Topic 3b,c
Electron Microscopy

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1.0 Introduction and History

What are they and Where did Electron Microscopes Come From?

- **Electron Microscopes are scientific instruments that use a beam of highly energetic electrons to examine objects on a very fine scale.**

- **Electron Microscopes were developed due to the limitations of Light Microscopes which are limited by the physics of light to 500x or 1000x magnification and a resolution of 0.2 mm.**

1.0 Introduction and History

What are they and Where did Electron Microscopes Come From?

- In the early 1930's this theoretical limit had been reached and there was a scientific desire to see the fine details of the interior structures of organic cells (nucleus, mitochondria...etc.).

- This required 10,000x plus magnification which was just not possible using current optical microscopes.

Dates

- The Transmission Electron Microscope (TEM) was the first type of Electron Microscope to be developed and is patterned exactly on the Light Transmission Microscope except that a focused beam of electrons is used instead of light to "see through" the specimen. It was developed by Max Knoll and Ernst Ruska in Germany in 1931.
- The first Scanning Electron Microscope (SEM) debuted in 1938 (Von Ardenne) with the first commercial instruments around 1965. Its late development was due to the electronics involved in "scanning" the beam of electrons across the sample.

Chronology

1897: J.J. Thompson **- Discovers the electron**
1924: Louis deBroglie
Identifies a wavelength to moving electrons - $\lambda = h / mv$
where: λ **= wavelength,** h **= Planck's constant** m **= mass,**
v **= velocity (For an electron at 60kV,** λ **= 0.005 nm)**
1926: H. Busch **- Magnetic or electric fields act as lenses for electrons**
1929: E. Ruska **- Ph.D thesis on magnetic lenses**
1931: Knoll & Ruska **- First electron microscope built**

1931: Davisson & Calbrick **- Properties of electrostatic lenses**

1934: Driest & Muller **- Surpass resolution of the OM**

1938: von Borries & Ruska
First practical EM (Siemens) - 10 nm resolution

1940: RCA **- Commercial EM with 2.4 nm resolution**

Microscopy and Related Techniques

- Light (optical) microscopy (LM) or (OM)
- Scanning electron microscopy (SEM)
  Energy dispersive X-ray spectroscopy (EDS) & Wavelength dispersive X-ray spectroscopy (WDS)
- X-ray diffraction (XRD)/X-ray fluorescence (XRF)
- Transmission electron microscopy/ Scanning transmission electron microscopy (TEM)/(STEM)

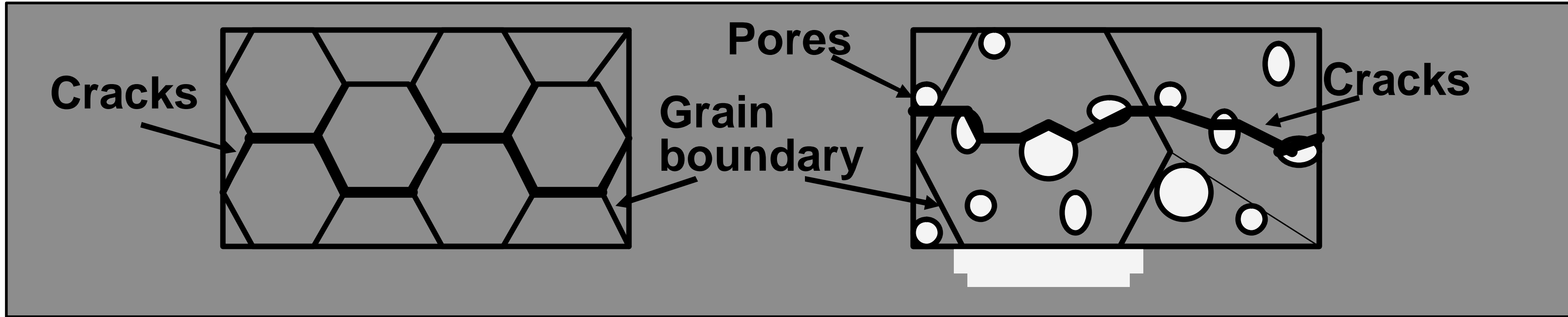
Surface Characterization Techniques

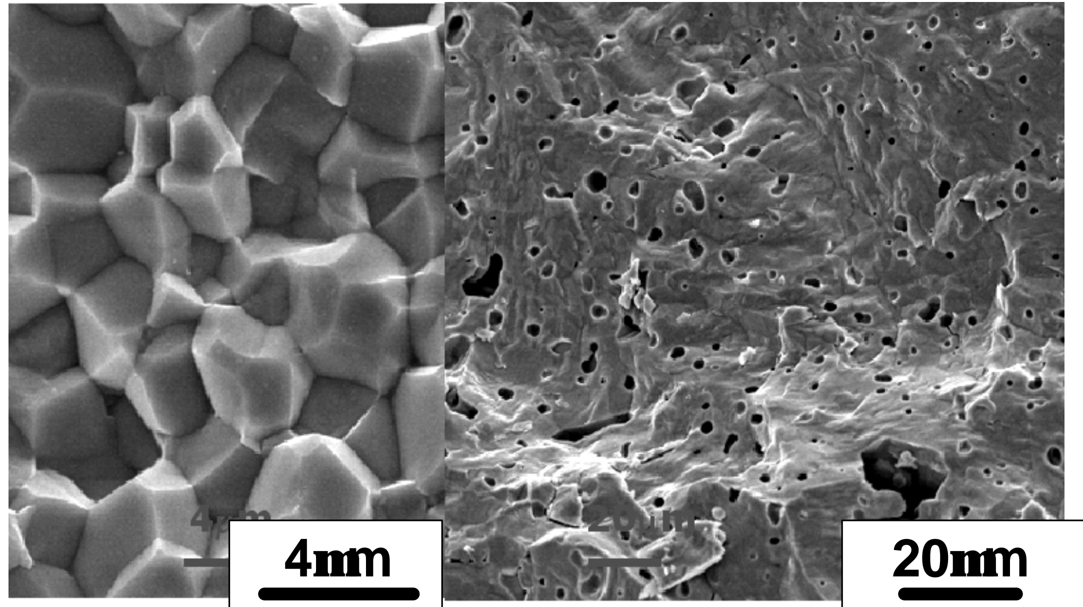
- Scanning probe microscopy (AFM & STM)

1.1 Characteristic Information

o **Topography**
The surface features of an object or "how it looks", its texture; direct relation between these features and materials properties
o **Morphology**
The shape and size of the particles making up the object; direct relation between these structures and materials properties
o **Composition**
The elements and compounds that the object is composed of and the relative amounts of them; direct relationship between composition and materials properties
o **Crystallographic Information**
How the atoms are arranged in the object; direct relation between these arrangements and materials properties

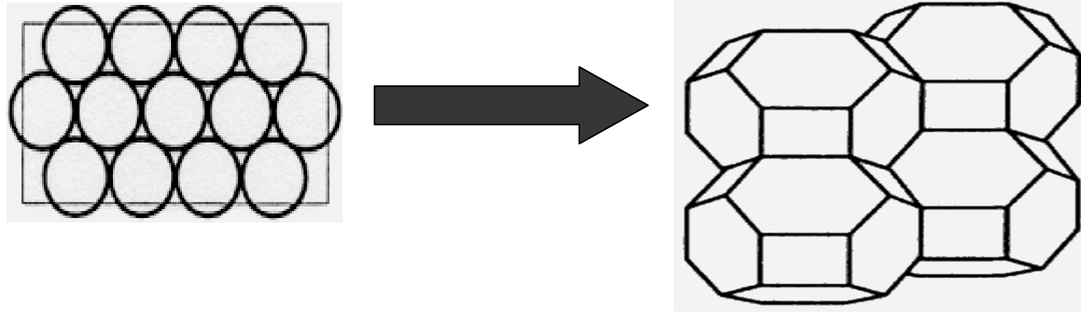
Identification of Fracture Mode

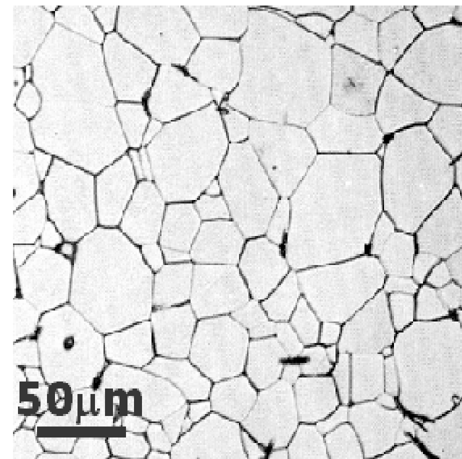




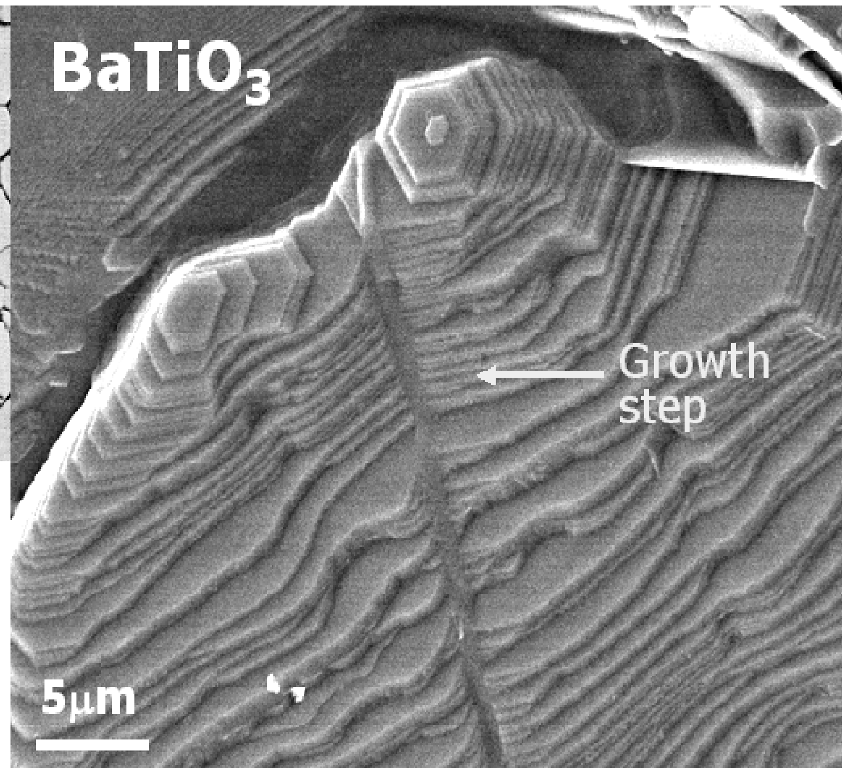
SEM micrographs of fractured surface of two $BaTiO_3$ samples.

OM and SEM





OM - 2D



SEM – 3D

How Fine can You See?

- Can you see a sugar cube? The thickness of a sewing needle? The thickness of a piece of paper? …
- The resolution of human eyes is of the order of 0.1 mm, 100mm » 4 mils.
- However, something vital to human beings are of sizes smaller than 0.1mm, e.g. our cells, bacteria, microstructural details of materials, etc.

Microstructural Features which Concern Us

- **Grain size: from <μm to the cm regime**
- **Grain shapes**
- **Precipitate size: mostly in the μm regime**
- **Volume fractions and distributions of various phases**
- **Defects such as cracks and voids: <μm to the cm regime**

Phase contrast microscopy works by introducing an additional phase shift between R_0 and R_1, creating a difference in amplitude of light at C and D. That is, contrast is created. This phase shift is introduced by placing a phase plate in the back focal plane of the objective lens. In positive phase contrast the first-order diffracted beam is shifted an additional 90° relative to the zeroth-order (direct) beam. The result is that R_1 is directed opposite to R_0 so that they subtract at image point C and give a minimum of intensity. At image point D, R_1 is further retarded by 90° with respect to R_0 and is therefore at right

Phase Contrast Microscopy - Phase contrast microscopy, first described in 1934 by Dutch physicist Frits Zernike, is a contrast-enhancing optical technique that can be utilized to produce high-contrast images of transparent specimens such as living cells, microorganisms, thin tissue slices, lithographic patterns, and sub-cellular particles (such as nuclei and other organelles). *In effect, the phase contrast technique employs an optical mechanism to translate minute variations in phase into corresponding changes in amplitude, which can be visualized as differences in image contrast.* One of the major advantages of phase contrast microscopy is that living cells can be examined in their natural state without being killed, fixed, and stained. As a result, the dynamics of ongoing biological processes in live cells can be observed and recorded in high contrast with sharp clarity of minute specimen detail.

http://www.microscopyu.com/tutorials/java/phasecontrast/microscopealignment/index.html

33.5 PHASE CONTRAST MICROSCOPY

Optical phase contrast microscopy is used with essentially transparent specimens and is probably most widely used with biological materials. It is important to have some degree of understanding of phase contrast microscopy with respect to materials characterization, not only for its direct use but also because of the analogy to phase contrast effects in electron microscopy. The subject is complicated and a full treatment is beyond the scope of this book. An attempt is made to present the basic ideas in enough detail to give a physical understanding of the origin of the enhanced contrast and the techniques for achieving such contrast. The treatment here is an abbreviated version of the simplified vector model given by Goldstein (Goldstein, 1982).

An incident beam of parallel light is shown illuminating a specimen in Figure 33.4. According to Huygens' construction each point of the specimen can be considered to emit a spherical wave and the resulting waves added to give the result of scattering of light by the specimen (Born and Wolf, 1975). The Abbe theory of image formation considers the behavior of a grating in the specimen to treat the formation of an image and its resolution and contrast. The figure shows a grating made up of transmitting portions (slots) and opaque portions (bars). A spherical wave is emitted from the center of each slot. The envelope of these waves gives the zeroth-order diffraction maximum, which is the direct beam. The drawing also shows the formation of the first-order diffraction maximum by interference of light at a distance one wavelength from the first slot with light at a distance two wavelengths from the second slot, and so on. This is the same effect as one-dimensional Bragg diffraction of X-rays or electrons.

Figure 33.5 shows the formation of a diffraction pattern and an image from the diffracted beams. Only the zeroth- and first-order diffracted beams are shown, but these are sufficient to illustrate the principles involved. Each diffracted beam consists of parallel light, and is brought into focus in the back focal plane of the objective lens. The position of focus in the back focal plane will depend on the angle that the diffracted beam makes with the optic axis of the microscope. Thus the direct beam is focused into a

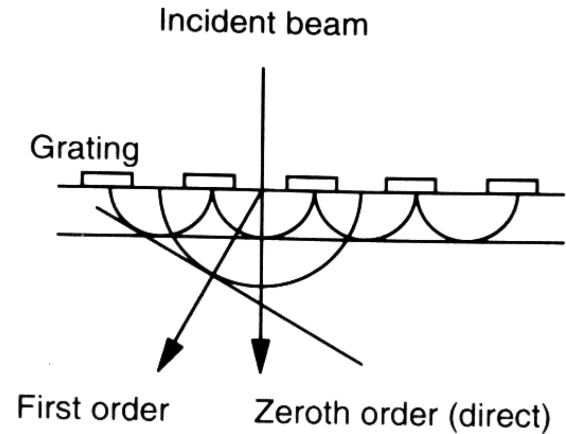


Figure 33.4 Diffraction from a grating

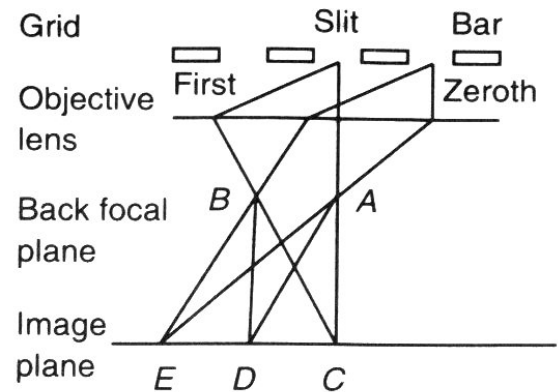


Figure 33.5 Diffraction pattern and image

diffracted spot at point A on the optic axis, while the first-order diffracted beam is focused into a diffraction spot at point B off the optic axis. The rays of light continue to form an image of the grating in the image plane. Points A and B are equidistant from point C (the back focal "plane" is approximately, but not strictly, a plane), and the rays from A and B constructively interfere to form an intensity maximum at C and produce an image of a slit. At point D the waves are completely out of phase and destructively interfere, forming an image of a bar of the grating. At point E the waves are again in phase and interfere constructively to form the image of another slit of the grating.

To understand the principle of phase contrast microscopy one considers the situation of bars and slits that are transparent but that have different indexes of refraction. If the indexes of refraction were the same, each bar would form a spherical wave centered on the bar, as do the slits in Figure 33.4. For the zeroth-order beam all the waves are in phase, giving a strong direct beam. For the first-order beam, the waves from the bars are exactly out of phase with the waves from the slits so that the intensity of the first-order diffracted beam is zero. The Huygens' construction and diffraction picture thus correctly describes the transmission of light through a specimen that is transparent and has a uniform index of refraction.

The phase relations are shown in more detail in Figure 33.6. The arrows show amplitude (length of arrow) and phase (angle of arrow with retardation taken counter-clockwise). At location A of Figure 33.5 (the central diffraction spot in the back focal plane of the objective lens), the phase is taken as zero and the arrow is labeled B_0 for bar, zeroth-order. Here, following Goldstein, a transparent bar has been taken to be on the optic axis and the slit has been taken for the moment to be opaque. At location B, the first-order diffraction from the bar B_1 is of the same magnitude and phase as B_0. Point C is located an integral number of wavelengths from both A and B so that B_0 and B_1 are parallel and equal in size. Point E is located a half-wavelength farther from A than C, and a half-wavelength closer to B than C. At E the waves will again constructively interfere. Point D is located one-quarter wavelength farther from A than C and one-quarter wavelength closer to B than C. Comparing the arrows at D with those at C it is apparent that B_0 has been rotated 90° clockwise (one-quarter wavelength retardation), and B_1 has been rotated 90° counterclockwise (one-quarter wavelength advance in phase). Vectors B_0 and B_1 cancel, giving zero intensity at D.

In the second portion of Figure 33.6 both bar and slit are taken to be transparent and to have equal index of refraction. At A the amplitudes from the bar B_0 and from the slit S_0 are of equal amplitude and in phase. At B the phase of S_1 is retarded by 180°, so that there is zero total first-order diffracted amplitude. At C the contributions are in phase and

add. At D both are retarded by one-quarter wavelength and still add. The result is uniform brightness.

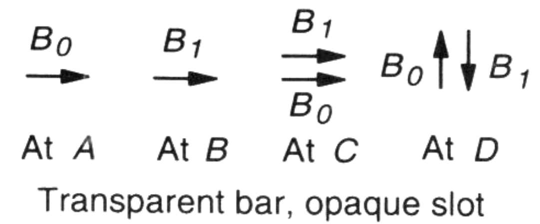
In the third portion of Figure 33.6 the bars are taken to have a higher index of refraction than the slits. This index difference causes both B_0 and B_1 to be retarded in phase by an angle φ. At point A, S_0 and B_0 add to a resultant R_0 as shown. At point B, S_1 and B_1 add to a resultant R_1. The magnitudes of all vectors are equal and the direction of S_1 is opposite to that of S_0. Using boldface for vectors and taking the dot product gives

$$\mathbf{R_0} \cdot \mathbf{R_1} = (\mathbf{S_0} + \mathbf{B_0}) \cdot (-\mathbf{S_0} + \mathbf{B_0}) = 0 \quad (33.9)$$

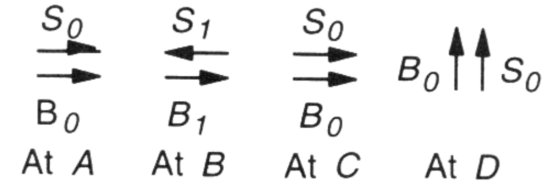
Thus R_0 and R_1 are perpendicular; i.e., the waves are $\pi/4$ out of phase.

The image at C will be formed by interference between R_0 and R_1 with no further change of phase and so will be twice OC in amplitude and phase. The image at D will be formed by R_0 and R_1 after a further phase change of $\lambda/4$ and so will be twice OD. The eye responds to intensity, which is the square of the amplitude, so that the same intensity will be seen at C and D and there will be no contrast in the image despite the difference in phase of the light at C and D.

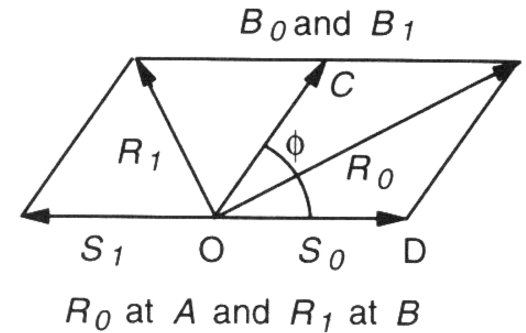
Phase contrast microscopy works by introducing an additional phase shift between R_0 and R_1, creating a difference in amplitude of light at C and D. That is, contrast is created. This phase shift is introduced by placing a phase plate in the back focal plane of the objective lens. In positive phase contrast the first-order diffracted beam is shifted an additional 90° relative to the zeroth-order (direct) beam. The result is that R_1 is directed opposite to R_0 so that they subtract at image point C and give a minimum of intensity. At image point D, R_1 is further retarded by 90° with respect to R_0 and is therefore at right



Transparent bar, opaque slot



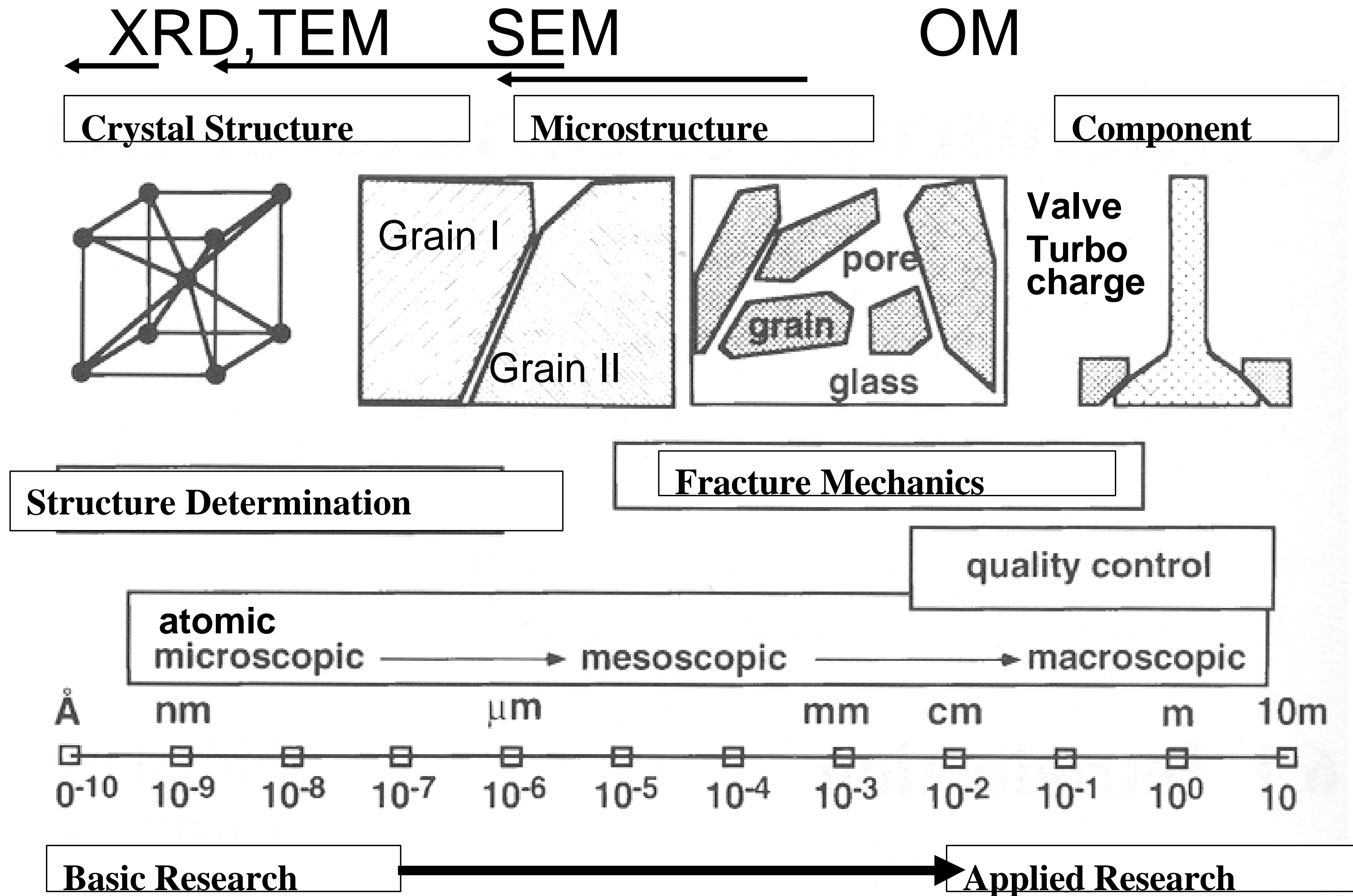
Transparent bar, transparent slot, equal index



Transparent bar, transparent slot, unequal index

Figure 33.6 Vector model of phase contrast

Scale and Microscopy Techniques



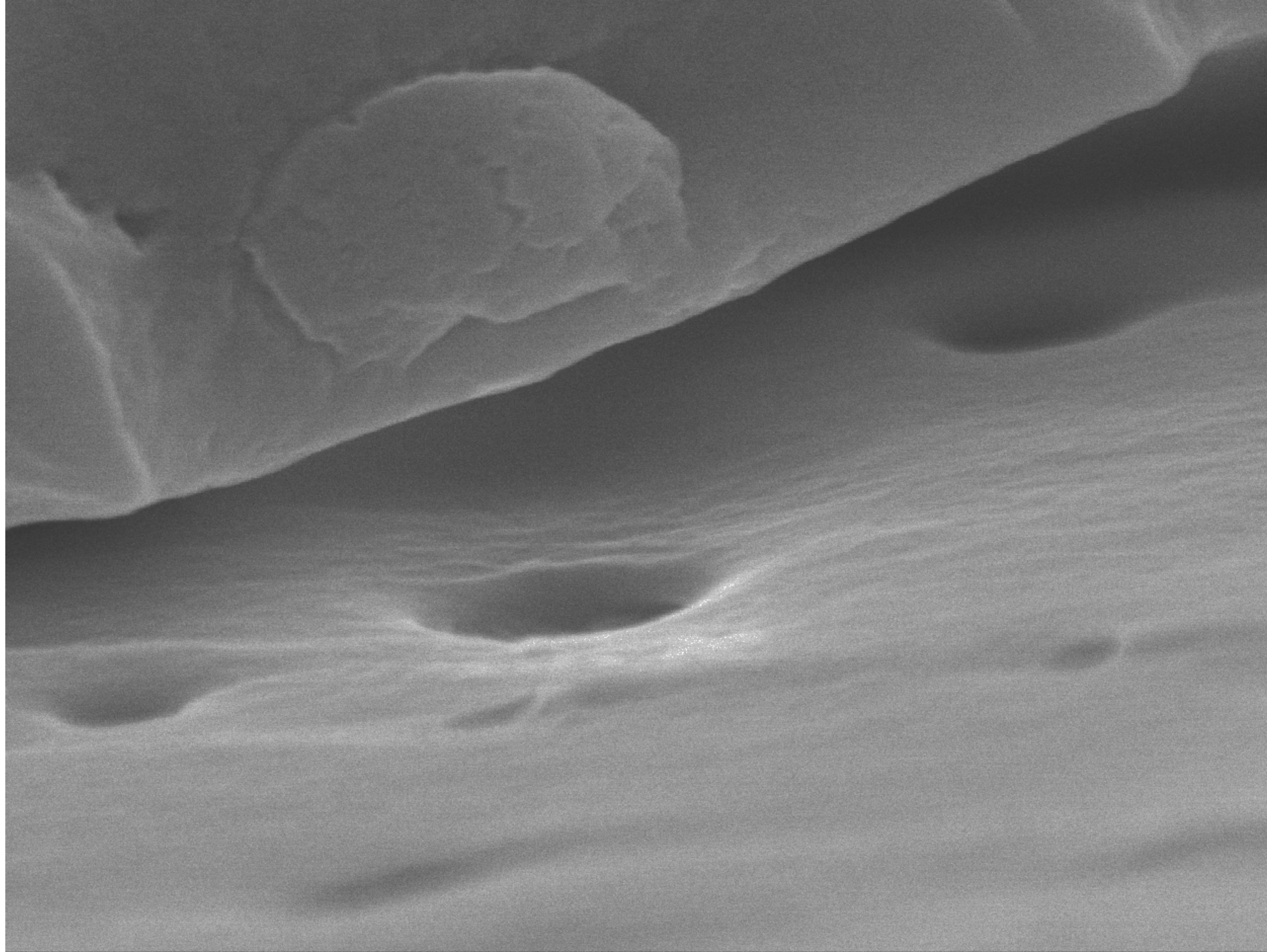
Microstructure ranging from crystal structure to Engine components (Si_3N_4)

Advantages of Using SEM over OM

	Mag	Depth of Field	Resolution
OM:	4x – 1000x	15.5mm – 0.19mm	~ 0.2mm
SEM:	10x – 500Kx	4mm – 0.4mm	1-10nm

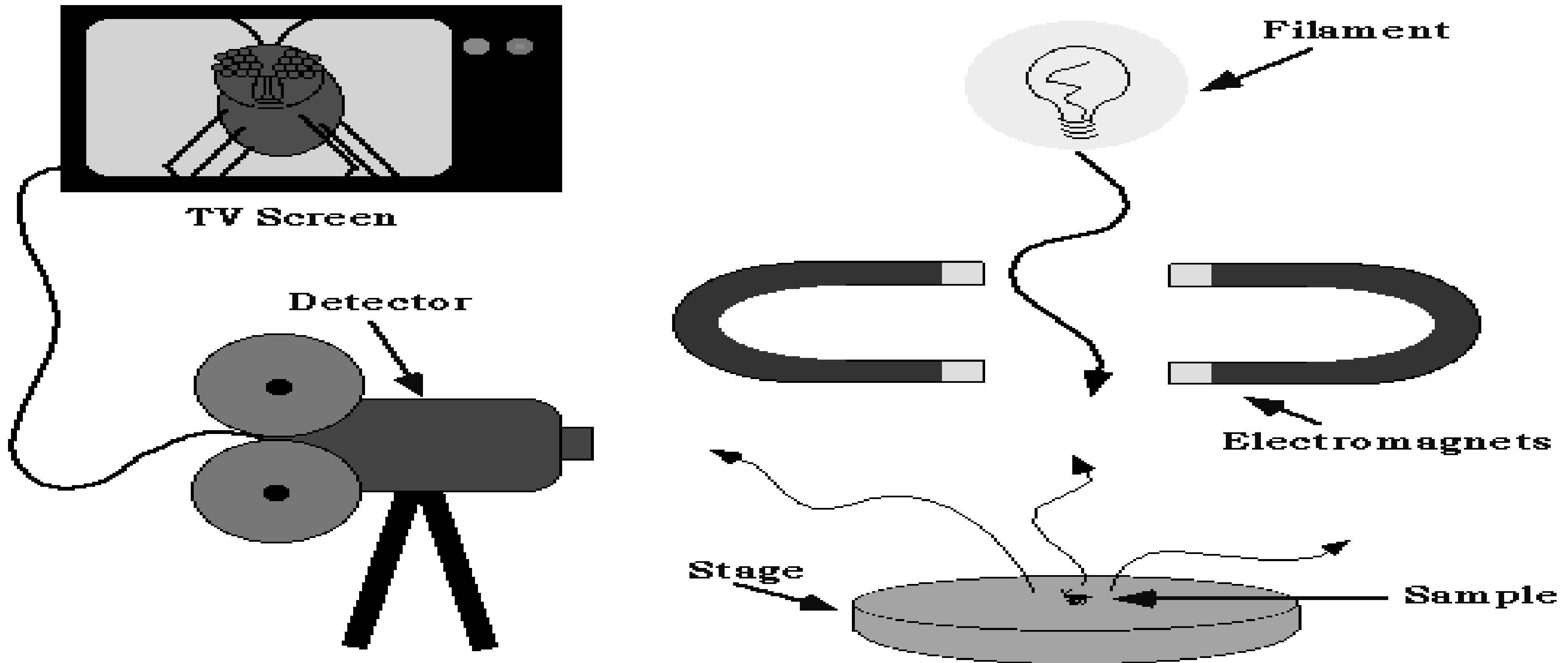
The SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time and produces an image that is a good representation of the three-dimensional sample. The SEM also produces images of high resolution, which means that closely features can be examined at a high magnification.

The combination of higher magnification, larger depth of field, greater resolution and compositional and crystallographic information makes the SEM one of the most heavily used instruments in research areas and industries, especially in semiconductor industry.


NONE
SEI
5.0kV
X50,000
100nm
WD 2.9mm

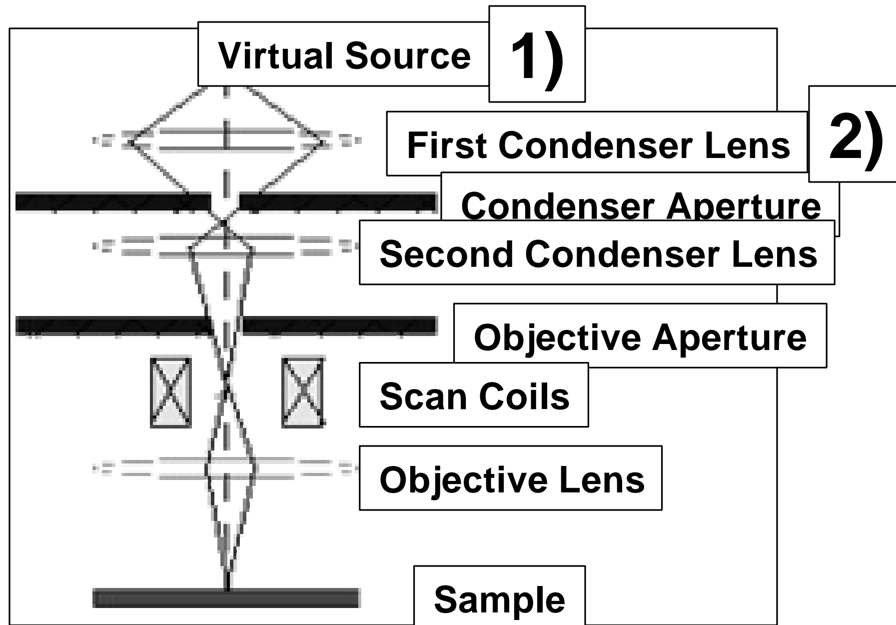
2.1 The Microscope Column

General Layout



In simplest terms, an SEM is really nothing more than a television. We use a filament to get electrons, magnets to move them around, and a detector acts like a camera to produce an image.

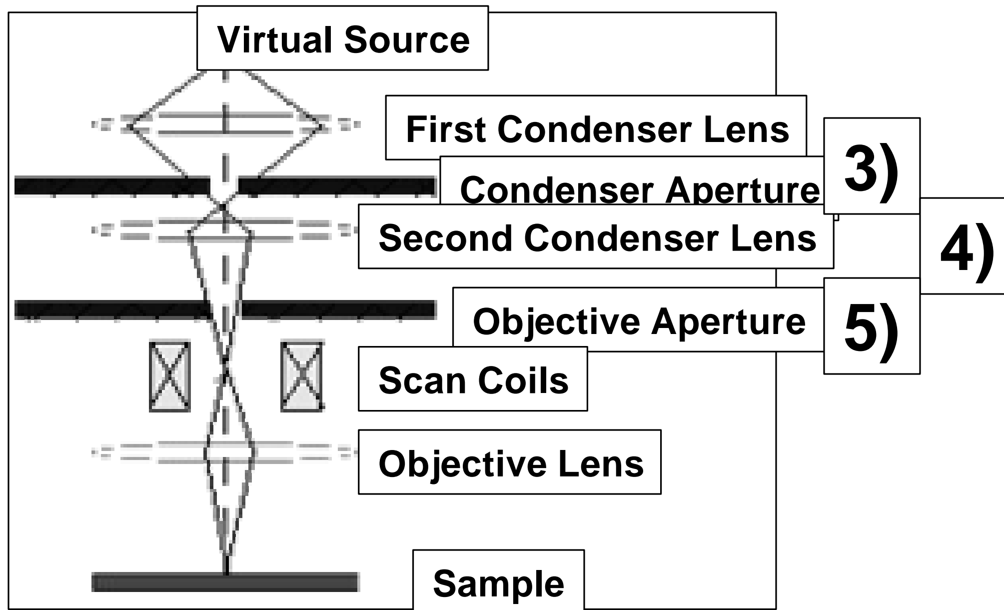
Scanning Electron Microscope



1) The "Virtual Source" at the top represents the electron gun, producing a stream of monochromatic electrons.

2) The stream is condensed by the first condenser lens (usually controlled by the "coarse probe current knob"). This lens is used to both form the beam and limit the amount of current in the beam. It works in conjunction with the condenser aperture to eliminate the high-angle electrons from the beam.

Scanning Electron Microscope

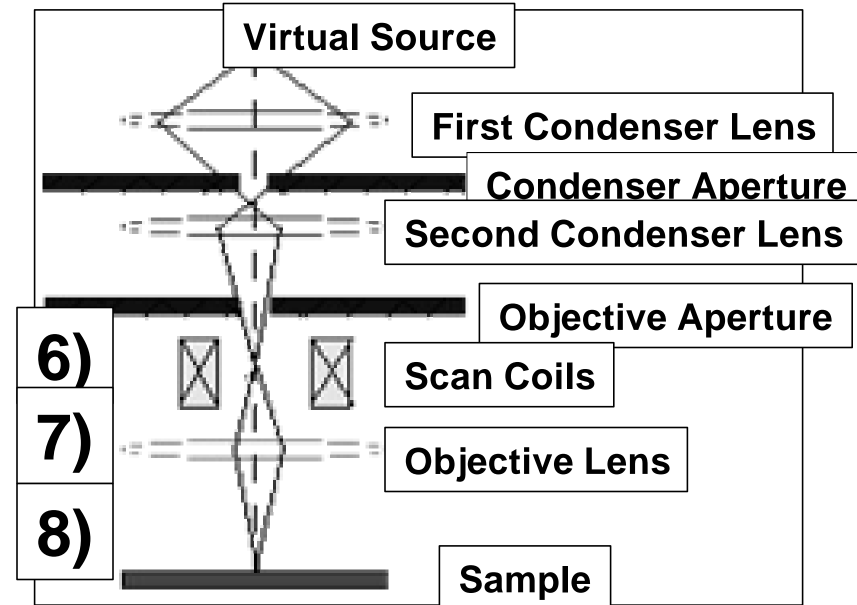


3) The beam is then constricted by the condenser aperture (usually not user selectable), eliminating some high-angle electrons.

4) The second condenser lens forms the electrons into a thin, tight, coherent beam and is usually controlled by the "fine probe current knob".

5) A user selectable objective aperture further eliminates high-angle electrons from the beam.

Scanning Electron Microscope

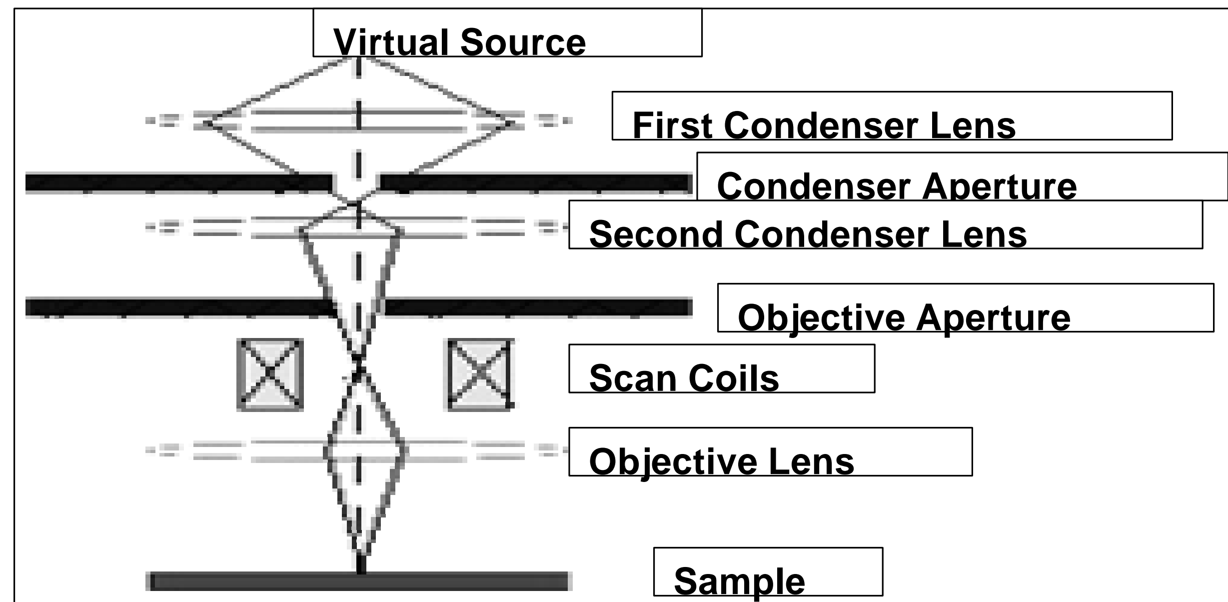


6) A set of coils then "scan" or "sweep" the beam in a grid fashion (like a television), dwelling on points for a period of time determined by the scan speed (usually in the microsecond range).
7) The final lens, the Objective, focuses the scanning beam onto the part of the specimen desired.
8) When the beam strikes the sample (and dwells for a few microseconds) interactions occur inside the sample and are detected with various instruments.

Scanning Electron Microscope

9) Before the beam moves to its next dwell point these instruments count the number of interactions and display a pixel on a CRT whose intensity is determined by this number (the more reactions the brighter the pixel).

10) This process is repeated until the grid scan is finished and then repeated, the entire pattern can be scanned 30 times/sec.



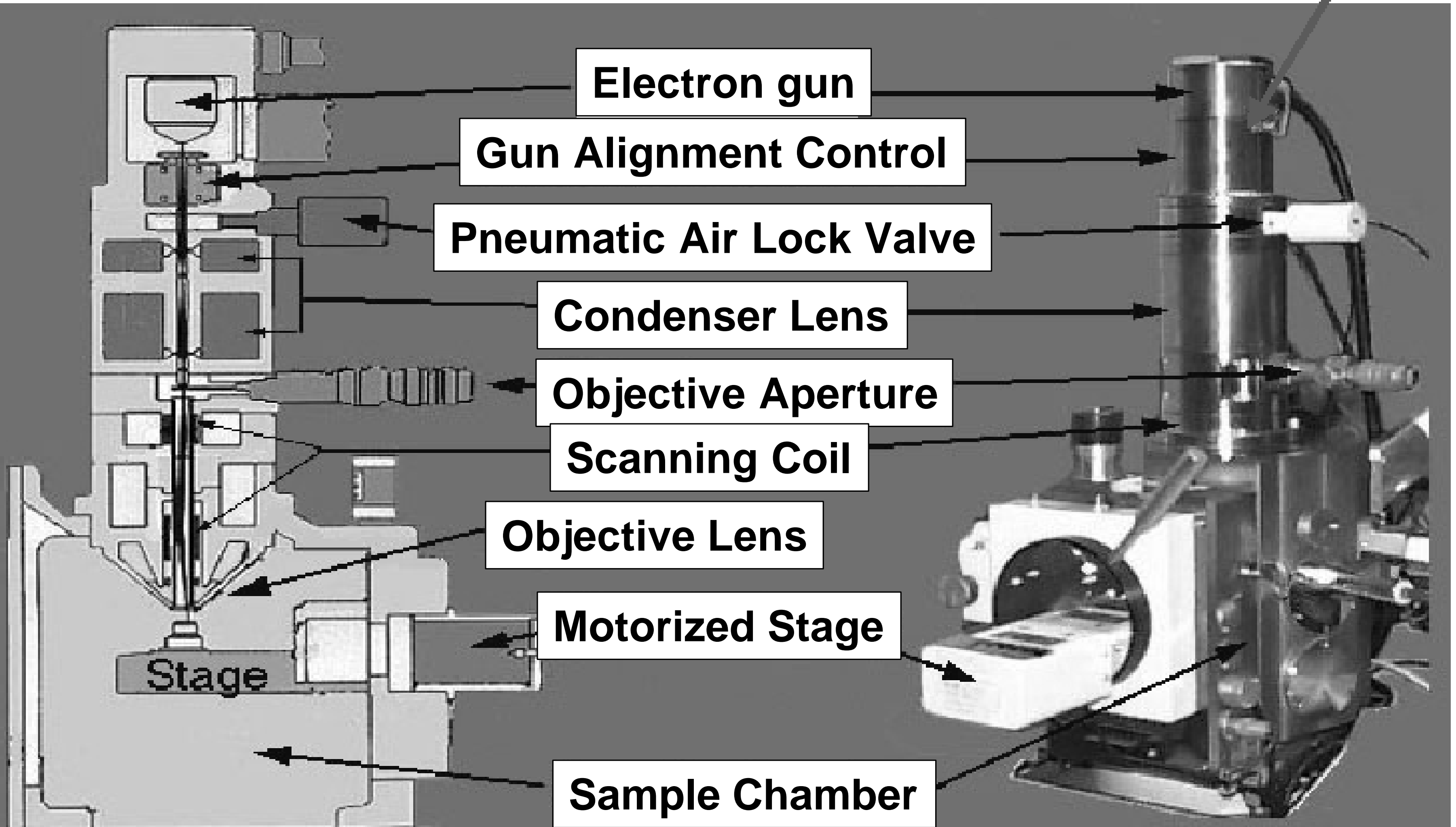
Parts of the Microscope

1. Electron optical column consists of:
   - electron source to produce electrons
   - magnetic lenses to de-magnify the beam
   - magnetic coils to control and modify the beam
   - apertures to define the extent of beam, prevent electron spray, etc.
2. Vacuum systems consists of:
   - chamber which “holds” vacuum
   - pumps to produce vacuum
   - valves to control vacuum
   - gauges to monitor vacuum

Parts of the Microscope

3. Signal Detection & Display consists of:
   - detectors which collect the signal
   - electronics which produce an image from the signal

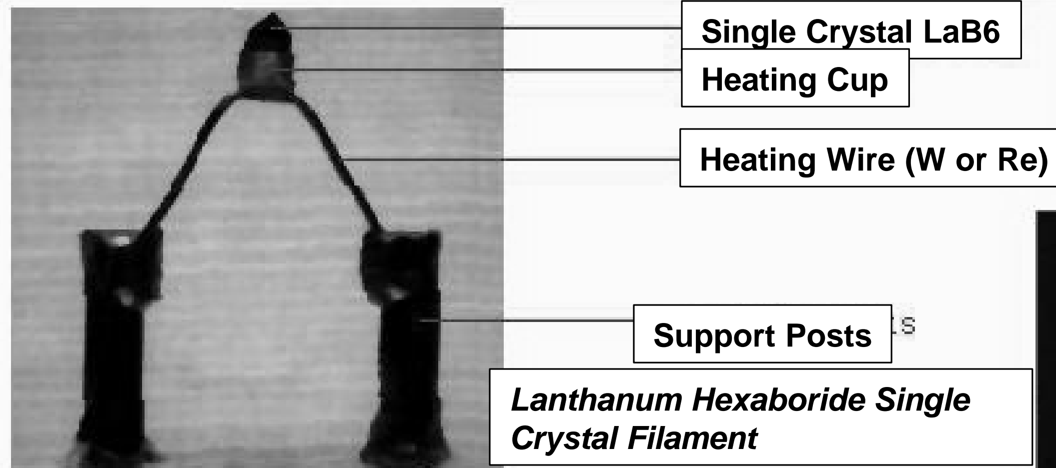
A Look Inside the Column

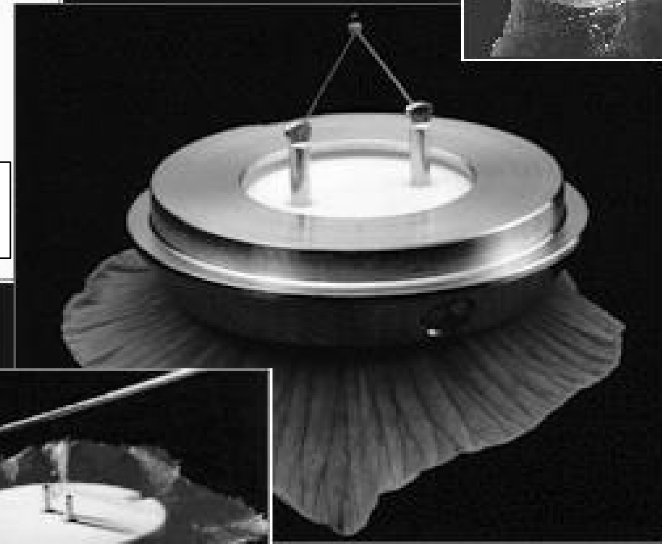


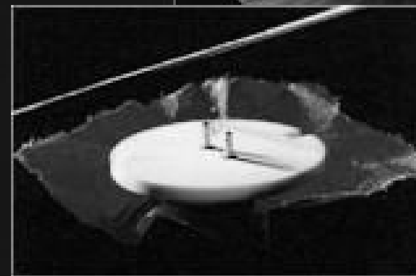
Getting a Beam

- A filament is heated to a high T to emit electrons.
- A bias (of up to 45 kV) between the filament and a target material draws the electrons to an anode.
- Up to this point, the process is identical to that used to form x-rays. However, instead of striking a target to form x-rays, the electrons are drawn towards an anode, where they go through a hole, forming a beam.
- This beam is attracted through the anode, condensed by a condenser lens, and focused as a very fine point on the sample by the objective lens.

Electron Beam Source





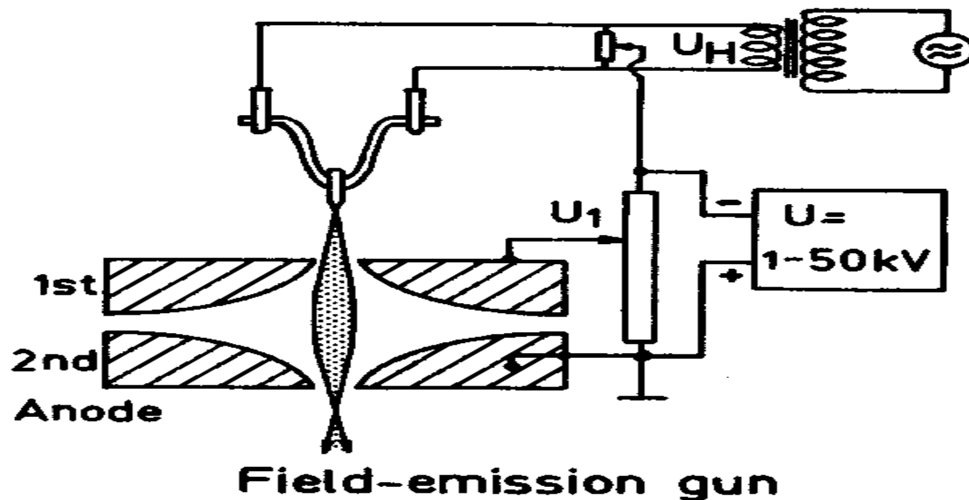


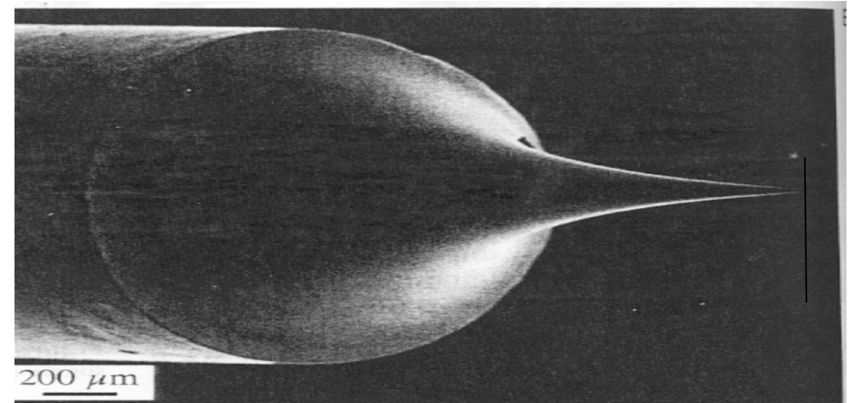
W / LaB_6 (Filament)

Thermionic or Cold Cathode (Field Emission Gun)

Field Emission Gun

- **The tip of a tungsten needle is made very sharp (radius < 0.1 μm)**
- **The electric field at the tip is very strong (> 10^7 V/cm) due to the sharp point effect**
- **Electrons are pulled out from the tip by the strong electric field**
- **Ultra-high vacuum (better than 10^{-6} Pa) is needed to avoid ion bombardment to the tip from the residual gas.**
- **Electron probe diameter < 1 nm is possible**

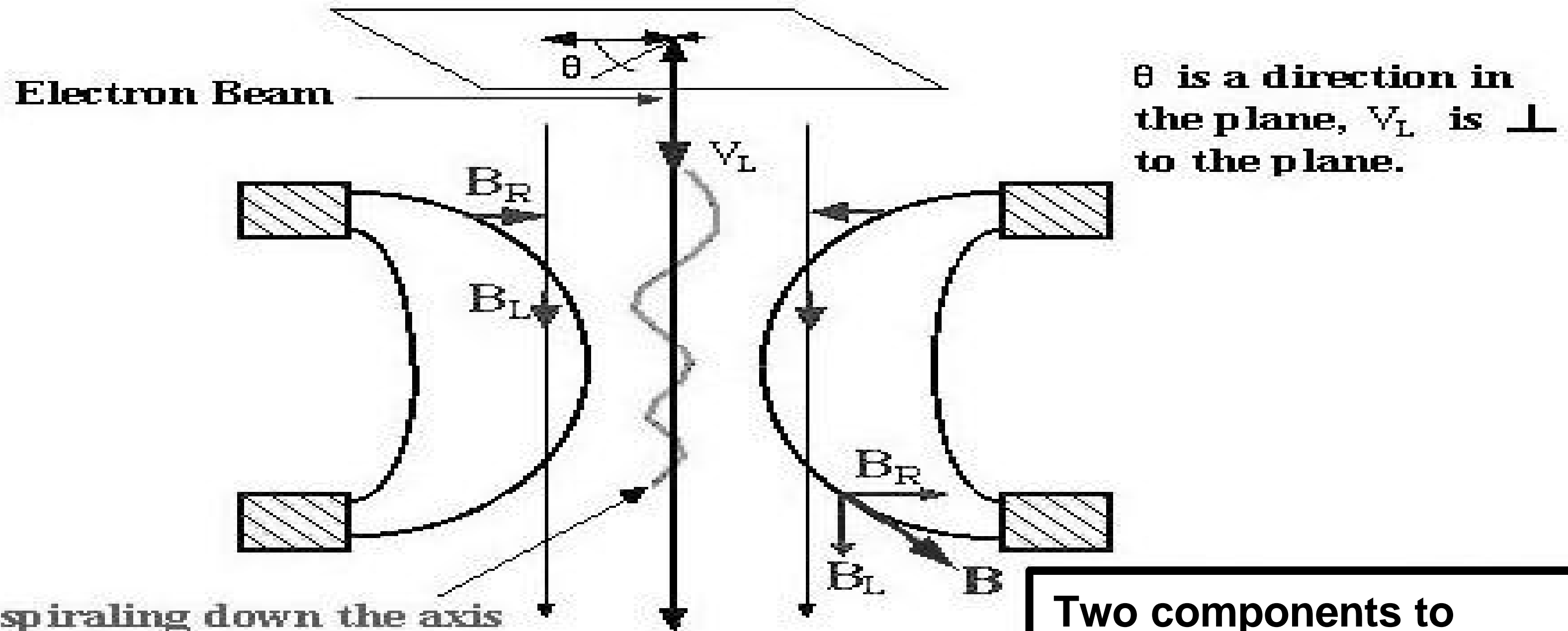




Electromagnetic Lenses

Lorentz force equation:

$$F = q_0 v \times B$$



Two components to the B field:
B_L = longitudinal component (down the axis)
B_R= radial component (perpendicular to axis)

Nonaxial electrons will experience a force both down the axis and one radial to it. Only electrons traveling down the axis feel equal radial forces from all sides of the lens. The unequal force felt by the off-axis electrons causes spiraling about the optic axis.

after "Electron Beam Analysis of Materials", M.H. Loretto

Electromagnetic Lenses

- **Condenser lens – focusing determines the beam current which impinges on the sample.**

- **Objective lens – final probe forming**

- **The objective lens determines the final spot size of the electron beam, i.e., the resolution of a SEM.**

Electromagnetic Lenses

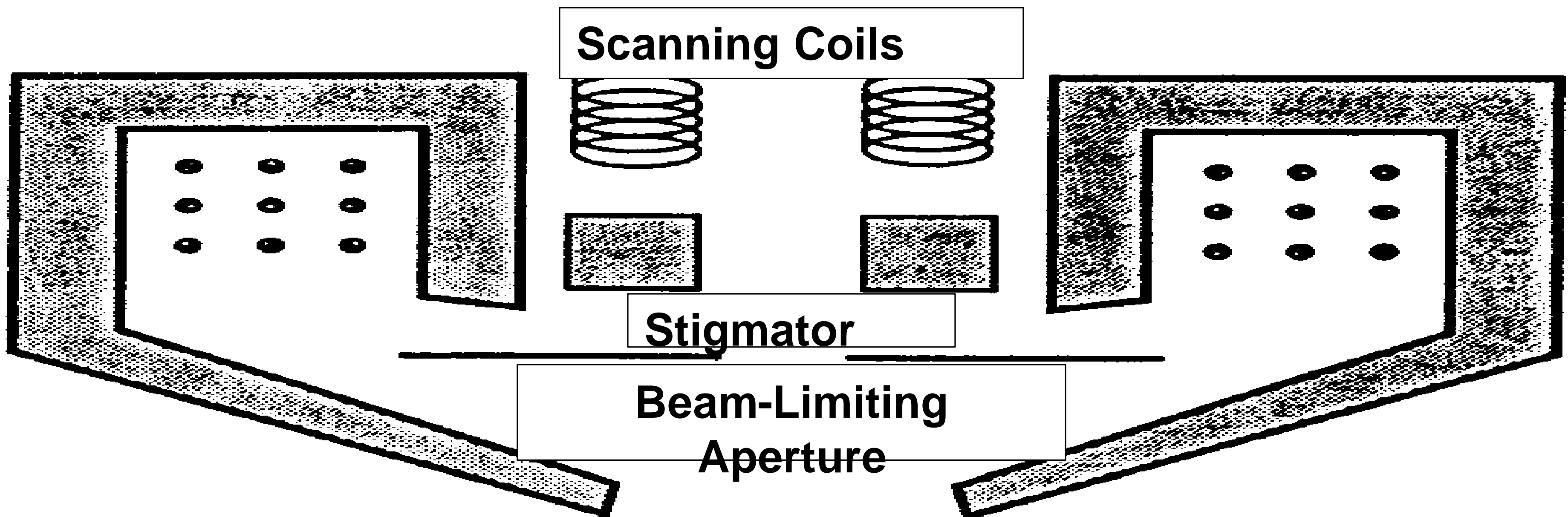
The Condenser Lens

- **For a thermionic gun, the diameter of the first cross-over point ~20-50μm.**
- **If we want to focus the beam to a size < 10 nm on the specimen surface, the magnification should be ~1/5000, which is not easily attained with one lens (say, the objective lens) only.**
- **Therefore, condenser lenses are added to demagnify the cross-over points.**

Electromagnetic Lenses

The Objective Lens

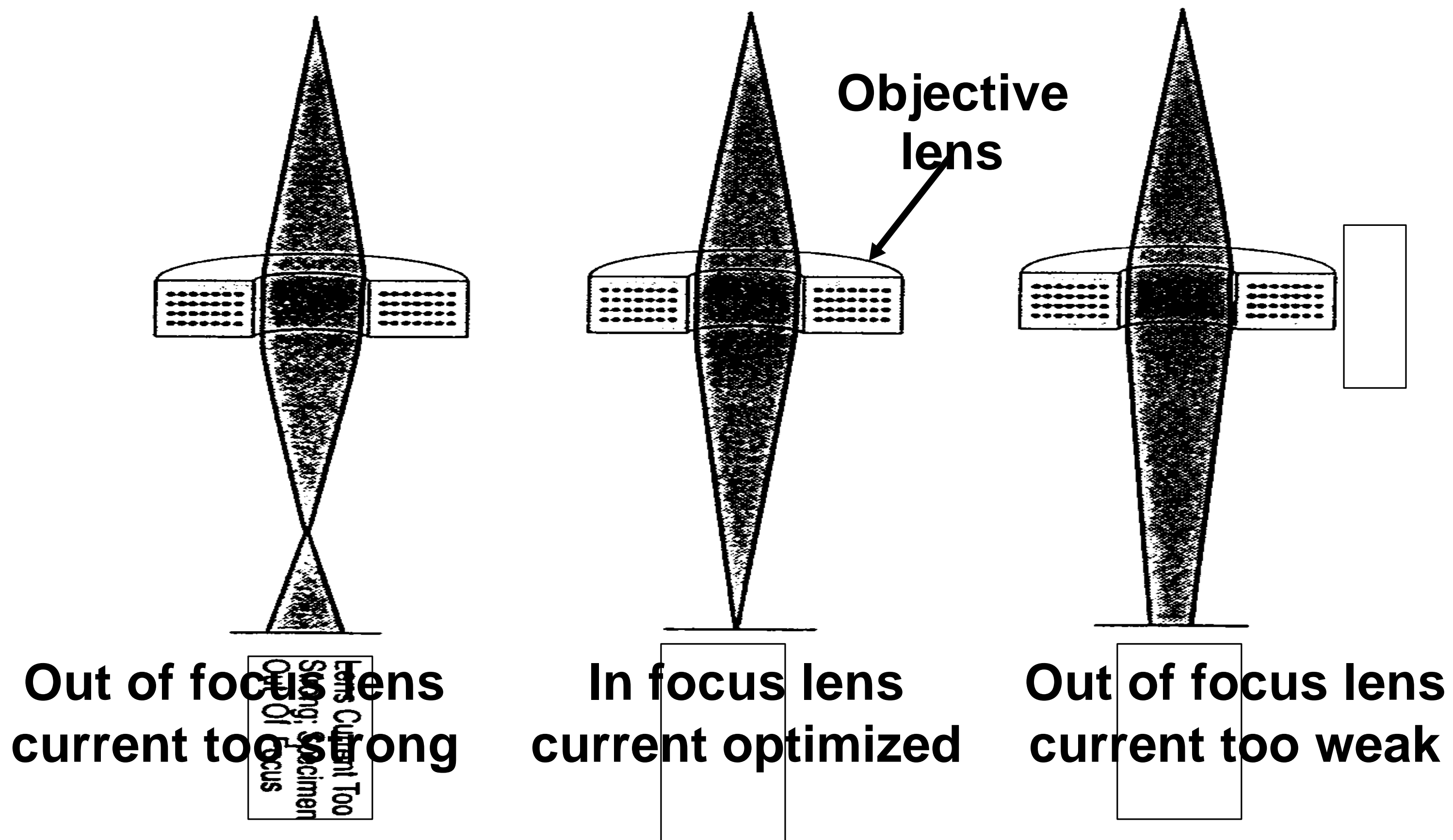
- The objective lens controls the final focus of the electron beam by changing the magnetic field strength
- The cross-over image is finally demagnified to an ~10nm beam spot which carries a beam current of approximately 10^{-9} - 10^{-13} A.



Electromagnetic Lenses

The Objective Lens - Focusing

- **By changing the current in the objective lens, the magnetic field strength changes and therefore the focal length of the objective lens is changed.**



Electromagnetic Lenses

The Objective Lens - Stigmator

- The objective lens is machined to very high precision and the magnetic field pattern is very carefully designed.

- However, the precision attainable by machining cannot match that required for controlling a beam with a 10 nm diameter.

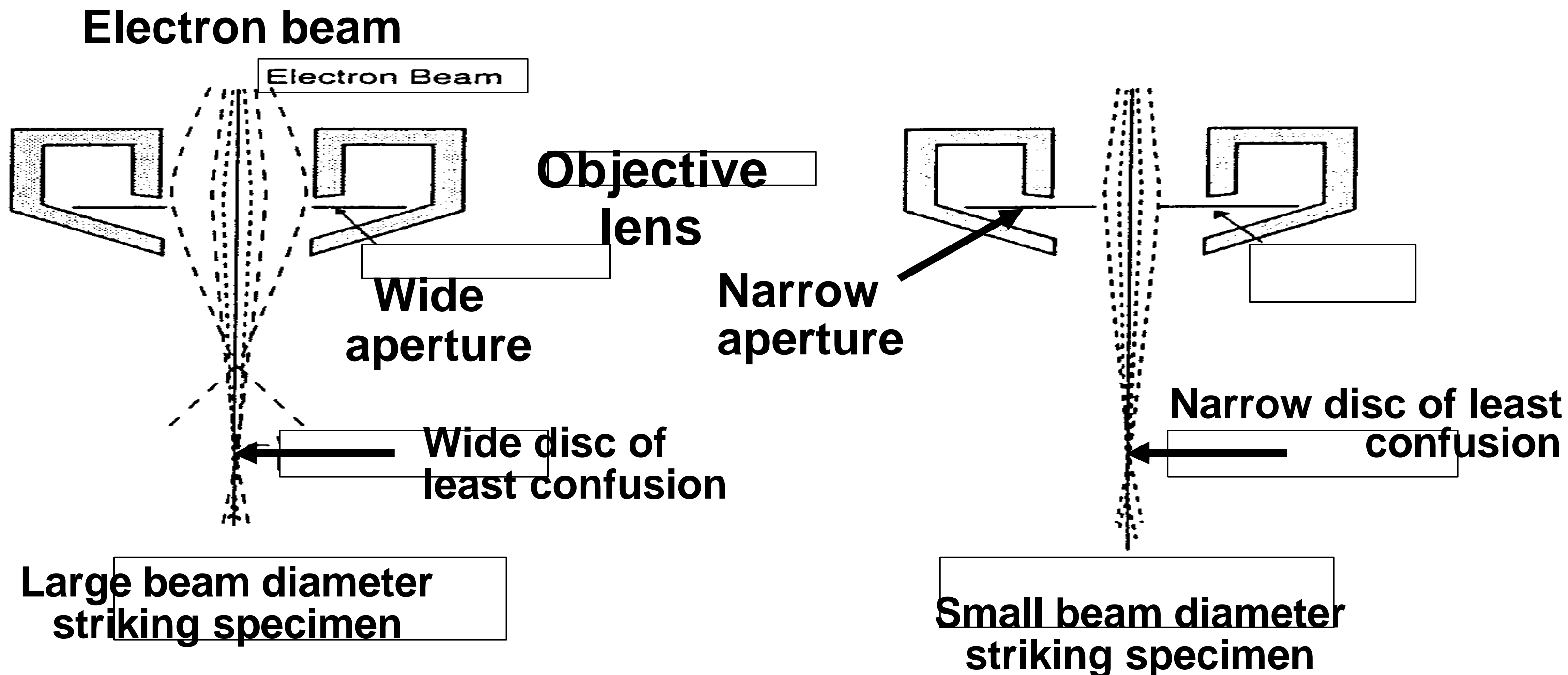
- The **stigmator**, which consist of two pairs of pole-pieces arranged in the X and Y directions, is added to correct the **minor imperfections** in the objective lens.

Electromagnetic Lenses

The Objective Lens - Aperture

- **Since the electrons coming from the electron gun have spread in kinetic energies and directions of movement, they may not be focused to the same plane to form a sharp spot.**

- **By inserting an aperture, the stray electrons are blocked and the remaining narrow beam will come to a narrow "Disc of Least Confusion"**

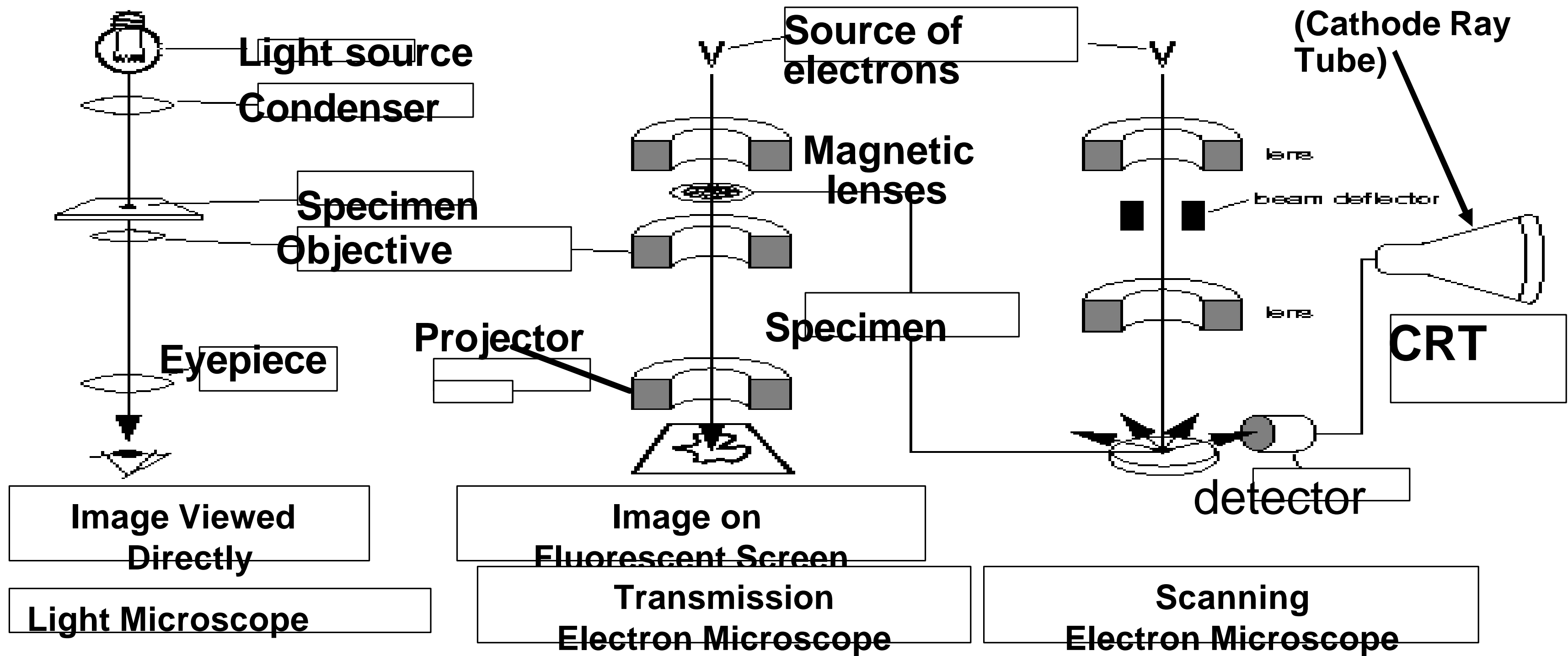


Electron vs. Optical Lenses

- e^-'s don't actually touch the lens
  No definite interface
- e^-'s rotate in the magnetic field
- e^-'s repel each other
- f µ H µ I
  - Focus and magnification controlled electronically
  - No physical movements
- e^- lenses can only be positive elements (converging)
- Can't correct e^- lens aberrations like you can with compound optical lenses
- e^- lenses always operate at small apertures

H = MFS

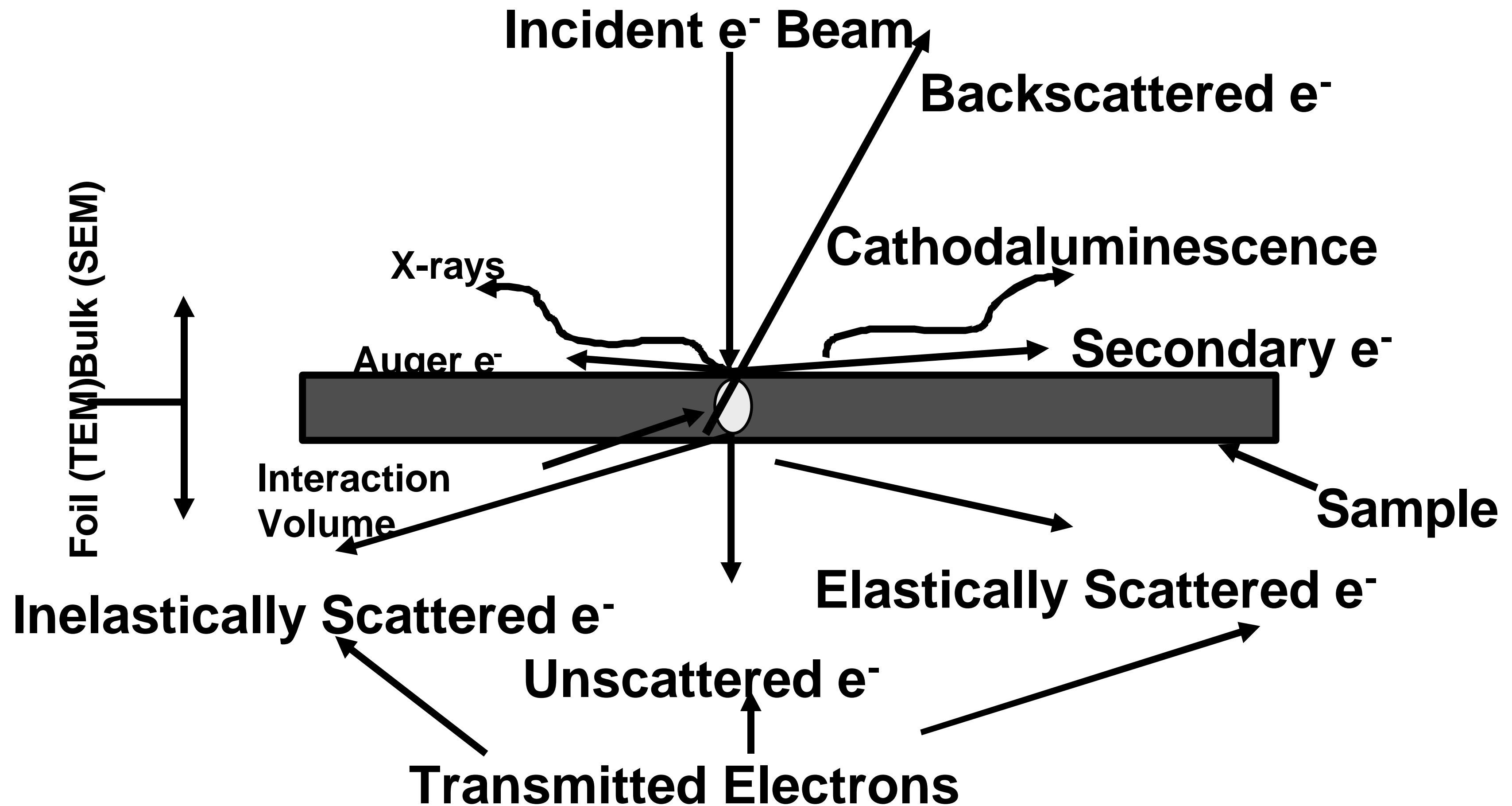
Comparison of OM,TEM and SEM



Principal features of an optical microscope, a transmission electron microscope and a scanning electron microscope, drawn to emphasize the similarities of overall design.

2.2 Signal Detection and Display

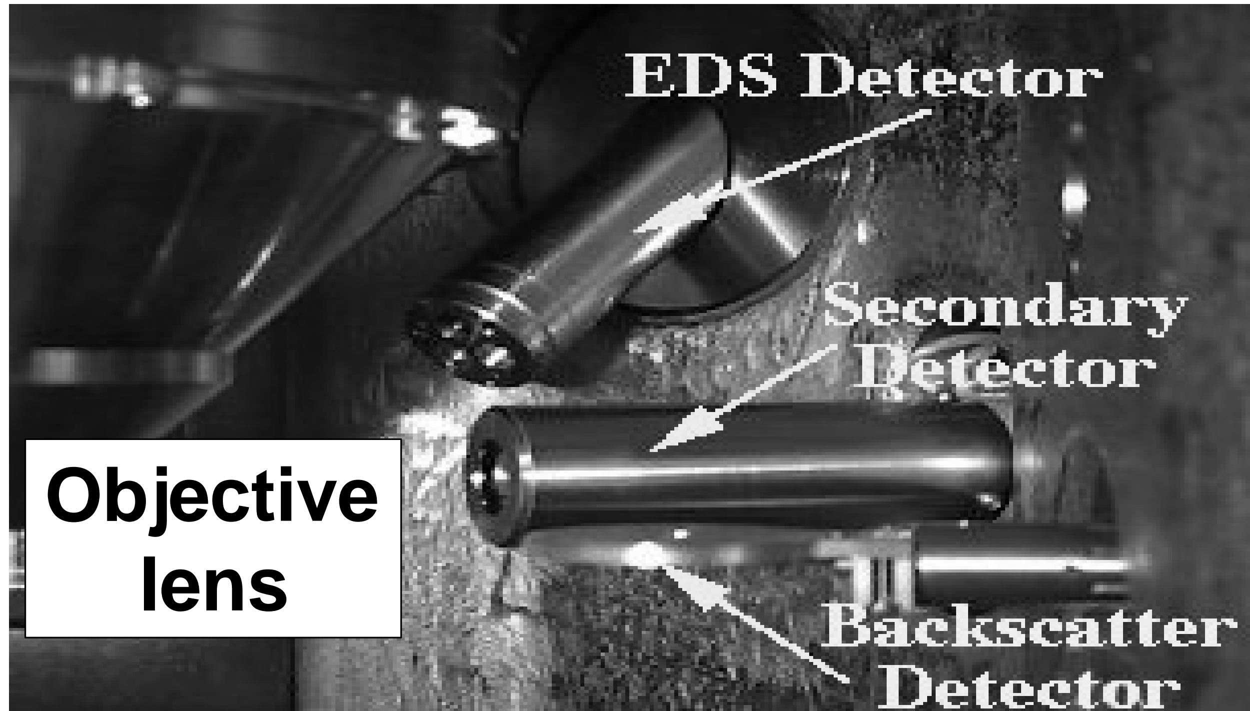
When an electron beam strikes a sample, a large number of signals are generated.

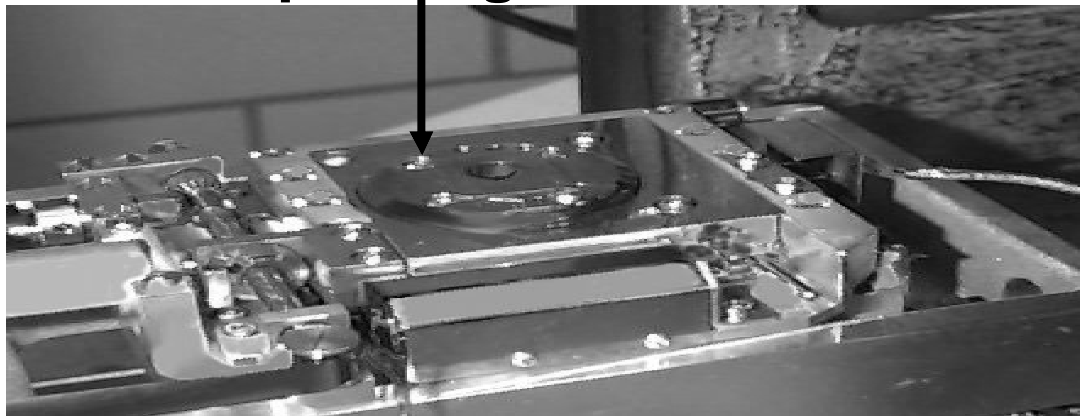


We can divide the signals into two *broad* categories:

a) electron signals, b) photon signals

Electron Detectors and Sample Stage





Example

Specimen Interaction Volume

The volume inside the specimen in which interactions occur while being struck with an electron beam. This volume depends on the following factors:

• Atomic number of the material being examined; higher atomic number materials absorb or stop more electrons and so have a smaller interaction volume.

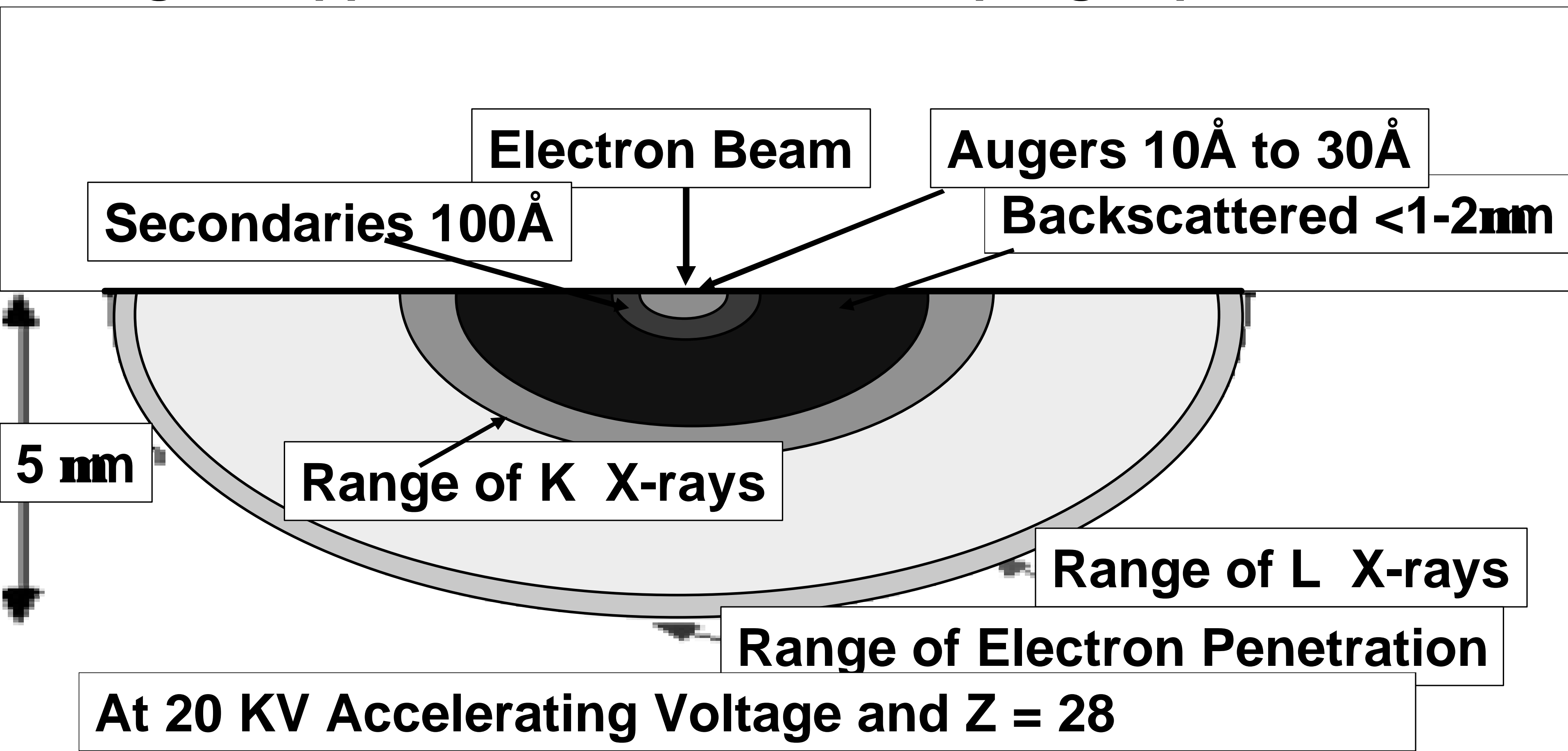
• Accelerating voltage: higher voltages penetrate farther into the sample and generate larger interaction volumes

• Angle of incidence for the electron beam; the greater the angle (further from normal) the smaller the volume

Specimen Interaction Volume

Below is an example of a typical Interaction volume for:

- **Specimen with atomic number 28, 20 kV**
- **0° degrees tilt, incident beam is normal to specimen surface noting the approximate maximum sampling depths for the**



Beam Interaction Simulations

Theoretical SrS density: 3.7g/cm^3, Long. range: 1062 Å, Long. straggle: 384 Å

SrS
5000 Å
Si
2 mm
15 kV

5000 Å
SrS
Si
10 kV
1 mm

SrS
5000 Å
Si
5 kV
2 mm

SrS
2 kV
0.5 mm
5000 Å

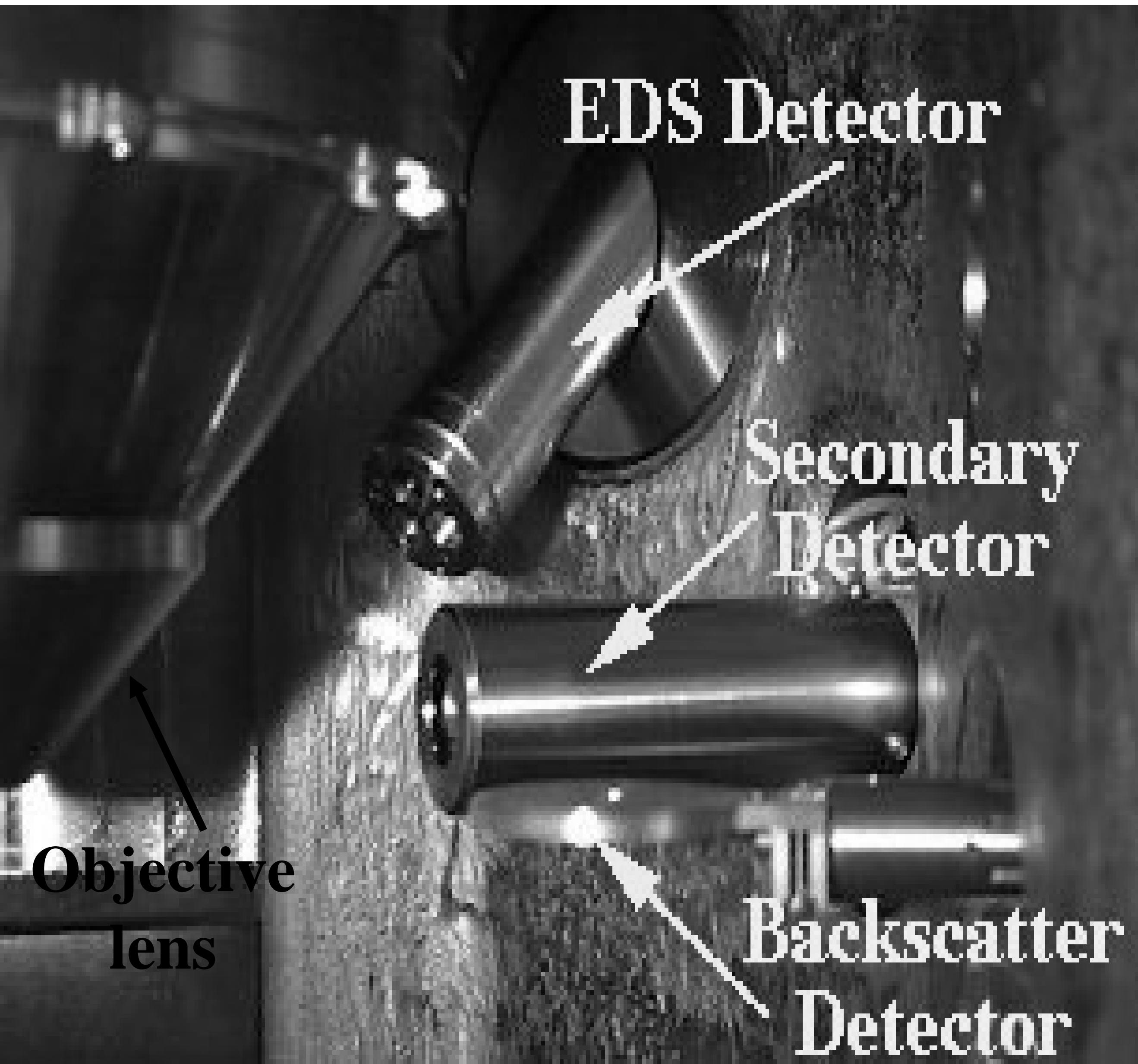
Signal Detection and Display

- **If you change the target material, the high and low energy peaks remain (although their intensity may change) while the low intensity peaks change position and are characteristic of the sample.**

- **The reason we produce this type of profile is because the incident electrons we send into the sample are scattered in different ways.**

- **There are two broad categories to describe electron scattering:**
  - **elastic: Backscattered electrons**
  - **inelastic: Secondary electrons**

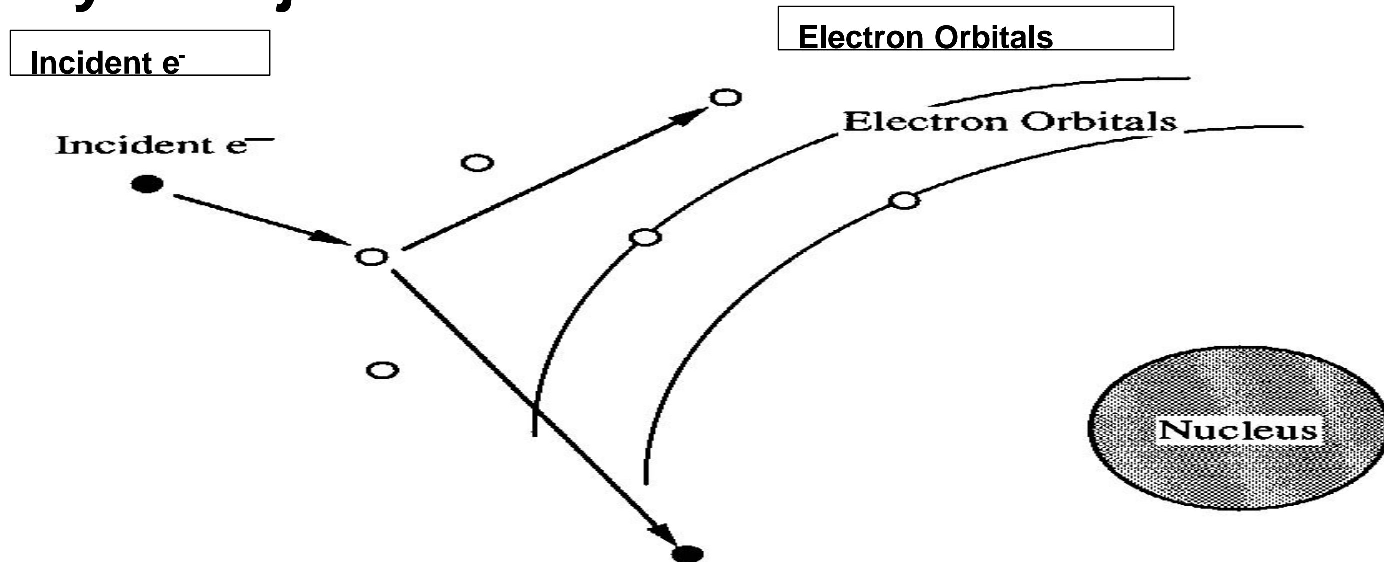
Electron Detectors



Example

Secondary Electrons

These electrons arise due to inelastic collisions between primary electrons (the beam) and loosely bound electrons of the conduction band (more probable) or tightly bound valence electrons. The energy transferred is sufficient to overcome the work function which binds them to the solid and they are ejected.



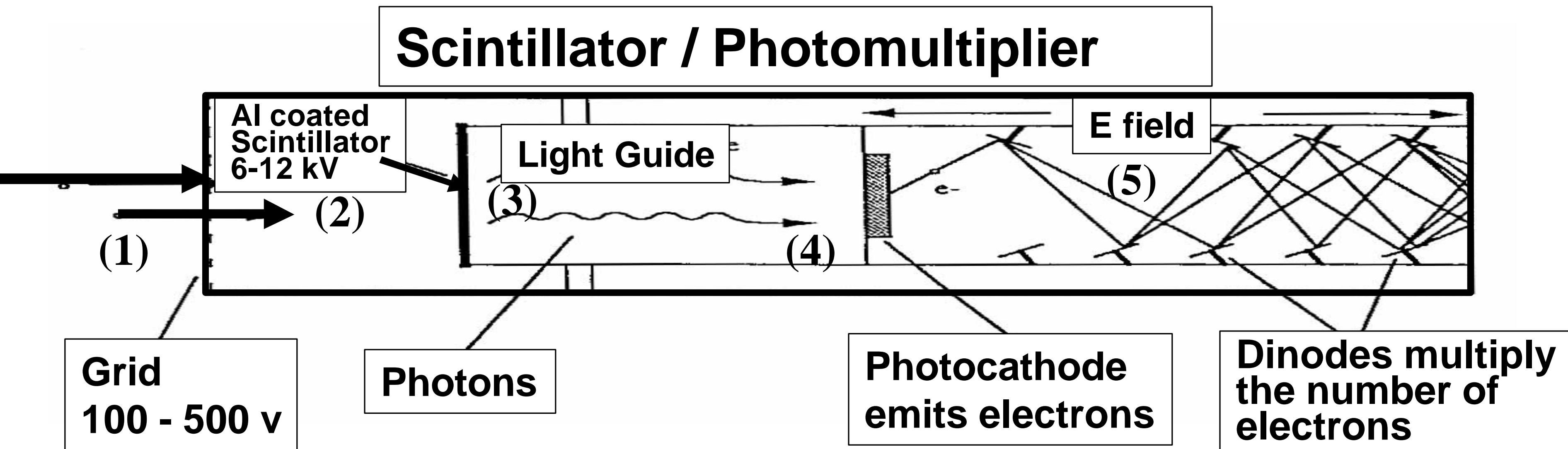
The interaction is Coulombic in nature and the ejected electrons typically have ~ 5 - 10 eV. 50 eV is an arbitrary cut-off below which they are said to be <u>secondary electrons</u>.

Detection

Remember, secondary electrons are low energy electrons. We can easily collect them by placing a positive voltage (100 - 300V) on the front of our detector. Since this lets us collect a large number of the secondaries (50 - 100%), we produce a "3D" type of image of the sample with a large depth of field.

The type of detector used is called a scintillator / photo-multiplier tube.

Detection Sequence

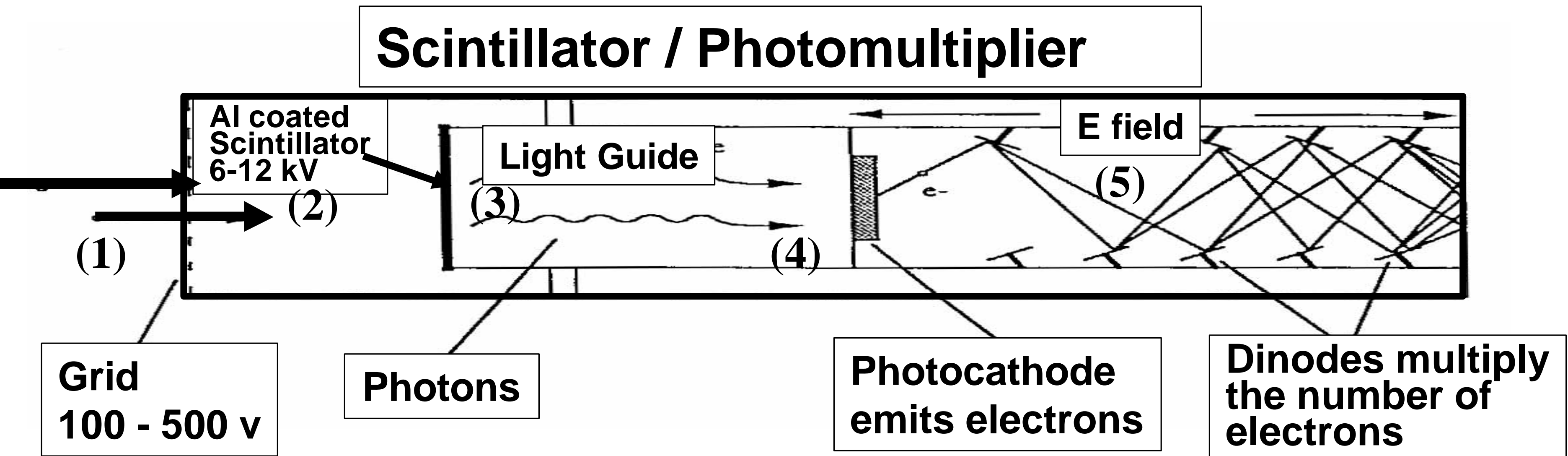


1. Secondary electrons (SE) are accelerated to the front of the detector by a bias voltage of 100 - 500 eV.

2. They are then accelerated to the scintillator by a bias of 6 - 12 keV, (10 KeV is normal).

3. Scintillator is doped plastic or glass covered with a fluorescent material (e.g. Europium). A thin (700Å) layer of Al covers it to prevent light from causing fluorescence. The 10keV potential allows the SE to get through the Al and fluoresce.

Detection Sequence

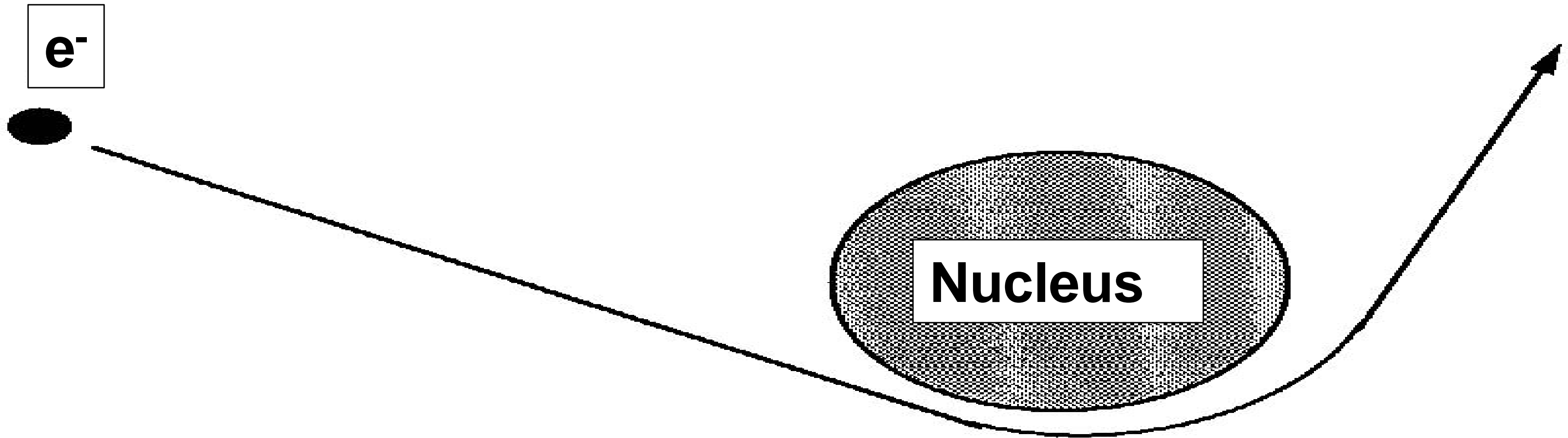


4. The light photons travel down the tube (guide) to a photo-cathode which converts them into electrons

5. The electrons move through the detector, producing more electrons as they strike dinodes. An output electron pulse is then detected.

Backscattered Electrons

Backscattered electrons (BSE) arise due to elastic collisions between the incoming electron and the nucleus of the target atom (i.e. Rutherford scattering). Higher Z, more BSE emitted.

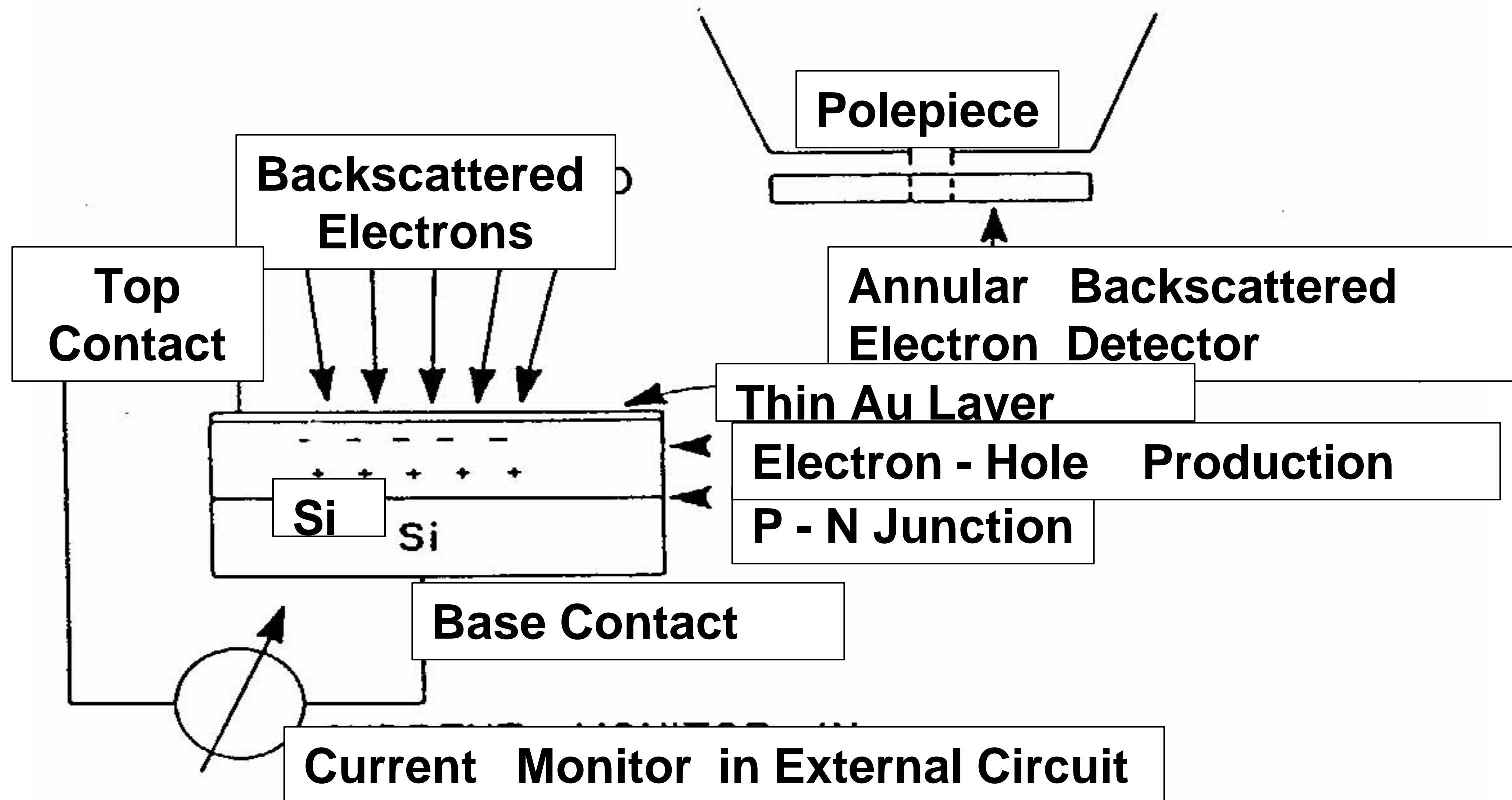


As the name implies, elastic scattering results in little (< 1 eV) or no change in energy of the scattered electrons, although there is a change in momentum (p). Since p = mv and the mass of the electron doesn't change, the direction of the velocity vector must change. The angle of scattering can range from 0 to 180°.

Detection

Since BSE have high energies, they can't be pulled in like secondaries. If you placed a potential on a grid to attract them, you would also attract the incident beam!!

The most common detector used is called a surface barrier detector. It sits above the sample, below the objective lens. BSE which strike it are detected.

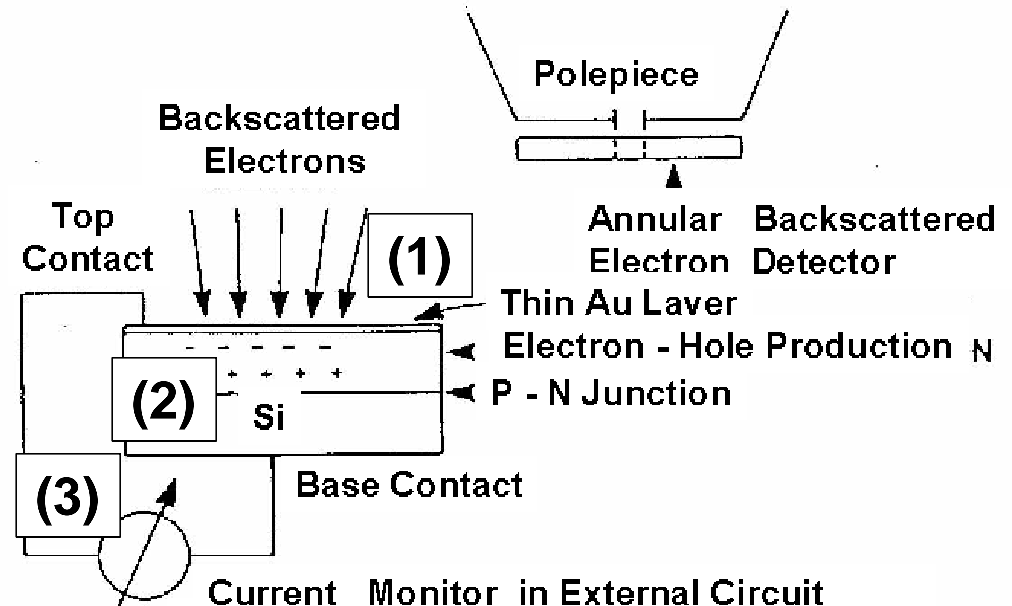


Detection Sequence

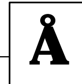
Surface barrier detectors are solid state devices made up of semiconducting materials. A semiconducting material has a filled valence band and an empty conduction band- similar to ceramic materials.

1. When a BSE electron strikes the detector, electrons in the material move from valence to conduction band.

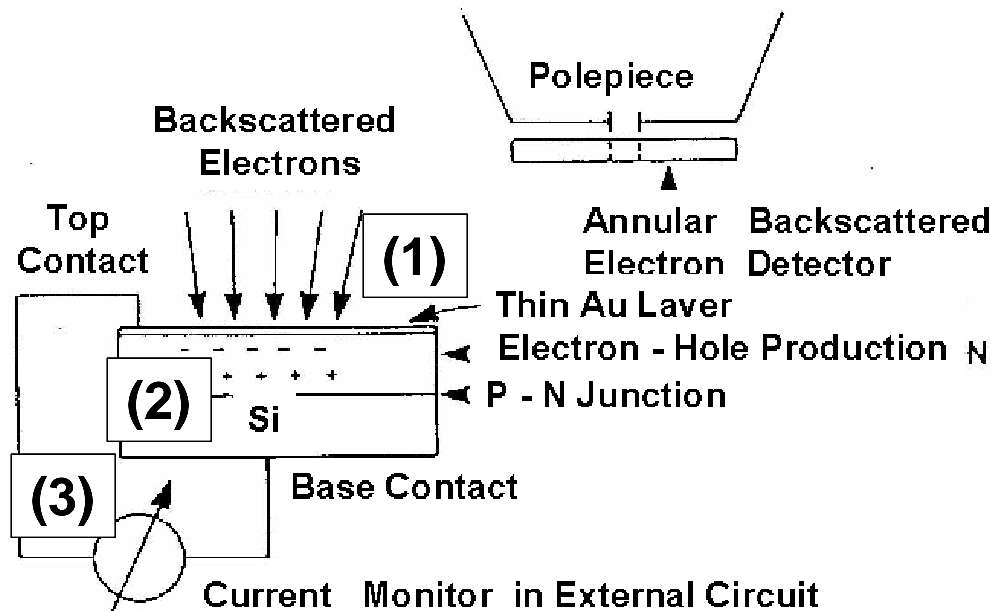
2. The electrons are now free to move in the conduction band or drop back into the valence band.



Detection Sequence

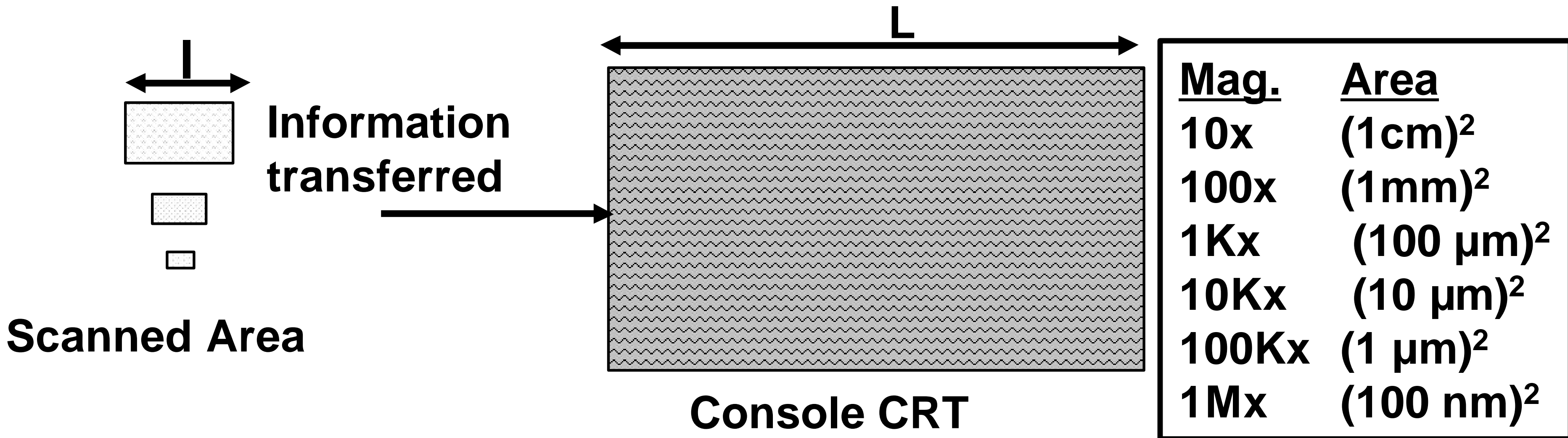


3. If a potential is applied, the e- and e+ can be separated, collected, and the current measured. The strength of the current is proportional to the number of BSE that hit the detector.



2.3 Operating Parameters
Magnification

An image is obtained by taking the signal from the sample and transferring it to a CRT screen. By decreasing the size of the scanned area (from which we get the signal), magnification is produced.



Mag.	Area
10x	$(1cm)^2$
100x	$(1mm)^2$
1Kx	$(100\ \mu m)^2$
10Kx	$(10\ \mu m)^2$
100Kx	$(1\ \mu m)^2$
1Mx	$(100\ nm)^2$

Magnification is determined by taking the ratio of the lengths of the scans:

Mag. = L/l

Resolution

Resolution is the ability to resolve two closely spaced points. While you may have to be at a high magnification to see small features, resolution is NOT the same as magnification.

One way to improve resolution is by reducing the size of the electron beam that strikes the sample:

$$d_{min} = 1.29C_s^{1/4}\lambda^{3/4}[7.92\ (iT/J_c)x10^9 + 1]^{3/8}$$

at low current:

$$d_{min} = 1.29C_s^{1/4}\lambda^{3/4}$$

J_c = current density of the source, λ = electron wavelength

C_s = spherical aberration, i = current, T = temperature,

Resolution

We can also improve the resolution by:

- **Increasing the strength of the condenser lens**
- **Decreasing the size of the objective aperture**
- **Decreasing the working distance (WD = the distance the sample is from the objective lens)**

Depth of Field (I)

The height over which a sample can be clearly focused is called the Depth of Field. The SEM has a large depth of field which produces the images that appear 3-dimensional in nature.

Depth of field is improved by:

- Longer working distance
- Smaller objective apertures
- Lower magnifications

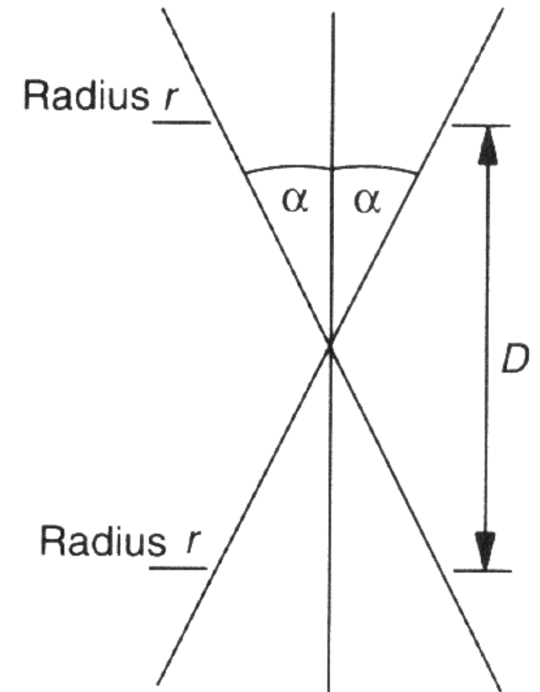
Depth of Field (II)

A beam having a semi-angle of convergence a will converge from a radius r to a focus and diverge again in a vertical distance D. For small a :

$$D = 2r / a$$

In a current high-resolution CRT's (spot size =0.1 mm = 100 microns), the focusing becomes objectionable when two pixels are fully overlapped, where the pixel size on the specimen is 0.1/M mm, where M is the magnification, which gives us the practical expression for the depth of focus/field:

$$D » 0.2 / aM \text{ (mm)}$$



Depth of Field (III)

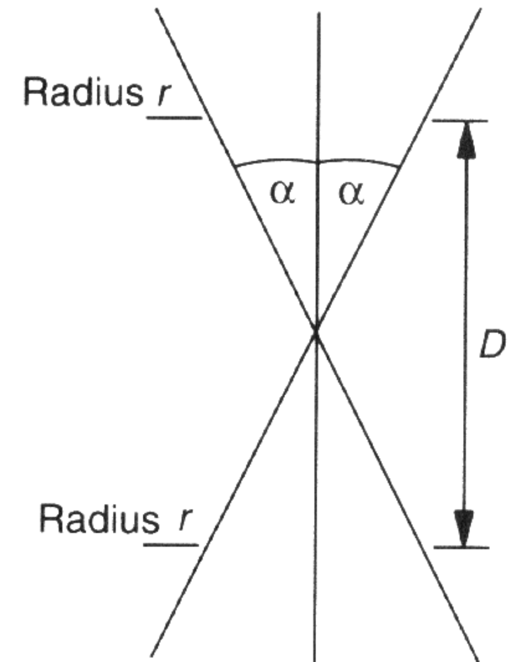
The angle a is determined by:

a = R/WD

R = radius of the aperture used, and WD is the working distance of the aperture from the specimen.

Table 34.2 **Depth of field in µm with 10-mm working distance**

Magnification	*With 100-µm aperture*	*With 400-µm aperture*
100	400	100
1000	40	10
10000	4	1
100000	0.4	0.1



Depth of Field vs. Resolution

As for the optical microscope, the depth of field and resolution have a reciprocal relationship:

• Improving resolution in conventional SEM’s leads to a smaller depth of field

• While increasing depth of field decreases resolution.useful for each particular sample.

3.0 Instrumentation

JEOL 6700F Ultra High Resolution Scanning Electron Microscope

3.1 Sample Preparation

Sample Coating

Q: Why ?

A: Charging:

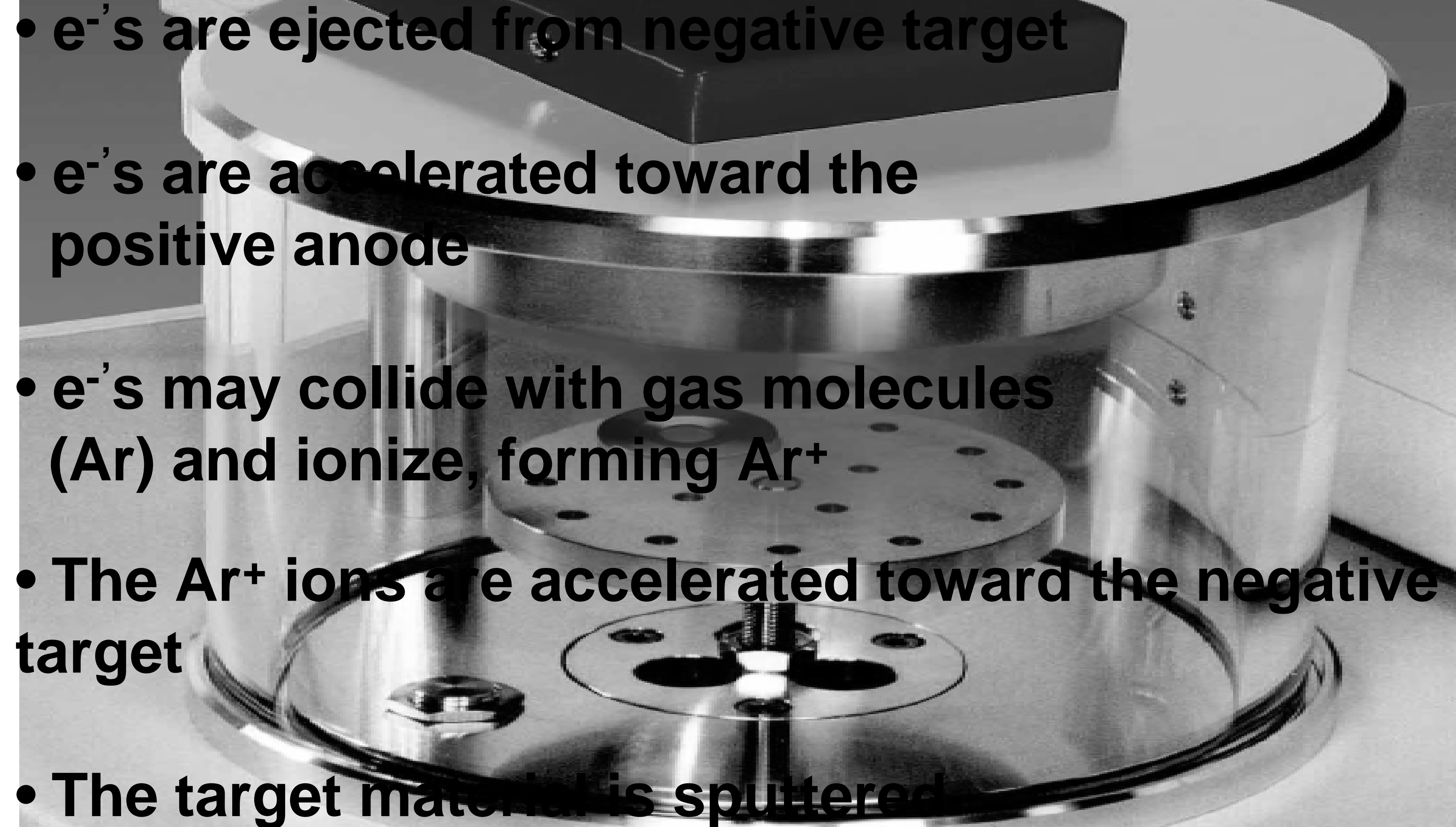
- **Deflection of SE's**
- **Increased emission of SE's in cracks**
- **Periodic SE bursts**
- **Beam deflection**

Solutions:

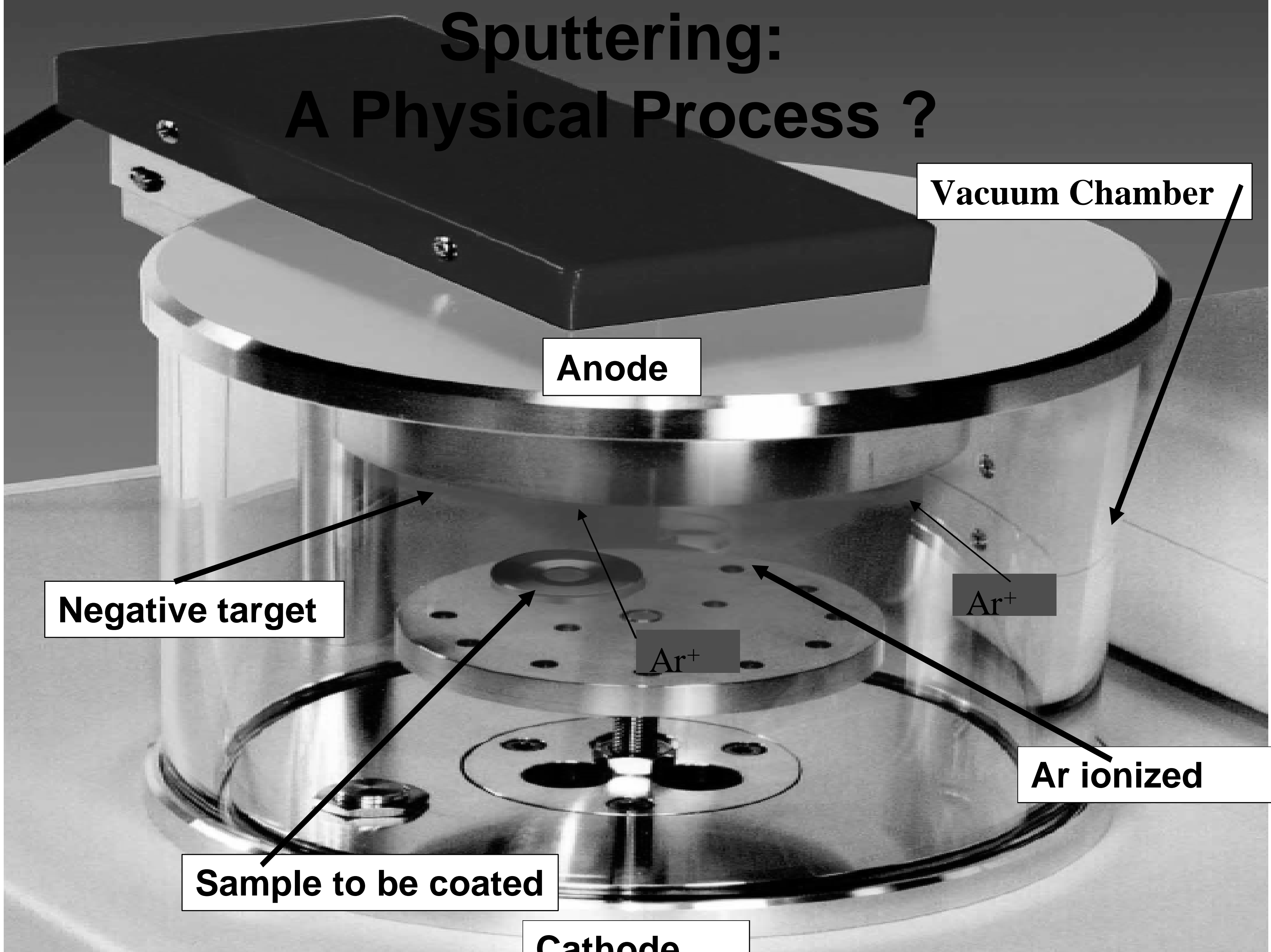
- **Sputter coating with C, Cr, or Au-Pd**

- **Carbon tape, carbon paint, In foil**

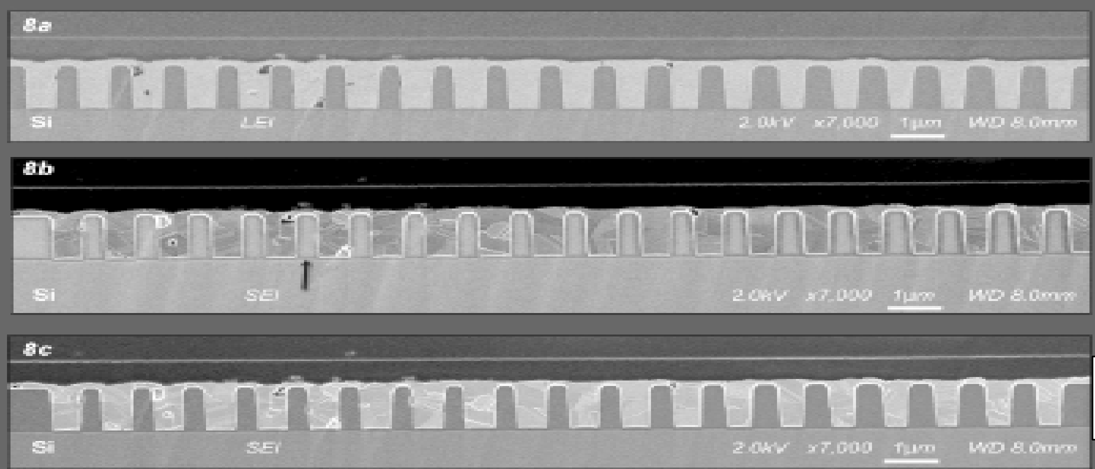
How It Works



- e^-'s are ejected from negative target
- e^-'s are accelerated toward the positive anode
- e^-'s may collide with gas molecules (Ar) and ionize, forming Ar^+
- The Ar^+ ions are accelerated toward the negative target
- The target material is sputtered


Sputtering:
A Physical Process ?
Vacuum Chamber
Anode
Negative target
Ar+
Ar+
Ar ionized
Sample to be coated
Cathode

Etching and Coating



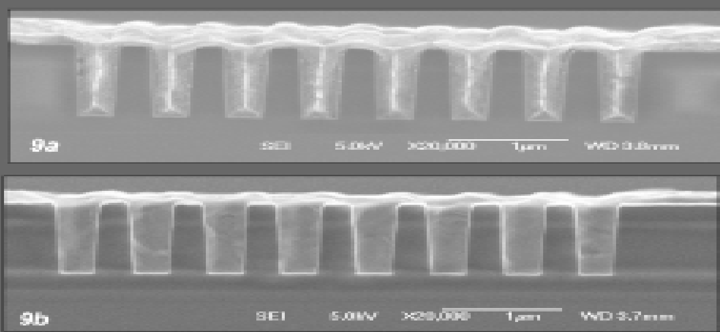
8. A sequence of SEM images taken from Cu contacts on a Si substrate after a variety of surface treatments in the PECS.

Figure 8a is taken after ion polishing and before etching. The specimen was coated with 20 Pt to prevent charging.

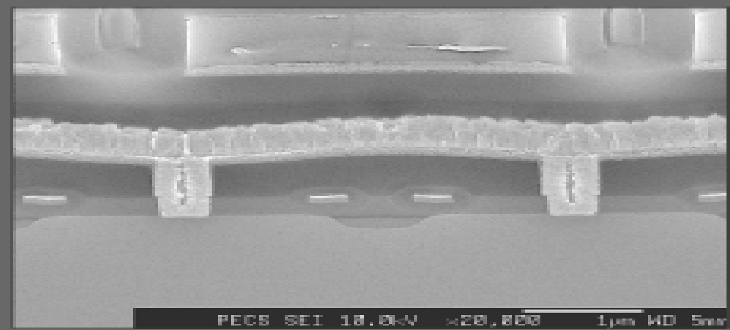
Figure 8b, as Figure 8a, but after ion beam etching only and no conductive coating. It is seen that there are some charging effects on SiO_2 areas between Cu contacts (see arrow).

Figure 8c, as Figure 8b, but after coating with ~20 coating. Note charging has been prevented.

Gatan PECS Model #682



9. Before (9a) and after (9b) etching SEM images of cleaved Cu contacts on Si substrate. Note that as all plastically deformed Cu areas are removed, more details of Cu contacts along with barrier layers are visible.



10. As cleaved IC device containing tungsten plugs and other features after etching and coating (Pt) in the PECS.

Beam Interaction Simulations

Theoretical SrS density: 3.7g/cm^3, Long. range: 1062 Å, Long. straggle: 384 Å

SrS

5000 Å

Si

2 mm

15 kV

SrS

5000 Å

Si

1 mm

10 kV

SrS

5000 Å

Si

5 kV

2 mm

SrS

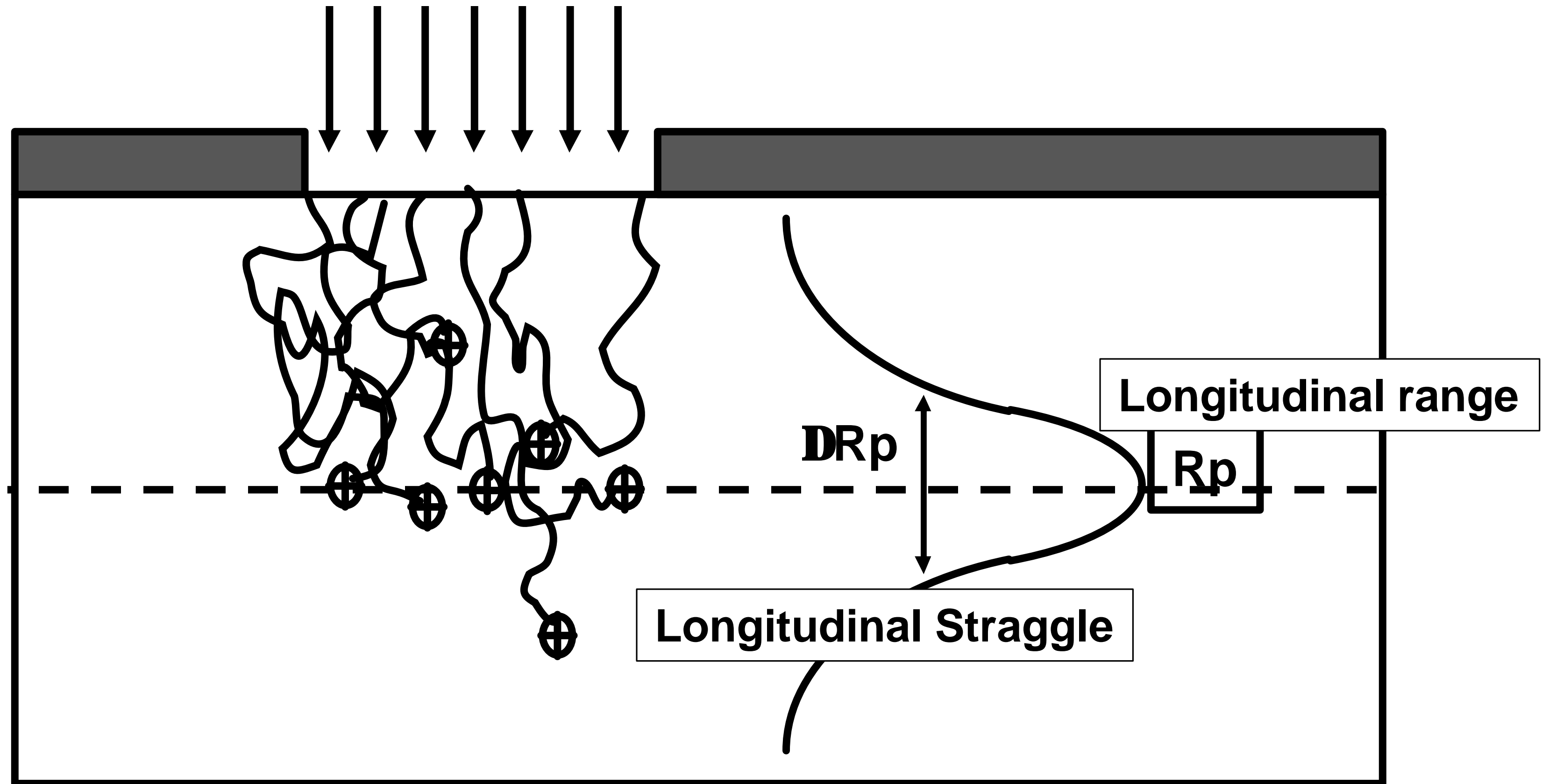
2 kV

0.5 mm

5000 Å

Random interactions with target atoms

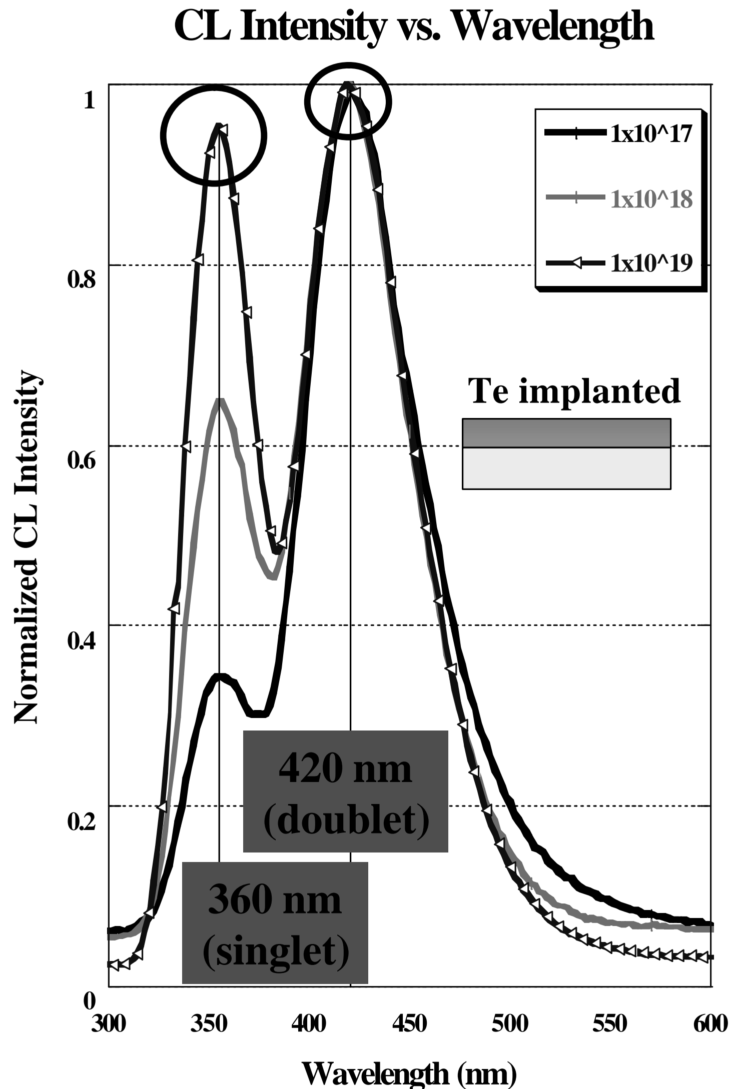
Ion, dose, energy



From LSS Theory:

$$R_p \approx \frac{R}{1+\left[\frac{M_T}{3M_i}\right]} \qquad \Delta R_p \approx \frac{2R_p}{3}\,\frac{\sqrt{M_i M_T}}{M_i+M_T}$$

Cathodoluminescence (CL)



CL Imaging at 15 kV, 10kX - 1kX
Sample: $1x10^{19} cm^{-3}$

light pipe

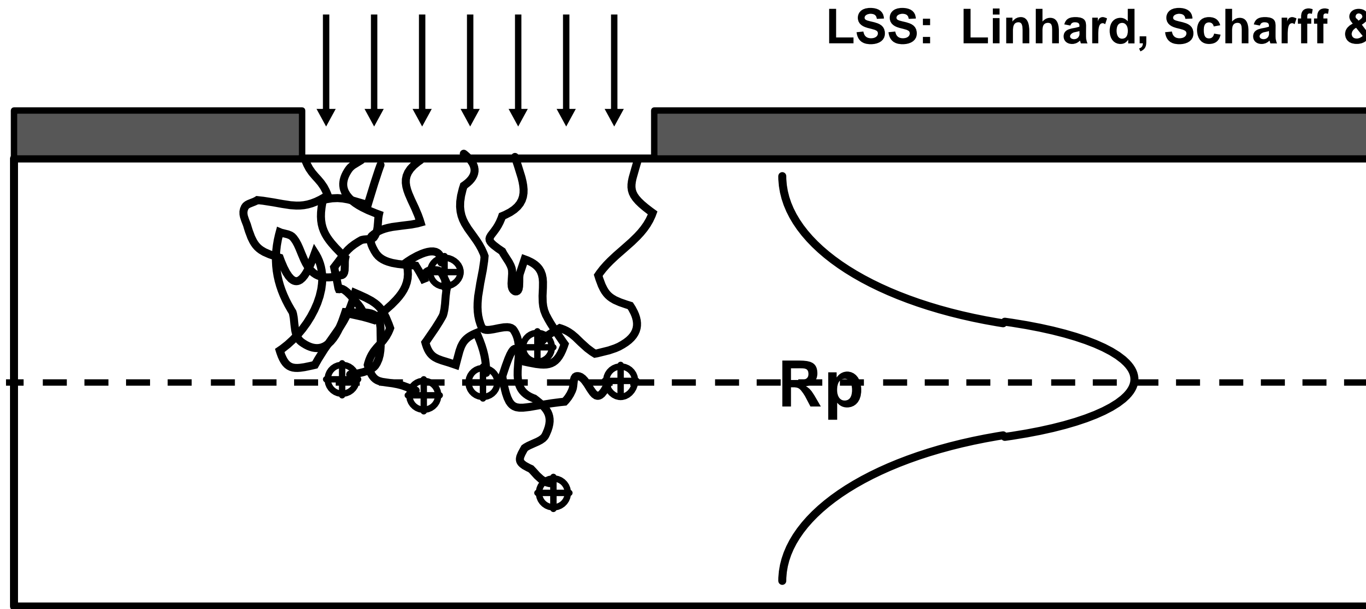
Sample

λ = 420 nm, 15 kV

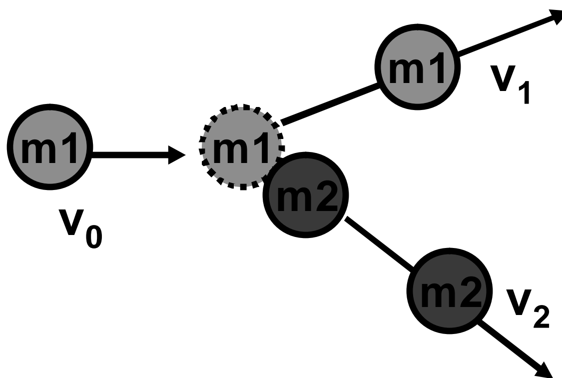
10 µm

LSS Theory of Ion Stopping

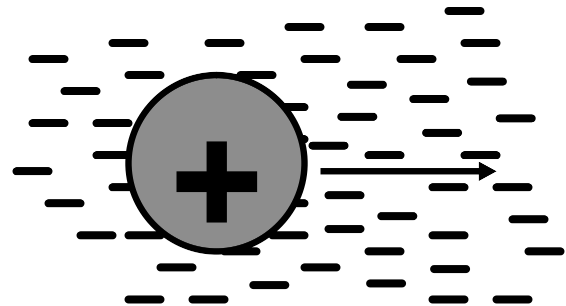
LSS: Linhard, Scharff & Schiott



Nuclear Stopping: Coulombic Scattering



Electronic Stopping



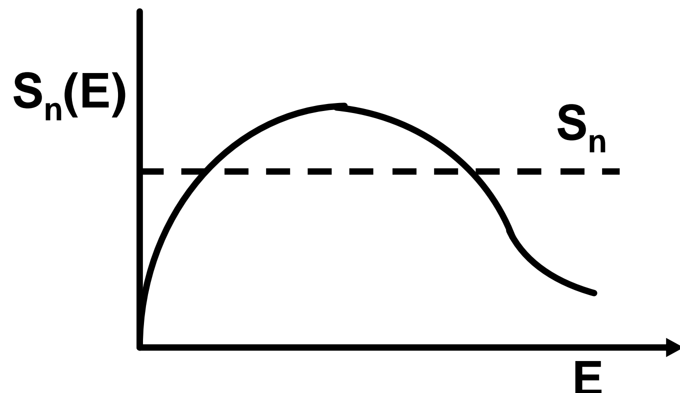
Nuclear Stopping

Energy loss per distance traveled as a function of energy:

$$S_n(E) = \left(\frac{dE}{dx}\right)_n$$

- **depends on incident ion and target atom nuclear charge (Z = # protons) and atomic masses (M)**

Energy loss due to interactions with atomic nuclei is basically a decreasing function of energy. At high kinetic energy (ion velocity) there is a very short interaction time for any absorption of energy by target atoms.



Approximate nuclear stopping near max of $S_n(E)$:

$$\frac{dE}{dx(n)} = S_n = N\frac{\pi^2}{2}e^2 a\frac{Z_1 Z_2 M_1}{M_1 + M_2}$$

where

N is the atomic density (atoms/Volume)

a ~ 1.4x10^{-2} nm, subscripts 1 and 2 refer to ion and target respectively, Z is atomic number and M is mass number

Electronic Stopping

Due to interactions with electrons in the target material

- like a drag force that is proportional to the ion velocity

Ion velocity $\propto$ (Energy)$^{1/2}$

$$S_e(E) = \left(\frac{dE}{dx}\right)_e \propto E^{1/2} = k_e E^{1/2}$$

where k_e is relatively independent of the incident ion

For silicon: $k_e \sim 10^7 (\text{ev})^{1/2}/\text{cm}$

Total rate of energy loss = $dE/dx = S_n(E) + S_e(E)$

LSS Calculations

e & r are dimensionless parameters

e a energy

r a range (R) (distance variable)

"like" dE/dx

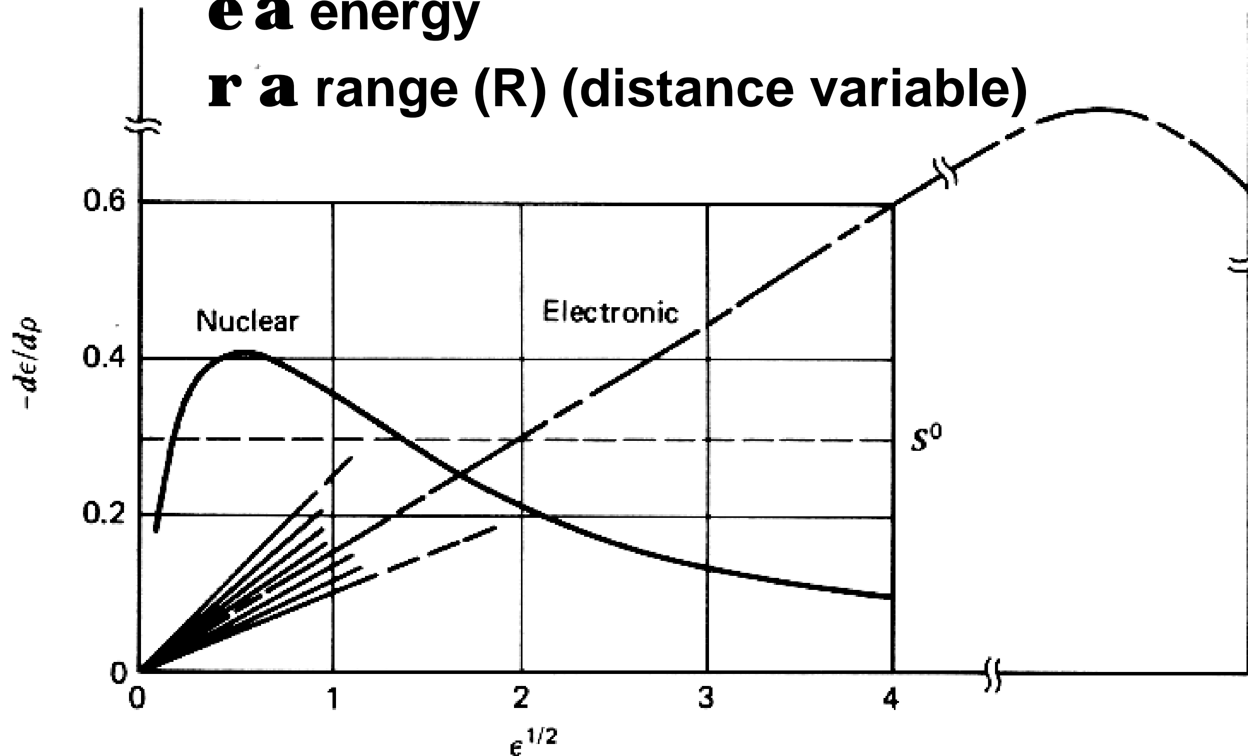


Fig. 6.8 Nuclear and electronic stopping power curves. From J. W. Mayer, L. Eriksson, and J. A. Davies, *Ion Implantation in Semiconductors* [7], 1970. Used with permission of Academic Press, Inc.

Nuclear & Electronic Stopping

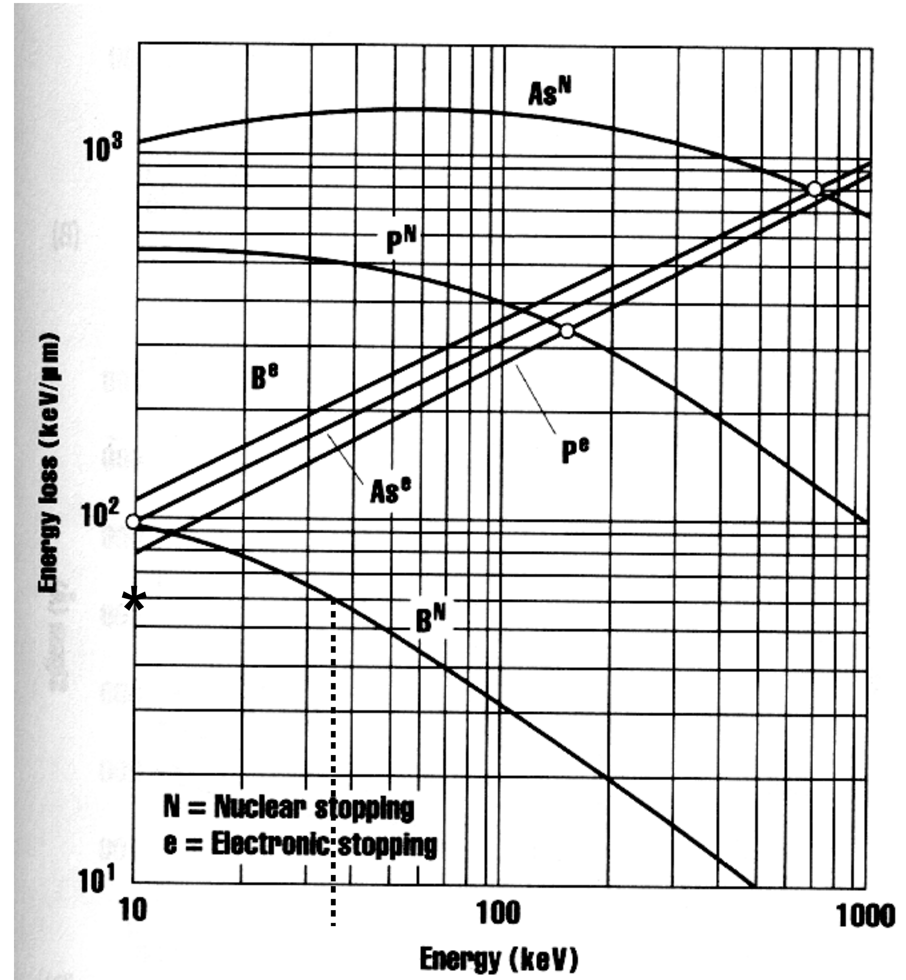
<u>Electronic stopping dominates:</u>

Light ions and high energies

<u>Nuclear stopping dominates:</u>

Heavy ions and low energies

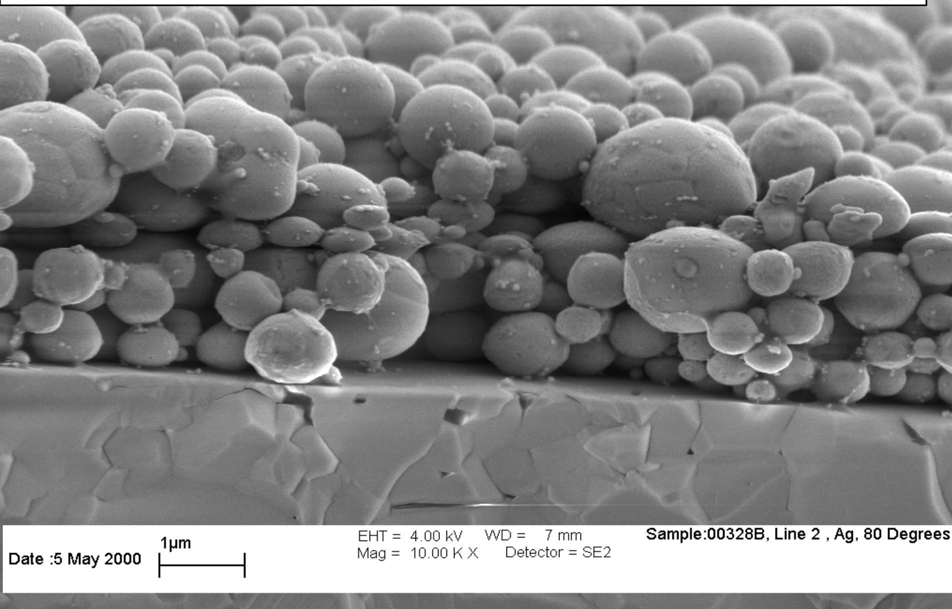
Implant damage occurs due to nuclear interactions. The extent of damage depends on $S_n(E)$.



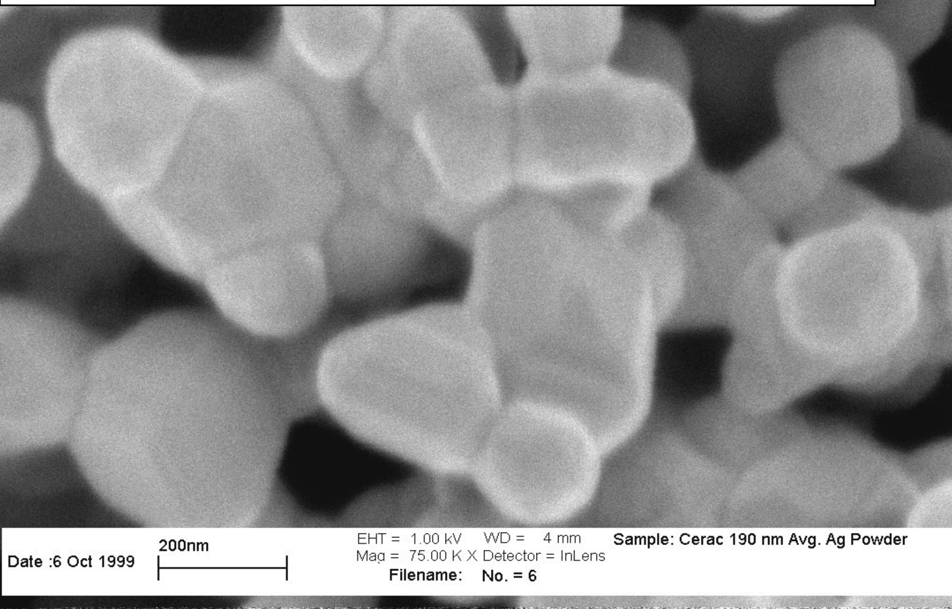
Artifacts and Examples

- Thermal damage
  - Sample temperature can rise up to 40°C
- Surface Etching
- Coating Film Adhesion
  - Not sticking, temperature sensitive
- Contamination
- Morphology Modification

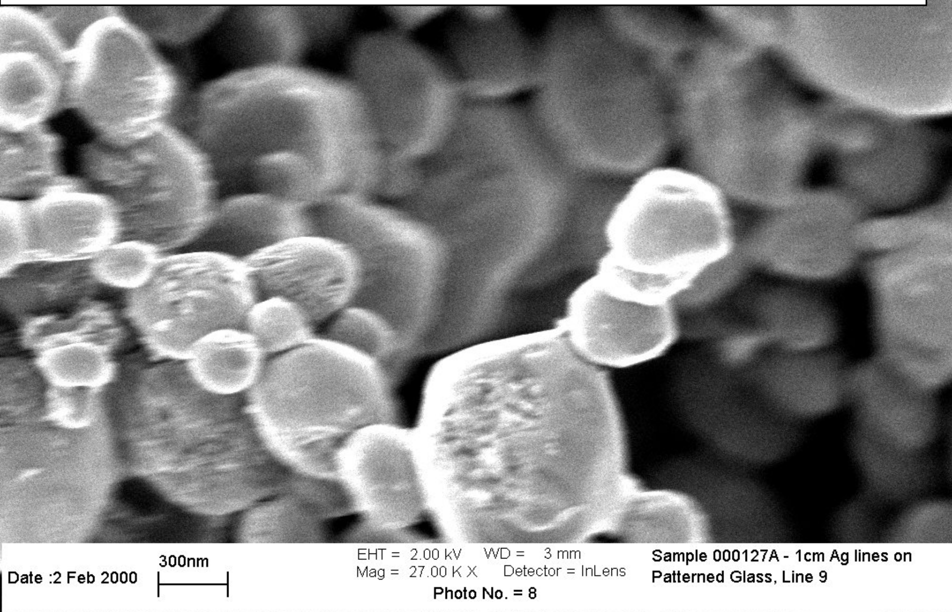
Ag powders written by a laser on glass



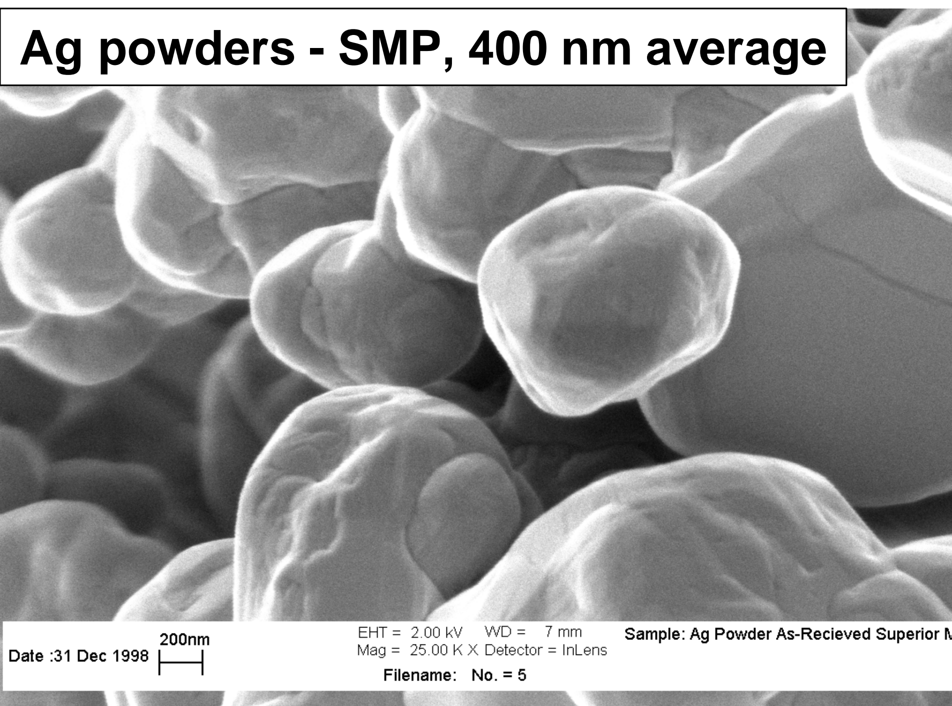
Ag powders - Cerac, 190 nm average



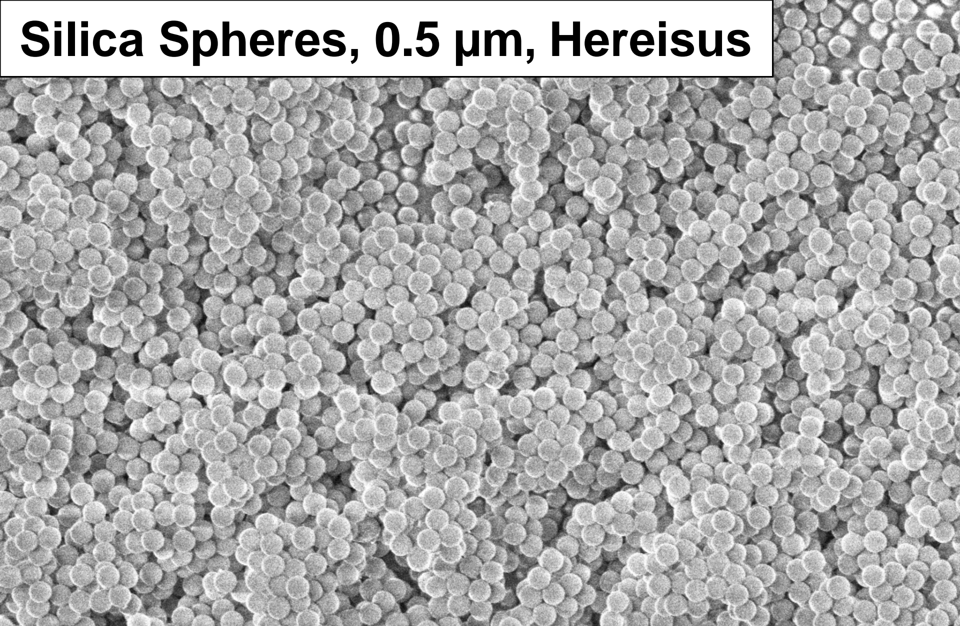
Ag powders written by a laser on glass

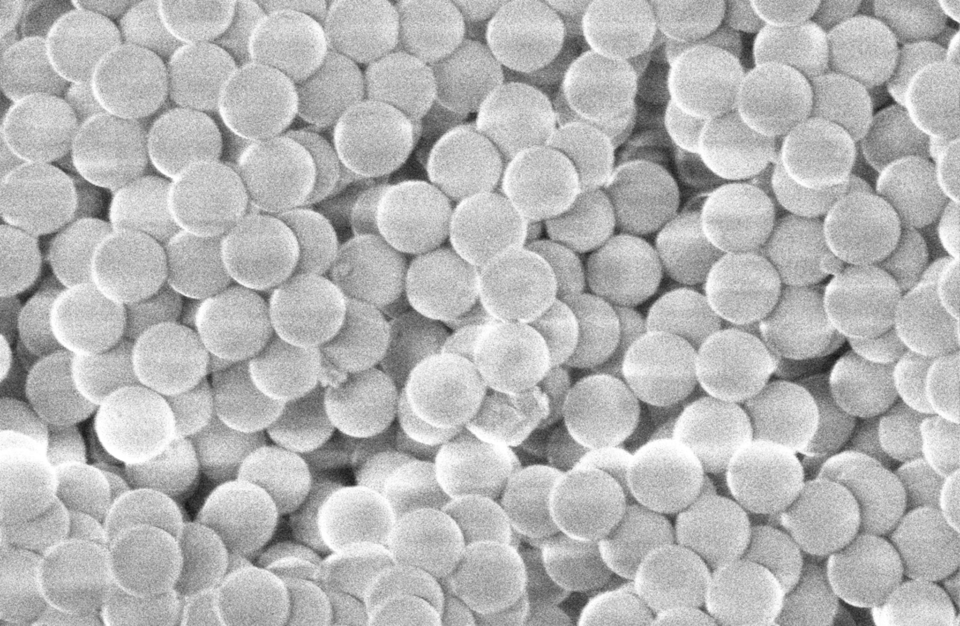


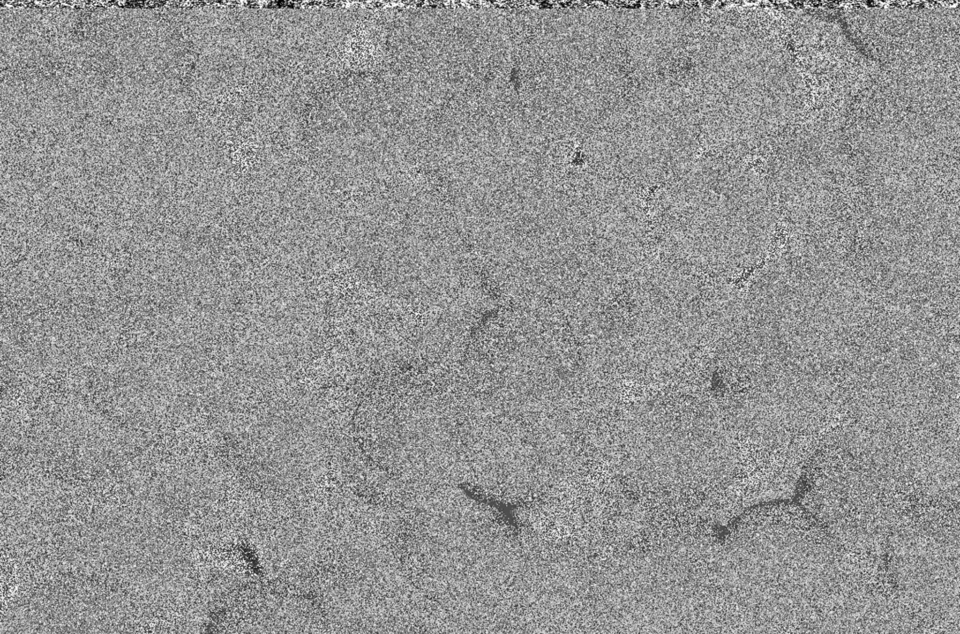
Ag powders - SMP, 400 nm average

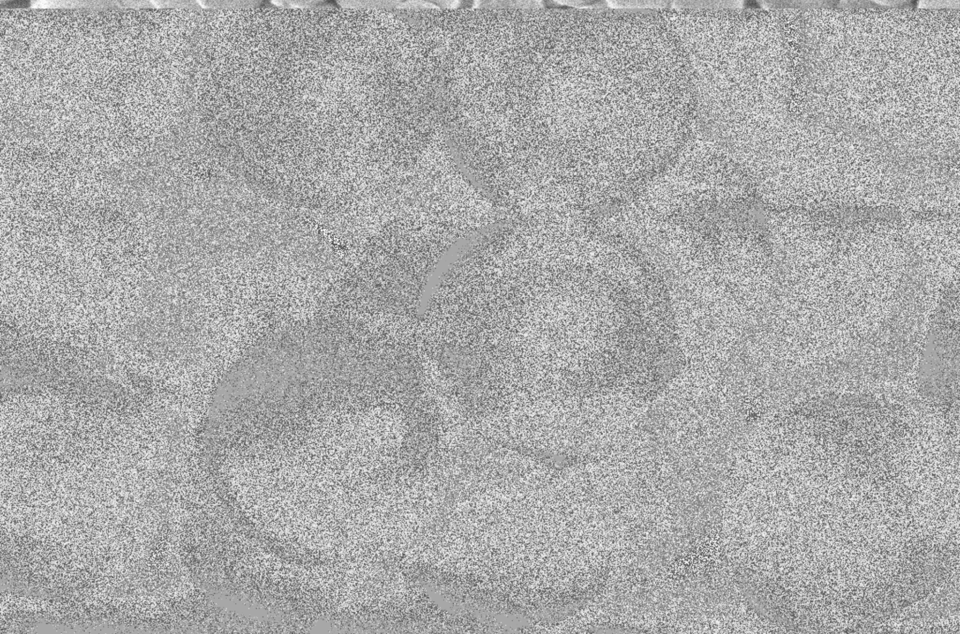


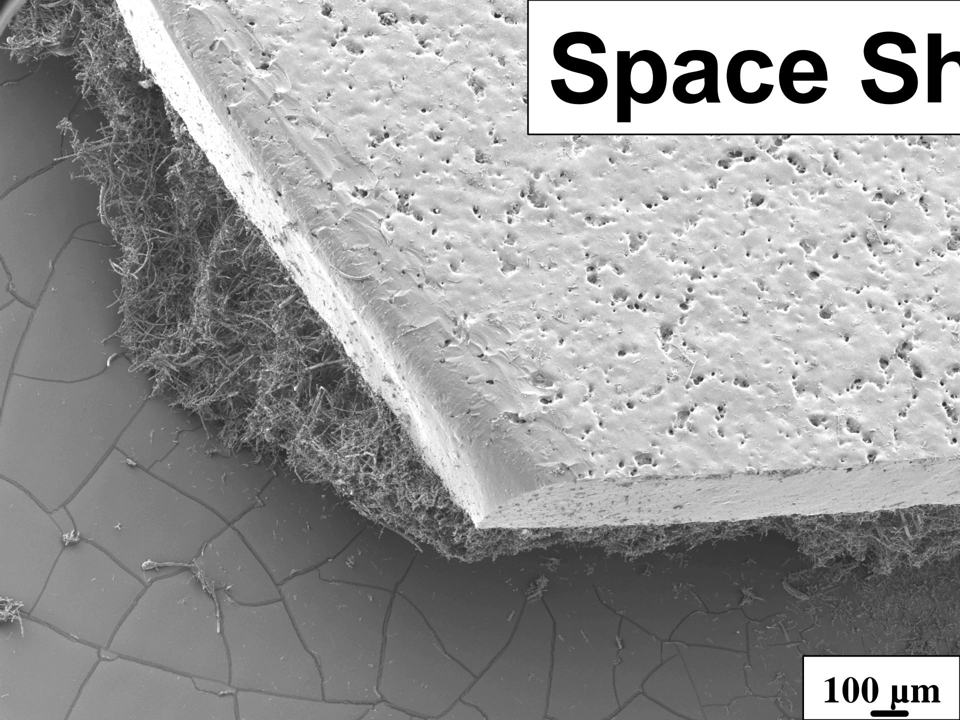
Silica Spheres, 0.5 µm, Hereisus

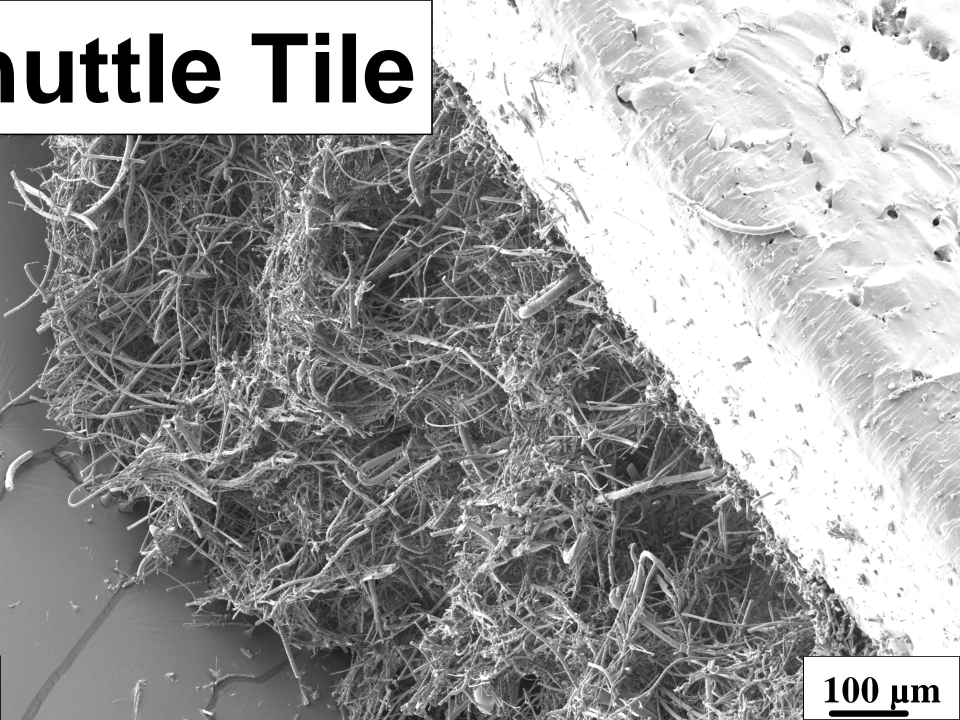


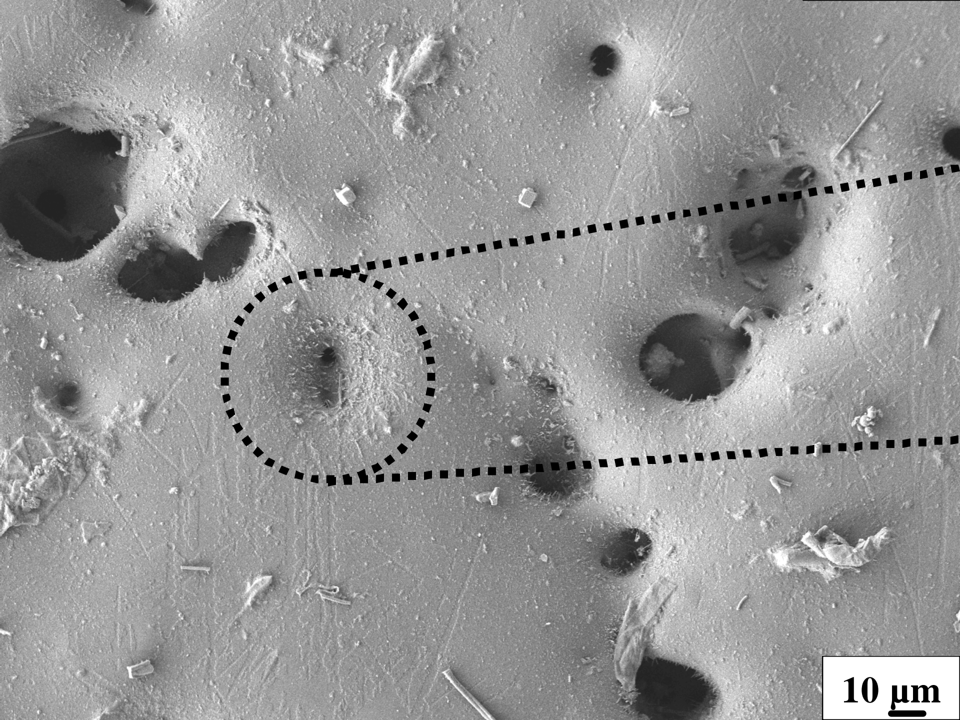


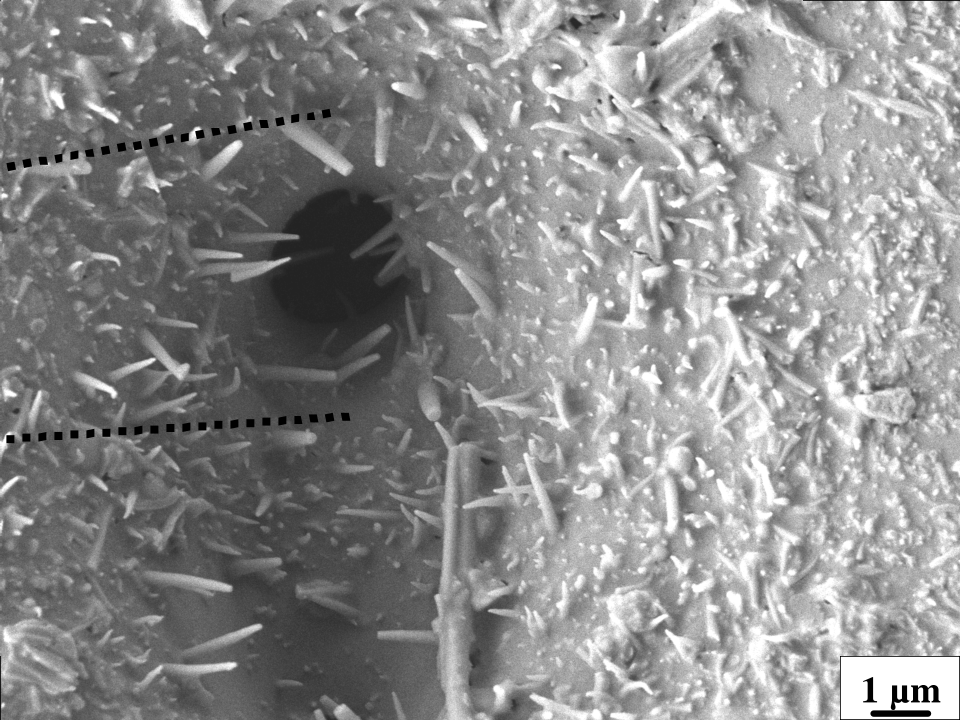




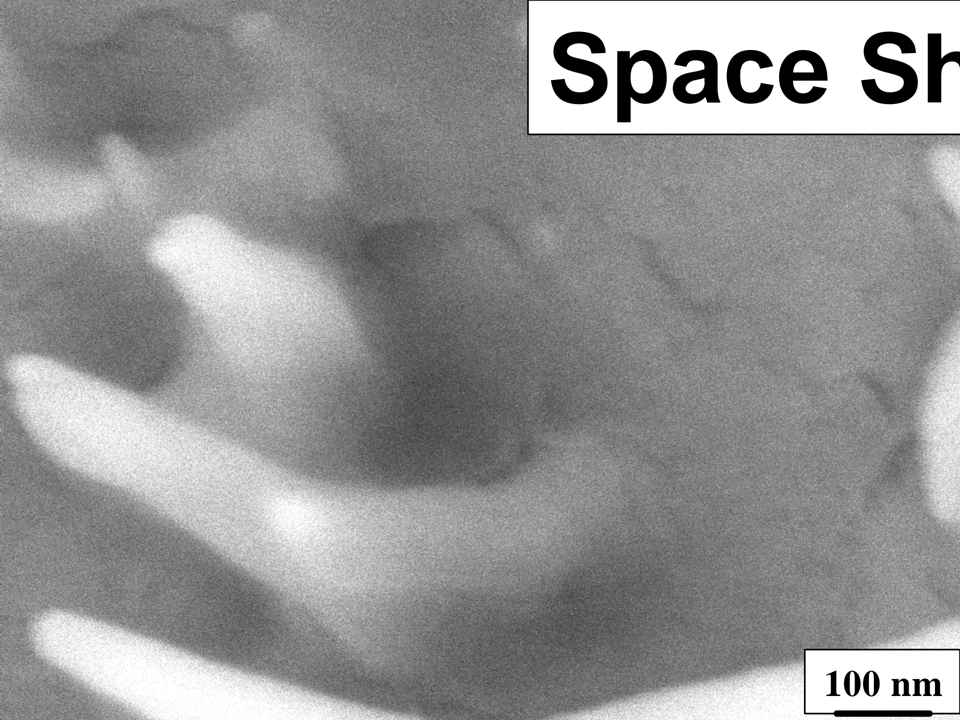

Space Shuttle Tile
100 µm

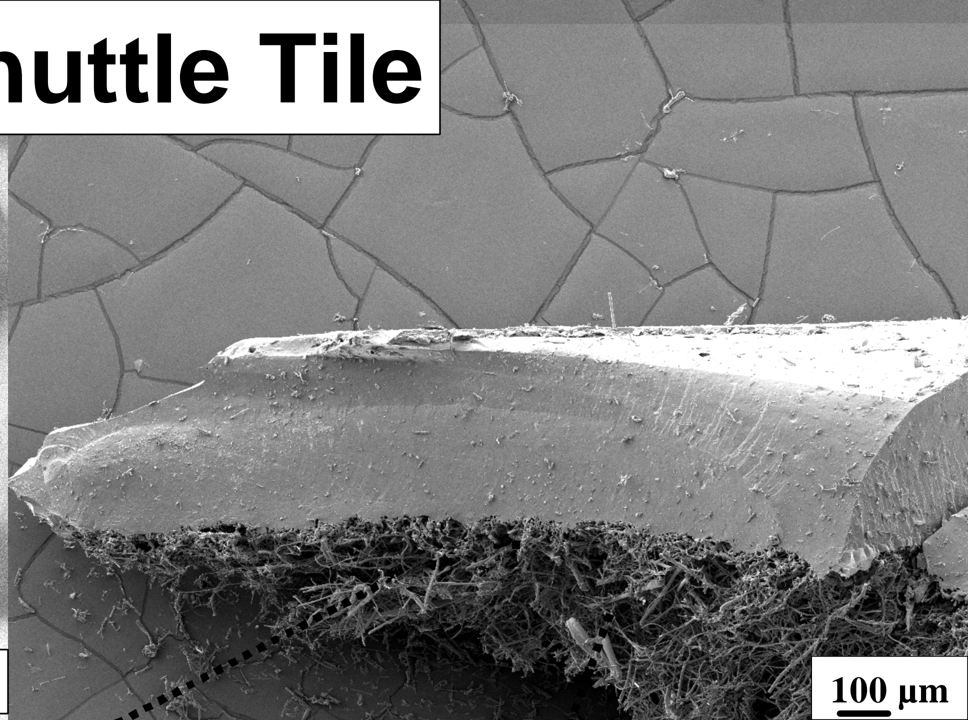

100 µm

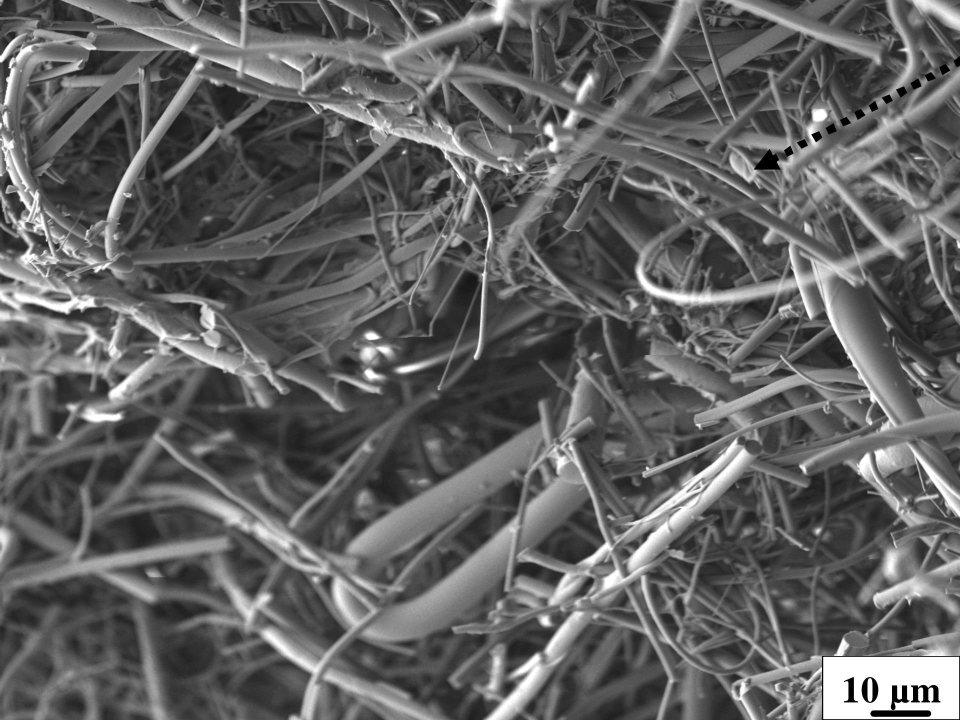

10 µm

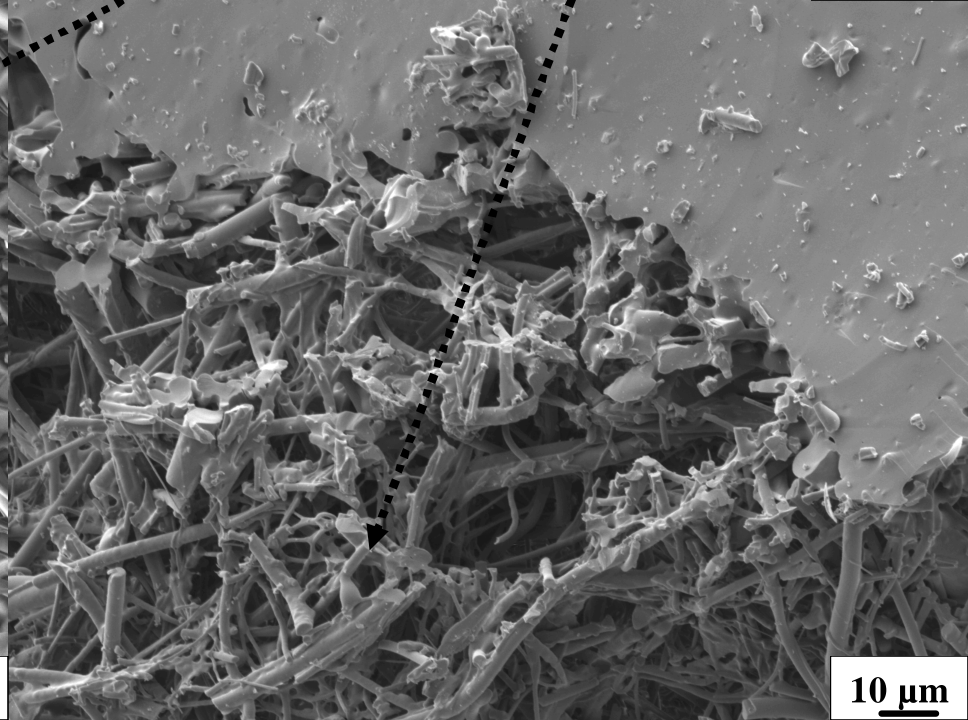

1 µm

Space Shuttle Tile

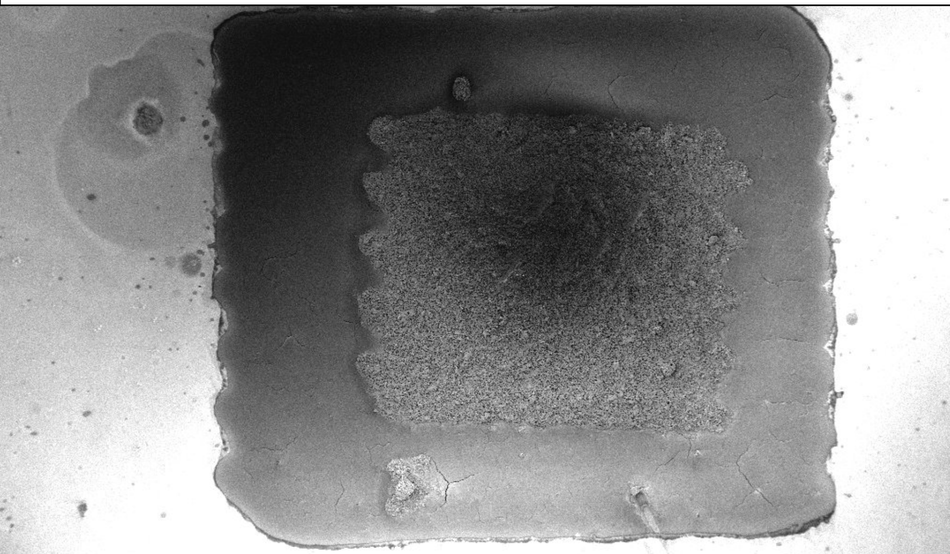


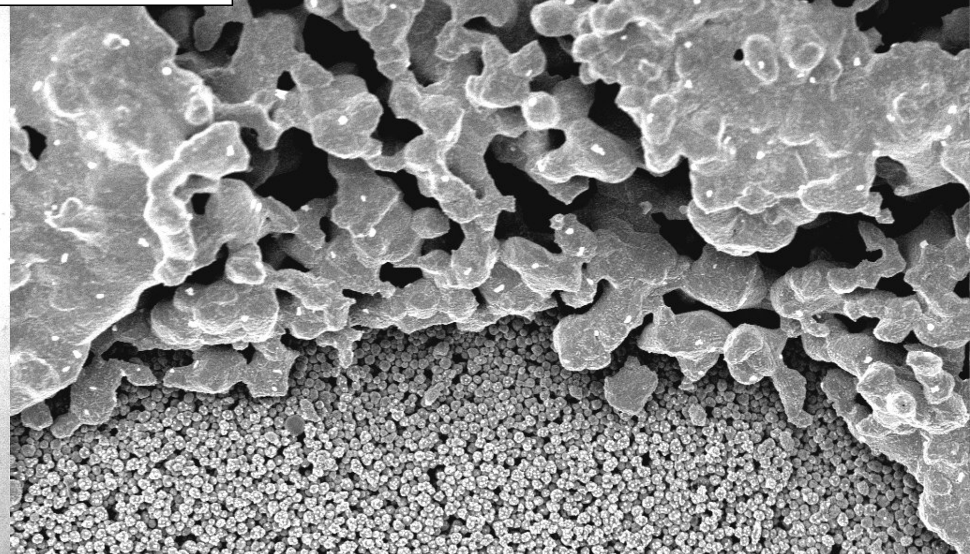


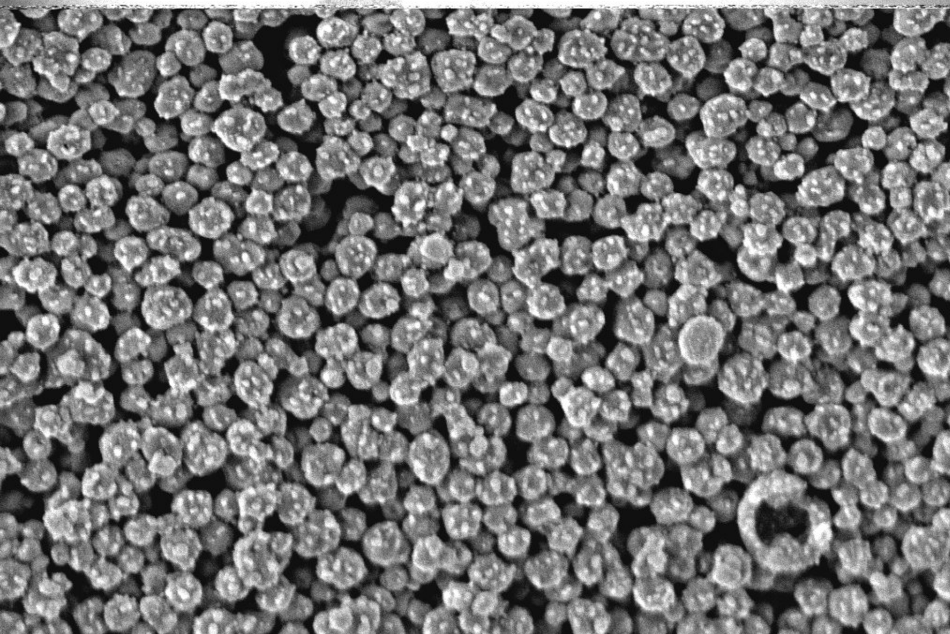


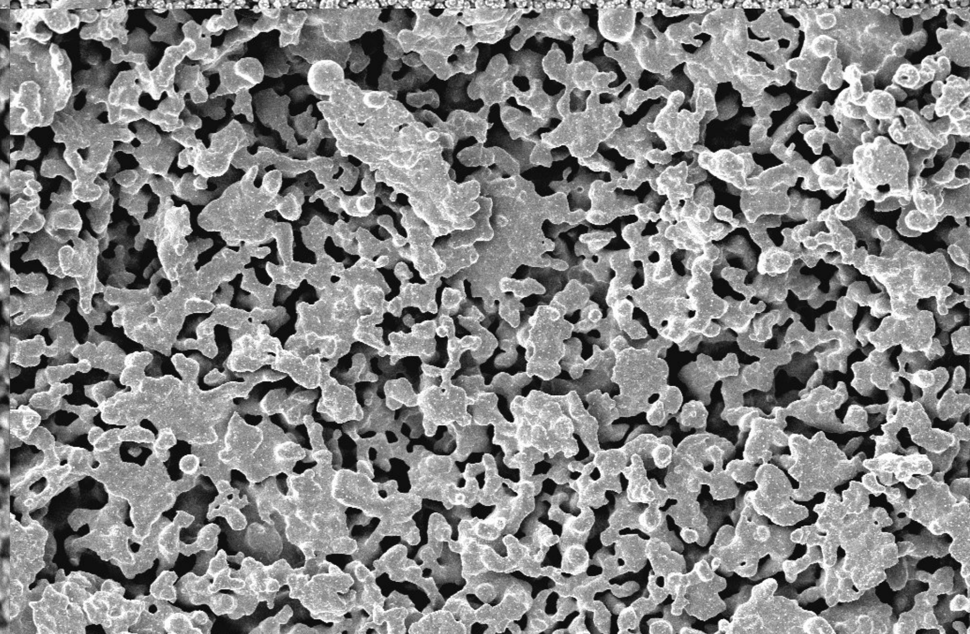


BTO and Ag powders written by a laser on Kapton









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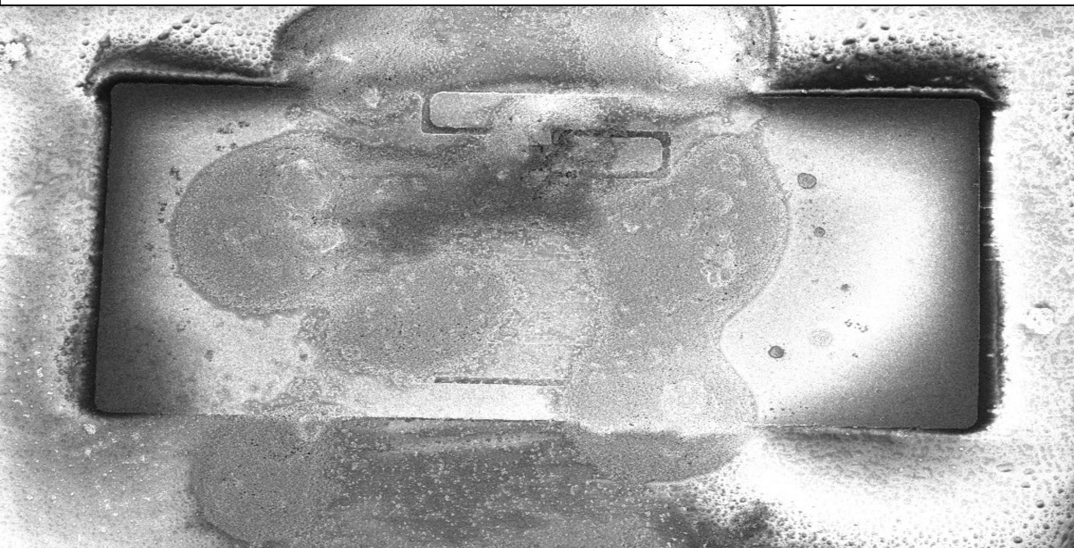
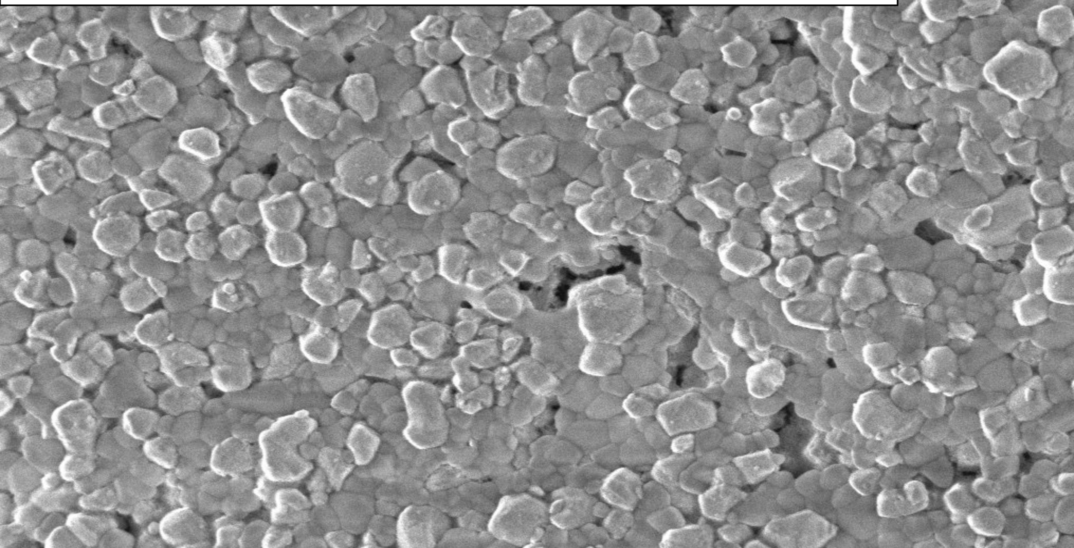
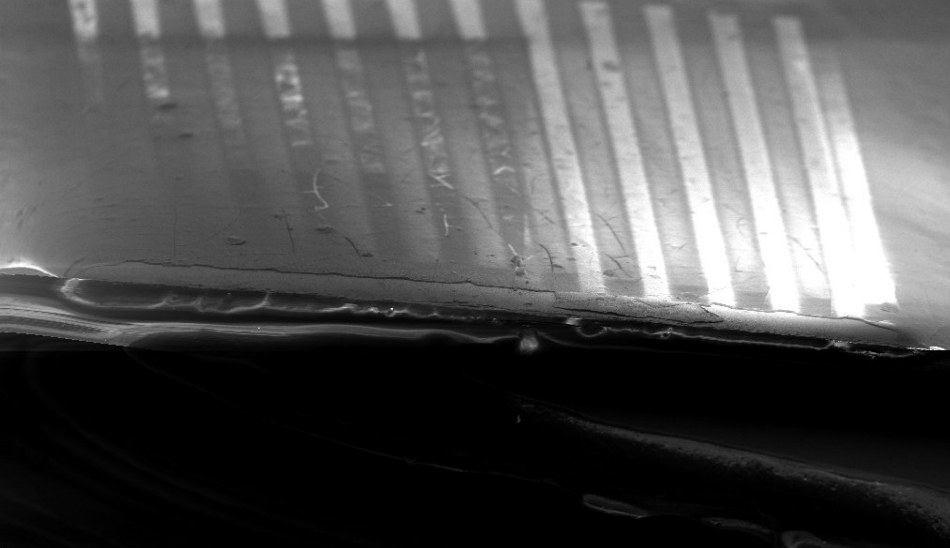
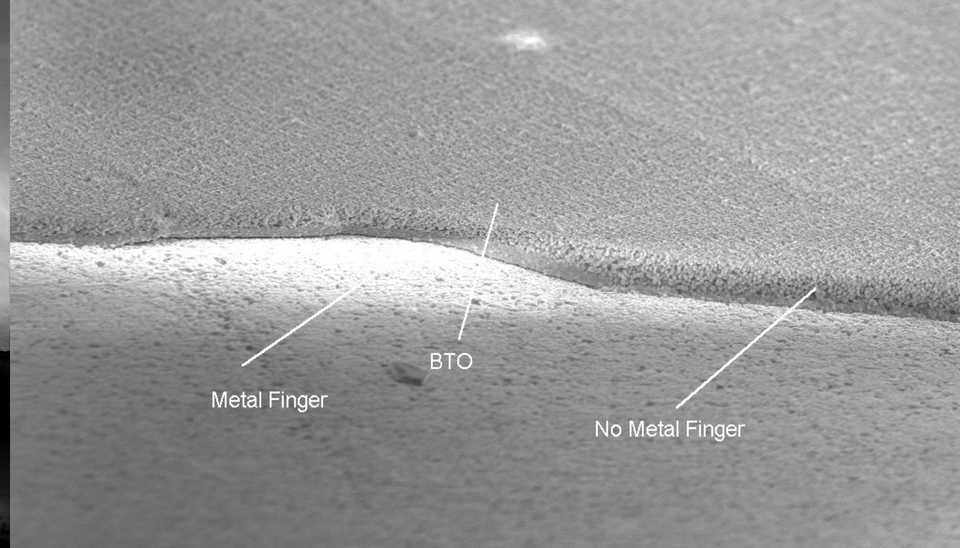


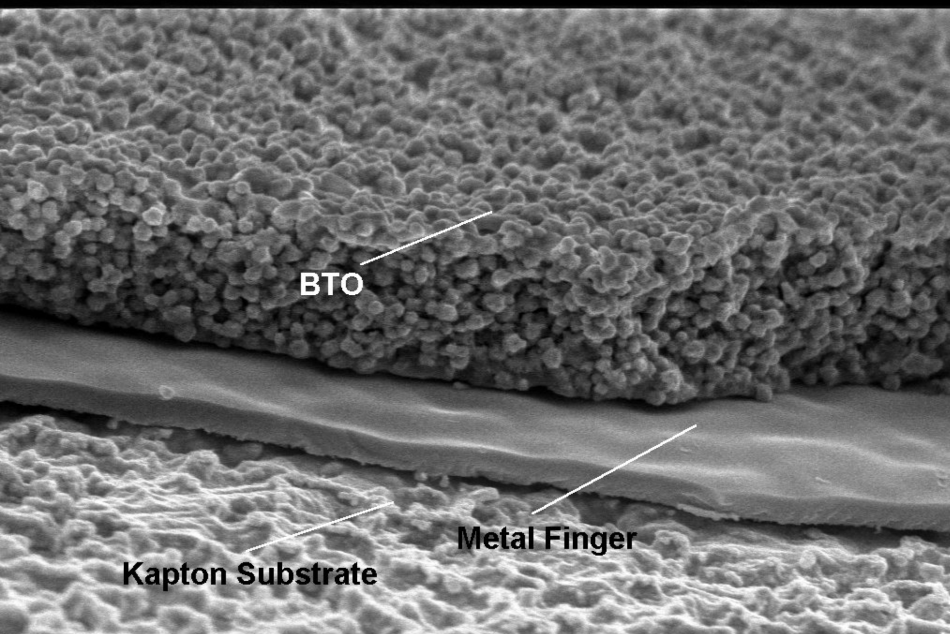
BTO sol gel matrix with BTO powders

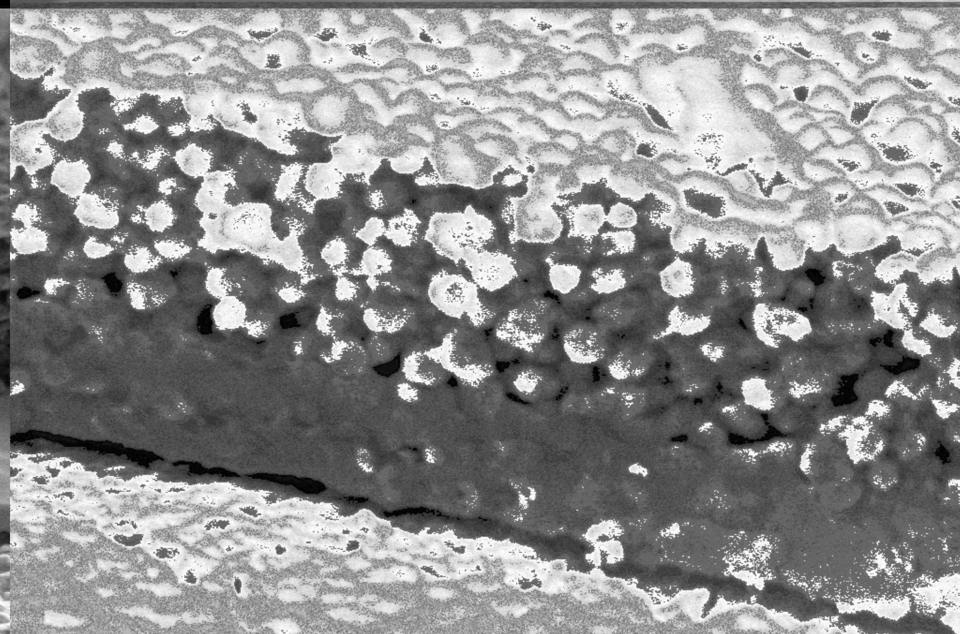


BTO and Ag powders written by a laser on Kapton









Summary

- The basic layout and operation of an SEM.
- The different imaging modes of the SEM.
- How an image is obtained
- Sample preparation
- Materials
- Vacuum levels
- Information: Imaging, EDS, WDS, EBL, beam current

Typical Thermionic Gun

A Thermionic Electron Gun functions in the following manner:

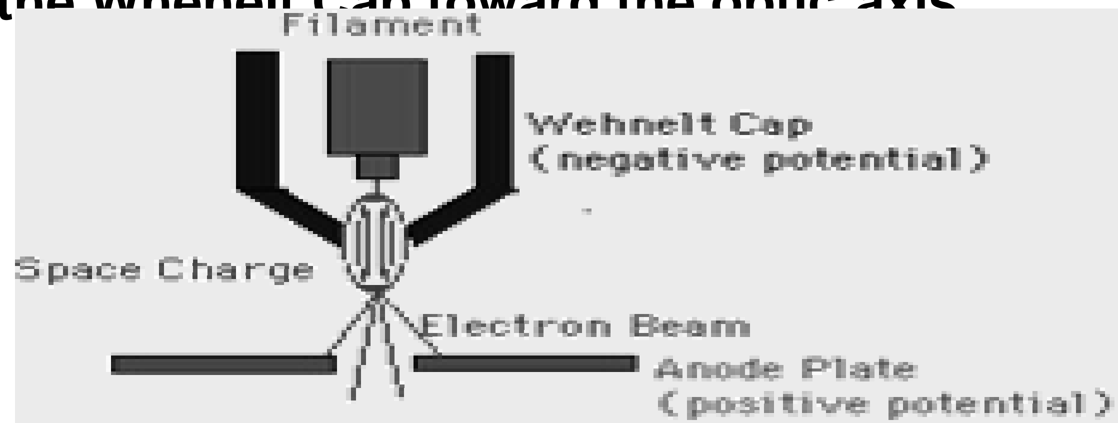
1) An positive electrical potential is applied to the anode.

2) The filament (cathode) is heated until a stream of electrons is produced.

3) The electrons are then accelerated by the positive potential down the column.

4) A negative electrical potential (~500 V) is applied to the Whenelt Cap.

5) As the electrons move toward the anode any ones emitted from the filament's side are repelled by the Whenelt Cap toward the optic axis (horizontal center).



Typical Thermionic Gun Cont'd

6. A collection of electrons occurs in the space between the filament tip and Whenelt Cap. This collection is called a space charge

7. Those electrons at the bottom of the space charge (nearest to the anode) can exit the gun area through the small (<1 mm) hole in the Whenelt Cap

8. These electrons then move down the column to be later used in imaging

This process insures several things:

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

Other Bulk Specimen Interactions

X-rays

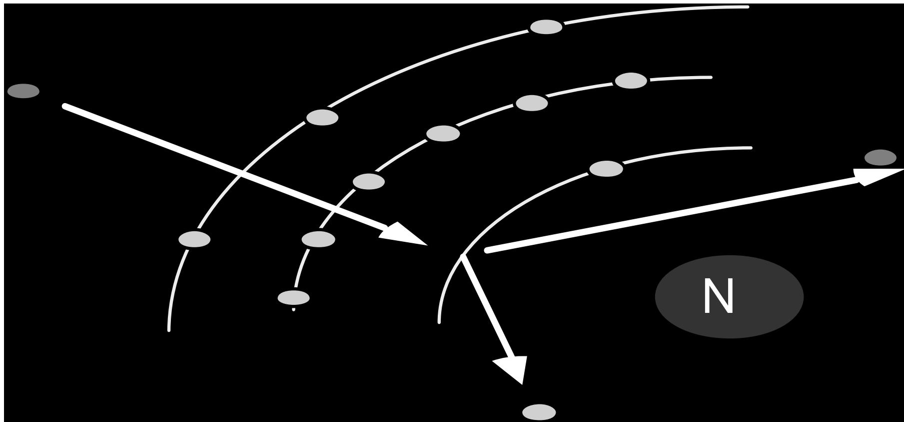
Source

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.

Utilization

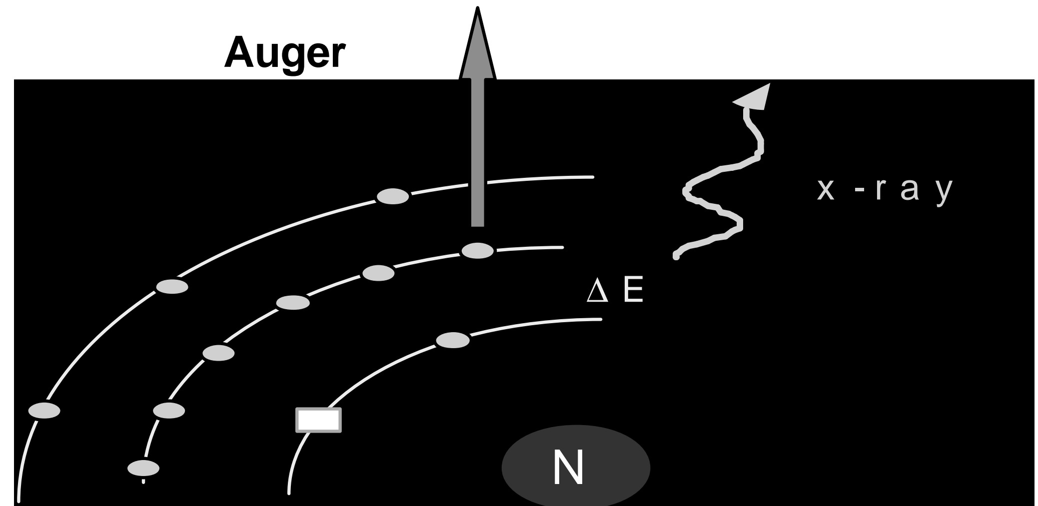
X-rays or Light emitted from the atom will have a characteristic energy which is unique to the element from which it originated. These signals are collected and sorted according to energy to yield micrometer diameter) of bulk specimens limiting the point-to-point comparisons available (see Interaction Volume).

Characteristic X-rays and Auger Electrons



We've already discussed how an electron collision can create an x-ray that is characteristic of the sample.

This same process also produces Auger electrons



Generally, in the SEM we are only concerned with the characteristic x-ray signal.

Other Bulk Specimen Interactions

Auger Electrons

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 from the same atom can "fall" to a lower energy, filling the vacancy. This creates an energy surplus in the atom which can be corrected by emitting an outer (lower energy) electron; an Auger Electron.

Utilization

Auger Electrons have a characteristic energy, unique to each element from which it was emitted from. These electrons are collected and sorted according to energy to give compositional information about the specimen. Since Auger Electrons have relatively low energy they are only emitted from the bulk specimen from a depth of <3 nm (see Interaction volume).

Thin Specimen Interactions

Unscattered Electrons

Source

• Incident electrons which are transmitted through the thin specimen without any interaction occurring inside the specimen.

Utilization

• The transmission of unscattered electrons is inversely proportional to the specimen thickness.

• Areas of the specimen that are thicker will have fewer transmitted unscattered electrons and so will appear darker.

• Conversely the thinner areas will have more transmitted and thus will appear lighter.

Thin Specimen Interactions

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.

Utilization

All electrons follow Bragg's Law and thus are scattered according to Wavelength=2*Space between the atoms in the specimen*sin (angle of scattering). All incident electrons have the same energy (thus wavelength) and enter the specimen normal to its surface. All incidents that are scattered by the same atomic spacing will be scattered by the same angle. These "similar angle" scattered electrons can be collated using magnetic lenses to form a pattern of spots; 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.

Thin Specimen Interactions

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

Utilization

Inelastically scattered electrons can be utilized two ways

•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.

•Kakuchi 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.

Characteristic X-rays

1) Core electron is knocked out

2) Outer shell electron drops down

3) Energy difference is emitted as an X-Ray

4) Electron shells have discreet energy levels

5) The X-Ray energy is characteristic of the particular element in question (energy levels)

X-Ray Detection: X-Ray intensity is μ Z

• Two types of detectors:

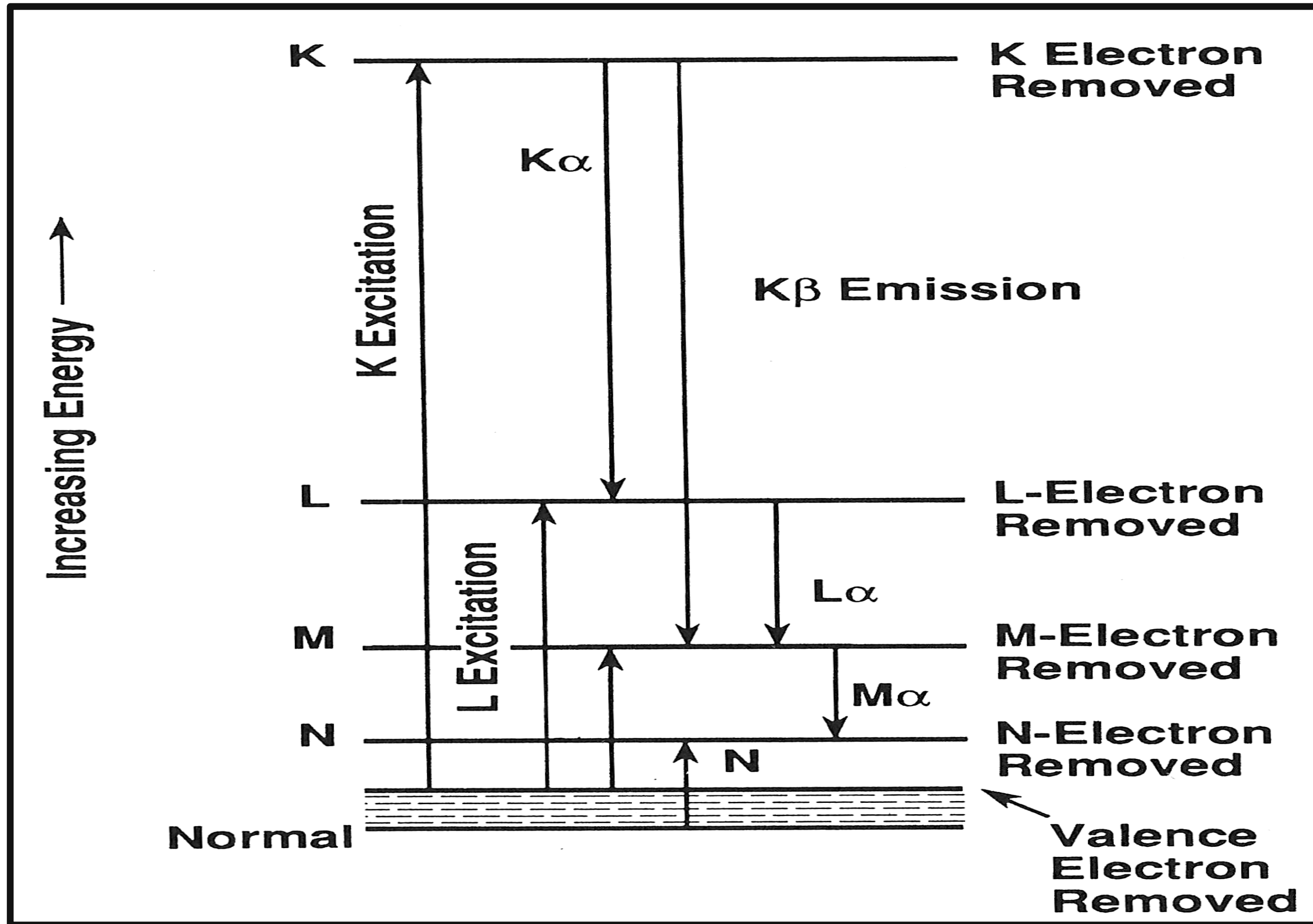
a) Wavelength dispersive:

- Separates X-Rays by wavelength

b) Energy Dispersive (EDXA, EDS, EDX):

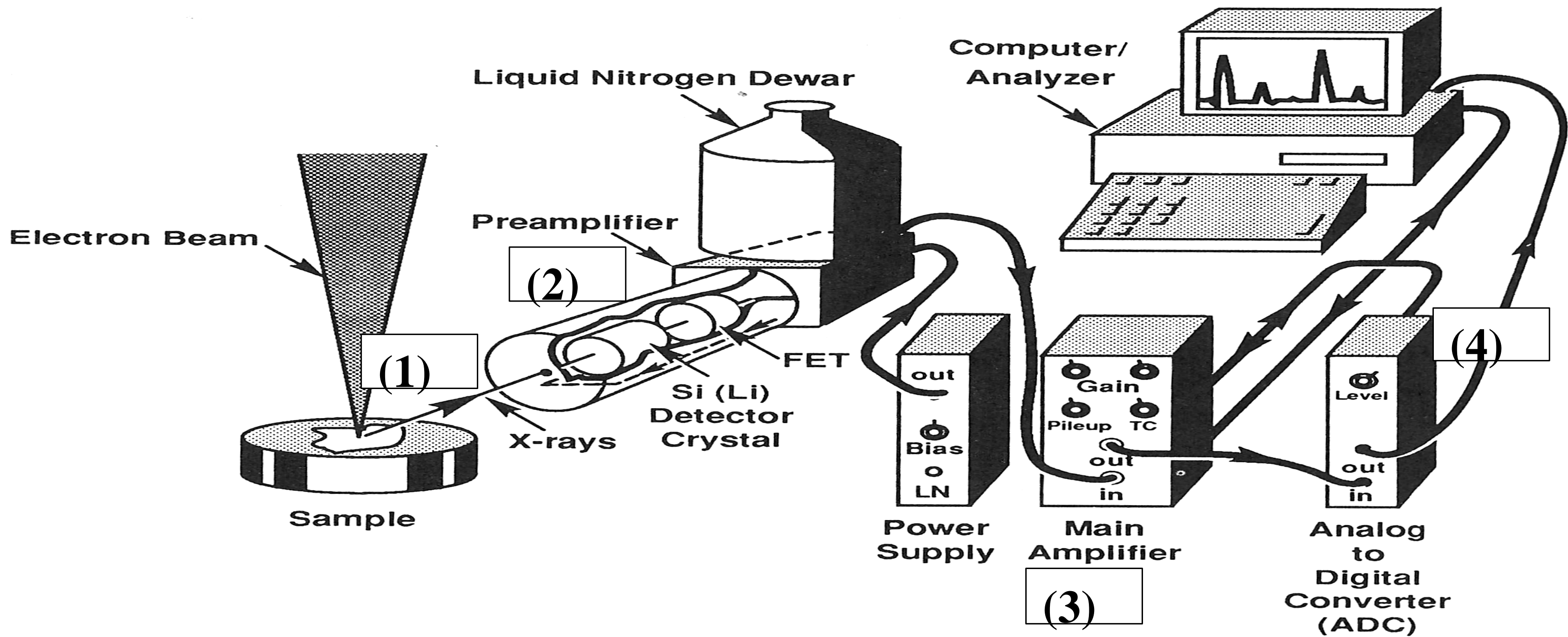
- Separates X-Rays by energy
- Can monitor one energy

Characteristic X-rays



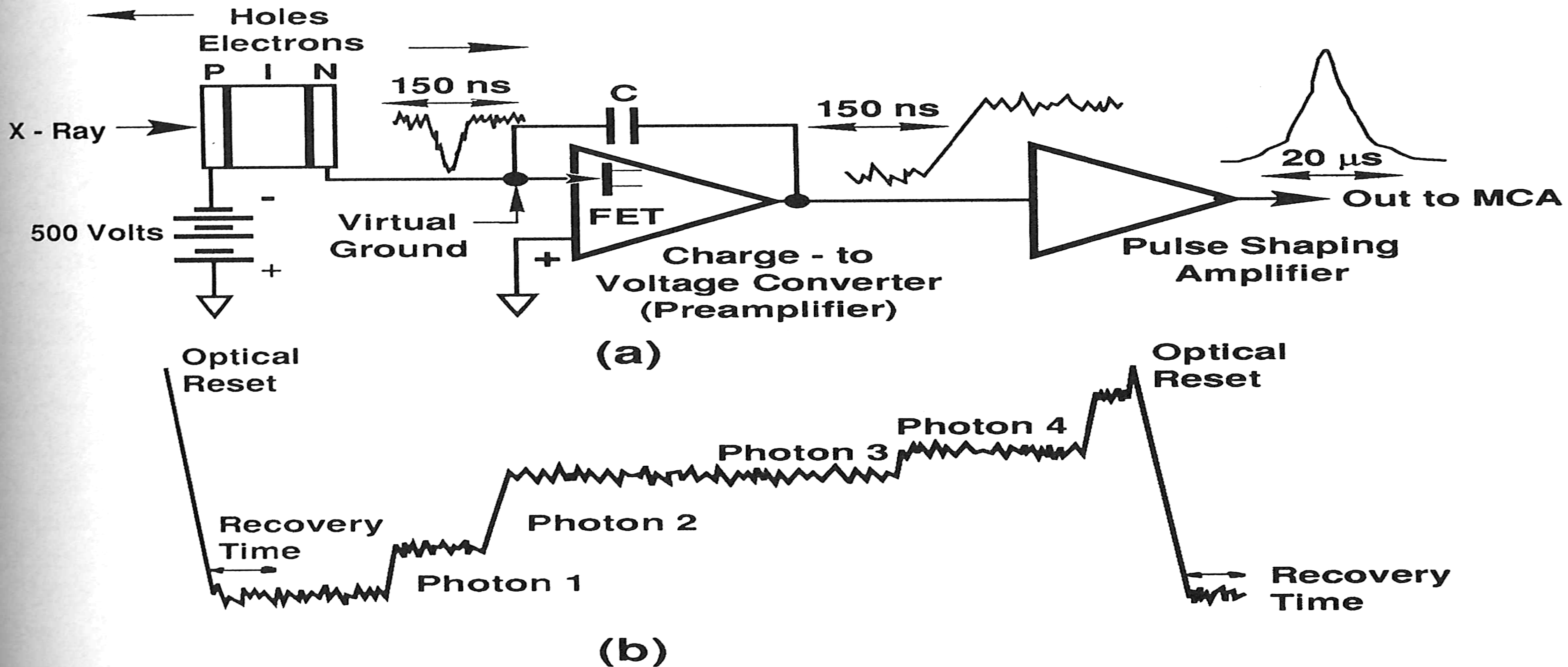
Energy-level diagram for an atom, illustrating the excitation of the K, L, M, and N shells and the formation of K_a, K_b, L_a, and M_a x-rays.

X-ray Detection I



1. **X-rays enter the detector and strike a Si crystal, creating e ⁻ / Å pairs. The number of e ⁻ / Å pairs is related to the energy of the incoming x-ray.**
2. **A potential is placed across the crystal to cause the e- to move to the + side and the Å to move to the - side, producing a charge pulse.**
3. **The charge pulse is amplified and sorted. The size of the pulse is again a function of the energy of the incoming x-ray.**
4. **The number pulses are plotted as a function of pulse size (which = energy of the x-ray).**

Charge to Voltage Conversion Process



(a) Representation of the detector charge-to-voltage converter, and pulse-shaping linear amplifier from an electronic perspective. (b) Output of the charge-to-voltage converter after the detection of a series of x-ray photons.