# Electron microscopy

PH 673

Nanoscience and nanotechnology

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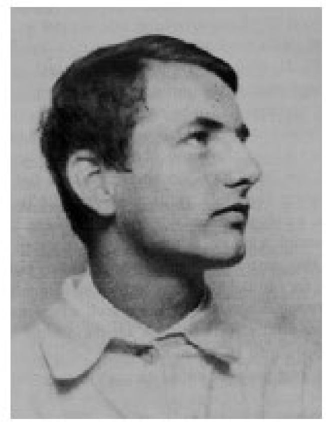
# TEM: the beginning

## Ruska & Knoll

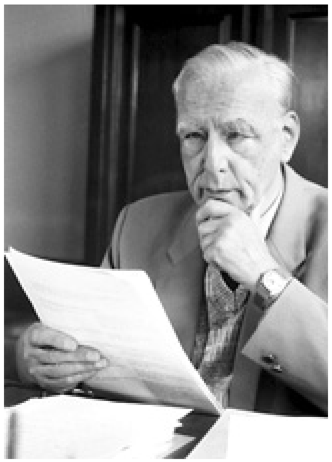
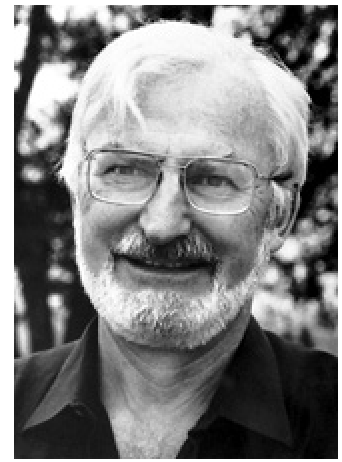
Fig. 6. The first electron microscope. The publication (48) contained only one cross-sectional drawing corresponding to Figure 5. This photograph, reproduced here for the first time, with M. Knoll and the author – was actually taken several years later, on 8 February 1944.

## Invention and Evolution of the Modern TEM

- In 1932, invented by E. Ruska *et al.*
- In 1986, Ruska received the Nobel Prize



# Nobel Prize in Physics 1986

**Ernst Ruska** (Fritz-Haber-Institut der Max-Planck-Gesellschaft, Germany)
**Gerd Binnig** (IBM Zurich Research Laboratory, Switzerland)
**Heinrich Rohrer** (IBM Zurich Research Laboratory, Switzerland)
The Nobel Prize in Physics 1986 was divided, one half awarded to Ernst Ruska *"for his fundamental work in electron optics, and for the design of the first electron microscope"*,the other half jointly to Gerd Binnig and Heinrich Rohrer *"for their design of the scanning tunneling microscope"*.

# Transmission Electron Microscope

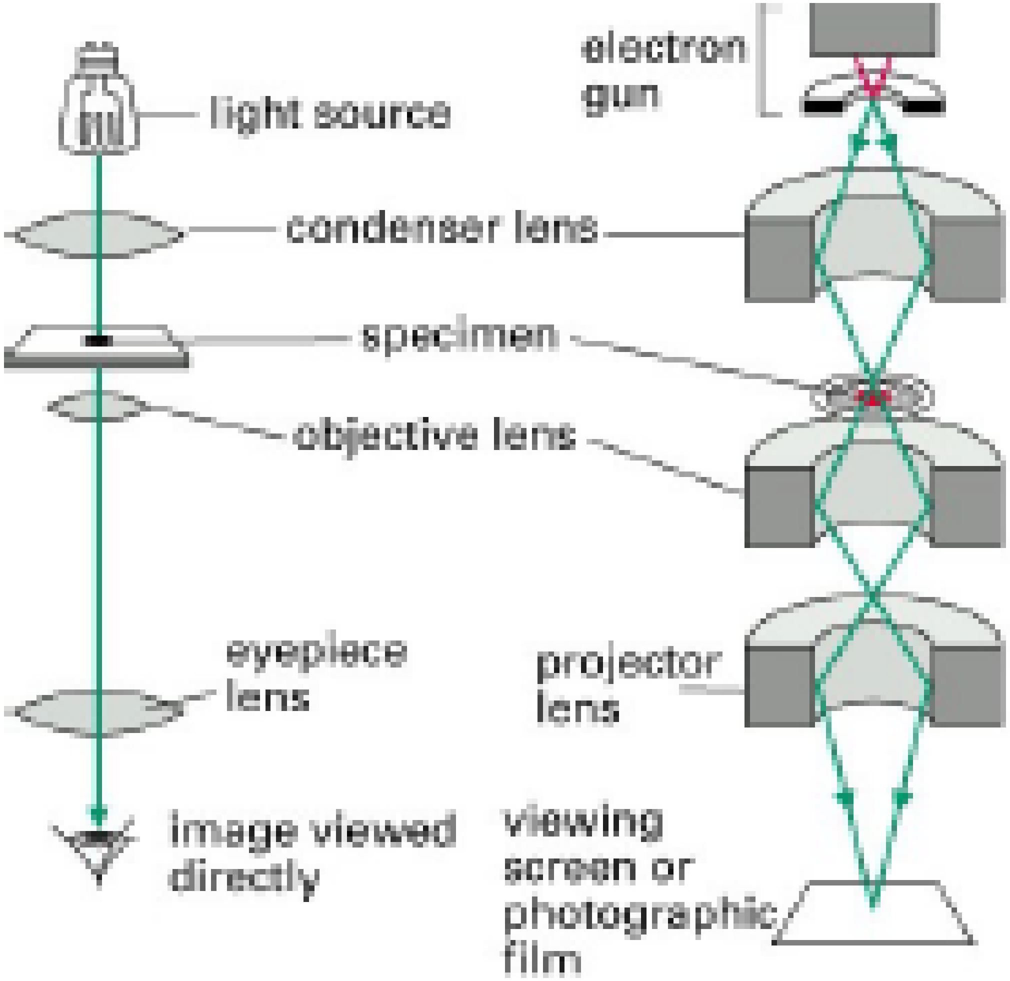
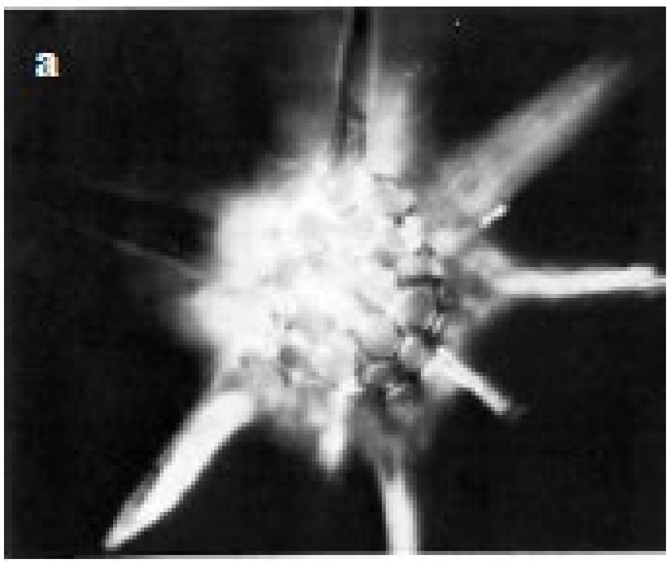


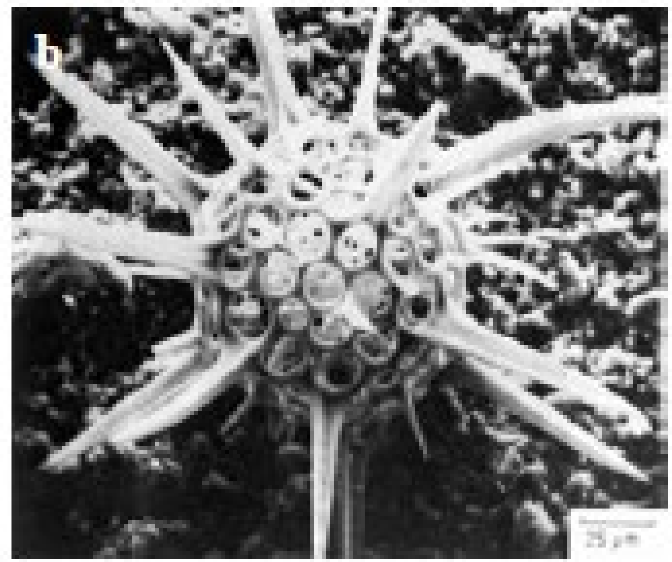

Figure 9–22. Molecular Biology of the Cell, 4th Edition.

# Optical vs electron microscopy

## Depth of Field or Depth of Focus




OM image




SEM image

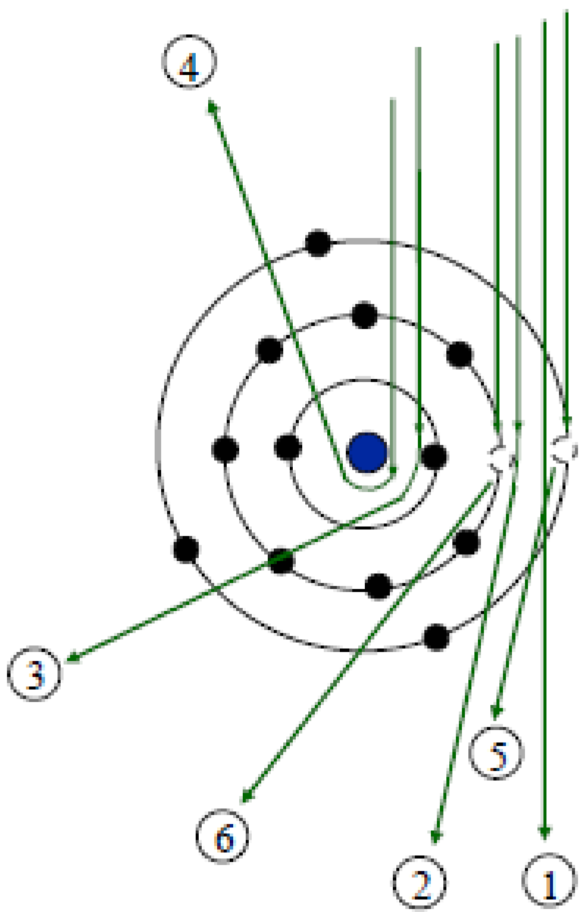
Radiolarian, taken from Goldstein *et al.* (1992)

# Basic electron optics

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

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

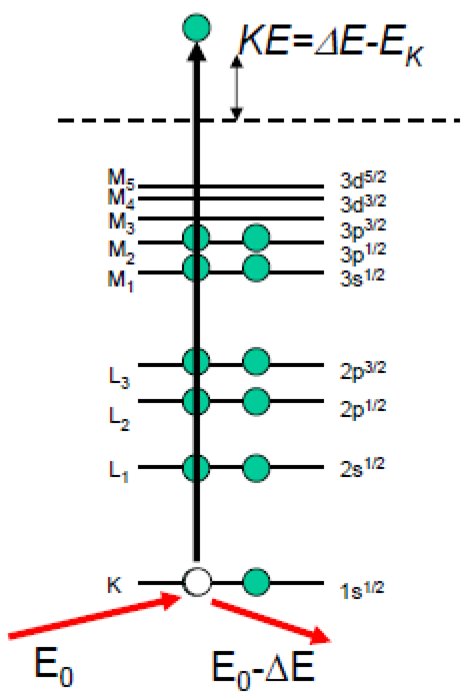
## Interaction with an Atom




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

# *Electron Shells and Transitions*

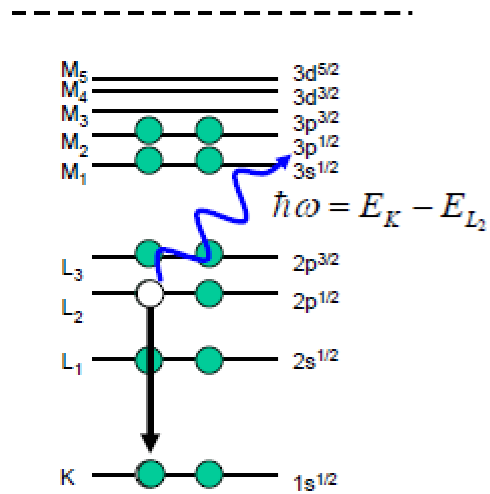
## 1) Ionization




Measured by EELS

(Electron Energy Loss Spectroscopy In a TEM)

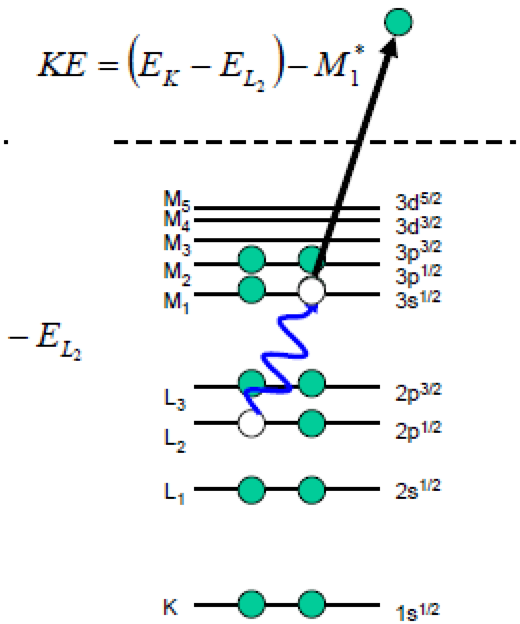
## 2a) X-ray Emission




Measured by EDS/XRF

(X-ray fluorescence in an SEM or TEM)

## 2b) Auger electron

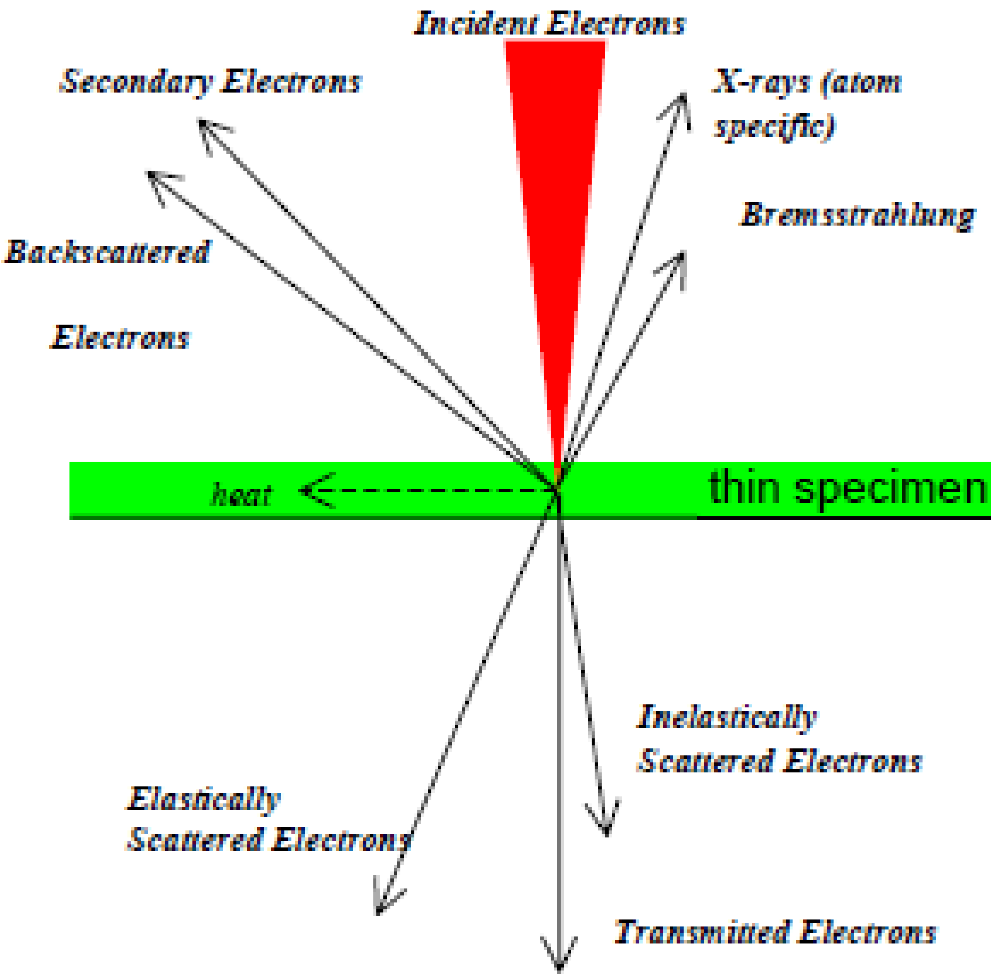



Measured by AES

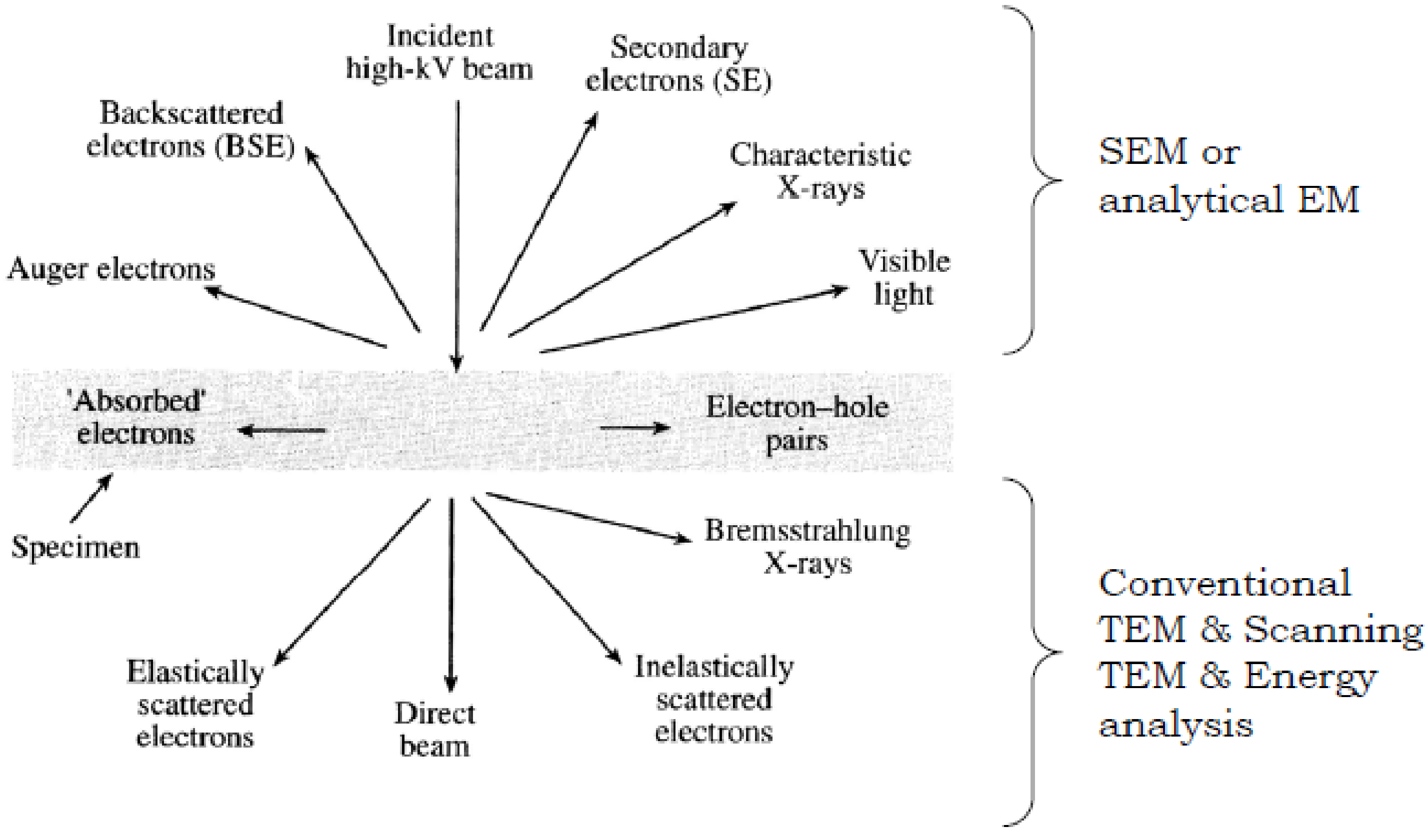
(Auger Spectroscopy in a SAM, Leaves atom doubly charged)

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

## Interaction with a thin specimen (TEM & STEM)

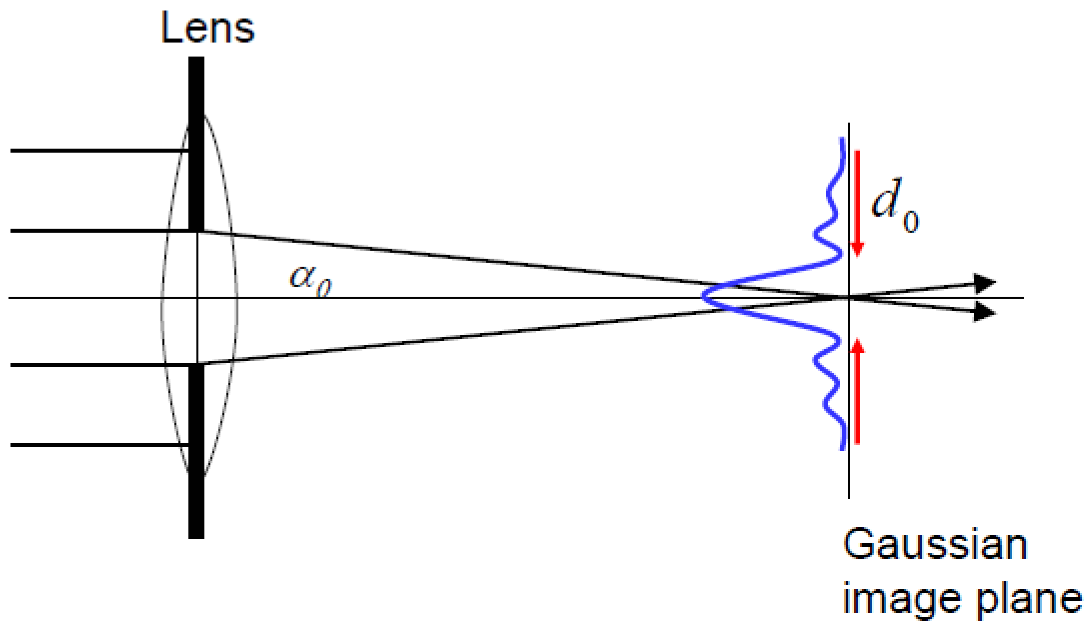


# Interaction of Electron with Samples



# *Resolution Limits Imposed by the Diffraction Limit*

(Less diffraction with a large aperture – must be balanced against $C_s$)




The image of a point transferred through a lens with a circular aperture of semiangle $\alpha_0$ is an Airy Disk of diameter $d_0 = \dfrac{0.61\lambda}{n\sin\alpha_0} \approx \dfrac{0.61\lambda}{\alpha_0}$

(0.61 for incoherent imaging e.g. ADF-STEM, 1.22 for coherent or phase contrast,. E.g TEM)

(for electrons, n~1, and the angles are small)

# *Electron Velocity and Wavelength*

De Broglie Wavelength: $\lambda = \frac{h}{p}$

Where *h* is Planck's constant
And *p=mv* are the momentum, mass and velocity of the electron

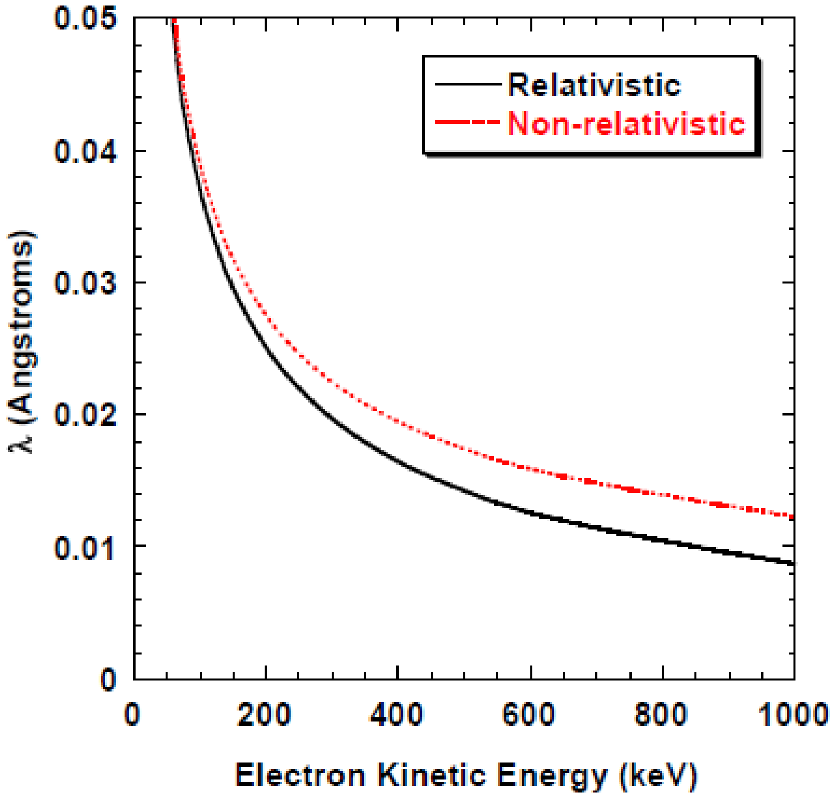
If an electron is accelerated through a potential *eV*, it gains kinetic energy

$\frac{1}{2}mv^2 = eV$ So the momentum is $mv = \sqrt{2meV}$

Electron wavelength $\lambda = \sqrt{\frac{h^2}{2meV}} = \frac{1.23nm}{\sqrt{V}}$ (V in Volts)

( relativistically correct form: $\lambda = \sqrt{\frac{h^2c^2}{eV(2m_0c^2 + eV)}}$ )

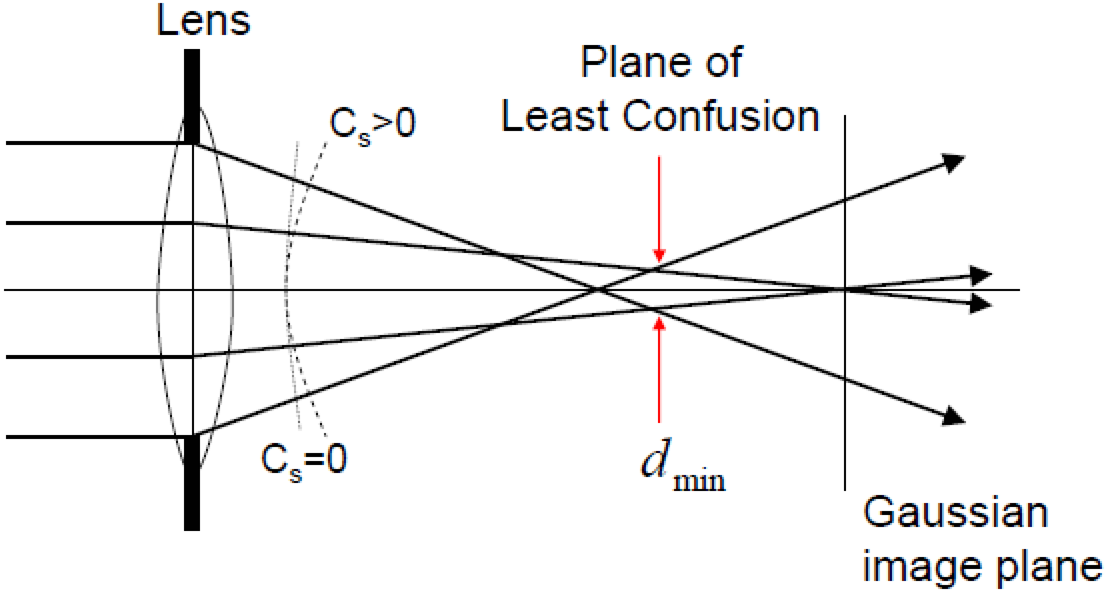
# Electron Wavelength vs. Accelerating Voltage




| Accelerating Voltage | v/c | λ (Å) |
|---|---|---|
| 1 V | 0.0019784 | 12.264 |
| 100 V | 0.0062560 | 1.2263 |
| 1 keV | 0.062469 | 0.38763 |
| 10 keV | 0.019194 | 0.12204 |
| 100 keV | 0.54822 | 0.037013 |
| 200 keV | 0.69531 | 0.025078 |
| 300 keV | 0.77653 | 0.019687 |
| 1 MeV | 0.81352 | 0.0087189 |

# *Resolution Limits Imposed by Spherical Aberration, $C_s$*

(Or why we can't do subatomic imaging with a 100 keV electron)



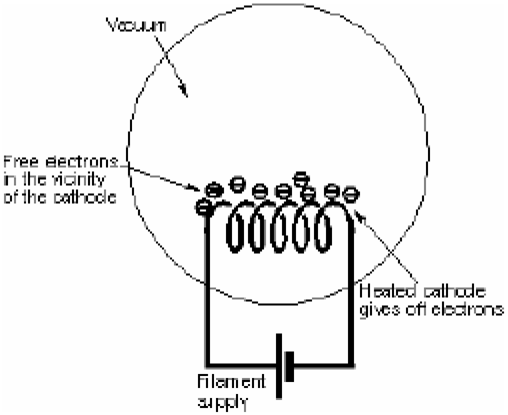

For $C_s$>0, rays far from the axis are bent too strongly and come to a crossover before the gaussian image plane.

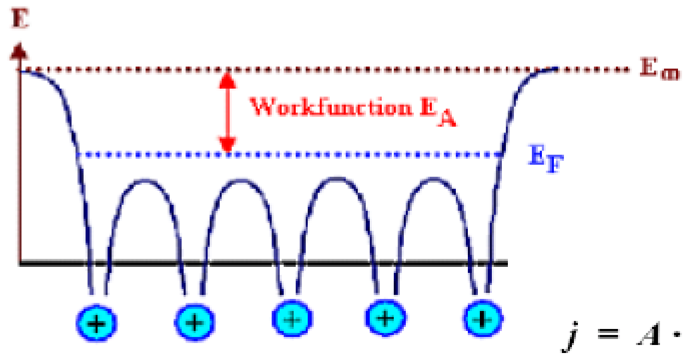
For a lens with aperture angle $\alpha$, the minimum blur is $d_{\min} = \frac{1}{2} C_s \alpha^3$

Typical TEM numbers: $C_s$= 1 mm, $\alpha$=10 mrad $\rightarrow$ $d_{min}$= 0.5 nm

# Electron sources

- Thermal emission



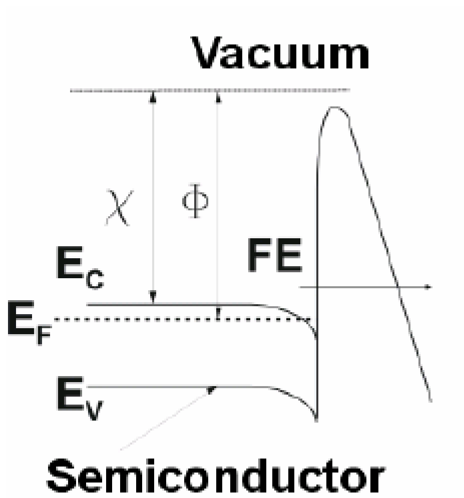



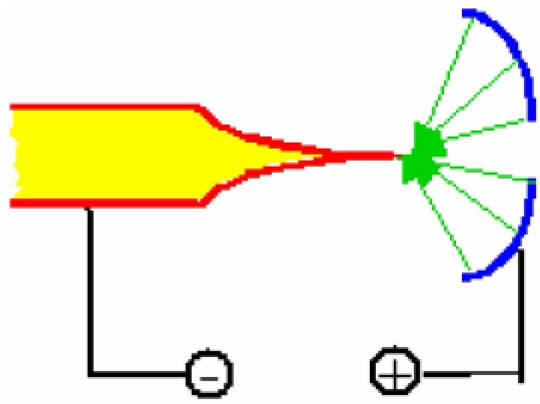

$$j = A \cdot T^2 \cdot \exp - \frac{E_A}{kT}$$

| Material | Fe | Ni | Pt | Ta | W | Cs | $LaB_6$ |
|---|---|---|---|---|---|---|---|
| $A$ [$Acm^{-2}K^{-2}$] | 26 | 30 | 32 | 55 | 60 | 162 | 25 |
| $E_A$ [eV] | 4,5 - 4,8 | 5,15 - 5,35 | 5,65 | 4,15 - 4,8 | 4,2 | 1,8 - 2,14 | 2,6 |
| $T_m$ [$^oC$] | 1 535 | 1 452 | 1 755 | 2 850 | 3 410 | 28,4 | 2 210 |

# Electron sources

- Field emission



Field emission starts for $E > 10^7$ V/cm
High current density: $J(E) = A \cdot E^2 \varphi \exp(-B \varphi^{1.5} / E)$

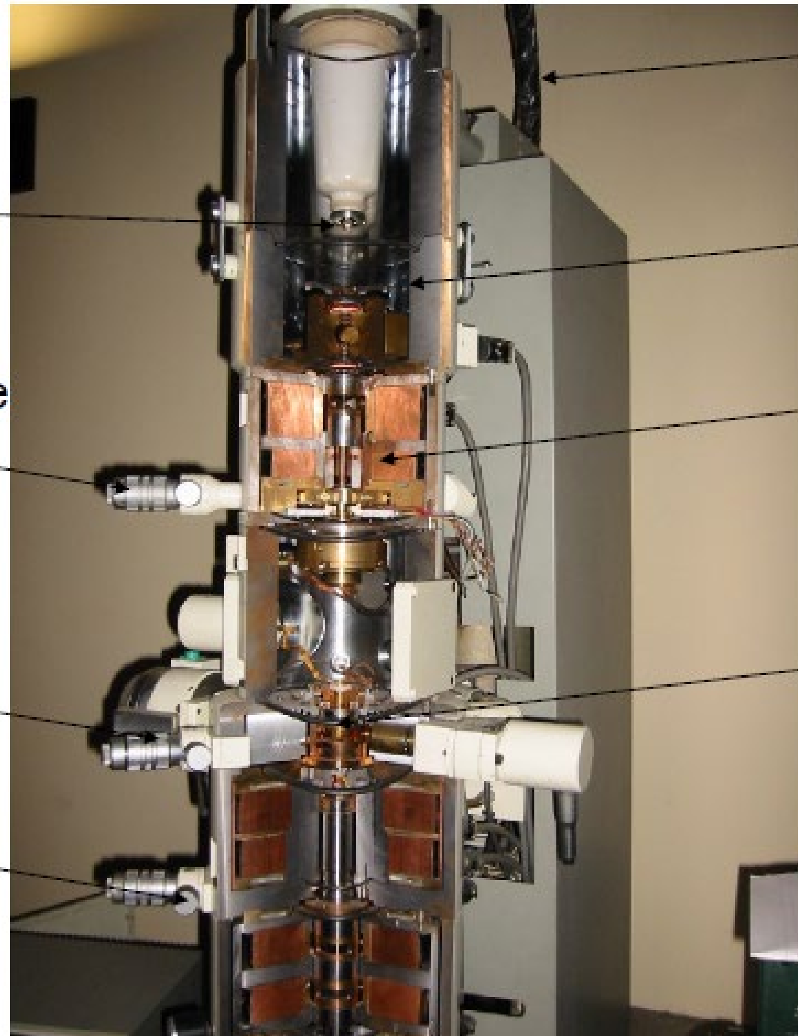
Strong nonlinear current-voltage characteristic
Very short switching time (t <ns)

Small spot size due to field enhancement at the tip apex

- # Electron beam sources

TABLE 2.1 Properties of the electron sources commonly used in electron beam lithography tools.

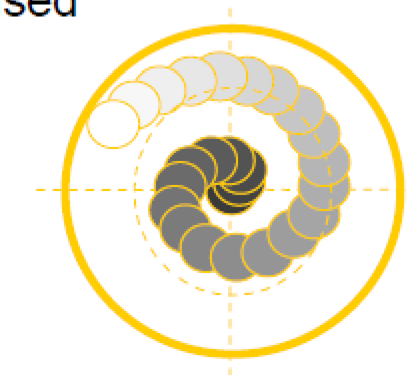
| source type | brightness ($A/cm^2/sr$) | source size | energy spread (eV) | vacuum requirement (Torr) |
|---|---|---|---|---|
| tungsten thermionic | $\sim 10^5$ | 25 um | 2-3 | $10^{-6}$ |
| $LaB_6$ | $\sim 10^6$ | 10 um | 2-3 | $10^{-8}$ |
| thermal (Schottky) field emitter | $\sim 10^8$ | 20 nm | 0.9 | $10^{-9}$ |
| cold field emitter | $\sim 10^9$ | 5 nm | 0.22 | $10^{-10}$ |


High tension cable (100-200 kV)
Filament
Accelerating stack
Double condenser lens
condenser aperture
objective aperture
Sample sits here
Selected area aperture
Viewing chamber

# Lenses

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

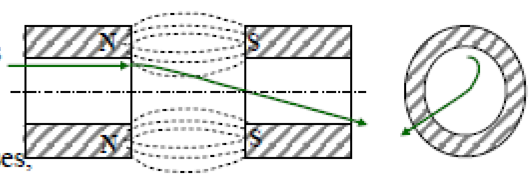
- Electromagnetic lenses are based on the fact the moving electrons are forced into a spiral trajectory, i.e. focused into one point



## Round Lenses

Magnetic lenses

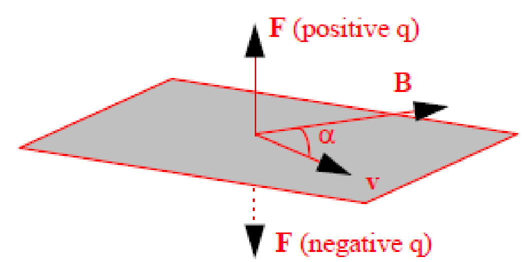
- change the direction of electrons
- magnifying (diverging)
- diminishing (converging)
- condenser lenses, objective lenses,
- intermediate lenses, projection lenses



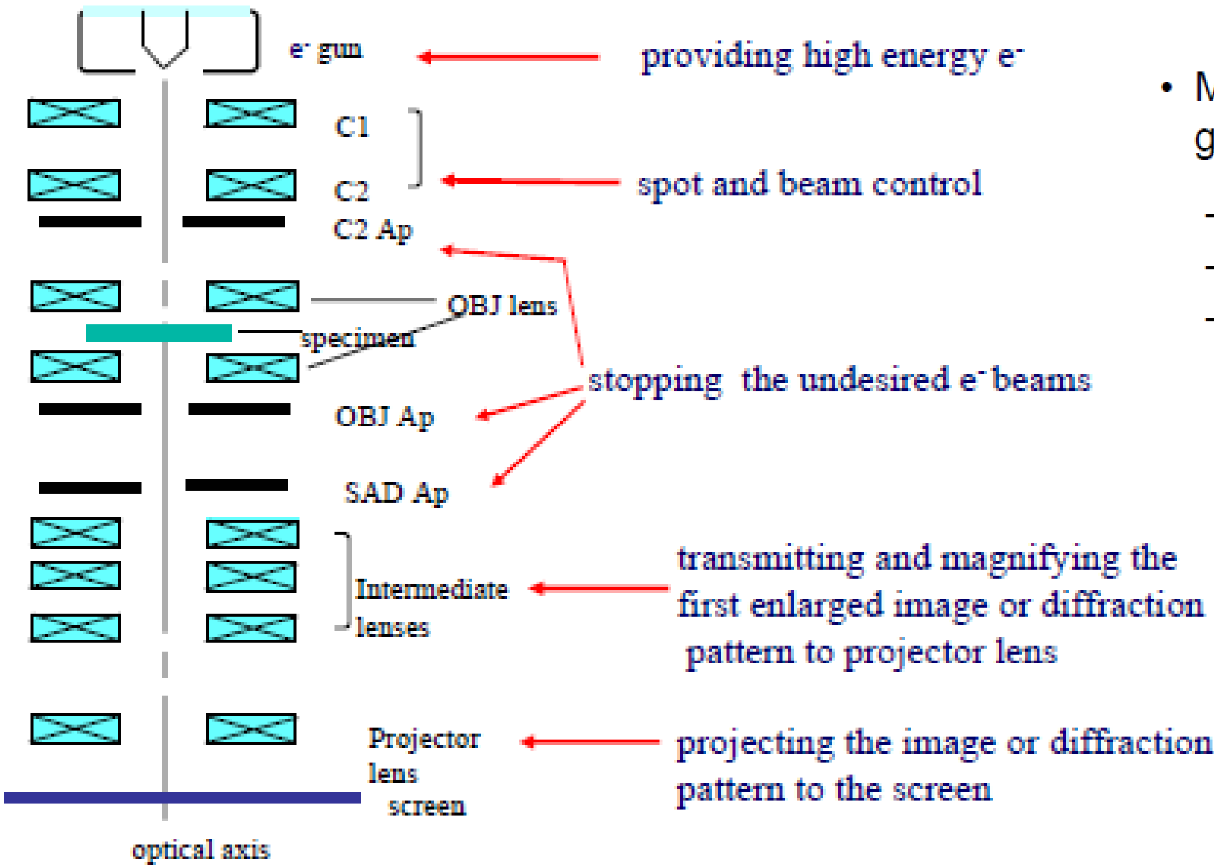

- Working Principle: Lorenz Force
  - electrons are only *deflected* by magnetic fields

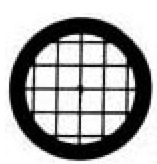
Electrostatic lenses: the Wehnelt cap

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



# Lens System of TEM



TEM grid

# Lens System & Microscope Resolution

- Microscope resolution is governed by: (for TEM)
  - wavelength of electrons
  - $C_s$ of objective lens
  - other lenses are less crucial ($\alpha$/M)

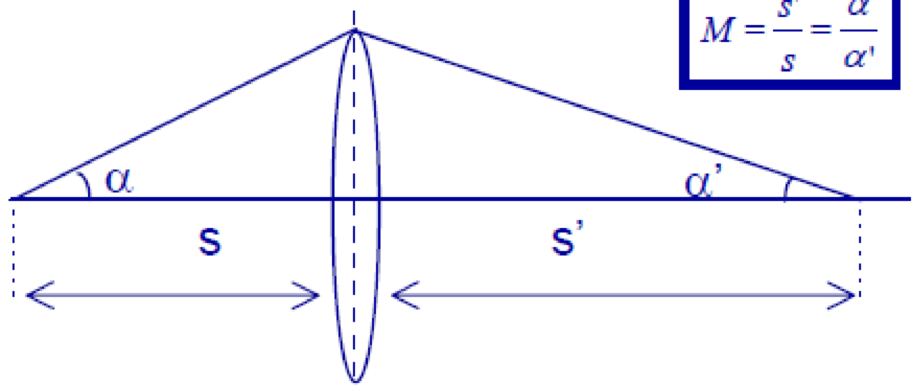
$$\delta = 0.66 \times C_s^{1/4} \lambda^{3/4}$$

## Electron Detectors

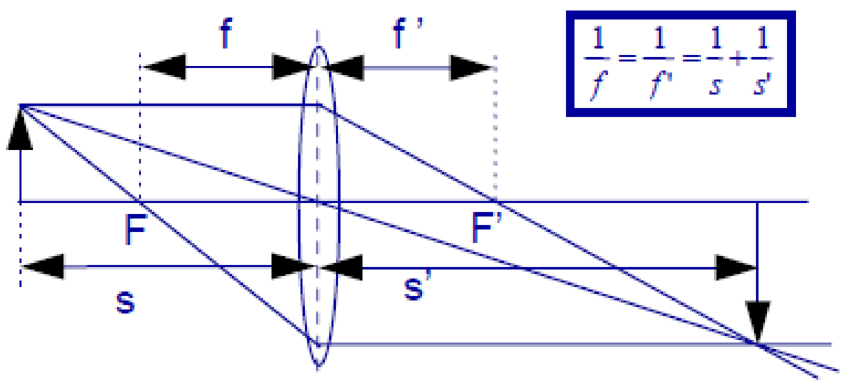
- TEM
  - phosphor screen, Film, CCD, Image Plate…
- SEM
  - SE detector, BE detector….
- STEM
  - BF detector, DF detector, ………

# Geometric (ray) optics

- Gaussian Law



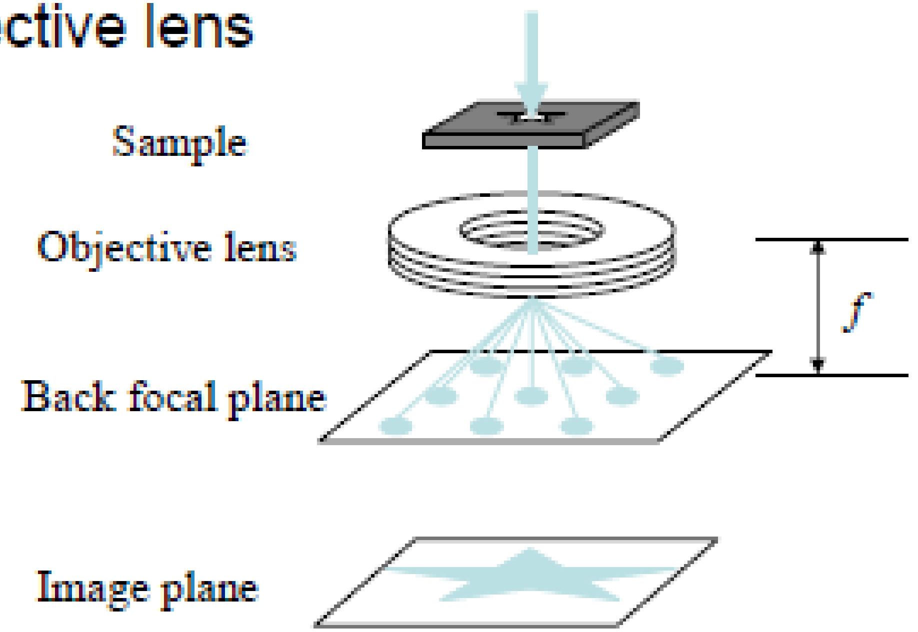

$$M = \frac{s'}{s} = \frac{\alpha}{\alpha'}$$



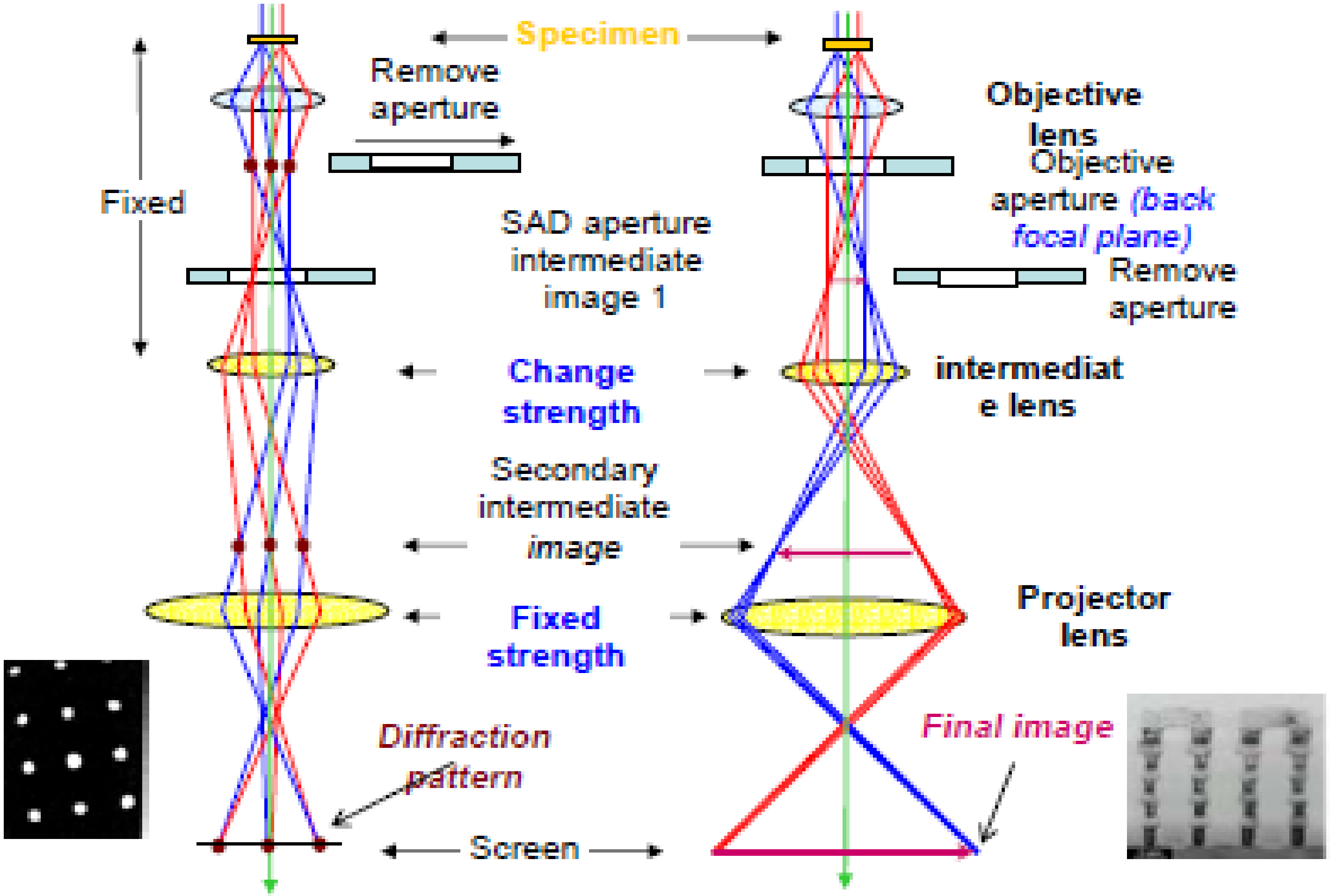

$$\frac{1}{f} = \frac{1}{f'} = \frac{1}{s} + \frac{1}{s'}$$

# Electron diffraction

- Diffraction pattern locates at the back focal plane of the objective lens

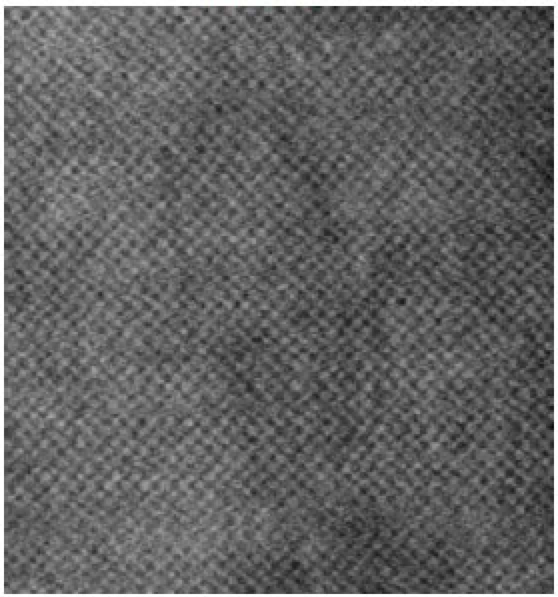


## Diffraction mode   Image mode

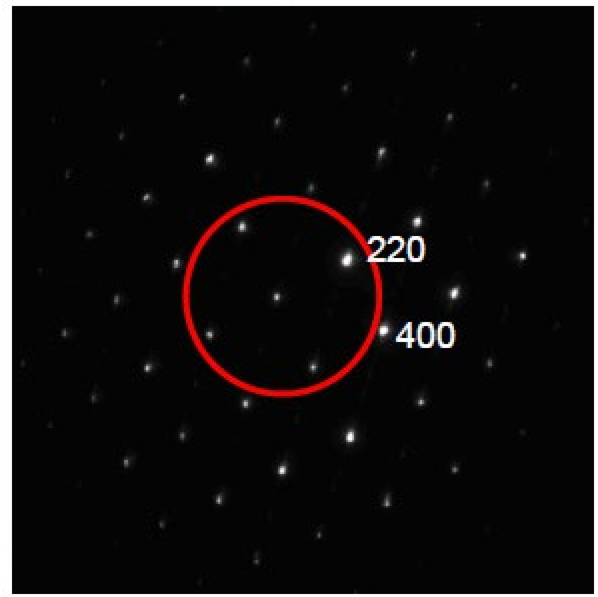


# *Electron Diffraction and Imaging a [100] Silicon Crystal*

Image



Diffraction Pattern



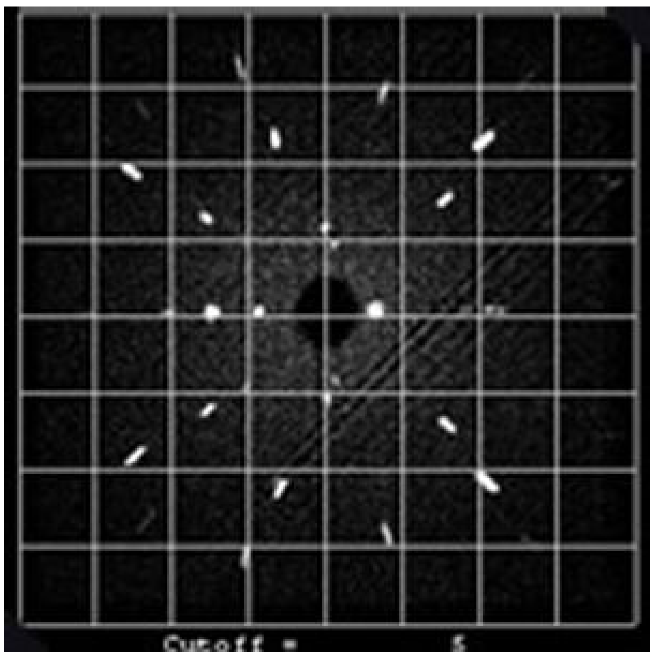

$\lambda$=0.0251Å

In Si $d_{220}$ = 1.92 Å

# *X-ray and Electron Diffraction from a Silicon Crystal*

Bragg's Law: $n\lambda = 2d\sin\theta$

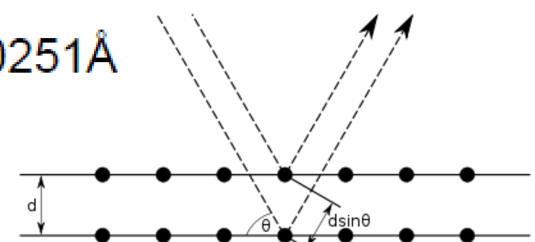
10 keV x-rays



$\lambda$=1.54 Å

200 keV Electrons

$\lambda$=0.0251Å




In Si $d_{220}$ = 1.92 Å

# Spot pattern

$$F_{hkl} = \sum_i f_i e^{2\pi i (hx_i + ky_i + lz_i)}$$

- Single crystal within the illumination area
- The regular arrangement of spots
- Spot brightness relates to the structure factor
- Spot position relates to the d-spacing

for fcc, the basis vector is

$$(x, y, z) = (0,0,0), \left(\frac{1}{2}, \frac{1}{2}, 0\right), \left(\frac{1}{2}, 0, \frac{1}{2}\right), \left(0, \frac{1}{2}, \frac{1}{2}\right)$$

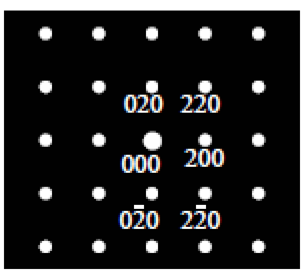
$$\text{so } F = f\left\{1 + e^{\pi i(h+k)} + e^{\pi i(h+l)} + e^{\pi i(k+l)}\right\}$$

$F = 4f$, if h, k, l are all even or all odd

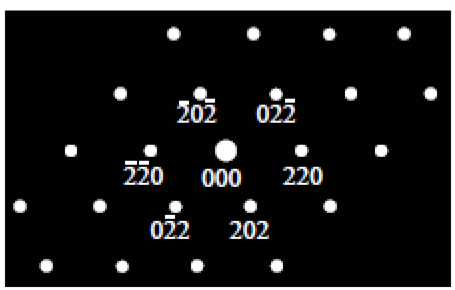
$F = 0$, if h, k, l are mixed even and odd

hkl – Miller indices

- Example 1: f.c.c

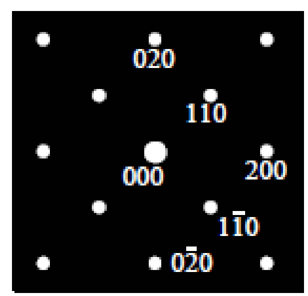



[001]




$[\bar{1}11]$

- Example 2: b.c.c

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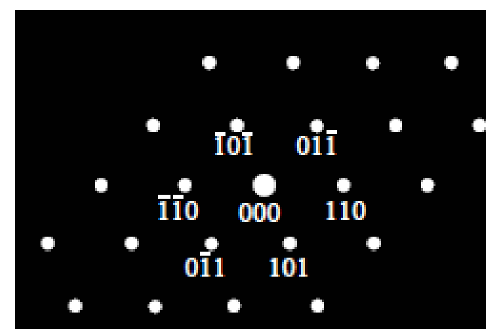
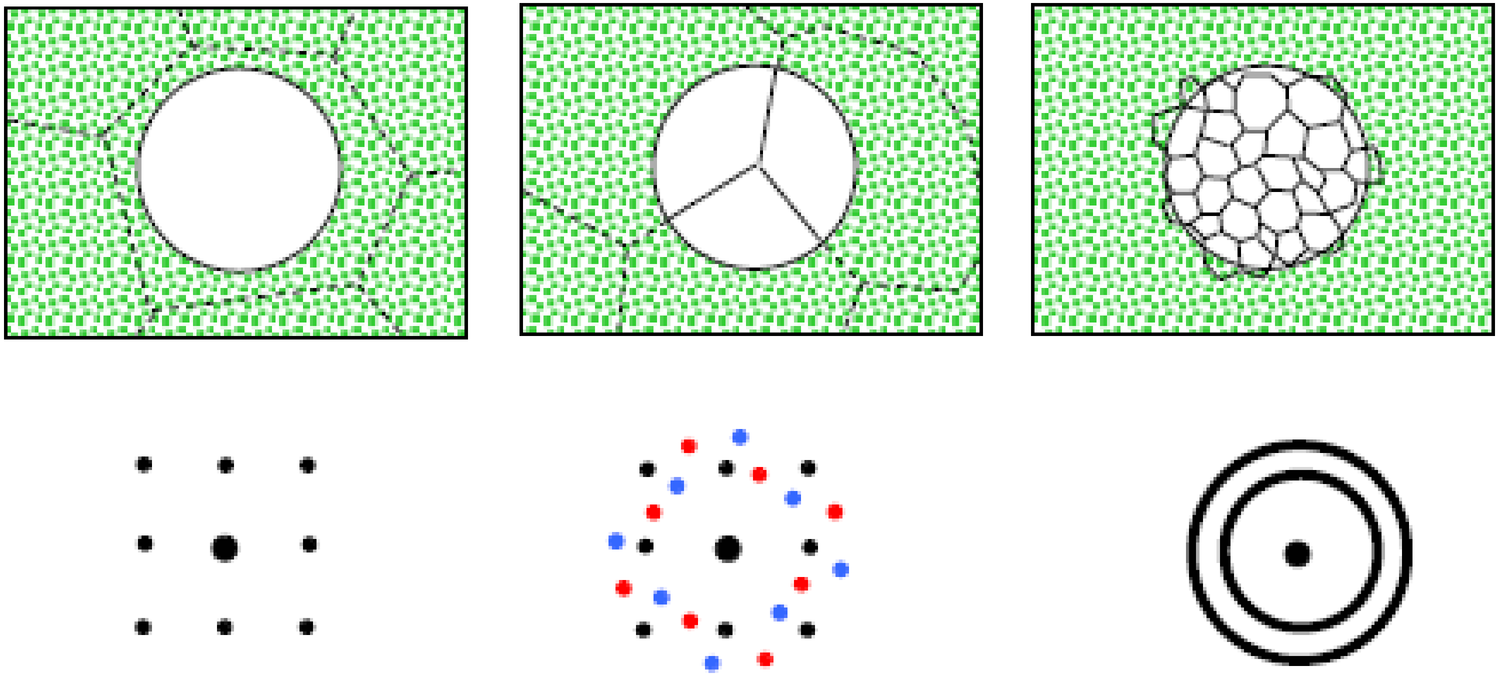


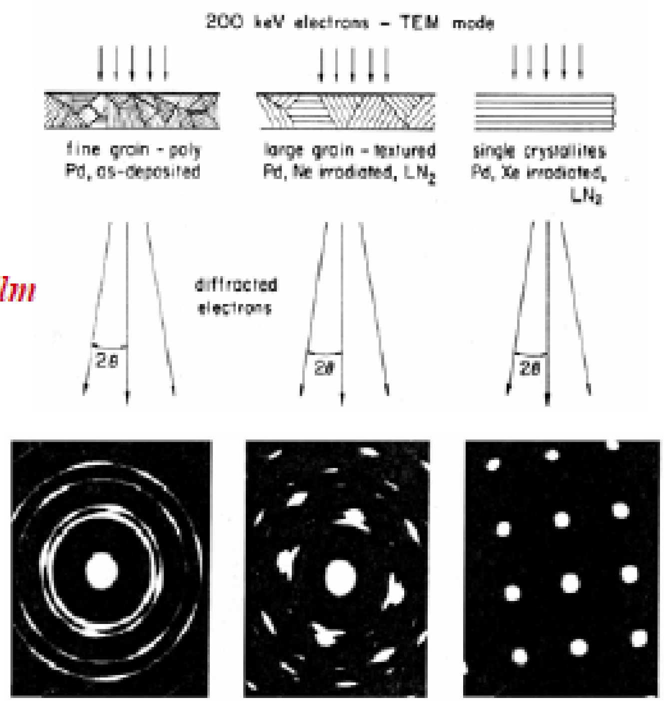

$[\bar{1}11]$

# Electron Diffraction Pattern--Spot to Ring



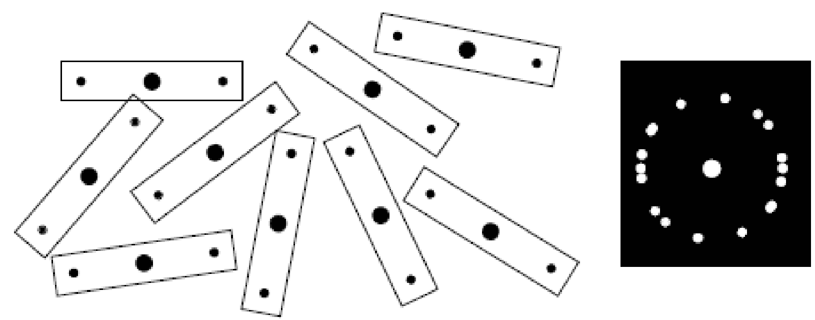
(a) (b) (c)

***Electron Beam Diffraction of a Pd film***



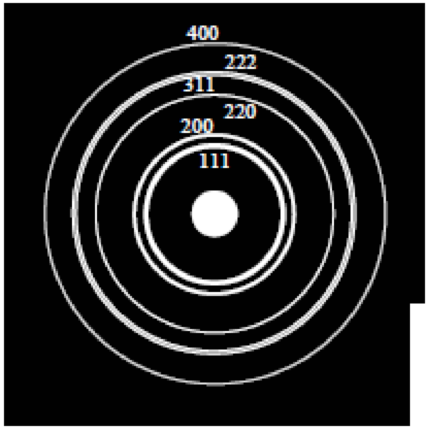

## Ring pattern

- Many fine particles in the illumination area, each of them is a single crystal and orientated randomly



# Ring pattern

- Typical polycrystalline Au diffraction pattern




# Ring pattern: what can we obtain

- d-spacing

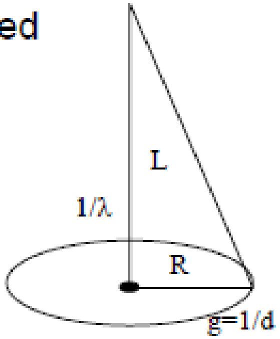
$$2Rd_{hkl} = L\lambda$$

  R: the measured ring radius

  $d_{hkl}$: the d-spacing being measured

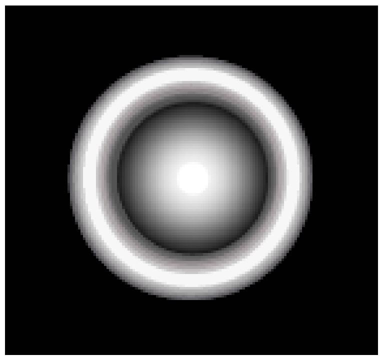
  L: camera length

  $\lambda$: wave length of electron beam
- Camera length calibration
- Crystalline / particle fineness

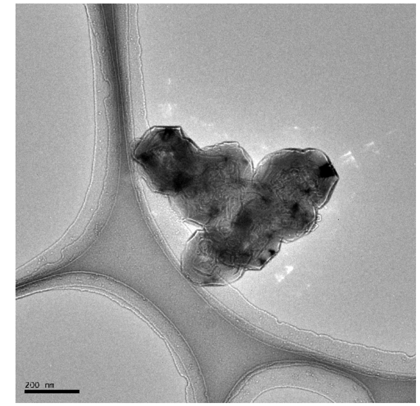


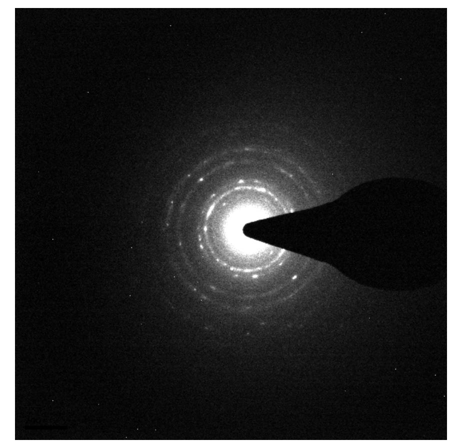
# Amorphous materials

- Diffused ring pattern
- Reflecting the short range ordered structure
- Often seen at contamination layer or on carbon support film



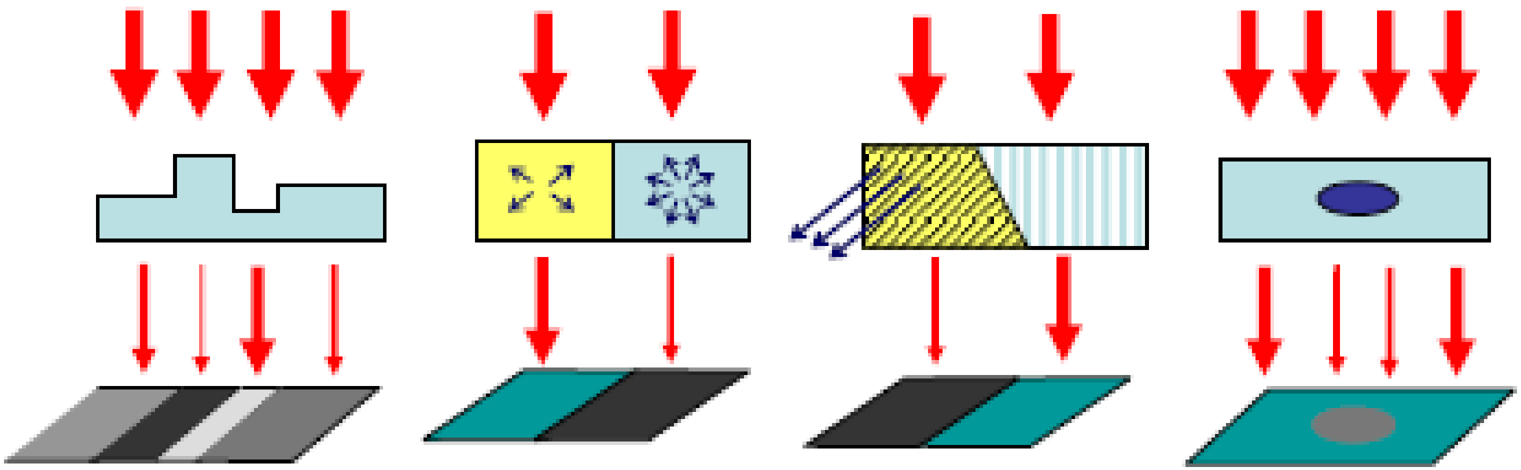
ADT-TES-F



d-spacings: 1.26 – 3 Å

# Major Factors affecting TEM Image Contrast



| Thickness | Atomic number | Orientation | Defects |
|---|---|---|---|
| *Scattering* | *Scattering* | *Diffraction* | *Scattering & diffraction* |

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# Four-Dimensional Electron Microscopy

Ahmed H. Zewail

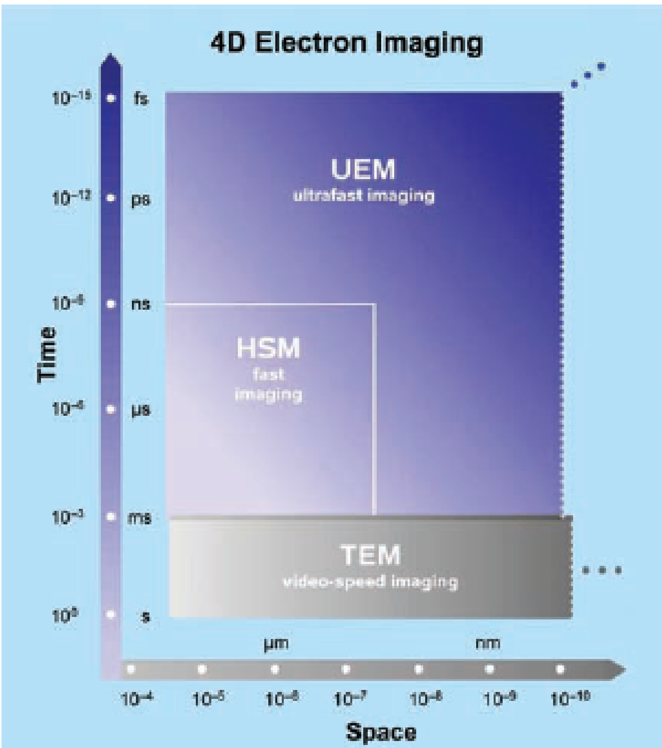



**Fig. 1.** 4D electron imaging. The resolution boundaries of ultrafast imaging are compared with those achieved in conventional TEM, limited by the speed of video camera, and, in high-speed microscopy (HSM), defined by the rectangle shown. The spatiotemporal scales of UEM achieved to date are outlined with possible future extensions. The approaches of single-electron and single-pulse imaging are fundamentally different because of the limiting problem of space-charge described in the text.

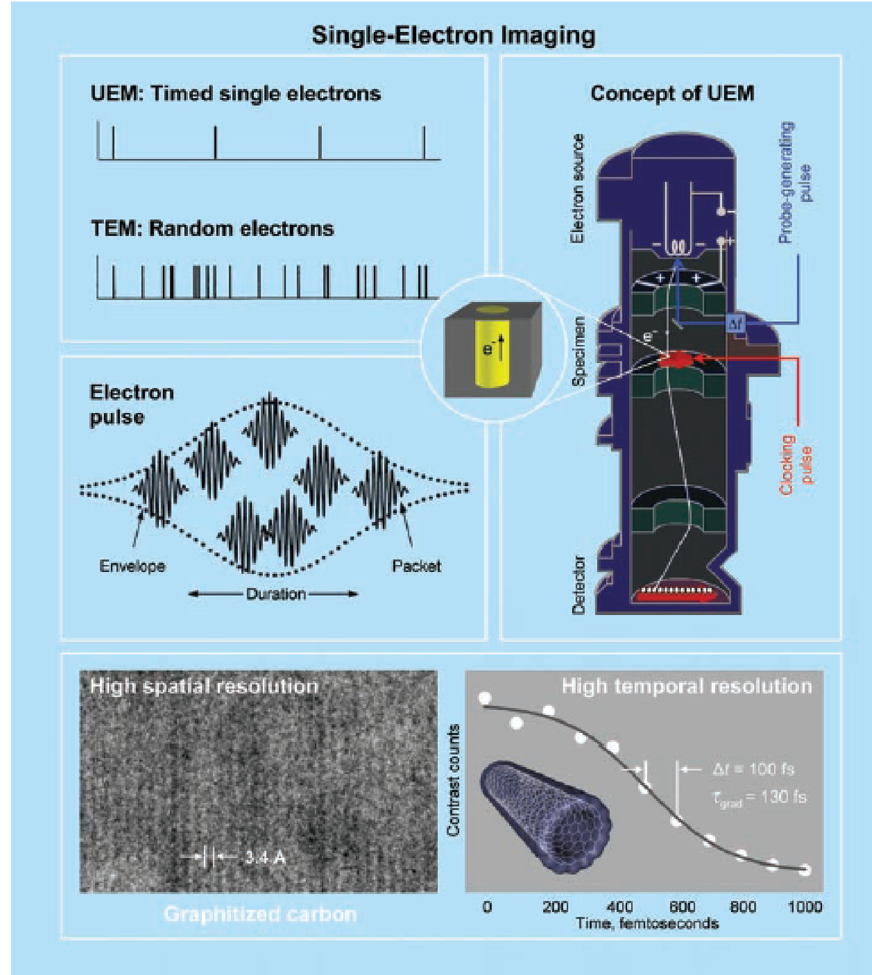


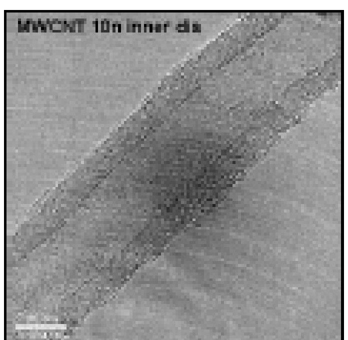

**Fig. 2.** Conceptual and experimental realization of single-electron imaging. Shown are schematics of timed single-electron packets (UEM) and random electrons (conventional TEM) used in imaging (upper left). Also depicted is the electron-pulse envelope together with individual electron packets (center left). A single-electron trajectory is schematized in the microscope together with an illustration of what is meant by a coherence volume (upper right; see text). The bottom panel displays a high-resolution UEM image of graphitized carbon with lattice-plane separations of 3.4 Å (left), and a temporal profile obtained from the images of evanescent fields in carbon nanotubes; the time steps are of 100-fs duration, and the rate of change is given by the gradient ($\tau_{grad}$ = 130 fs), which is determined by the materials response and pulse widths involved (see the section on near-field UEM and Fig. 5). The experimental data are from (*10*, *46*).

# Scanning Electron Microscope

- Sequential imaging similar to the optical scanning confocal microscope
- Can be used in reflection or transmission modes (STEM)

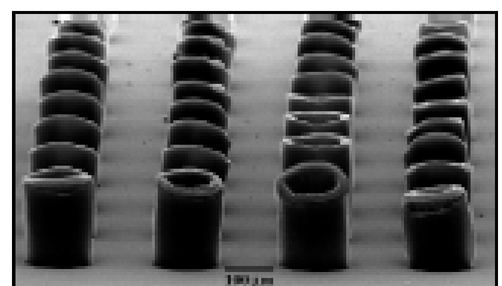
**TEM**

- Resolution: atomic
- Can determine 3-D structure
- Be careful of $e^-$ - $e^-$ interactions




**SEM**

- Resolution: atomic
- Surface features
- Requires Vacuum
- Often must coat specimens



NASA nanotech group

# SEM capabilities

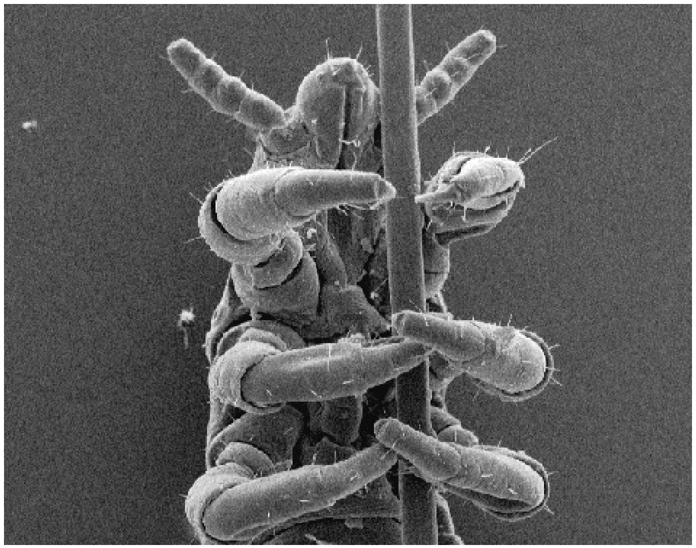
**Topography**
**The surface features of an object or "how it looks", its texture; direct relation between these features and materials properties**

**Morphology**
**The shape and size of the particles making up the object; direct relation between these structures and materials properties**

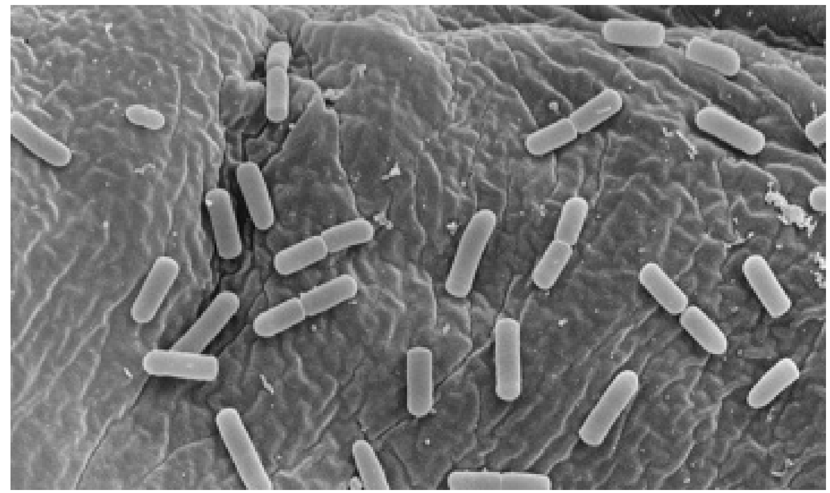
**Composition**
**The elements and compounds that the object is composed of and the relative amounts of them; direct relationship between composition and materials properties**

**Crystallographic Information**
**How the atoms are arranged in the object; direct relation between these arrangements and material properties**

# Some SEM images



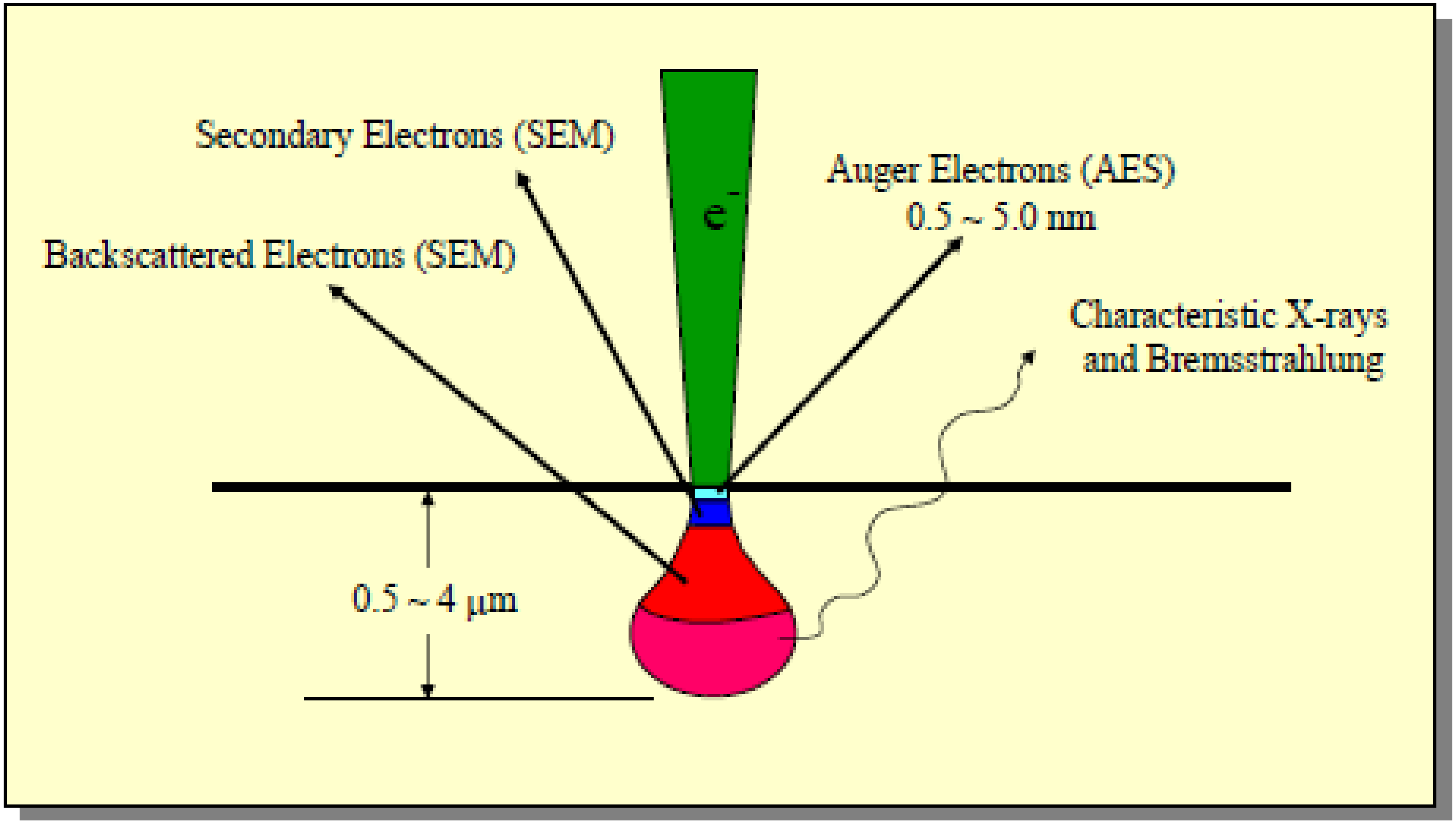
Insect climbing up a straw



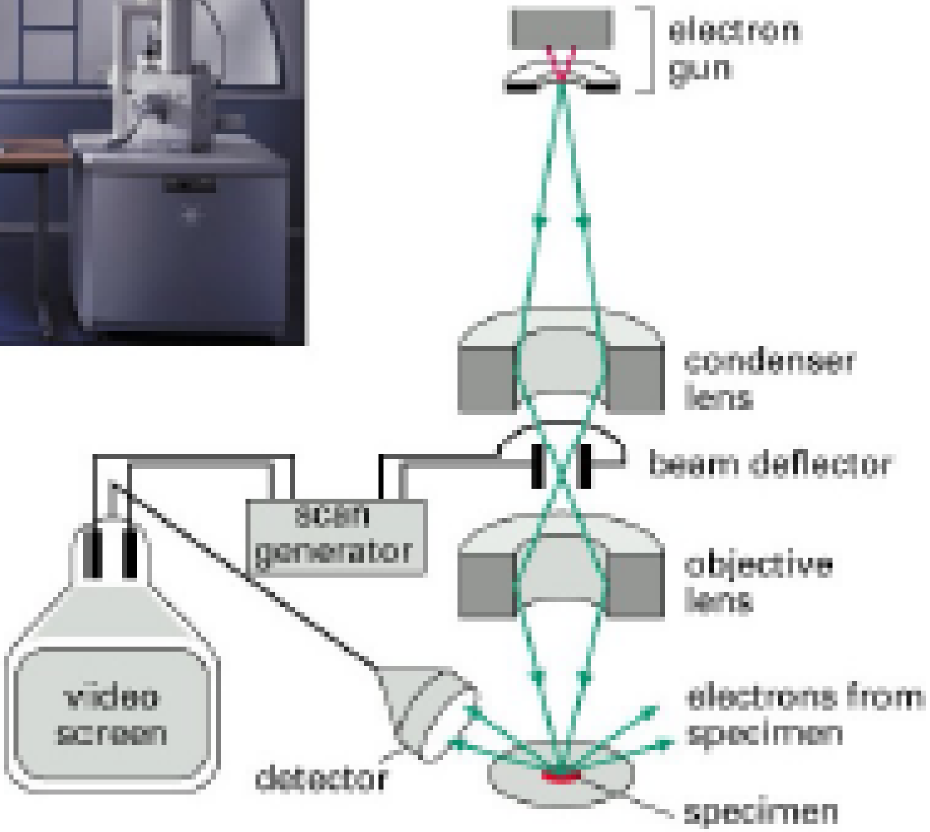
bacteria distributed over the surface of shredded lettuce leaf at the end of its shelf life

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

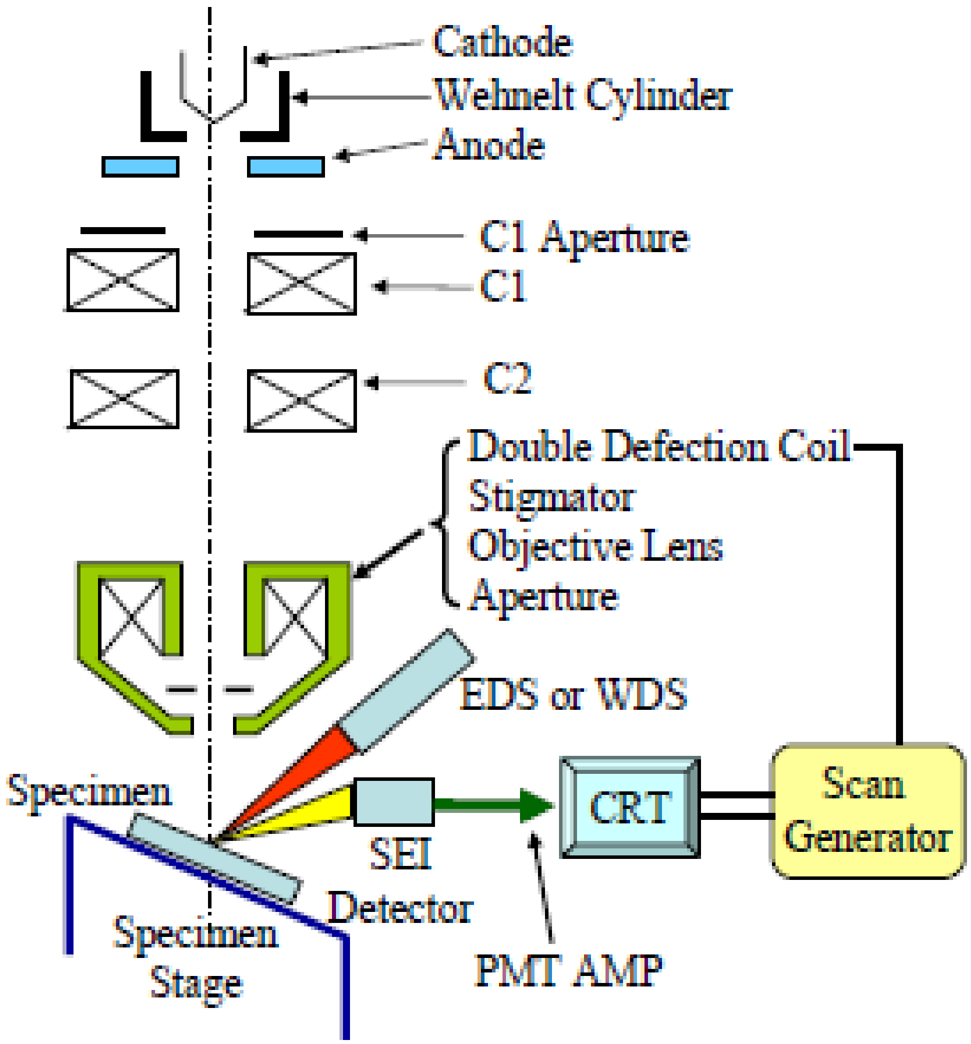
## Interaction with a thick specimen (SEM)



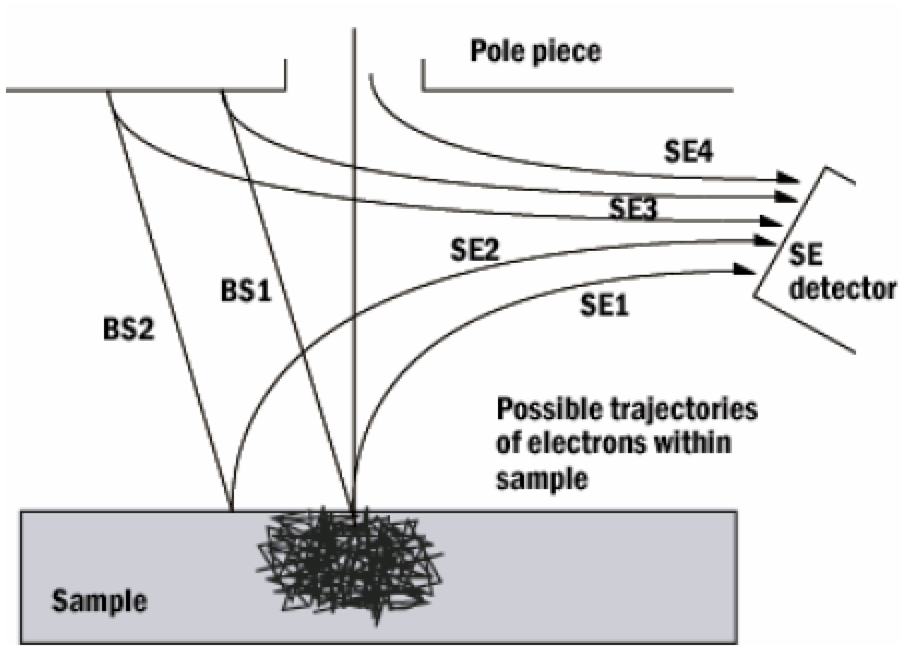
# Scanning Electron Microscope



# Lens System of SEM

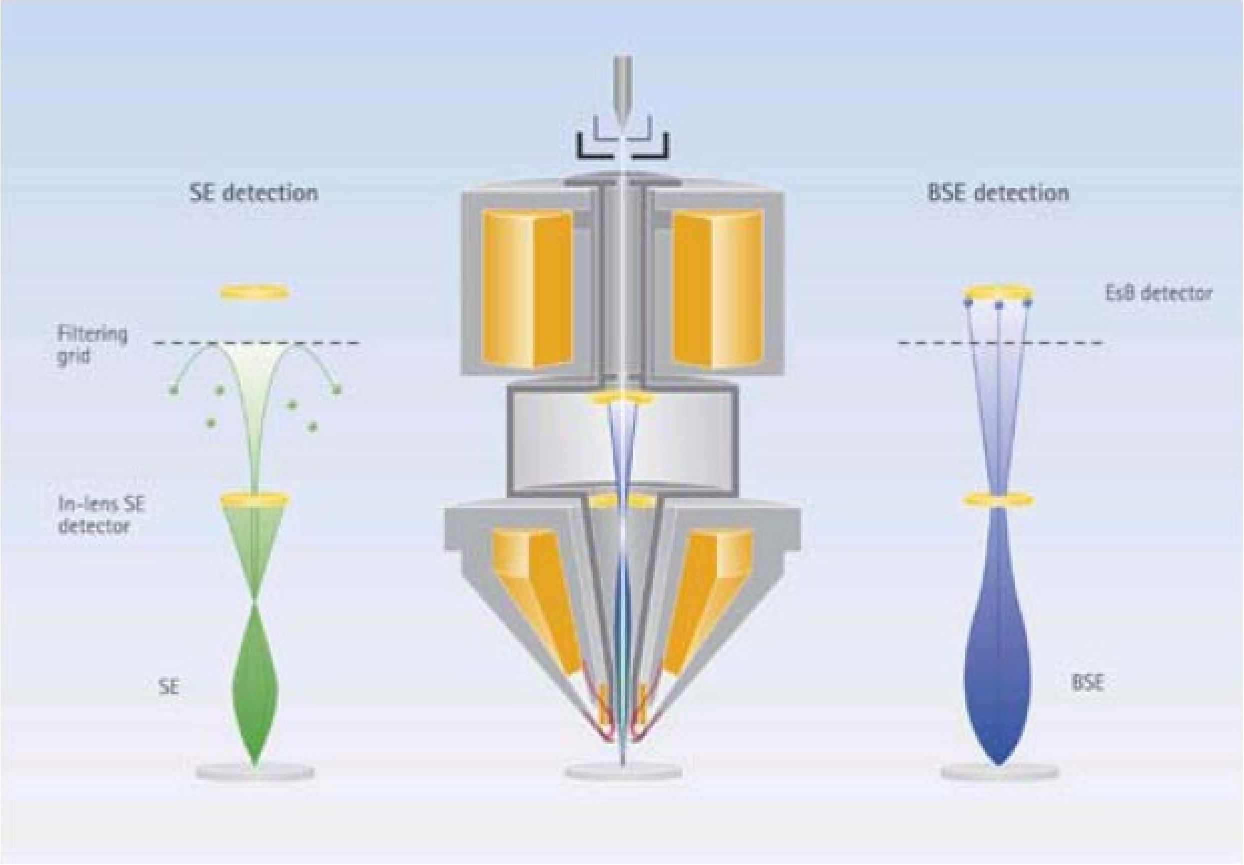


# Detectors: Secondary Elec. Det.

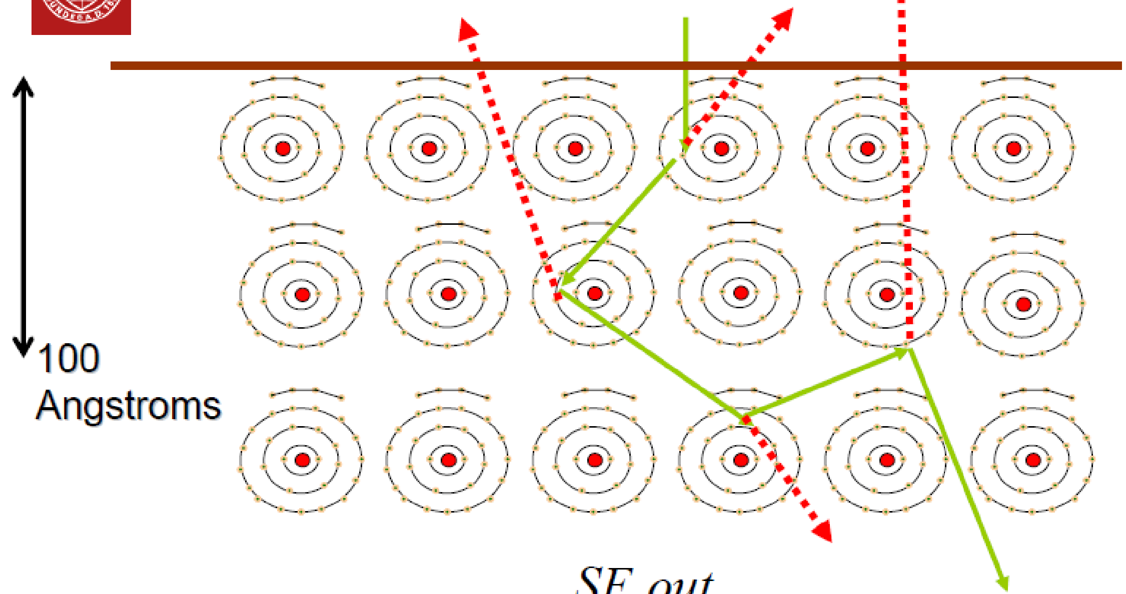



- Secondary electrons generally low energy (50-100 eV)
- Trajectories can be bent easily by biasing the detector
- Low Z contrast

# Energy filtered detection of electrons

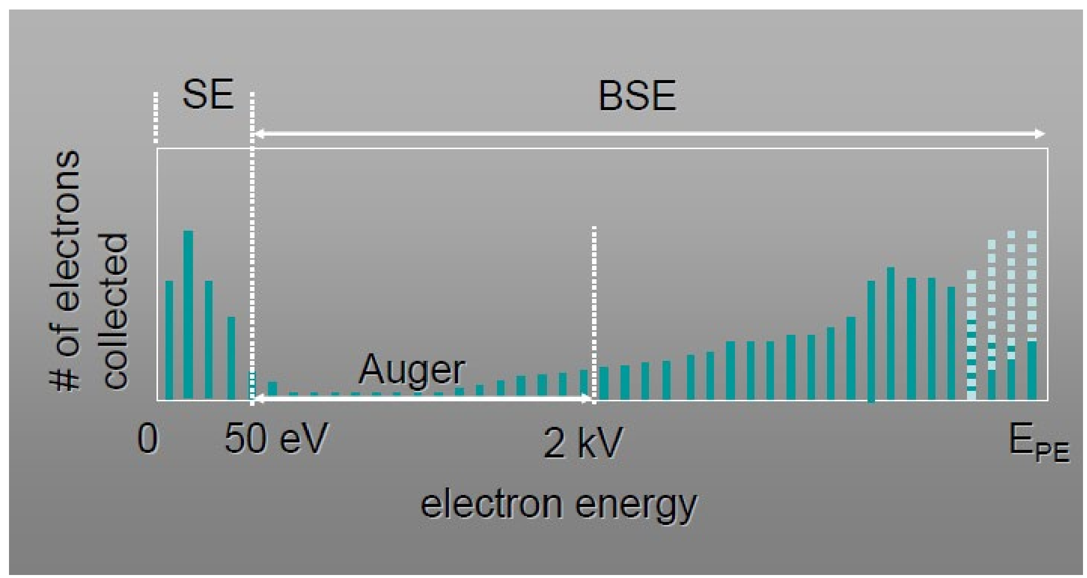


# *One Primary Electron In Can Create Several SEs Out at Low Accelerating Voltages*



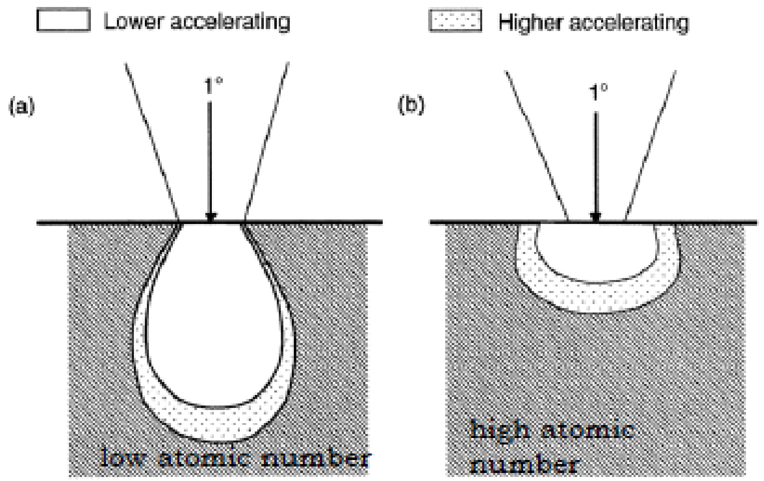

Secondary Electron Yield Coefficient

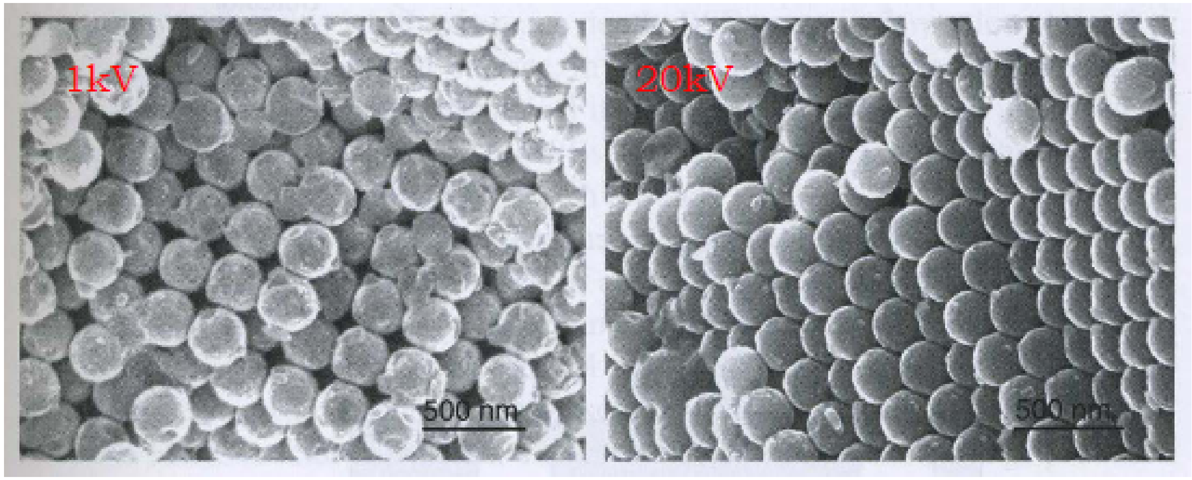
$$\delta = \frac{SE\ out}{PE\ in}$$



# Secondary electrons

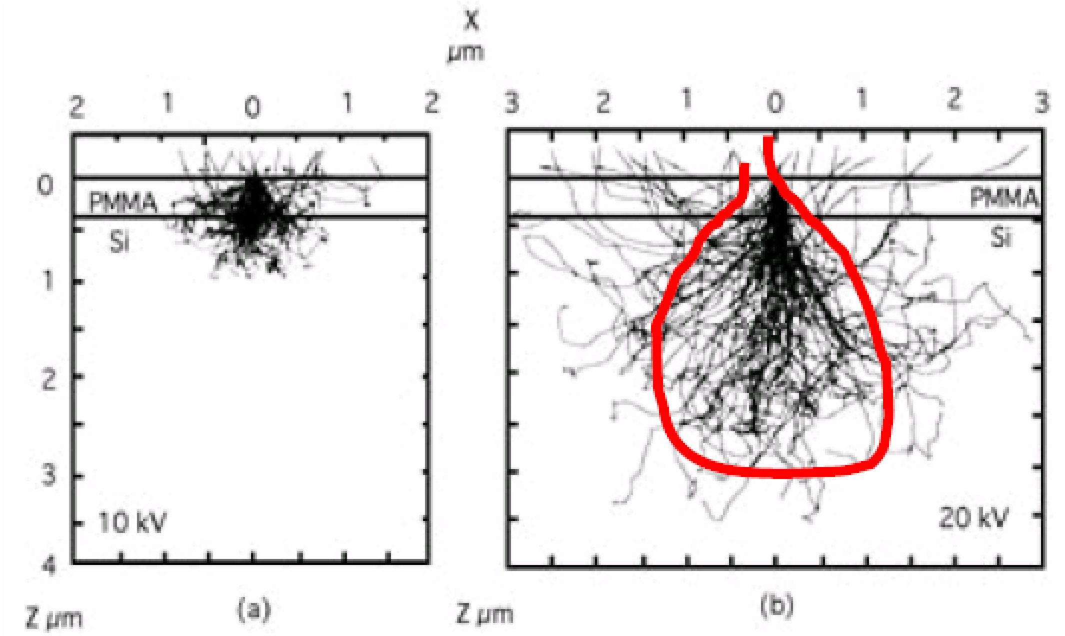
- Low energy
- Topographic contrast (surface texture and roughness)
- Resolve surface structure down to 10nm
- Excitation region depends on the accelerating voltage



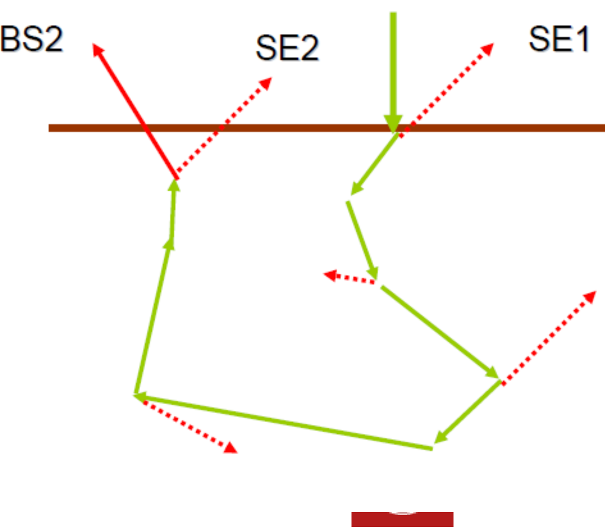



# Electron Beam and Sample Interaction

- Depends on energy of beam, material of the sample. The beam penetrates the sample
- Beam Spot size isn't everything



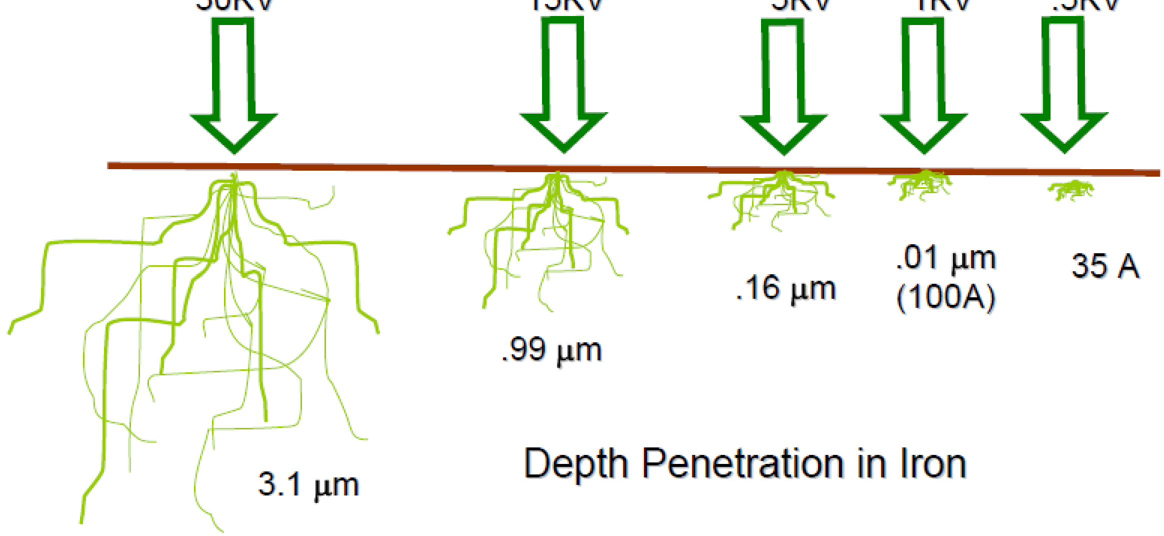
## *Path of the Electron Beam*




## *Kanaya-Okayama Depth Penetration Formula*

$$R = \frac{0.0276\ A\ E^{1.67}}{(Z^{0.89}\ \rho)}\ \mu m$$

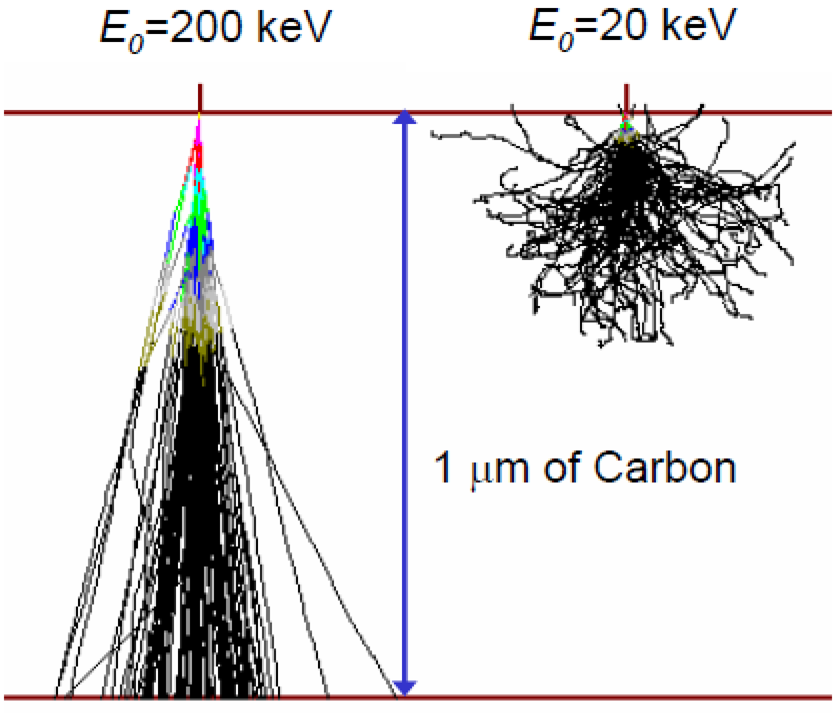
R= Depth Penetration
A= Atomic Weight (g/mole)
E= Beam Energy (KV)
Z= Atomic number
$\rho$ = density $(g/cm)^2$




Depth Penetration in Iron

(predictions from the KO formula)

# *Beam Spreading*




Electron Range (in μm):

$$R \approx \frac{0.064}{\rho} E_0^{1.5}$$

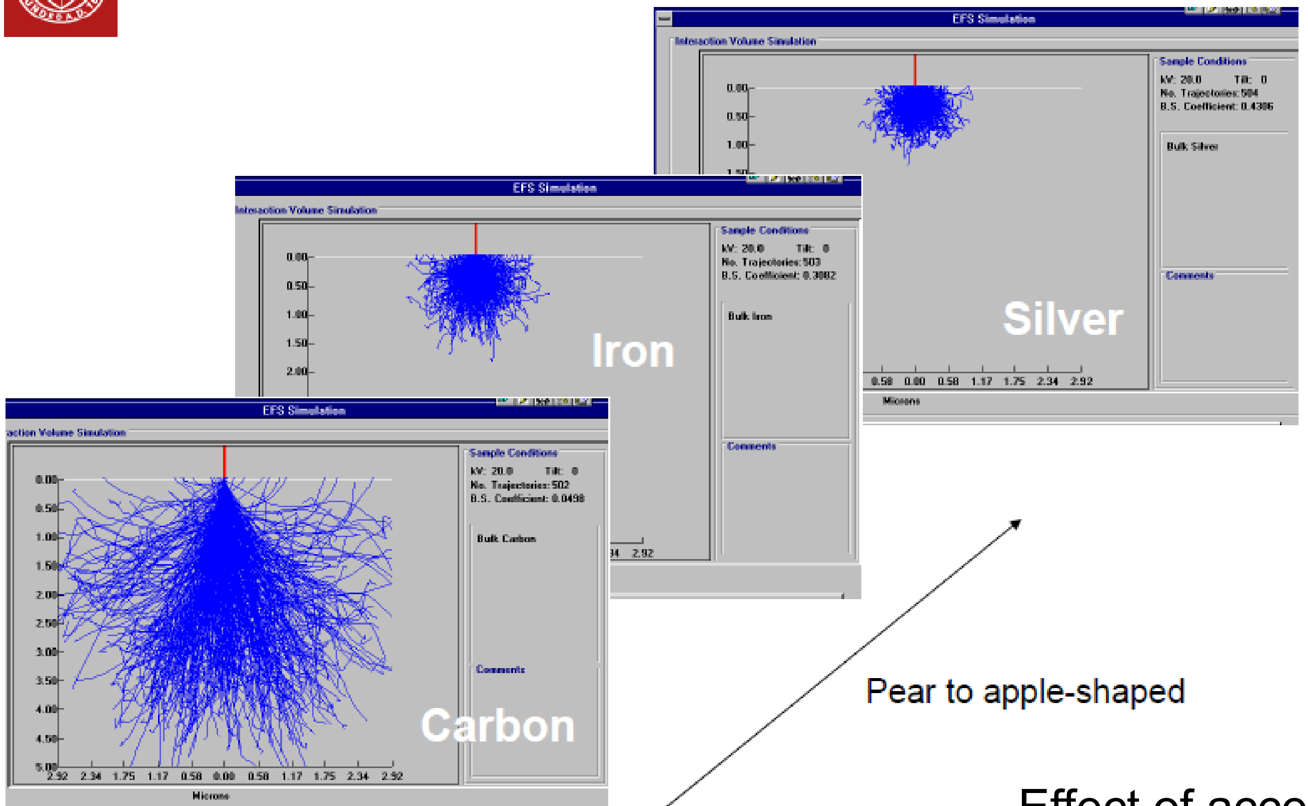
(density ρ in g/cm³, $E_0$ in keV)

R~ 100 μm at 200 keV

David Joy's simulation code is available at http://web.utk.edu/~srcutk/htm/simulati.htm
A more detailed simulator can be found at http://www.gel.usherbrooke.ca/casino/What.html

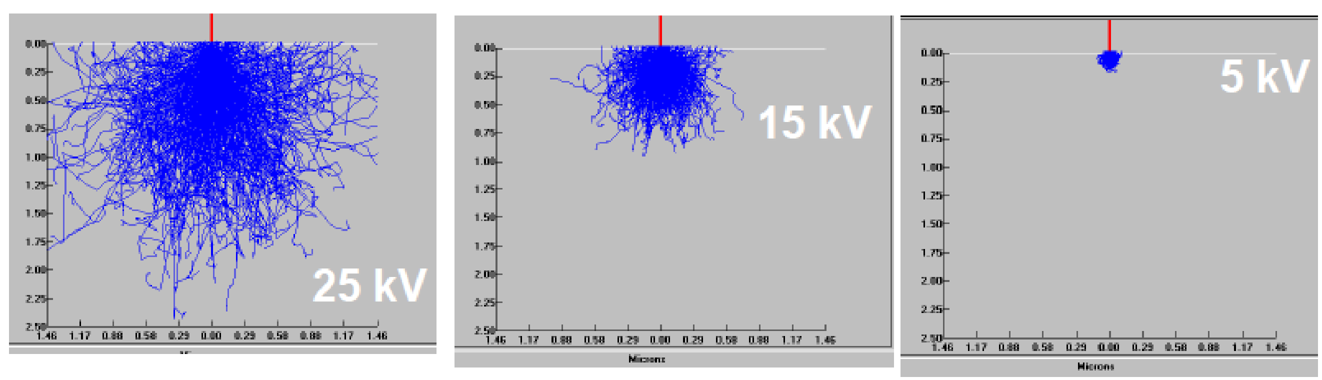
# *Interaction Volume –Sample Composition*

(20 kV incident beam in all 3 cases)




Pear to apple-shaped

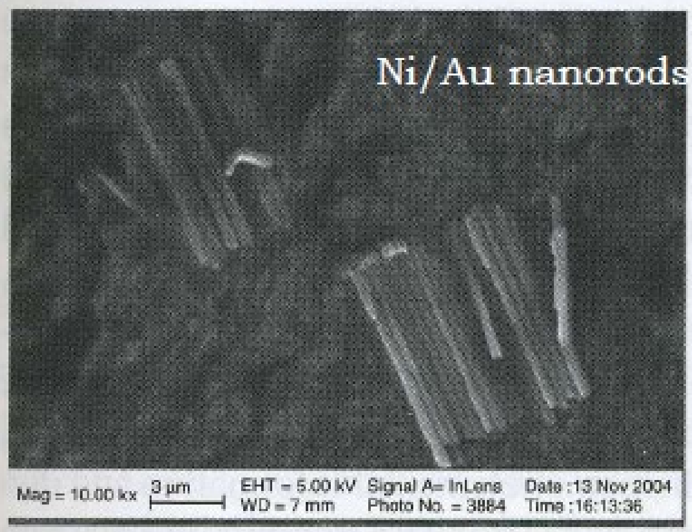
## Effect of accelerating voltage



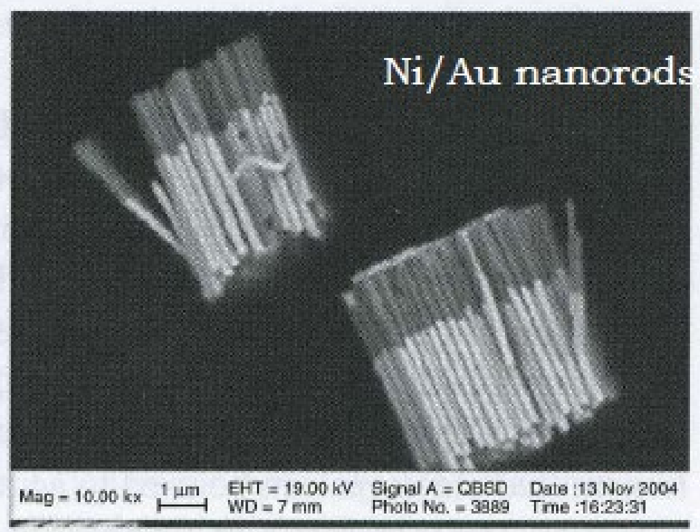
# Backscattered electrons

- High energy
- Both Compositional and Topographic information
- Atomic number contrast
- Lateral resolution is worse than secondary electron image

Secondary electron image

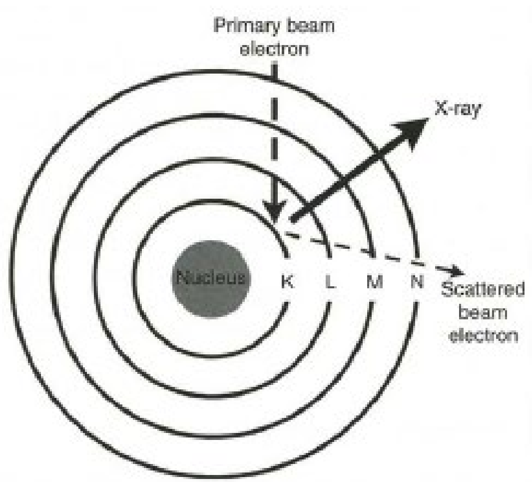


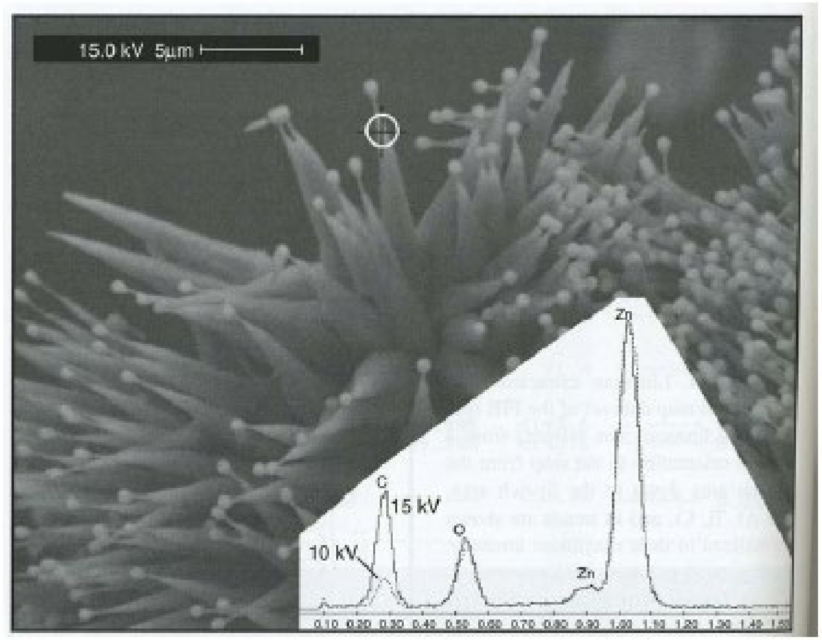

Backscattered electron image



# Characteristic X-ray

- Chemical information of sample
- Energy Disperse X-ray Spectroscopy (EDS)

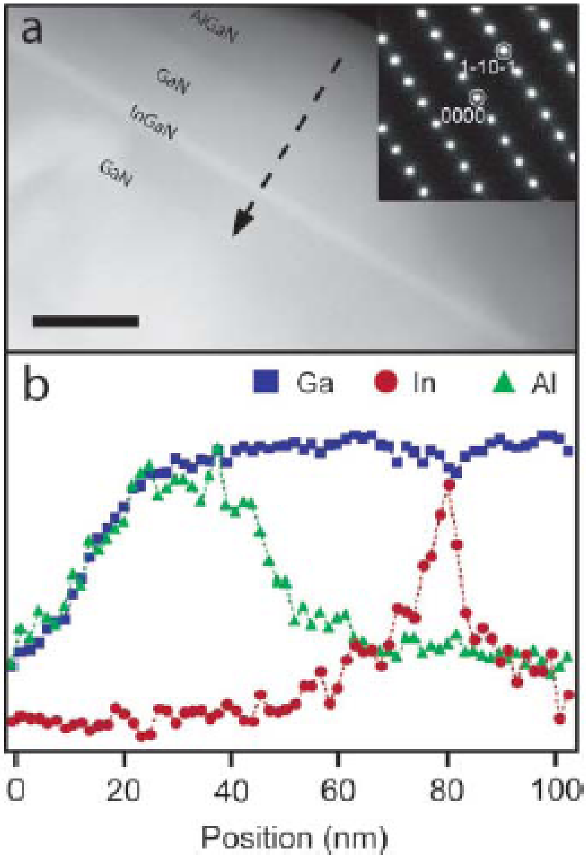


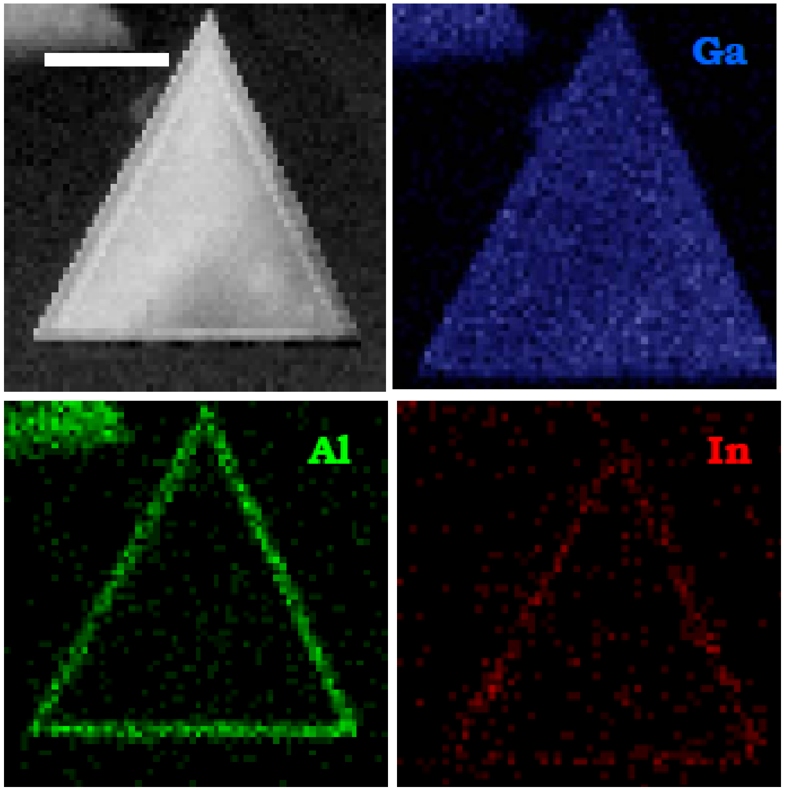

Detection area is limited by the resolution of SEM (accelerating voltage of electron)

# Energy Disperse X-ray Spectroscopy (EDS)

Line scan

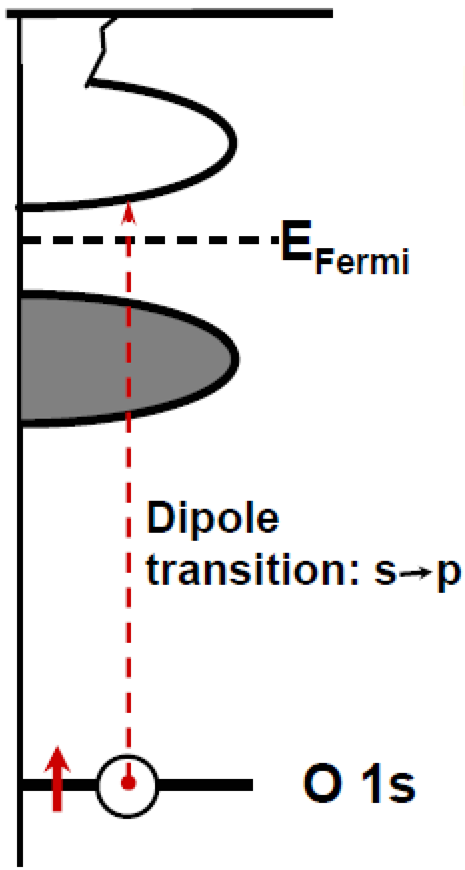



Elemental mapping




Highly resolved spatial distribution of elements in specimen

# Core-Level Electron Energy Loss Spectroscopy




EELS measures a local density of states partitioned by

- •site - as the probe is localized,
- •element - the core level binding energy is unique

- probes the conduction band
- provides local electronic information

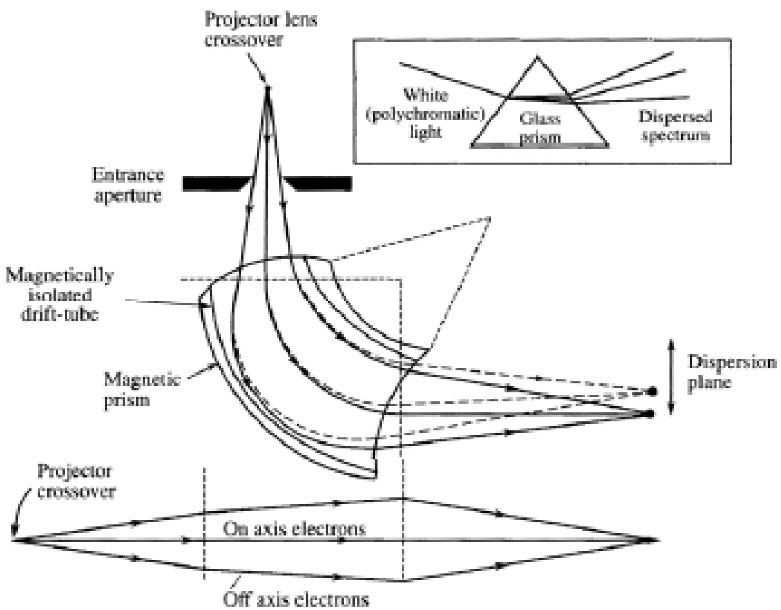
In the first Born approximation, the partial cross section for the inelastic scattering of an electron wave packet (with initial group velocity $v$), undergoing a momentum transfer $\vec{q}$ and losing energy $E$,[58–60] is given by

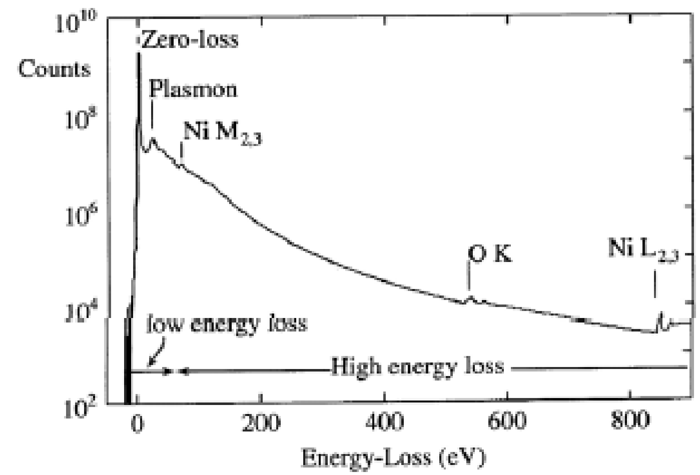
$$\frac{d^2\sigma(E,q)}{dE\,dq} = \frac{8\pi e^4}{\hbar^2 v^2}\frac{1}{q}\sum_{i,f} |\hat{\epsilon}_q \cdot \langle f|\vec{r}|i\rangle|^2 \delta(E - E_f + E_i) + \cdots$$

*Some subtleties as to which density of states is measured see Muller, Singh and Silcox, Phys Rev* ***B57****, 8181 (1998)*

# Electron Energy Loss Spectroscopy (EELS)

Magnetic prism spectrometer



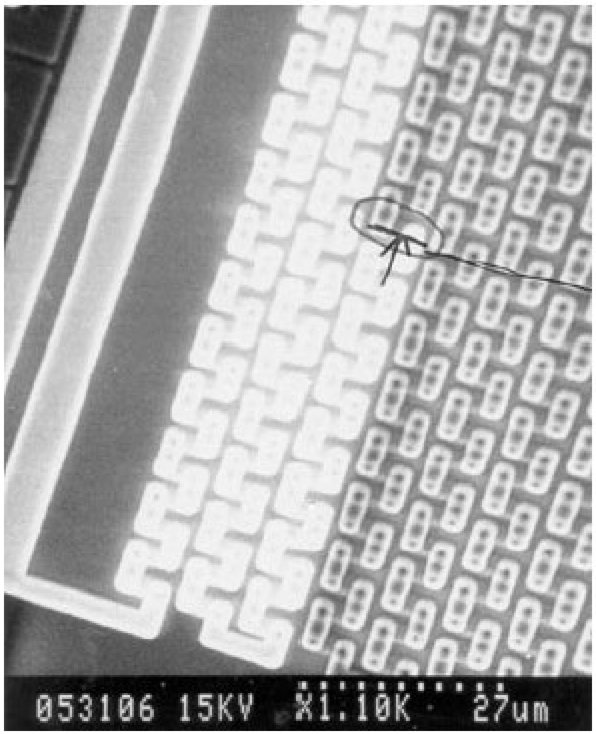




- Absorption spectroscopy
- Inelastic scattered electrons

- Complementary to EDS
- High energy resolution
- Atomic composition, chemical bonding, valence and conduction band electronic properties and surface properties
- Ability to fingerprint different forms of the same element

- *EDS*
  - *Low collection efficiency (small solid angle)*
  - *Good peak/background*
  - *Good for high-Z elements*
  - *SEM (~500 nm) or TEM (~10 nm)*
  - *Stray x-rays in the column can be mistaken for trace elements*
- *EELS*
  - *TEM only & thin samples*
  - *High collection efficiency (~90%)*
  - *Poor peak/background (esp in thick samples)*
  - *Best for low-Z elements (large signals)*
  - *Bonding information for Z<33*
  - *High spatial resolution (0.1 – 1 nm)*

# Voltage contrast with SE




*The floating end of the via chain is bright because of trapped negative charge causes secondary electrons to be repelled.*

*The remainder of the chain is neutral, and thus darker.*

(http://www.acceleratedanalysis.com/hepvc.html)

## Sample Coating

Q: Why ?

A: Charging:

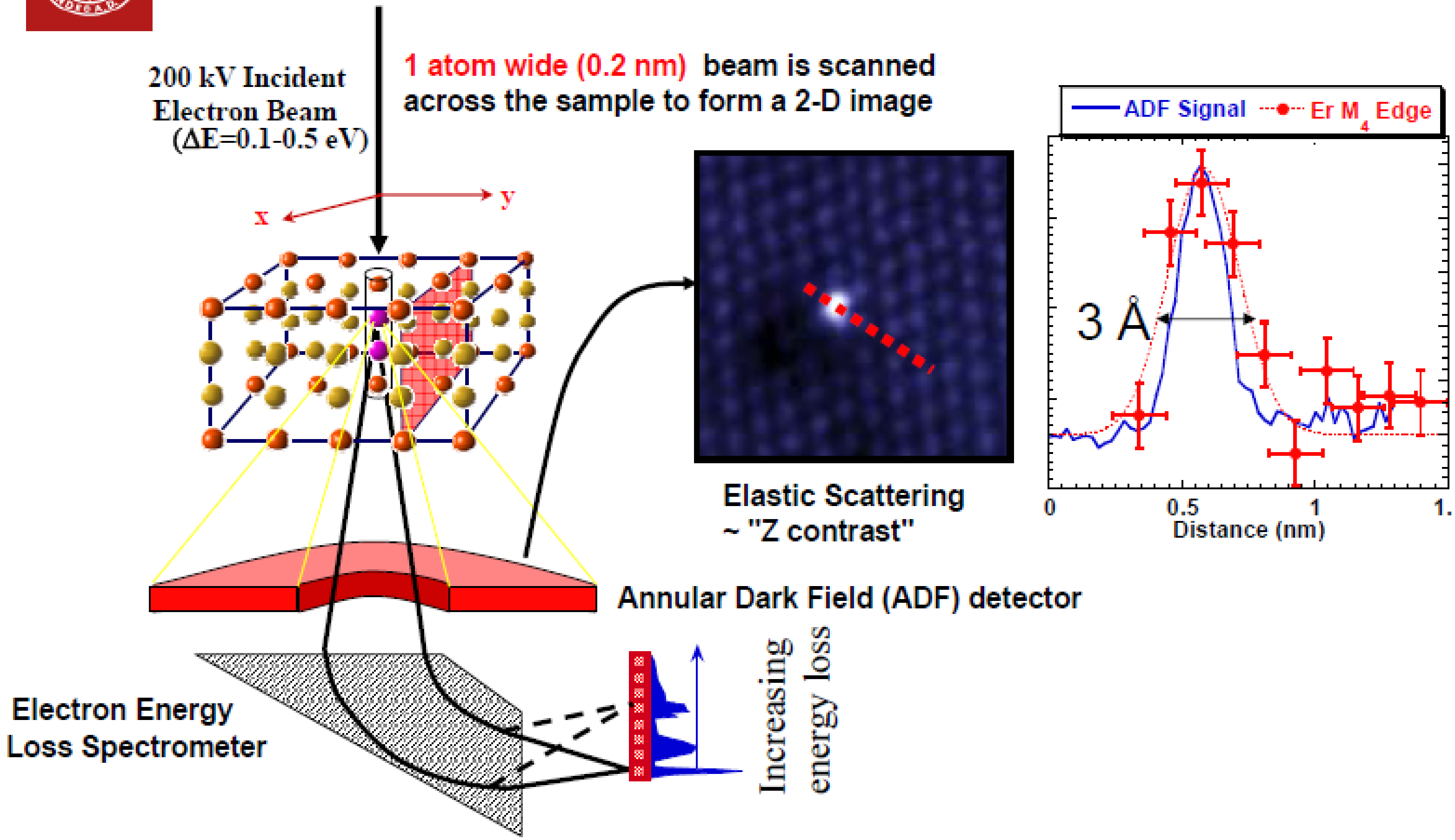
- Deflection of SE's
- Increased emission of SE's in cracks
- Periodic SE bursts
- Beam deflection

Solutions:

- Sputter coating with C, Cr, or Au-Pd

- Carbon tape, carbon paint, In foil

# Scanning Transmission Electron Microscopy




Single atom Sensitivity: P. Voyles, D. Muller, J. Grazul, P. Citrin, H. Gossmann, *Nature* **416** 826 (2002)
U. Kaiser, D. Muller, J. Grazul, M. Kawasaki, *Nature Materials*, **1** 102 (2002)

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# LETTER

doi:10.1038/nature09718

## Grains and grain boundaries in single-layer graphene atomic patchwork quilts

Pinshane Y. Huang[1]*, Carlos S. Ruiz-Vargas[1]*, Arend M. van der Zande[2]*, William S. Whitney[2], Mark P. Levendorf[3], Joshua W. Kevek[4], Shivank Garg[3], Jonathan S. Alden[1], Caleb J. Hustedt[5], Ye Zhu[1], Jiwoong Park[3,6], Paul L. McEuen[2,6] & David A. Muller[1,6]

ADF-STEM

TEM