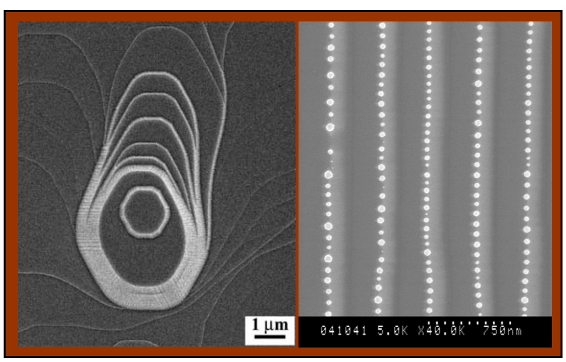
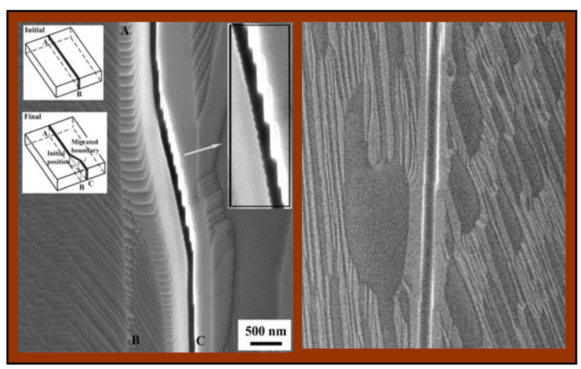
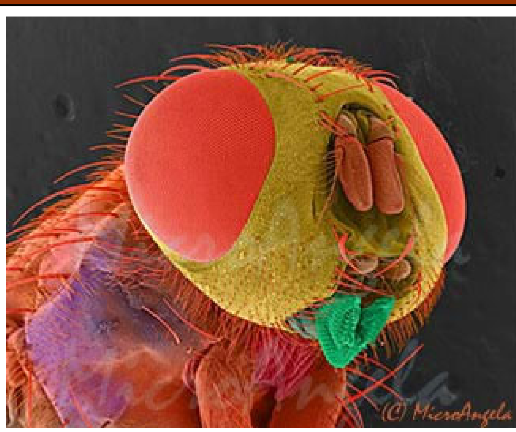
MR202

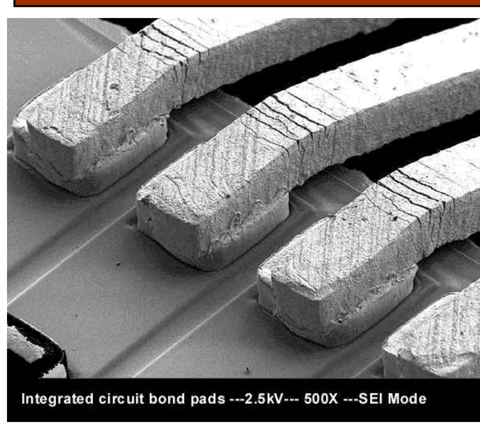
Electron Microscopy in Materials Characterization

General Introduction, Resolution, Limits on resolution, Lens aberrations
Introduction to SPM/SEM, Electron Optics – Electron Guns and Lenses, Probe diameter and probe current
Electron-Specimen Interactions, Interaction volume, elastic and inelastic scattering
Basics of SEM imaging, Imaging modes, Detectors, Image contrast, Image processing
XEDS and WDS – Principles and practice, Basics
Case studies in Materials Science – Imaging and Analysis
Newer Techniques – EBSD, LVSEM, ESEM
Sample Preparation and a special note about digital imaging/processing









Microscope

Used to 'see' objects not visible to the human eye

Eye can 'resolve' objects ~ 0.1mm apart

For anything closer, we need a means of magnifying

Note : ***BIG*** difference between 'seeing' and 'resolving'

Seeing a car approaching (from its headlights)
Resolving the two headlights as separate sources of light

Optical System - Components

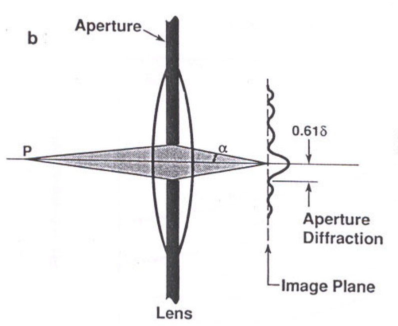
Source of Radiation - Visible-light

System of lenses and apertures

Any system used to form an image uses lenses and apertures that have a certain dimension

Limit to resolution arises from the phenomenon of ***diffraction***

Point object is not mapped on to a point - spread out



Diffraction from a single slit
Intensity $\sim (\sin(x)/x)^2$
A big maxima surrounded by smaller maxima

Rayleigh Criterion

Profiles from two adjacent point will overlap

To be able to resolve two points as distinct

$$\theta_R = \sin^{-1}(1.22\lambda/d)$$

This is the ***diffraction-limited*** resolution limit

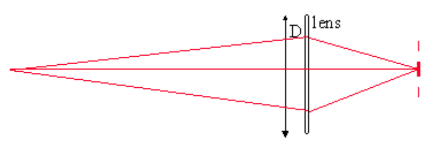
To increase resolution

Large d

Small λ

Rayleigh Criterion: a picture

The lens will focus the light to a fuzzy DOT rather than a true point.



Rayleigh Criterion: an example

- Consider the (ideal) resolving ability of the eye
- Estimate D, the diameter of the pupil
- Use λ = 550 nm (middle of visible spectrum)
- Now calculate the minimum angle the eye can resolve.
- Now calculate how far apart two points of light can be if they are 5 meters away.

Rayleigh Criterion: an example

- with D = 5 mm and λ = 550 nm,

  $\theta_{limit} = \sin^{-1}(1.22 \times 5.5 \times 10^{-7}\,\text{m}/.005\,\text{m})$

  $= 7.7 \times 10^{-3}$ degrees

  = .46 arc minutes

  so $x/L = \tan(\theta_{limit})$, and

  $x = 5\text{m} * \tan(7.7 \times 10^{-3}$ degrees$) =$ **.67 mm**

Limits on Resolution

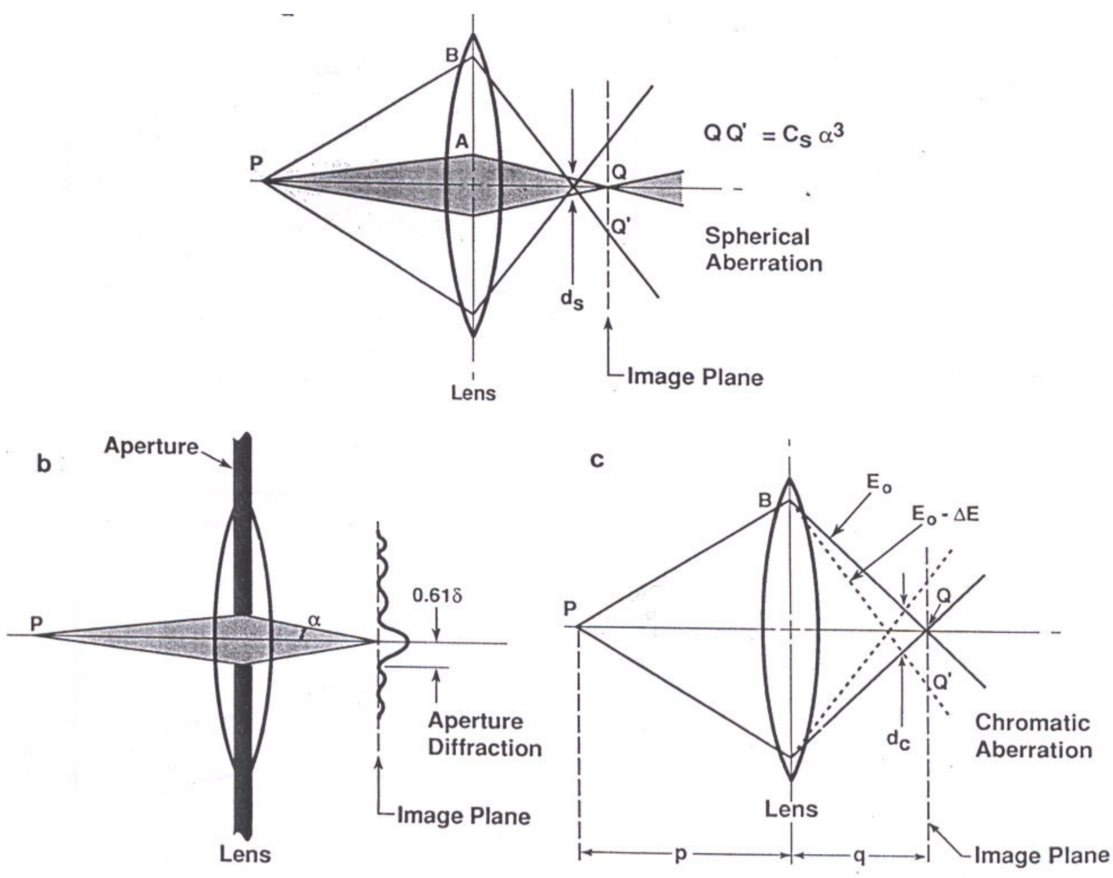
What diameter of telescope would you require to read the numbers on a license plate from a spy satellite?

Limits on Resolution: Telescope

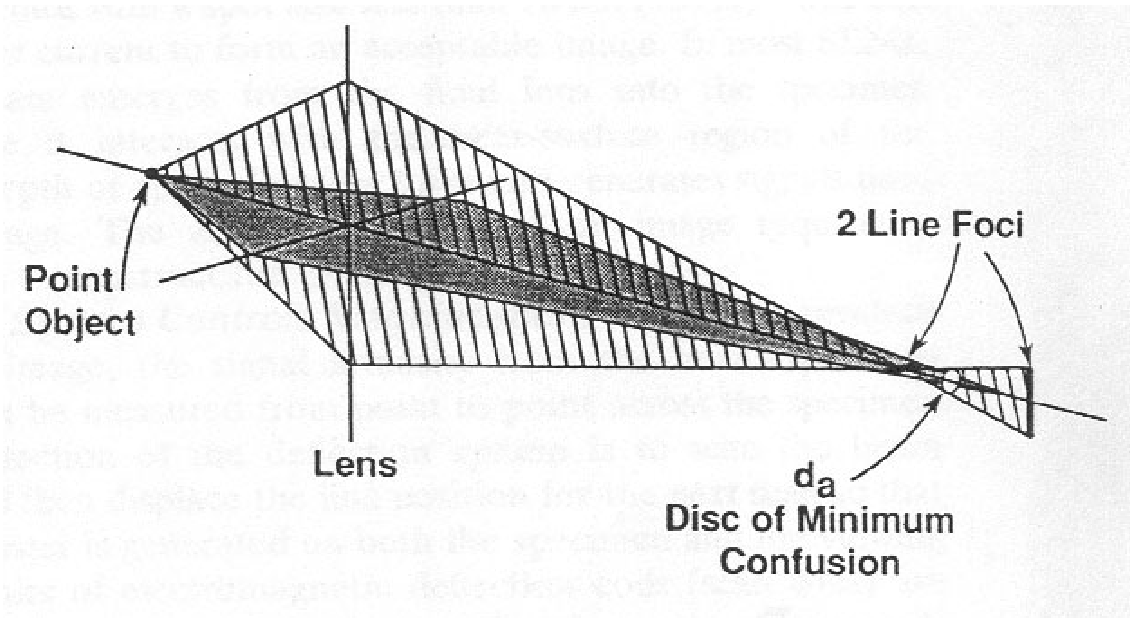
- need to resolve an "x" size of about 1 cm
- "s" is on order of 100 miles or 150 km
- θ_{limit} then must be (in radians)
  $= 1 \text{ cm} / 150 \text{ km} = 7 \times 10^{-8}$
- $\theta_{limit} = 1.22 \times 5.5 \times 10^{-7} \text{ m} / D \; = \; 7 \times 10^{-8}$

so $D = 10$ m (Hubble has a 2.4 m diameter)

Lens Aberrations - Other limiting factors for resolution



Astigmatism



Electrons as Waves - The particle-wave duality

Electron moving with a velocity 'v' has a wavelength associated with it

$$\lambda = h/mv \sim 12.247/E(kV)$$

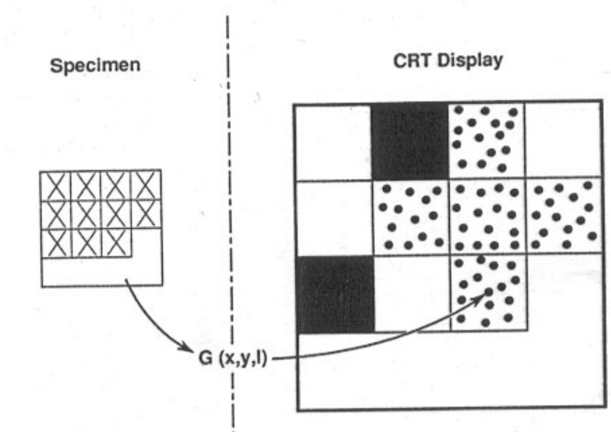
<u>Typical wavelengths</u>

E	λ
100 kV	0.037 A
200 kV	0.025 A

Basics of SEM Operation

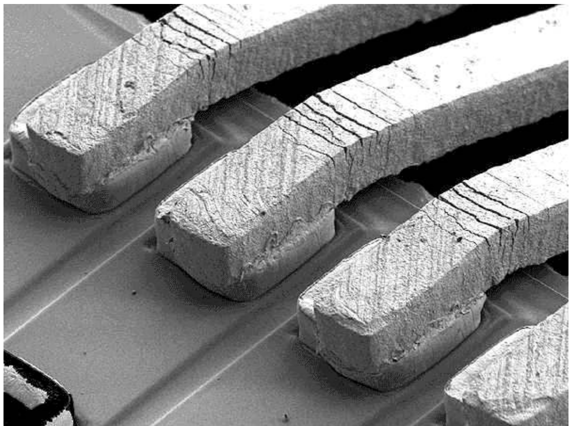
- Electron gun produces a 'beam'
  - *Thermionic/Field-emission guns*
- Produce a 'tight' spot on the specimen surface
  - *Condenser and Objective lenses*
- Scanning coils 'raster' the beam across the specimen
  - *Size of scan -> Magnification*
- Electron-specimen interactions
  - *Produces a wide variety of signals*
- Detectors to collect the signal
  - *Different detectors for different signals*

Magnification

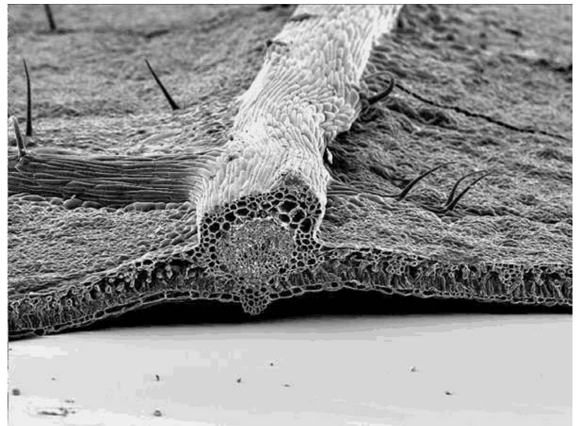


Magnification = D/d

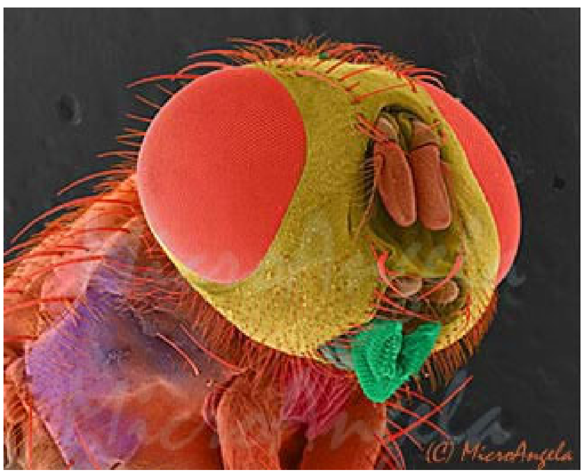
Magnification	Area on Sample	Pixel Size
10x	1cmx1cm	10 μm
100x	1mmx1mm	1 μm
10kx	10μmx10μm	10 nm
100kx	1μmx1μm	1 nm

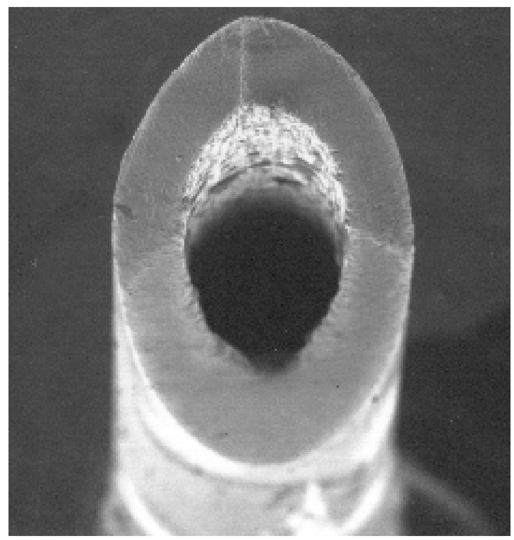


Integrated circuit bond pads ---2.5kV--- 500X ---SEI Mode

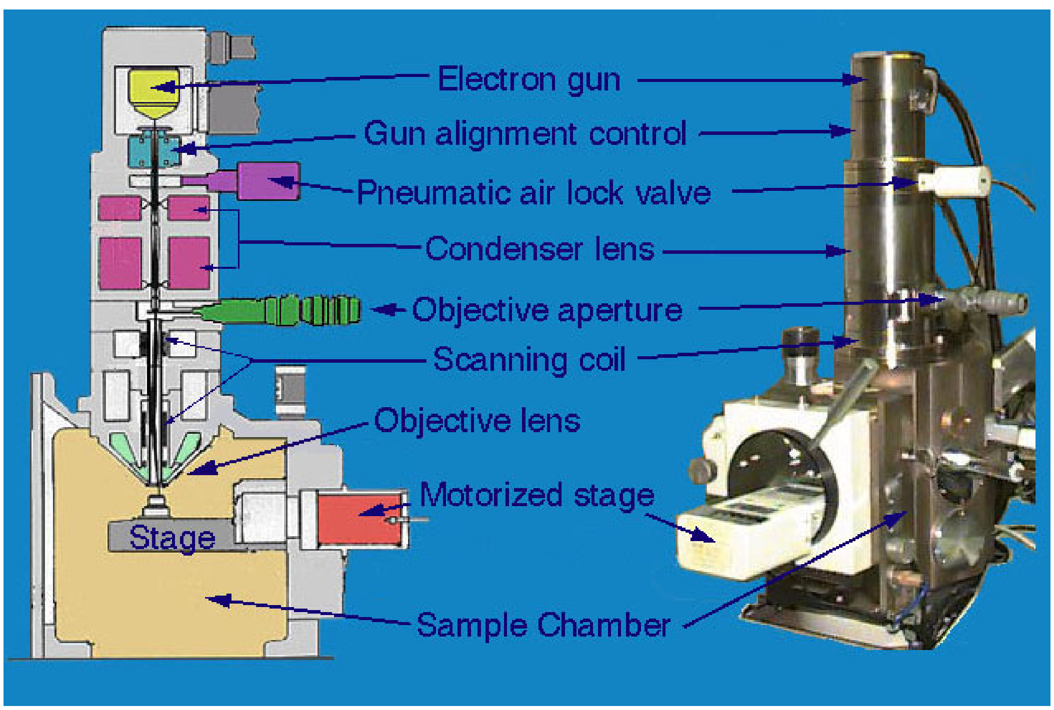


Leaf Cross Section ---10KV--- 100X--- SEI Mode

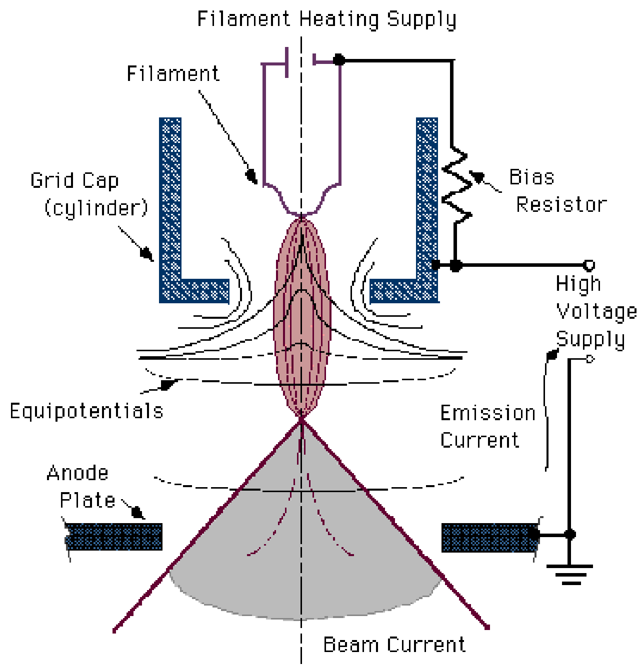




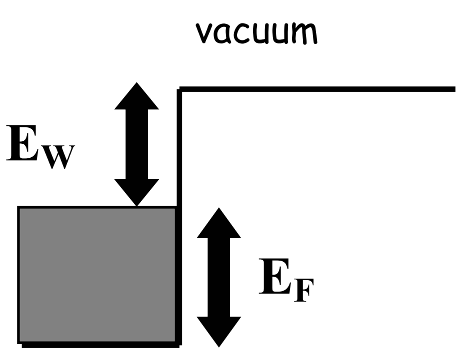
Some Examples of SEM Images


Electron gun
Gun alignment control
Pneumatic air lock valve
Condenser lens
Objective aperture
Scanning coil
Objective lens
Motorized stage
Stage
Sample Chamber

Electron Gun



Thermionic Emission



$$J_c = A_c T^2 \exp(-E_w/kT)$$

Richardson Equation

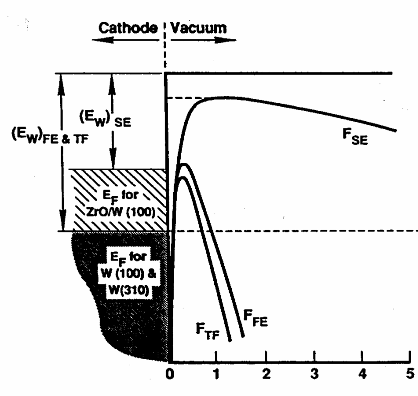
E_w = 4.5 eV for W, at
T = 2700 K
J = 3.4 A/cm^2

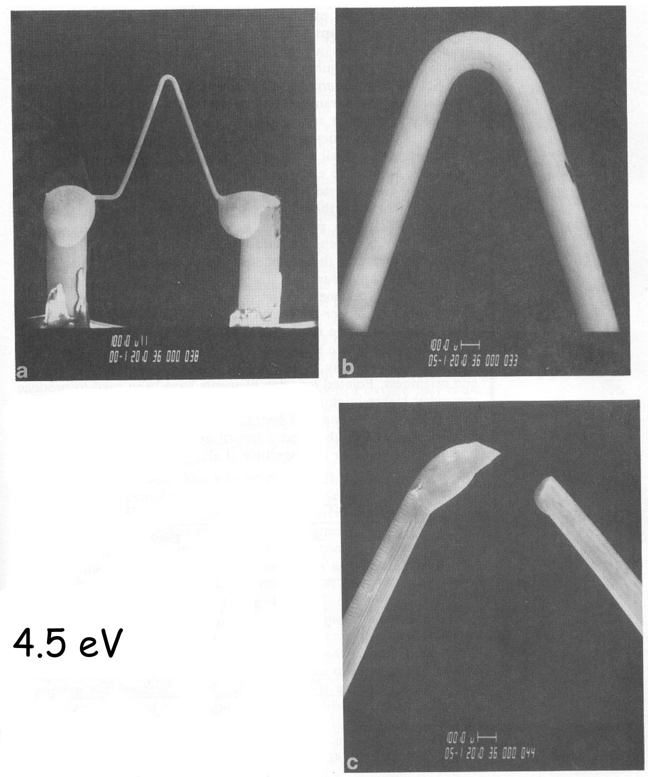
Use 'thermal' energy to excite electrons from a metal

Field Emission

Tip with small radius of curvature

Very high electric fields

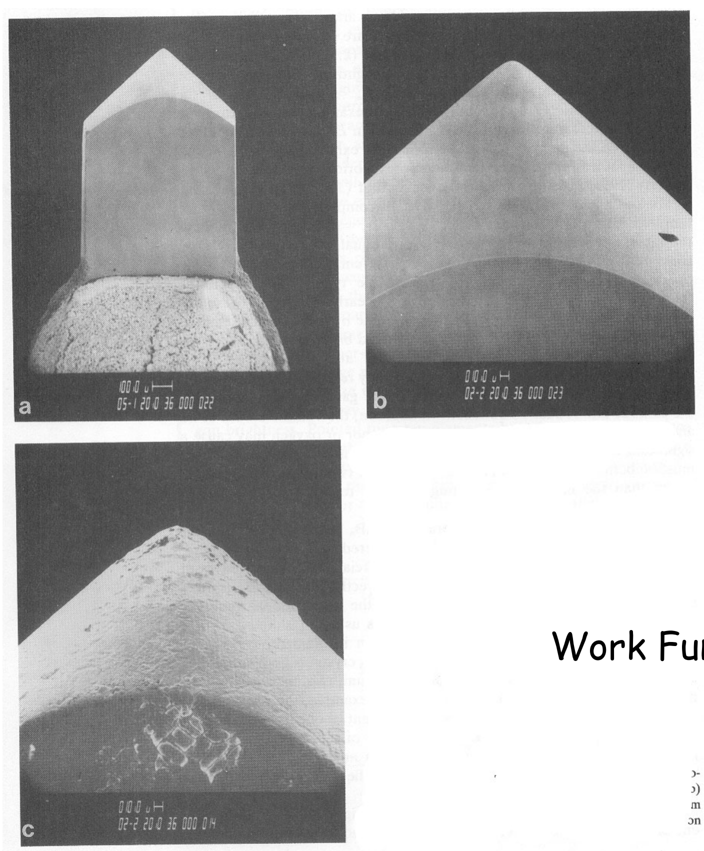




Tungsten
Hairpin
Filament

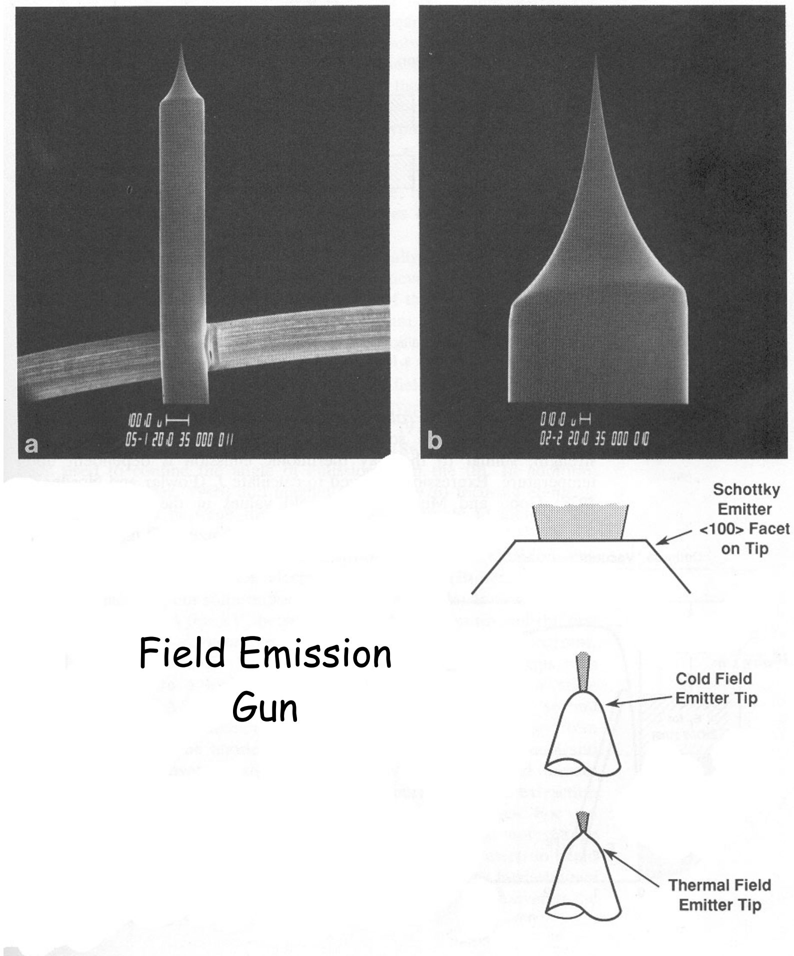
Work Function = 4.5 eV

7



LaB_6
Filament

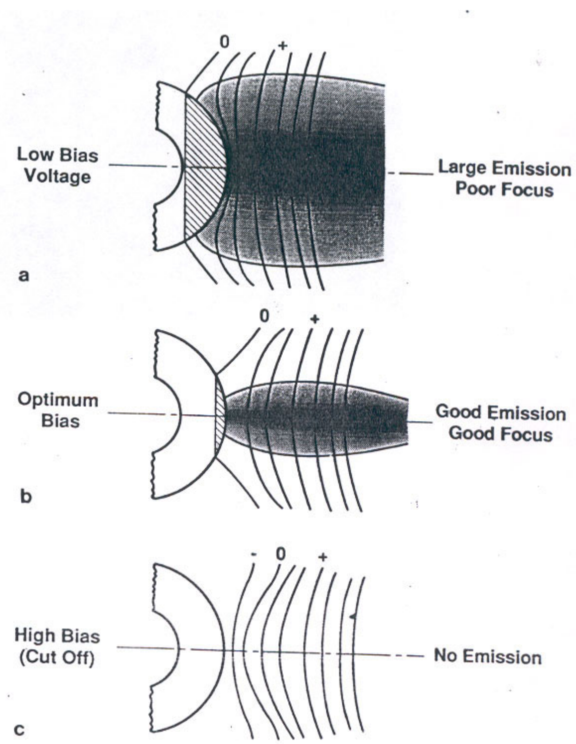
Work Function = 2.5 eV



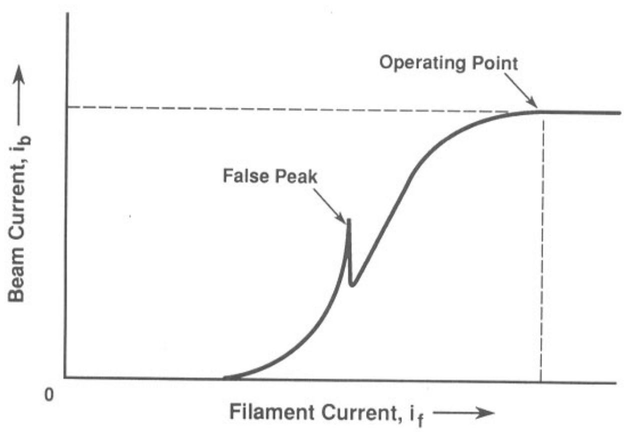
Field Emission
Gun

Comparison of Electron Sources
(Brightness = Current/area/solid angle)

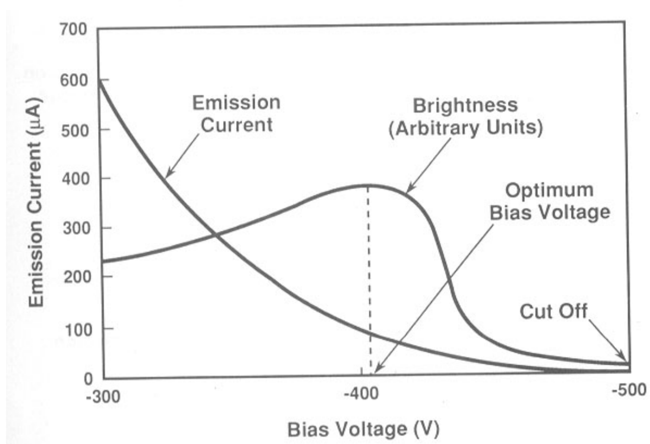
Source	Brightness (A/cm^2sr)	Source Size	Lifetime (h)	Vacuum Level
Tungsten filament	10^5	30-100 um	40-100	10^{-5}Torr
LaB_6 <100> (2.5 ev)	10^5	5-50 μm	200-1000	10^{-7}Torr
Cold FE	10^8	< 5nm	> 1000	10^{-10}Torr
Thermal FE	10^8	< 5 nm	> 1000	10^{-9}Torr
Schottky	10^8	15-30 nm	> 1000	

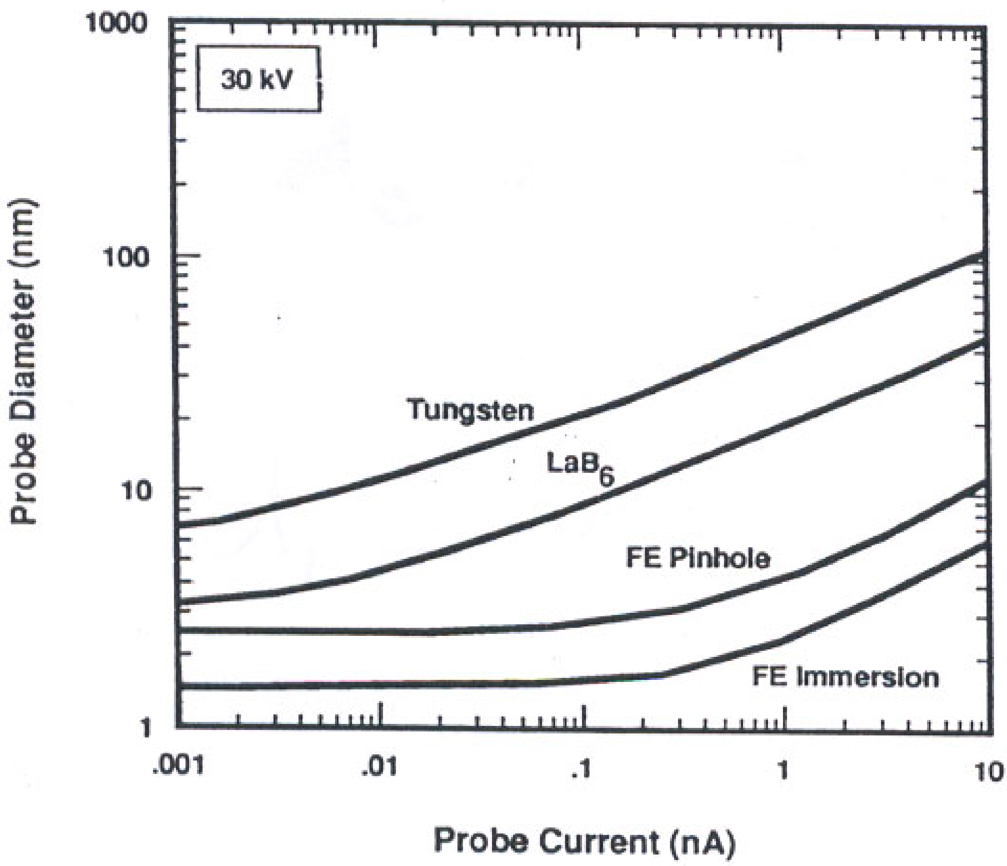


Effect of Bias on the filament emission



This is what happens when you turn the filament knob

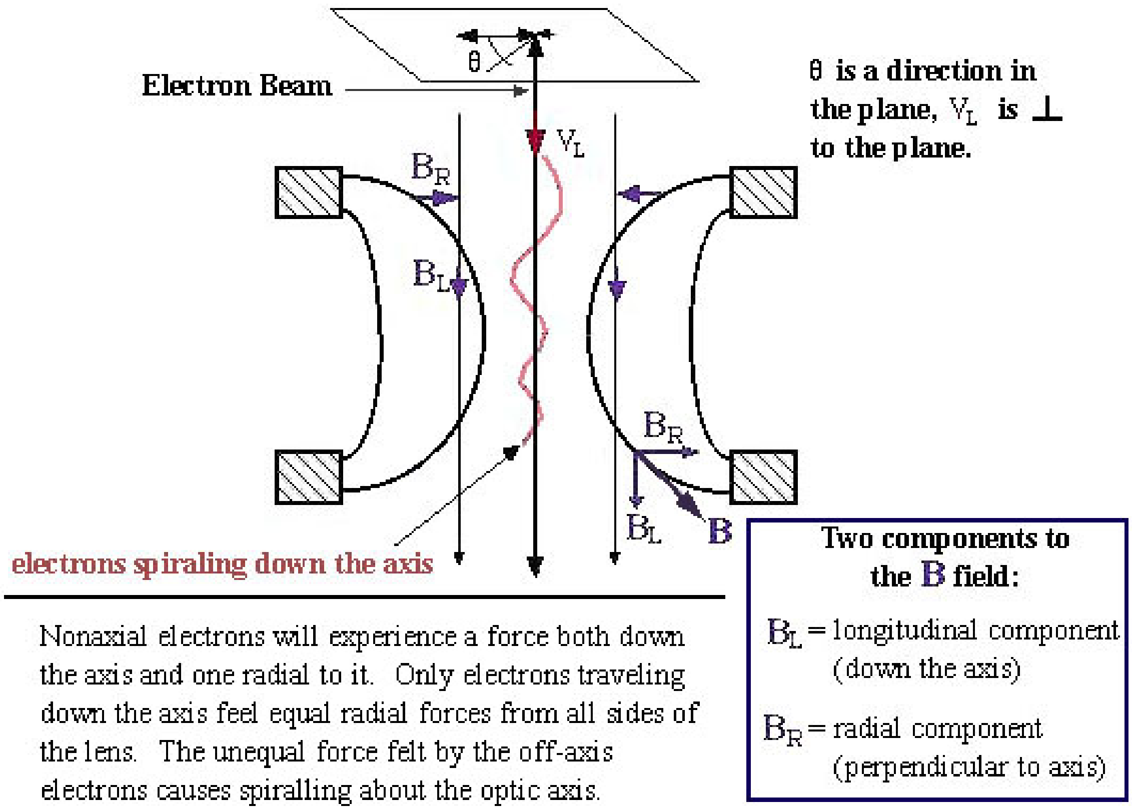




Comparison of Electron Sources

Note that a very small FE probe carries a much larger current compared to a W filament.

Lenses in the Electron Microscope



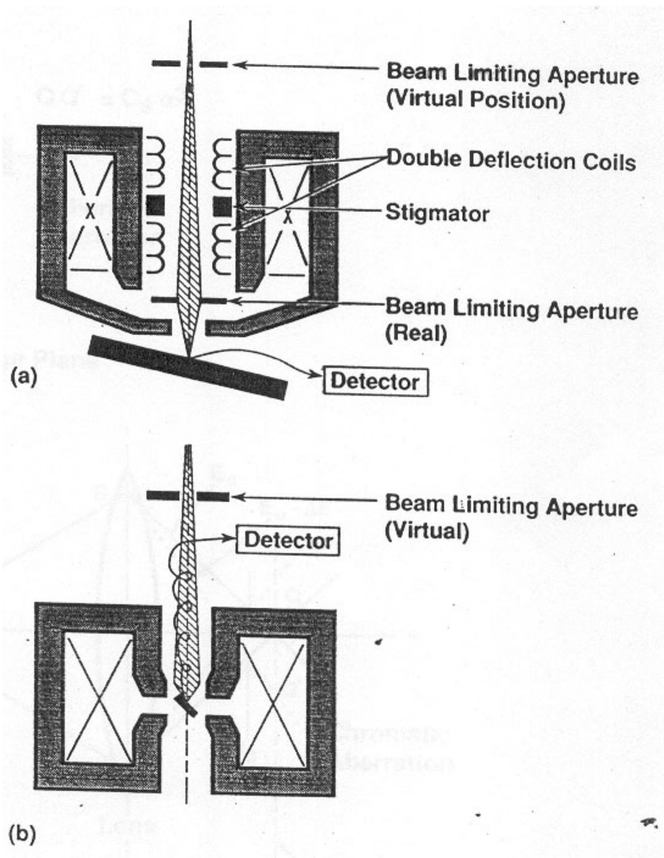
The 'Wehnelt ' is the first lens in the SEM

Two sets of lenses in the SEM

Larger current through lens => Smaller Focal Length

Condenser lenses – condense the beam to give smaller beam size

Objective lens – final 'probe-forming' lens in the SEM
Larger currents pass through this lens (compared to the condenser lens) – needs to be cooled.



Large working distance

Two types of Objective Lenses

Very small working distance

(a) Pin-hole lens – variable working distance, no size limitation of sample, good depth of field

(b) Small focal length => High Resolution

Three important parameters for image formation

Probe Size -

Smaller the probe size, higher the resolution
Smaller the probe size, lower the brightness

Probe Current/Brightness -

Imaging requires 'large' currents

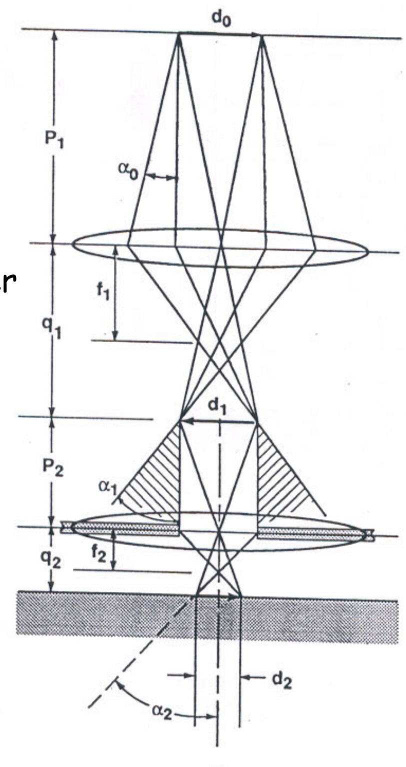
Convergence Angle -

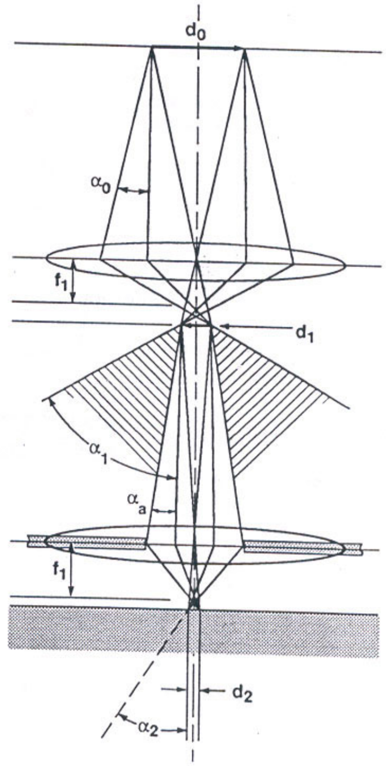
Depth of field increases as angle decreases
Aberrations decrease as angle decreases
Brightness decreases as angle decreases

These three parameters are NOT independent of each other

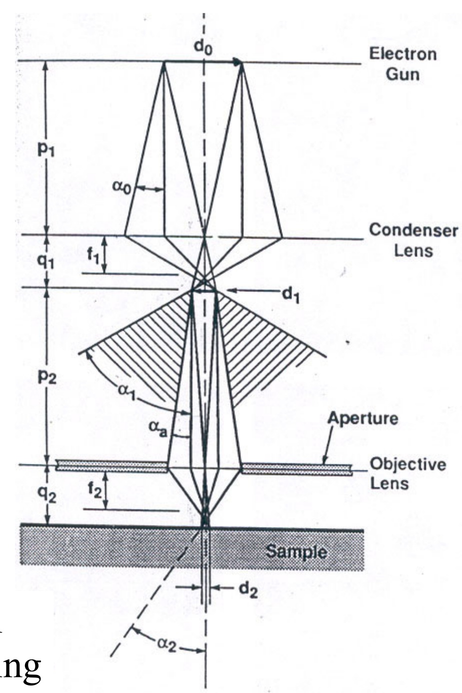
Probe Size d_p – diameter of beam as it falls on the specimen
Brightness β – Current density/solid angle
Probe convergence α_p – angle made by the cone of electrons with the central axis at the sample surface

Weak condenser lens

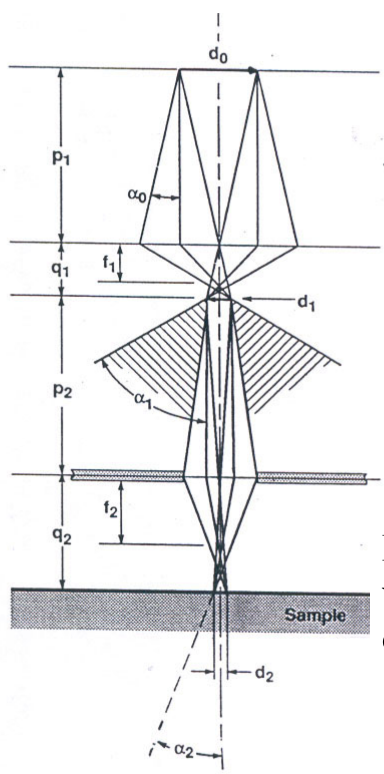




Strong condenser lens



Small working distance



Large working distance