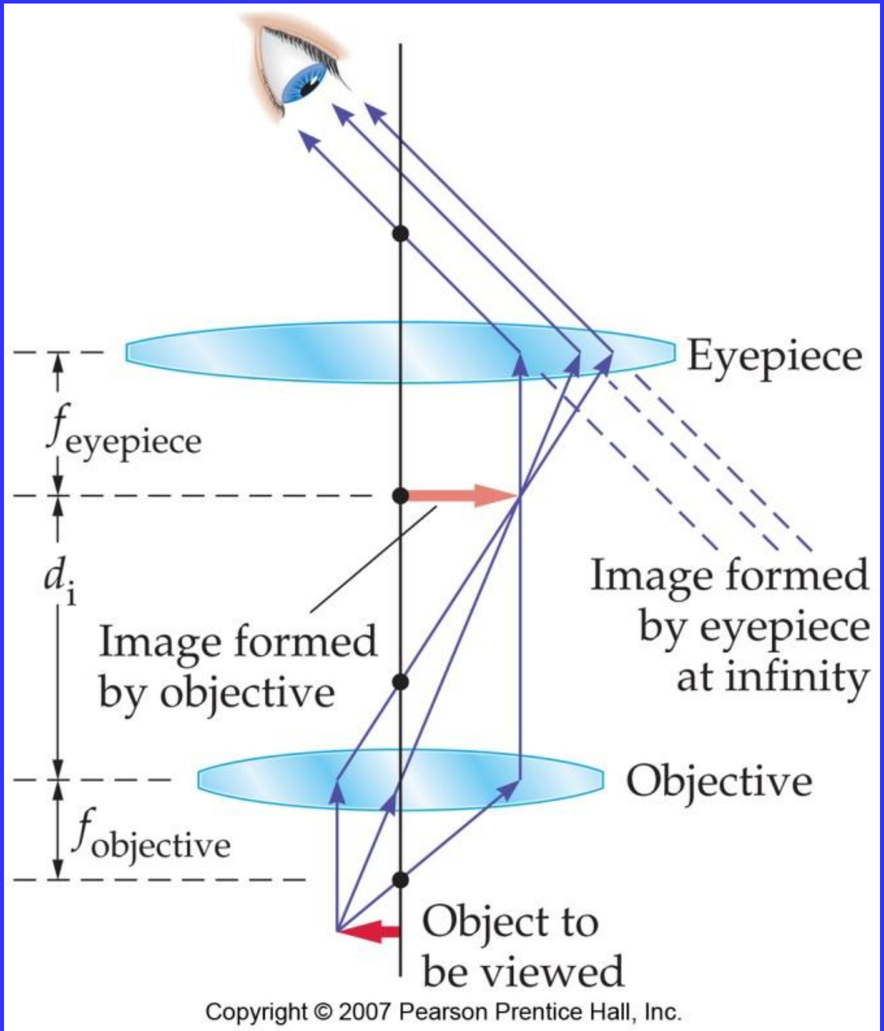
An early compound microscope.

- Röntgen discovered x-ray
- J. J. Thompson in 1897 discovered the electron

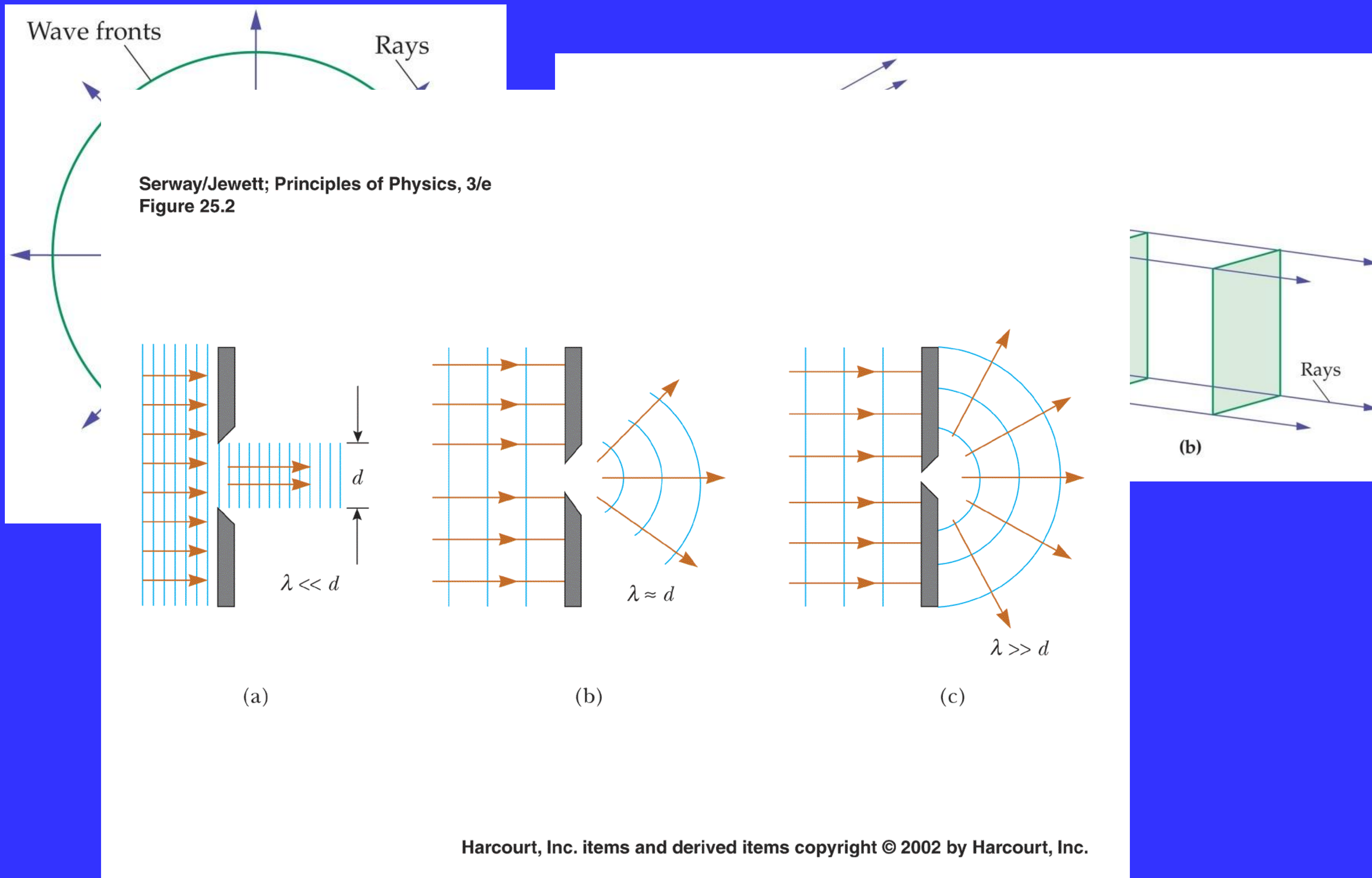
MICROSCOPY: HISTORY

- Microscopy: Image Formation

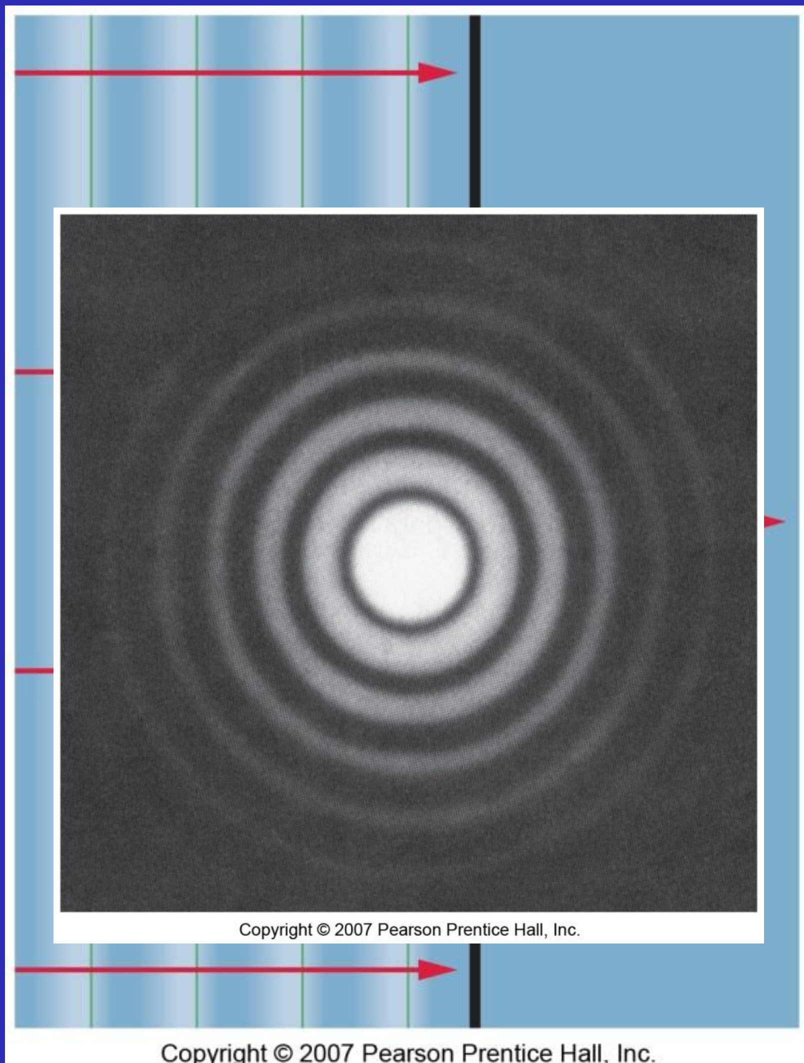


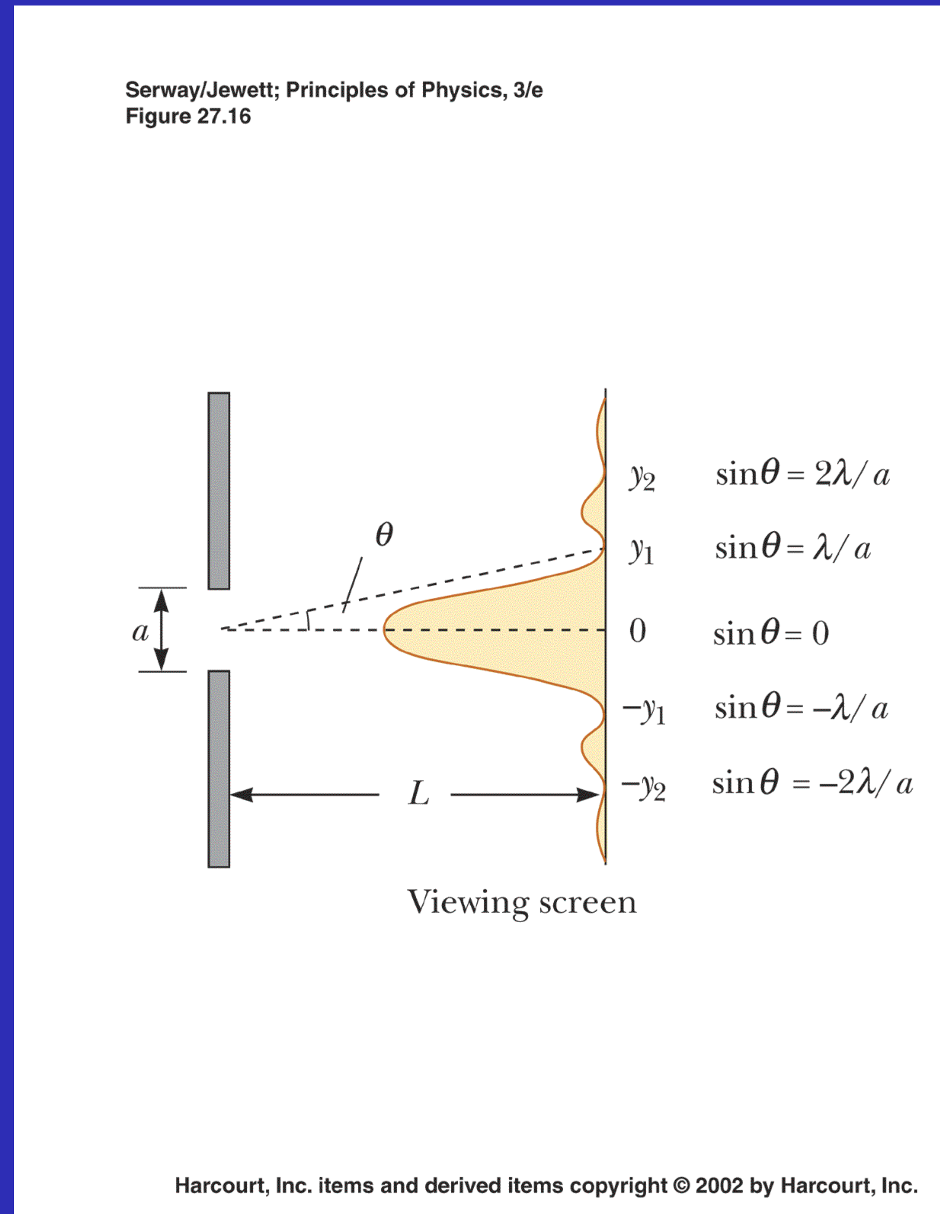


MICROSCOPY HISTORY: WAVE BEHAVIOR

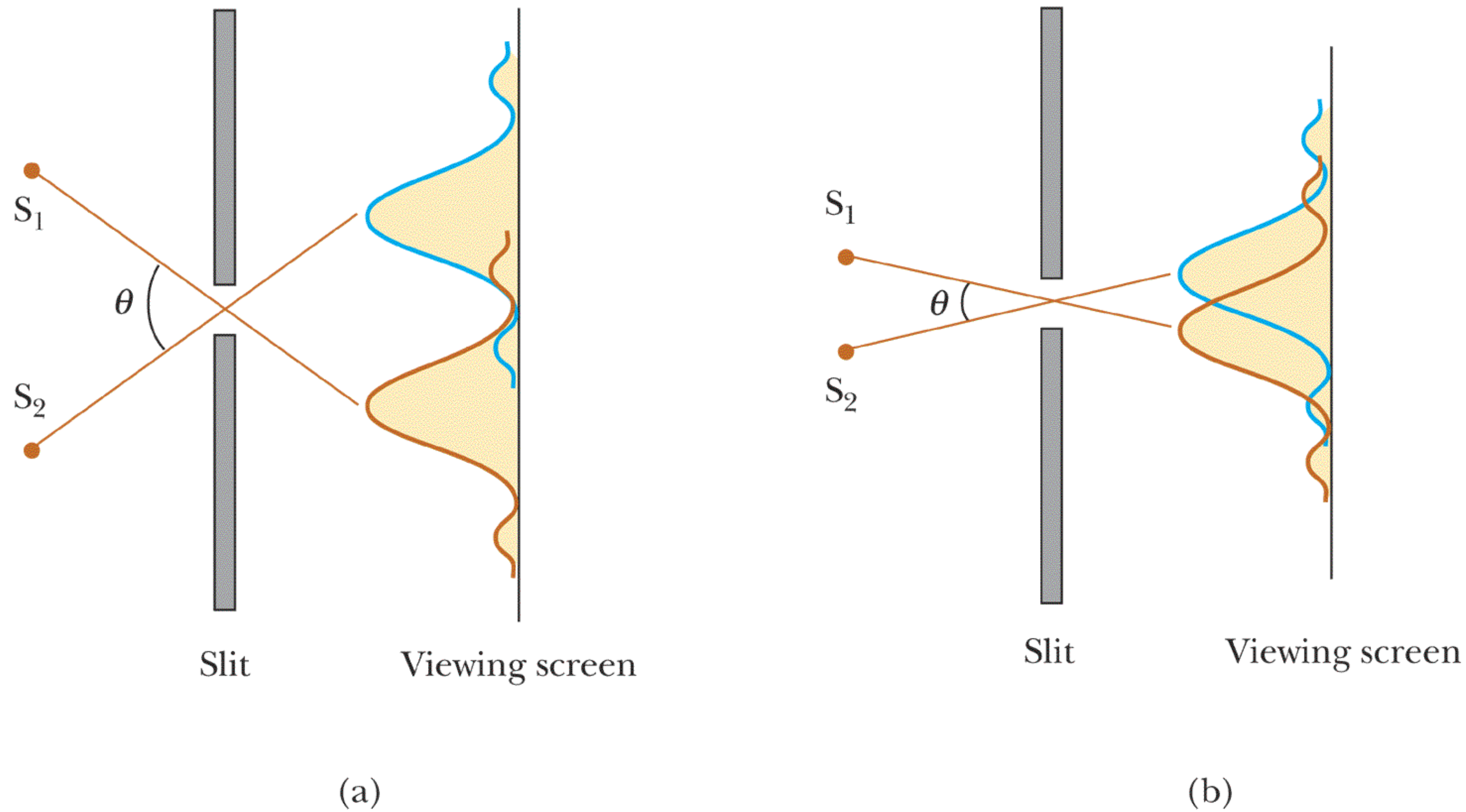


MICROSCOPY HISTORY: DIFRACTION

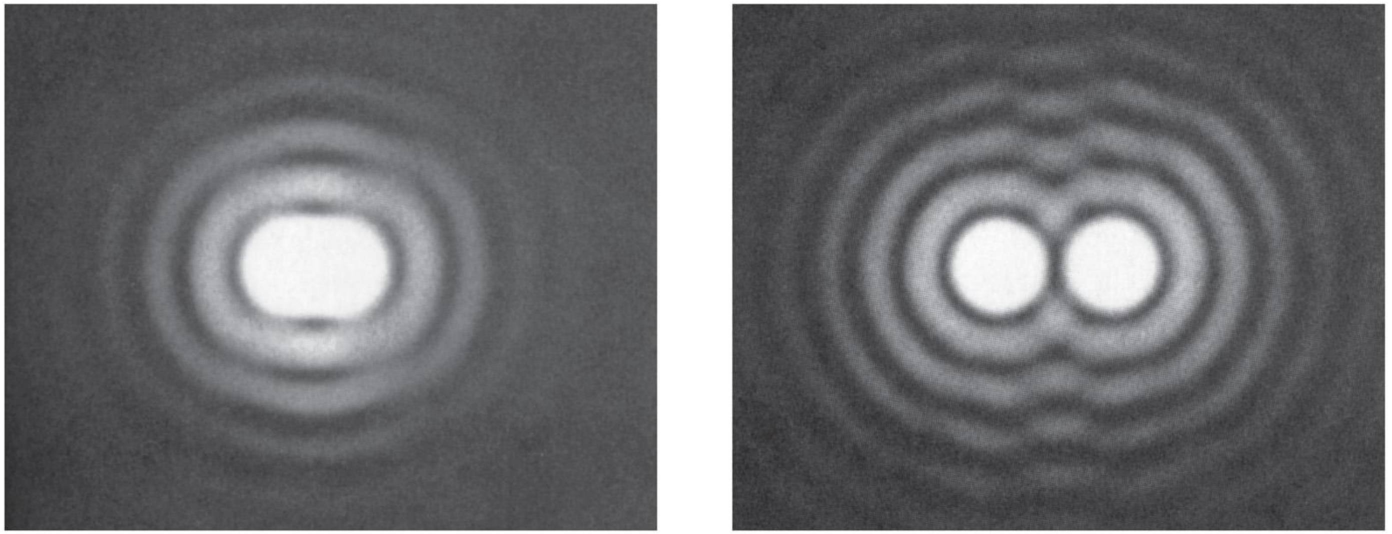




MICROSCOPY: RESOLUTION POWER



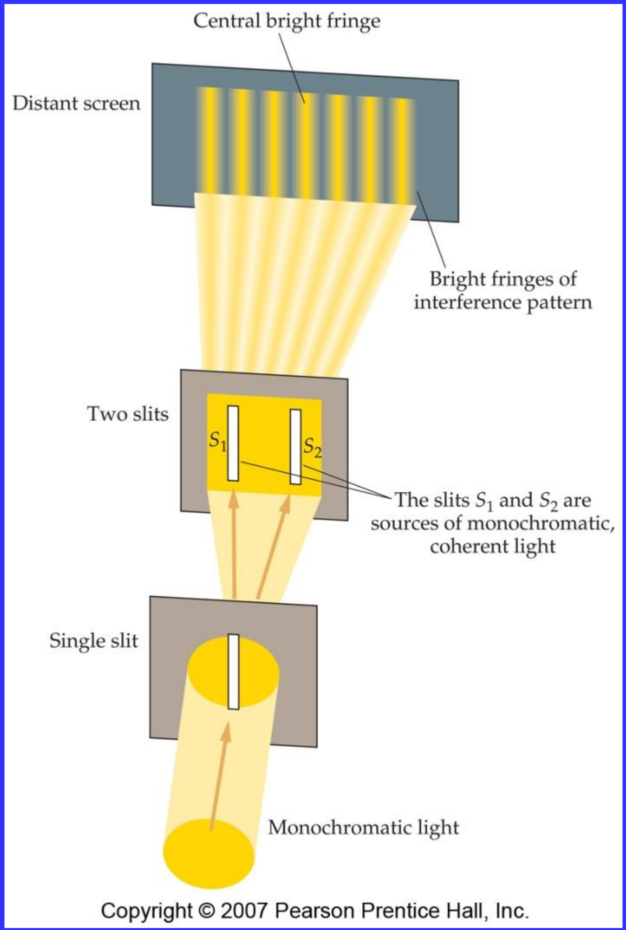
MICROSCOPY: RESOLUTION POWER

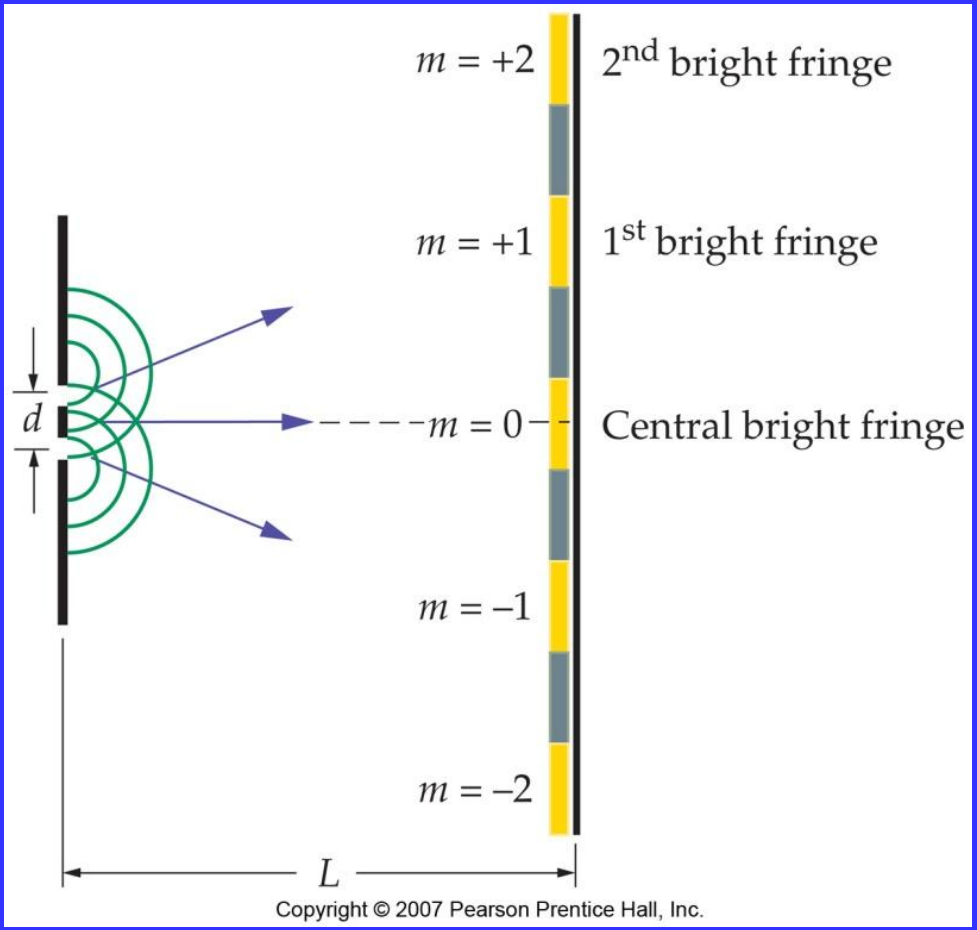


$$\delta = \frac{0.16\lambda}{\mu \sin\beta}$$

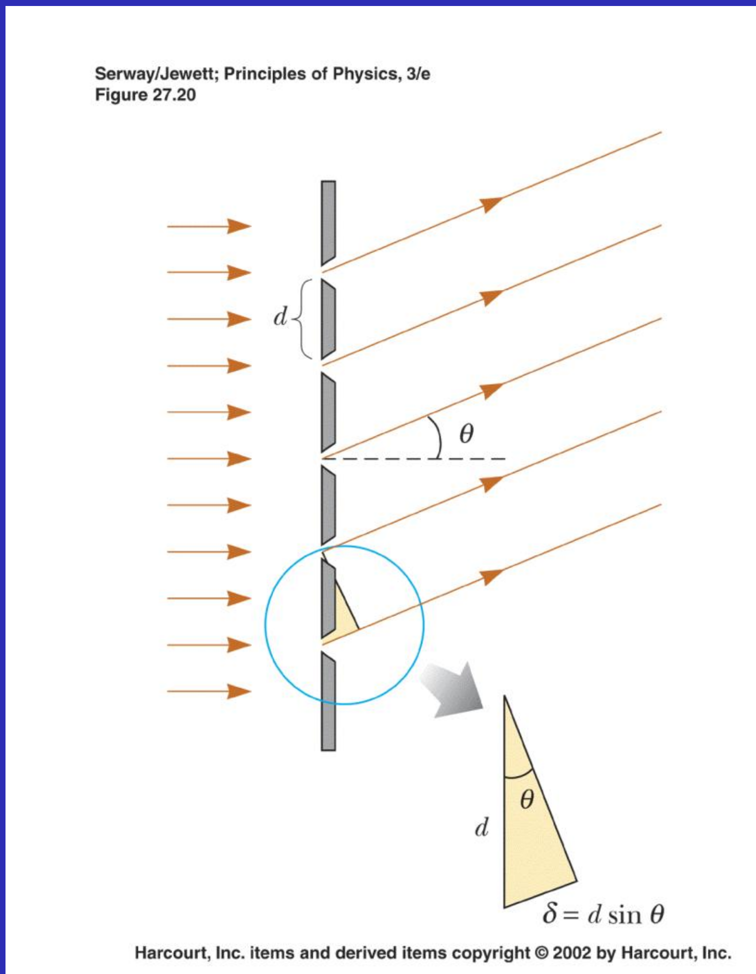
- J.J. Thompson in 1897 discovered the electron
- Röntgen discovered x-ray

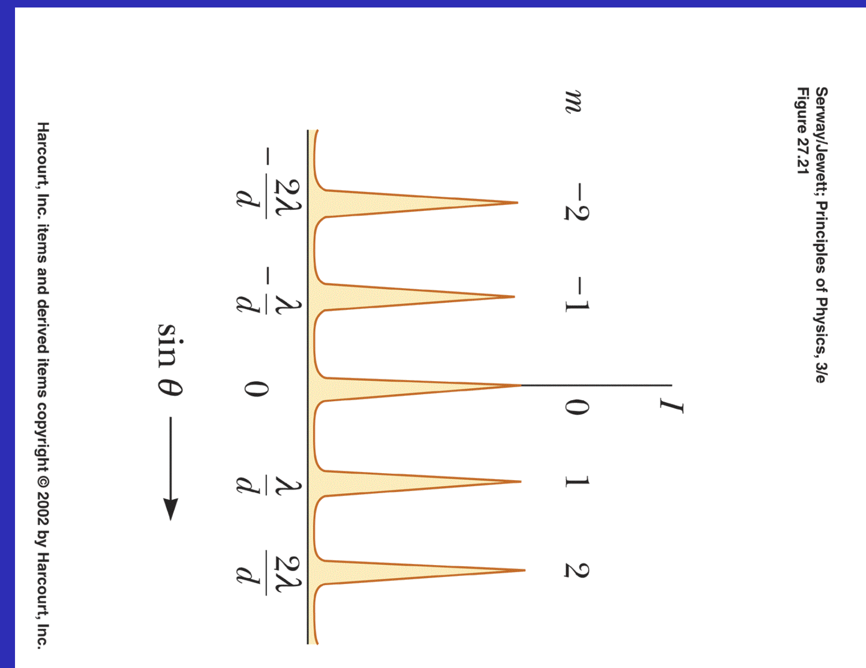
MICROSCOPY: SUPERPOSITION AND INTERFERENCE PATTERN



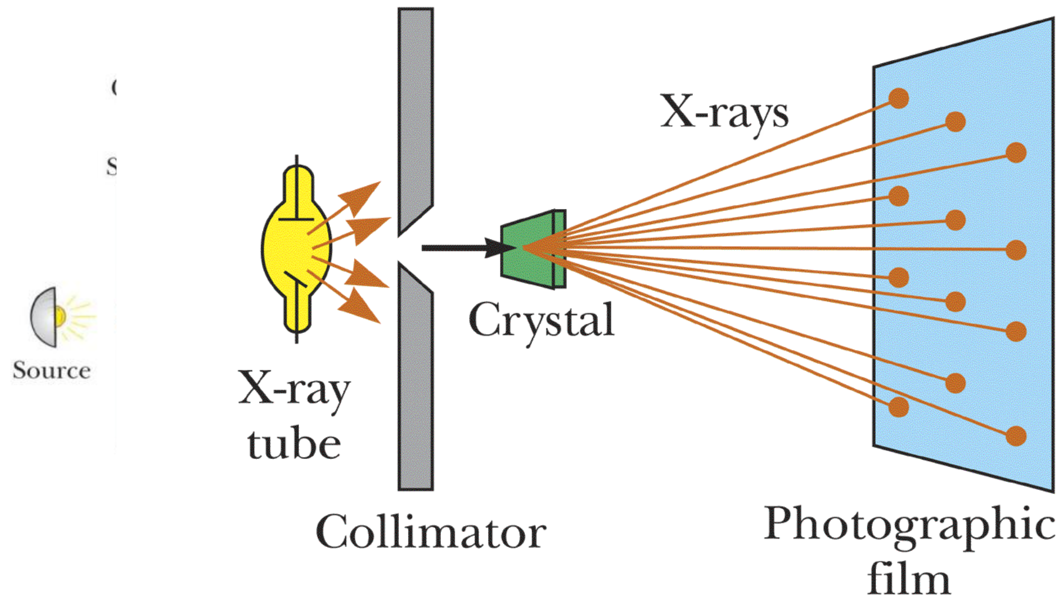


MICROSCOPY: INTERFERENCE PATTERN

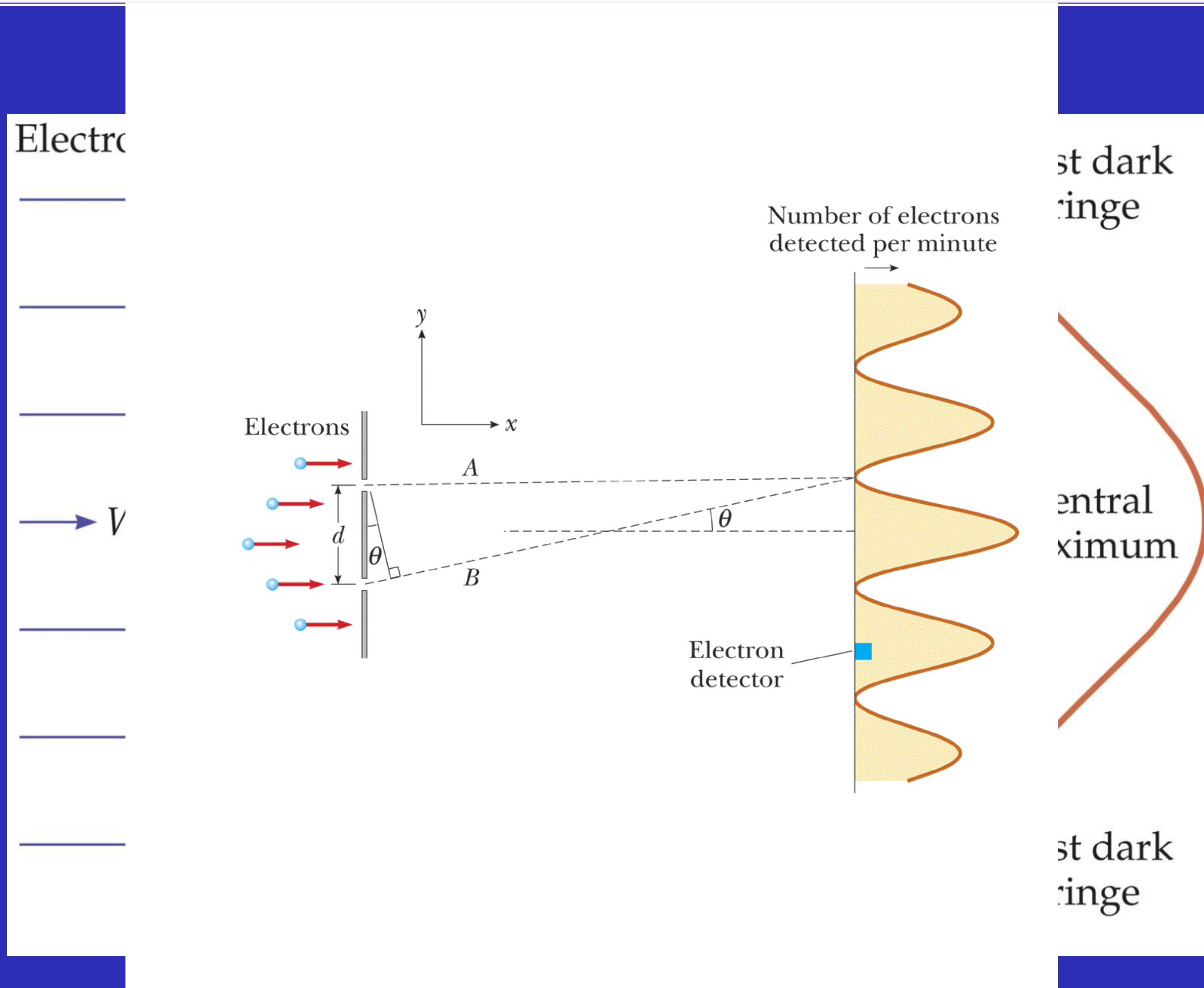




MICROSCOPY: INTERFERENCE AND DIFRACTION



DIFRACTION PATTERN OF ELECTRONS



MICROSCOPY: HISTORY

20th Century Microscopy:

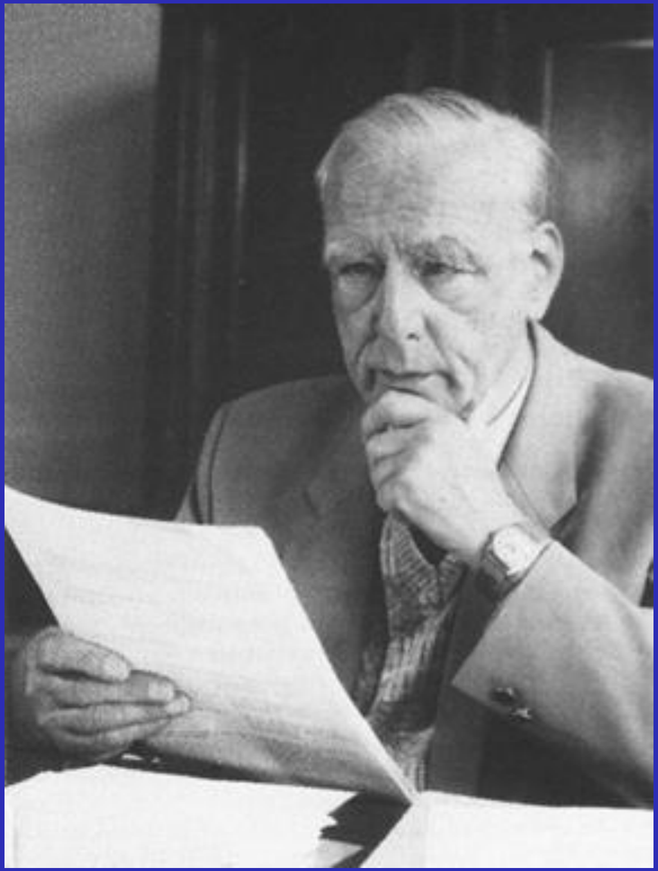
✓ 1925 – *Broglie* – electron has wave-like characteristic

✓ 1926 – Busch – Ruska Magnetic or electric fields act as lenses for electrons

✓ 1927 – *Davison, Germer, Thomson, Reid*: **experimental electron diffraction: electron microscope idea!**

✓ 1931: Knoll - Ruska – First Transmission Electron Microscope (TEM)

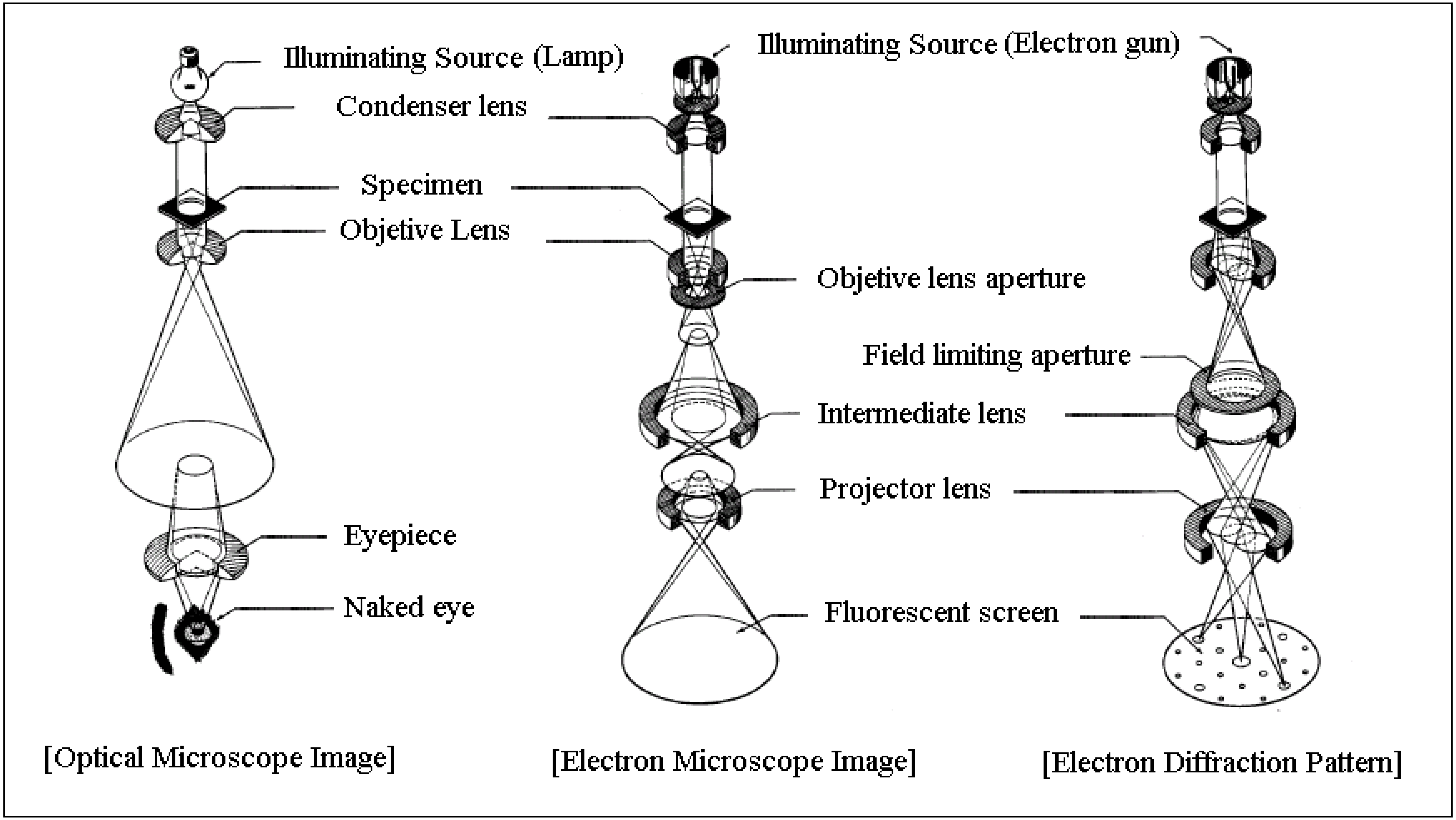
✓ *1934: surpassed resolution limit of the light microscope*



Max Rushka in his later years.

LIGHT MICROSCOPE – ELECTRON MICROSCOPE

Comparison of Image Formation



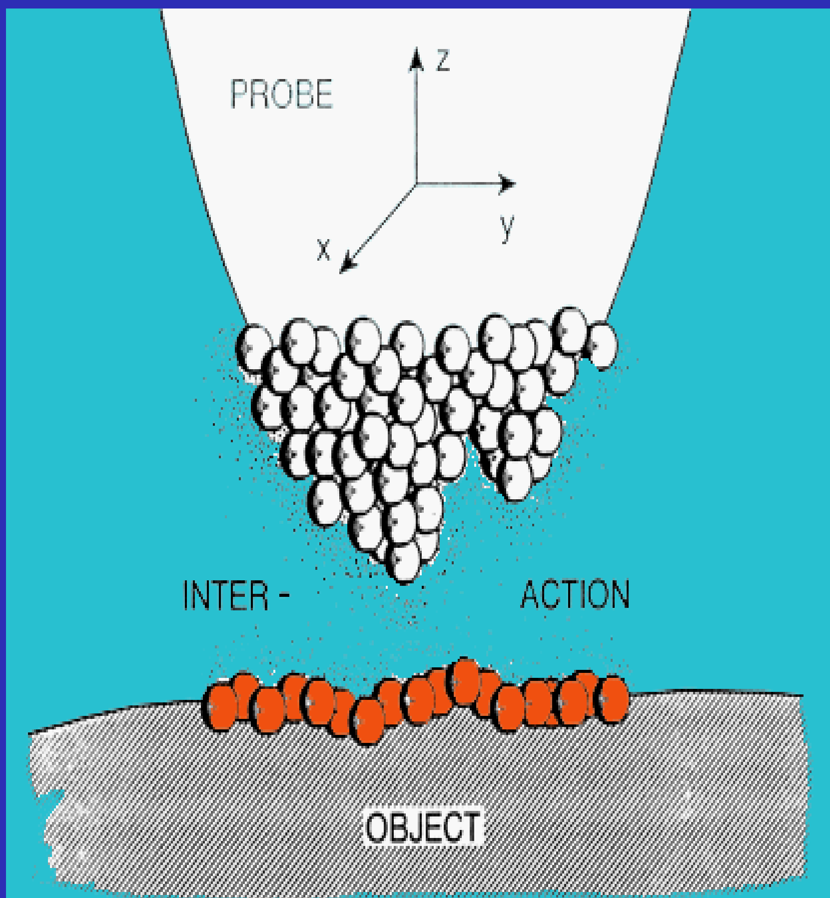
ELECTRON MICROSCOPY: HISTORY (2)

- 20th Century
- ✓ 1935 - Knoll - concept of a SEM
- ✓ 1938 – STEM
- ✓ 1940 - 1945: 10 nm - 2.8 – 1.0 nm resolution in TEM
- ✓ 1942 – first surface solid specimen SEM image

Figure considered unexciting!

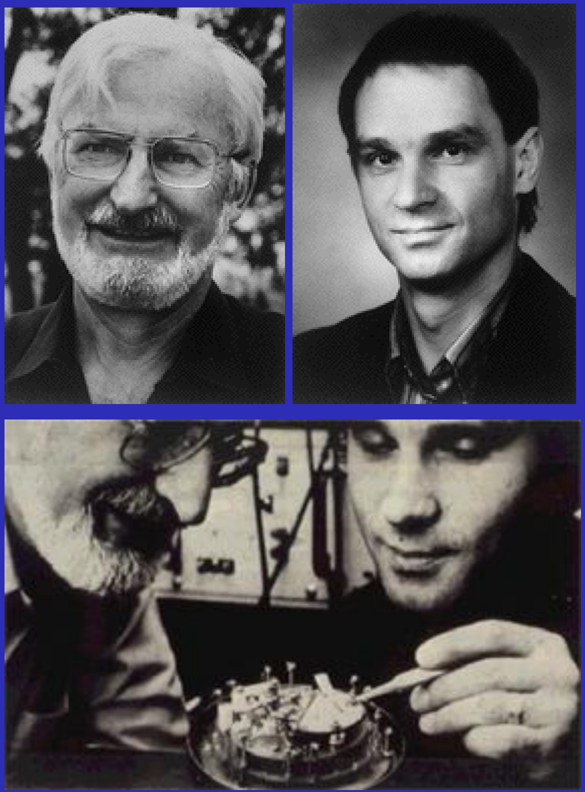
Scanning Probe Microscopes

Use the sense of touch to create images



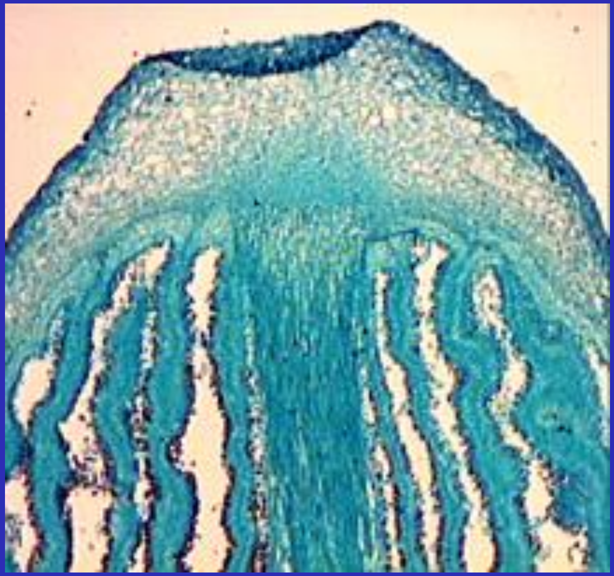
MICROSCOPY: HISTORY

- **20th Century Microscopy:**
  - ✓ 1982: Heinrich Rohrer and Gerd Binnig in Scanning Probe Microscope (SPM) extends the "sense of touch" - Scanning Tunneling Microscope (STM)
  - ✓ 1985 Atomic Force Microscope (AFM)
  - ✓ 1986 Nobel Prize Rohrer – Binning – Rushka
- **Today:** ***EM – SPM most efficient and versatile tools for the characterization of materials***



Gerd Binnig (left) and Heinrich Rohrer

Views by different microscopes

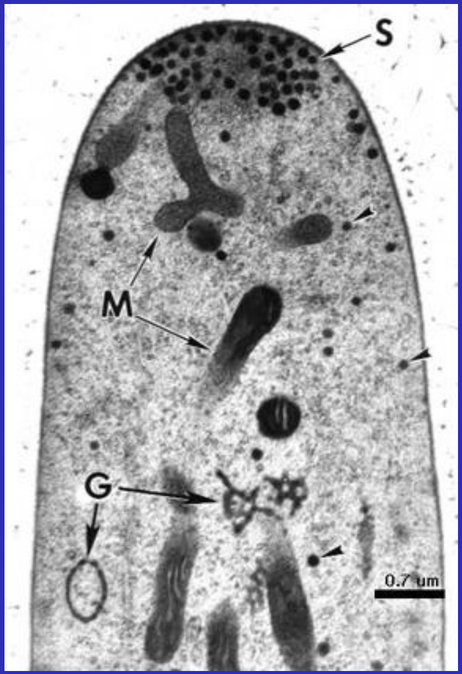


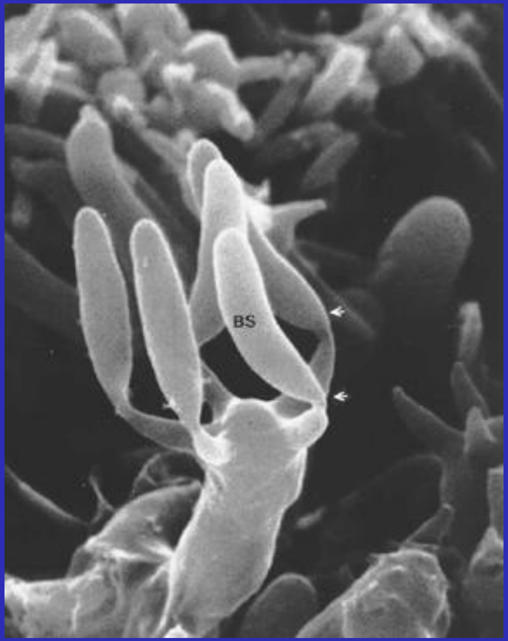
Mushrooms with out any magnification 1:1

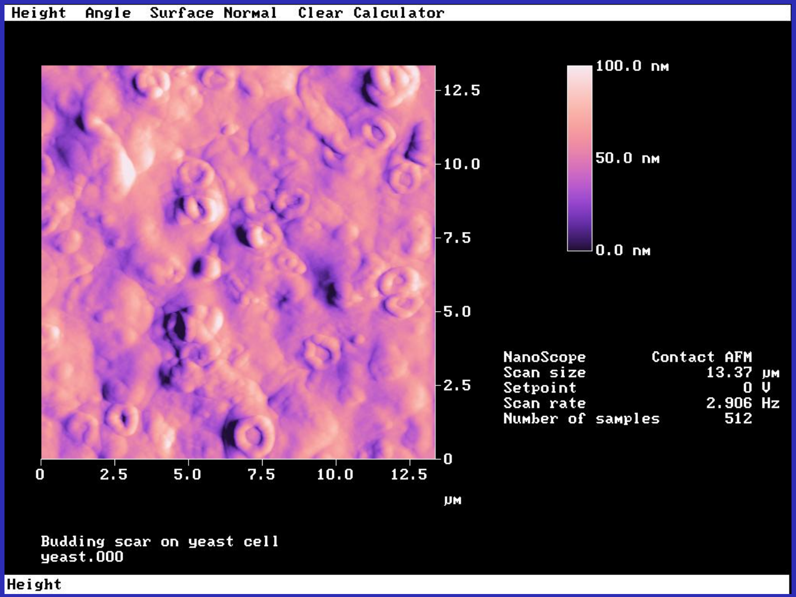
Light microscopy picture of cross section mushrooms 1:50

Mushrooms thread-like cells seen by light microscope 1:100

Views by different microscopes





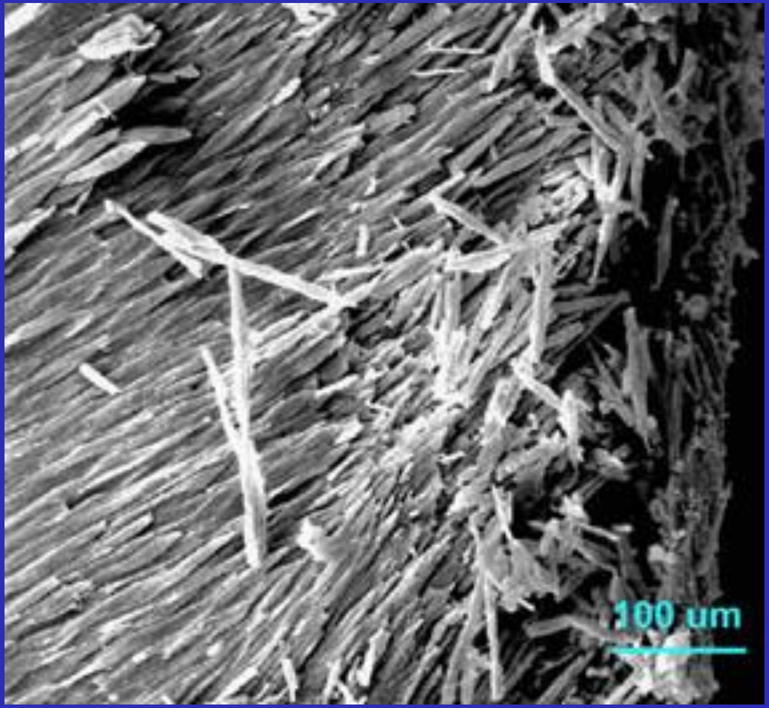


Organelles in the cell are more obvious in a TEM 1:1000

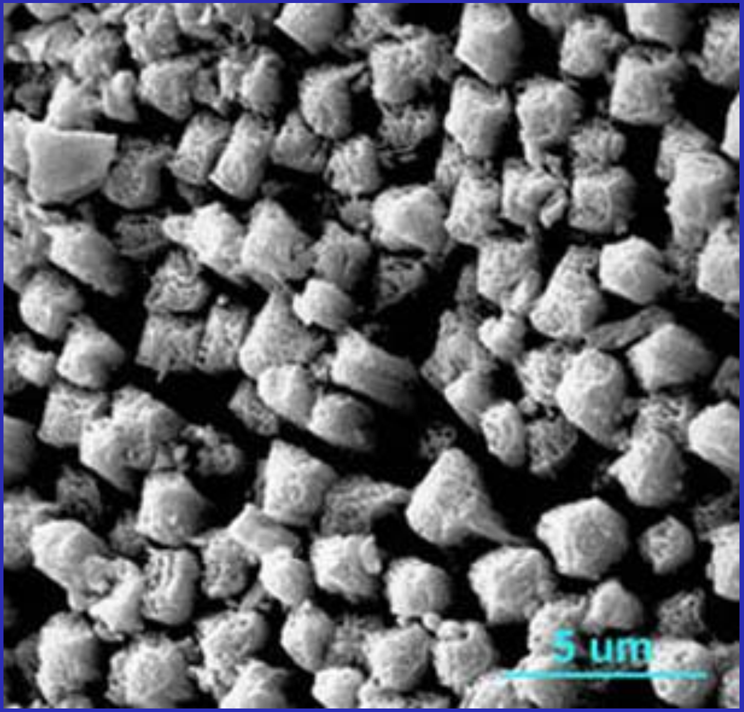
Spore-producing basidium visualized by SPM 1:250

different kind of fungus visualized by SPM

Views by different microscopes

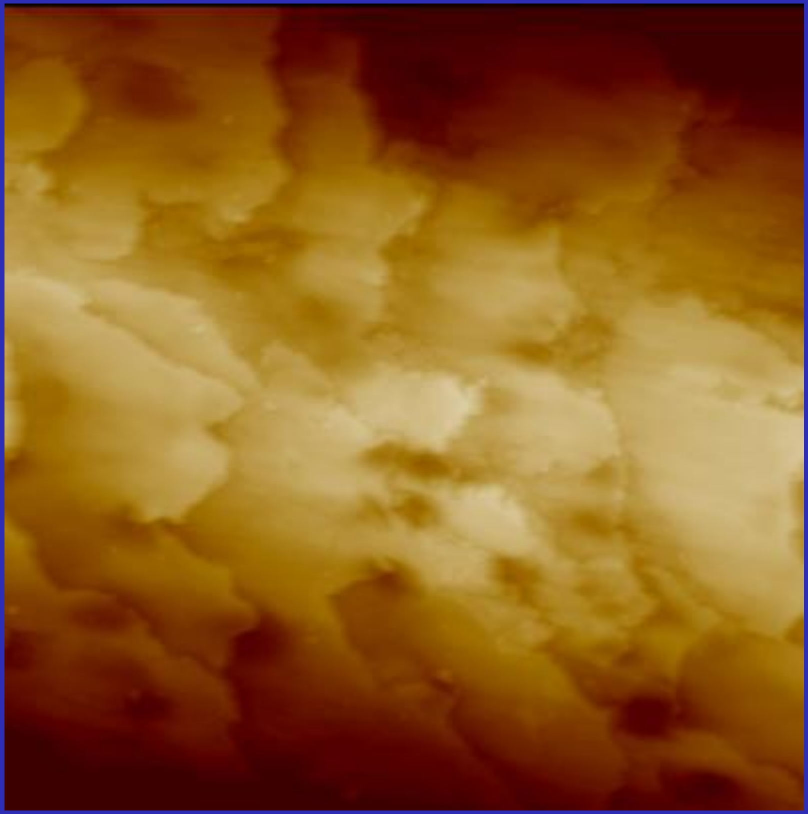


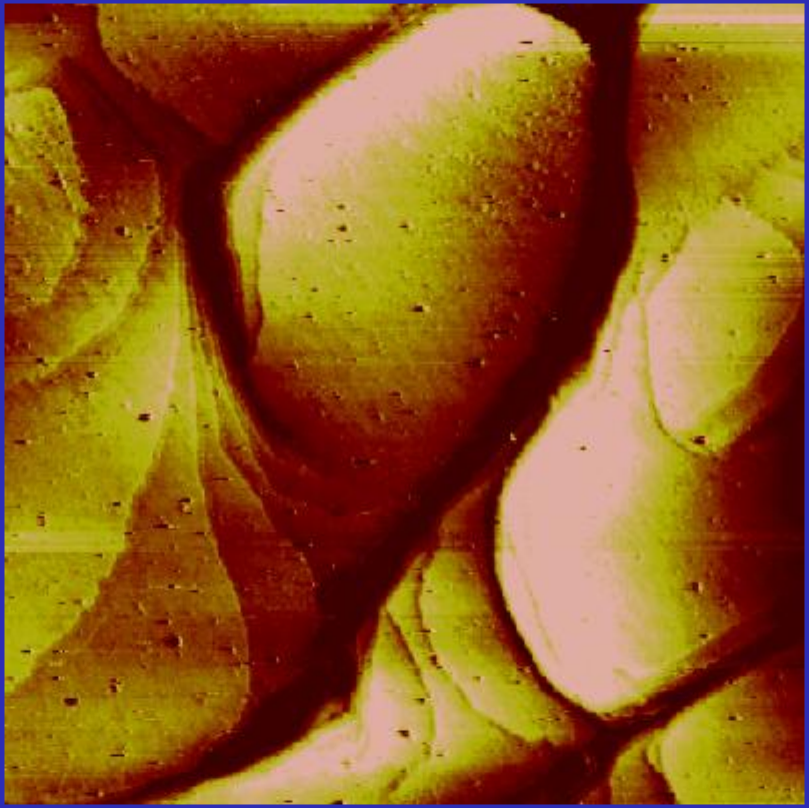
Fibrous texture of a mussel sea shell surface seen with SEM



Fibers at higher SEM magnification

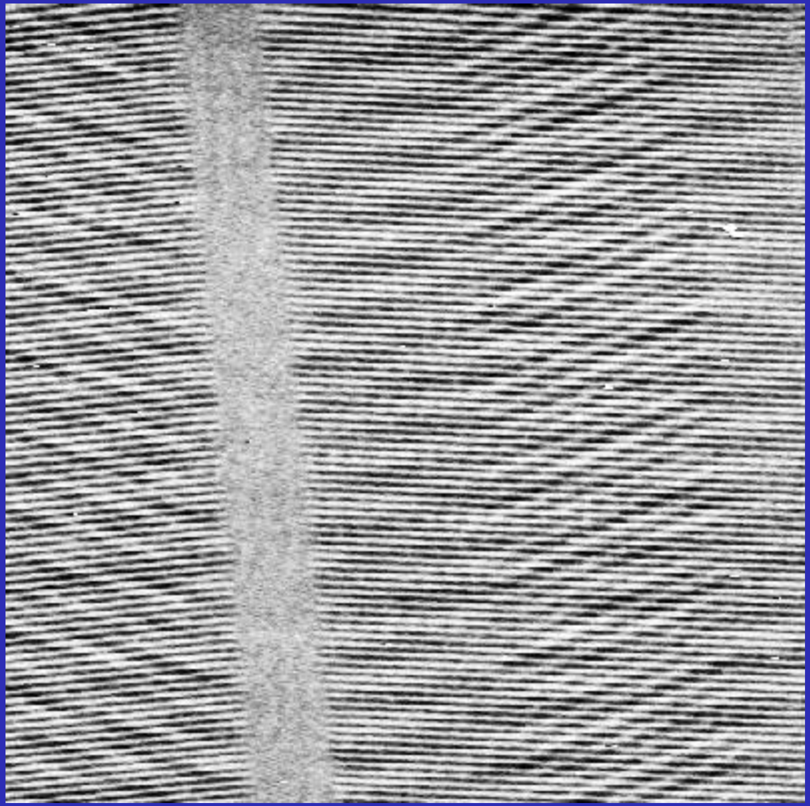
Views by different microscopes

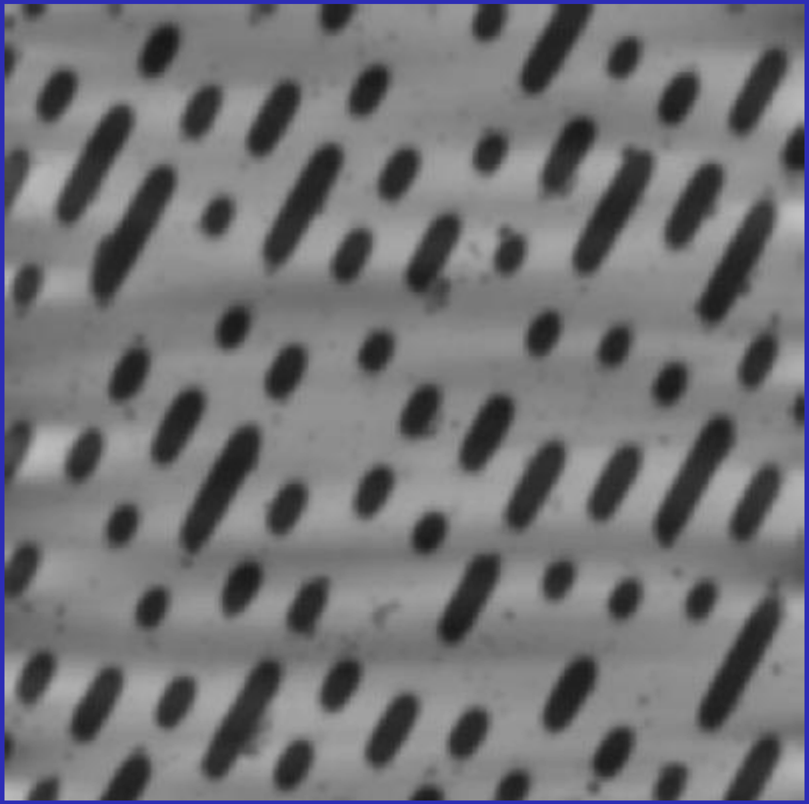




AFM images of horse hair and Gold film

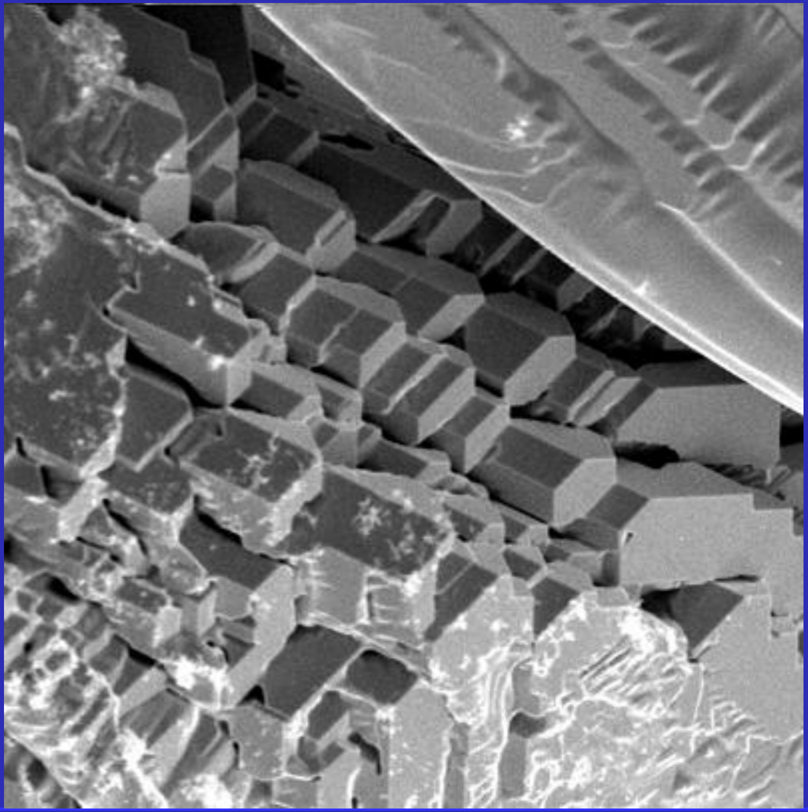
Views by different microscopes





STM images of video tape surface and CD room surface

Views by different microscopes

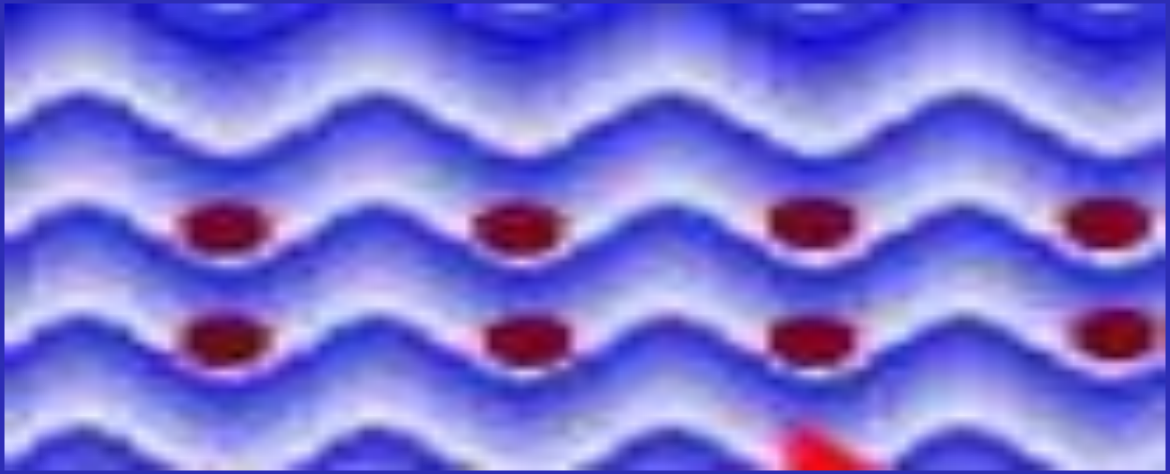
 

afm images: bacterium - tungsten

ELECTRON MICROSCOPY

- History
- Why to use electrons?
- Resolution Power

WHY USE ELECTRONS?



Because visible light has wavelengths that are hundreds of nanometers long we can not use optical microscopes to see into the nano world.

SOME FUNDAMENTAL PROPERTIES OF ELECTRONS (1)

Electrons show both particle and wave characteristics

- Broglie´s wave-particle duality:
  λ: wavelength; p: particle momentum
  h: Planck´s constant

$$\lambda = \frac{h}{p}$$

- In the TEM: momentum is imparting by accelerating it through a potential drop V, giving a kinetic energy

$$eV = \frac{1}{2} m_0 v^2$$

- relationship between wavelength and accelerating voltage V :

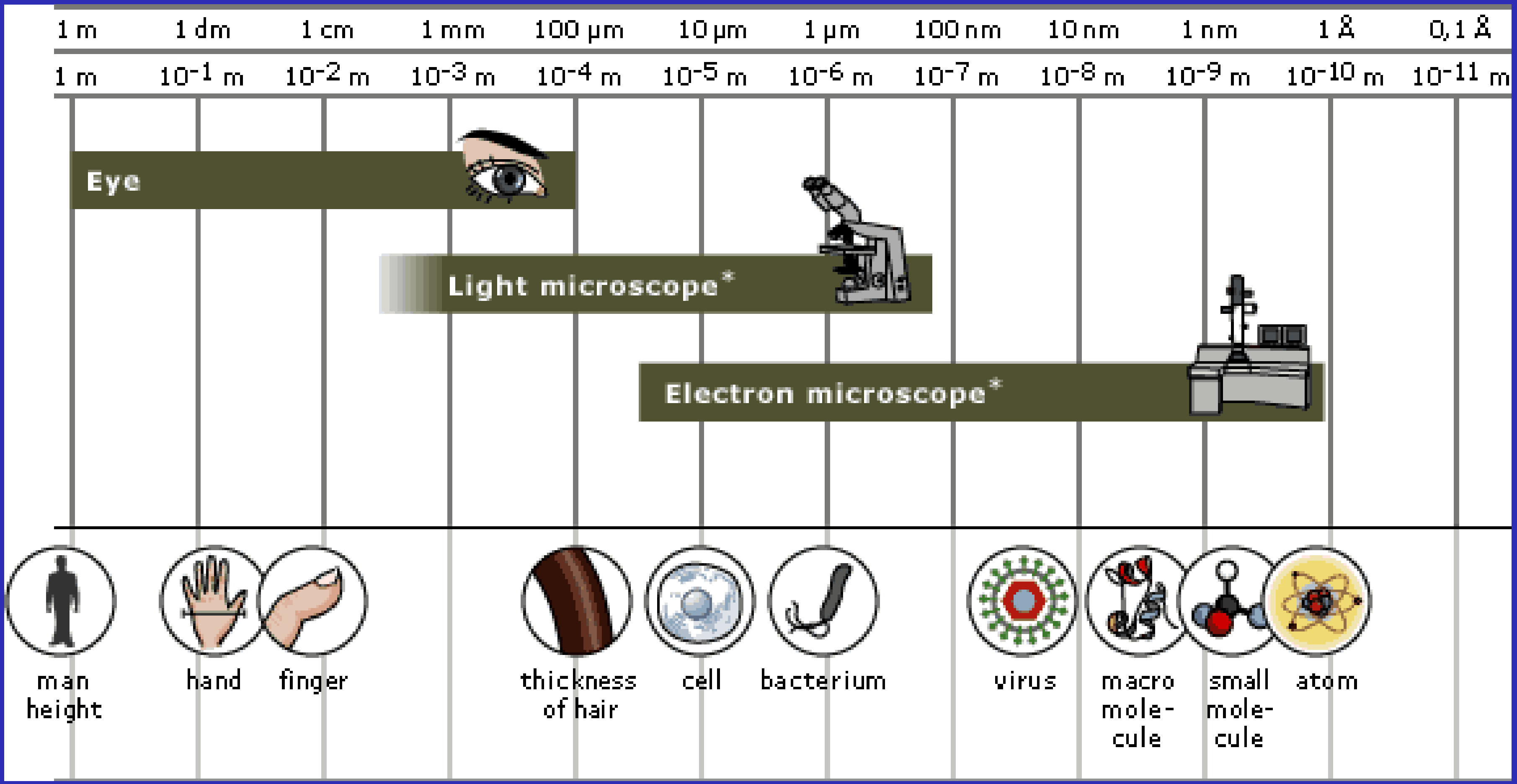
$$\lambda = \frac{h}{\left[2m_0 eV \left(1 + \frac{eV}{2m_0 c^2} \right) \right]^{1/2}} \approx \frac{h}{\left(2m_0 eV \right)^{1/2}}$$

ELECTRON PROPERTIES AS A FUNCTION OS ACCELERATING VOLTAGE

V (kV)	Non-relativisty λ (nm)	relativisty λ (nm)	Mass (*m_0)	Velocity (*10^8 m/s)
100	0.00386	0.00370	1.196	1.644
200	0.00273	0.00251	1.391	2.086
400	0.00193	0.00164	1.783	2.484
1000	0.00122	0.00087	2.957	2.823

Charge	-1.6×10^{-19} C
Rest mass m_0	9.109×10^{-31} kg
Rest energy (m_0c^2)	511 keV
Kinetic energy (for each 1V potential)	1.602×10^{-19} Nm
Planck´s constant (h)	6.626×10^{-34} Nms
Speed of light in vacuum	2.998×10^8 m/s

MICROSCOPY AND THE CONCEPT OF RESOLUTION



WHY USE ELECTRONS?

- 1925 – *Broglie* – electron has wave-like characteristic:

$$\lambda_e << \lambda_{light}$$

$$\lambda_{e-}(\mathrm{nm}) \approx \frac{1.22}{\sqrt{\mathrm{E}(\mathrm{eV})}}$$

$$E = 100keV \therefore \lambda_{e-} \approx 0.0004nm$$

Image resolution?

MICROSCOPY AND THE CONCEPT OF RESOLUTION
DEFINITIONS

RESOLUTION: ability to distinguish closely spaced points as separate points. **RESOLUTION LIMIT:** smallest separation of points which can be recognized as distinct.

Resolving power *is a property of the instrument and is a quantity that may be estimated on theoretical grounds.*

Resolution *is equal to or poorer than the resolving power and is the quantity observed under any given set of experimental conditions.*

MICROSCOPY AND THE CONCEPT OF RESOLUTION

DEFINITIONS

The maximum magnification of an instrument is limited according to the following relationship:

$$Maximum \cdot Magnification = \frac{resolving \cdot power \cdot eye}{resolving \cdot power \cdot microscope}$$

The main limiting factor in the TEM, with respect to achieving the theoretical resolving power of the instrument, concerns the nature of the imaging lenses and the process of image formation.

MICROSCOPY AND THE CONCEPT OF RESOLUTION

Image resolution - Rayleigh criterion

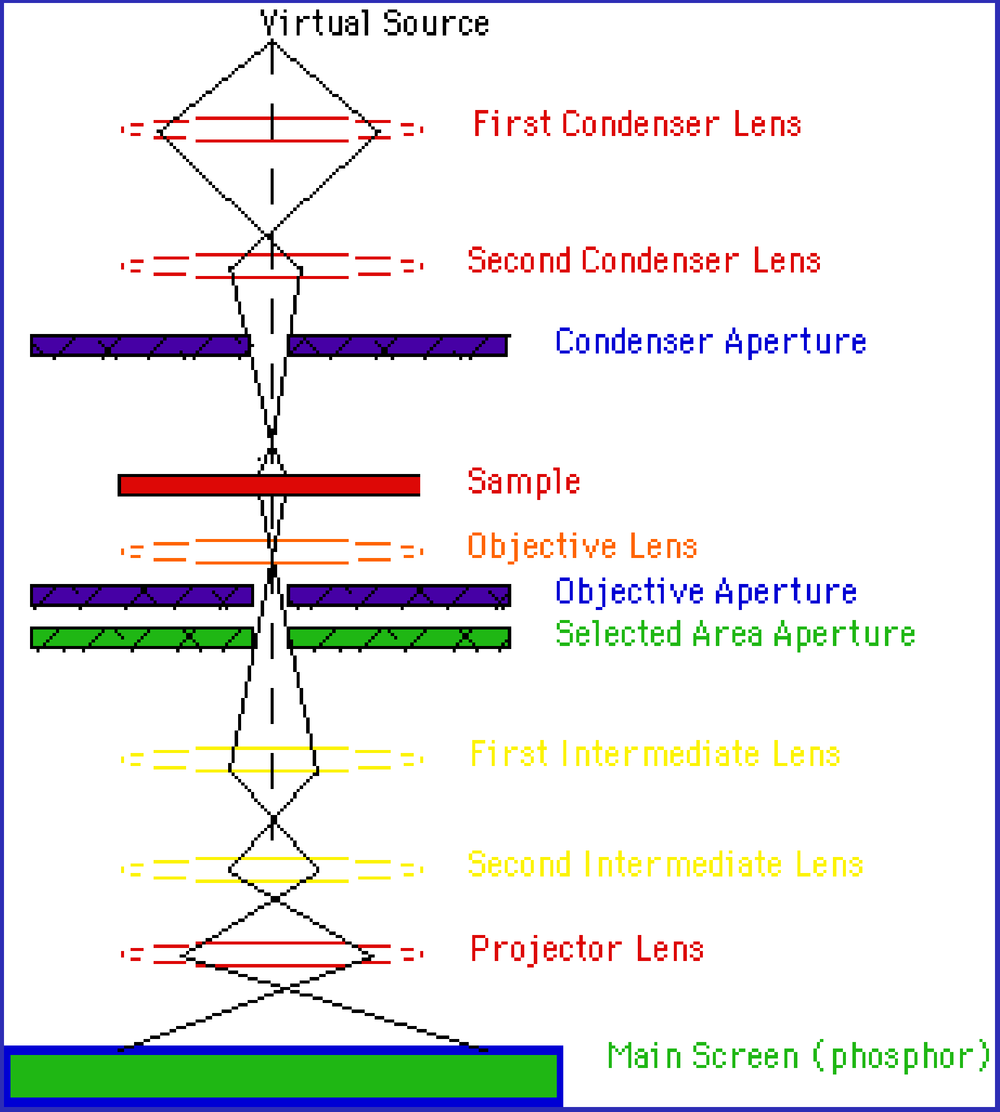
$$\delta = \frac{0.16\lambda}{\mu \sin \beta}$$

Angular aperture of the lens

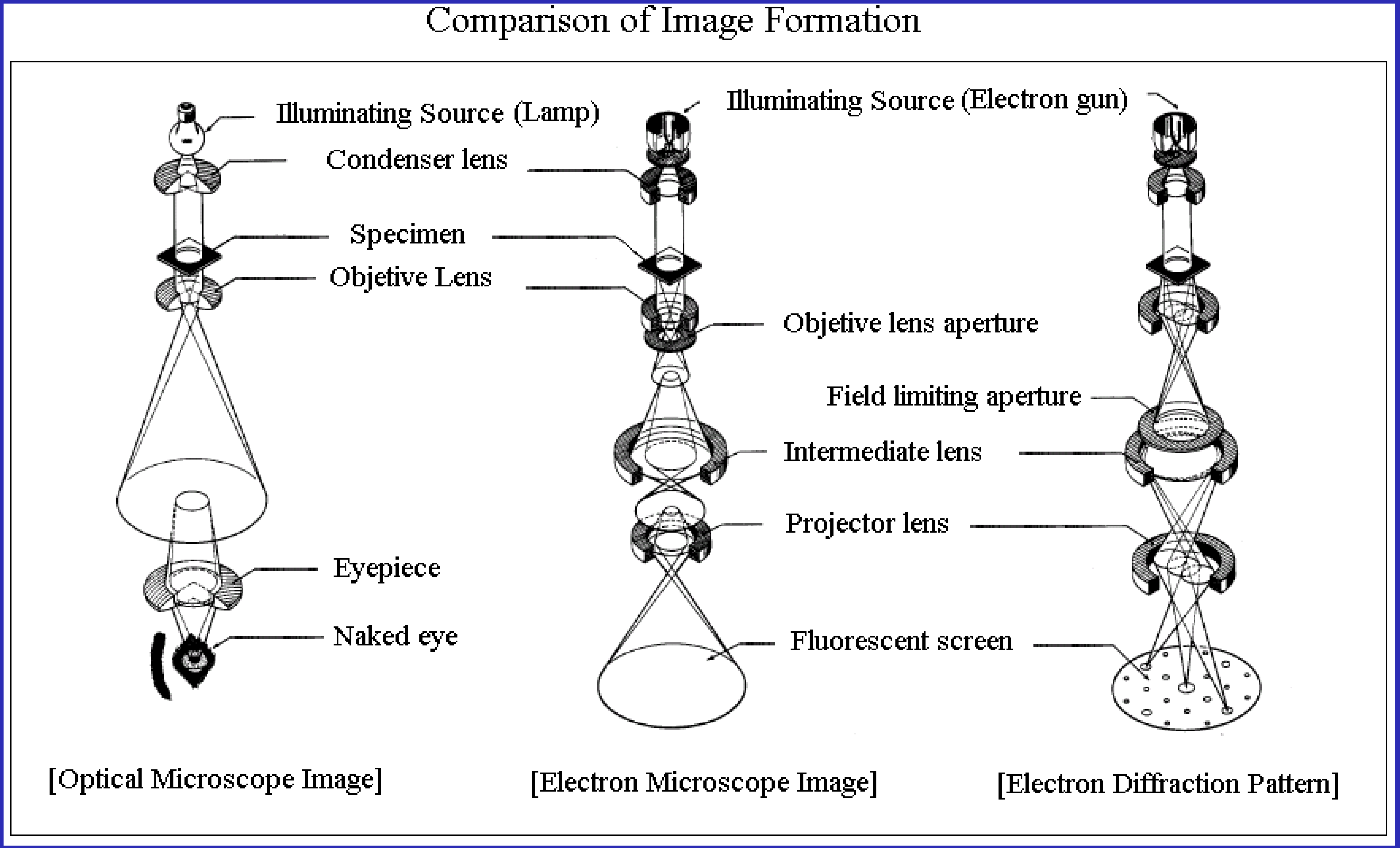
The aperture determines the total amount of radiation arriving from the object which can be focused to form an image. The aperture thus controls the ability of the lens to gather information about the object. This depends on the angle of the cone of rays it is able to accept from the object.

OPTICAL VS ELECTRON MICROSCOPY

TEM SKETCH


Virtual Source
First Condenser Lens
Second Condenser Lens
Condenser Aperture
Sample
Objective Lens
Objective Aperture
Selected Area Aperture
First Intermediate Lens
Second Intermediate Lens
Projector Lens
Main Screen (phosphor)

ANALOGIES WITH LIGHT MICROSCOPE



OPTICAL VS ELECTRON MICROSCOPY: SIMILARITIES

1) **Illumination system:**
   i) Source: emits radiation
   ii) Condenser lens: focuses the beam on the specimen.

2) **Specimen stage:** situated between the illumination and imaging systems.

3) **Imaging system:** Lenses
   i) objective lens: focuses the beam after it passes through the specimen and forms an image
   ii) the projector lens(es): magnifies a portion of the intermediate image to form the final image.

4) **Image recording system:** Converts radiation into a permanent image - photographic emulsion- that can be viewed

OPTICAL VS ELECTRON MICROSCOPY: DIFFERENCES

1) Optical lenses:

LM: glass with fixed focal lengths

EM: electro magnetic lenses - focal length which can be changed by varying the current through the coil

2) Magnification

LM: provided by different power objective lenses

EM: focal length of the objective remains fixed while the focal length of the projector lens is changed to vary magnification.

3) Depth of field,

LM: small - different focal levels can be seen

EM: large - means that the entire (thin) specimen is in focus simultaneously.

OPTICAL VS ELECTRON MICROSCOPY: DIFFERENCES

4) Mechanisms of image formation vary (phase and amplitude contrast).

5) Position of the Source:

LM: source is generally at the bottom

EM: source at the top of the instrument

6) Enviroment:

LM: Air

EM: VACUUM - so **most** biological specimens must be dehydrated (i.e. dead !!) – BUT

ENVIROMENT SEM –TEM - STEM

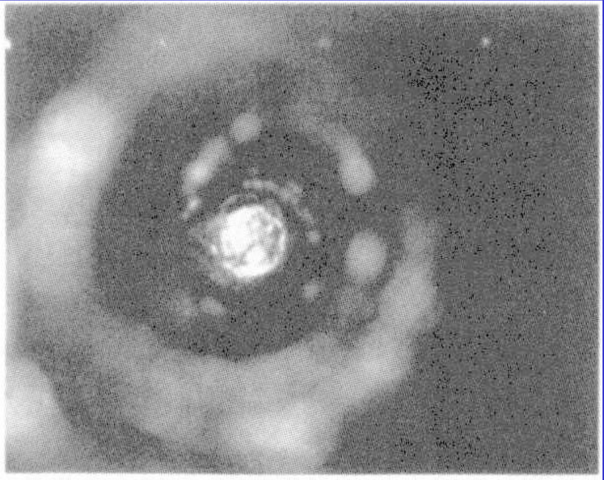
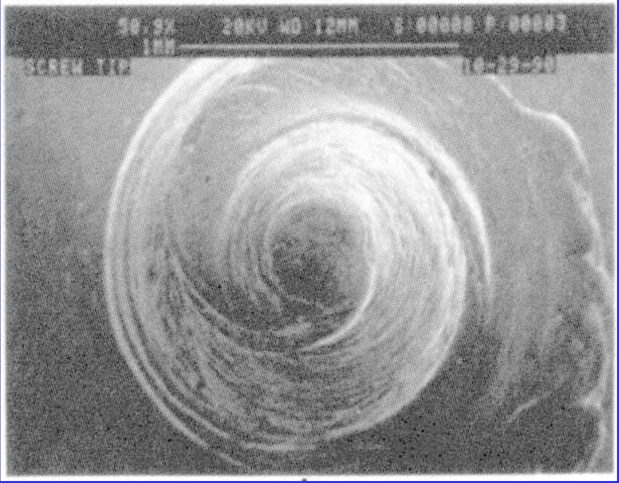
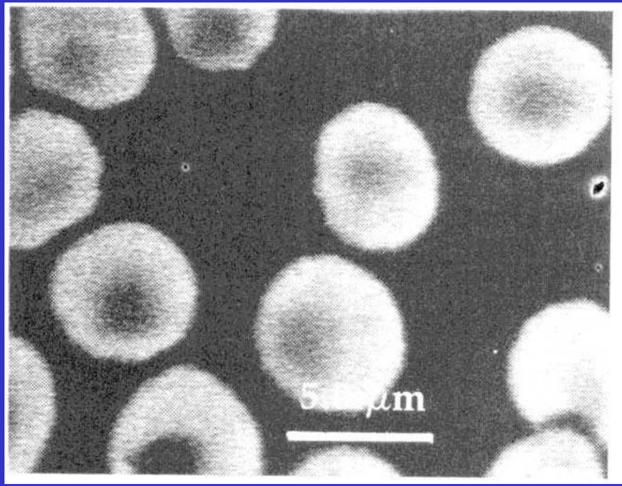
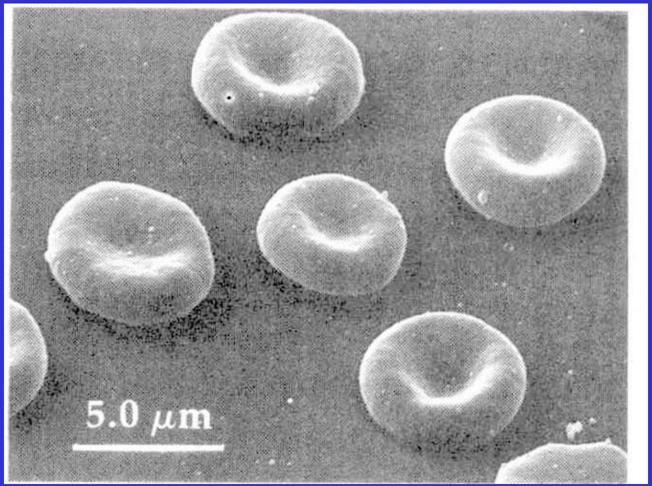
OPTICAL VS ELECTRON MICROSCOPY: DIFFERENCES

7) TEM specimens (biological) are rapidly damaged by the electron beam.

8) TEMs can achieve higher magnification and better resolution than LMs.

9) Price tag!!! (100x more than LM)

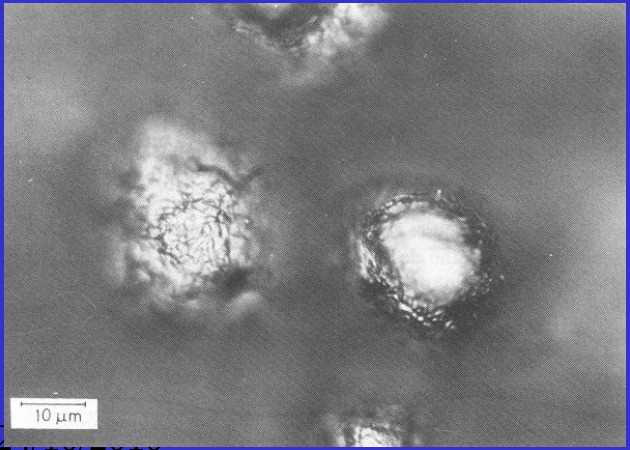
SEM Images: Improved Depth of Focus

	Optical Image	SEM Image	
screw			From Brundle
cells		 	From Flegler

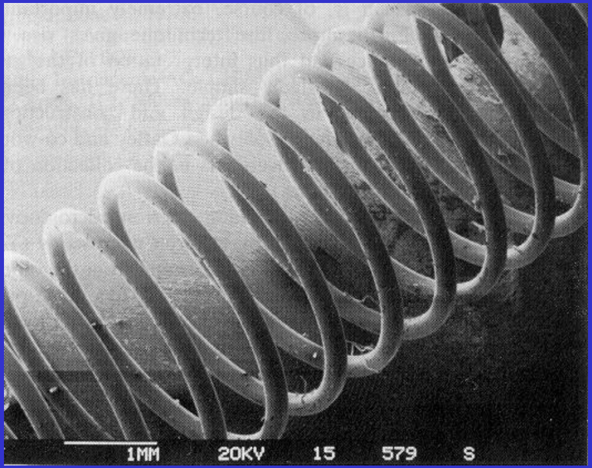
SEM provide higher depth of focus compared to OM

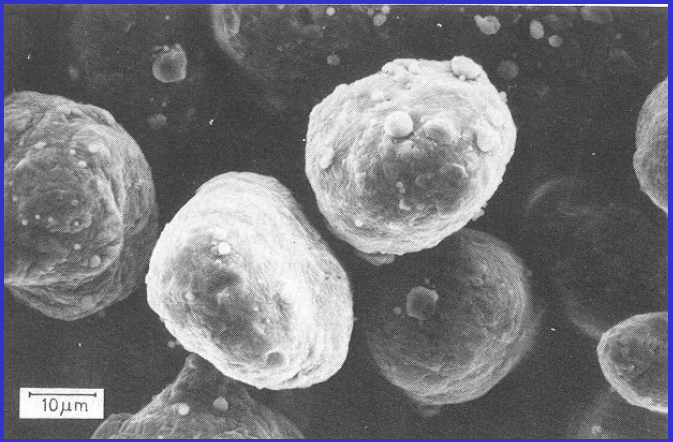
Advantages of SEM over light microscope

Optical Image

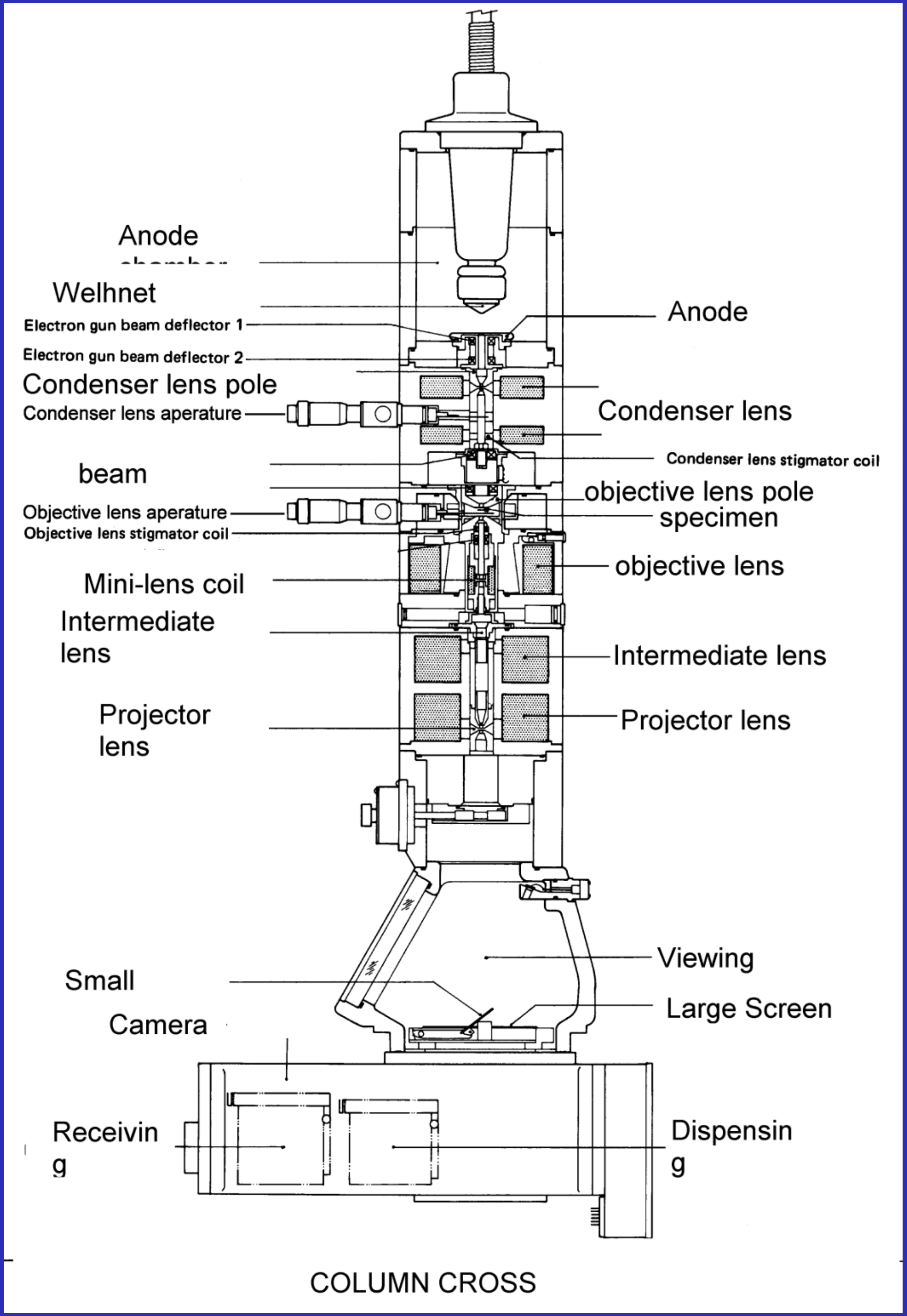


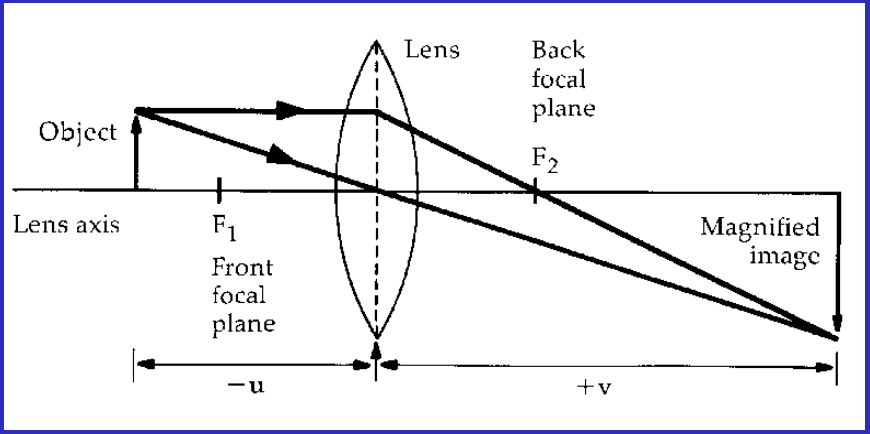
SEM Image



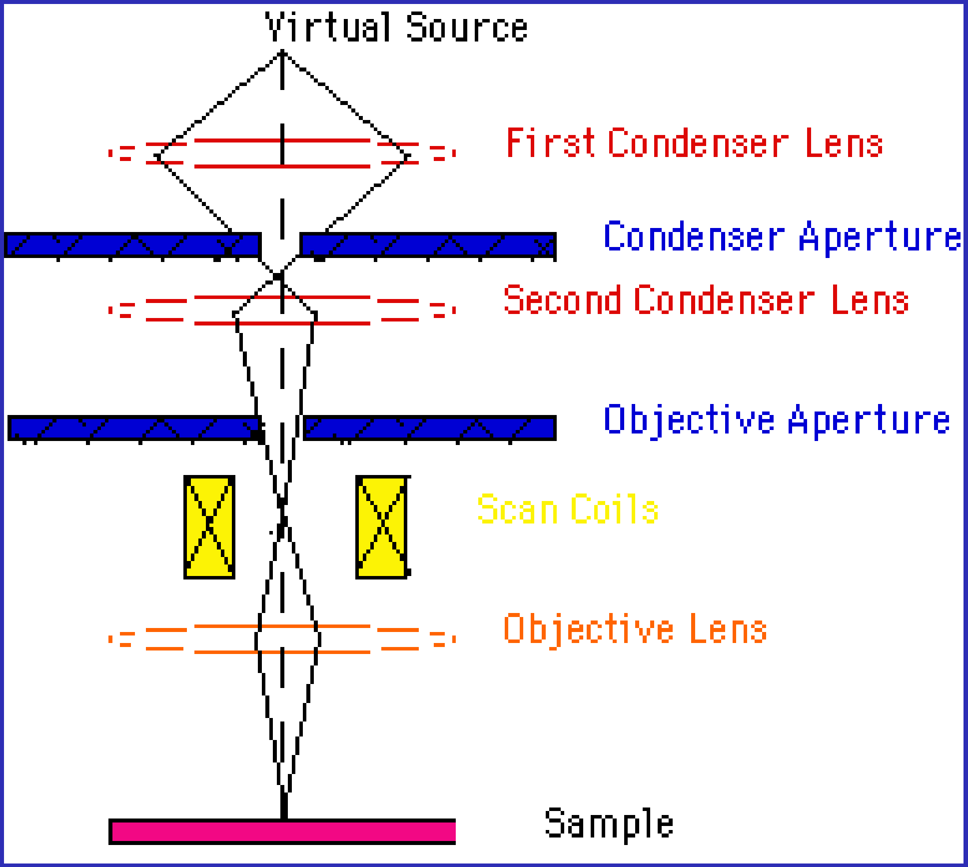


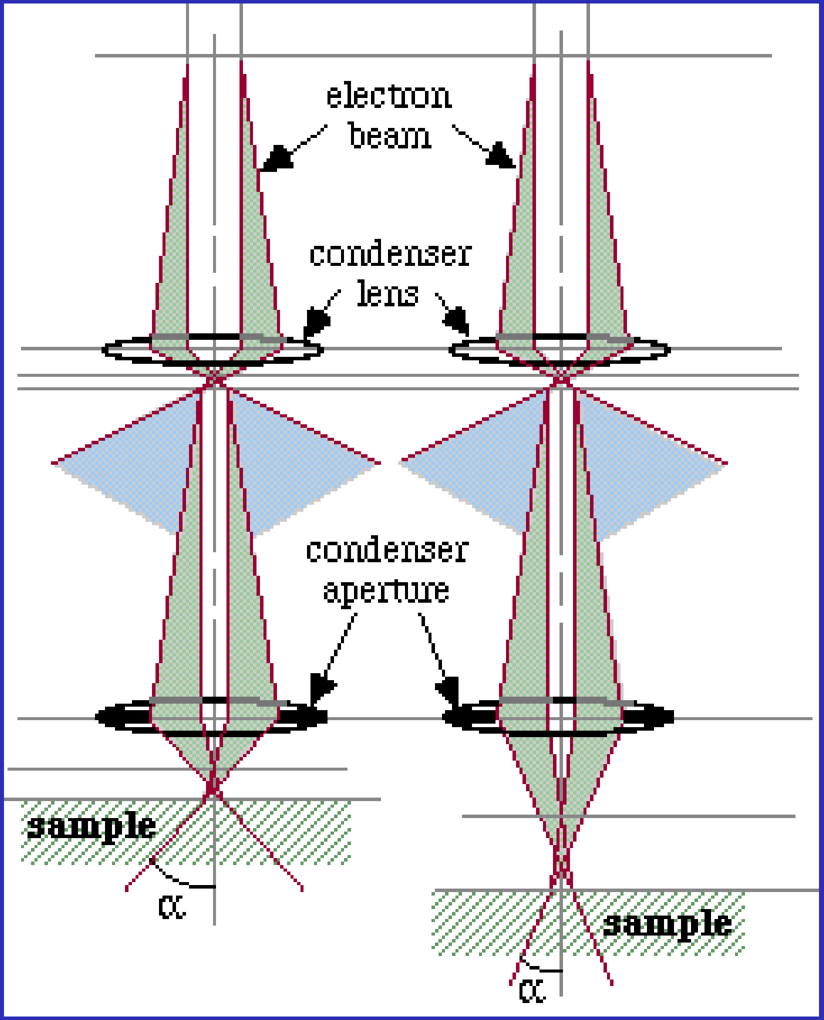
ELECTRON MICROSCOPE (1)



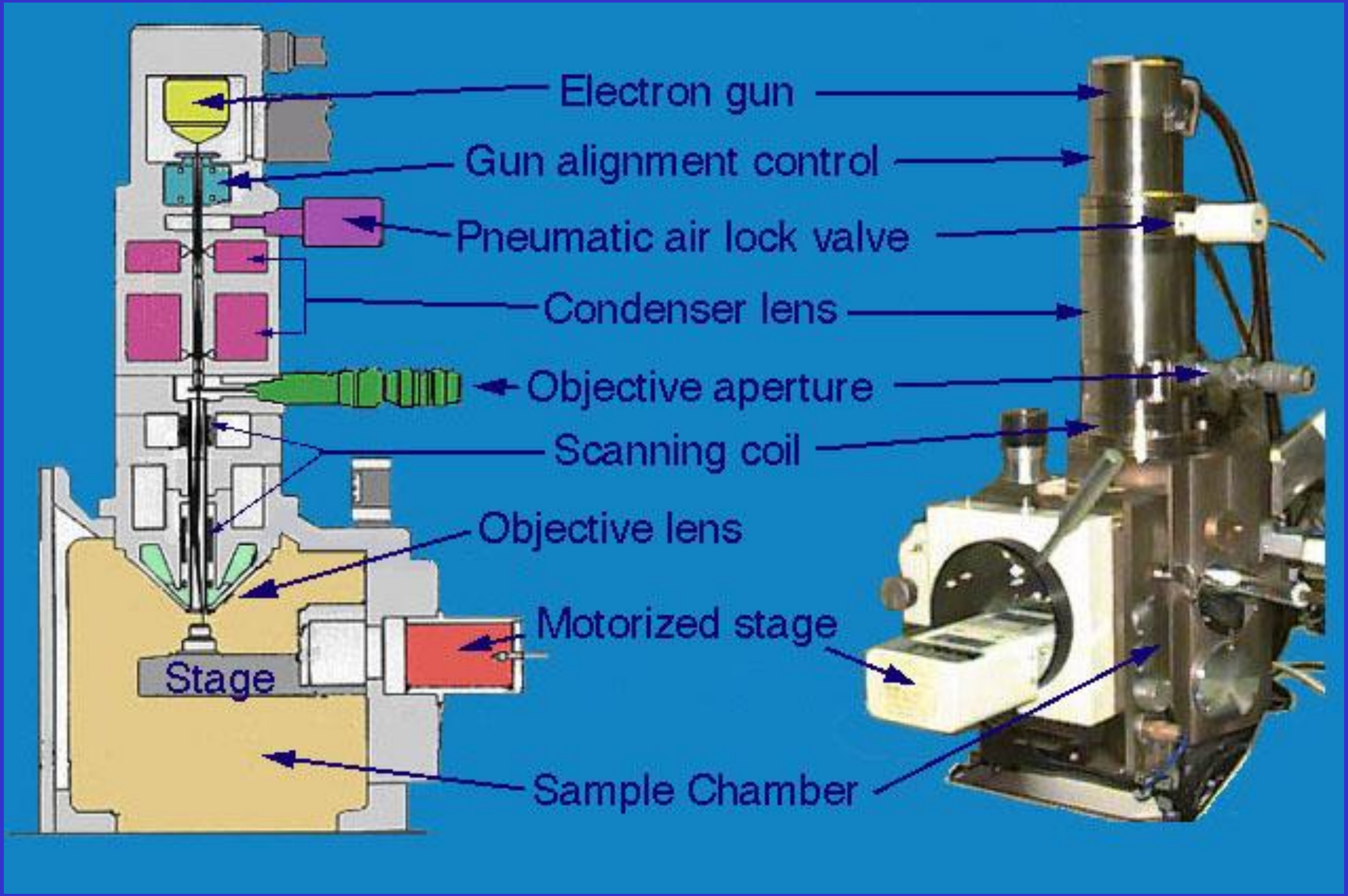


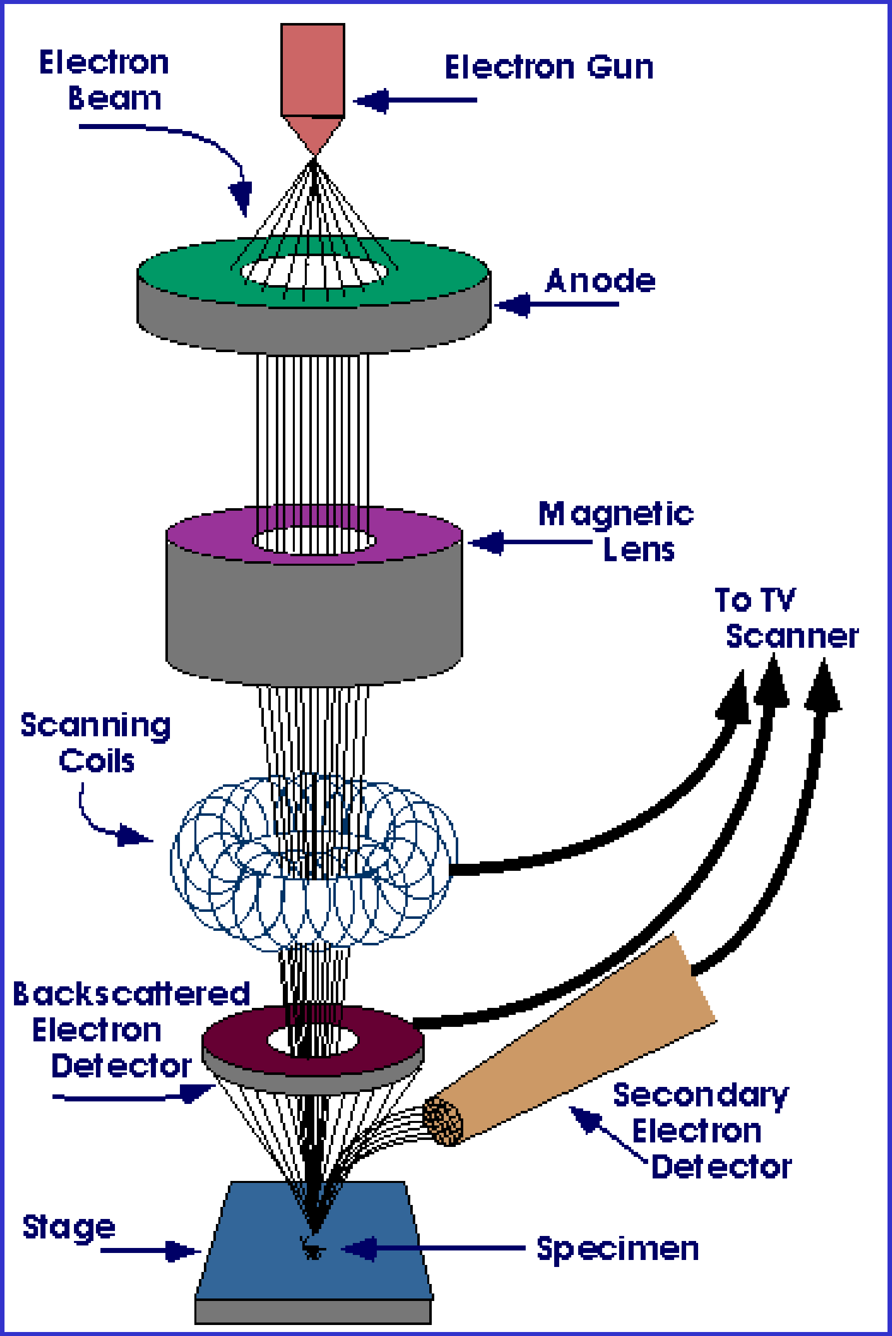
RAY DIAGRAM - SEM





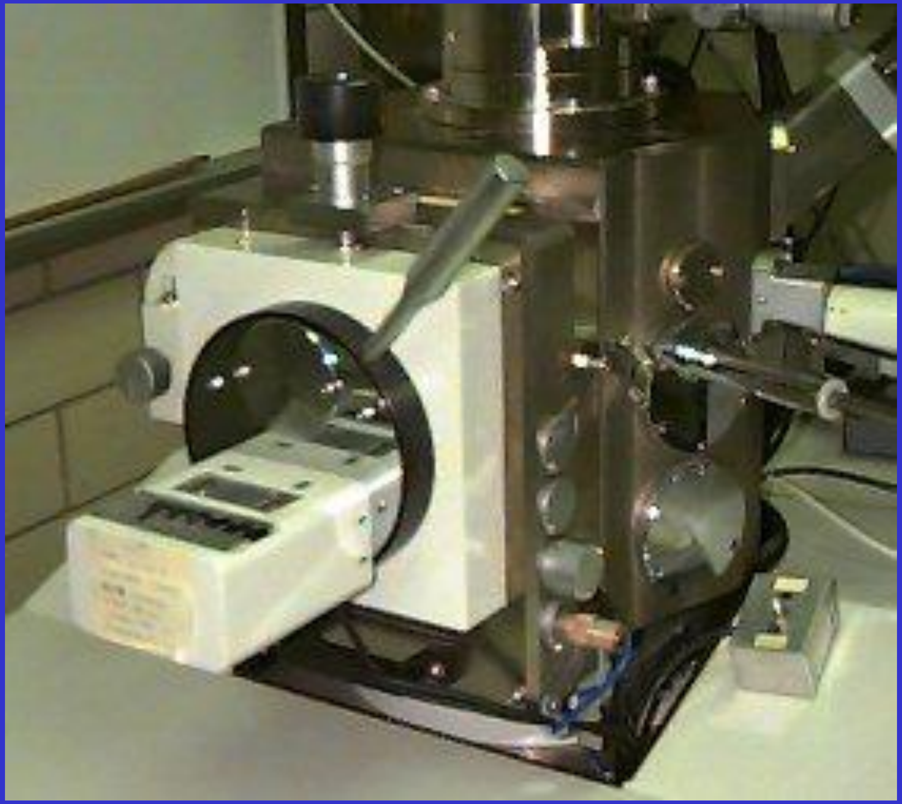
ELECTRON MICROSCOPES: SEM

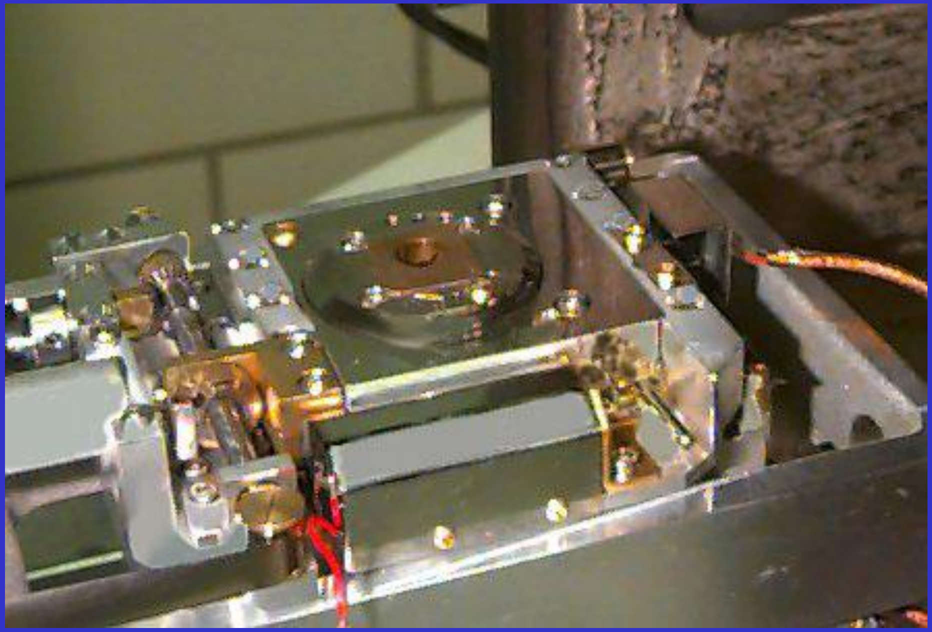




http://mse.iastate.edu/microscopy/path2.html

SEM SAMPLE CHAMBER





Lens Aberrations And Other Non-Ideal Imaging Properties

- spherical aberration
- astigmatism

Because of the rotation effect of magnetic lenses:

- anisotropic astigmatism
- anisotropic distortion

Other factors in image defects

- chromatic aberration
- rotational chromatic aberration
- and space-charge distortion

VIEWING NANOWORLD

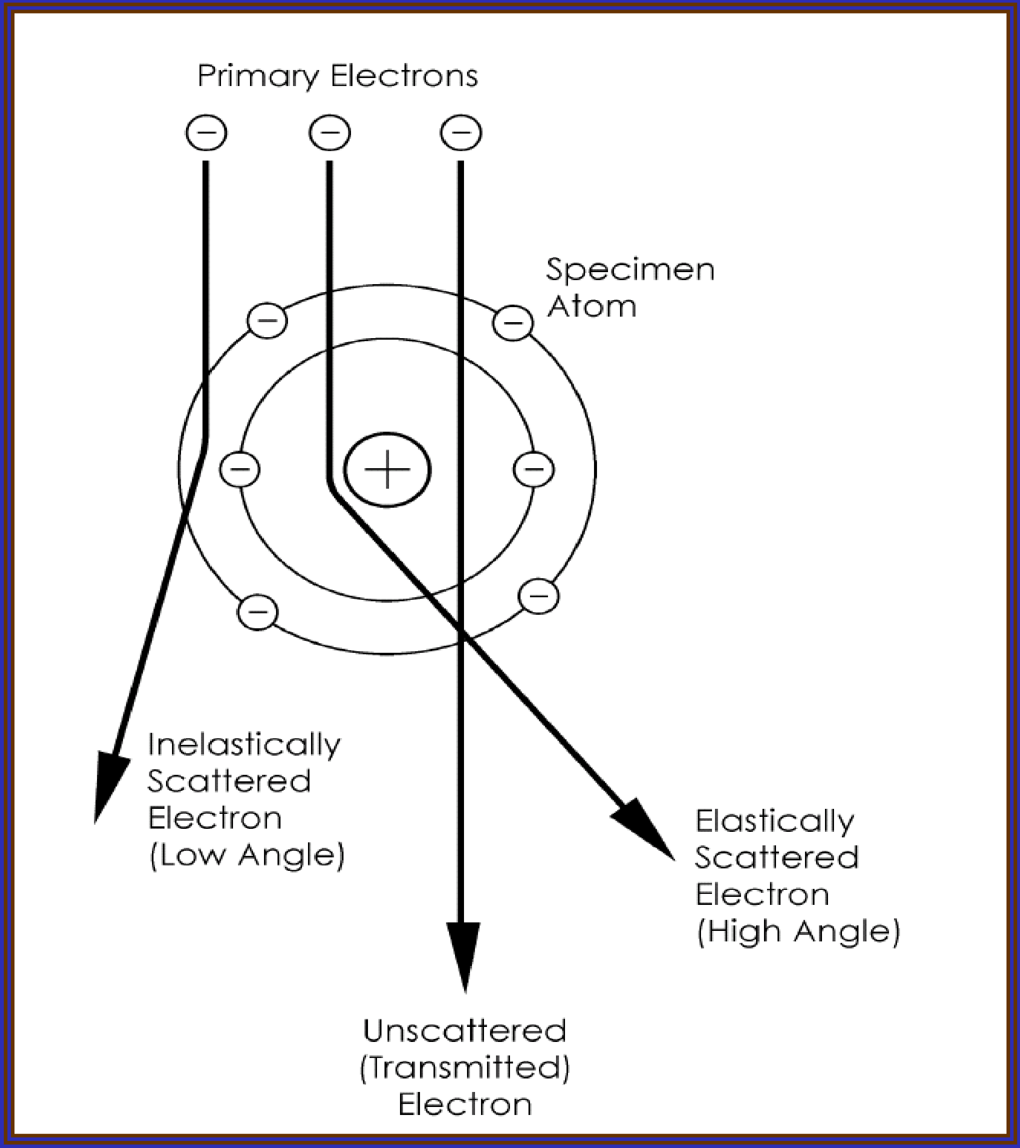
- Introduction
- Fundamentals
  - * Radiation – Matter Interaction
  - * Force microscopy
- Scanning Electron Microscopy – SEM
- Scanning Probe Microscopy SPM
- Transmission Electron Microscope -TEM
- High Resolution Electron Microscope HRTEM

BEAM SPECIMEN INTERACTION

SOME FUNDAMENTAL PROPERTIES OF ELECTRONS (2)
Electron beam interaction

- Electrons are one type of ionizing radiation
- Ionizing radiation: radiation capable of removing one of the tightly bound inner-shell electrons from the attractive field of the nucleus
- Ionizing radiation produces a wide range of secondary signals from the specimen

SOME FUNDAMENTAL PROPERTIES OF ELECTRONS (2)
Electron beam interaction



Interaction between electron beam and sample

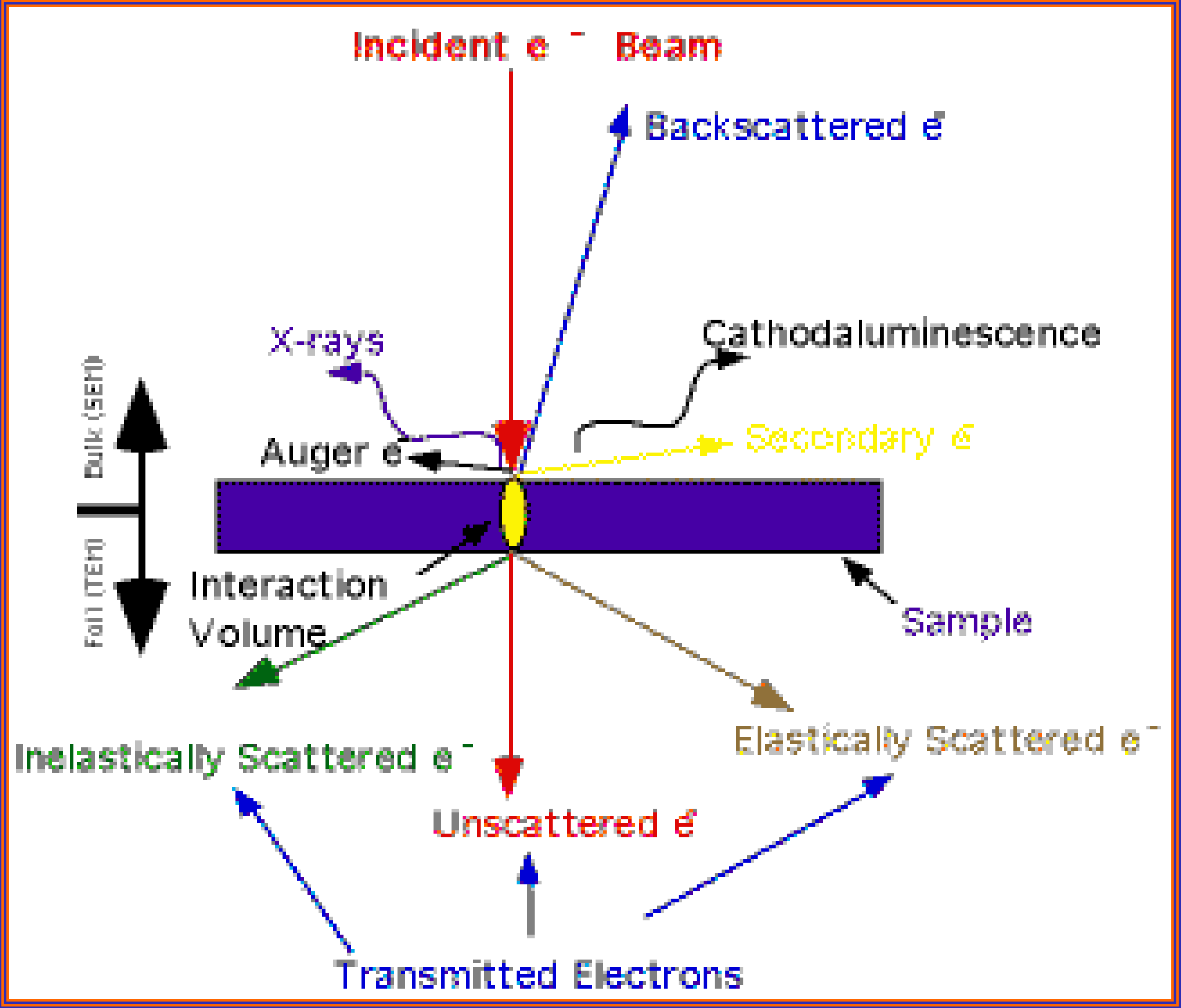
The elastic interactions:

- ✓ incident electrons and nucleus
- ✓ a large-angle deflection of incident electrons.
- ✓ little energy loss

The inelastic interactions:

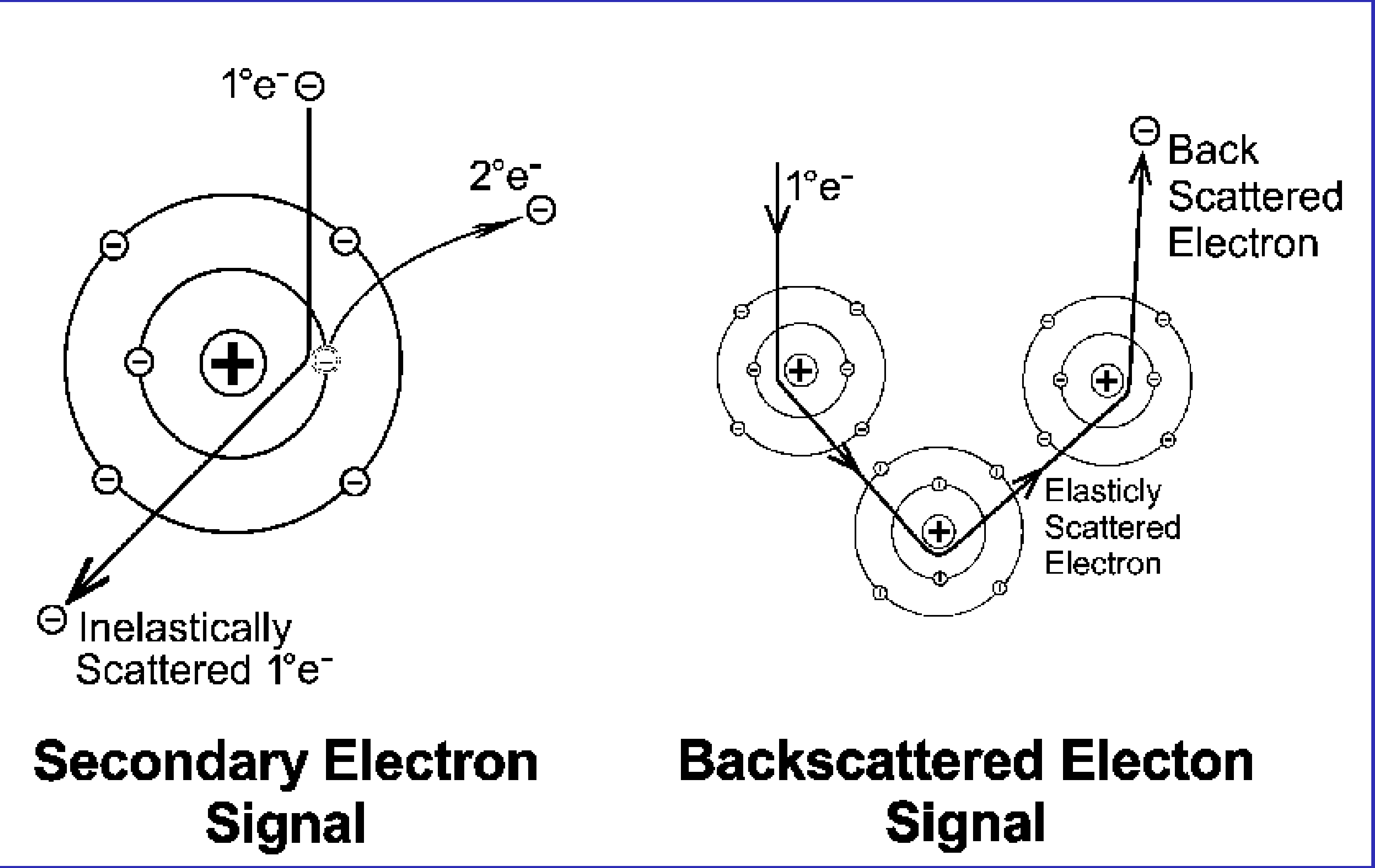
- ➢ incident electrons and orbital shell electrons
- ➢ a small-angle deflection of incident electrons.
- ➢ heavy energy loss

SPECIMEN INTERACTION

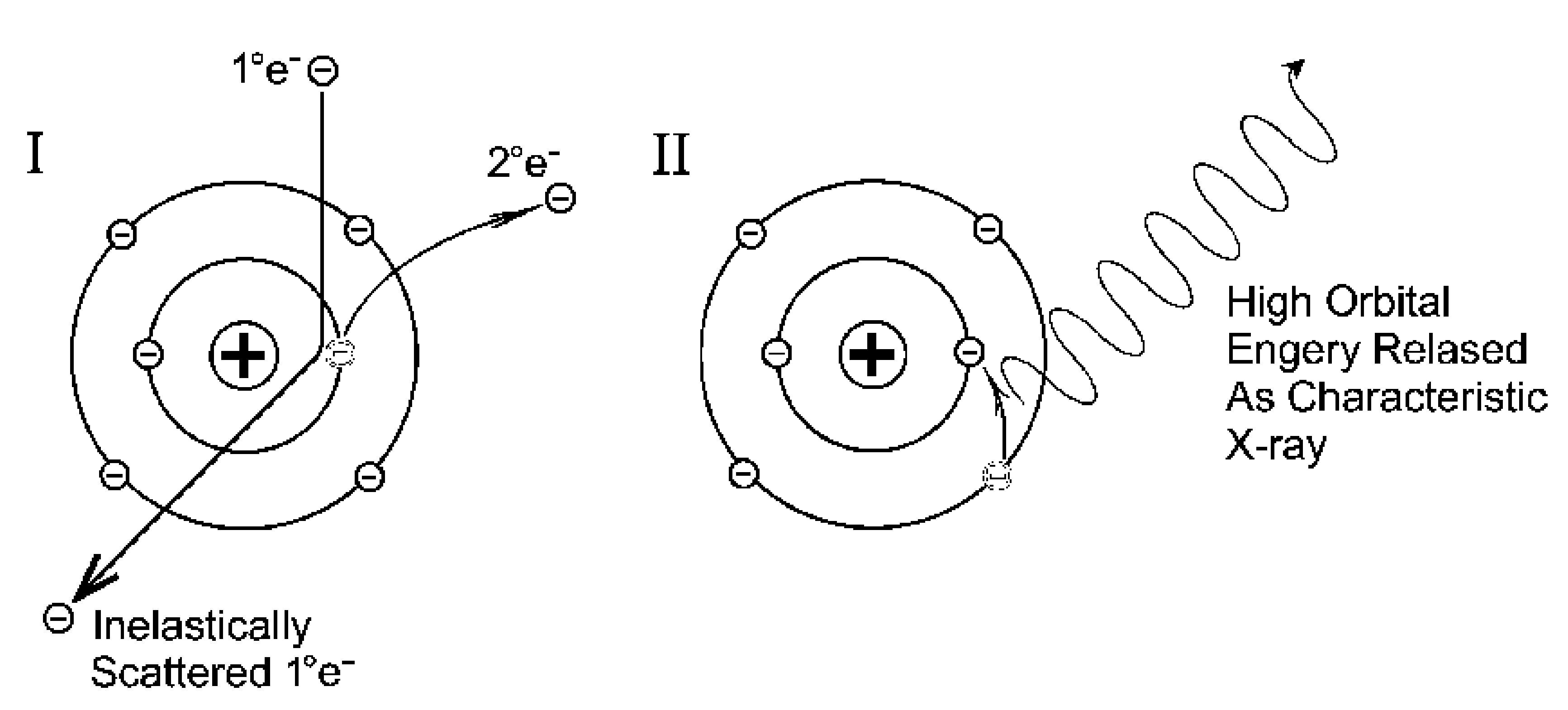


Specimen interaction is what makes ***Electron Microscopy*** possible

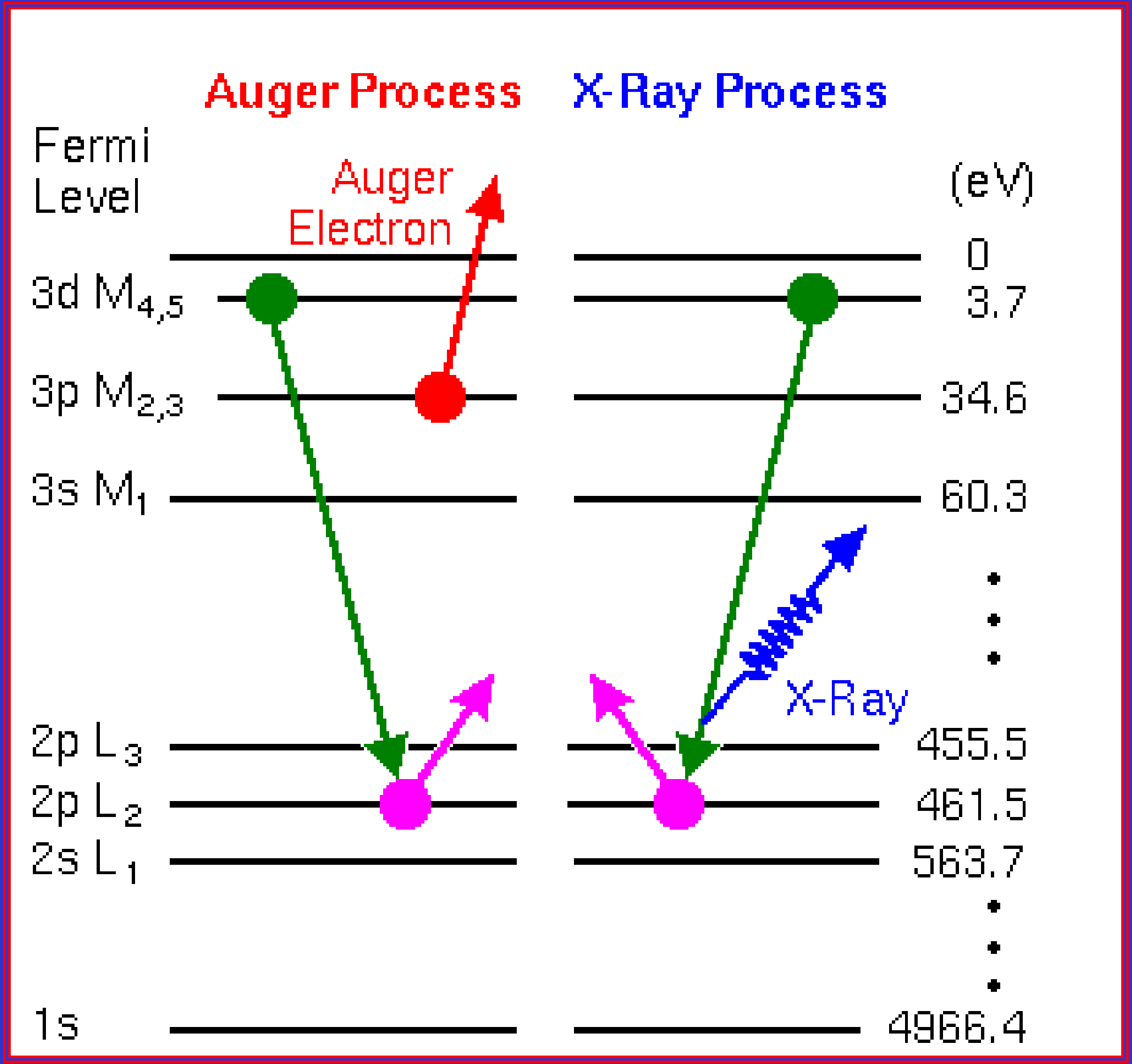
SCANNING ELECTRON MICROSCOPE: FUNDAMENTALS



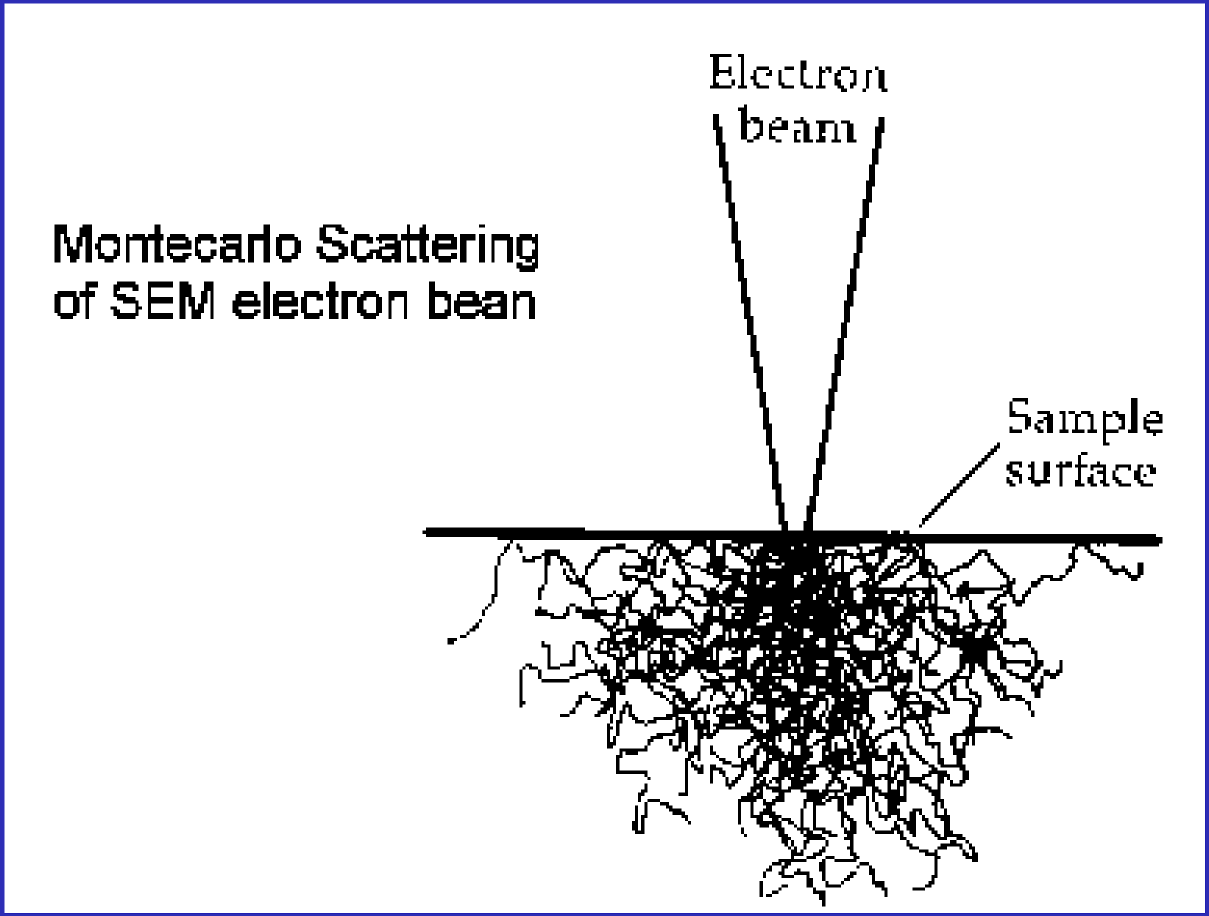
SEM FUNDAMENTALS: CHARACTERISTIC X-RAY SIGNAL



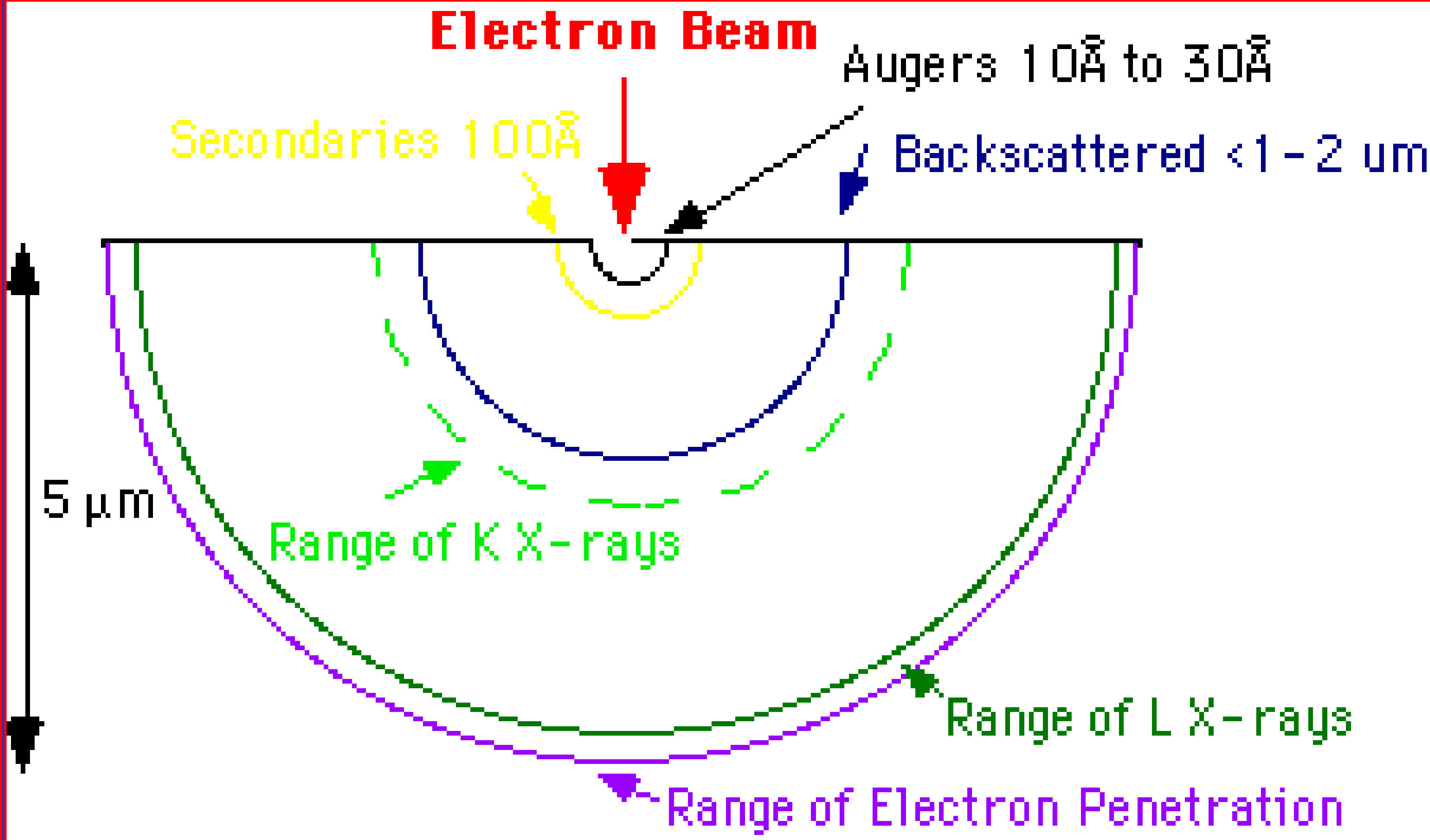
SEM - AUGER ELECTRONS



SEM: INTERACTION VOLUMES



SEM: GENERATION DEPTHS



At 20 KV Accelerating Voltage and Z=28

VIEWING NANOWORLD

- Introduction
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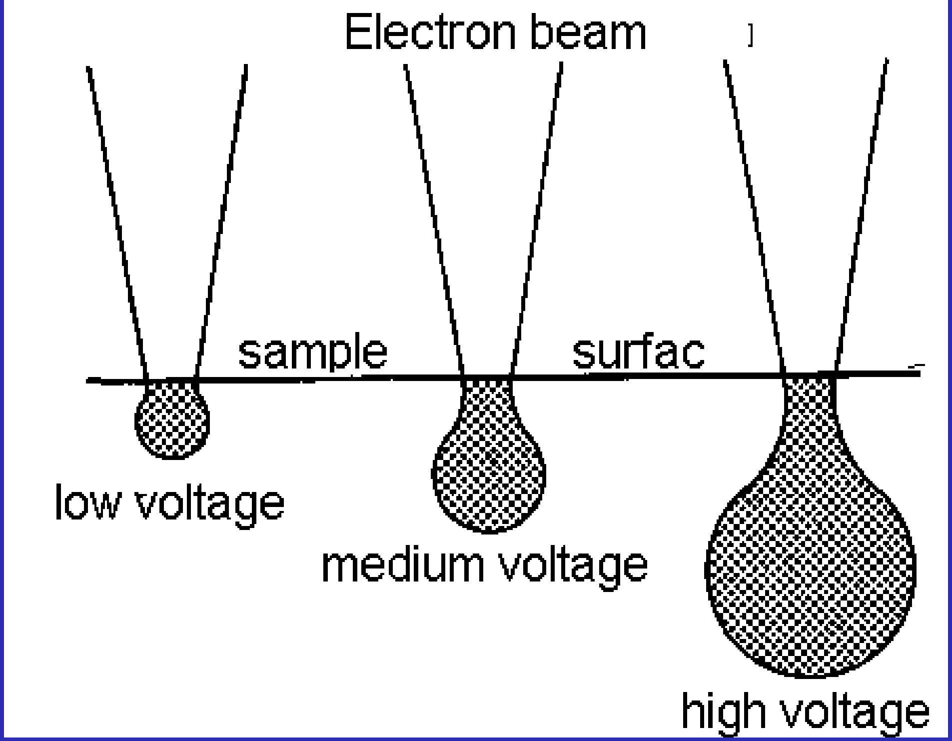
  * Force microscopy
- Scanning Electron Microscopy – SEM
- Transmission Electron Microscope -TEM
- High Resolution Electron Microscope HRTEM
- Scanning Probe Microscopy SPM

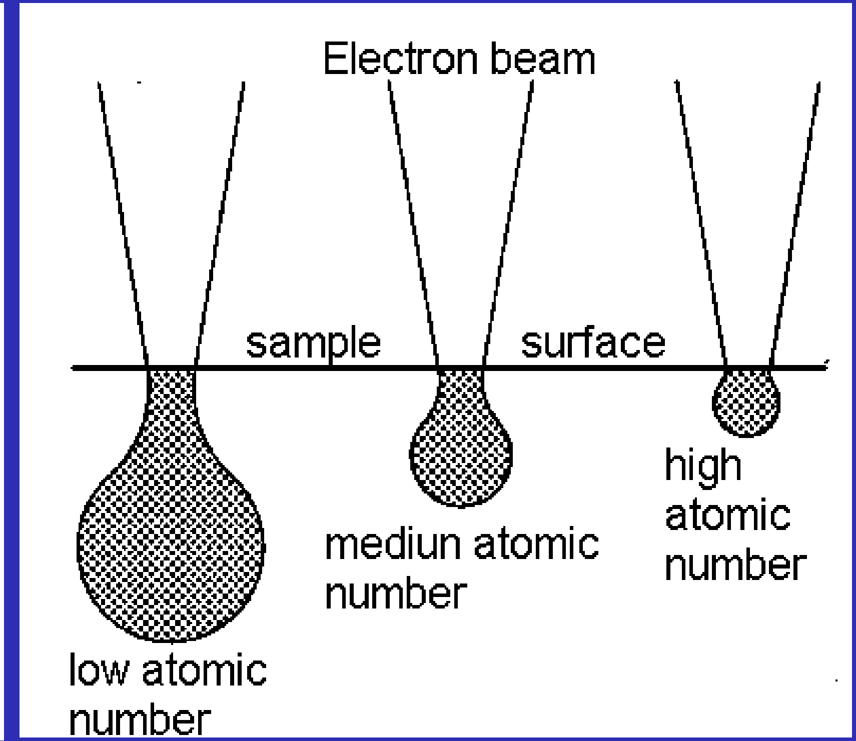
SEM: GENERATION DEPTHS

- The volume inside the specimen in which interactions occur while being struck with an electron beam

- Volume depends on
  - ✓ atomic number of the material
  - ✓ accelerating voltage
  - ✓ angle of incidence for the electron beam; the greater the angle (further from normal) the smaller the volume

SEM FUNDAMENTALS: DEPTH OF SCATTERING





VOLTAGE

higher voltages penetrate farther into the sample and generate larger interaction volumes

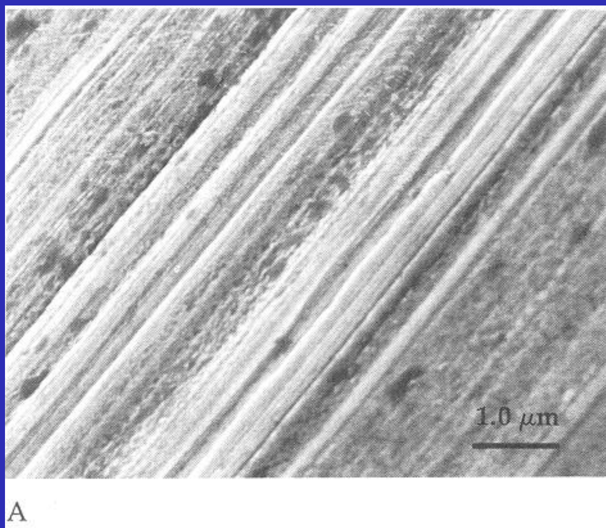
ATOMIC NUMBER

higher atomic number materials absorb or stop more electrons and so have a smaller interaction volume

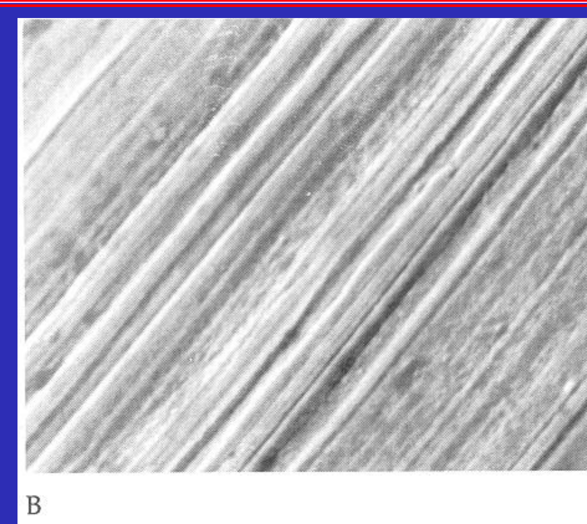
Effect of acceleration voltage on resolution

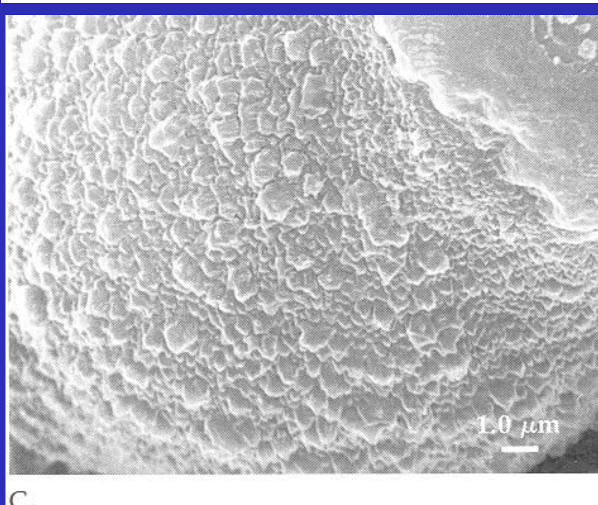
- Higher voltage generates shorter wavelength of electrons → better resolution.
- Higher voltage causes an increase of the volume of electrons/specimen interactions → worse resolution

Effect of acceleration voltage on resolution

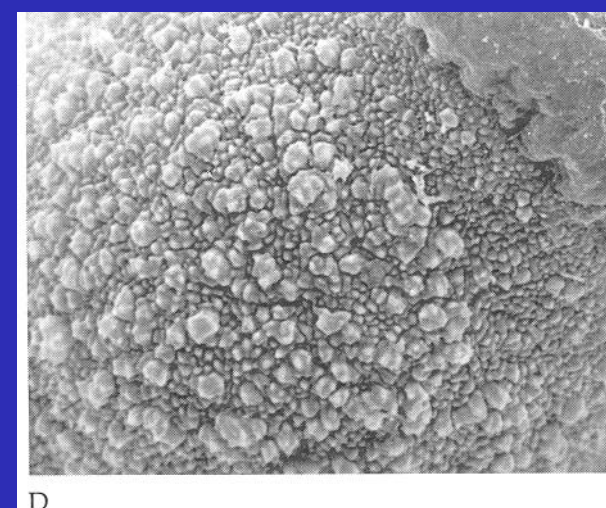


Gold





Red-oak pollen



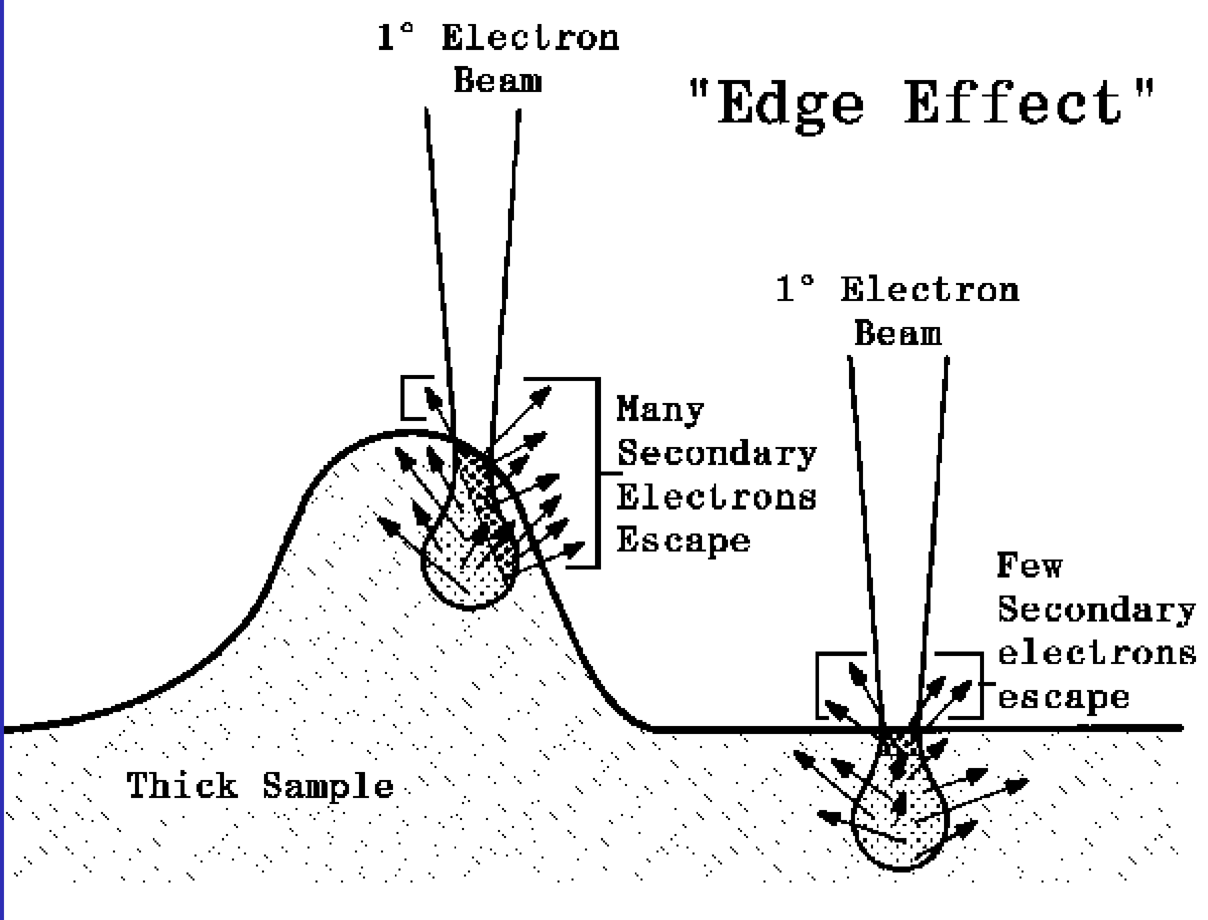
35 kV

10 kV

SEM – Secondary electrons (SE) - SOURCE

- Produced by inelastic interactions.
- SE energy level is only 3 ~ 5 eV.
- Can be easily collected
- The maximum escape depth is about 5 nm in metal and 50 nm in insulators.

SEM FUNDAMENTALS: EDGE EFFECT



SEM – Backscattered electrons (BSE) - SOURCE

- Produced by elastic interactions.
- BSE energy is about 60 ~80 % of incident one.
- Special detector is required to collect BSE.
- The maximum escape depth varies inversely with the average atomic number
- The backscattering coefficient η increases with increasing atomic number Z.

SEM – UTILIZATION

- **Contrast** is the ratio of the change in signal between any two points on the specimen and the average signal.

- ***Topographic Contrast: -- mainly given by secondary electron signal,*** which is sensitive to the surface structure of specimens.

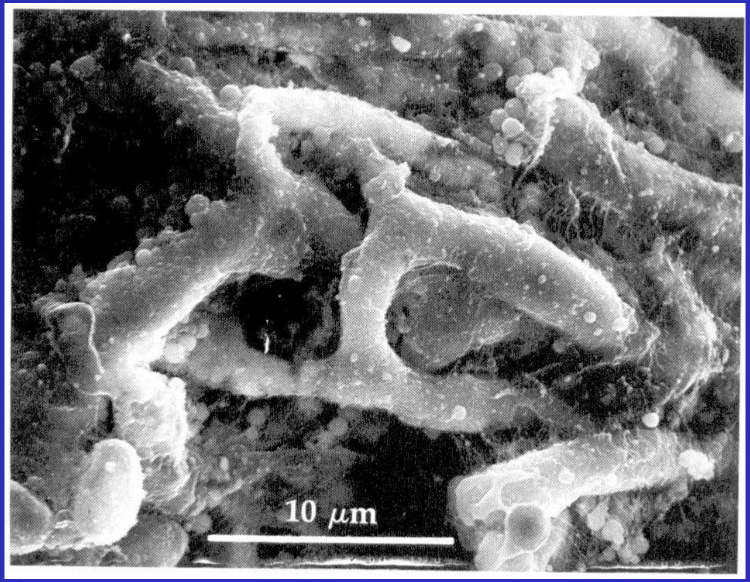
SEM – UTILIZATION

- ***Compositional Contrast:* -- given by the backscattered electron signal,** which change with the differences in atomic number, i.e., nuclear charge of the atoms composing the specimen.

- The higher the atomic number**, the greater the backscattering.** In smooth plane surfaces we are dealing entirely with compositional contrast

SEM Images: Contrast Mechanisms

Secondary e^-



Backscattered e^-

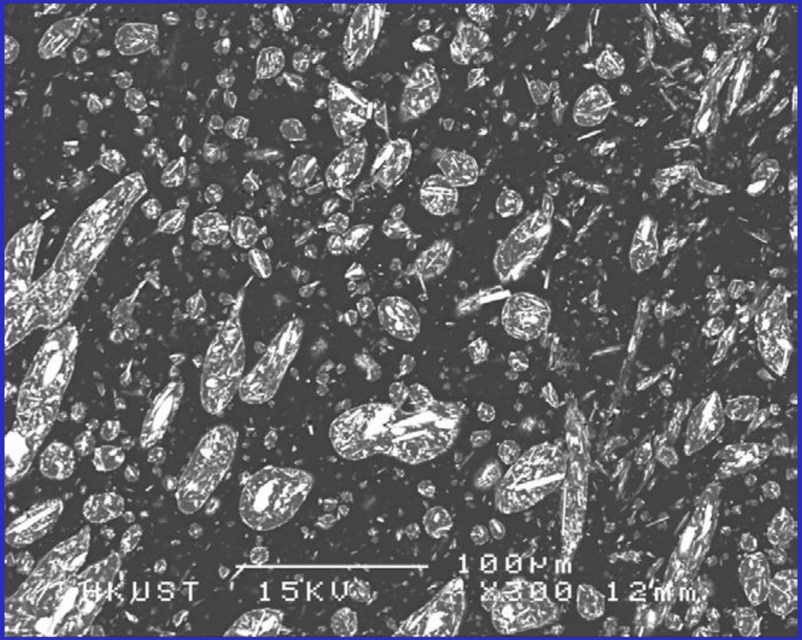
Fungal hyphae with Ag preferentially deposited at polysaccharides

Secondary electrons indicate sample topography, whereas backscattered electrons indicate sample composition.

Comparison of SE and BSE Images

BSE image: Provides the atomic number contrast (also lower resolution of topographic contrast).

SE Image



BSE Images

Contrast Development in the SEM

Major Influences on Contrast

i. the incidence angle of impinging electrons onto the specimen.

ii. the collecting angle from the specimen to the positively charged collector.

Contrast Development in the SEM

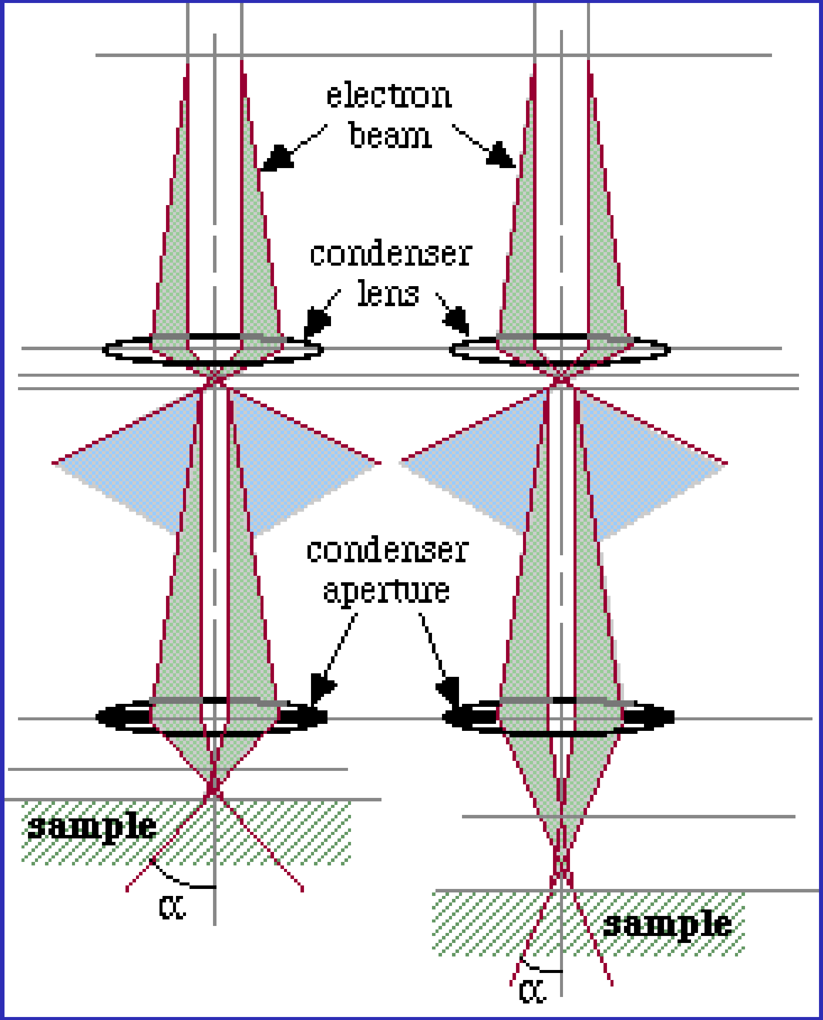
Machine Variables for Contrast Improvement:

i. Reducing working distance

ii. Using smaller aperture

iii. Using larger electron beam size

Contrast Development in the SEM



SEM – CHARACTERISTIC X-RAY – SOURCE

- ***Caused by the de-energization of the specimen atom after a secondary electron is produced.***
- ***Since a lower (usually K-shell) electron was emitted from the atom during the secondary electron process an inner (lower energy) shell now has a vacancy.***
- ***A higher energy electron can "fall" into the lower energy shell, filling the vacancy.***
- ***As the electron "falls" it emits energy, usually X-rays to balance the total energy of the atom so it***

SEM – CHARACTERISTIC X-RAY – UTILIZATION

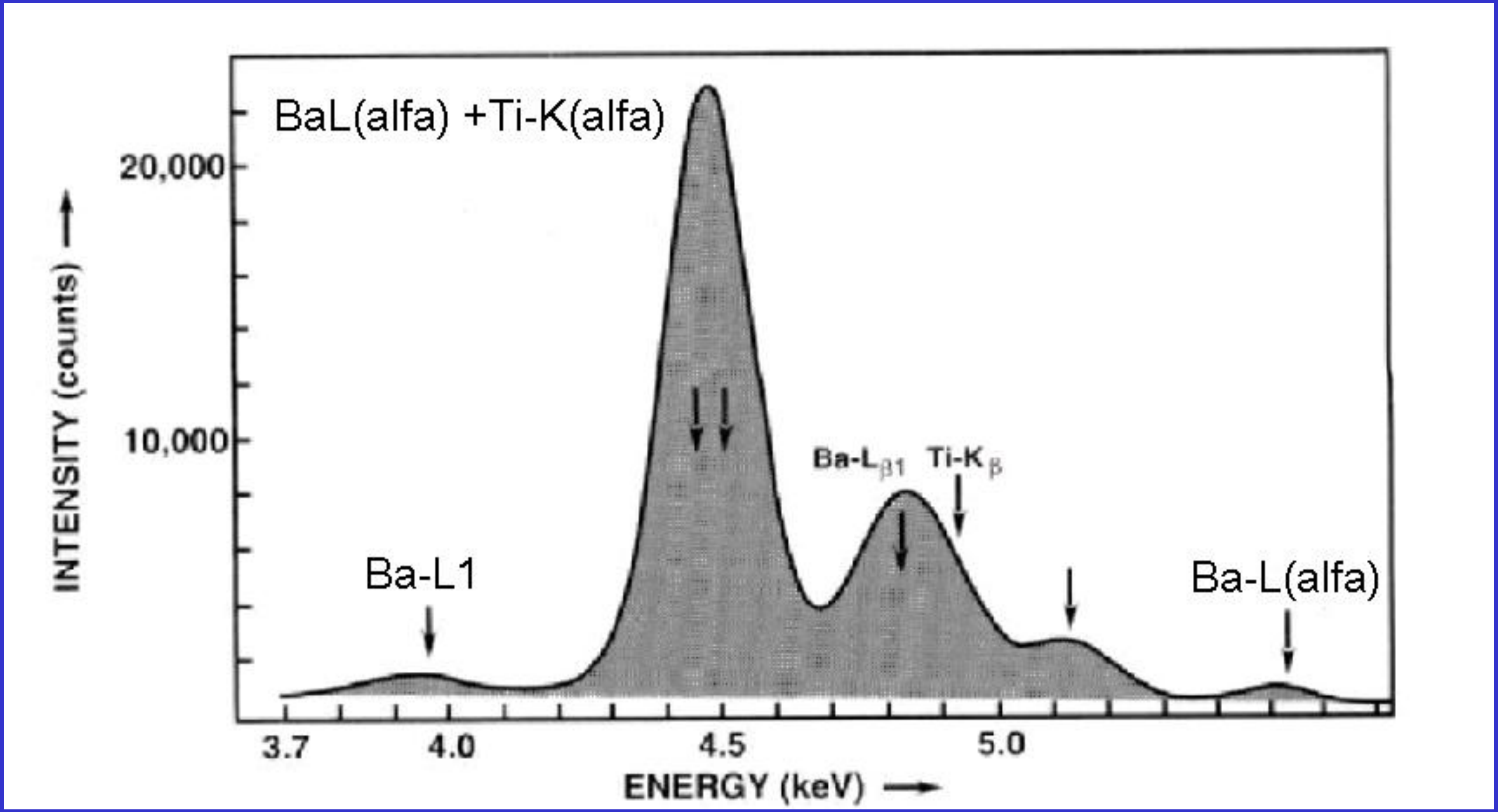
- ***X-rays or Light emitted from the atom will have a characteristic energy which is unique to the element from which it originated.***

- ***These signals are collected and sorted according to energy to yield micrometer diameter) of bulk specimens limiting the point-to-point comparisons available***

SEM – CHARACTERISTIC X-RAY – UTILIZATION

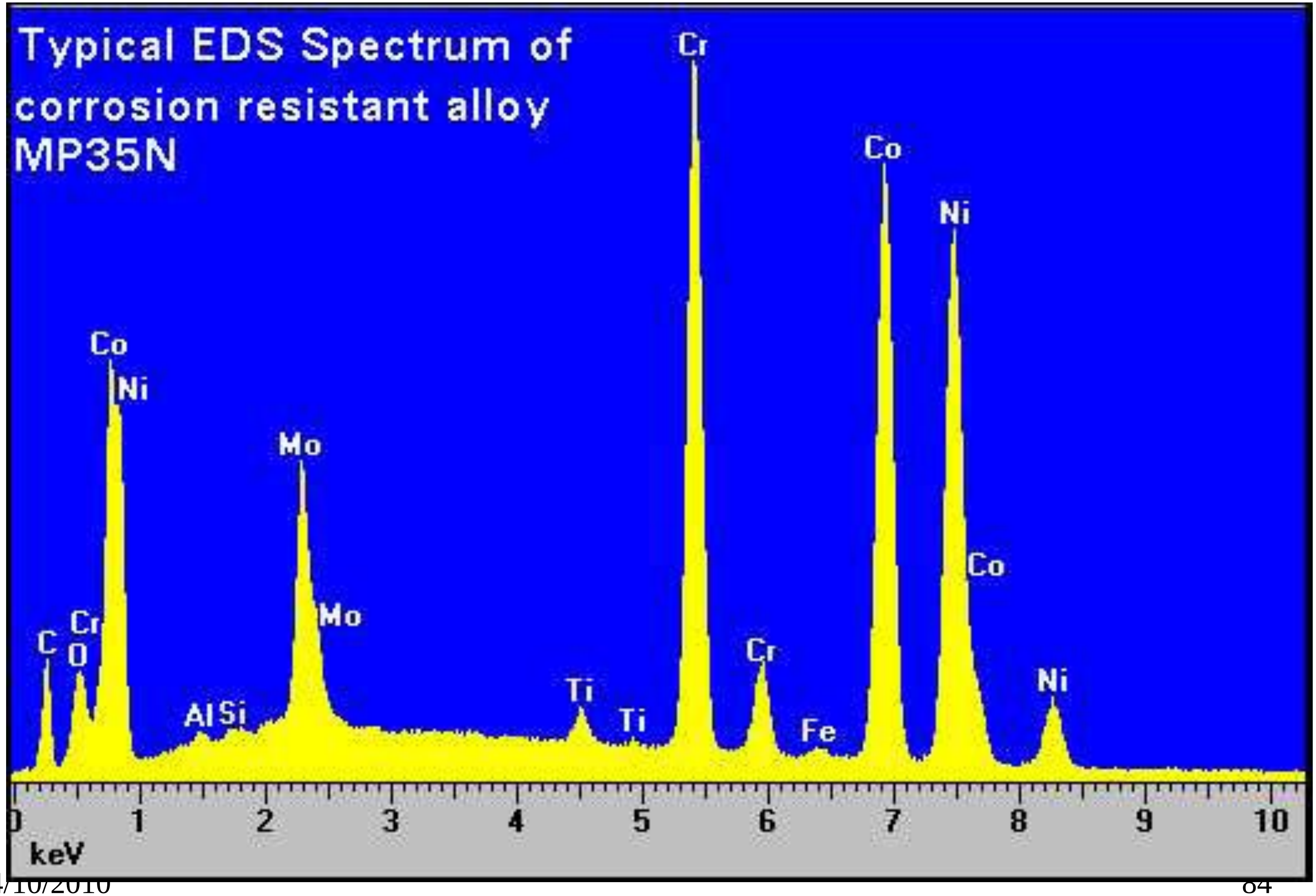
- Large escape depth due to difficulty of x-ray absorption
- Energy and wavelength of x-ray can be used for chemical analysis:
- Energy-dispersive spectroscopy, EDS
- Wavelength-dispersive spectroscopy, WDS

EDS SPECTRA

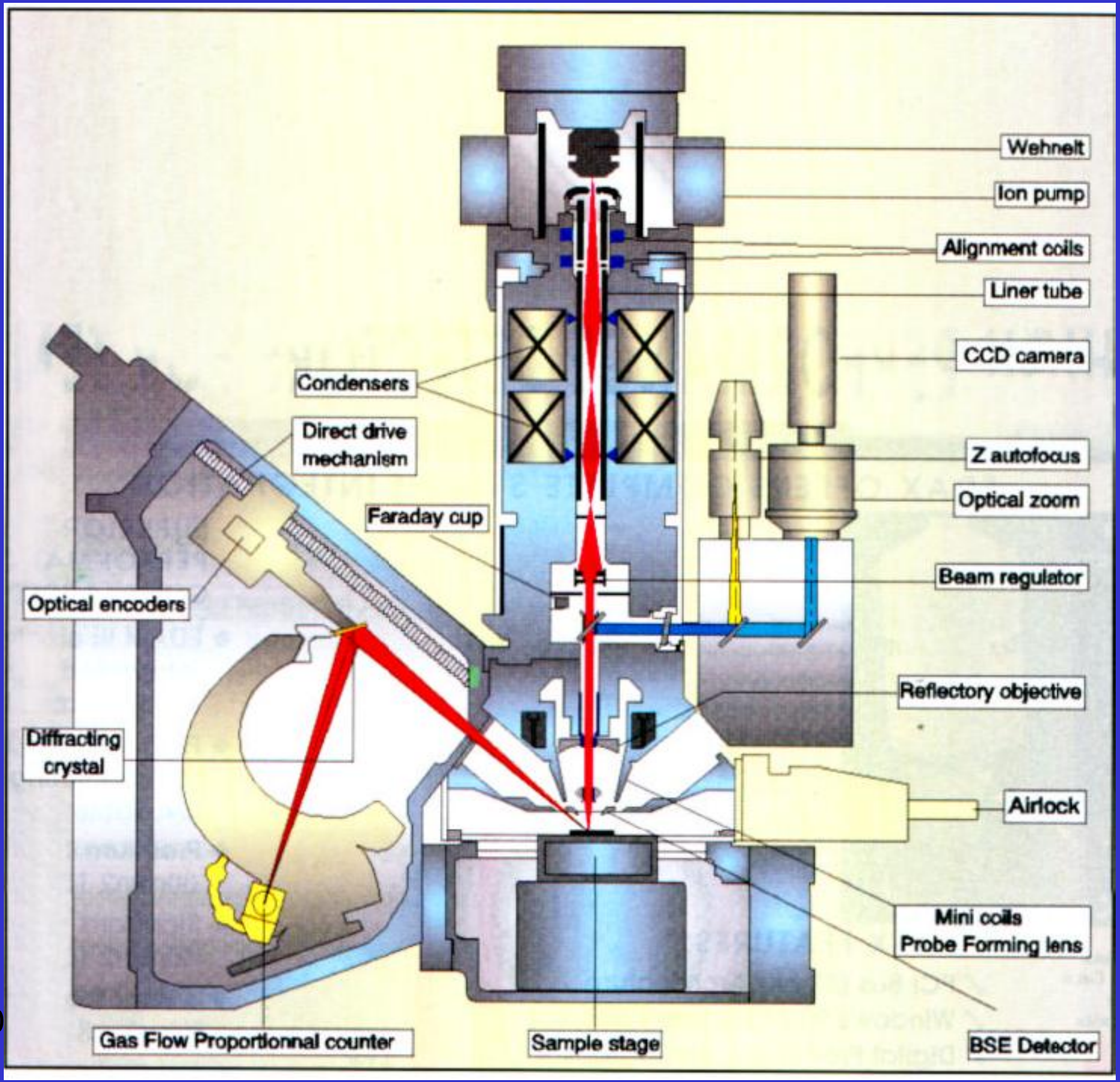


BaL(alfa) +Ti-K(alfa)
20,000
10,000
INTENSITY (counts)
Ba-L1
Ba-Lβ1
Ti-Kβ
Ba-L(alfa)
3.7
4.0
4.5
5.0
ENERGY (keV)

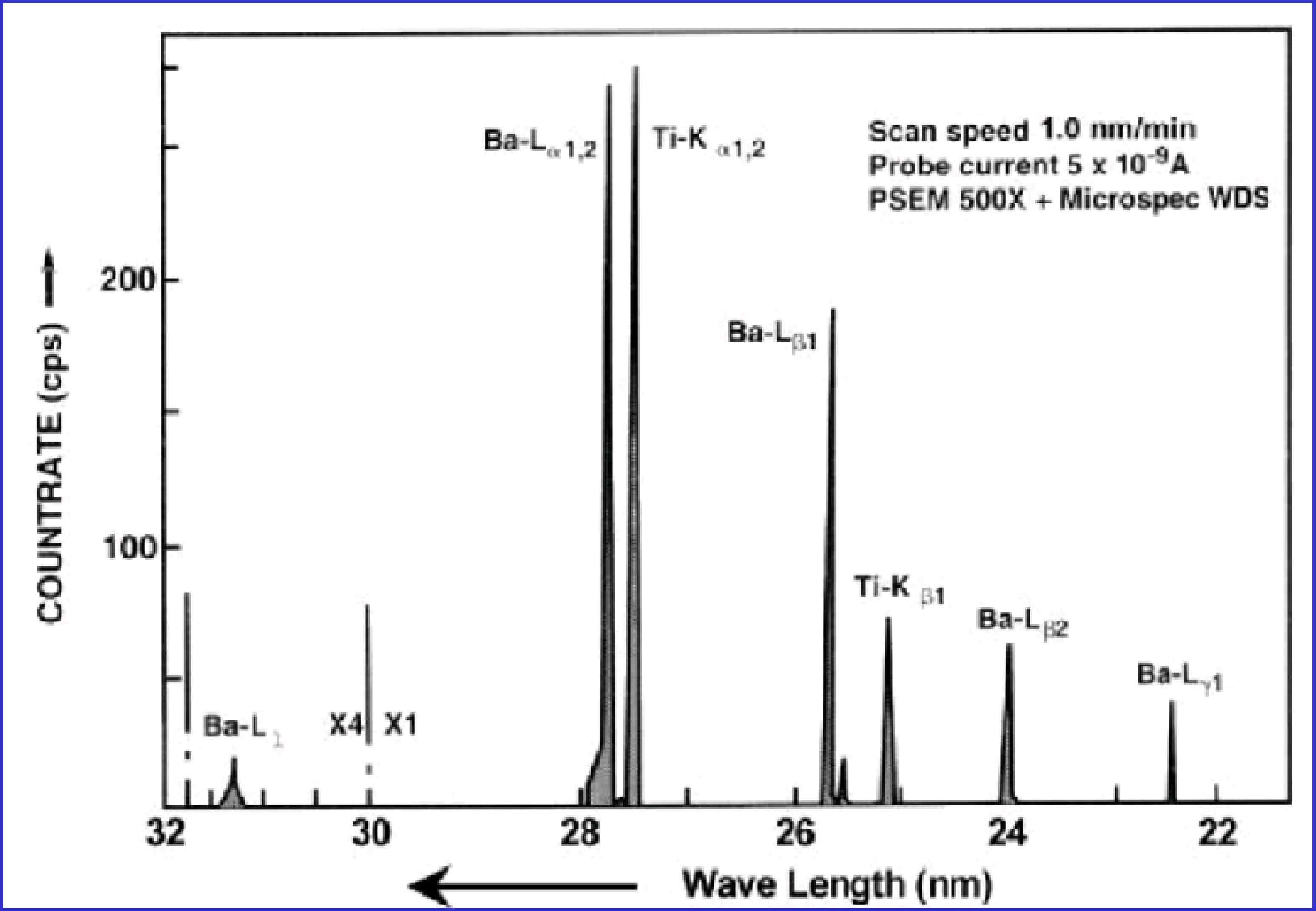
EDS EDS spectrum for corrosion resistant alloy MP35N



SEM WDS

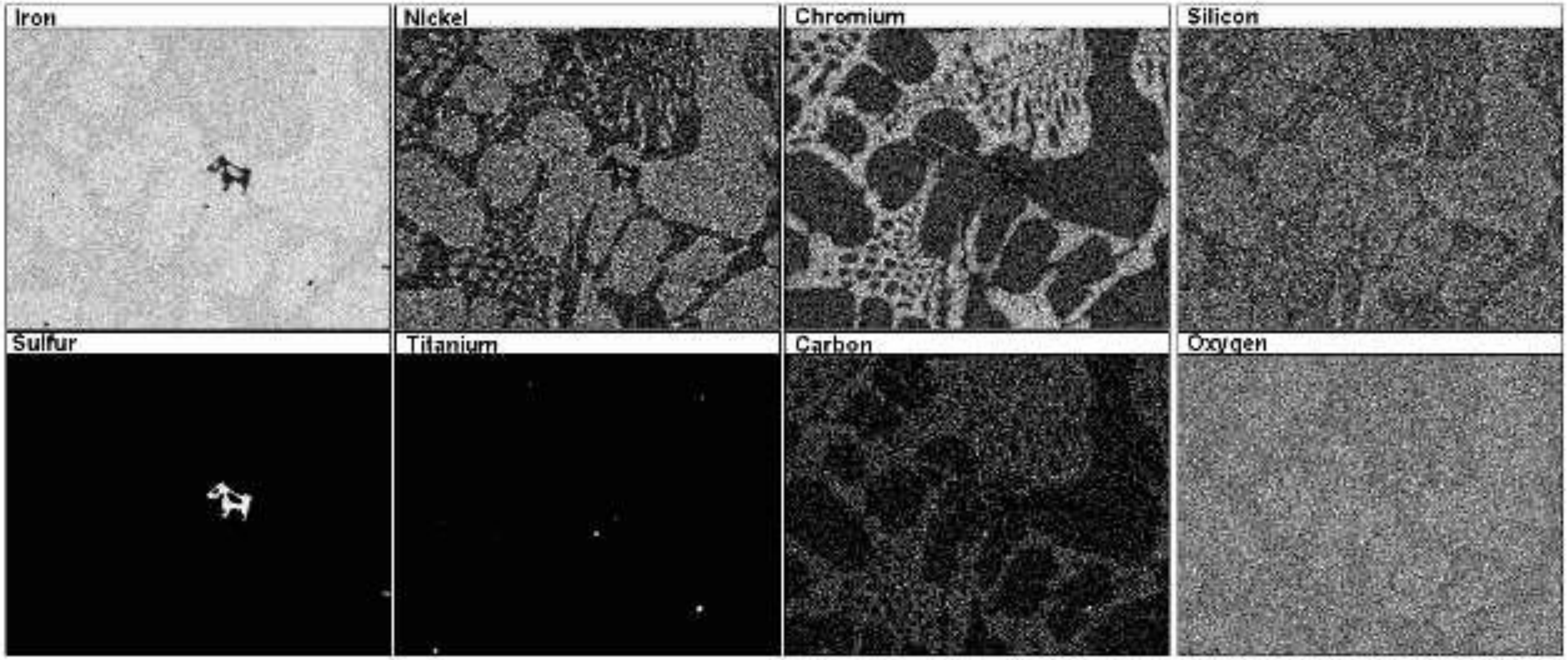

Wehnelt
Ion pump
Alignment coils
Liner tube
CCD camera
Condensers
Direct drive mechanism
Z autofocus
Optical zoom
Faraday cup
Beam regulator
Optical encoders
Reflectory objective
Diffracting crystal
Airlock
Mini coils
Probe Forming lens
Gas Flow Proportionnal counter
Sample stage
BSE Detector

WDS SPECTRA

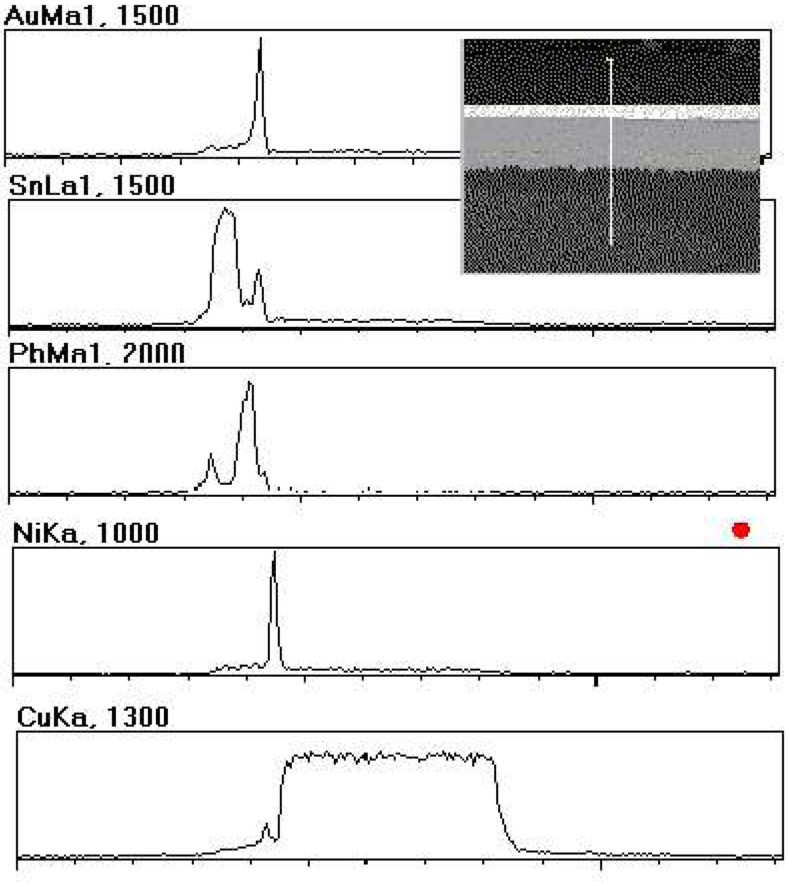


Scan speed 1.0 nm/min
Probe current 5 x 10-9A
PSEM 500X + Microspec WDS
Ba-Lα1,2
Ti-K α1,2
Ba-Lβ1
Ti-K β1
Ba-Lβ2
Ba-Lγ1
Ba-L
X4
X1
200
100
COUNTRATE (cps)
32
30
28
26
24
22
Wave Length (nm)

EDS Elemental map of polished metallographic cross-section of high-chromium white-iron casting



EDS EDS line profile

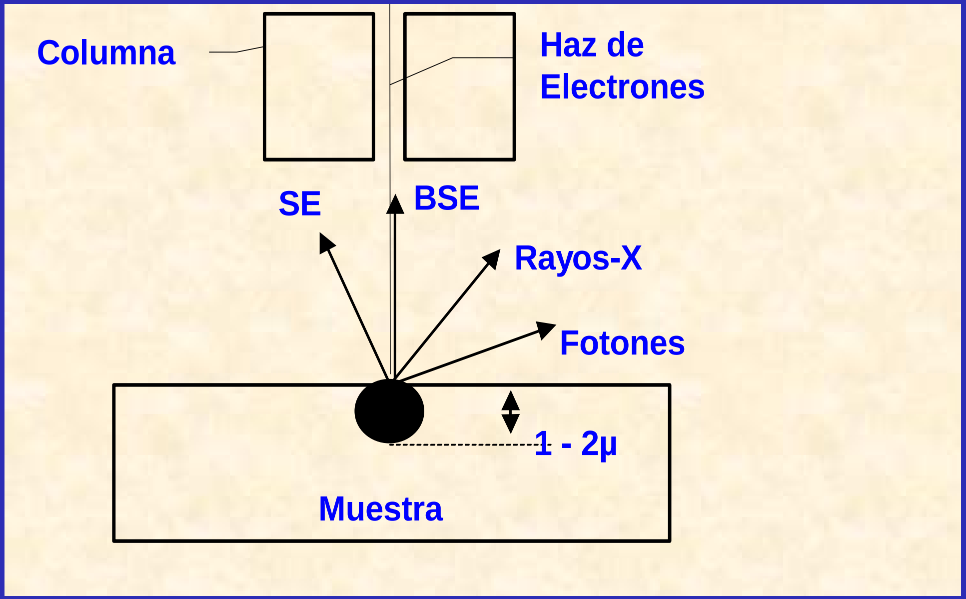


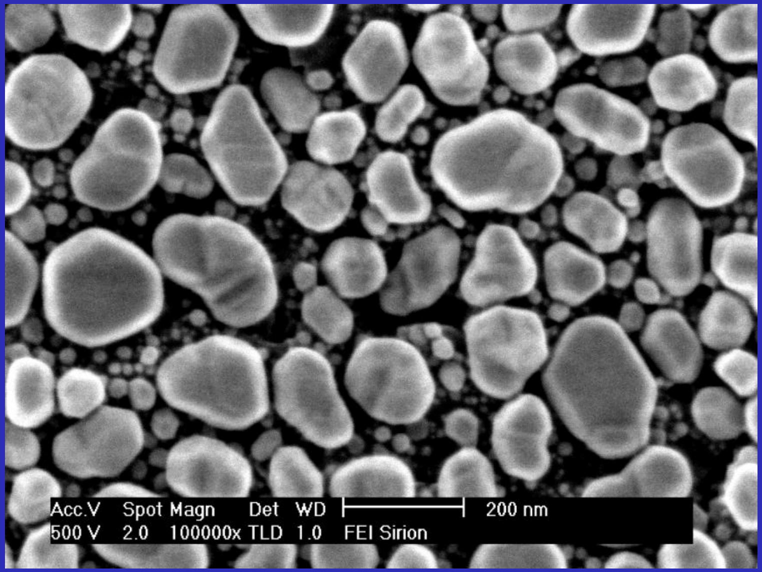
Means To Improve Resolution

- using secondary electron (SE) image, as the optimum resolutions are: 5 nm for SE; 25 nm for BSE and 2 μm for X-ray mapping
- long exposure times, leading to more electrons being detected
- reducing lens aberrations to minimum

Haz de Electrones = SEM

- El microscopio electrónico de barrido nos permite obtener información superficial de la muestra.
  - Caracterización con electrones secundarios, electrones retro-dispersados y rayos-X.

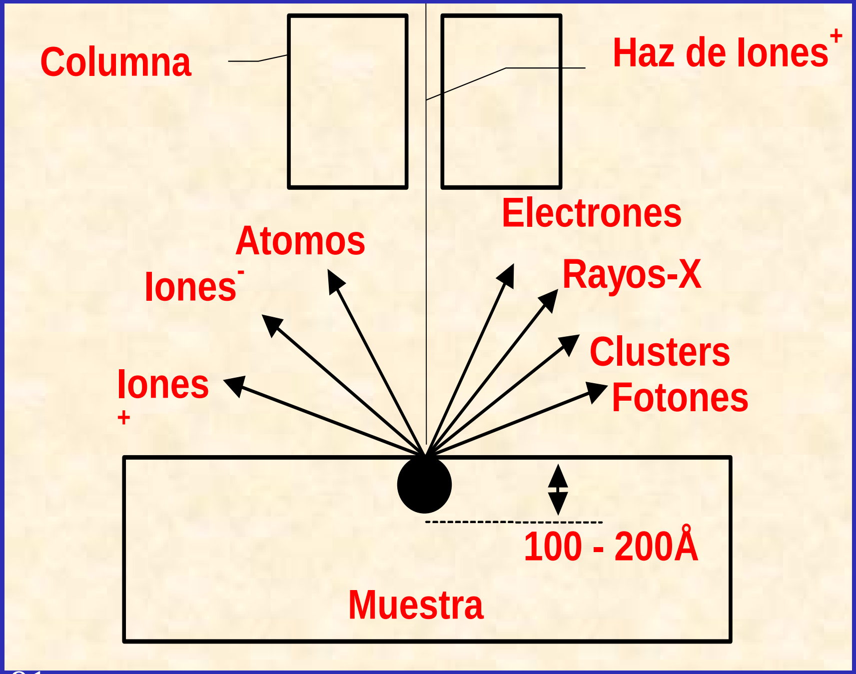


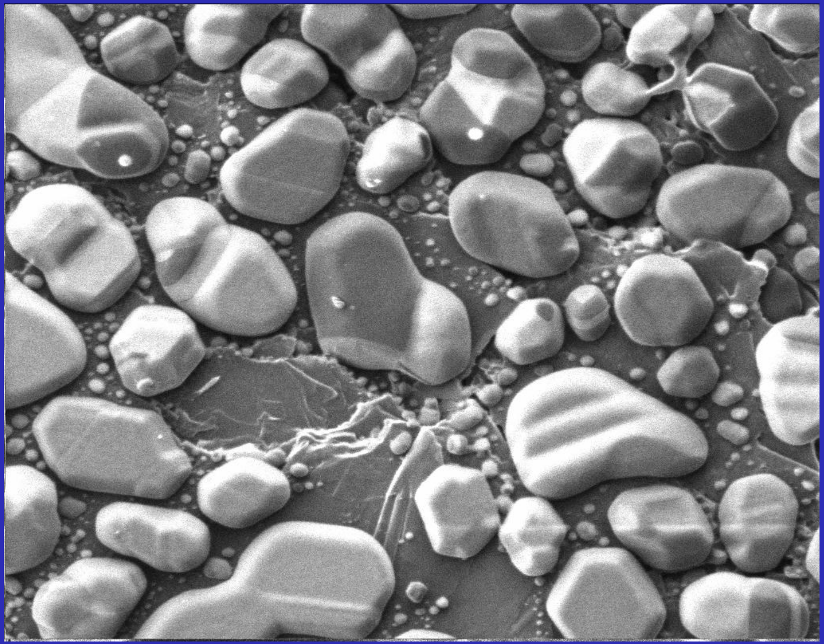


Haz de Iones = FIB

El FIB o microscopio iónico de barrido nos permite obtener además de la información superficial, información en la tercera dimensión.

Caracterización con electrones secundarios, iones secundarios (contraste por canalización)





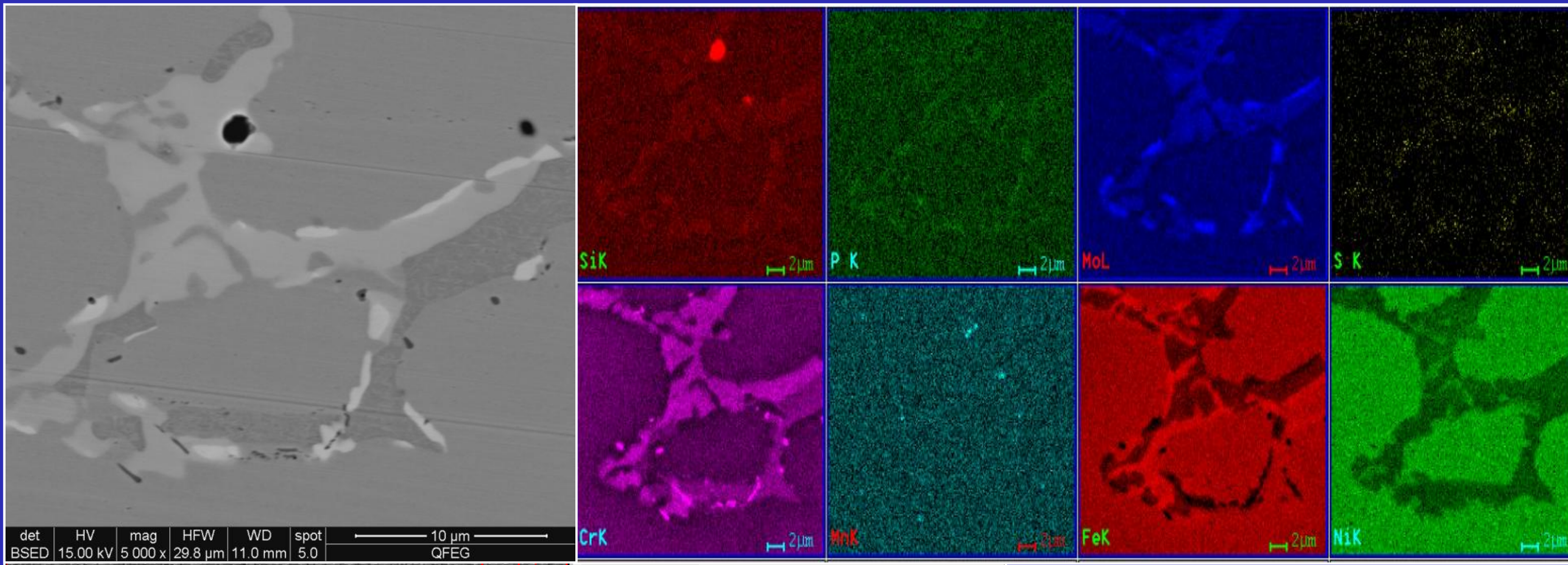
SEM - FIB

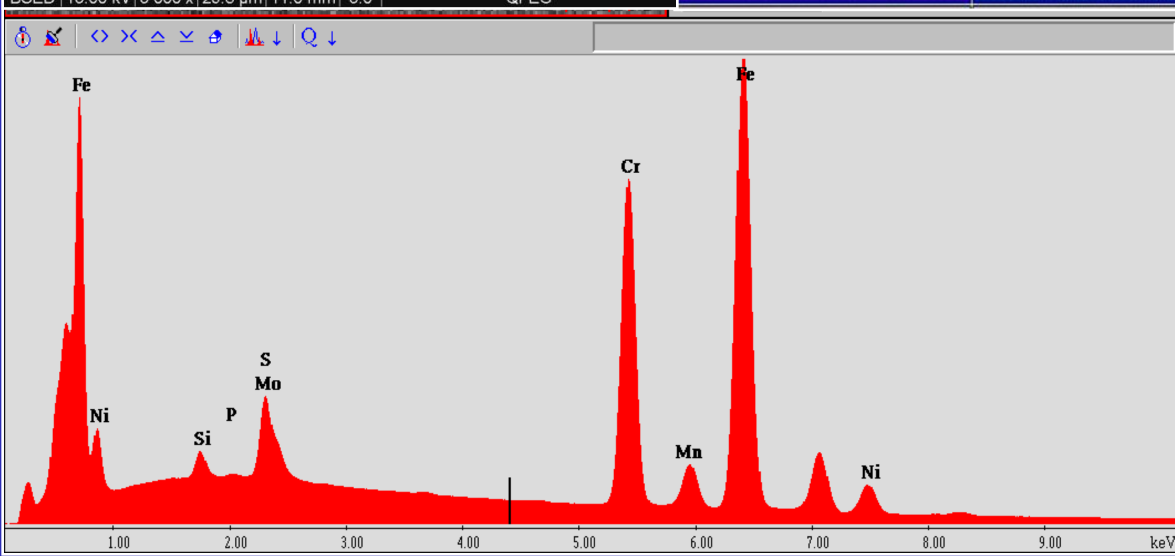
- Los electrones son reemplazados por iones
- Los iones son más grandes que los electrones
- No penetración dentro del material no ionización
  - No se generan rayos-X
- Los iones son más pesados que los electrones
  - Mueven átomos y los remueven (efecto sputtering / devastado)
- Los iones tienen carga positiva – los electrones carga negativa
  - Su uso simultáneo evita el efecto de carga

¿Qué hace un FIB que no hace un SEM?

- Remueve material
- La imagen de electrones secundarios muestra contraste en el material
- Ofrece imagen de contraste por canalización
- Prepara muestras TEM in-Situ

¿Qué hace un SEM que no hace un FIB?

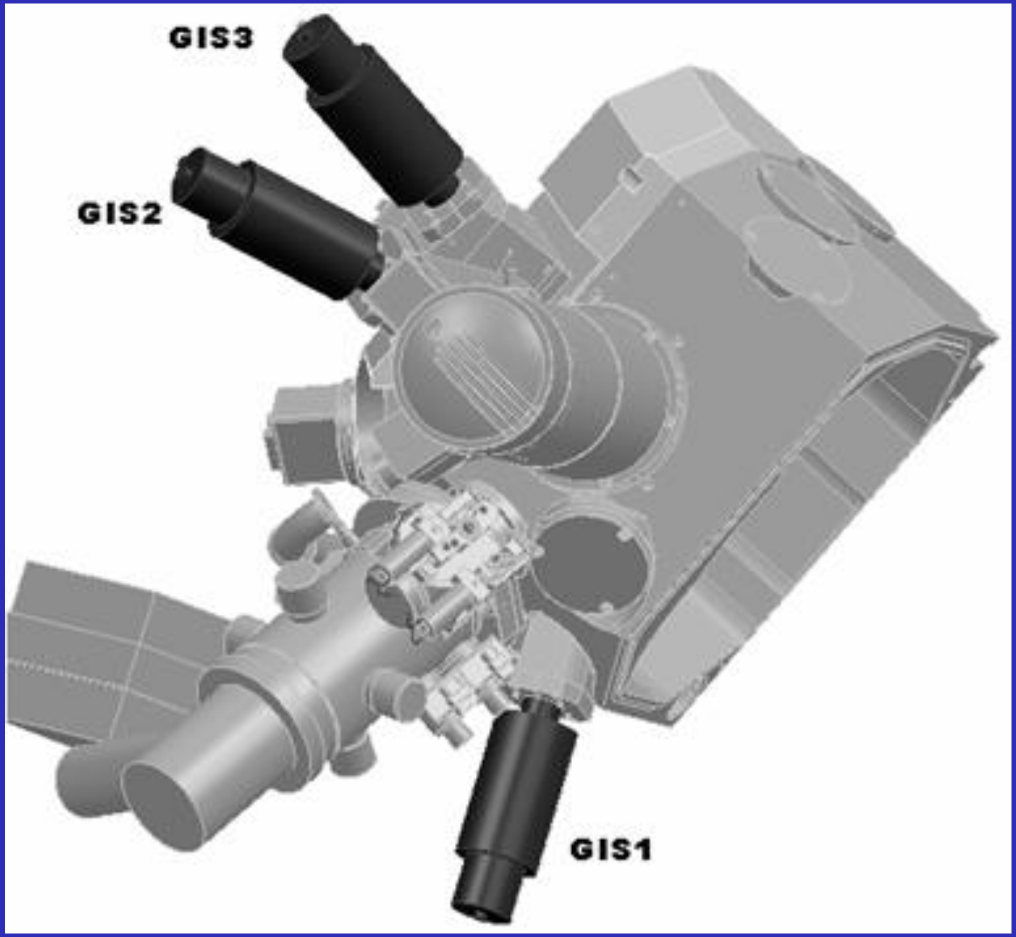




Element	Wt %	At %
C K	3.7	15.14
SiK	0.65	1.14
P K	0.1	0.16
MoL	4.86	2.49
CrK	23.88	22.56
MnK	0.25	0.23
FeK	59.99	52.78
NiK	6.56	5.49
Total	100	100

Dual Beam

SEM y FIB combinados



Dual Beam

- SEM y FIB combinados
  - Imagen generada con el SEM
  - Efecto sputtering con FIB
- Posibilita la exploración en 3 dimensiones
  - Información debajo de la superficie
  - Caracterización de material y su estructura
- Un punto de coincidencia
- Imagen simultánea y remoción de material (modo SPI)

Dual Beam

Vista interna cámara de muestras DualBeam

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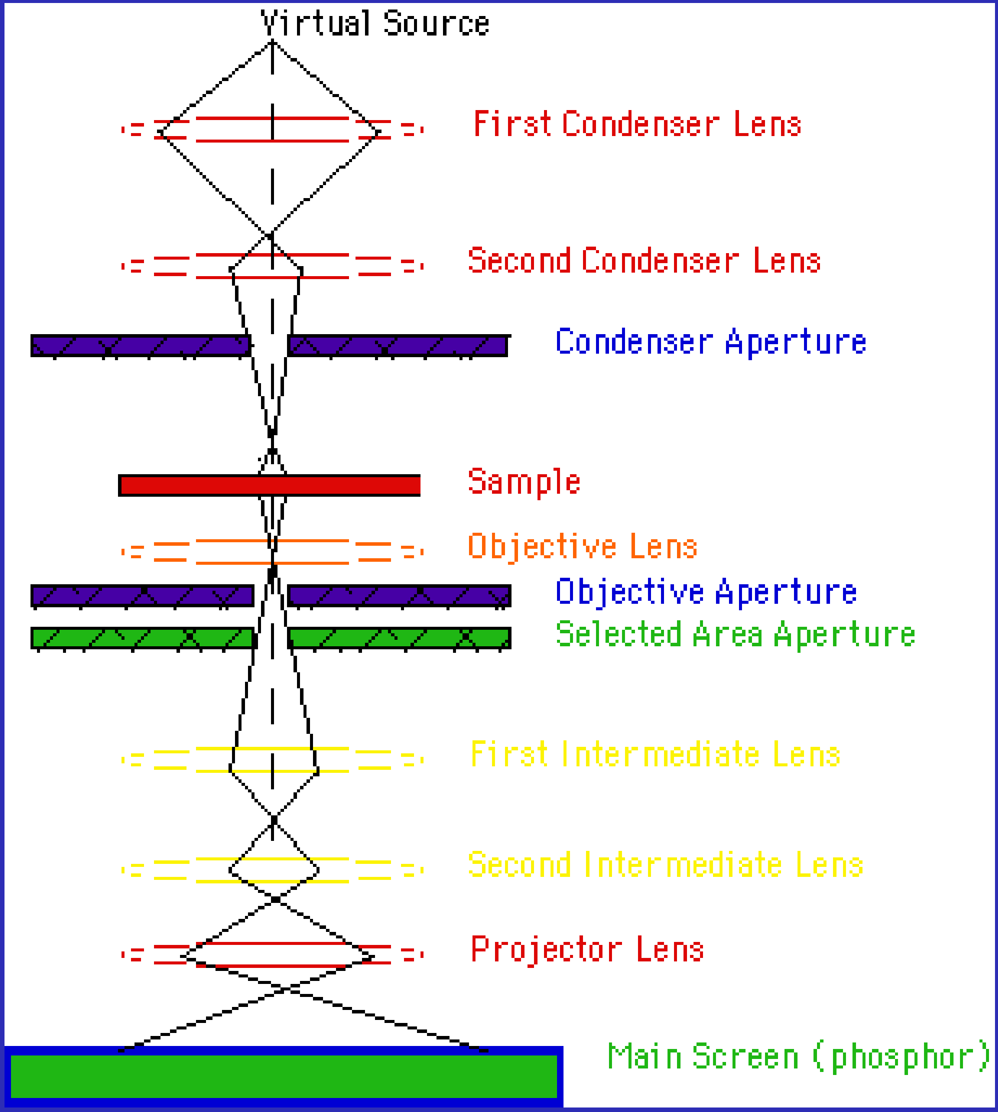
- High Resolution Electron Microscope HRTEM

TRANSMISSION ELECTRON MICROSCOPES

DESIGN

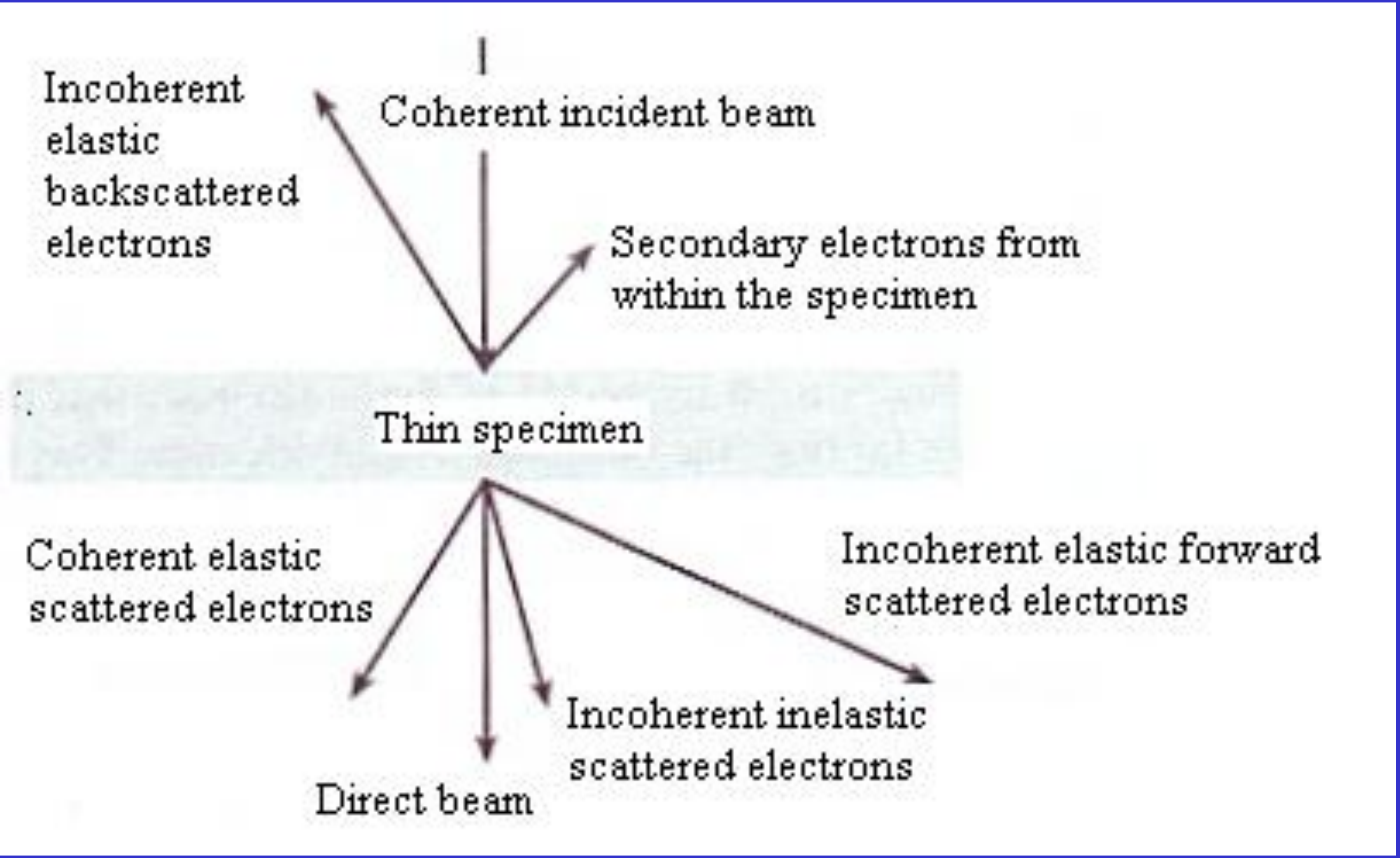
http://mse.iastate.edu/microscopy/path2.html

TEM SKETCH



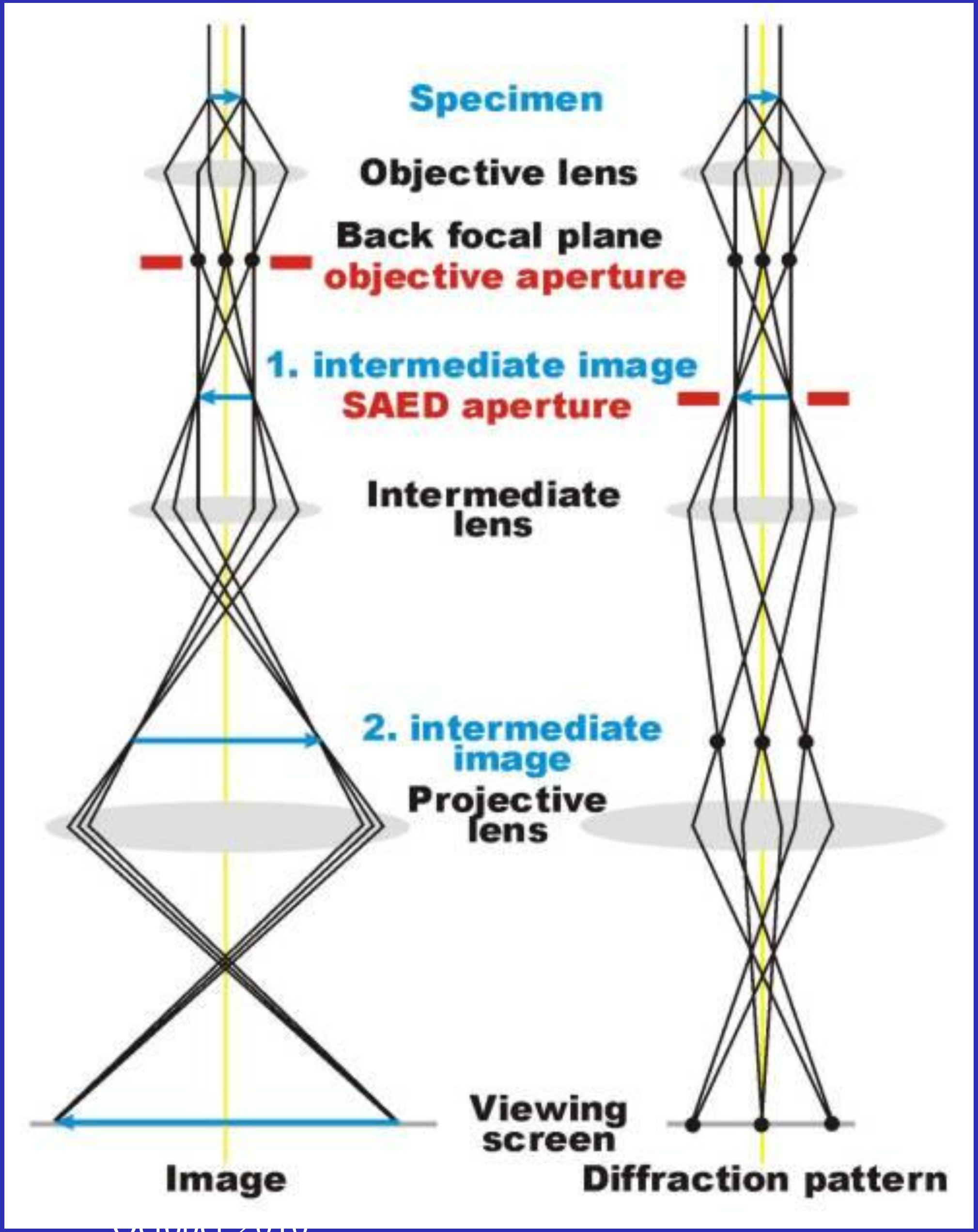
http://mse.iastate.edu/microscopy/path2.html

SOME FUNDAMENTAL PROPERTIES OF ELECTRONS (4)



ELECTRON MICROSCOPE

- Electrons emitted from a nearly perfect point source (the space charge)
- Electrons for imaging will all have similar energies (monochromatic)
- Only electrons nearly parallel to the optic axis will be allowed out of the gun area



Elastically Scattered electrons: Utilization

Elastically Scattered electrons: Source

Incident electrons that are scattered (deflected from their original path) by atoms in the specimen in an elastic fashion (no loss of energy). These scattered electrons are then transmitted through the remaining portions of the specimen

Elastically Scattered electrons: Utilization

All electrons follow Bragg's Law: each spot corresponding to a specific atomic spacing (a plane)

This pattern can then yield information about the orientation, atomic arrangements and phases present in the area being examined

Mechanisms of Image Formation

Scattering and diffraction of electrons is mainly responsible for image formation.
Absorption of electrons plays a minor role in image formation.

Mechanisms of Image Formation

- Mass-thickness contrast (biology and polymer specimens)
- Diffraction contrast (major image formation mechanisms)
- Phase contrast (high resolution image of crystal lattice)

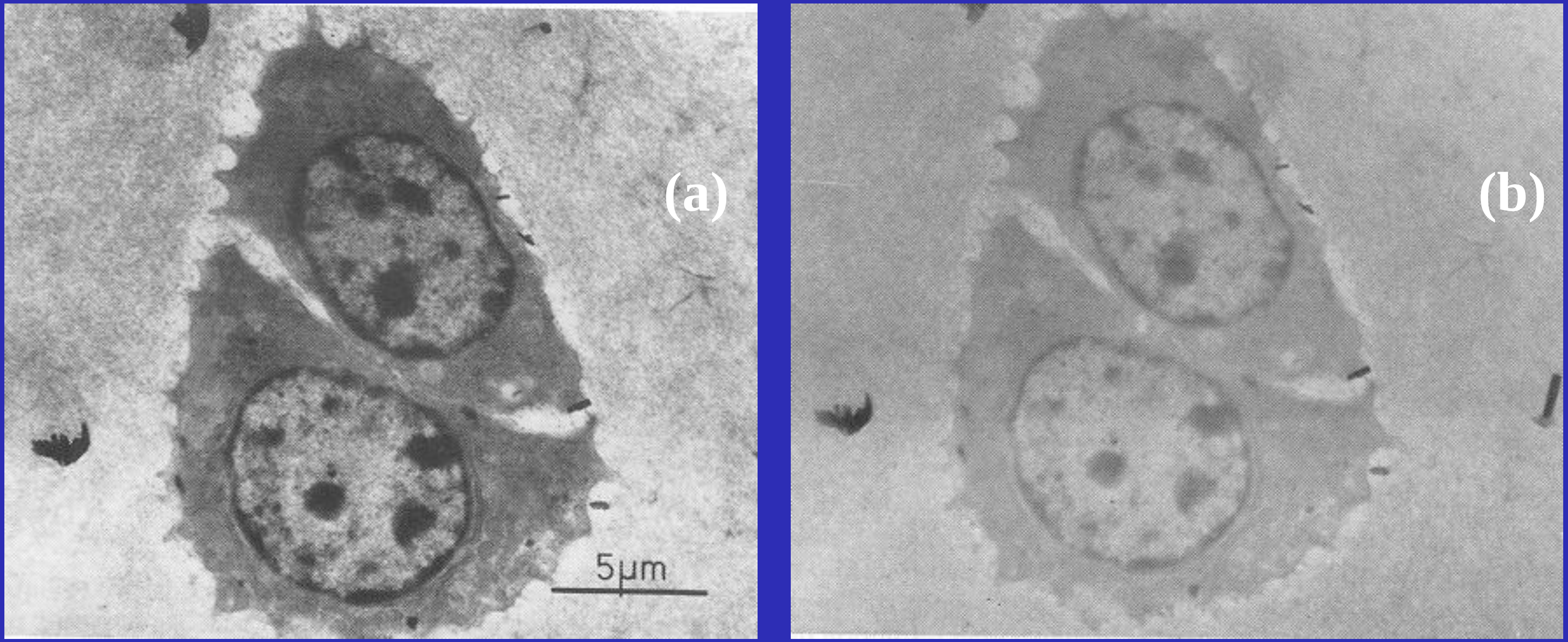
Mass-thickness contrast

The factors affecting electron scattering

- atomic number of specimen atoms
- thickness of specimen
- density of specimen
- opening of objective aperture (size of aperture)
- energy of electrons (acceleration voltage)

Mass-thickness contrast

Using a objective aperture can enhance the mass-thickness contrast



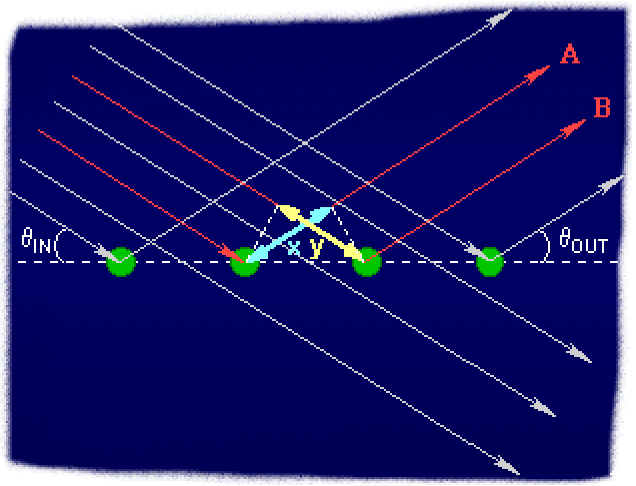
(a) with and (b) without an objective aperture in position

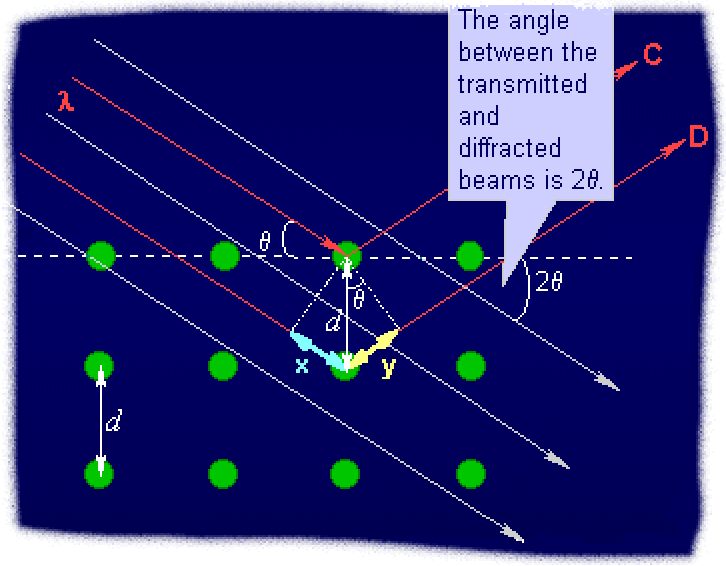
Diffraction contrast

The constructive interference occurs when the Bragg equation is obeyed.

$$n\lambda = 2d \sin \theta$$

Diffraction: the Bragg Law





Diffraction is an interference effect which leads to the scattering of strong beams of radiation in specific directions.

- Diffraction from crystall is described by

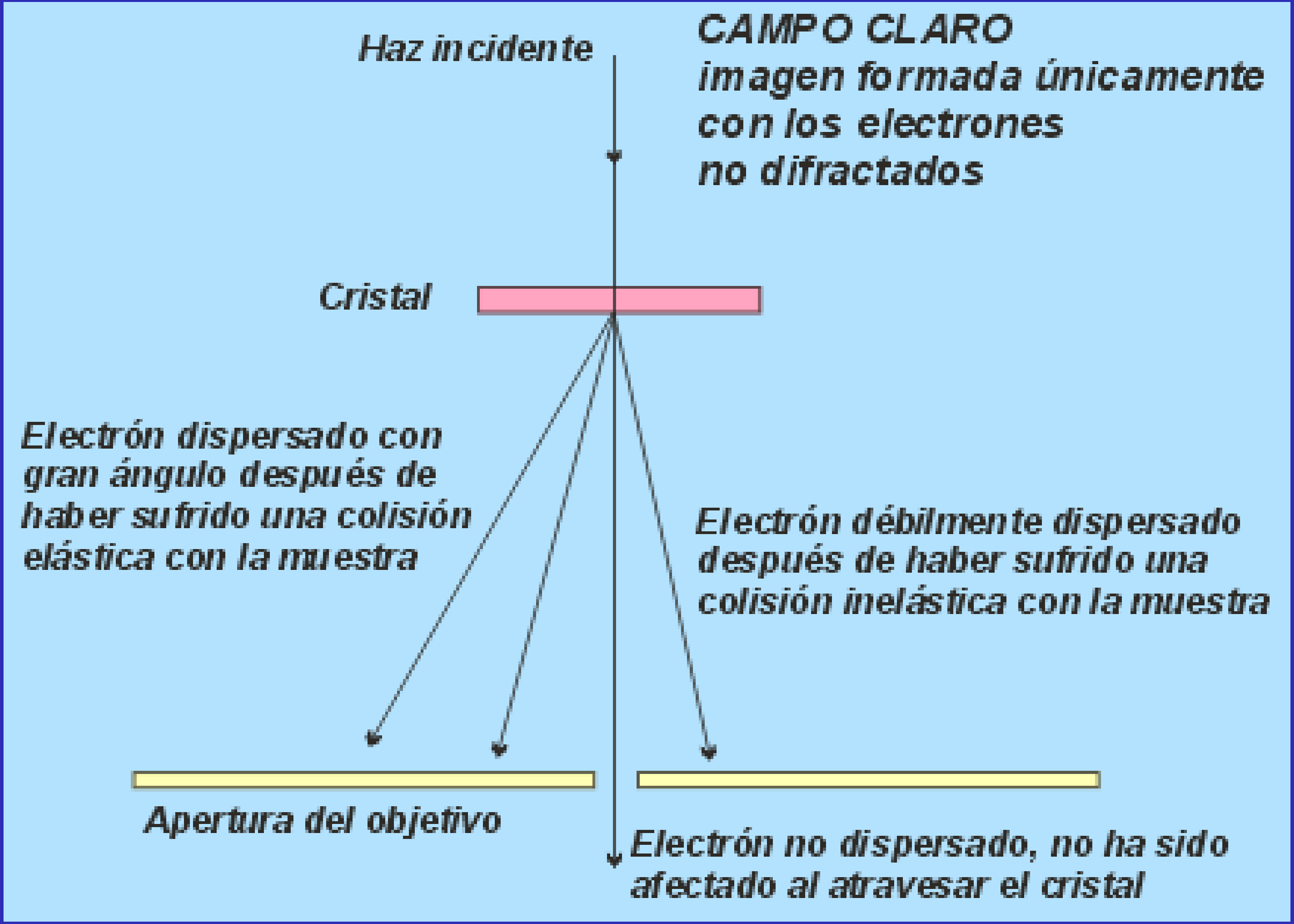
$$n\,\lambda = 2\,d_{hkl}\sin\theta$$

where n is an integer (the order of scattering), λ is the wavelength of the radiation, d_{hkl} is the spacing between the scattering entities (e.g. planes of atoms in the crystal) and θ is the angle of scattering

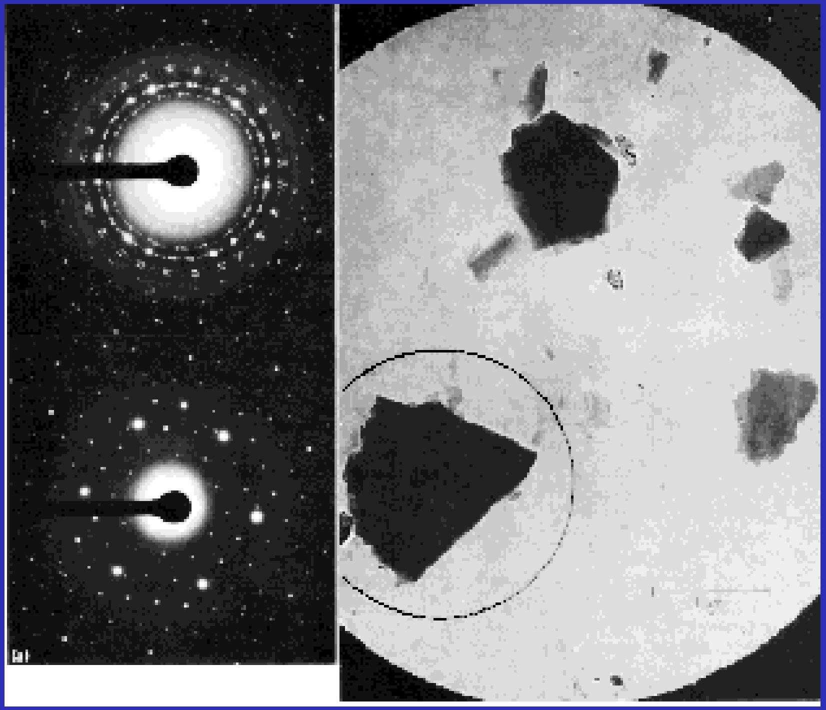
Diffraction contrast

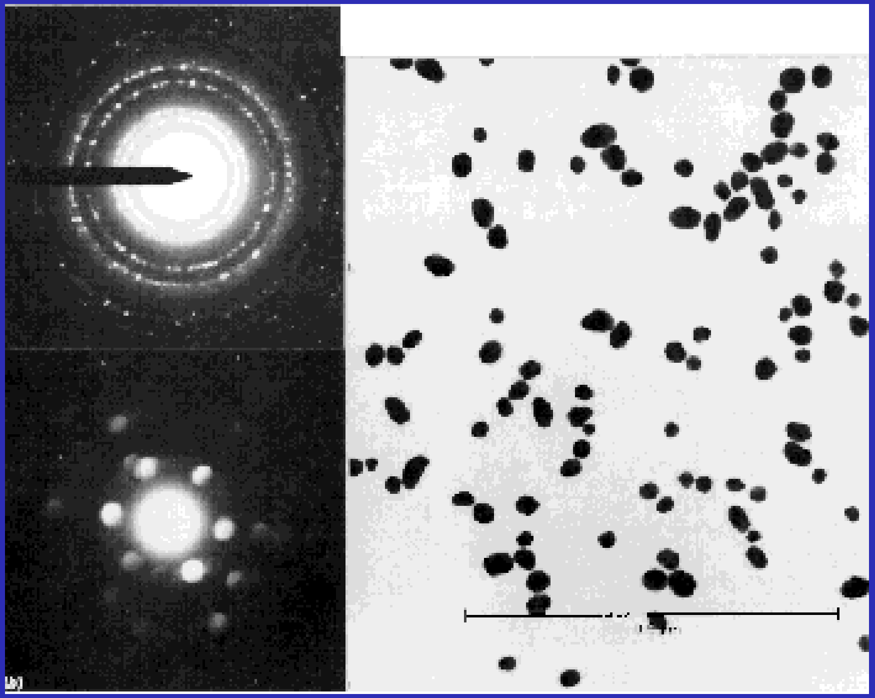
- The contrast is generated when the electron beam does not pass through aperture due to the Bragg diffraction of crystalline specimen.
- When the Intensity Ir is removed from the primary beam Io by the Bragg reflection, the image of the respective sample area appears dark.

DIFFRACTION PATTERN(1)

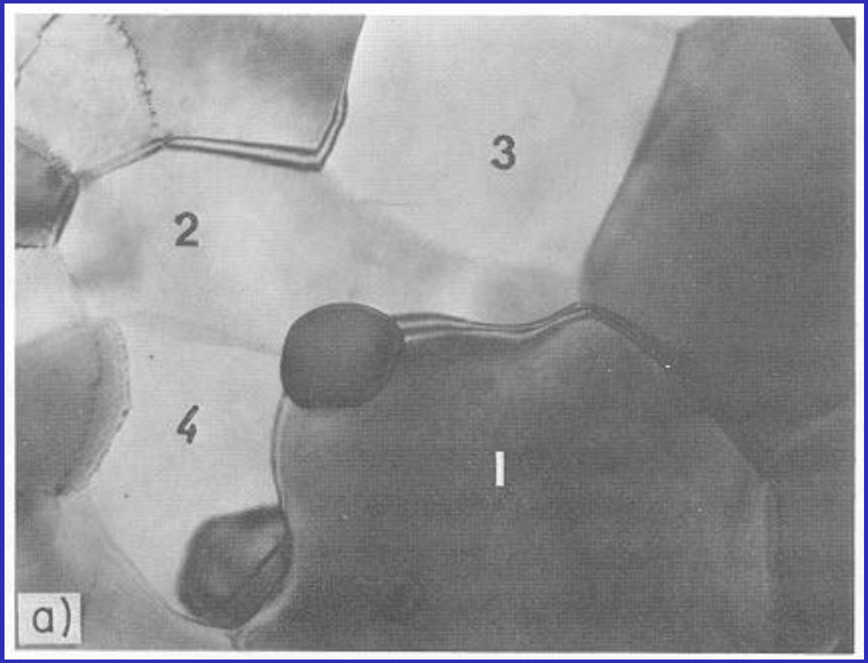


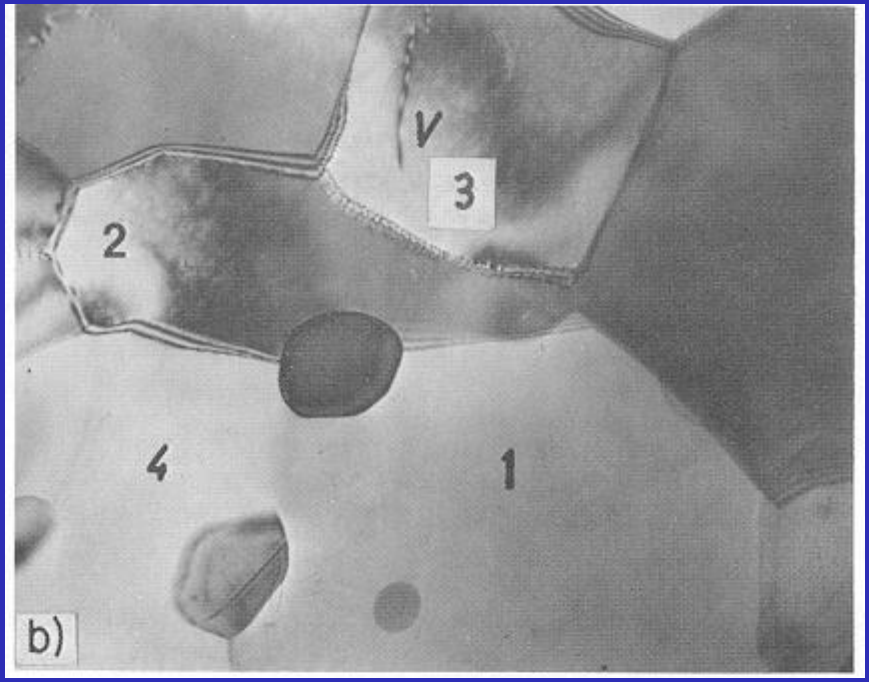
ELECTRON MICROSCOPE: OBJECTIVE LENS





Diffraction contrast: examples





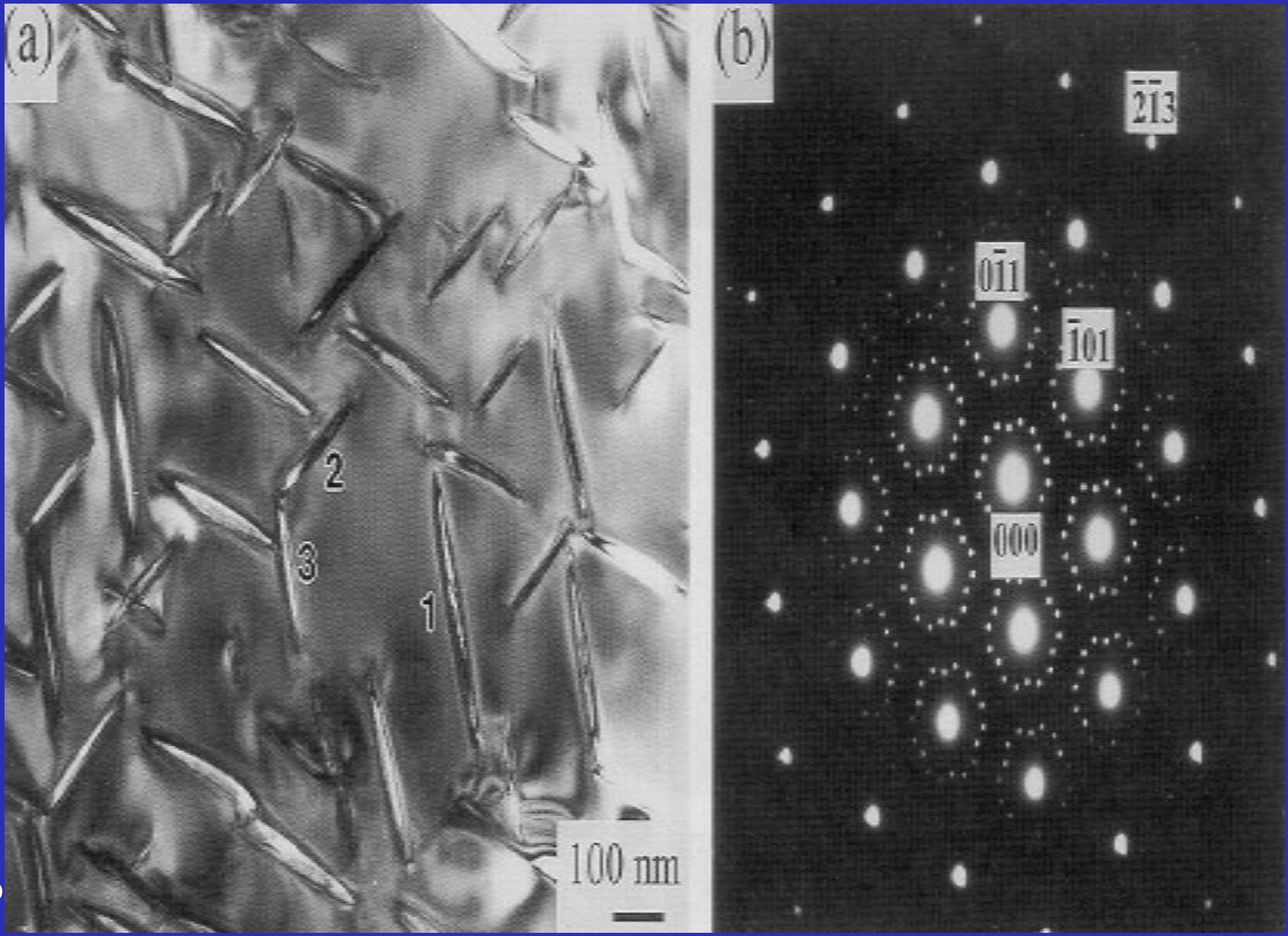
Titling the specimen changes the contrast of grains

Diffraction contrast: examples

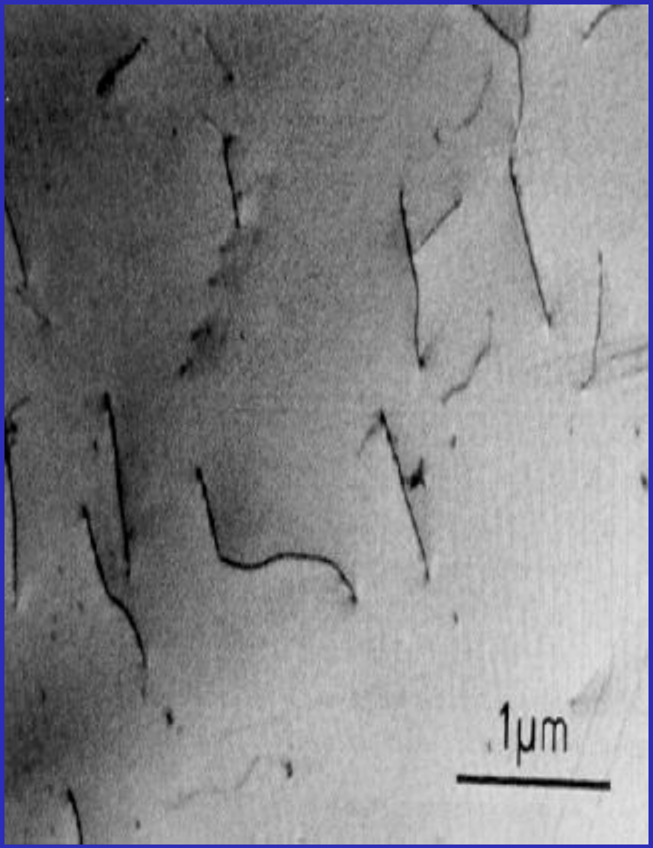
The amorphous and crystalline phases are clearly seen in a semi crystalline polymer thin film

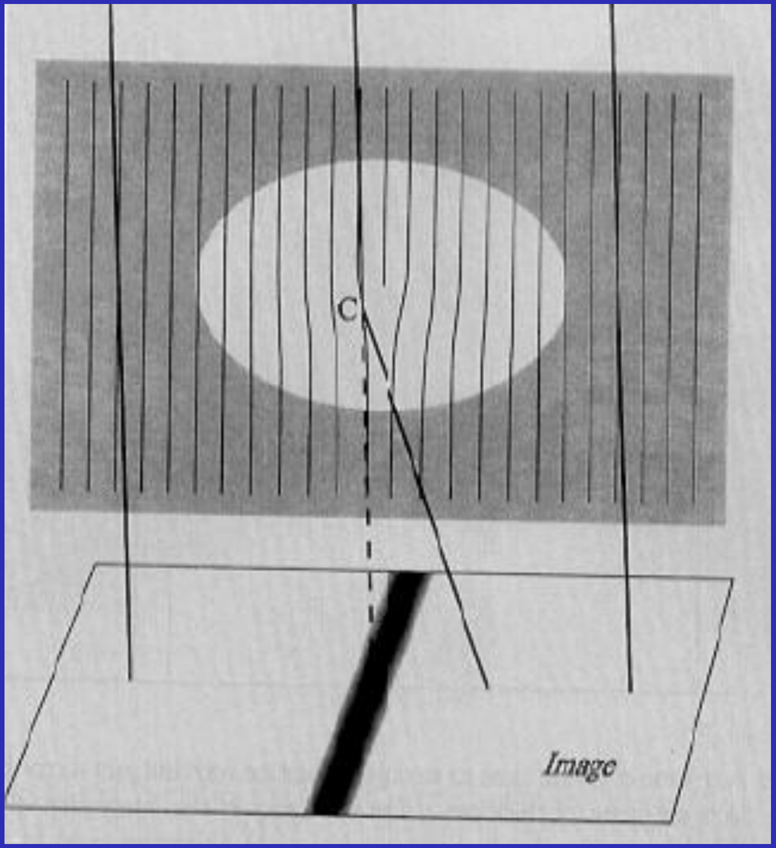
TEM Imaging: Matrix/precipitates & Diffraction

Ti3Ni4 and R-phase in TiNi



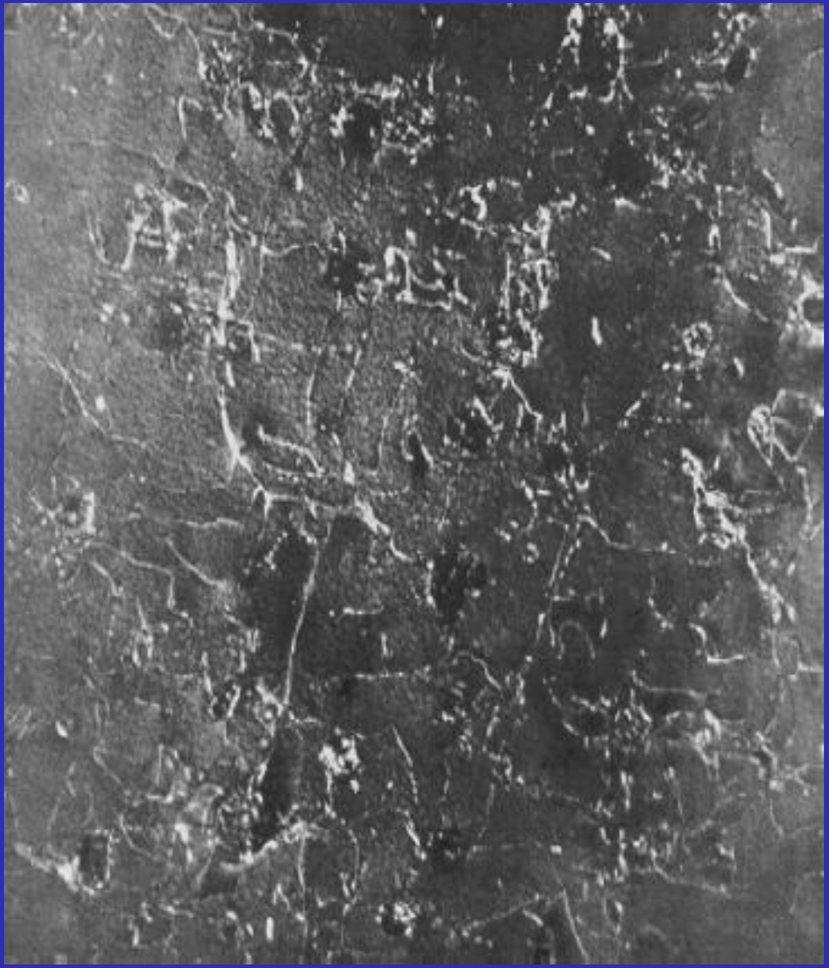
TEM Imaging: BF image of Dislocations





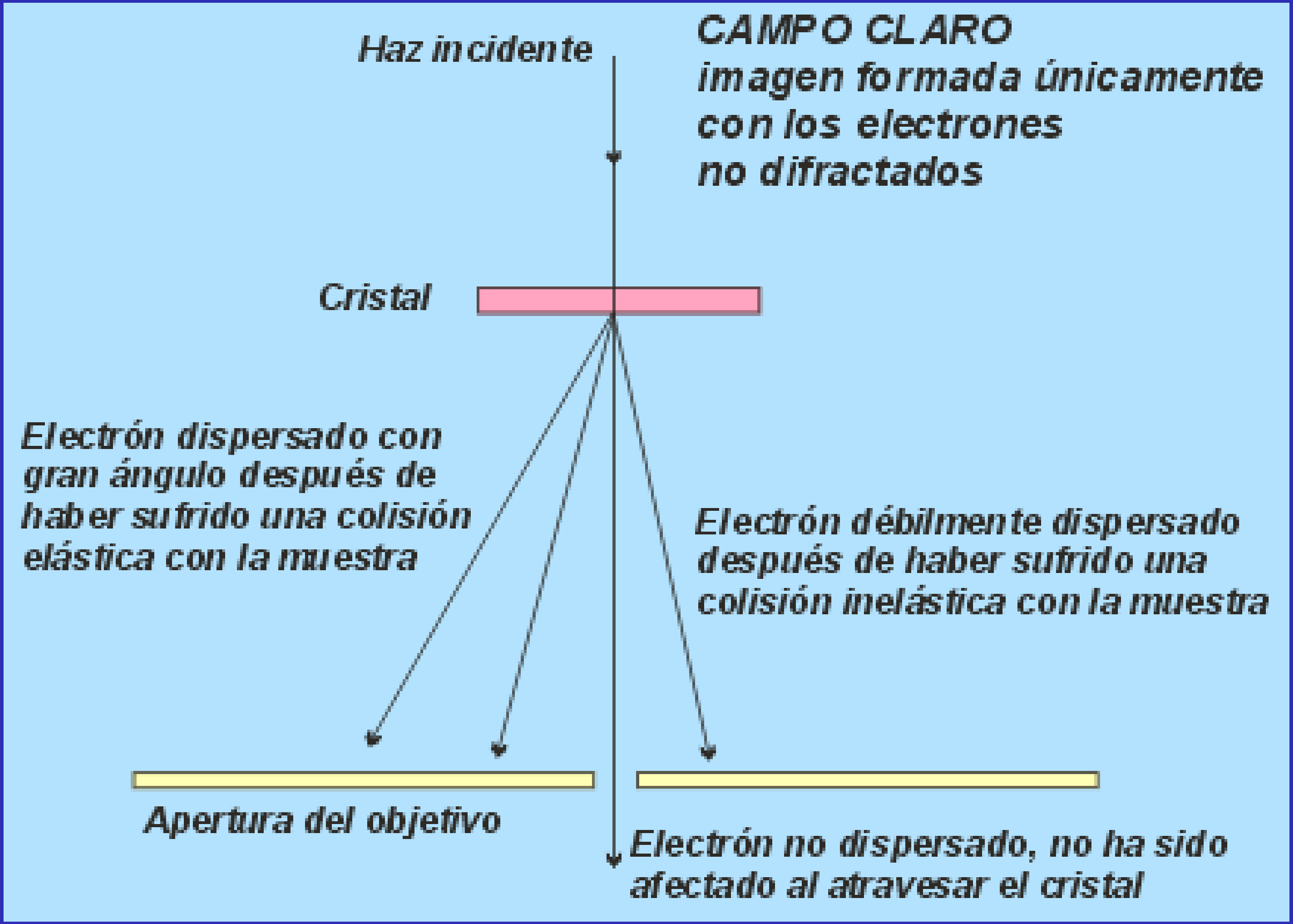
TEM Imaging: BF and DF images

Dislocations in Al-4wt.%Cu



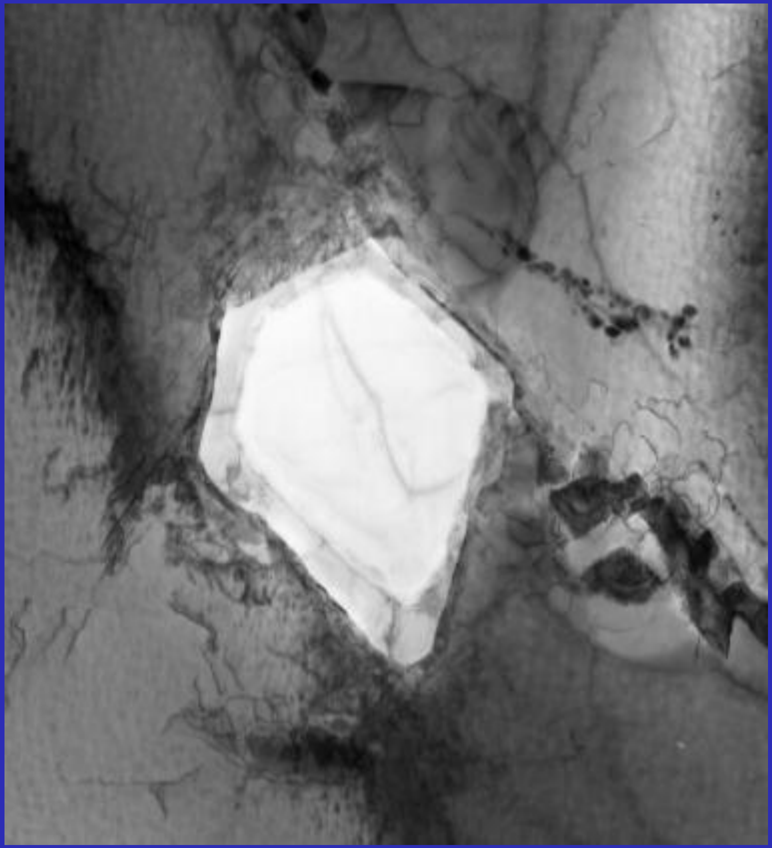
DF image

DIFFRACTION PATTERN(1)



TEM Imaging: DF images

Differently oriented inclusions (precipitates)



Three directions of inclusions in one area of an Al-Cu alloy

Mass-thickness and Diffraction contrast

Both mass-thickness and diffraction contrast are based on the amplitude contrast mechanisms because they employ only the amplitudes of the scattered waves.

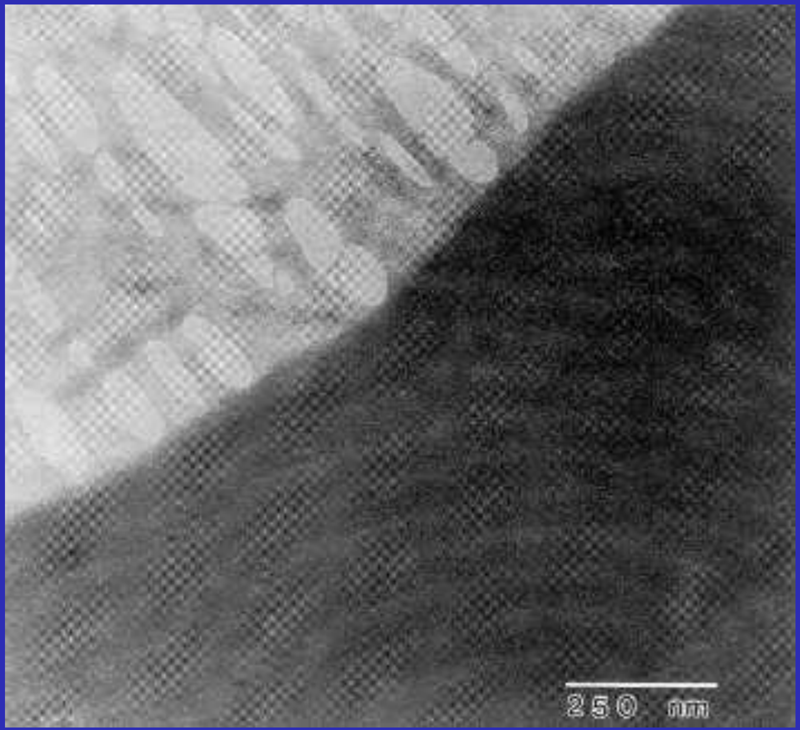
Phase contrast

Phase contrast results from interference between waves of different phases.

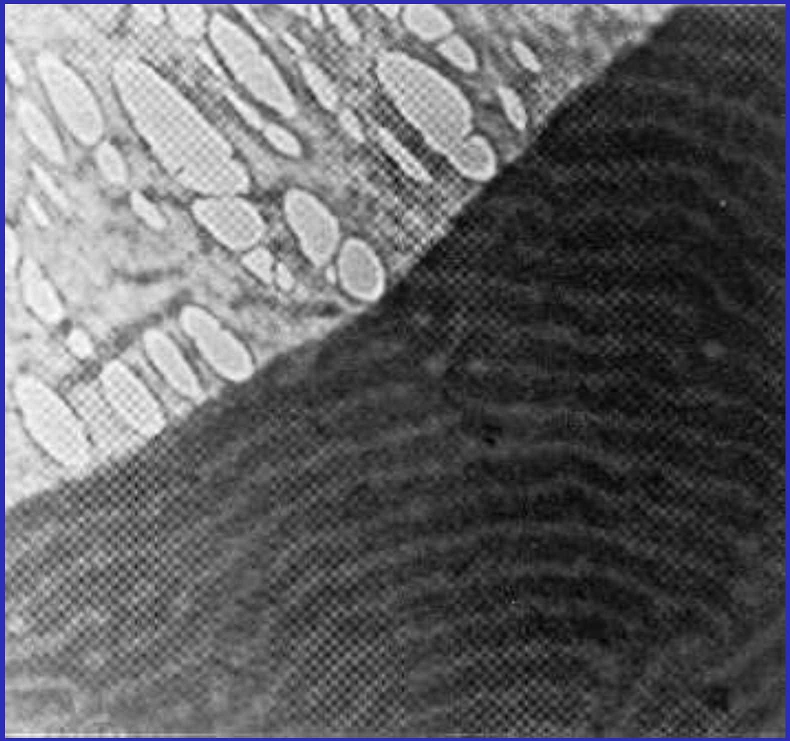
- Most electron scattering involve a phase change.
- Two or more diffracted beams are allowed to pass through the objective aperture.
- Each pair of beams, which interferes, will in principal give rise to a set of fringes in the images.

Phase contrast. examples

Without phase contrast



With phase contrast

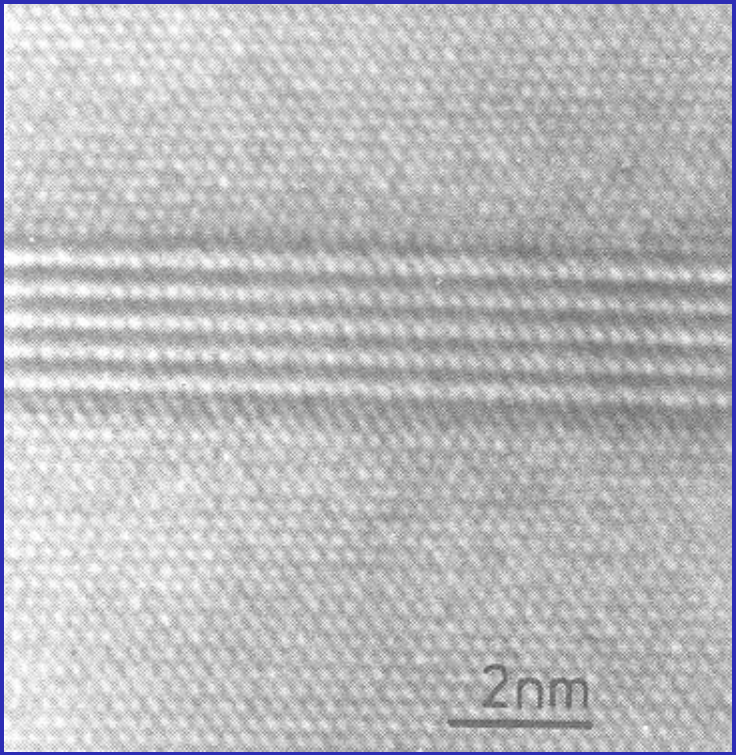


Phase contrast

Use of two (or more) scattered beams:

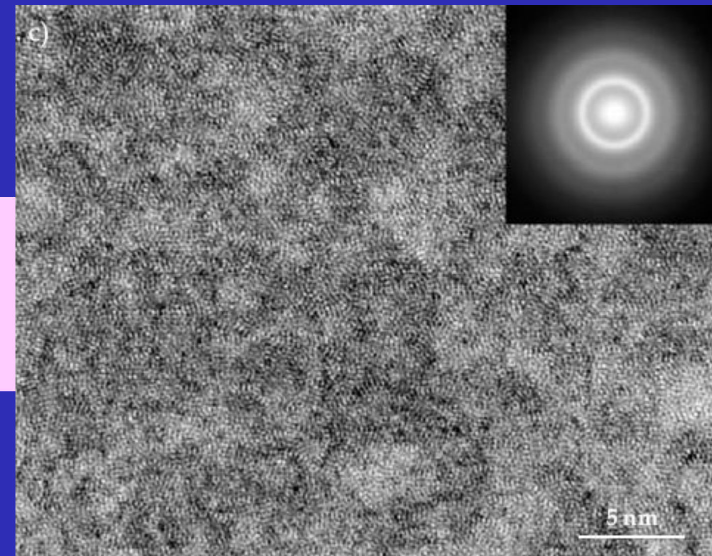
✓ Forwarded beam - Diffracted beam or Diffracted beams

Interference of the beams shows the lattice fringe image of the material



Identificación de estructuras cristalinas en recubrimientos duros

Recubrimiento de WC/C en vista de planta, mostrando su patrón de difracción.



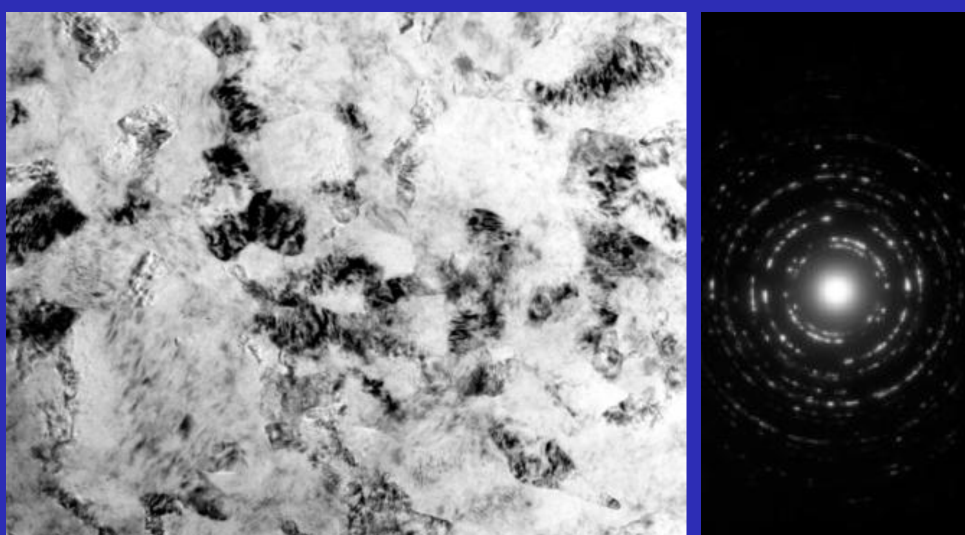
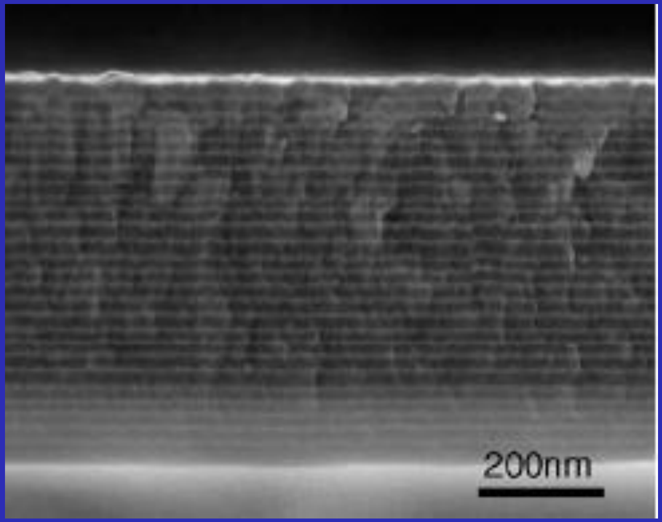
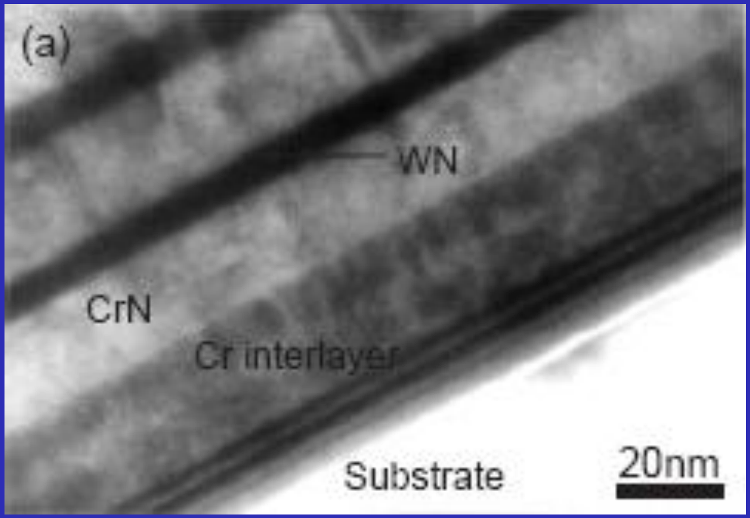


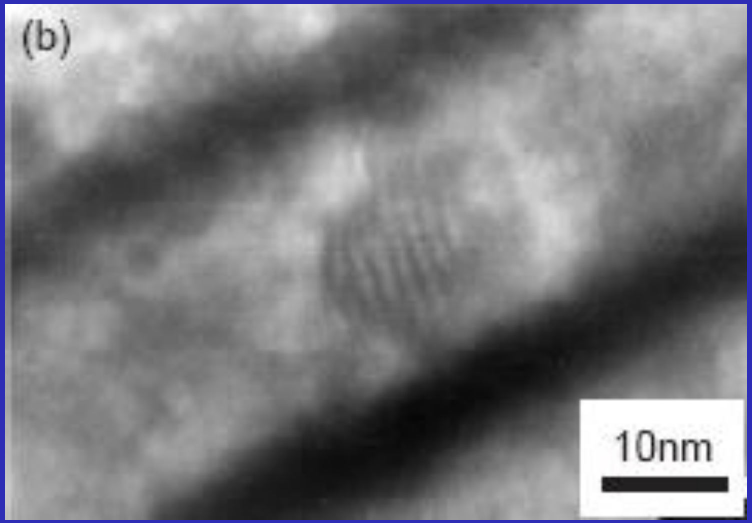
Imagen en campo claro y patrón de difracción de un recubrimiento de TiN.[7]

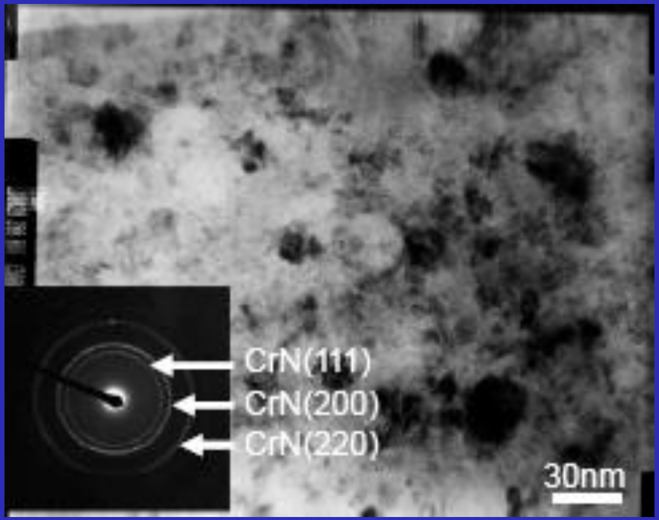
Recubrimientos de nanocapas de CrN/WN y nanocompuesto de CrWN





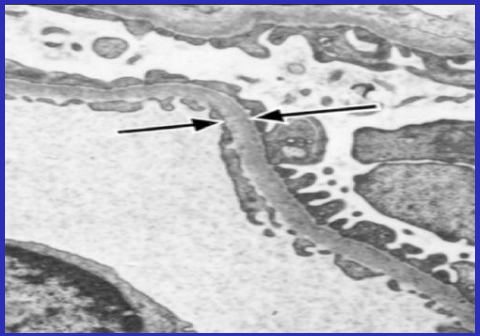
Recubrimiento de nanocapas de CrN/WN con un periodo de bicapa de 24 nm.

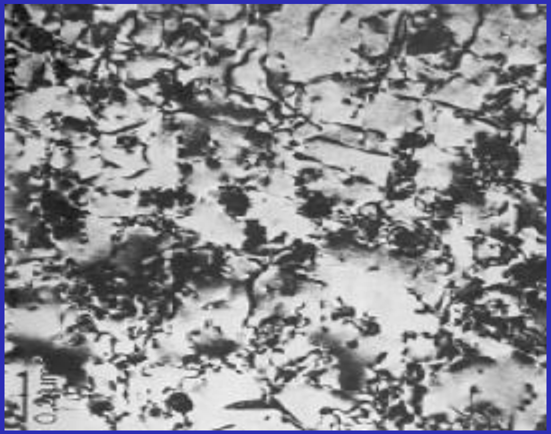




Recubrimiento de CrWN.

Use of a TEM: Take-away message





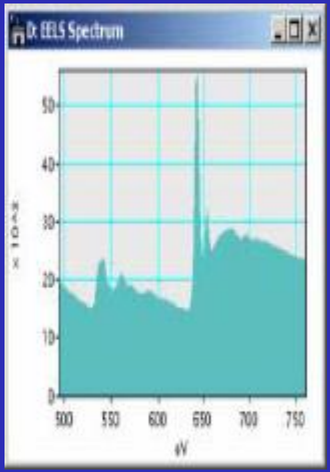
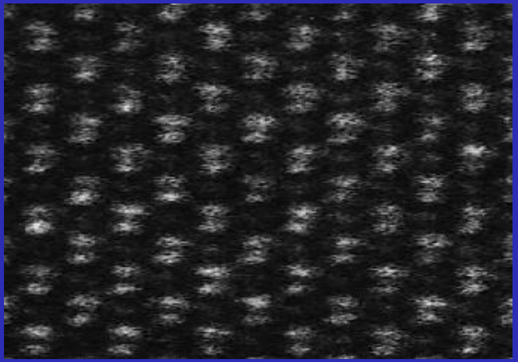
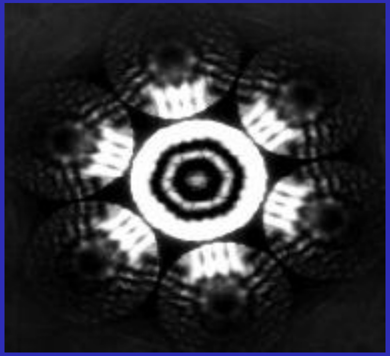
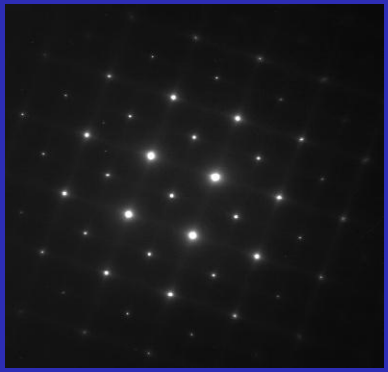


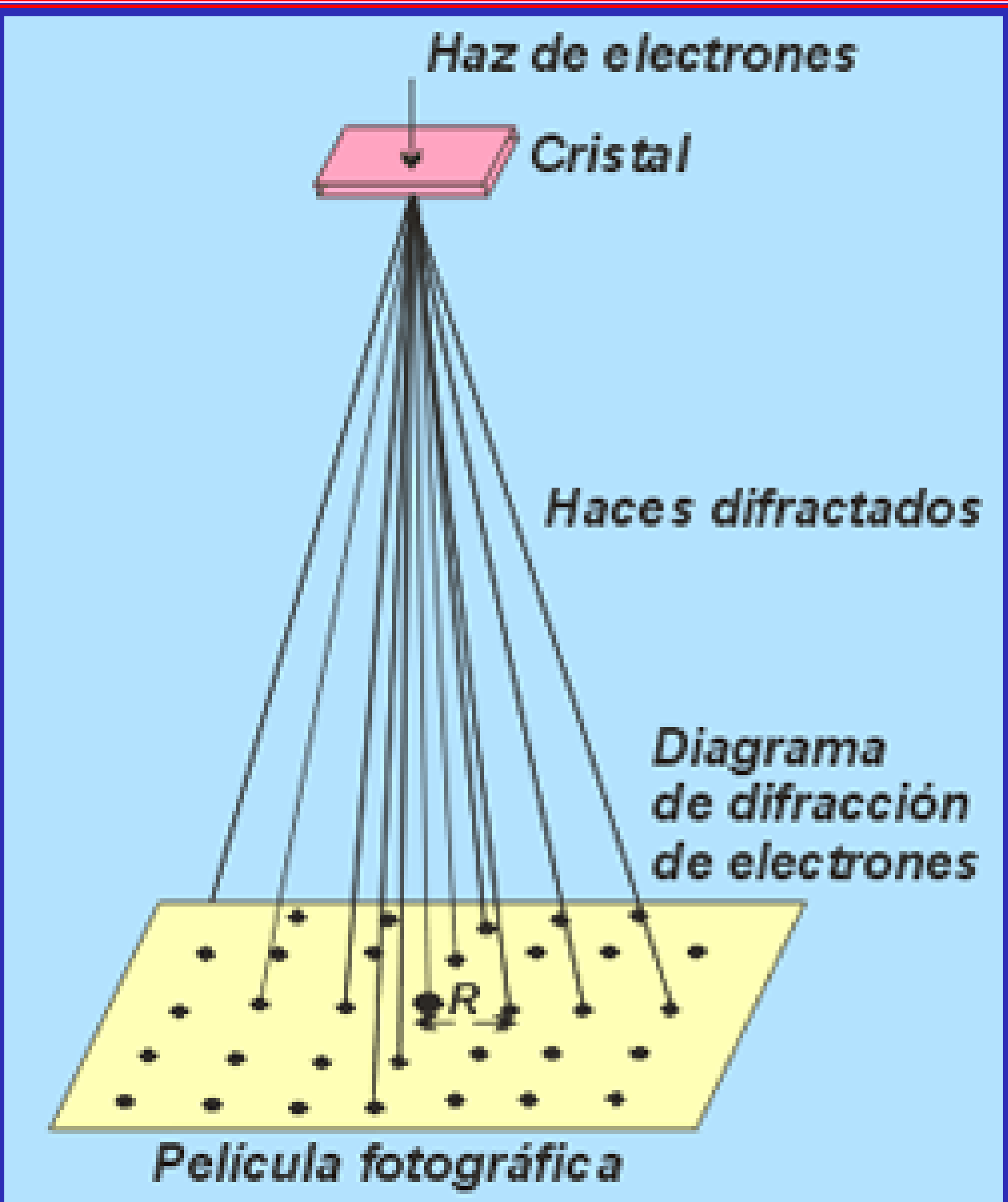
Image
Structural
Chemical





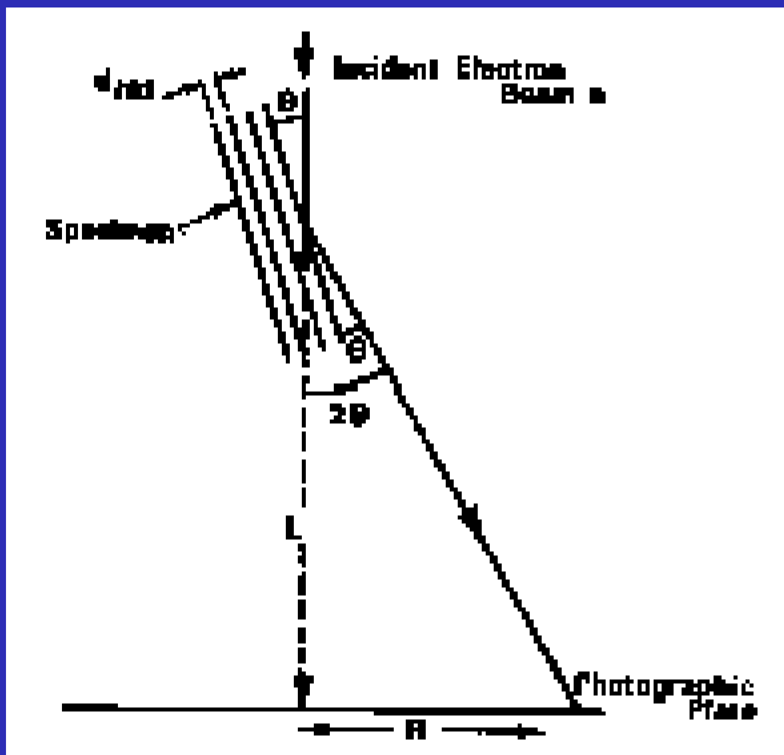


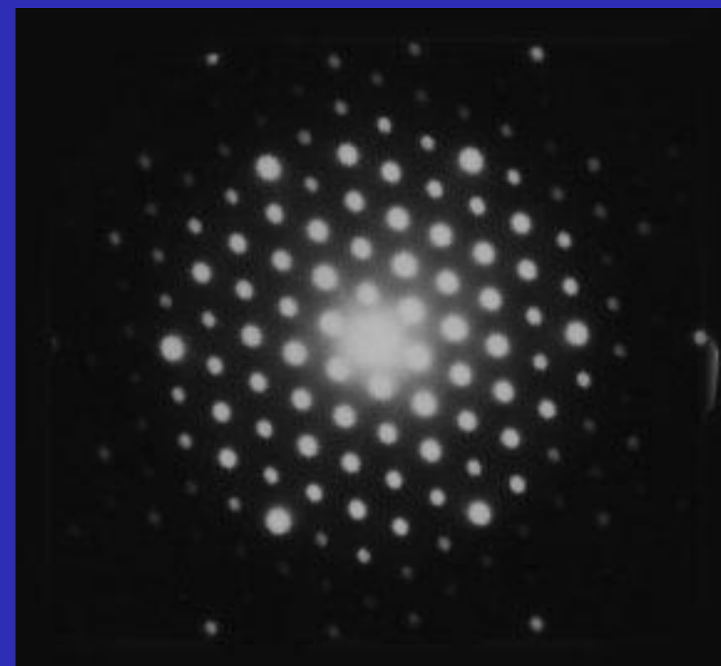
DIFFRACTION PATTERN(1)



DIFFRACTION PATTERN(1)

A typical electron diffraction pattern for a crystalline specimen is shown here





Indexing: It is often possible to index an electron diffraction pattern by noting its symmetry. A reciprocal lattice has the same symmetry as its real lattice

Inelastically Scattered Electrons: Source

Incident electrons that interact with specimen atoms in a inelastic fashion, loosing energy during the interaction. These electrons are then transmitted trough the rest of the specimen

Inelastically Scattered Electrons: Utilization

Inelastically scattered electrons can be utilized two ways:

- ***Electron Energy Loss Spectroscopy***
- ***Kikuchi Bands***

Inelastically Scattered Electrons: Utilization

Electron Energy Loss Spectroscopy:

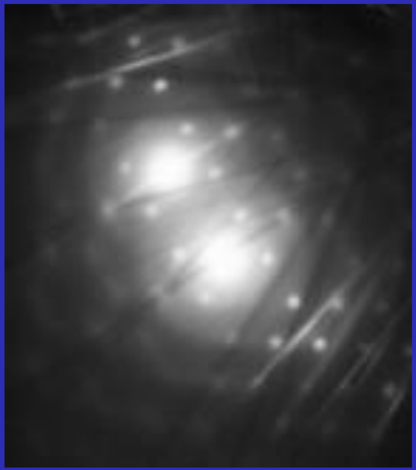
The inelastic loss of energy by the incident electrons is characteristic of the elements that were interacted with. These energies are unique to each bonding state of each element and thus can be used to extract both compositional and bonding (i.e. oxidation state) information on the specimen region being examined

Inelastically Scattered Electrons: Utilization

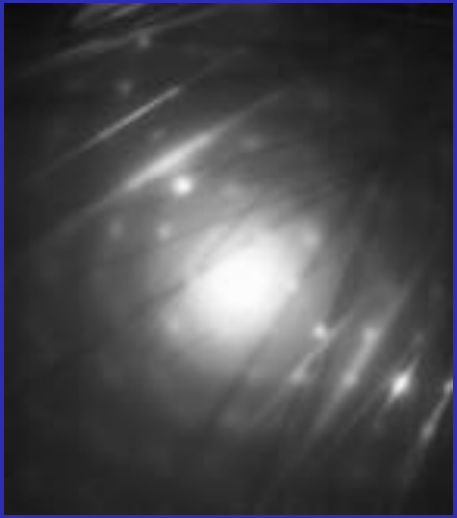
***Kikuchi Bands*:**

Bands of alternating light and dark lines that are formed by inelastic scattering interactions that are related to atomic spacings in the specimen. These bands can be either measured (their width is inversely proportional to atomic spacing) or "followed" like a roadmap to the "real" elasticity scattered electron pattern

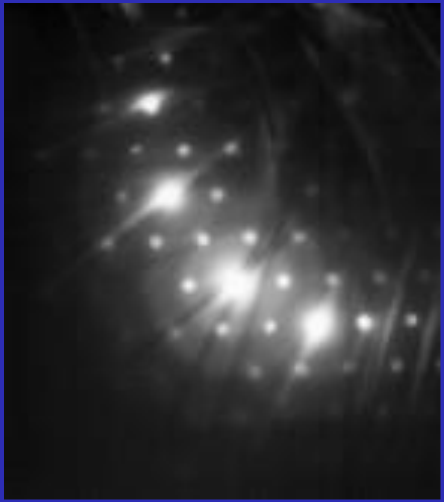
Kikuchi Lines (3)



The Kikuchi lines pass straight through the transmitted and diffracted spots. The diffracting planes are therefore tilted at **exactly** the Bragg angle to the optic axis



The crystal has now been titled **slightly away** from the Bragg angle, so that the Kikuchi lines no longer pass through the transmitted and diffracted spots

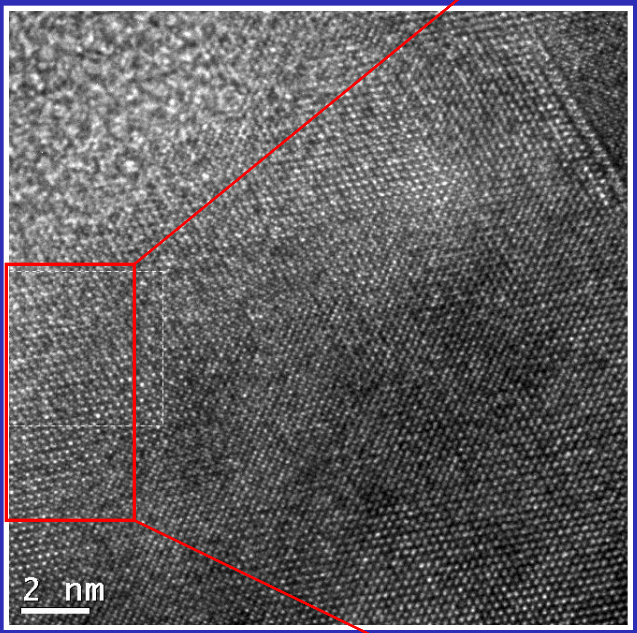


The spacing of the pair of Kikuchi lines is the same as the spacing of the diffracted spots from the same plane. However, the position of the lines is very sensitively controlled by the orientation of the specimen and Kikuchi lines are often used to set the orientation of a crystal in the TEM to an accuracy of 0.01 degrees

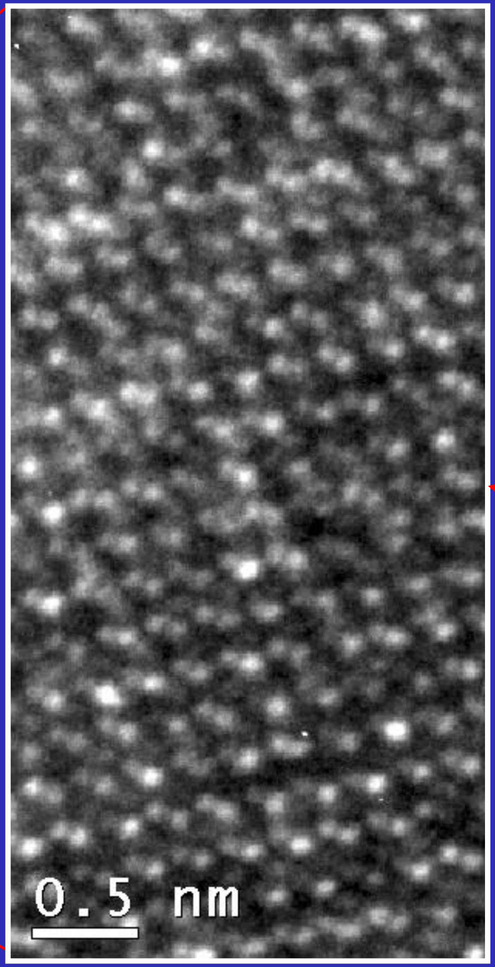
- Visión del átomo

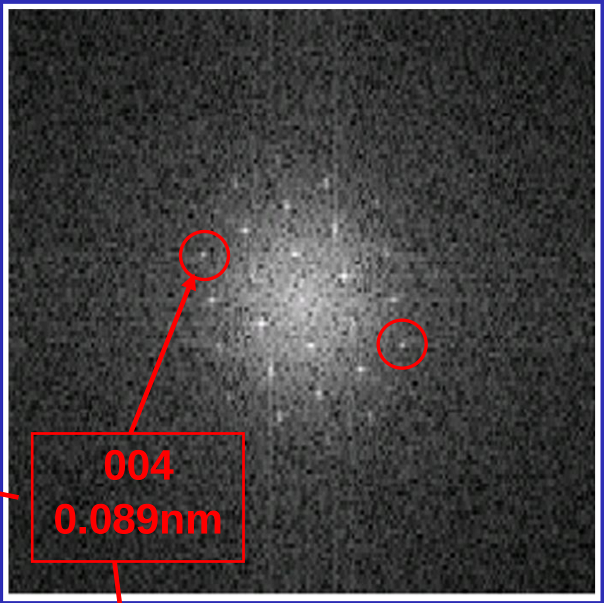
Twins en Diamante <110>

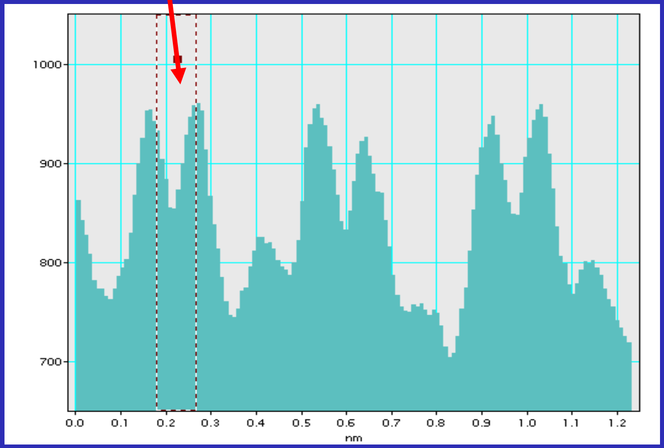
TITAN image Cs corrector @300kV



Images B.Freitag, Sample C. Kisielowski NCEM

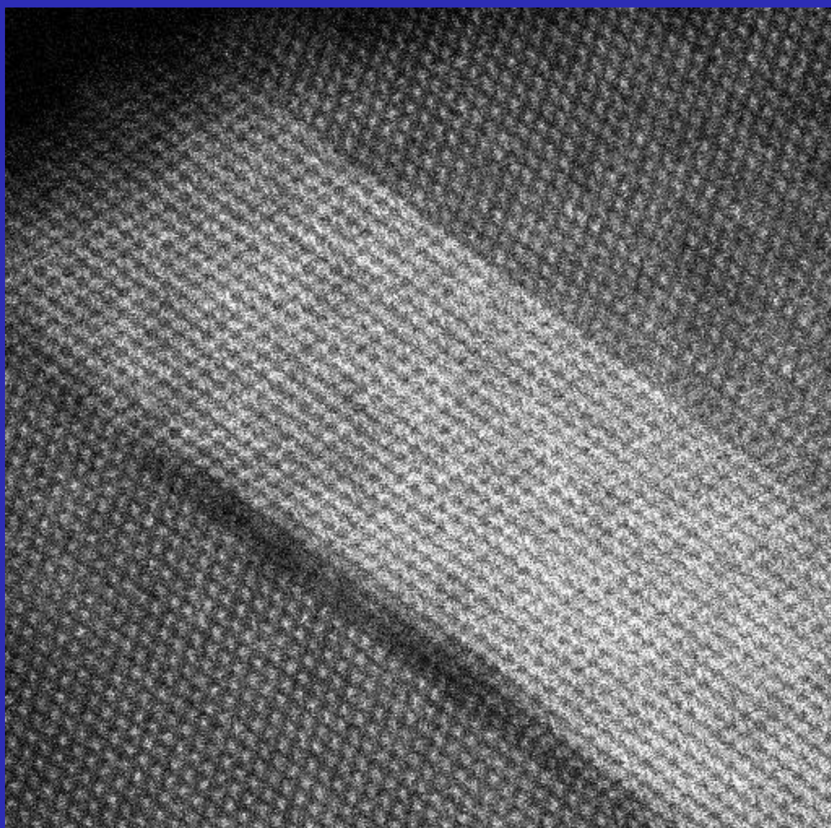


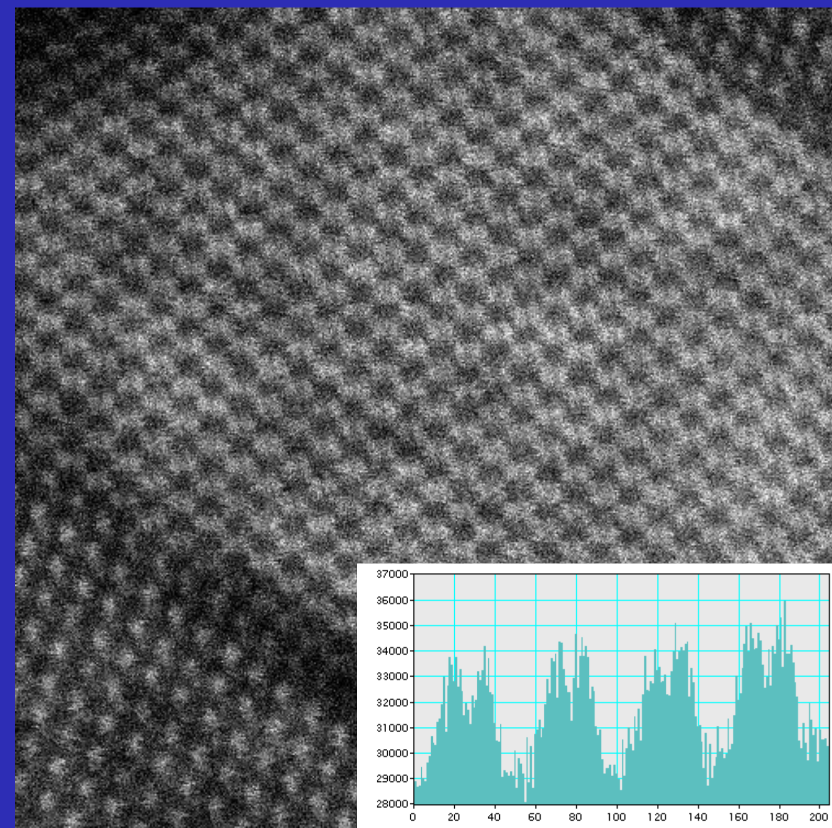




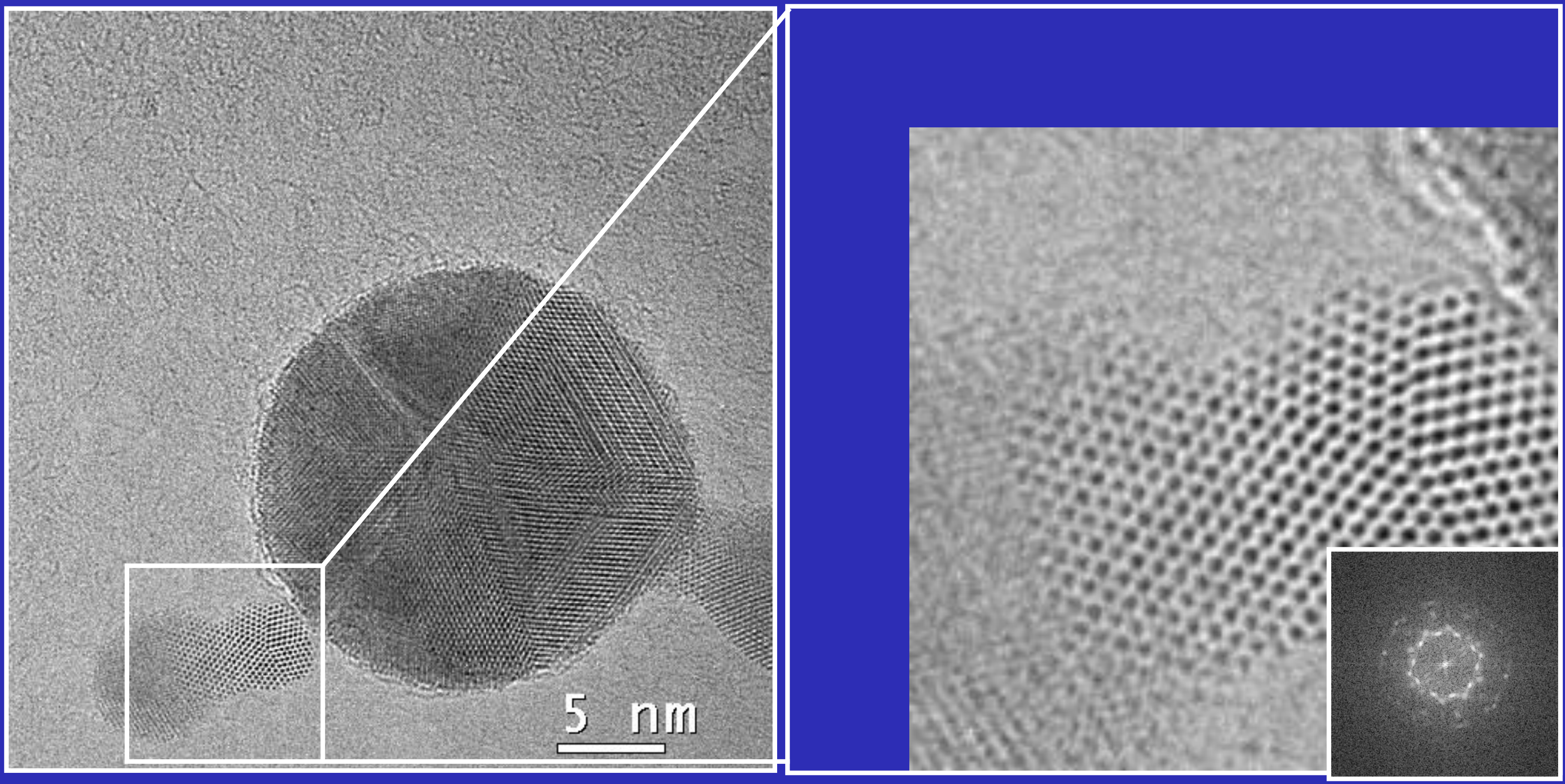
Distancia entre dobletes (Dumbbell) (0.089 nm)

Dobletes de Diamante, resueltos a 0.089nm
Se observa también un defecto en el límite del twin





Nanopartículas de Au



Images B. Freitag,

Algunas consideraciones

La información obtenida es muy local (evaluándose una porción muy pequeña de material): debe emplearse TEM después de otras técnicas de caracterización microestructural que permitan cierta "perspectiva".

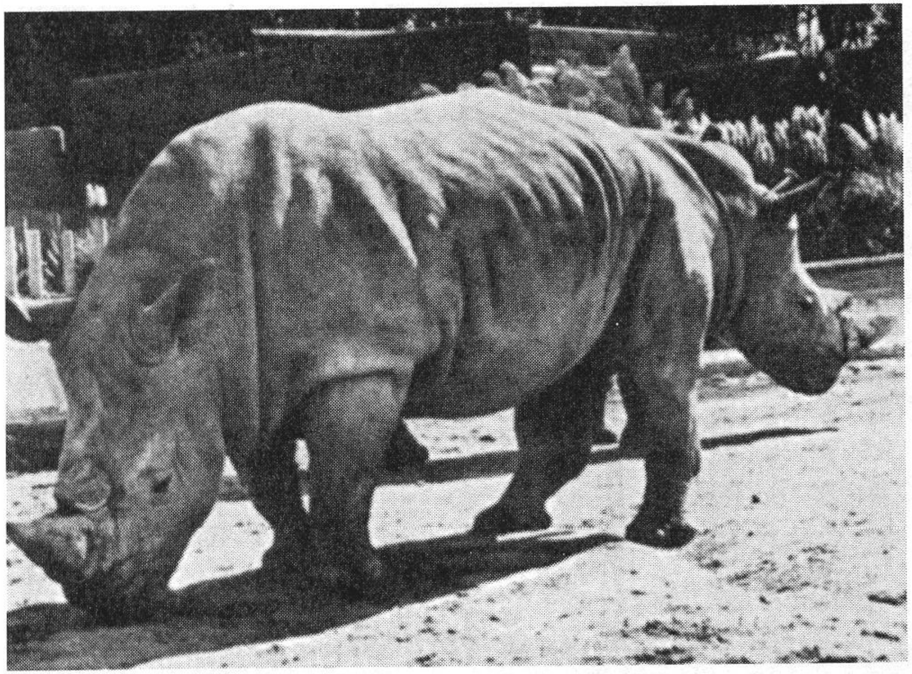


Figure 1.7. Photograph of two rhinos taken so that, in projection, they appear as one two-headed beast. Such projection artifacts in reflected-light images are easily discernible to the human eye but similar artifacts in TEM images are easily mistaken for "real" features.

Algunas consideraciones

- Debemos interpretar una imagen bidimensional con información tridimensional.
- El haz de electrones al interaccionar con la muestra puede dañarla o alterarla (“artefactos”)
- Deben prepararse muestras finas ? transparentes al haz de electrones (= 100 nm, = 50 nm para HRTEM).
- La nuestra debe ser representativa del material

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5. Electron microscopy in the Department of Materials. http://www-em.materials.ox.ac.uk/