Lecture 13 SEM Techniques

Outlines:
- **Fundamentals of Scanning Electron Microscope**
- **Assembled Column of the Scanning Electron Microscope**
- **Electron Emission**
- **Lens Aberrations**
- **Specimen Preparation**

- **Fundamentals of Scanning Electron Microscope**

1. How does it work

TEM (Transmission Electron Microscope)

a. **Forms a true image of the specimen, as does the light microscope**
b. **Some of the image transmitted through the specimen, while some is absorbed by the specimen.**

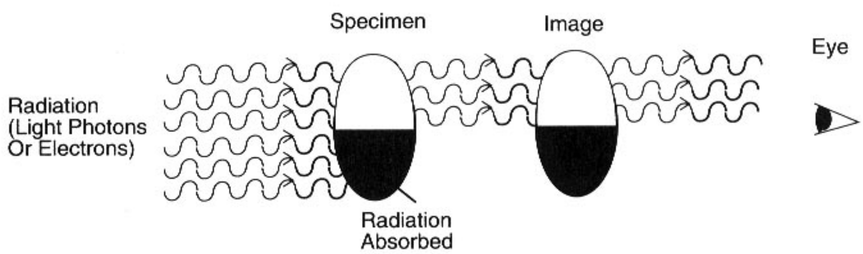


Figure 1-2 The light and electron microscope produce a true image of the specimen by selectively absorbing or transmitting light photons or electrons.

SEM (Scanning Electron Microscope)

a. **Does not produce a true image of the specimen.**
b. **Produces a point-by-point reconstruction of the specimen, similar to the TV.**
c. **Signal emitted from the specimen when it is illuminated by the high-energy electron beam (secondary electron, 5-50 nm into specimen).**
d. **The number of electrons escaping from a pixel is determined by the <u>topography of the specimen surface</u> and the <u>atomic number of the elements</u>. (Rougher surface and higher atomic number elements produce more electrons)**
e. **3-5 nanometers is the minimum diameter of the electron beam that still has enough electrons to produce an image.**

f. Depth of focus about 500 times greater than a light microscope, because of very narrow electron beam that illuminates the specimen.

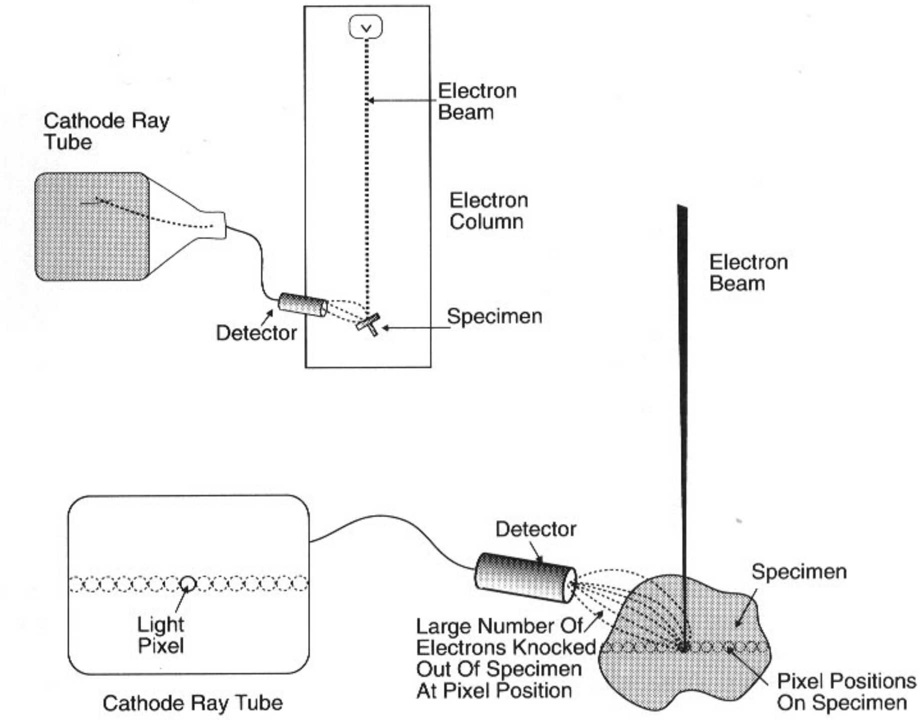


Figure 1-5 The gray level of each pixel on the viewing face of a cathode ray tube of a scanning electron microscope is determined by the number of electrons that are ejected from specimen atoms as the atoms are being illuminated by the electron beam in the column. The larger the number of electrons knocked out of the specimen atoms, the lighter is the gray level of the pixel.

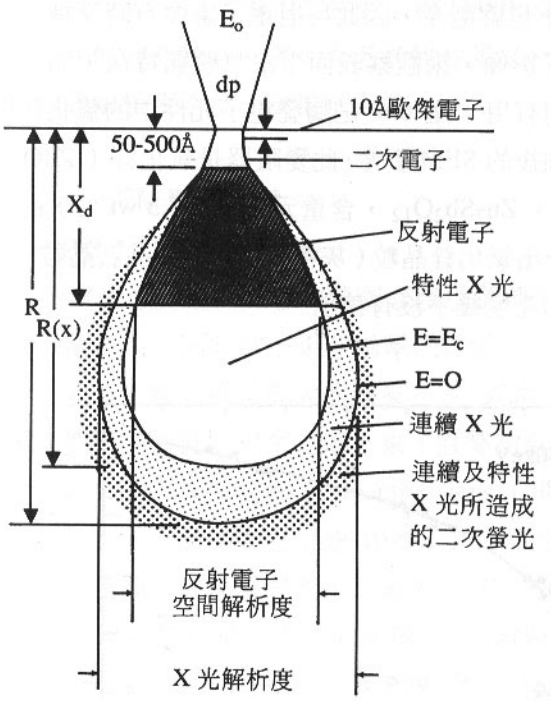


圖 6-2
電子束撞擊試片時，各種訊號產生範圍及空間解析度示意圖

2. Modern SEM

a. **Development of SEM began in 1935 by Max Knoll at the Technical University in Berlin.**

b. **Including the following sub-systems:**

Electron gun, Electron Lenses, Lens aberrations and stigmators, Detector, image process, Vacuum system, X-ray detector.

Figure 1-15 A digital scanning electron microscope, the AMRAY 1830I. (*Photograph courtesy of AMRAY*)

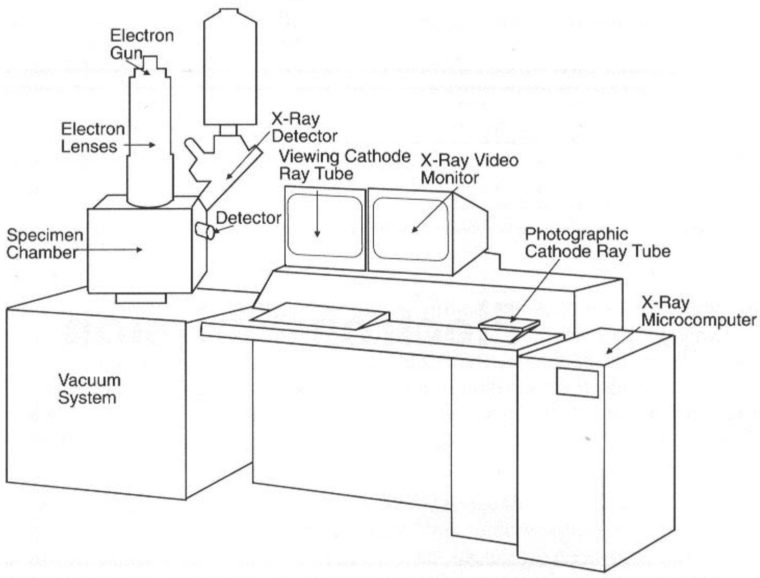


Figure 1-16 Diagram of the AMRAY 1830I showing the component parts of a scanning electron microscope fitted with X-ray microanalytical equipment.

◆ Assembled Column of the Scanning Electron Microscope

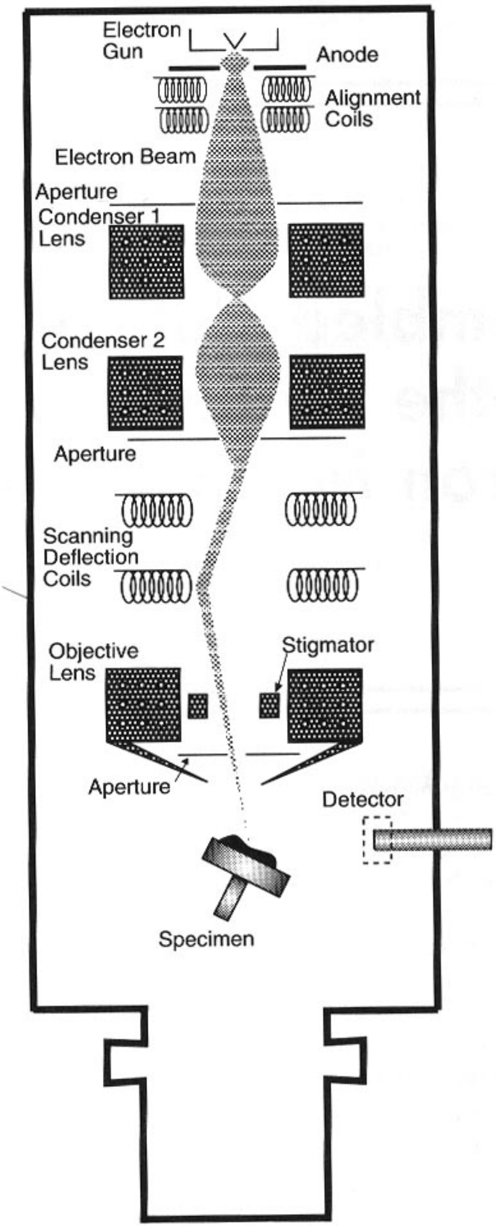


Figure 5-1 Organization of the colu
of a scanning electron microscope.

1. Electron Gun

a. **Field emission or thermionic emission (tungsten or lanthanum hexaboride).**

b. **Vacuum isolation valve under the gun.**

2. Anode

a. **Voltage varied from 0 to 30,000 V, defining the acceleration voltage of the electrons.**

b. **More electrons produce clearer image, but greater electron penetration thus worse resolution.**

c. **The higher the accelerating voltage, the less the electrons affected by the magnetic field and the further the crossover point.**

3. Beam (shift and tilt) alignment

a. **Two pairs of deflector coils are placed in the column to align the electron beam directly over the optic axis.**

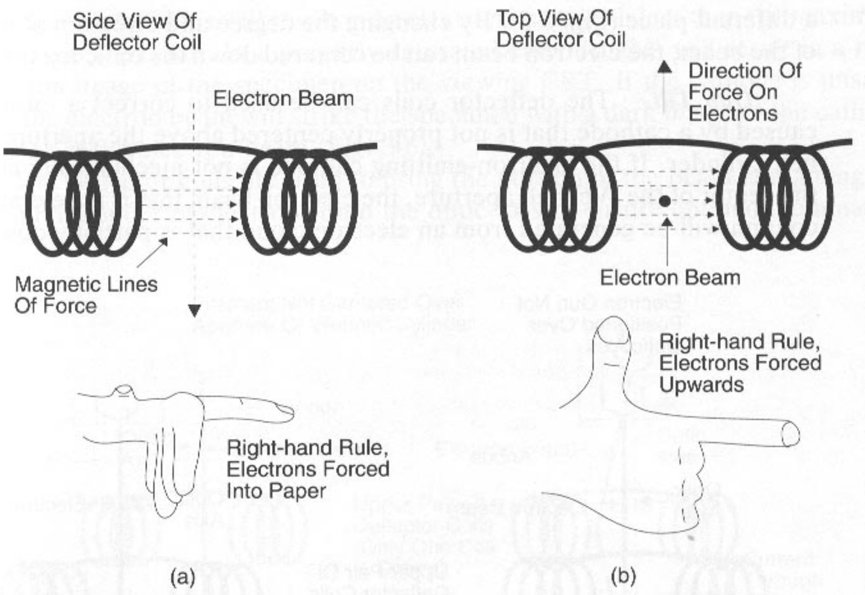


Figure 5-3 (a) Side view and (b) top view of deflector coils showing the direction of force on electrons traveling through the magnetic lines of force generated by the deflector coils. The electrons are subjected to a force perpendicular to the magnetic lines of force and the direction of the electron beam.

b. Gun shift

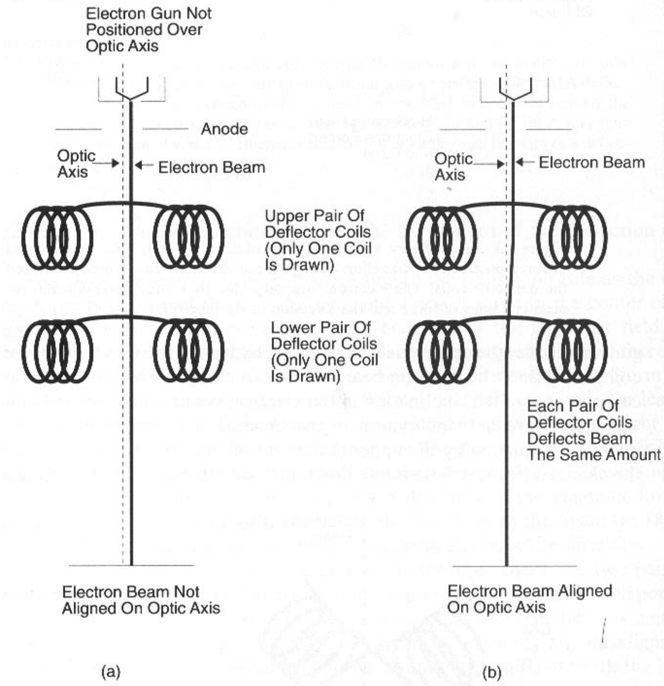


Figure 5-5 Gun shift. The set of deflector coils is used to align the electron beam with the optic axis of the column. Note that only one deflector coil of each pair is drawn. The electron gun is not precisely positioned over the optic axis. (a) Without any current through the deflector coils, the electron beam does not follow the optic axis of the column. (b) When the proper amount of current is passed through the deflector coils, the beam becomes aligned with the optic axis. The electron beam is bent the same amount by each deflector coil, so the electron beam is parallel to the optic axis.

c. Gun tilt

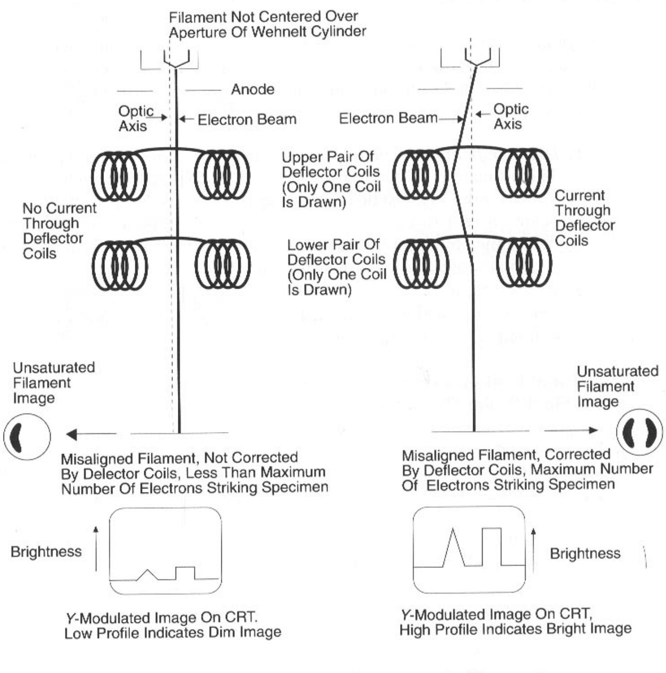


Figure 5-6 Deflector coils can be used to correct a misaligned electron beam caused by the cathode not being in the center of the Wehnelt cylinder aperture. (a) No current is passing through the deflector coils and the beam is misaligned. (b) Current is passing through the deflector coils, so the electron beam travels parallel to the optic axis after passing through the lower set of deflector coils.

4. Lenses and apertures

a. Two condenser lenses and one objective lens. Each lens usually has one aperture.

b. Lens used to demagnification electron beam and focus on specimen.

Demagnification: resolving power depends on the beam diameter=> using condenser lenses to demagnify electron beam. (Demagnify beam size from 50 μm into 0.01 nm, 500 times)

Focus: Objective lens is used to bring the electron beam to crossover at the specimen.

Working distance: short working distance produces shallow depth of focus; long working distance produces deep depth of field.

c. Design of the objective lens

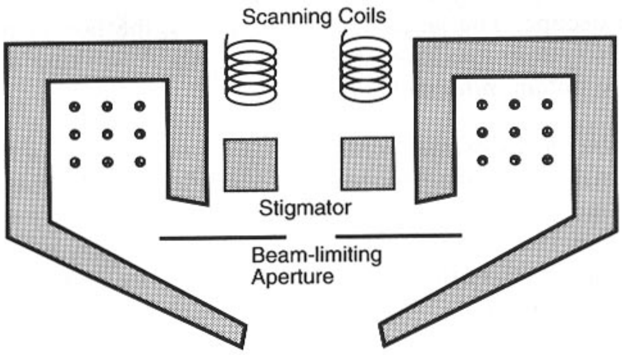


Figure 5-10 Structure of an objective (probe-forming lens) in the column of a scanning electron microscope. The outer pole piece extends inward toward the specimen.

d. Current in electron beam: The condenser lenses in combination with the aperture in the probe-forming lens determines the current in the final probe spot striking the specimen. Stronger condenser lens, greater the demagnification, and less electron current. Minimum current need for detection: 10^{-12} A.

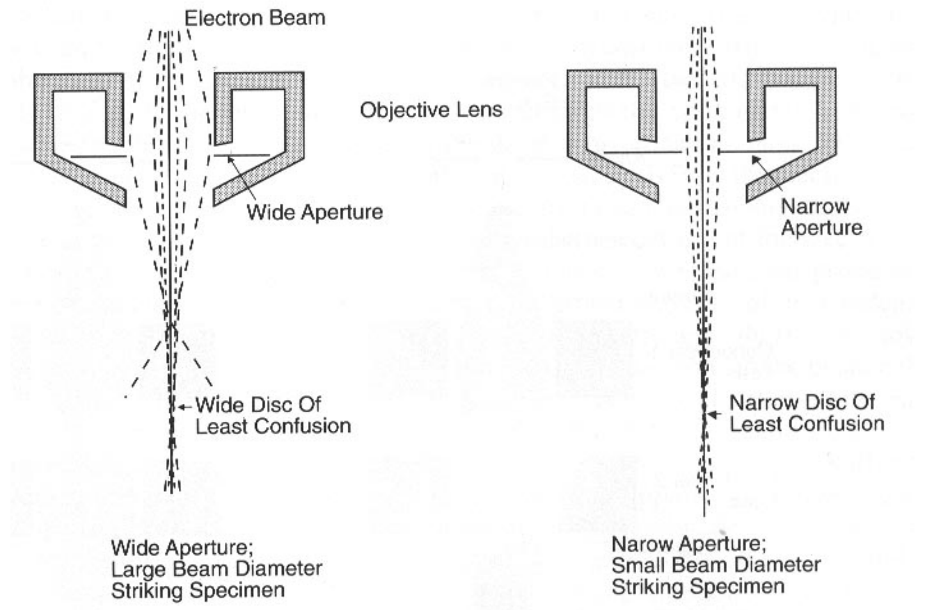


Figure 5-11 Effect of using different-sized apertures in the objective (probe-forming) lens on the size of the electron beam striking the specimen. A wide aperture results in a large beam spot because of the large disc of least confusion produced by spherical aberration. A narrow aperture results in a small beam spot because of the reduction in spherical aberration. The angles and size of the electron beam have been exaggerated for illustration purposes.

5. Focusing

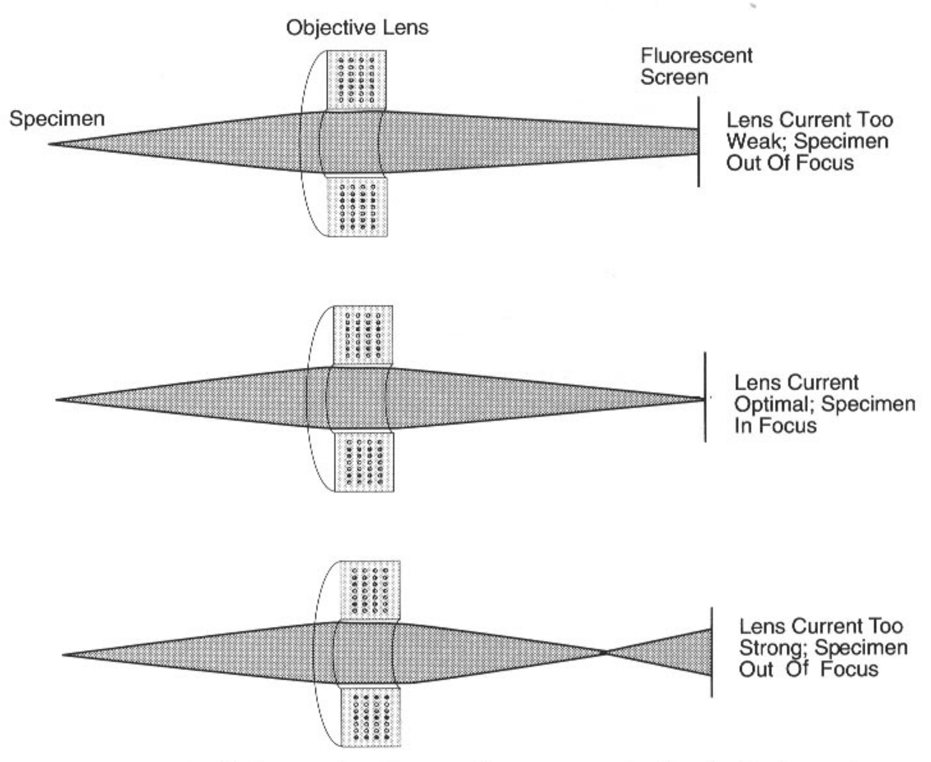


Figure 5-18 In the scanning electron microscope, varying the electrical current to the objective lens changes the magnetic field and allows the operator to bring the electron beam to crossover on the specimen. When the electron beam crosses over at the specimen, the specimen is in focus.

6. Apertures

a. **A hole in a nonmagnetic refractory metal (gold, silver, platinum, or molybdenum)**
b. **Two types: thick aperture strips or discs and thin foil apertures**

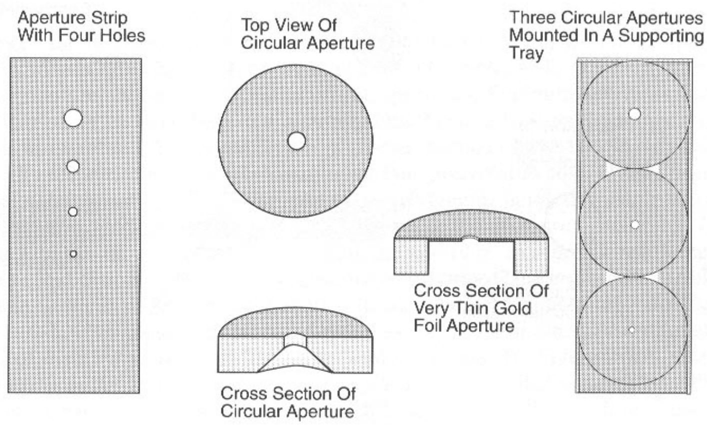


Figure 5-19 Aperture strips and discs.

Thick aperture strips or discs: 100-500 μm for condenser lens apertures, and 10-60μm for objective lens. Contamination is the major problem; from the gas adhere to aperture by charge, causing astigmation.

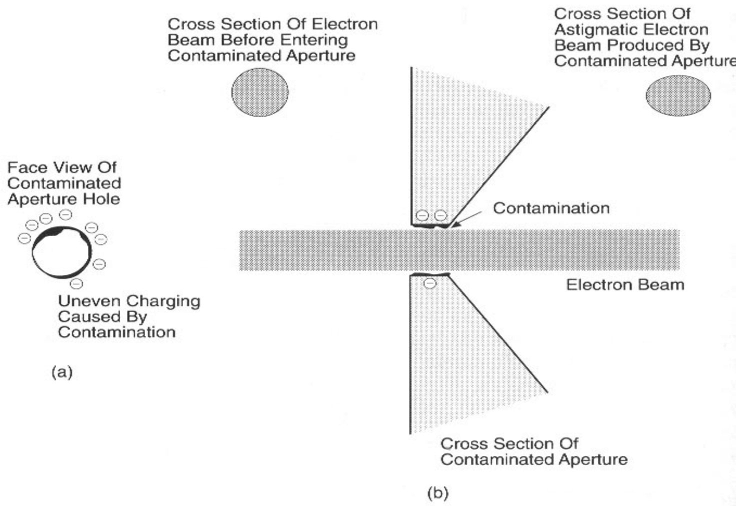


Figure 5-20 Contamination of an aperture. (a) Face view of a contaminated aperture showing the uneven deposition of contamination around the hole. In the electron beam the contamination charges unevenly, resulting in an uneven distribution of negative charges. (b) Effect of a contaminated aperture on an electron beam passing through the aperture. The aperture removes those stray electrons not aimed directly at the aperture. Those electrons that pass through the aperture are affected by the uneven charge on the aperture due to the contamination. This results in an uneven electric field on the beam and an astigmatic beam that is elliptical in cross section after it has passed through the aperture.

<u>Thin foil apertures:</u> 0.5 μm thick gold foil with hole.

7. Objective Stigmator

a. To correct for astigmatism of the beam by magnetic field.

8. Scanning coil

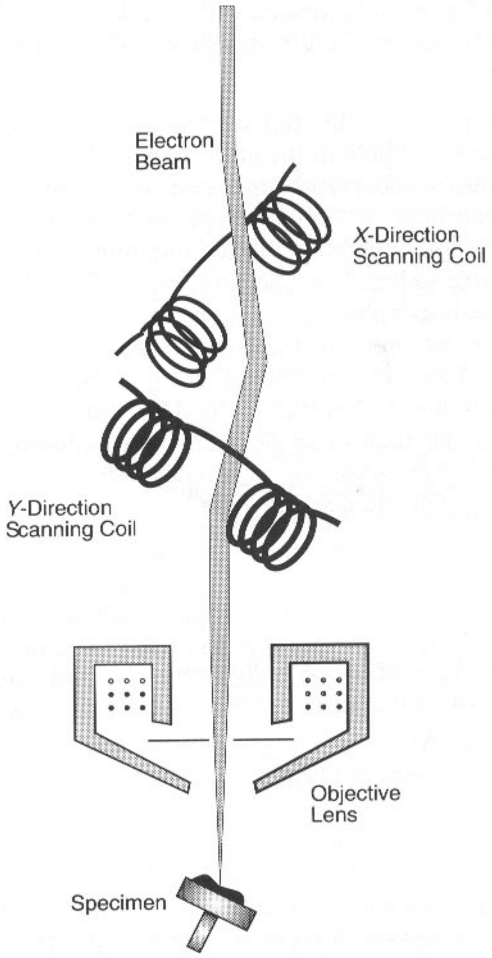


Figure 5-21 Scanning coils and objective lens. The electron beam is deflected by the scanning coils so th it is elbowed and manipulated to pa near the center of the objective lens aperture to strike the specimen.

- **Electron Emission**

1. Types

a. Thermionic emission (by heat)

b. High-field emission or field emission (by strong electrical field)

c. Electromagnetic radiation (photoelectric emission)

d. Atomic particles (secondary emission)

Only a. and b. are used for electron microscopes.

e. **<u>Work function</u>: the potential in volts that must be overcome for the electron to escape the surface of the metal, depending on atomic radius, crystal spacing, texture of the metal surface, and cleanliness.**

f. **The larger atom, larger crystal space, rougher surface, and cleaner surface give lower work function, thus the greater the emission of electrons.**

2. Thermionic emission

a. **Using heat to raise the energy of electrons**

b. **Requirement of materials for thermionic emitters: sufficient low work function, high melting point, stable at high temperatures, long operation life (less evaporation), economical.**

c. **Work function for tungsten: 4.5 eV.**

d. **Tungsten suffering from poisoning from water vapor and electronegative gases (such as oxygen)**

e. **Direction heating: current, indirect heating: cathode heated by outside source.**

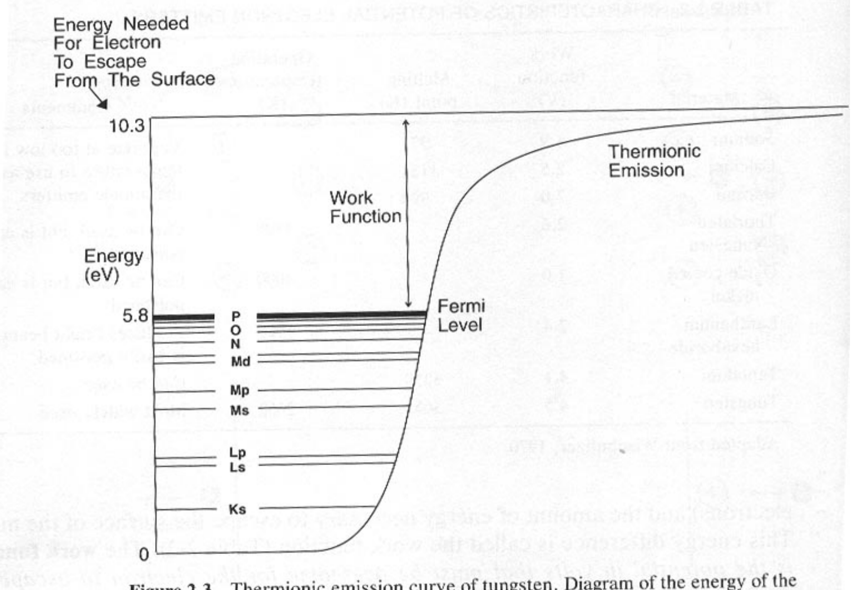


Figure 2-3 Thermionic emission curve of tungsten. Diagram of the energy of the orbital electrons of tungsten showing that the work potential is the difference between the energy necessary for the electrons to escape from the surface of the metal and the energy of the highest-orbital electrons.

3. Electron guns

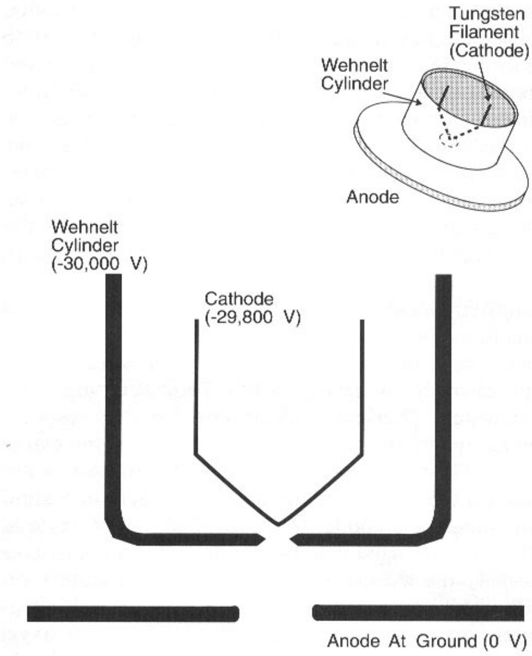


Figure 2-4 Biased electron guns, showing the position of the cathode, Wehnelt cylinder, and anode.

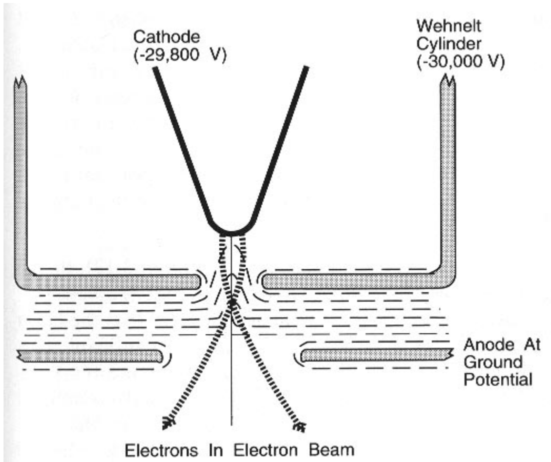


Figure 2-11 Path of the beam electrons in a self-biased gun.

a. Tungsten cathode is a wire about 100 μm in radius, and heated by 2.5 A at 1 V giving about 2700K. Apex of the V becomes the hottest part and the effective electron source.

b. Wehnelt cylinder provides electrostatic field to help on focus the electron beam.

c. Anode usually set as ground to prevent shorting.

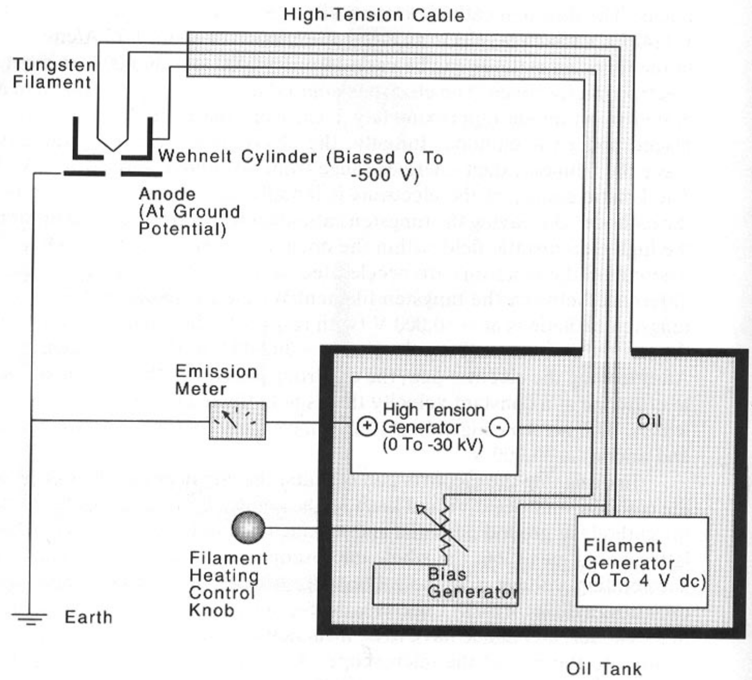


Figure 2-6 Setup of an electron microscope with a self-biased electron gun.

d. Operation of self-biased gun

(1). Emission of electrons occurs primarily form the hot filament tip. Little Wehnelt voltage, electron beam pass through anode and down to specimen, and the elongated spot representing the image of the filament.

(2). Tip and the area of the rear have been heated; Wehnelt cylinder has moderate voltage, forcing electron away and producing a cloud of beam electrons. Beam spot is still the image of the filament. (Central spot surrounded by uncompleted ring)

(3). Saturation point (tungsten filament at 2700K), where an increase in filament heating current results in increased electron emission but does not result in an increase in beam current. Space charge of the electron cloud is strong and the electron beam formed is only from the cloud instead of directly from the surface of the filament.

(4). Beam current is not increased by increasing the filament heating current. The increased negative bias of the Wehnelt cylinder forces a greater portion of the electrons back onto the tungsten filament.

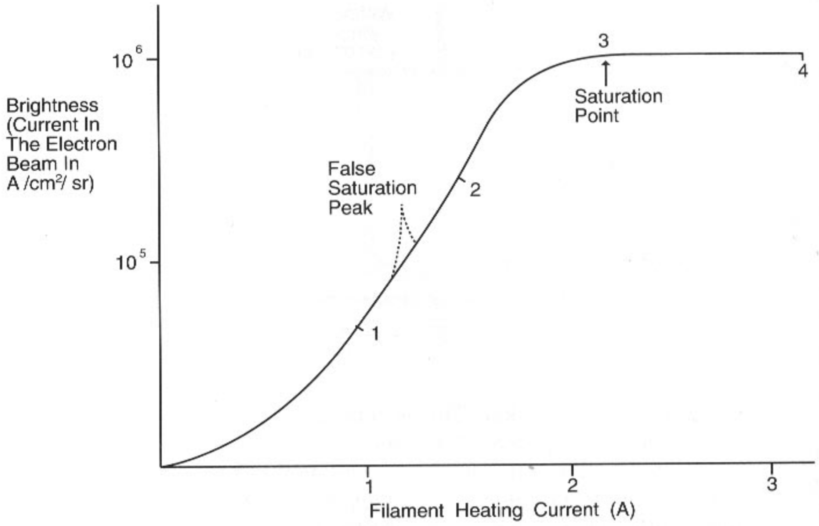


Figure 2-12 Relationship between the brightness of the electron beam and the filament heating current in a self-biasing gun with a tungsten cathode.

e. False Saturation peaks

As the filament heating current is increased, peripheral parts of the filament reach the saturation emission temperature before other areas, giving false peak.

f. Distance between the tungsten Cathode and the Wehnelt Cylinder:

When the tungsten filament is close to the aperture of the Wehnelt Cylinder, a large number of electrons are pulled from the accumulated electron cloud in front of the filament to produce a bright beam with a high beam current, thus a high filament heating current is needed to produce a saturated beam current.

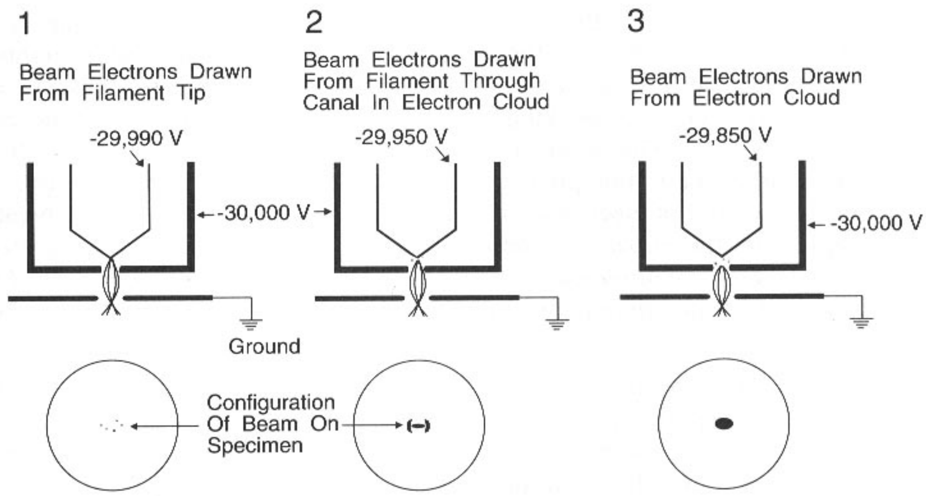


Figure 2-13 Potential of the electron gun at positions 1 to 3 in Fig. 2-12. The configuration of the beam on the specimen is also shown for each point.

g. **Failure of the tungsten filament**

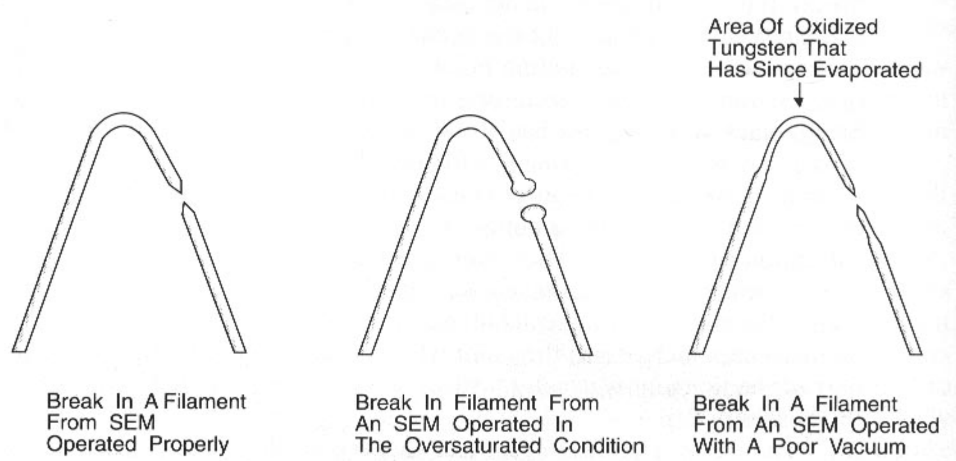


Figure 2-17 Condition of a failed tungsten filament operated under different conditions.

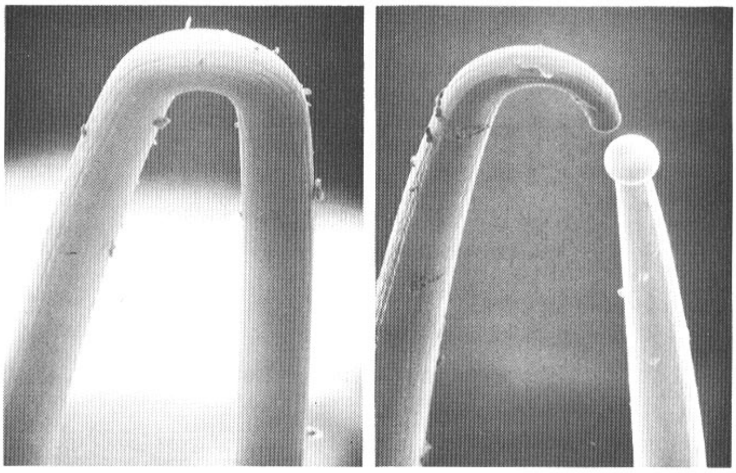


Figure 2-18 Scanning electron micrographs of (a) an unused tungsten filament and (b) a tungsten filament that has failed after being used at oversaturation. The failed filament has a ball of tungsten that was formed when the tungsten melted.

4. Lanthanum Hexaboride (LaB_6) cathode

a. **LaB_6 cathodes have the advantage of a long life of producing an electron beam with brightness 5-10 times higher than tungsten filament. Low work function (2.4 eV).**

b. **Disadvantages: expansive, need high vacuum to prevent poisoning.**

c. **Operating at 200K, 100 times brighter.**

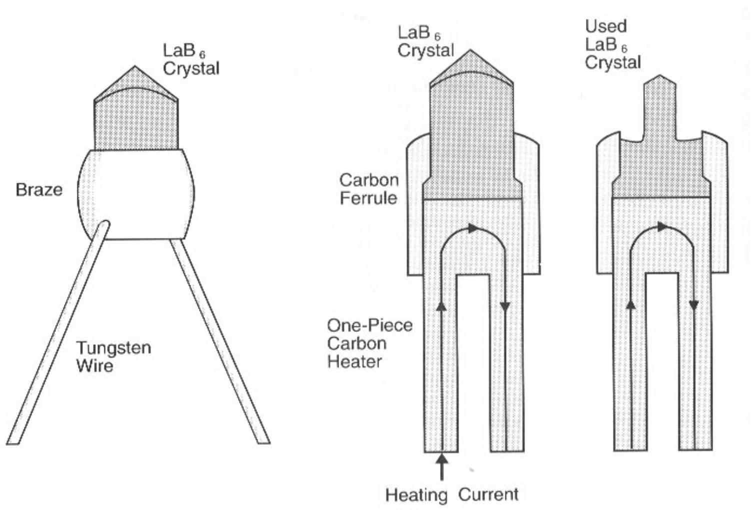


Figure 2-22 An electron gun containing a lanthanum hexaboride cathode.

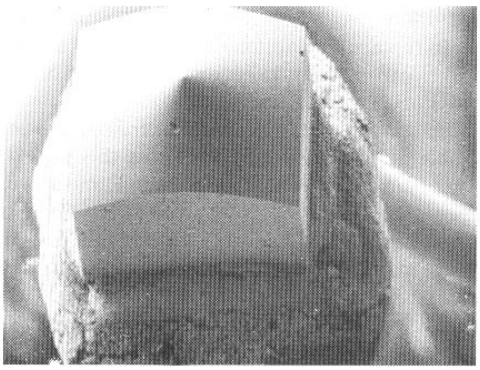


Figure 2-23 Scanning electron micrograph of the tip of a lanthanum hexaboride crystal that has been mounted to form a cathode for an electron gun.

Field Emission

d. Electron emission occurs when a cold metal surface in a vacuum is subjected to an electron-accelerating field on the order of 10^8 V/cm—field emission.

e. It is independent of temperature, and emission current increases with the increase of the field follow an exponential law.

f. Current densities are about 10^6 A/cm^2 for a field emission source, compared to 10 A/cm^2 for a thermionic tungsten source.

g. Smaller beam size can be produced, thus increase resolution.

$$Field = \frac{Volts}{Radius} = \frac{V}{R} = \frac{1000V}{50nm} = 2\times10^{8} V/cm$$

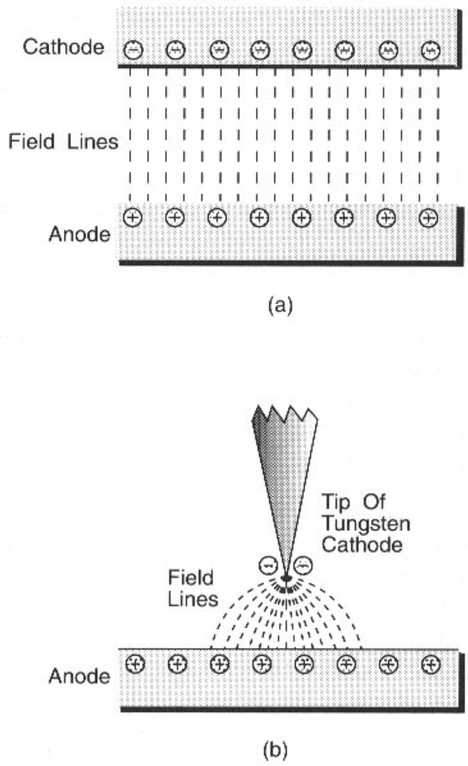


Figure 2-25 How a very high electric field can be generated at the tip of a fine point on a tungsten cathode: (a) When a potential is applied across two plates, the charges are spread along the plates and a disperse electric field results. (b) When the cathode is a fine tip, the field lines from the anode concentrate at the tip, resulting in the formation of a very strong electric field at the tip of the cathode.

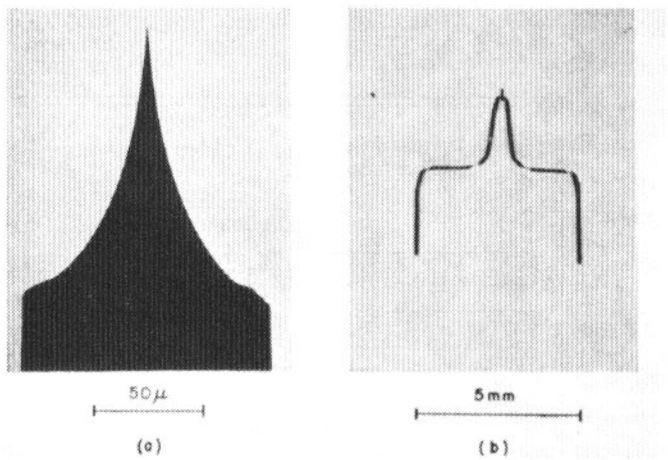


Figure 2-26 A tungsten cathode with an etched tip suitable for field emission. (a) Shadowgraph showing the configuration of the tip. (b) An etched tungsten tip mounted on a hairpin filament. (*From* A. V. Crewe and others, *Rev. Sci. Instruments* 39:576–584, 1968). (*Photograph courtesy of American Institute of Physics*)

- **Lens Aberrations**

  1. Spherical Aberration

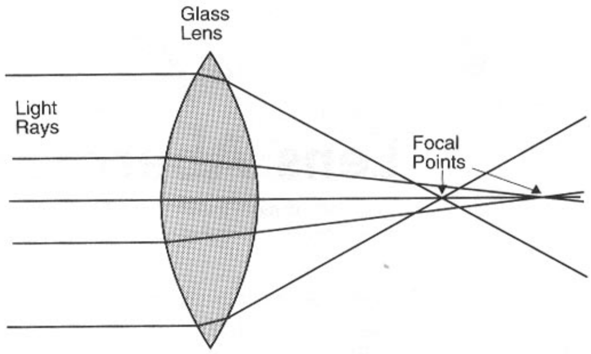
  a. Light rays or electrons that pass through the center of the lens converge at a focal point that is farther from the lens when compared to the focal point of those light rays or electrons that pass closer to the edge of the lens.

  b. In electromagnetic lenses, the strength of the magnetic field decreases as the distance from the windings of the

electromagnetic lens increases.

c. **Spherical aberration is proportional to the third power of the aperture angle, so introduce aperture in the electron path to limit spherical aberration.**

d. **Decrease focal length also reduce spherical aberration.**



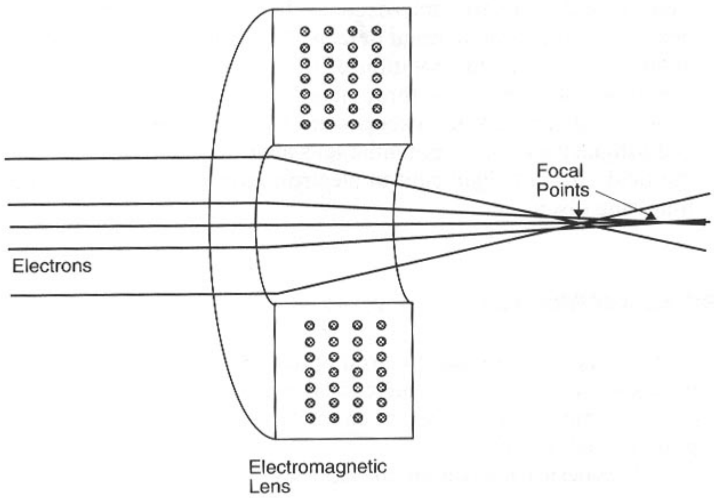


Figure 4-1 Spherical aberration arises when light rays or electrons passing through a lens are brought to different focal points. The differential bending of the light rays or electrons is due to varying lens strength.

2. Chromatic Aberration

a. **Occurs when lenses deflect light rays or electrons of different wavelengths to different degrees.**

b. **In electron lenses, electrons with different velocities enter the lenses at the same place and are bent to different focal points. Due to three factors: (1) variation in the emission velocity of the electrons as they leave the electron gun. (Not very important) (2) Variation in the accelerating voltage (3) variation in lens currents.**

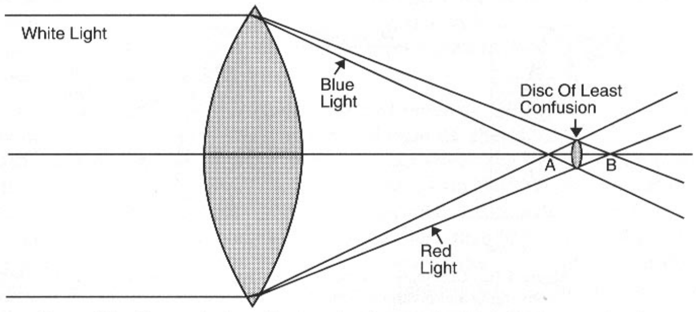


Figure 4-8 Chromatic aberration in a glass lens results when shorter-wavelength light rays are bent more than longer wavelengths as the light rays pass through the glass lens.

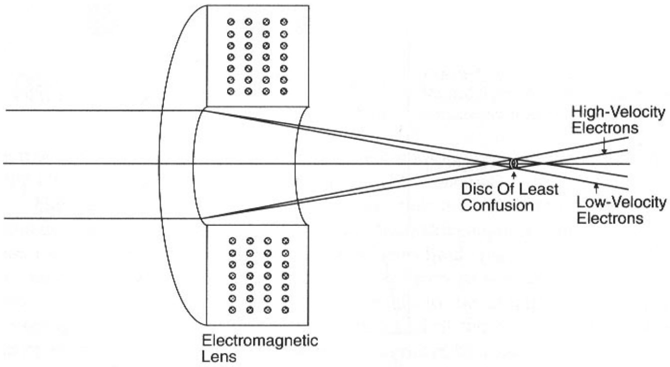


Figure 4-11 Chromatic aberration in an electromagnetic lens. The higher-velocity electrons are deflected less by the magnetic field than the lower-velocity electrons, resulting in a series of focal points for the different-velocity electrons.

3. Astigmatism

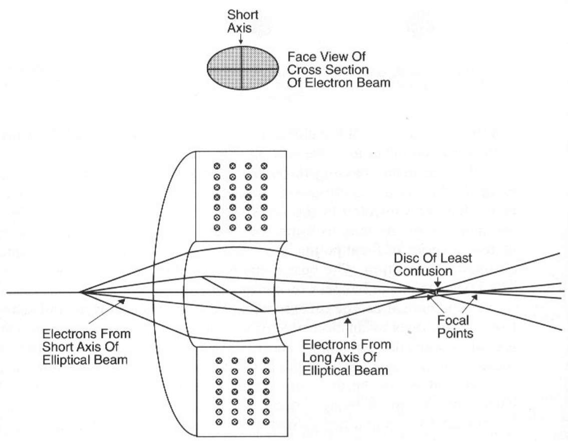


Figure 4-15 An electron beam that is elliptical in cross section will result when the electron beam passes through an electromagnetic lens that has an asymmetric magnetic field. This results in the formation of a continuous series of focal points past the lens.

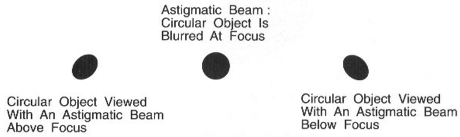


Figure 4-16 A circular object appears elongated in an astigmatic image.

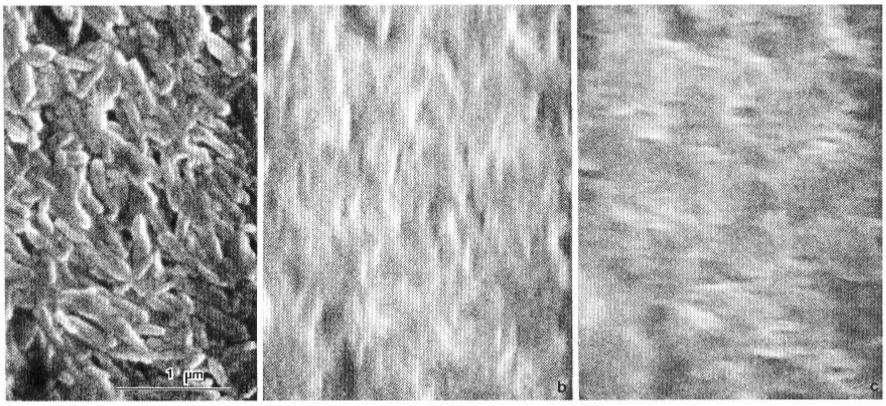


Figure 4-17 Scanning electron micrographs of the same area of magnetic tape taken at an accelerating voltage of 30,000 V. (a) An in-focus micrograph showing no astigmatism. (b) and (c) Images taken with an astigmatic electron beam showing how the orientation of details of the specimen changes 90° as the operator goes from overfocus to underfocus. (b) An astigmatic overfocused image showing the orientation of details primarily in the vertical plane. (c) An underfocused astigmatic image showing the orientation of details primarily in the horizontal direction.

a. Astigmatism is the inability of a lens to bring rays of light or electrons to a point, and is the most important factor limiting resolution in electron optical system comprising objective lens.

b. The cross section of electron beam becomes elliptical, not circular, when the short axis of the cross section is subjected to a stronger electrostatic or magnetic field in the lens.

c. Reasons: (1). Nonsymmetrical fields in electron lenses, (2). Dirty aperture or lenses.

d. Stigmators:

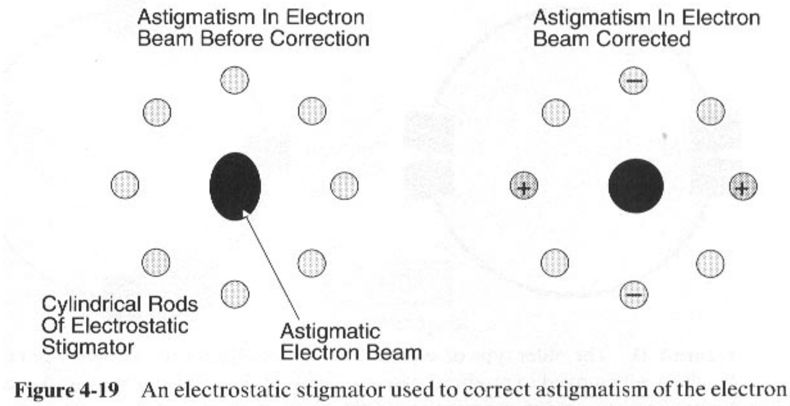


Figure 4-19 An electrostatic stigmator used to correct astigmatism of the electron beam by applying an electrostatic field to push and pull the electron beam into a circular cross section.

◆ **Specimen Preparation**

1. Dry specimens

Clean surface, coating the specimen with a 10-20 nm layer of a metal to reduce charging problem.

2. Specimens containing volatile components

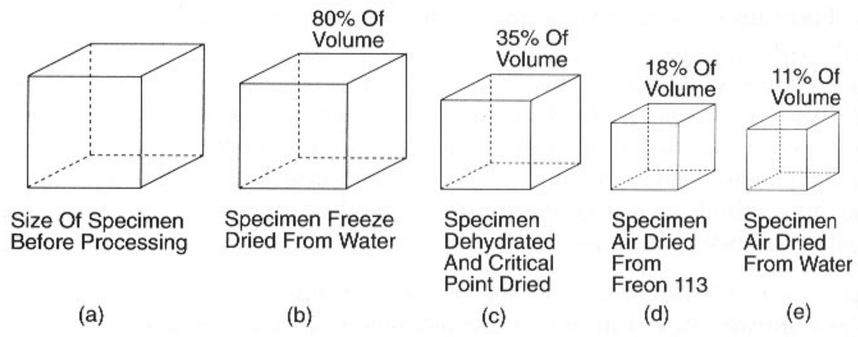


Figure 11-19 (a, b) Minimal shrinkage of a specimen results from rapid freezing followed by freeze drying. Specimens chemically fixed and critical point dried (c) have less shrinkage than specimens that are air dried (d, e). (*Redrawn from* A. W. Robards and U. B. Sleyter, Low temperature methods in biological electron microscopy, in *Practical Methods in Electron Microscopy* (A. M. Glauert, ed.), Vol. 10, Elsevier Publishing Co., New York, 1985.)

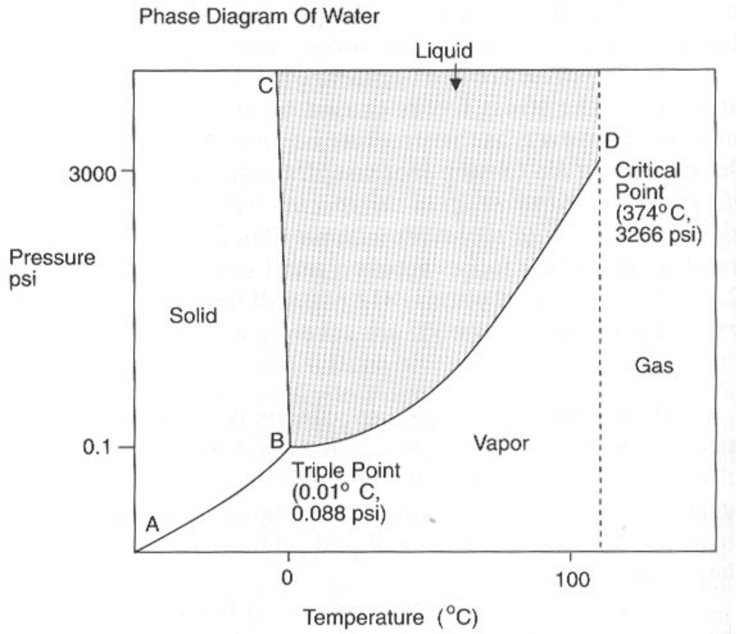


Figure 11-14 Phase diagram of water.

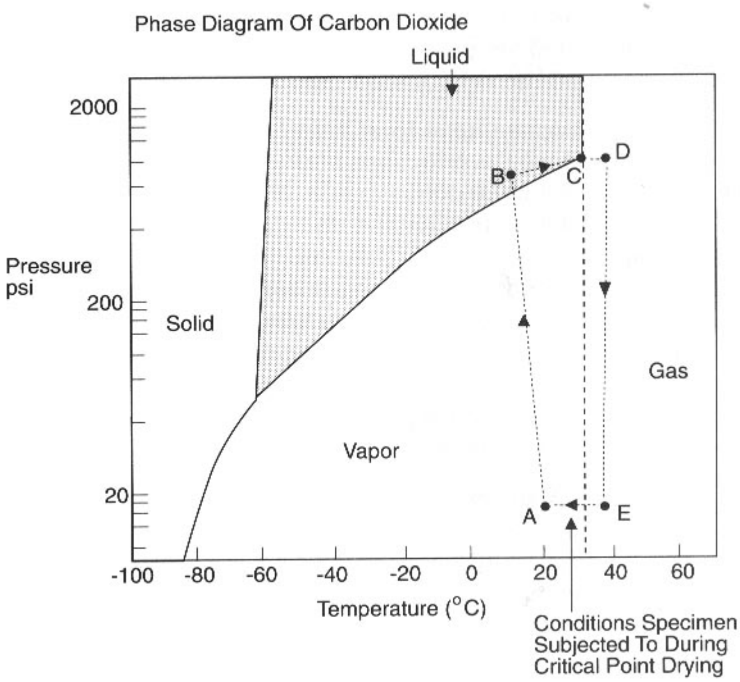


Figure 11-18 Phase diagram of carbon dioxide showing conditions that a specimen is subjected to during critical point drying. (A) The specimen in the dehydration fluid at room temperature and pressure. (B) The specimen in the critical point dryer with full cylinder pressure of carbon dioxide in the pressure chamber. (C) The specimen at the critical point. (D) The specimen above the critical temperature so that all the carbon dioxide is a gas. (E) The dry specimen at room pressure ready to be removed from the critical point dryer.

References:

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2. 汪建民主編, “材料分析”, 中國材料科學學會, 1998.