Do it with
electrons !

Microscopy

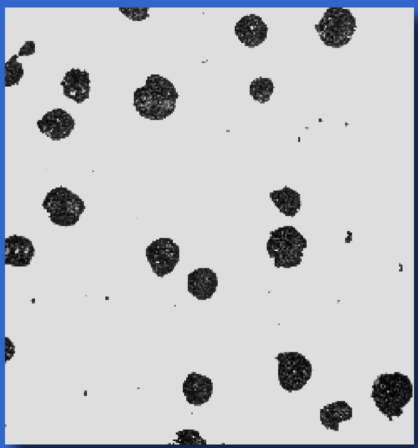
Structure determines properties

We have discussed crystal structure (x-ray diffraction)

But consider now different level of structure

Microstructure (微观结构)- also affects properties

Grey cast iron (灰色生铁) - rather brittle



Ductile iron - highly ductile

Microscopy

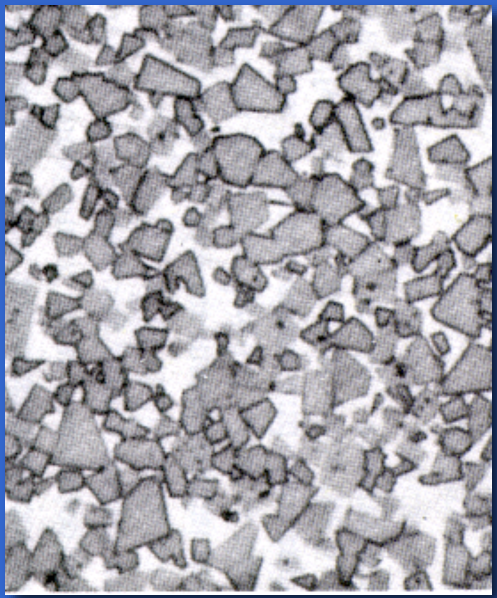
Structure determines properties

We have discussed crystal structure (x-ray diffraction)

But consider now different level of structure

Microstructure - also affects properties

Cemented WC (碳化钨)
cutting tool



Microscopy

Structure determines properties

We have discussed crystal structure (x-ray diffraction)

But consider now different level of structure

Microstructure - also affects properties

Ferroelectric domains in $BaTiO_3$

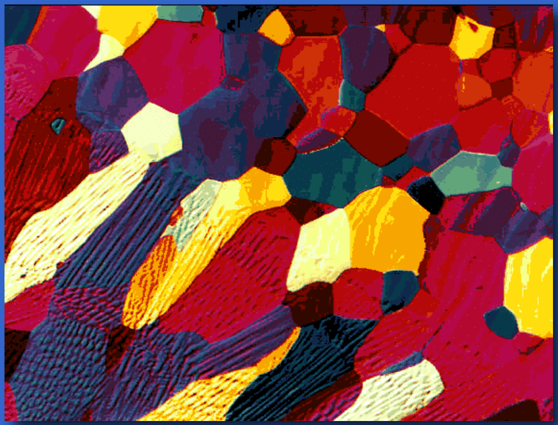
Microscopy

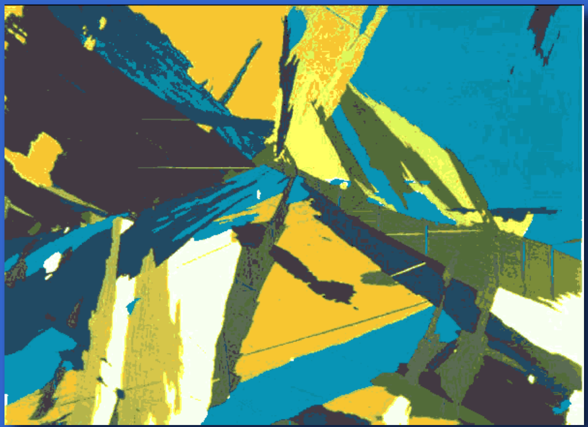
Structure determines properties

We have discussed crystal structure (x-ray diffraction)

But consider now different level of structure

Microstructure - also can be 'art (美术)'





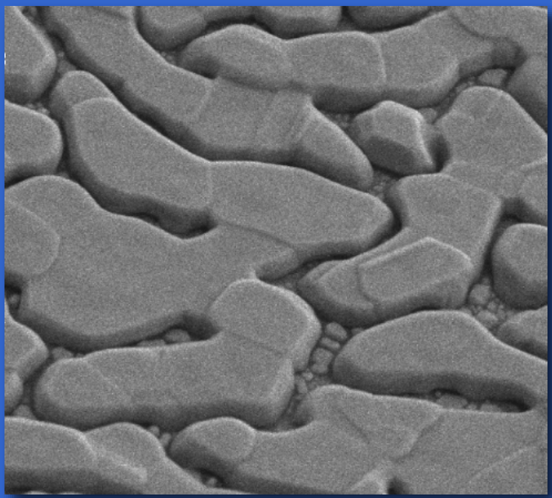
Electron microscopy

SEM - scanning electron microscopy

tiny electron beam scanned across surface of specimen

backscattered (背散射) or
secondary electrons (二次电子) detected

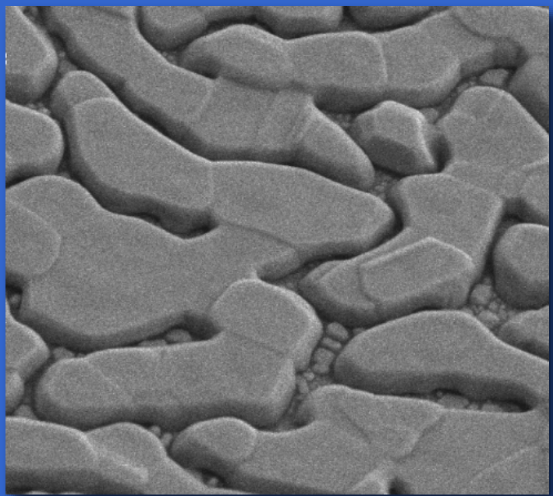
signal output to synchronized display



Electron microscopy

SEM - scanning electron microscopy

- Magnification range 15x to 200,000x
- Resolution of 50 Å
- Excellent depth of focus
- Relatively easy sample prep



SEM - scanning electron microscopy

Electron gun

Don't make x-rays - use electrons directly

Wavelength:

NOT $\lambda = hc/E$
(massless photons)

$\lambda = h/(2m_{electron}qV_o)^{1/2}$
(non-relativistic)

$\lambda = h/(2m_{electron}qV_o + q^2V_o^2/c^2)^{1/2}$
(relativistic (相对论的))

SEM - scanning electron microscopy

$$\lambda = h / (2m_{electron} q V_o + q^2 V_o^2 / c^2)^{1/2}$$

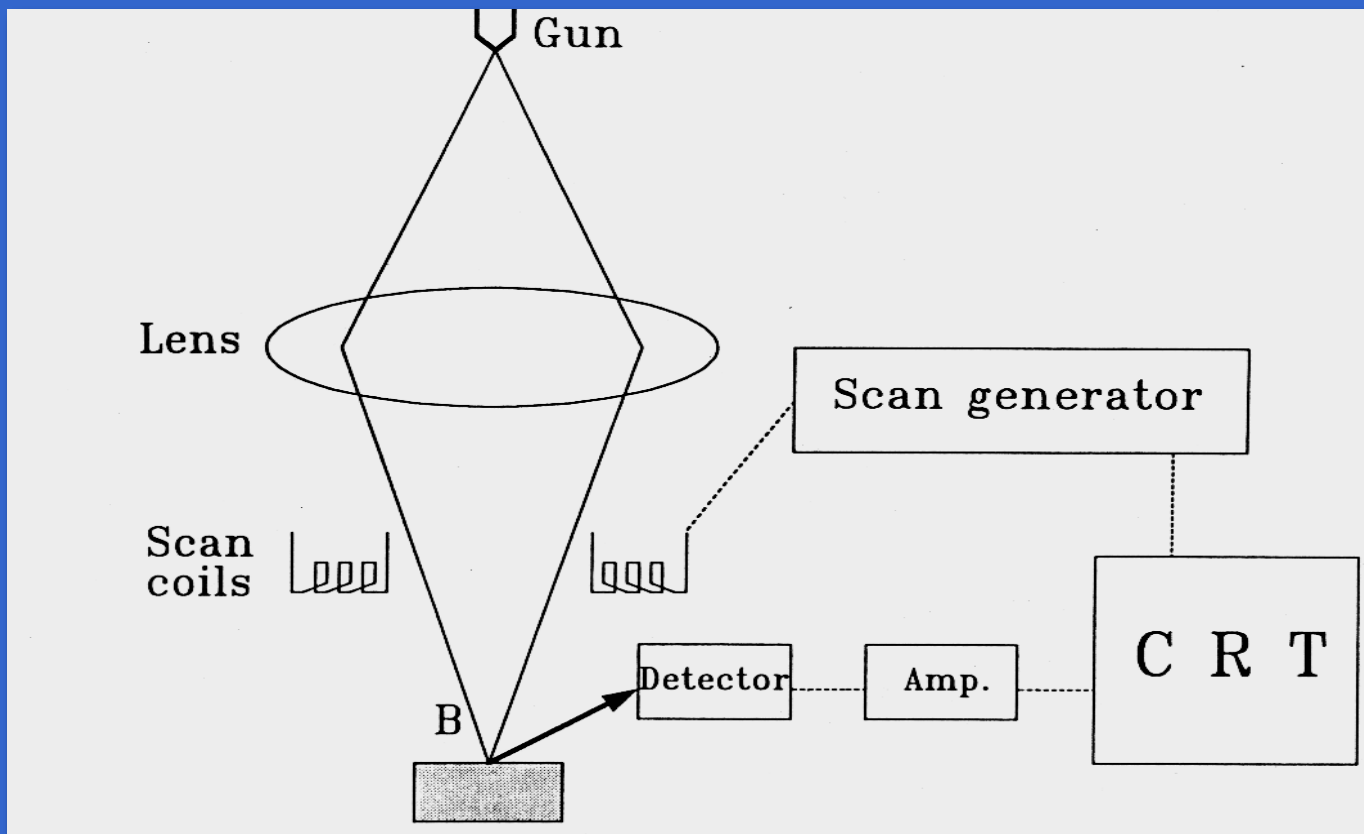
$$\lambda = 1.22639 / (V_o + 0.97845 \cdot 10^{-6} V_o^2)^{1/2}$$

λ(nm) & V_o(volts)

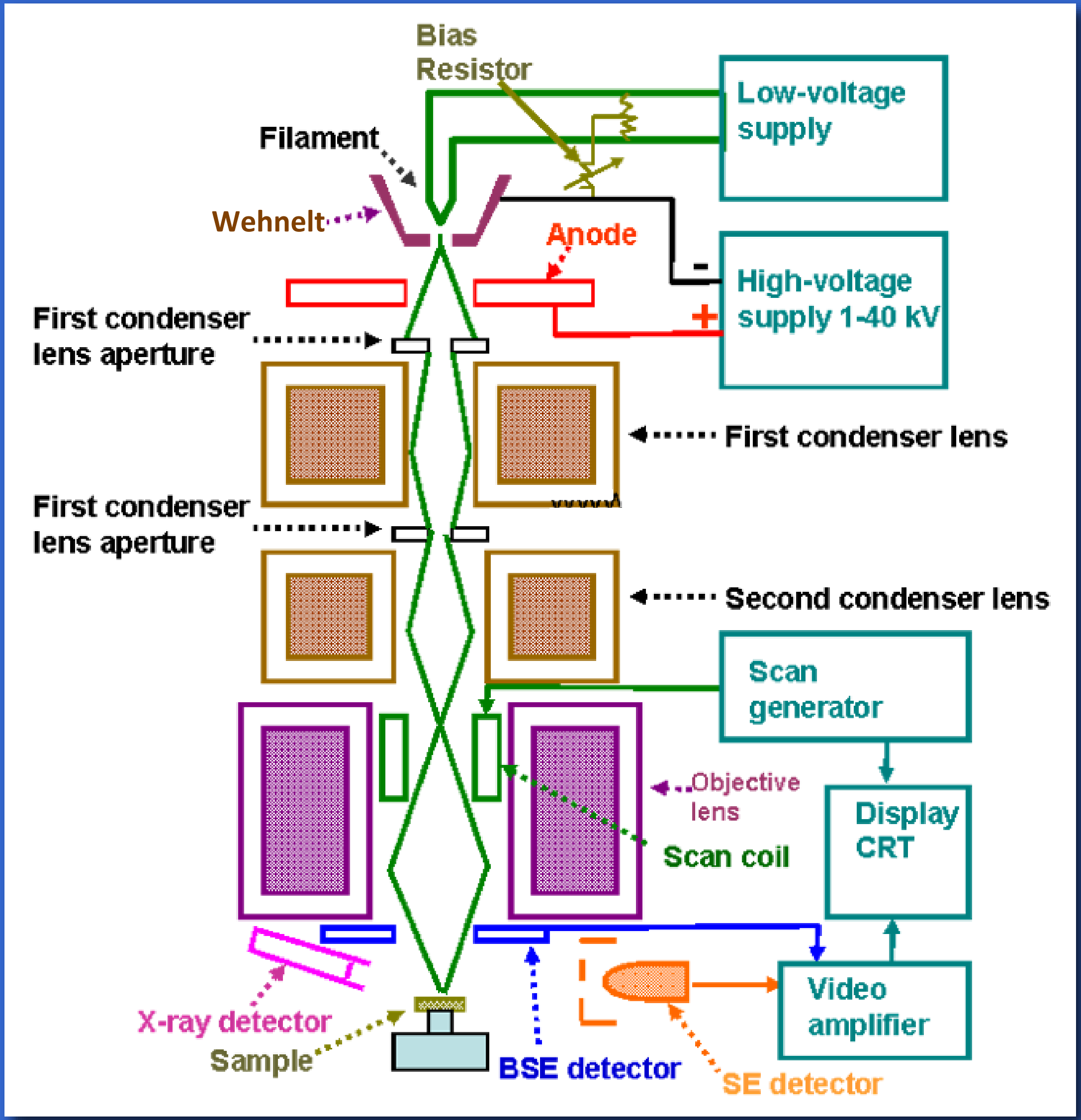
10 kV ——> 0.12 Å

100 kV ——> 0.037 Å

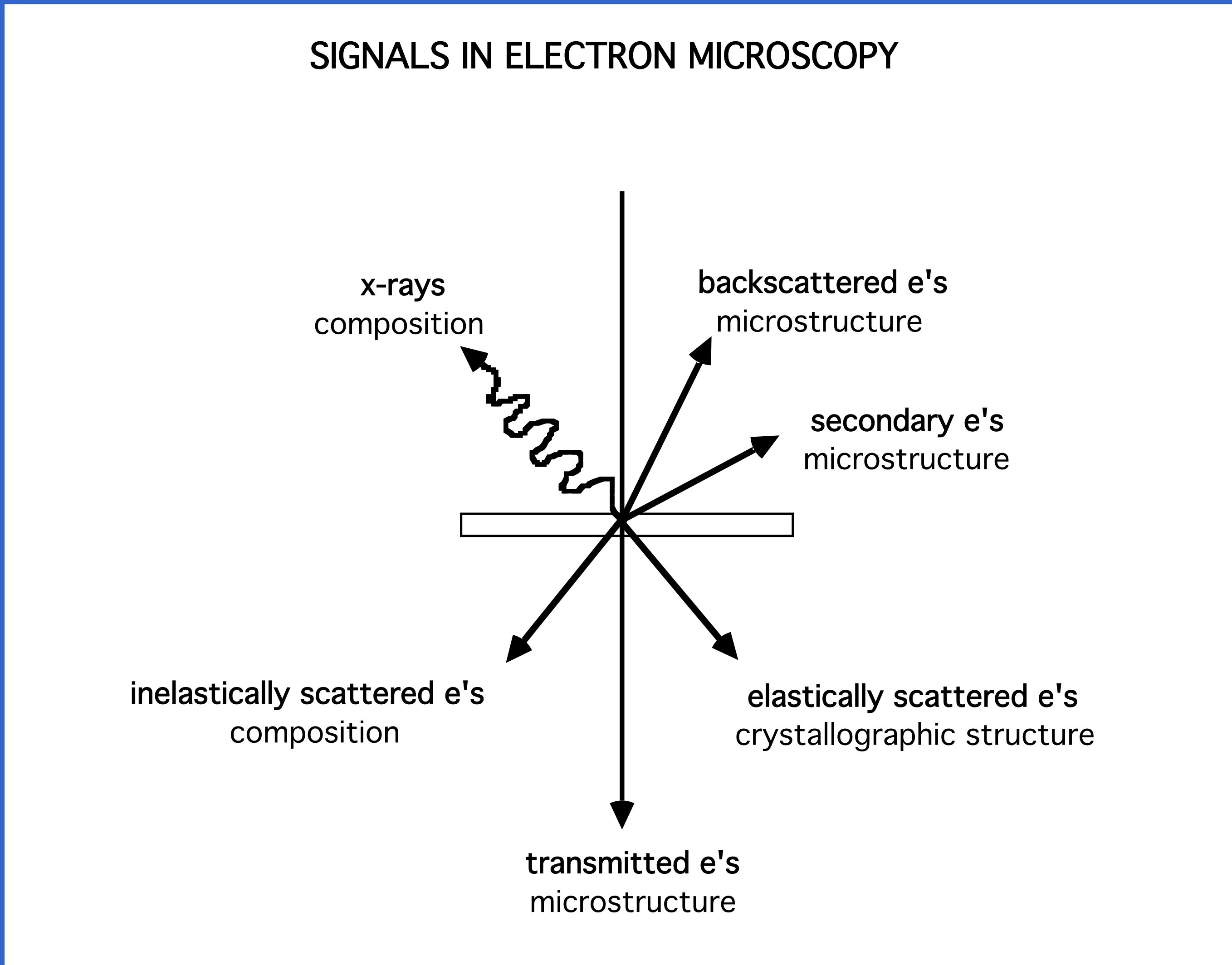
SEM - scanning electron microscopy



SEM - scanning electron microscopy

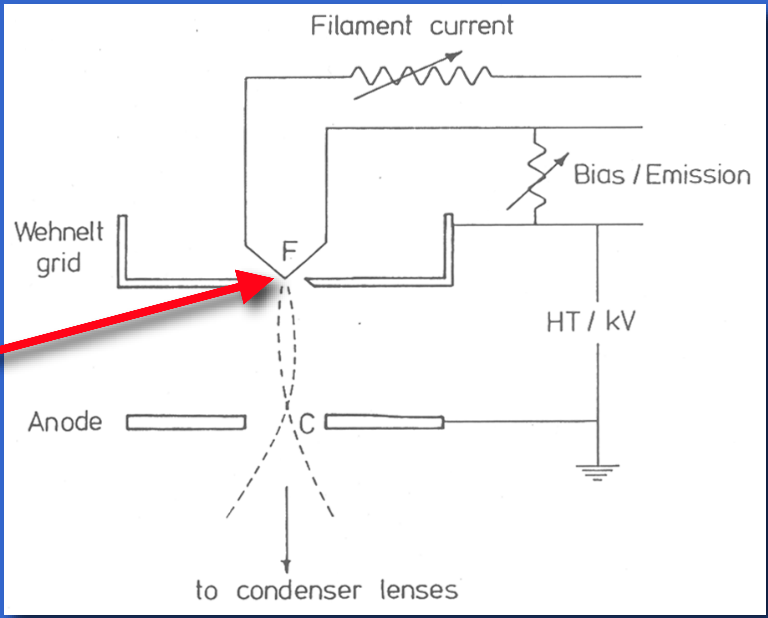


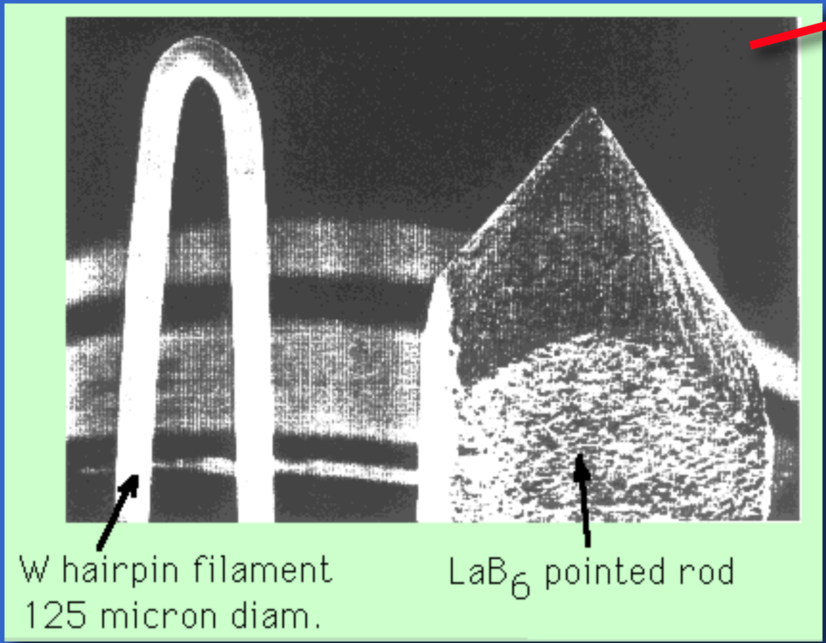
SEM - scanning electron microscopy



SEM - scanning electron microscopy

Electron gun





Electron emitter

SEM - scanning electron microscopy

$$\lambda = h/(2m_{electron}qV_o + q^2Vo^2/c^2))^{1/2}$$

Effects of increasing voltage in electron gun:

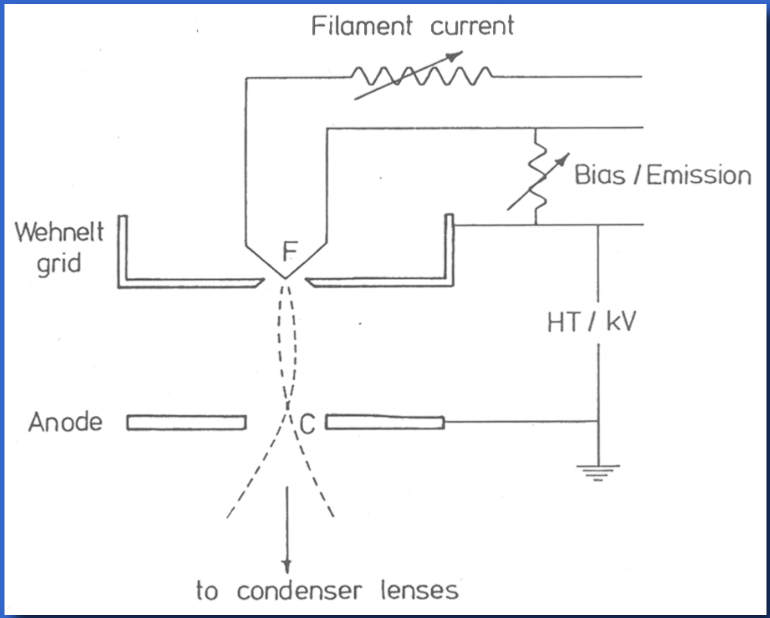
Resolution increased (λ decreased)

Penetration increases

Specimen charging increases (insulators)

Specimen damage increases

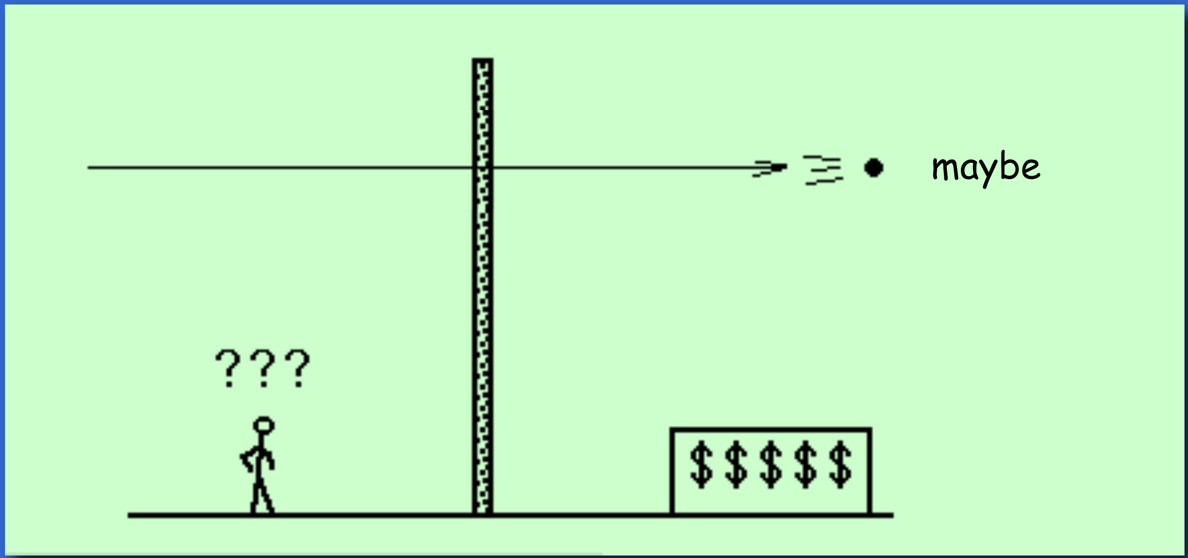
Image contrast decreases



SEM - scanning electron microscopy

Field emission electron source:

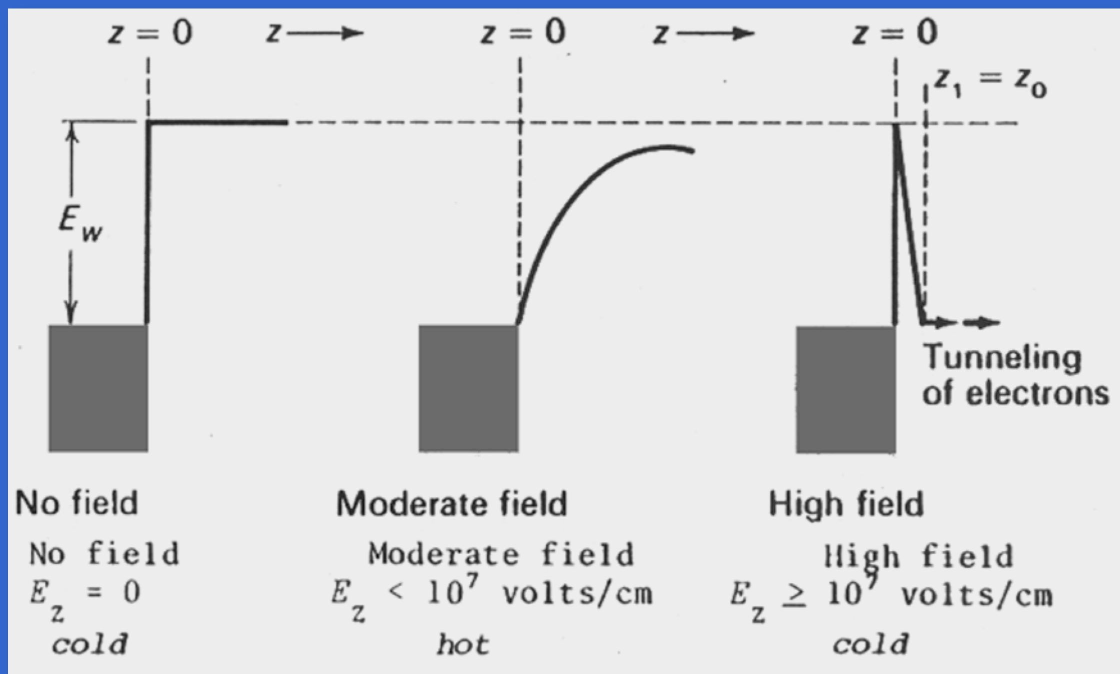
High electric field at very sharp tip causes electrons to "tunnel"



SEM - scanning electron microscopy

Field emission electron source:

High electric field at very sharp tip causes electrons to "tunnel"



SEM - scanning electron microscopy

Field emission electron source:

High electric field at very sharp tip causes electrons to "tunnel"

cool tip ——> smaller ΔE in beam
improved coherence

many electrons from small tip ——> finer
probe size, higher current densities (100X >)

problems - high vacuum, more $$$, fussy

SEM - scanning electron microscopy

Lenses

electrons focused by Lorentz force from electromagnetic field

$$\boldsymbol{F} = q\boldsymbol{v} \times \boldsymbol{B}$$

effectively same as optical lenses

Lenses are ring-shaped

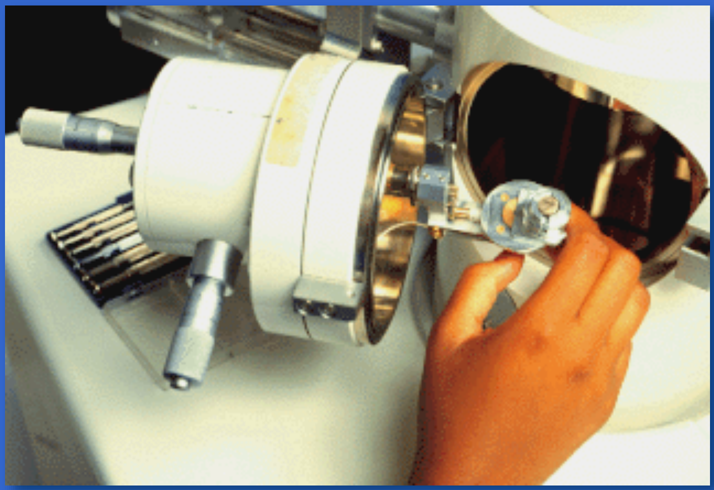
coils generate magnetic field
electrons pass thru hollow center

lens focal length is continuously variable

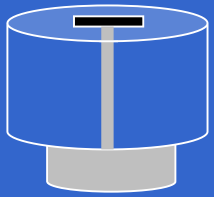
apertures control, limit beam

SEM - scanning electron microscopy

Specimen



Conducting -
- little or no preparation
- attach to mounting stub
  - for insertion into instrument
- may need to provide
  - conductive path with Ag paint



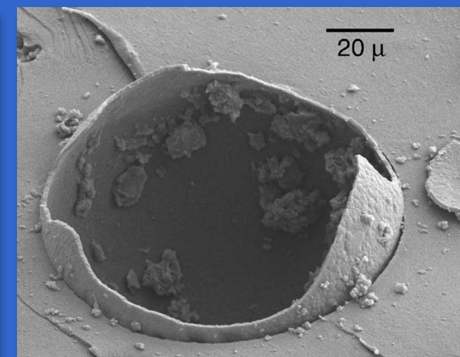
Non-conducting -
- usually coat with conductive very thin layer (Au, C, Cr)

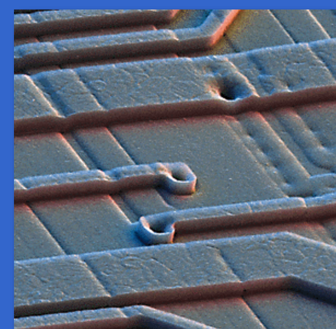
SEM - scanning electron microscopy

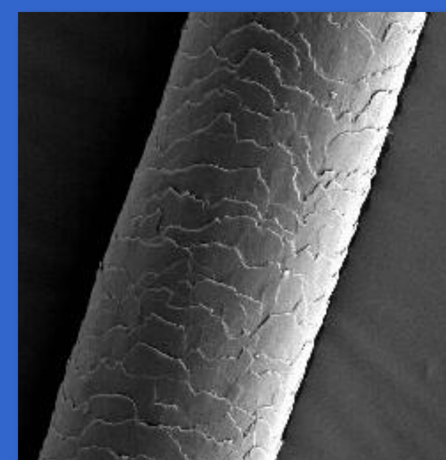
Specimen

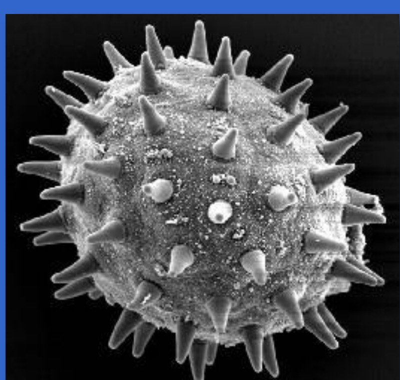
Can examine

- fracture surfaces
- electronic devices
- fibers
- coatings
- particles
- etc.







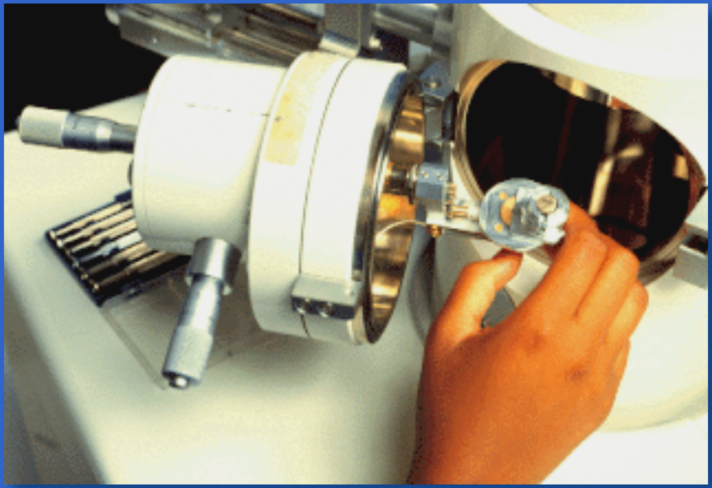


SEM - scanning electron microscopy

Specimen

Can be tilted, translated

Specimen size limited by
size of sample chamber



SEM - scanning electron microscopy

Specimen

What comes from specimen?

Backscattered electrons

Secondary electrons

Fluorescent X-rays

high energy
compositional contrast

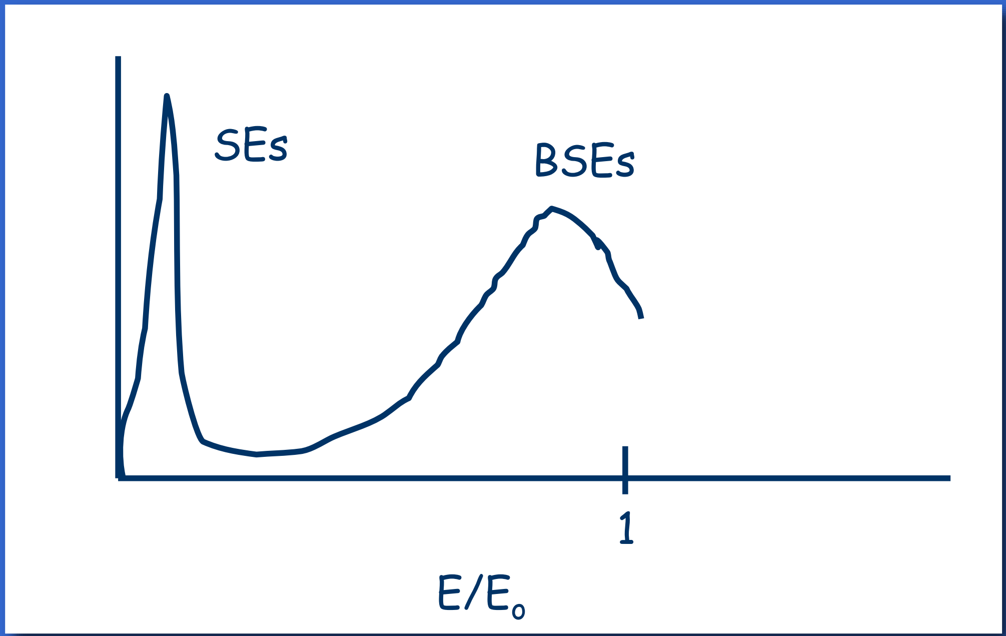
Brightness of regions in image increases as atomic number increases (less penetration gives more backscattered electrons)

low energy
topographic contrast

composition - EDS

SEM - scanning electron microscopy

Electron energy distribution



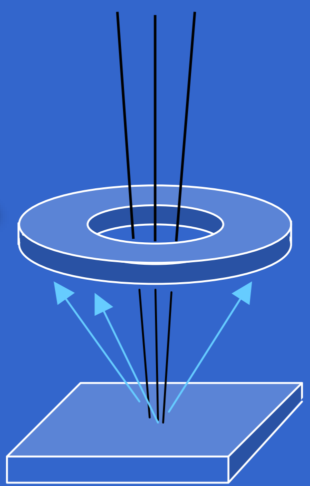
SEM - scanning electron microscopy

Backscattered electron detector - solid state detector

electron energy up to 30-50 keV

annular around incident beam

repel secondary electrons with
— biased mesh



images are more sensitive to chemical composition (electron yield depends on atomic number)

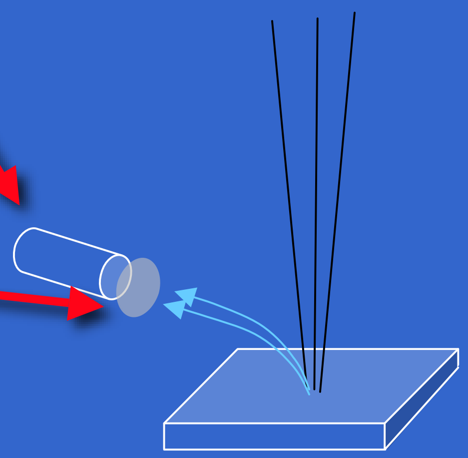
line of sight necessary

SEM - scanning electron microscopy

Secondary electron detector - scintillation detector

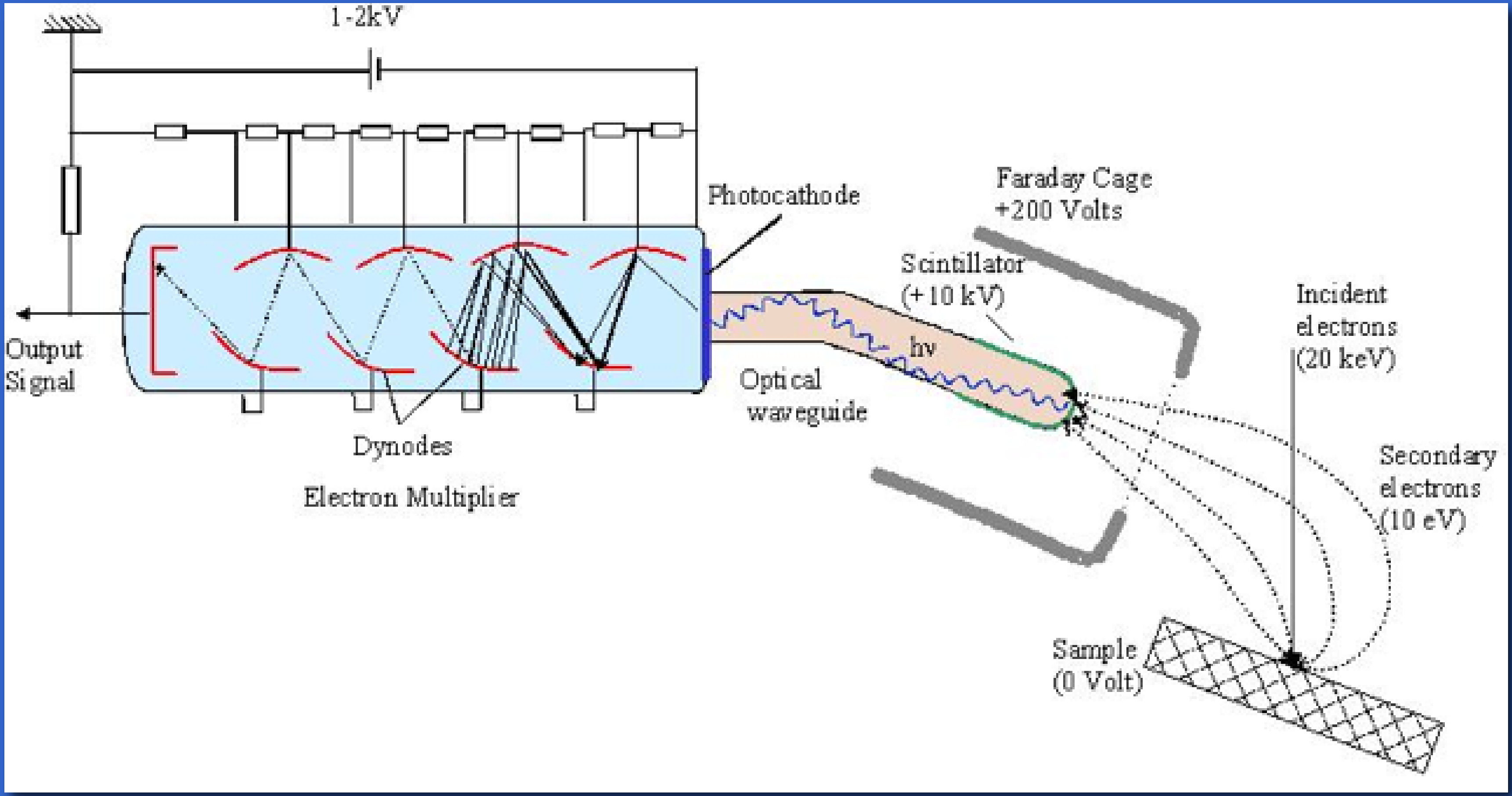
+ bias mesh needed in front of detector to attract low energy electrons

line of sight unnecessary



SEM - scanning electron microscopy

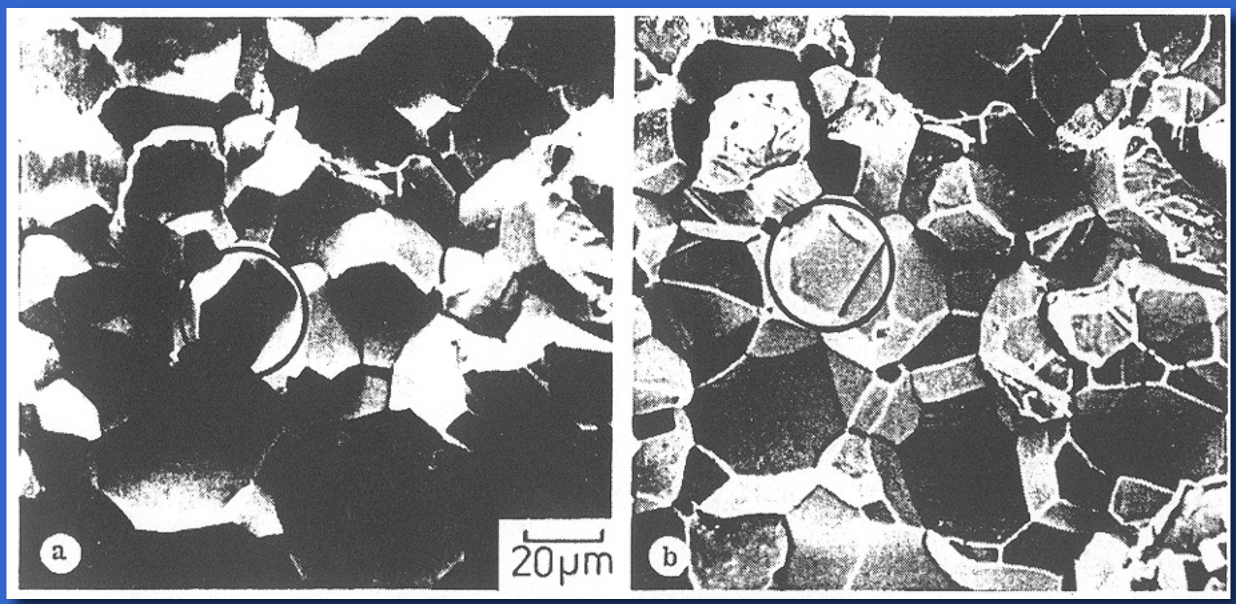
Secondary electron detector - scintillation detector



SEM - scanning electron microscopy

Choose correct detector- topography example

Fracture surface in iron



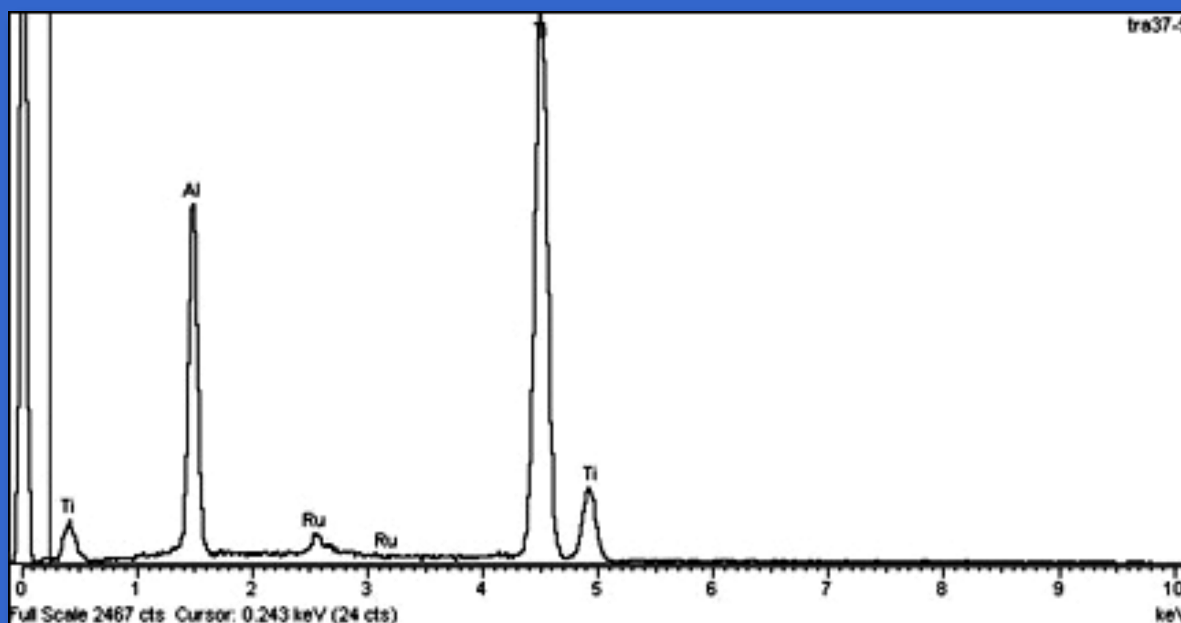
backscattered electrons   secondary electrons

SEM - scanning electron microscopy

Composition - what elements present at a particular spot in specimen?

Use solid state detector

Do energy scan for fluorescent X-rays



SEM - scanning electron microscopy

Composition mapping - x-ray fluorescence

Use solid state detector set for X-ray energy for a particular element in specimen

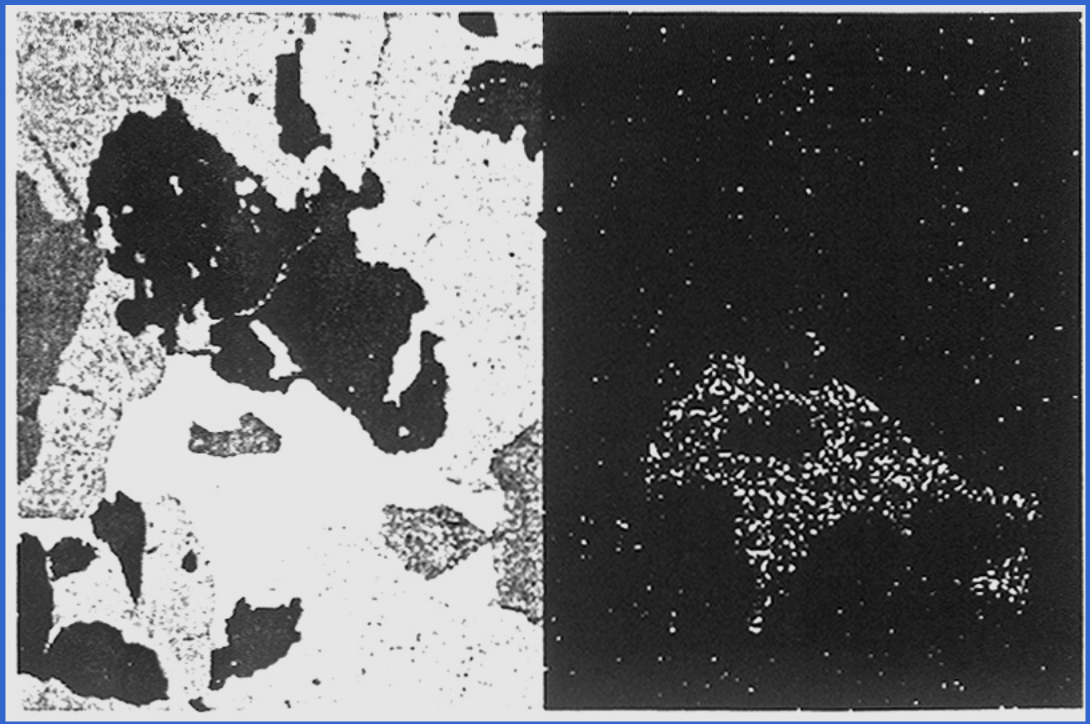


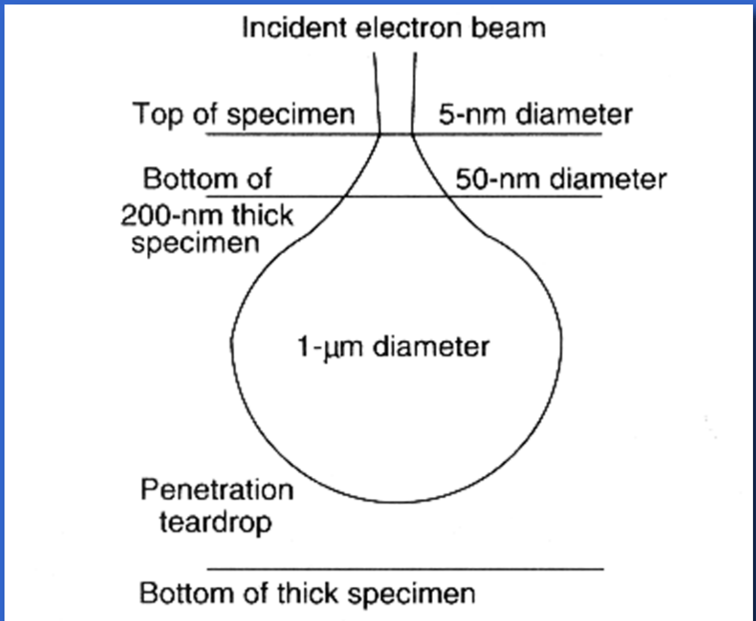
image X-ray map

SEM - scanning electron microscopy

Interaction volume

Backscattered electrons come from whole volume (high energy)

Secondary electrons come from neck only (low energy)



Electron penetration in specimen

SEM - scanning electron microscopy

Contrast

Comes from any kind of interaction with electron beam
- topography
- composition
  - elements
  - phases
- grain (crystal) orientation

charging affects contrast

SEM - scanning electron microscopy

Magnification

specimen

CRT

l_s = e beam scan distance on specimen

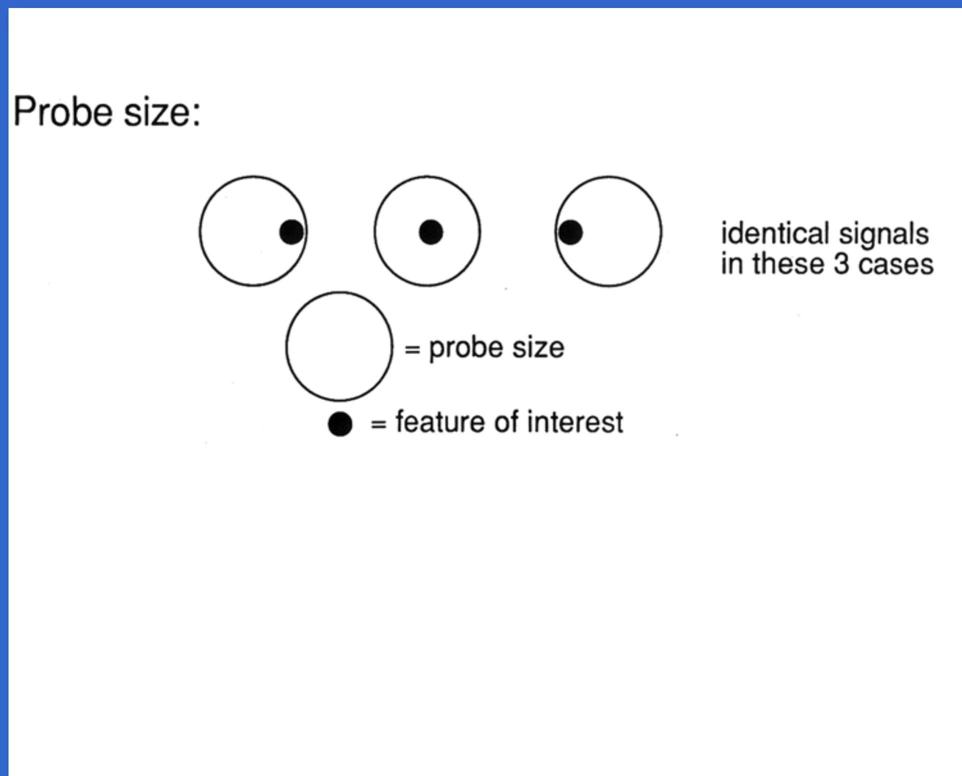
L_D = sweep distance on monitor

$$\text{Mag.} = \frac{L_D}{l_s}$$

SEM - scanning electron microscopy

Resolution

Determined by
probe size

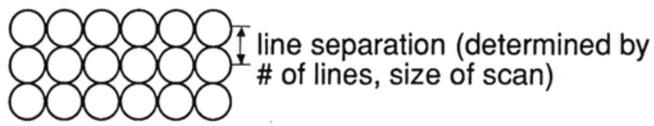


SEM - scanning electron microscopy

Resolution

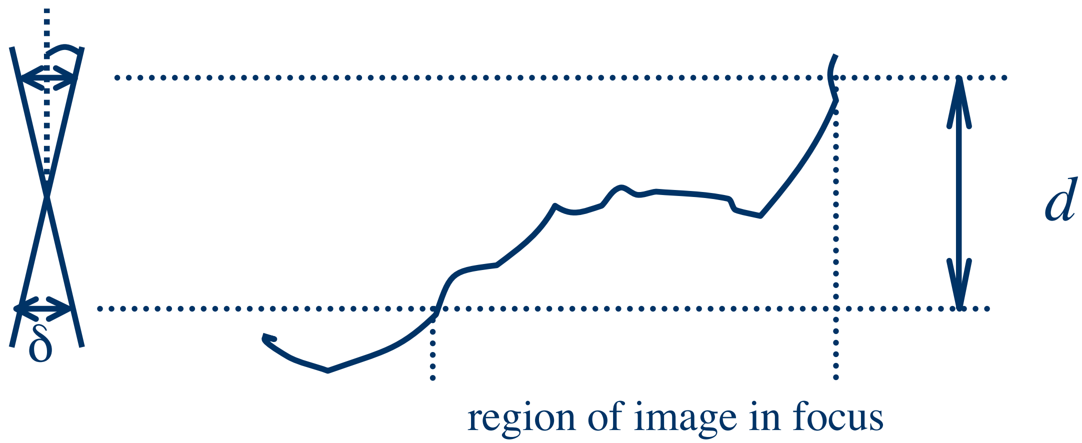
Determined by
probe size

Optimum probe size:



SEM - scanning electron microscopy

Depth of field



d = depth of field
δ = required spatial resolution
α = convergence angle

$$\tan\alpha = \frac{0.5\delta}{0.5d} = \frac{\delta}{d}$$

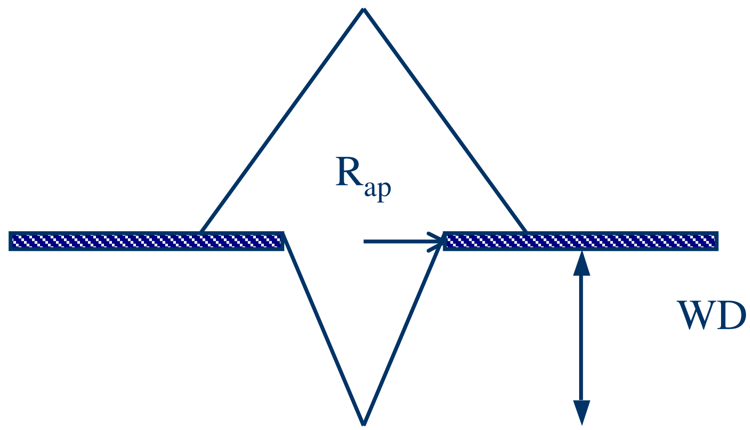
For small angles, $\tan\alpha = \alpha$

$$d = \frac{\delta}{\alpha}$$

Can control depth of field (d) with convergence angle (α)

SEM - scanning electron microscopy

Depth of field



$$\tan\alpha = \frac{R_{ap}}{WD}$$