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# Questions/Answers

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## Chapter 1

1.1 What are the advantages of the SEM over optical microscopy?

Answer:

Advantages: Higher resolution and greater depth of field and microchemical analysis

Disadvantages: Expensive, images lack color, difficult to image wet/live samples, analysis takes more time, and equipment maintenance is relatively tedious.

1.2 What are the different types of samples imaged in the SEM?

Answer:

Metals, alloys, semiconductors, polymers, coatings, ceramics, rocks, sand, corrosion products, catalysts, membranes, carbon nanotubes, nanopowders tissues, cells, insects, leaves

1.3 List various applications of the SEM.

Answer:

Materials identification, materials science, forensic science, metallurgical and electronic materials failure analysis, corrosion science, rock mineralogy, geosciences, nanodevices, polymer science, catalysis, semiconductor design, desalination, life sciences, and oil and gas mining

1.4 Name the industries where SEM is used.

Answer:

Academic and research, oil and gas, power generation, metals and alloys, industrial manufacturing, automobile, aero, aerospace, petrochemical, geosciences, nanotechnology, semiconductor, computer, chemical process industry, mining

1.5 What was the imaging resolution achieved in the earliest SEM? What is the routinely achieved resolution nowadays?

Answer:

50 nm, 1 nm

1.6 Why the scanning electron microscope is so named?

Answer:

It is an instrument that is used to magnify small objects using an electron probe that is scanned across the surface of the object.

1.7 Define resolution limit.

Answer:

It is the smallest distinguishable distance separating two objects.

1.8 What is a useful magnification? Why simply magnifying objects is not enough to keep revealing fine details?

Answer:

It is the maximum magnification beyond which the image becomes blurred and the objects are not resolved clearly.

It is not possible to keep revealing newer details in an object by simply increasing the magnification. Fine details in an image cannot be resolved beyond a certain magnification. This is due to limitations imposed by the resolving power of the imaging technique as well as that of the human eye.

1.9 Who is credited with the invention of the first scanning electron microscope? State the approximate resolution achieved by the SEMs constructed in the early days.

Answer:

German physicist Max Knoll introduced the concept of a scanning electron microscope in 1935. He proposed that an image can be produced by scanning the surface of a sample with a finely focused electron beam. Another German physicist Manfred von Ardenne went on to produce the earliest scanning electron microscope in 1937.

1.10 Can SEM produce color images? State the reason for your answer.

Answer:

No. The probe used in the SEM is electrons whose wavelength does not fall within the visible light spectrum.

1.11 What capability of the SEM enables it to produce 3-D-like images?

Answer:

Large depth of field

## Chapter 2

2.1 Calculate the maximum theoretical brightness for W filament operated at 20 kV accelerating voltage and a temperature of 2700 °K. Take current density  $J_c$  of W cathode as  $3.4 \text{ A/cm}^2$ .

Answer:

$$\beta_{\max} = \frac{J_c e V_0}{\pi k T}$$

$$9.2 \times 10^4 \text{ A} \cdot \text{cm}^{-2} \cdot \text{sr}^{-1}$$

2.2 Determine the brightness of an electron beam that has a current of 3 mA, probe diameter of 0.5 mm, and convergence angle of 0.04 radians.

Answer:

$$\beta = \frac{4i_p}{\pi^2 d_p^2 \alpha_p^2}$$

$$\beta = 3.04 \times 10^9 \text{ A} \cdot \text{cm}^{-2} \cdot \text{sr}^{-1}$$

2.3 What is the brightness  $\beta$  of a probe that has a current density of  $8 \times 10^3 \text{ A/cm}^2$  and a convergence angle of 0.05 radians?

Answer:

$$\beta = \frac{j_c}{\pi \alpha^2}$$

$$\beta = 1 \times 10^6 \text{ A} \cdot \text{cm}^{-2} \cdot \text{sr}^{-1}$$

2.4 What are the four main types of electron guns used in the SEM?

Answer:

Tungsten filament thermionic emission gun

LaB6 thermionic emission gun

Schottky field emission gun

Cold field emission gun

2.5 Name the three main parts of the thermionic emission electron gun.

Answer:

Emitter (cathode, negative electrode, filament)

Grounded plate (anode, positive electrode)

Surrounding grid cover (Wehnelt cup, control electrode)

2.6 List at least three required characteristics of the electron gun.

Answer:

High brightness

Small source size

Low energy spread

Beam stability

2.7 List three advantages and disadvantages of W filament.

Answer:

Advantages:

Low cost.

Reliable.

Replacement is fairly straightforward.

Disadvantages:

Large energy spread, and  $\Delta E$  is 3eV.

High work function of 4.7eV.

Insufficient beam current.

Short service lifetime.

2.8 Describe the working of thermionic emission W filament.

Answer:

The filament is heated resistively to 2400 °C to exceed work function of W.

Negative bias exists between the filament and Wehnelt cylinder.

Wehnelt cylinder focuses the beam and controls electron emission.

Electrons make a crossover of diameter  $d_0$  (50 microns) between Wehnelt cylinder and anode.

Gun is at high negative potential, and anode is at ground (zero) potential resulting in electron acceleration.

Anode allows only a fraction of electrons to pass through.

Bias resistor keeps the negative bias at the Wehnelt cylinder at an optimum level resulting in a stable beam.

2.9 What is the difference between filament, emission, probe, and beam current?

Answer:

The current provided to the filament is called “filament heating current”  $i_f$ .

Total emitted current (100 mA) from the filament is “emission current”  $i_e$ .

The fraction of emission current leaving thru the anode is “beam current”  $i_b$ .

Current decreases at every lens and aperture; the final current measured at specimen is called “probe current”  $i_p$ .

2.10 List three advantages of LaB<sub>6</sub> filaments over W filaments.

Answer:

Five to ten times brighter than W filaments

Improved image quality

Longer service life ( $\approx 1000$  h)

2.11 List three disadvantages of LaB<sub>6</sub> filaments over W filaments.

Answer:

About ten times more expensive

Require higher vacuum to work

Chemically reactive when it gets hot

2.12 Describe shortly the working of the field emission electron gun.

Answer:

The field emission cathode is usually a wire of single-crystal tungsten fashioned into a sharp point of about 100 nm or less. If the tip is held at negative 3–5 kV relative to the anode, the applied electric field at the tip is so strong that the potential barrier for electrons becomes narrow in width. This narrow barrier allows electrons to “tunnel” directly through the barrier which leave the cathode without requiring any thermal energy to lift them over the work function barrier.

2.13 List three advantages of field emission gun over the LaB<sub>6</sub> emitter.

Answer:

Field emission gun is 100 times brighter than a LaB<sub>6</sub> emitter.

Service life is up to 2000 h.

Field emission guns produce fine probe sizes with large probe currents allowing for high spatial resolution of microscopes.

2.14 List two disadvantages of field emission gun over W or LaB<sub>6</sub> guns.

Answer:

High cost

High vacuum requirement, i.e.,  $10^{-9}$  to  $10^{-10}$  Pa

2.15 What is an aperture disc, and what is its function?

Answer:

Apertures: It is a strip of metal (Mo or 95%Pt-5%Ir alloy) with precision-drilled holes of various sizes, e.g., 10, 50, 100, and 500  $\mu\text{m}$ .

Function: The aperture discs are located at critical positions within the SEM column and serve to block off-axis beam electrons from reaching the specimen. It limits or controls the number of electrons passing through the column.

2.16 List two types of electromagnetic lenses in the SEM and their functions. How are they controlled?

Answer:

Condenser lenses: Demagnify the electron beam by regulating the current in the condenser lens coils. It is controlled by the knob labeled as “spot size.”

Objective lenses: To focus the beam onto the specimen surface and form the image. The objective lens is controlled by adjusting the “focus” control knob during the SEM operation.

2.17 What does strong and weak condenser lens signify? What effect it has on the spot size?

Answer:

Weak condenser strength results in higher current passing through, i.e., bigger probe size (large spot size).

Strong condenser strength results in lower current passing through, i.e., smaller probe size (small spot size).

2.18 What is spherical aberration, and how can it be corrected?

Answer:

The ability of electromagnetic lenses to focus the beam into a fine symmetrical probe is limited by defects called lens aberrations. Electrons near the edge of the lens are bent more strongly than the ones away from the edge resulting different points of focus. It can be corrected by using a small aperture.

2.19 What is chromatic aberration? Is it higher at low kV or high kV?

Answer:

Electrons of different energies focus at different focal points. Less energetic electrons will be bent more strongly by the lens. It is enhanced at low kV.

2.20 How does an astigmatized image appear? How can it be corrected?

Answer:

The image appeared stretched in one direction due to astigmatism, which can be removed using astigmator coils.

2.21 Where are scan coils located, and what are their function/purpose?

Answer:

Scan coils are located within the objective lens assembly in the electron column. They scan the electron beam from left to right across the surface of the specimen. They are used to control the magnification of the image.

2.22 Calculate the probe diameter of the electron beam with an energy of 20 keV as it passes through a condenser lens set at a magnetic field strength ( $B$ ) of 1.0 tesla. Take electron mass ( $m$ ) and charge ( $e$ ) as  $9.109 \times 10^{-31}$  kg and  $1.602 \times 10^{-19}$  Coulombs, respectively.

Answer:

$$d_0 = \frac{1}{2B} \sqrt{\frac{2mV_0}{e}}$$

0.24  $\mu\text{m}$

2.23 What unit dimension is used to express spherical aberration coefficient,  $C_s$ ?

Calculate the beam diameter formed at the Gaussian image plane in an SEM equipped with an immersion lens that has a focal length of 5 mm. Take convergence angle to be 0.005 radians, and ignore other lens aberrations.

Answer:

Millimeters.

The beam diameter at the Gaussian image plane is measured as  $2 C_s \alpha^3$   
 $2 \times 5 \times (0.005)^3 = 1.25$  nm.

2.24 Calculate the minimum probe size of an electron beam with an energy of 20 keV, probe current of 3 mA, the brightness of  $3.04 \times 10^9 \text{ A} \cdot \text{cm}^{-2} \cdot \text{sr}^{-1}$ , and a spherical aberration of 2 mm (take  $K = 1$  and  $\lambda = 0.008 \text{ nm}$ ).

Answer:

$$d_{p,\min} = K C_s^{1/4} \lambda^{3/4} \left( \frac{i_p}{\beta \lambda^2} + 1 \right)^{3/8}$$

125.46 nm

2.25 What is the solid angle of a detector? How does it affect the efficiency of the signal collection?

Answer:

The size of the detector is described by the solid angle ( $\Omega$  – omega) which is the ratio of the area of the face of the detector to the square of the radial distance to the beam impact point,  $\Omega = A/r^2$ .

2.26 Name different components of Everhart-Thornley detector (ETD) and describe their functions.

Answer:

Faraday cage: Secondary electrons are pulled toward the detector by applying a bias ( $-50\text{V}$  to  $+250 \text{ V}$ ) to F placed at the front end of the detector.

Scintillator: The scintillator (S) surface is doped plastic/glass or crystalline  $\text{CaF}_2$  doped with europium with a  $+10\text{--}12\text{kV}$ . The electrons are converted into light (photons) upon striking the scintillator surface.

Light guide: The light then passes through a total internal reflection plastic or glass pipe (light guide, LG).

Photomultiplier: The first photocathode of PM converts the photons into an electric signal (electrons) that is amplified (up to  $\times 10^6$  gain) to levels suitable for image formation on a CRT tube or computer monitor.

2.27 What are secondary and backscattered electrons, and what is their origin?

Answer:

SE = secondary electrons (low-energy electrons belonging to sample)

B = backscattered electrons (high-energy electrons originally belonging to the electron beam)

2.28 Where is backscattered electron detector usually located?

Answer:

It is located directly above the sample (below the objective lens cone).



2.29 Why can't we use rapid scan rates with a solid-state BSE detector?

Answer:

The detector response time is slow for BSE solid-state detector. Rapid scan rates can be employed with a scintillator-type BSE detector.

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## Chapter 3

3.1 Define the picture element and pixels. What parameters are stored in the computer for each pixel? How can we compare the size of the picture element and pixel?

Answer:

Discrete locations on the specimen where the electron beam dwells to generate signals are called picture elements. Each picture element will have a specific value of location ( $x, y$ ) and intensity  $I$ . The signals are then processed by the detector and are used to form an image on a screen at corresponding locations called pixels. A pixel in an image is larger than the corresponding picture element on a specimen by a magnitude that equals the magnification used to take that image.

3.2 State the benefits of digital imaging.

Answer:

- Digital imaging technology is efficient and cost-effective and easily lends itself to storage and further handling or processing.
- Several images or frames can be acquired and averaged to reduce noise or charging effects.
- Digital images are stored in memory and can be displayed on the screen without having to continuously scan the beam on the specimen surface thus reducing the probability of beam-induced contamination or damage in sensitive specimens.

3.3 How is the size of the picture element in the specimen determined?

Answer:

Pixel length is determined by dividing the horizontal length of the image with the number of image pixels present in the horizontal direction. The picture element is calculated by dividing the pixel size by the value of magnification.

$$L_{\text{picture element}} = \frac{L_{\text{pixel}}}{M}$$

3.4 How does the size of the interaction volume affect the image resolution?

Answer:

Interaction of the probe with the specimen gives rise to large interaction volume. If the diameter of the projected area of interaction is comparable to the size of one picture element in the specimen, the image will be in sharp focus. If the area extends to several picture elements, the image will become blurred since information from several locations will overlap.

3.5 What practical steps can be taken to increase the signal-to-noise ratio?

Answer:

Increase probe current, reduce image scan rate, perform frame averaging, increase solid angle of electron collection, and carry out post-acquisition smoothing.

3.6 What is the contrast formed by a particle emitting 500 secondary electrons compared to 100 SEs ejected from the background?

Answer:

$$C = \frac{S_A - S_B}{S_A}$$

$$C = 0.8 = 80\%$$

3.7 Why are secondary electrons suitable to study surface features?

Answer:

Secondary electrons are produced from near the surface of the specimen and hold information about the surface features. They are suitable to study the topography of a material.

3.8 What happens when +200 V and at -50 V bias voltage is applied on the Faraday cage of an E-T detector?

Answer:

At +200 V setting, the E-T detector attracts all SEs and only line-of-sight BSEs. At -50 V, the E-T detector rejects all SEs and receives line-of-sight BSEs.

3.9 What is edge effect? Why does it occur?

Answer:

Higher incidence angle allows greater penetration of the beam into surface regions such that the escape distance toward one side of the beam decreases and the number of secondary electrons emitted from the specimen increases. Higher secondary electron emission results in a brighter contrast. This is why steep surfaces, protrusions, and edges within a specimen tend to appear brighter compared to the flat surfaces. This is called *edge effect*.

3.10 Why is an E-T detector commonly known as SE detector?

Answer:

Images formed by the E-T detector have a strong SE component due to the positively biased Faraday cage.

3.11 What is interaction volume and why is it important?

Answer:

Elastic and inelastic scattering events make electrons penetrate into the depth and spread laterally across the breadth of the specimen resulting in a relatively large “interaction volume.” This volume encompasses most of the scattering events. This is the volume of the specimen within from all imaging, and the microchemical information is extracted directly affecting the spatial and analytical resolution of the SEM.

3.12 Describe the effect of beam energy and atomic number of the specimen on the shape and size of interaction volume.

Answer:

The higher is the beam energy ( $E_0$ ), the greater is the depth and breadth to which the electrons can travel into the specimen as they lose energy at a lower rate which is proportional to  $\frac{1}{E_0}$ . Increasing beam energy also reduces its probability to scatter elastically (as a function of  $\frac{1}{E_0^2}$ ) thus penetrating deeper into the specimen. The trajectories of the electrons near the specimen surface are straight resulting in widening of the interaction volume away from the surface. High beam energy is more likely to form a teardrop interaction volume, and low beam energy tends to form a hemispherical shape.

For specimens with high  $Z$ , the elastic scattering is greater resulting in a deviation of the electrons from their original path more quickly and reducing the distance that they travel into the specimen. This results in a hemispherical shape of the interaction volume. On the other hand, elastic scattering and the rate of energy loss per unit length are lower in low atomic number targets due to which beam electrons manage to maintain their straight trajectories for larger depths in the specimen. This results in the formation of a large interaction volume taking the form of a *teardrop*.

3.13 How does beam energy and  $Z$  affect electron range?

Answer:

Electron range or depth of beam penetration increases with increasing beam energy and decreasing  $Z$ .

3.14 What is the difference between BSE and SE?

Answer:

SE are low-energy (<50 eV) electrons that belong to the target material. They are ejected out due to inelastic scattering of the electron beam close to the surface of the specimen. BSE are high-energy (several keV) electrons belonging to the original beam that enters the specimen and gets elastically scattered at large angles to emit out.

3.15 What is the origin of compositional contrast?

Answer:

Compositional contrast is formed by BSE. Scattering of BSE strongly depends on the atomic number of the specimen material. Target materials with high atomic number show a high degree of elastic scattering resulting in high angles of deflection and large backscattering effect. Two phases with different atomic numbers present within a specimen will exhibit different values of backscatter coefficient. This will result in a contrast where the phase with a high atomic number will appear relatively brighter (due to a larger number of backscatter electrons ejecting out of this phase), while phase with a low atomic number will appear relatively dark. This forms the basis for a compositional or atomic number or  $Z$  contrast.

3.16 Calculate the natural BSE contrast formed between two adjacent phases of pure Au and Al.

Answer:

Backscatter coefficient is given as:

$$\eta = -0.0254 + 0.016Z - 1.86 \times 10^{-4}Z^2 + 8.3 \times 10^{-7}Z^3$$

$$\eta_{\text{Au}} = 0.48$$

$$\eta_{\text{Al}} = 0.15$$

From Eq. 3.3, contrast =  $\frac{\eta_{\text{Au}} - \eta_{\text{Al}}}{\eta_{\text{Au}}} = 0.69$  or 69%.

3.17 Why does backscatter coefficient increase with specimen tilt?

Answer:

As the angle of inclination between the beam and specimen is narrowed (i.e., tilt,  $\theta$  increases), a greater number of electrons can escape the specimen surface as backscattered electrons. The surface, when tilted, lies closer to the forward scattering direction of electrons.

3.18 What can be done to maximize BSE signal if the detector is placed (a) directly above and (b) to the side of the specimen?

Answer:

- (a) Image the specimen at zero-degree tilt.
- (b) Image the specimen by tilting it toward the detector.

3.19 How does the variation of spatial distribution of BSE with the atomic number of the target material affect the image?

Answer:

Low atomic number targets will exhibit larger depth and lateral dimensions from which the BSE are derived. This will add to the noise component of the image leading to the degradation of its resolution.

3.20 How much of a backscattered contrast between two phases is appropriate?

Answer:

Contrast between 1% and 10% is appropriate. Less than 1% is difficult to interpret. More than 10% is considered strong.

3.21 What is the origin of trajectory component in BSE image?

Answer:

Features facing the detector will appear bright in the BSE image. The features facing away from the detector will appear relatively dark even though they have the same density. This is due to the fact that BSE emitted from the specimen have straight trajectories and continue their travel in the direction they are originally emitted. Apart from the atomic number  $Z$  component, this characteristic adds a trajectory component to the BSE image. If the specimen is flat, the BSE trajectories will not vary, and the contrast will solely depend on the  $Z$  component. If the surface is rough, the BSE trajectories will vary as per orientation of features, and the image contrast will be composed of  $Z$  and trajectory components. Separation of  $Z$  and trajectory components present in the BSE image can generate BSE compositional or topography images independently.

3.22 Why is the spatial resolution of the BSE image worse than that of SE image?

Answer:

The spatial resolution of backscattered electron images varies between 50 and 100 nm for beam energies of 10–20 keV that are employed during routine imaging. This is directly related to the comparably large volume within the specimen from where the backscattered electrons are derived to form the image. At low beam energy of 1 keV, the information volume of SE and BSE becomes

comparable. For high-resolution microscopy, low-loss BSE are used which are ejected from the area immediately surrounding the point of beam incidence. These electrons undergo single or lesser number of scattering events and represent high-resolution signal.

3.23 Summarize the factors that influence BSE contrast formation.

Answer:

By convention, the energy of the BSE is generally defined as  $50 \text{ eV} < E_{\text{BSE}} \leq E_0$ .

Specimens with large  $Z$  will have high BSE energy distribution. Light elements will show low-energy distribution.

The depth and lateral dimensions of interaction volume formed in a given target material increase with an increase in beam energy.

Specimens with a high  $Z$  show a high degree of elastic scattering resulting in high angles of deflection and large backscattering coefficient  $\eta$ . Light elements exhibit small  $\eta$ . Due to this reason, a phase with a high  $Z$  will appear bright, while a phase with a low  $Z$  will appear dark. This is called compositional or atomic number or  $Z$  contrast.

Backscattered signal from the specimen can be increased by tilting the specimen in such a way that the incidence angle of the beam with the specimen surface is small, i.e., at large tilt angle  $\theta$ .

At zero-degree specimen tilt, the maximum backscattered electrons are emitted along the beam. BSE emitted at small angles relative to the specimen are small in number, following a cosine law.

The collection efficiency of a detector for an untilted specimen can be maximized by placing it directly above the specimen.

BSE topographic contrast can be obtained by collecting BSE signals separately by two segments A and B which are located at opposite sides of the specimen. The image formed by these segments will differ as per the orientation of the features. Features normal to segment A will appear bright in A and dark in B. Features normal to B will appear bright in B and dark in A. Subtraction (A-B) of these images will reveal topographic contrast.

3.24 An electron beam with primary current  $i_B$  of  $2 \mu\text{A}$  gives SE current  $i_{\text{SE}}$  of  $0.6 \mu\text{A}$  and BSE current  $i_{\text{BSE}}$  of  $0.4 \mu\text{A}$ . Calculate the specimen current  $i_{\text{sp}}$  and SE yield  $\delta$  and BSE yield  $\eta$  for this beam.

Answer:

Primary beam current,  $i_B = i_{\text{SE}} + i_{\text{BSE}} + i_{\text{sp}}$

Specimen current,  $i_{\text{sp}} = i_B - i_{\text{SE}} - i_{\text{BSE}}$

$i_{\text{sp}} = 2 - 0.6 - 0.4 = 1.0 \mu\text{A}$

SE yield,  $\delta = \frac{i_{\text{SE}}}{i_B} = 0.3$

BSE yield,  $\eta = \frac{i_{\text{BSE}}}{i_B} = 0.2$

3.25 Why is escape depth of SE smaller in metals compared to insulators?

Answer:

The escape depth depends on the type of material. It is smaller (around 1 nm) in metals and up to 20 nm in insulators. This is due to the fact that SE generated within the specimen are inelastically scattered due to the presence of a large number of conduction electrons in metals. This scattering prevents the SE generated within greater depths of metals to escape the surface. Due to a lack of electrons in insulators, inelastic scattering of generated SE is not significant thus allowing them to reach and escape the specimen surface from greater depths.

3.26 Compare escape depth of BSE and SE.

Answer:

The probability of escape of secondary electrons as a function of specimen depth  $Z$  decreases sharply with depth. In the range of 10–30 keV, secondary electron escape depth is around  $100\times$  smaller than the backscattered electron escape depth.

3.27 What is the energy distribution of SE?

Answer:

Although, the secondary electron energy can be up to 50 eV, 90% of secondary electrons have an energy  $<10$  eV. The distribution of the secondary electron energy is generally peaked in the range (2–5 eV).

3.28 Which signal dominates in an image,  $SE_1$  or  $SE_2$ , and why?

Answer:

The contribution of the  $SE_1$  signal in secondary electron emission is greater than  $SE_2$  signal in light elements. This is due to the fact that backscattering is low in light elements due to their small atomic size. Therefore, the main component of the total SE signal is generated by the primary beam incident on the specimen surface. In heavy elements,  $SE_2$  signal dominates since backscattering is prominent due to large atomic size.

3.29 How does  $SE_3$  signal represent BSE contrast?

Answer:

The number of  $SE_3$  signal emanated from the hardware of the SEM specimen chamber depends on the number of BSE originating from the specimen.  $SE_3$  increases when BSE increases and  $SE_3$  decreases when BSE decreases. Thus,  $SE_3$  is an indication of atomic number contrast in the image.

3.30 What is the effect of beam energy on SE yield?

Answer:

Secondary electron emission is higher at lower incident beam energy. There is a significant increase in secondary electron emission below 5 keV. This is due to the fact that secondary electrons have low energy and can only escape the specimen if they are generated near its surface. At low keV, the penetration of incident beam is shallow, and most of the secondary electrons are generated near the specimen surface. This enables them to escape resulting in a higher secondary electron coefficient ( $\delta$ ) at lower beam energy.

3.31 How low beam energy be used to obtain high-resolution images?

Answer:

At low beam energy, the SE<sub>2</sub> electrons are generated closer to the beam impact point decreasing the range of SE<sub>2</sub> signal. SE<sub>2</sub> spatial distribution approaches that of SE<sub>1</sub> under such conditions. Due to their close proximity to the beam impact point, both types of signals now serve to make up the high-resolution image. The use of low beam energy, therefore, allows exerting better control on interaction volume and image contrast and facilitates the acquisition of high-resolution images rich in near-surface information.

3.32 Does SE yield change with Z?

Answer:

For practical considerations, the secondary electron emission can be considered to be independent of atomic number and does not change significantly from light to heavy elements.

3.33 What is the effect of specimen tilt on SE yield?

Answer:

When the specimen is tilted at increasing angles  $\theta$ , secondary electron coefficient  $\delta$  increases obeying a secant relationship.

3.34 List the factors that affect topographic contrast.

Answer:

Sharp edges and tilted features in a specimen yield more SE as secondary coefficient  $\delta$  increases as a secant function of tilt  $\theta$ . Sharp edges, therefore, appear bright in SE image. Tilted surfaces in a specimen will also generate a higher number of BSE as backscatter coefficient  $\eta$  increases significantly with the angle of tilt. This



will, in turn, increase the  $SE_2$  signal also adding a number component to the topographic contrast formed in the SE image. Moreover, tilted features will generate BSE in a direction defined by the beam direction and surface normal of the feature. This adds a trajectory component to the SE image.

The yield of SE from tilted surfaces is not significantly different in various directions, so the trajectory component in this regard is negligible.

The Everhart-Thornley detector receives line-of-sight BSE from features of specimen facing the detector. These BSE will also form part of the SE image.

Elements with high atomic number have a greater yield of backscattered electrons compared to elements with lower  $Z$ . This adds a number component to the topographic contrast, and high  $Z$  elements, therefore, may appear brighter in the SE image.

Contrast in SE image is enhanced at lower accelerating voltage. Charge accumulation or buildup in partly coated areas of the specimen can also result in an increase in contrast.

Magnetic areas in a specimen may either deflect or attract the beam to affect the yield of SE.

### 3.35 Fracture surface is best imaged using SE or BSE?

Answer:

Fracture surface is usually imaged to observe the topography of material. Therefore SE signal is more adequate for fracture surface imaging. SE gives better image resolution and can be collected efficiently from surfaces facing away from the E-T detector. Various features within the fracture surface such as fatigue striations, dimples, ridges, beach marks, crevices, etc. can be identified unambiguously.

### 3.36 What imaging mode suits a specimen with two phases widely separated by $Z$ ?

Answer:

It is more appropriate to use BSE imaging to observe the contrast between two phases with different  $Z$  as SE yield does not differ appreciably with  $Z$ .

## Chapter 4

4.1 What is the resolution of the SEM at 30, 20, 10, 5, and 1 keV if convergence angle is 0.01 radian?

Answer:

$$d = \frac{0.753}{\alpha\sqrt{V}} \text{ where } d \text{ is the resolution limit}$$

At 30 keV,  $d = 0.435 \text{ nm}$

At 20 keV,  $d = 0.532 \text{ nm}$

At 10 keV,  $d = 0.753 \text{ nm}$

At 5 keV,  $d = 1.065 \text{ nm}$

At 1 keV,  $d = 2.38 \text{ nm}$

4.2 Summarize the operating conditions that favor high-resolution microscopy.

Answer:

Short working distance produces a small probe size and reduces lens aberrations that serve to improve resolution. High accelerating voltage should be used as it produces a small probe size, unless surface features are to be imaged in which case low accelerating voltage is suitable as it limits the size of the excitation volume. Smallest probe current that gives adequate signal-to-noise ratio combined with frame averaging should be used. Intermediate final aperture is suitable to balance out aberration and diffraction effects.

4.3 Calculate the wavelength of the electrons that have an energy of 10 keV.

Answer:

$$\lambda = \sqrt{\frac{1.5}{V}} \text{ nm}$$

$$\lambda = 0.0122 \text{ nm}$$

4.4 Summarize the operating conditions that favor high depth of field.

Answer:

Large working distance, small final aperture (e.g., small convergence angle), and small magnification favor images with large depth of field.

4.5 Why depth of field in the SEM is far larger than in the optical microscope?

Answer:

The depth of field is large because the convergence/divergence angle formed in the SEM is small (in milliradians). Due to this reason, the probe size remains small for longer depth ( $z$ - $d$  axis).

4.6 Find the depth of focus of a beam when the angle of convergence is 0.2 radian and magnification is 10,000 $\times$ .

Answer:

$$D = \frac{200}{aM} \mu\text{m}$$

$$D = 0.1 \mu\text{m}$$

4.7 Describe the relationship between the probe current and size.

Answer:

Probe diameter increases with probe current at all accelerating voltages. At high accelerating voltage, the probe diameter is smaller compared to low accelerating voltage.

4.8 Summarize the advantages/disadvantages of the use of high/low probe current.

Answer:

High probe current results in smooth images but degraded image resolution. It can also induce beam damage. Low probe current can realize high image resolution due to small probe size, and the specimen is susceptible to less beam damage. Very low probe currents give rise to grainy images which tend to hide surface details. A critical level of probe current is required to achieve an acceptable contrast in the image.

4.9 What is the advantage of FESEM for high-resolution use?

Answer:

Field emission electron guns can concentrate a large amount of current in small probe resulting in high brightness and small spot size.

4.10 State the benefits/drawbacks of using large working distance during microscopy.

Answer:

Large WD increases the depth of field due to smaller convergence angle. It also allows the specimen to be observed at small magnifications. Large WD lowers the spatial resolution due to increased probe diameter. The signal strength at large WD decreases, and the image can appear relatively noisy.

4.11 What is the positive charging effect on the specimen surface?

Answer:

A charge balance is achieved when incident electrons impinging upon the specimen ( $i_B$ ) are equal to those leaving the sample ( $i_{sp} + \eta + \delta$ ). If the number of incident electrons is higher than the emitted electrons, the sample will charge negatively

which is the usual case. If the number of emitted electrons become more than the incident electrons at localized specimen surface, then positive charging takes place at that region. This does not pose much difficulty as the positive charge created at the specimen surface is neutralized by the SE emitted from the specimen and pulled back toward the surface.

#### 4.12 How is charging recognized?

Answer:

Charging effect may present itself in many forms such as unusual contrast (fluctuation in image intensity such as excessive brightness/darkness in images), horizontal lines on images, beam shift and image distortion (spherical objects appear flat), etc.

#### 4.13 What is the most effective and common used method to combat charging effects?

Answer:

Sputter coat with a thin metal layer.

#### 4.14 What means can be adopted to prevent contamination during imaging?

Answer:

Contamination from the instrument is reduced by employing dry pumps or installing a vapor trap in the pump backing line that can control hydrocarbon contamination originating from vacuum pumps. In addition, cold traps can be employed to seize contaminants, and the SEM chamber is purged with dry nitrogen gas during specimen exchange. Contamination from the specimens can be reduced by properly handling (e.g., use of gloves and completely dry specimens) the specimens and using minimum amount of adhesive tapes, conductive paints, embedding agents, and resins. Size of outgassing biological or hydrocarbon volatile specimens should be kept to a minimum.

#### 4.15 How to combat beam radiation damage during microscopy of sensitive specimens?

Answer:

Use low accelerating voltage, decrease probe current, reduce exposure time, use low magnifications/large scan areas, and apply conductive coatings such as of gold, carbon, etc. at the specimen surface to improve thermal conductivity.

4.16 How does EMI present itself? How can it be reduced?

Answer:

EMI results in image distortion usually at high magnifications. Edges of objects appear jagged. EMI is reduced by installing screening cage and EMI cancellers that generate electromagnetic radiation of the similar magnitude but in the opposite direction.

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## Chapter 5

5.1 Why is low-voltage imaging beneficial?

Answer:

As the beam energy is lowered, the specimen interaction volume decreases sharply resulting in a high-resolution high-contrast SE and BSE signal emanating from fine features close to the specimen surface giving rise to images with greater surface detail. Due to the small interaction volume generated at low kV, both SE and BSE signals produced are of a high spatial resolution giving rise to stronger image contrast. A high-resolution image is obtained by separating the high-resolution SE and BSE signals from low-resolution SE and BSE signals, respectively. Production of low-resolution signals such as SE<sub>2</sub>, SE<sub>3</sub>, and BSE farther away from the probe is eliminated. Effects of specimen charging and edge brightness are also reduced at low beam energies. This imaging technique is also suitable for beam-sensitive specimens as it minimizes radiation damage.

5.2 State the challenges to low-voltage microscopy. What can be done to encounter these?

Answer:

Boersch effect (defocusing at crossover), decreased gun brightness, increased chromatic aberration, increased diffraction at the aperture, and contamination buildup relative to the low depths from which the signals are generated. Low energy beams are also susceptible to electromagnetic interference effects. If the beam current and gun brightness are kept constant, operation at low kV results in a significantly larger spot size resulting in decreased resolution.

Microscopes equipped with high brightness source with low energy spread and an immersion lens help to maintain reasonable image contrast. It is advisable to employ short working distance during imaging at low voltages to mitigate the effects of lens aberration and any extraneous electromagnetic field present in the work environment. The rate of contamination buildup can be reduced by avoiding high magnification, focusing and removing astigmatism in an area other than that used for imaging and not using spot or reduced area raster mode. Clean specimen chamber with a high-quality vacuum system, stable and vibration-free platform,

and proper shielding from electromagnetic influences has enabled imaging at a few tens of volts.

### 5.3 What is energy filtering and how is it useful?

Answer:

Electron energy filtering separates the low-energy SE from the high-energy SE and BSE. The final image can be selected to be composed of mainly SE or BSE or combination of both depending on the detected signal. Low-energy SE can be rejected or its detection can be controlled in combination with BSE by regulating the extent of negative bias on the control electrode. Low-energy SE are primarily responsible for charging effects. The ability to filter low-energy from the high-energy signal enables better control during low voltage imaging.

### 5.4 How does beam deceleration work and what is its benefit?

Answer:

In beam deceleration technique, the electron beam is kept at high energy as it passes through the SEM column. Once it exits the final lens, the beam is decelerated before it strikes the specimen surface. Beam deceleration is accomplished by applying a negative bias (up to  $-4$  kV) to the stage which sets up an electrical field between the specimen and the detector, acting as an additional electrostatic lens working to retard the beam accelerating voltage immediately before it hits the specimen. The energy with which the beam lands onto the specimen surface is known as landing energy and is equal to accelerating voltage minus stage bias.

By maintaining the beam at high energy during its movement through the column and lenses, large energy spread, *Boersch* effect, and chromatic aberrations are avoided. The beam lands on the specimen surface with lesser energy which serves to reduce beam penetration and interaction volume. With this technique, greater flexibility in the selection of beam voltages becomes available. It enables detection of electrons scattered at shallow depths emphasizing its surface features. It improves microscope resolution and contrast at low accelerating voltages. Beam deceleration is a relatively simple technique that can be incorporated within the existing electron sources and columns eliminating the need for a separate SEM system.

### 5.5 What purpose does imaging at low vacuum mode serve?

Answer:

SEM in low vacuum mode serves to analyze damp, dirty, or insulating samples. High vacuum is unsuitable for these sample types since they tend to charge up or degas.

5.6 Describe the working principle of low vacuum mode.

Answer:

In low vacuum mode, gas or water vapor is injected into the specimen chamber around the specimen surface area. High-energy electron beam penetrates the water vapor with some scatter and interacts with the specimen surface. Secondary and backscattered electrons emanating from the specimen strike the water molecules and produce secondary electrons which in turn produce more secondary electrons upon interaction with the surrounding water molecules. Water molecules are changed into positive ions as a result of this interaction with incident beam and secondary/backscattered electrons emerging from the specimen. Positive bias applied to a detector accelerates secondary electrons toward the detector, while positive ions are pushed toward the negatively charged areas on the specimen. The water vapor thus serves to produce positive ions and also increase the number of secondary electrons resulting in gas amplification. Generation of positive ions and their movement toward the negatively charged areas of the sample neutralizes the negative charge accumulated at the specimen surface.

5.7 Why is SE detector not used in low vacuum mode generally?

Answer:

Secondary electron image is usually not available in low vacuum mode because SE interact with the water vapors immediately upon emitting from the specimen. Also, conventional E-T detector relies on a high bias (+10 kV) applied upon the scintillator to enable electron-to-photon conversion. Such high bias can easily ionize water vapor and arc the detector to ground. Thus, the use of E-T detector for SE imaging is ruled out, and BSD is generally employed to undertake BS imaging.

5.8 Is there a way to undertake SE imaging in low vacuum mode?

Answer:

Gaseous secondary electron detector (GSED) is used in low vacuum mode. GSED is a positively biased electrode which is mounted on the objective pole piece and can be dismounted after use. The positive bias of up to +600 V is applied on the GSED to attract secondary electrons. Due to gas amplification caused by collisions with water vapor, the current collected by the detector is hundreds or even thousands time greater than the original signal. The detector is also placed closer (a few mm) to the specimen compared to an E-T detector, thus collecting SE efficiently. Positive bias on GSED also drives positively ionized water molecules toward the specimen to effectively neutralize the accumulated negative charge at specimen surface.

5.9 How does degraded vacuum in the chamber (in low vacuum mode) not affect the vacuum in the column?

Answer:

A conical pressure limiting aperture is provided at the center of the detector to sustain low vacuum in the chamber while maintaining a high vacuum in the electron column. The smaller the bore size in the center of the GSED, the higher the pressure that can be maintained in the specimen chamber. For instance, one-half of a millimeter can support a pressure of 1.3 kPa.

5.10 What is the beam skirt and how its effect can be diminished?

Answer:

Electron beam on its way to the specimen gets scattered due to the presence of water vapors in low vacuum mode. The scattered electrons move away from the focused beam and strike the specimen surface at a point away from the probe. This results in broadening of the electron beam which takes on a *skirt-like* form.

This scattering effect can be reduced by employing an extension tube with pressure-limiting aperture mounted at the end. This long tube is fitted to the objective pole piece. Electrons enter this tube after emanating from the objective lens assembly. In this manner, the distance (gas path length) that the electrons have to travel in gas vapor is reduced, resulting in less scatter.

5.11 How is FIB used to sputter and deposit materials? How is imaging performed using FIB?

Answer:

The focused ion beam (FIB) is an instrument that uses positively charged heavy ions (such as gallium instead of electrons) to raster the specimen surface. Ions are heavier than electrons and carry a greater momentum. The use of heavy ions makes it easier to remove material from the specimen. Therefore, FIB is used for sputtering, etching, or micromachining of materials. It is also useful for milling, deposition, and ablation of materials.

Different gases can be injected into the system near the surface of the specimen to deposit required materials on the sample surface.

When the focused ion beam interacts with the surface of the material, it results in the generation of secondary ions, secondary electrons, and neutral atoms. Information from secondary electrons and secondary ions help in the formation of an image in the same manner as that in the SEM. The resolution of the image can be as high as 5 nm.

5.12 What is the difference between the bright field and HAADF contrast in the STEM?

Answer:

The bright field image is formed by collecting electrons that are scattered at small angles and are centered on the optic axis of the microscope while passing through



the specimen (e.g., *direct beam*). The *incoherent* dark-field image is formed by (off-axis) electrons scattered at high angles and shows atomic number and mass-thickness contrast. The detector that collects the strongly scattered electrons to form a high STEM image contrast is called high-angle annular dark-field (HAADF) detector.

5.13 Which detector in the SEM can be used for structural analysis?

Answer:

Electron backscatter diffraction (EBSD) detector

5.14 How is EBSD used to obtain structural information?

Answer:

When electron beam strikes its surface, specimen acts as a divergent source of high-energy backscattered electrons which are incident upon sets of parallel lattice planes present within the crystal and are scattered in a manner that satisfies the Bragg's equation. This type of scattering is termed as electron diffraction. For each set of lattice planes for which the above Bragg condition is fulfilled, the diffracted beams emerge out of the specimen in all directions in the form of a cone. Two cones are formed for each set of lattice planes, one at the front and the second at the rear of the lattice plane. These cones intersect the phosphor screen as two dark lines bordering a bright band which represent a family of parallel planes with a specific value of  $d$ -spacing. The distance between two lines is inversely proportional to the  $d$ -spacing for that specific plane. From this information, the lattice parameter of the specimen material can be determined.

5.15 State the applications of E-beam lithography.

Answer:

Electron beam lithography is the commonly used method for precise patterning in nanotechnology. Generally, it could be used in the nanoelectromechanical system (NEMS), quantum structures, magnetic devices, solid-state physics, biotechnology, and transport mechanisms. It is used in the fabrication of many functional devices and products such as IC fabrication mask, nano-transistors, nano-sensors, and biological applications such as biomolecular motor-powered devices.

5.16 How does EBID work?

Answer:

Electron beam-induced deposition (EBID) is a process of decomposing gaseous molecules by an electron beam leading to deposition of nonvolatile fragments onto a nearby substrate. The electron beam is typically provided by scanning electron microscope (SEM), which brings about high spatial resolution and most likely to generate free-standing, three-dimensional structures. The electron beam

interacts with the material resulting in the emission of secondary electrons which in turn decompose molecular bonds of precursor gaseous materials resulting in deposition.

#### 5.17 What is cathodoluminescence?

Answer:

A particular class of materials can emit light (photons of characteristic wavelengths in ultraviolet, visible, and infrared ranges) when bombarded with an electron beam in the SEM. This phenomenon is known as cathodoluminescence (CL) which occurs when atoms in a material excited by high-energy electrons in the beam return to their ground state thus emitting light.

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## Chapter 6

### 6.1 Distinguish between characteristic and continuous x-rays.

Answer:

Primary electron beam ejects inner shell electron of specimen atom creating a vacancy in the orbital and turns the atom into an ion of the excited state. This vacancy is filled when an outer shell electron is transferred to the inner shell, which brings the atom to its ground state with an accompanying release of energy equal to the difference in the binding energy of the two shells. This excessive energy is released in the form of an x-ray photon. Characteristic x-rays have sharply defined energy values and occupy distinct energy positions in the x-ray spectrum which are unique to the element they emanate from. Distinct energy positions of x-ray lines form the basis for microchemical analysis where different elements in a specimen material are identified based on unique orbital transition energy.

Primary electron beam also decelerates (brakes) due to interaction with atomic nuclei of the specimen. The energy loss due to deceleration is emitted as photons of energy ranging from zero to the maximum energy supplied by incident electrons thereby forming a continuous electromagnetic spectrum called *continuum* or *white radiation* or *bremsstrahlung* (*braking radiation*). Continuum is generated due to a combination of all atoms in a specimen and appears as background in an x-ray spectrum. Since it is not unique to a particular element, it is devoid of any unique feature and cannot be used for microchemical analysis.

### 6.2 What is the notation used for characteristic x-rays?

Answer:

X-ray lines are denoted by the shell from where the electron was originally ejected (i.e., shell of innermost vacancy) such as K, L, M, etc. This is followed by a line

group written as  $\alpha$ ,  $\beta$ , etc. If the transition of electrons is from L to K shell, transition line is designated as  $K_\alpha$ . If the transition is from M to K shell, it is designated as  $K_\beta$ . Since the energy difference between K and M is larger than that between K and L,  $K_\beta$  is of higher energy than  $K_\alpha$ . Lastly, a number is written to signify the intensity of the line in descending order such as 1, 2, etc. Therefore, the most intense K line is written as  $K_{\alpha 1}$ , and the most intense L line is denoted as  $L_{\alpha 1}$ .

### 6.3 What is the significance of Moseley's law?

Answer:

In 1913, the English physicist Henry Moseley discovered that when the atomic number changes by one, the energy difference between the shells varies in a regular step. The energy of photon can be given by Moseley's law below:

$$E = A(Z - C)^2$$

where  $E$  is the energy of the x-ray line,  $Z$  is the atomic number, and  $A$  and  $C$  are constants with specific values for K, L, M, etc., shells. This forms the basis for identification of elements in materials using x-rays. The above relationship describes energy required to excite any series of transition lines. For instance, x-ray photons of the highest energy in an atom are emitted from  $K_\alpha$  shells. This energy equals the binding energy of 1s electron which in turn is proportional to  $Z^2$  as described above. This energy will be different for each element (depending on its atomic number) and can be used to identify it.

### 6.4 What is the excitation potential?

Answer:

The minimum energy required to eject an electron from an atomic shell is known as excitation potential ( $E_c$ ). As the size of the atom increases (e.g., from light to heavy elements), the energy required to excite any particular transition line also increases. For instance,  $E_c$  for Ni  $K_\alpha$  is much higher than that for Al  $K_\alpha$ . The excitation potential of K shell is higher than other shells. In addition, the excitation potential of K shell increases extensively with a small increase in the atomic number.

### 6.5 Define cross-section of inner shell ionization.

Answer:

It is the probability for an incident beam electron to be scattered inelastically by an atom per unit solid angle  $\Omega$ . It is denoted by  $\sigma$  or  $Q$ . The cross section decreases as the primary electron energy  $E_0$  increases. Also, it is lower for elements with the higher atomic number since the critical excitation energy increases with  $Z$ .

6.6 What is the x-ray range?

Answer:

X-ray range is the depth of x-ray production within the interaction volume. It mainly depends on the beam energy, critical excitation energy, and the specimen density.

6.7 What is x-ray spatial resolution?

Answer:

X-ray spatial resolution is defined as the maximum width of the interaction volume generated by electrons or x-rays projected up to the specimen surface. The low atomic number and low-density specimens allow deeper and wider electron beam penetration and generation of x-ray lines which degrades the x-ray spatial resolution achieved.

6.8 Why is mass depth used instead of linear depth?

Answer:

The use of the mass depth term  $\rho z$  is more common than the use of linear depth term  $z$  because the mass depth eliminates the need for distinguishing different materials because of their different densities when illustrating the relation with the depth distribution  $\varphi(\rho z)$ .

6.9 What is the primary mechanism of x-ray absorption?

Answer:

X-rays are primarily absorbed in specimen material by photoelectric absorption. An x-ray photon loses all its energy to an orbital electron, which is ejected with a kinetic energy equal to the difference in photon energy and critical ionization energy required to eject the electron.

6.10 An EDS detector has beryllium (Be) window which is 8- $\mu\text{m}$  in thickness.

Calculate how much fluorine  $K_{\alpha}$  radiation can pass through this window onward to be detected by the detector. The mass absorption coefficient for F  $K_{\alpha}$  radiation in Be is 2039  $\text{cm}^2/\text{g}$  [1].

Answer:

$$I = I_0 \exp \left[ - \left( \frac{\mu}{\rho} \right) (\rho t) \right]$$

$$\frac{I}{I_0} = \exp \left[ - \left( 2039 \frac{\text{cm}^2}{\text{g}} \right) \left( 1.848 \frac{\text{g}}{\text{cm}^3} \right) (8 \times 10^{-4} \text{cm}) \right]$$

$$\frac{I}{I_0} = 0.05 = 5\%$$

Only 5% of the F  $K_\alpha$  radiation will pass through the Be window used in this example. It is therefore not practical to employ such a window in an EDS detector to detect fluorine  $K_\alpha$  radiation.

6.11 What is secondary fluorescence and what is its significance?

Answer:

When primary electron beam penetrates a specimen, it ionizes atoms to generate characteristic x-ray photons. These photons, while they are out of the specimen, may interact with other specimen atoms to cause secondary ionization, resulting in the generation of additional characteristic x-rays or Auger electrons. The process by which x-rays are emitted as a result of interaction with other x-rays is called *secondary x-ray fluorescence*.

X-ray fluorescence can complicate quantification of elemental concentrations present within specimen material. For example, the  $K_\alpha$  x-ray of Cu element has a value of 8.05 keV, and it can be generated by  $K_\alpha$  x-ray of Zn that exists in a brass sample. In 70Cu-30Zn alloy, more than expected Cu  $K_\alpha$  and less than anticipated Zn  $K_\alpha$  x-rays will be generated due to the fluorescence effects. In this way, Cu will be overrepresented, and Zn will be underreported unless corrections are made to the calculations. X-ray fluorescence acquires importance in alloys that have elements with similar  $Z$  because it affects the relative amount of characteristic x-rays emanating from compounds.

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## Chapter 7

7.1 List advantages of an EDS detector.

Answer:

An EDS detector is simple, robust, versatile, and easy to use and does not take up a large amount of space. Its functionality is seamlessly integrated into SEM operation. It undertakes a simultaneous analysis of all elements.

7.2 What is the energy resolution of the EDS detector? How is it measured?

Answer:

The ability to distinguish between peaks in the EDS spectrum is the energy resolution of the EDS detector. It is usually measured at FWHM using Mn $K_\alpha$  (5.9 keV) as the reference peak. The energy resolution of modern EDS detector is approx. 122 eV.

7.3 What can be done to improve x-ray collection efficiency for EDS?

Answer:

The detector is placed close to the sample. Detector with a large surface area is used.

7.4 What is the purpose of a window in the EDS detector?

Answer:

The window is used to protect the detector from the SEM environment. It has the adequate mechanical strength to withstand pressure variations inside the SEM chamber during specimen exchange. It also protects the detector crystal surface from visible radiation. It also prevents any contaminants to move into the detector which functions in vacuum at low temperatures.

7.5 Describe shortly the history of the development of the EDS window.

Answer:

Until 1982, the only available window was made of beryllium usually around 8  $\mu\text{m}$  in thickness. This window would absorb x-rays of energy less than 1 keV, thus preventing the detection of light elements such as boron, oxygen, nitrogen, carbon, etc. To enable light element detection, an "ultrathin" window (UTW) made of thin (tens to a few hundred nanometers) organic film Formvar coated with gold was used instead of beryllium. This window is unable to withstand atmospheric pressure, and the detector assembly is kept under vacuum. The window can be removed altogether, and the detector can be used in a "windowless" mode. However, this leaves the detector exposed to contamination. In this situation, if the SEM chamber is vented, hydrocarbon condensation and ice formation will occur on the detector surface. The light will also be transmitted onto the semiconductor surface.

Presently, the ultrathin window of polymer covered with a thin layer of evaporated Al and supported with Si grid at the detector side is used as a standard. Due to grid support, the window is able to withstand the pressure of 1 bar in the SEM chamber. Support structure blocks part of the low-energy radiation thus reducing the detector efficiency to some extent. The grids are therefore designed to have up to 80% area available for x-ray transmission. This type of window can transmit low-energy ( $\approx 100$  eV) x-rays and is a preferred choice for light element analysis. Evaporated Al coating serves to restrict the passage of light through the polymeric material which otherwise exhibits high optical transparency. Modern EDS detectors routinely detect elements from beryllium to uranium.

7.6 Explain how EDS detector works.

Answer:

X-ray photons striking the detector surface ionizes Si atom through photoelectric effect creating electron-hole pairs. Upon application of a bias voltage between the

thin gold contacts present at opposite ends of the semiconductor, these electrons and holes move in opposite directions toward the collection electrodes creating a charge pulse. The mean energy required to create one electron-hole pair (one electric pulse) in undoped Si is taken as 3.86 eV. The number of charge pulses generated in the detector can be counted, and the x-ray photon energy responsible for this pulse output is calculated by multiplying this number by 3.86. For instance, if the pulse output count is 1642, the x-ray energy that would produce such a number will be  $1659 \times 3.86 = 6403$  eV or 6.4 keV. This energy corresponds to  $K_{\alpha}$  x-ray line which is emitted when an electron transitions from L to K shell in the Fe atom. The energy value is fixed for this particular transition, and thus whenever a magnitude of pulse equaling the number of 1659 is measured, Fe is identified as a possible constituent of the specimen under examination. The greater the number of times this particular value of pulse count is generated, the higher is the elemental concentration of Fe in the material.

7.7 What is the dead time?

Answer:

EDS detector's capacity to receive and process x-ray photons is not unlimited. While one x-ray event is received and processed, other simultaneous incoming x-rays are not processed. The duration for which these x-ray signals are not processed is known as "dead time."

7.8 List four overlapping peaks in the EDS spectrum.

Answer:

$SK_{\alpha,\beta}$ - $MoL_{\alpha}$ ,  $CrK_{\beta}$ - $FeK_{\alpha}$ ,  $MnK_{\alpha}$ - $CrK_{\beta}$ , and  $TaM_{\alpha,\beta}$ - $SiK_{\alpha,\beta}$

7.9 How does escape peak form?

Answer:

X-ray photons emanating from specimen enter the detector and ionize Si releasing K-type x-ray photons. If this transition occurs closer to the detector surface, the photons can escape the detector. This will decrease the energy of the x-rays emanating from the specimen by an amount equal to that required for Si K transition event. Due to this event, an escape peak is generated in the x-ray spectrum at energy  $E_{(\text{specimen})} - E_{SiK_{\alpha}}$ . For instance, if Cu is the specimen material tested, an escape peak at  $8.04 (E_{CuK_{\alpha}}) - 1.74 (E_{SiK_{\alpha}}) = 6.3$  keV can form in the x-ray spectrum

7.10 What is Castaing's first approximation?

Answer:

Castaing's first approximation assumes that peak intensities in eds spectrum are generated proportional to the respective concentrations of elements.

$$\frac{C_{i \text{ (unknown)}}}{C_{i \text{ (standard)}}} \approx \frac{I_{i \text{ (unknown)}}}{I_{i \text{ (standard)}}} = k_i$$

### 7.11 What is matrix effects?

Answer:

Measured or detected intensity of x-rays is not equal to generated intensity due to absorption or fluorescence of x-rays generated within the specimen. This variation between generated and detected values of x-ray intensity is governed by the composition of the specimen matrix and is known as matrix effects.

#### Atomic Number Effect

Consider measuring the low concentration of a light element  $i$  mixed with a high concentration of a heavy element  $j$  in a multielement specimen. A large proportion of beam electrons entering the specimen shall be backscattered by heavy element  $j$  and leave the specimen. These electrons shall be unavailable to generate x-rays from within the light element  $i$ . In this way, the concentration of  $i$  shall be underestimated if its intensity is compared with that originating from a standard with 100% pure element  $i$ . This is called the atomic number effect and needs to be corrected.

#### Absorption Effect

The greater the depth at which x-rays are generated, the greater is the proportion that is lost due to absorption within the specimen. Mass absorption coefficient depends on the composition of specimen analyzed. Generally, correction for mass absorption is the biggest correction made during quantitative microchemical x-ray analysis. Especially, light elements such as C, N, and O are strongly absorbed in heavy matrices and need to be accounted for in calculations. Absorption can be decreased by using the minimum incident beam energy required to generate characteristic x-rays resulting in lesser beam penetration and lower path lengths ( $t$ ) that x-rays need to traverse to reach specimen surface.

#### Fluorescence Effect

Characteristic x-rays generated as a result of the interaction between the electron beam and the specimen can be absorbed within the specimen matrix and cause ionization of atoms resulting in the emission of further characteristic x-rays. This fluorescent effect takes place only if the critical excitation energy of absorbing atoms is less than the energy of generated x-rays. This effect will result in an increase in the measured x-ray intensity by the SEM detector since now both the original x-rays as well as the x-rays generated due to fluorescence are measured. Correction required due to fluorescence effect is usually smaller compared to that for atomic number and absorption in ZAF corrections. In some cases, fluorescence can result in erroneous peaks in the x-ray spectrum.



7.12 State the working principle of WDS technique.

Answer:

The sample, crystal, and the detector are positioned on a circle (called Rowland circle). The sample is located at the bottom of the specimen chamber. The crystal and the detector are made to move on the Rowland circle during analysis. During this movement, the distance between the crystal and the specimen is always kept equal to the distance between the crystal and the detector. The characteristic x-rays with specific wavelength emanate from the constituent elements of the sample due to orbital transitions and strike the surface of the crystal that has a fixed  $d$ -spacing. During the course of crystal movement, it is probable that x-rays (with specific  $\lambda$ ) emanating from a particular element in the specimen and upon striking the crystal (with fixed  $d$ ) at an angle  $\theta$  satisfy Bragg's equation resulting in diffraction. Upon diffraction, the amplitude of the x-rays will increase manifold resulting in an increase in the intensity of the x-rays at the diffraction angle  $\theta$ . This increase in intensity is measured by the detector and appears as a peak in the x-ray spectrum recorded in the computer. The movement of the crystal is precisely controlled and monitored thereby providing an exact measure of  $\theta$  at the time of diffraction. The  $d$  value is identified as the crystal used is known. From Bragg's equation,  $\lambda$  is then measured. Working backward, element emanating x-ray with this particular wavelength is identified as the latter is specific to that element.

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## Chapter 8

8.1 What is meant by sampling?

Answer:

Sampling involves identifying and extracting a suitable area in the sample that adequately represents its structure, morphology, and chemistry. Any sample preparation method should not damage the sample or contaminate it. The sample should be transported to the SEM in a box or wrapped up in a dry material. It should be labeled and stored properly.

8.2 Why certain liquids are used during sectioning?

Answer:

Liquids are used for cooling and lubrication during sectioning.

8.3 How is grinding different than polishing?

Answer:

Mechanical grinding is carried out using a series of abrasive materials such as SiC from rough to fine grit size (e.g., 120-, 240- to 400-, and 600-grit papers).

Grinding eliminates material rapidly and reduces the damage caused by the sectioning of the specimen. By the end of grinding process, any cutting marks or scratches would have disappeared from the surface. However, grinding introduces its own thin layer of damage to the surface regions.

The polishing process uses loose abrasive (e.g., diamond paste,  $\text{Al}_2\text{O}_3$ , SiC, c-BN, etc.) between the specimen and hard fabric (e.g., nylon) fixed on a rotating wheel. The rubbing action results in abrasion of the specimen. Polishing is undertaken to impart a flat scratch-free mirrorlike (up to 1 mm) finish to the surface of the specimen. Polishing process with fine grain sizes eliminates only a minor amount of material. After polishing, the sample can be observed in the SEM.

8.4 What is the purpose of impregnation during sample preparation of ceramics and geological materials?

Answer:

The aim of impregnation is to reduce pullouts and ease sample preparation in ceramics. This way the degree of porosity in a sample can be accurately determined. In geological materials, impregnation with low viscous resin is used to provide the necessary mechanical strength to fragile materials in order to withstand preparation procedure. Furthermore, the porous material is filled to avoid entrapment of polishing materials which degas in vacuum later.

8.5 Why is sputter coating process preferred for imaging? Why is C coating suitable for elemental analysis?

Answer:

Sputter coating results in uniformly distributed fine-grained thin coatings. Carbon coating is preferred during chemical analysis since it does not interfere with different elemental peaks in an EDS spectrum.

8.6 What is the difference between dry and wet potting?

Answer:

Dry potting is used for specimens that are dried before preparation. In this way, cracks caused by drying shrinkage no longer remain a concern. Moreover, this method is used when specimens need to be prepared in a short period of time. Wet potting is used to prepare sections after polishing when the material is not dry. In this way, cracking due to drying shrinkage does not occur because the material is still wet. If cracks are observed in the material, it will be due to physical or chemical processes.

8.7 Why are polymers difficult to image? What can be done to overcome these difficulties?

Answer:

Challenges faced during imaging of polymers include beam damage due to radiation sensitivity, charging, beam contamination, and low contrast. Chemical analysis using EDS/WDS is also difficult since polymers are prone to damage at high probe currents.

Contrast can be enhanced with etching, staining, and replication of polymers. The use of conductive tape and conductive coating allows for electrons dissipation and minimizes beam damage. Replication is also used to completely avoid beam damage especially for highly sensitive samples, whereby a replica is scanned by SEM instead of the sensitive sample.

8.8 What is the difference between microtome and ultramicrotome?

Answer:

Microtome can produce samples of 5  $\mu\text{m}$  in thickness, while ultramicrotome can produce thin slices of 200 nm.

8.9 How is a polymer replica prepared?

Answer:

The specimen is placed onto a thick tape, and a mixture of silicone and a curing agent is poured over the polymer. After curing, the mixture is peeled off and observed in the SEM.

8.10 What is staining of polymers?

Answer:

Staining involves the introduction of heavy atoms into the polymeric sample so as to enhance its density and hence the contrast. Staining can be undertaken by physical or chemical means.

8.11 Why is it difficult to examine biological materials in the SEM?

Answer:

Biological samples are difficult to examine due to their nonuniform structure, organic makeup, and their water content. Radiation damage can occur in the sample. Basic steps involved in the preparation of biological materials are fixation, drying, and coating.

8.12 Explain fixation in biological samples.

Answer:

Fixation is used to stabilize and prevent the structure of the biological sample from decomposition in order to preserve its nature during analysis. This process involves several important stages such as prevention of decomposition of a sample by enzymes activities and removal of the water content of a sample through dehydration processes. Common fixation methods have been divided into two major groups including chemical fixation and physical fixation.

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# Index

## A

- Abbe's equation, 51, 129, 133
- Accelerating voltage, 2, 5, 8, 9, 11, 17, 18, 20, 23, 27, 30, 33, 37, 39, 45, 46, 50–52, 66, 87, 124, 125, 133, 137, 139, 147, 149, 160, 161, 163, 164, 167–169, 172, 177, 181–183, 185, 186, 189, 192, 194, 206, 208–211, 218, 222, 250, 251, 261, 273, 291, 295, 296, 298, 299, 301, 322
- Apertures, 2, 15, 17, 18, 22, 24, 26, 29, 31, 37, 39, 41, 42, 45, 47, 48, 50, 51, 63, 65, 86, 117, 129, 133, 135, 138, 139, 142, 143, 154, 157, 159, 160, 167, 172, 176–179, 182, 190–193, 196, 197, 202, 204, 207, 220, 267, 301
- Atom model, 86–87, 233–234
- Atomic number ( $Z$ ) contrast, 9, 95, 97–112

## B

- Backscattered electron detector
  - advantages, 69, 70
  - channel plate detector, 71
  - collection efficiency, 66, 70, 71, 99, 101, 104, 105, 112
  - drawbacks, 69, 70
  - scintillator BSE detector, 71
  - SSD, 66, 70
  - working principle, 66–69
- Backscattered electron imaging
  - applications, 111–112
  - COMPO, 108, 111
  - limitations, 112
  - SHADOW, 108
  - TOPO, 108, 111
- Backscattered electrons
  - energy distribution, 98
  - origin, 68, 95, 190
  - spatial distribution, 106

- spatial resolution, 110
- yield, 97
- Backscattered electron yield
  - directional dependence, 101–103
  - effect of atomic number, 98–100
  - effect of beam energy, 98
  - effect of crystal structure, 101
  - effect of tilt, 101–102
- Beam damage, 8, 149, 168, 171, 175, 208, 273, 289, 299, 338, 346, 349
- Beam energy, 22, 27, 34, 46, 51, 64, 66, 71, 77, 88, 90–91, 94, 95, 97–99, 105, 110, 116, 118–120, 122, 133–136, 139, 140, 147, 164, 169, 171, 181, 182, 186, 194, 198, 208, 238, 239, 246–252, 254, 261, 263, 273, 275, 284, 289, 291–293, 298, 299, 322
- Boersch* effect, 182, 186
- Bright field (BF), 207–209

## C

- Cathodoluminescence, 13, 61, 123, 175, 181, 230
- CeB<sub>6</sub> emitter, 30
- Charging
  - mechanism, 224
  - methods to reduce, 166
- Cold field emitter
  - advantages, 34
  - drawbacks, 34
  - service lifetime, 33–34
  - working principle, 32–33
- Compositional contrast, 7, 9, 77, 95, 97, 106, 109, 111, 182
- Computer control, 4, 15, 16, 71, 72, 202, 210, 225
- Condenser lens
  - beam current, 39, 52, 189

- Condenser lens (*cont.*)  
demagnification, 37, 39, 40  
spot size, 36, 39, 40, 52, 138, 175, 189
- Contamination, 20, 27, 29, 30, 33, 34, 36, 41, 73, 75, 79, 120, 137, 138, 146, 166, 170, 171, 173, 174, 178, 182, 190, 224, 267, 271, 273, 299
- Contrast formation  
COMPO, 108, 111  
compositional contrast, 7, 9, 77, 95, 97, 106, 109, 111, 182  
SHADOW, 108  
TOPO, 108, 111  
Z contrast, 99, 100, 106–110, 112
- D**
- Dark field, 206–209
- Depth of field, vii, 6, 8, 31, 43, 129, 136, 146, 152–154, 156, 157, 171, 187, 347, 349
- Digital imaging  
advantages, 133, 134  
picture element, 78, 82, 83, 134, 143  
pixel, 80–82
- Directly heated cathode, 23, 28
- Dwell time, 52, 53, 79, 165, 175, 285, 287
- E**
- Edge effect, 124–125, 146, 147, 169–171
- Effective probe diameter, 50–52
- Elastic scattering, 86, 87, 89–91, 94, 95, 97, 98, 106, 113, 116, 122, 167, 204, 246–248, 250, 253, 256, 258, 291, 335
- Electromagnetic interference (EMI), 76, 138, 169, 182
- Electromagnetic lens  
design, 37  
magnetic field, 37, 38, 41, 42  
material, 42  
working principle, 38
- Electron backscatter diffraction  
Bragg's equation, 211, 212, 303  
brief history, 211  
crystallography, 210, 215, 217  
crystal orientation, 213, 216  
diffraction, 9, 210, 211, 215, 218  
electron backscatter pattern, 211, 215  
Euler map, 216  
experimental set-up, 213–215  
grain map, 213  
grain shape, 9, 210, 213, 215  
inverse pole figure maps, 216  
Kikuchi pattern, 210, 213–215  
Kossel diffraction, 211  
orientation map, 217, 218  
phase distribution, 9, 334  
selected area channeling pattern, 211  
texture, 210, 216, 227  
transmission EBSD, 218  
transmission Kikuchi diffraction, 218  
working principle, 212
- Electron beam-induced deposition (EBID), 181, 225
- Electron beam lithography  
computer-aided design, 221  
electron beam resist, 222  
imprint lithography, 219  
lift-off, etching, 222, 223  
optical lithography, 219  
pattern design, 222  
raster scanning, 221  
vector scanning, 221  
working principle, 222
- Electron column, 4, 15–20, 24, 34, 37, 39, 52, 56, 59, 72, 74, 75, 159, 190, 192, 193, 301
- Electron range, 93–94, 98, 105, 116, 136, 140, 250–252
- Electron source/gun  
brightness, 17–18  
energy spread, 20  
size, 19  
stability, 20
- Energy dispersive x-ray spectroscopy  
absorption effect, 293  
advantages, 272  
atomic number effect, 291–292  
background correction, 275  
Be window, 259, 267  
Castaing's first approximation, 290–291, BNF–290  
dead time, 276  
drawbacks of, 272  
EDS detector, 11, 58, 60, 238, 259, 265–268, 270–272, 276–278  
EDS resolution, 277  
escape peaks, 281–282  
field-assisted transistor, 34  
first principles standards analysis, 298  
fitted standards standardless analysis, 298  
fluorescence effect, 293–294  
full width half maximum, 276  
full width tenth maximum, 277  
internal fluorescence peak, 283  
line scan, 285–288

- low-voltage EDS, 299–300
  - matrix effects, 291–294
  - minimum detectability limit, BNF–300
  - multichannel x-ray analyzer, 271
  - overlapping peaks, 278
  - peak acquisition, 273
  - peak broadening, 278–ENF
  - peak distortion, 278–279
  - peak identification, 273–274
  - Phi-Rho-Zi correction, 295
  - pulse height analysis, 270
  - qualitative analysis, 265, 273
  - quantitative analysis, 261, 265, 290, 294, 296, 299, 300
  - sensitivity, 265, 272, 275, 299, 300
  - spot analysis, 53, 284
  - standardless EDS analysis, 296–299
  - sum peaks, 282–ENF
  - working principle, 267–272
  - x-ray map, 284–285
  - ZAF correction, 295–296, BNF–295
- E**
- Everhart-Thornley (E-T) detector
    - advantages, 64
    - backscattered electron signal, 59, 60, 63
    - Faraday, 61
    - Faraday cage, 60, 61, 63, 122
    - lateral displacement, 126–127
    - light guide, 60, 61, 71
    - photomultiplier tube, 61, 85, 123, 228
    - scintillator, 61, 65
    - secondary electron signal, 59, 115, 124, 181
    - solid angle of collection, 62, 64, 66, 83, 106, 183, 208, 272
    - working principle, 59–62
- F**
- Field emission electron gun
    - advantages, 31, 32
    - drawbacks, 31, 32
    - quantum tunneling, 31
    - working principle, 30–31
  - Focused ion beam
    - detector, 204
    - electrospray technique, 202, 203
    - gas field ion source, 203
    - instrumentation, 204
    - introduction, 199
    - ion imaging, 205
    - ion-solid interaction, 205
    - ion sources, 203
    - lens, 204
- liquid metal ion source, 203
  - stage, 204
  - Taylor cone, 202, 203
  - volume plasma source, 203
- G**
- Gas path length, 196
  - Gaseous secondary electron detector (GSED), 192, 193
- H**
- Heater, 28, 71, 74, 75
  - High-angle annular dark field (HAADF) detector, 207, 209
  - High-resolution imaging, 31, 42, 45, 64, 75, 135, 140, 147, 160, 163, 170, 174, 175, 321
  - High-tension (HT) tank, 74, 170
  - History of the SEM, 12
- I**
- Indirectly heated cathode, 28
  - Inelastic scattering, 86, 88–90, 94, 95, 106, 113, 114, 116, 122, 167, 204, 246–248, 250, 256, 258, 291, 335
  - Infrared (IR) camera, 58
  - Interaction volume
    - effect of atomic number, 91–92
    - effect of beam energy, 90–91
    - effect of tilt, 93
  - Ion beam-induced deposition (IBID), 224
  - Ion imaging, 205
- L**
- LaB<sub>6</sub> emitter
    - advantages, 28–29
    - drawbacks, 30
    - flats, 28, 29
    - material, BNF–29
    - tip design, 29
  - Lens aberrations
    - astigmatism, 48–50
    - chromatic aberration, 45–47
    - diffraction at aperture, 47–48
    - spherical aberration, BNF–45
    - stigmator, 48, 160
  - Light microscopes, 1, 2, 6, 11, 51, 144, 310, 350, 352

Limitations of the SEM, vii, 8

Low vacuum

gas path length, 196

history, 191

introduction, 189

low vacuum detector, 193

mean free path, 193

skirt, 194, 195

working principle, 192

Low voltage imaging

beam deceleration, 164

detectors, 186

E x B filter, 184

energy-filtering, 183

energy selective backscatter (EsB)

detector, 183

low angle backscattered electron (LABe)

detector, 186

r-filter, 185

upper electron detector (UED), 183

## M

Maintenance of the SEM, 168, 170, 178

Mean free path (MFP), 72, 114, 193, 261

Microchannel plate (MCP), 204

Monte Carlo simulation, 90–93, 101, 102, 253, 292

## N

Numerical aperture, 129

## O

Objective lens

immersion lens, 42

pinhole lens, 41–ENF

real aperture, 43, 160

snorkel lens, 42

virtual aperture, 43, 160

Oligo Scattering, 194

Operational parameters

accelerating voltage, 147

alignment, 159, 160

beam energy, 147

objective aperture, 157

probe current, 151

specimen tilt, 158

spot size, 151

working distance, 154

## P

Pauli exclusion principle, 233, 234

Plural scattering, 194

Pressure-limiting aperture (PLA), 190–193, 196, 197, 344

## R

Resolution limit

Buxton criterion, 132

edge resolution, 132

Houston criterion, 132

maximum spatial frequency, 133

radial intensity distribution, 132

Rayleigh criterion, 131

Schuster's criterion, 132

Sparrow criterion, 132

Richardson law, 22

## S

Safety requirements

emergency, 179

handling, 179

radiation, 179

Sample preparation

amorphous polymers, 334

bacteria, 353

cast, 325, 326

chemical fixation, 349, 350, 352

cleaning, 311

coating, 322

crystalline polymers, 335, 336, 341

drying, 174, 325

dye impregnation, 331

embedding, 312, 326

epoxy impregnation, 331

etching, 315

evaporation, 321

fixing, 316

fracturing, 316

grinding, 312

handling, 323

heart tissue, 352

impregnation, 315

insects, 354

lapping, 312

mounting, 312

physical fixation, 349, 351

polishing, 312

polymers, 173, 337, 339, 345



- replica, 325, 326
  - replication, 345
  - sampling, 310
  - sectioning, 310
  - sputter coating, 320
  - staining, 345, 351
  - stem cells, 353
  - storage, 323
  - stub, 165, 173, 174, 311, 316, 322, 323, 326
  - thermoplastics, 333
  - thermosets, 334
  - Wood's metal, 331
  - Scan coils
    - magnification, 53–56
    - raster, 4, 52, 53
    - working distance, 53–55
  - Scanning electron microscopy (SEM)
    - alignment, 177
    - image acquisition, 177
    - maintenance, 177
    - operation, vii, 31, 41, 56, 71, 72, 129, 138, 160, 178
    - sample insertion, 174
    - sample preparation, 173
    - sample size, 173
  - Schottky field emitter
    - advantages, 36
    - design, 35, 36
    - drawbacks, 36
    - material, 35, 37, 219
    - operation, 36
  - Secondary electron imaging
    - examples, 122, 130, 163, 210
    - factors that affect, 77, 124, 191
    - irregular samples, 126
    - particles, 126
    - topographic contrast, 113–127
  - Secondary electrons
    - energy distribution, 114
    - escape depth, 113, 114
    - origin, 95, 96
    - SE<sub>1</sub>, 115–117
    - SE<sub>2</sub>, 115–117
    - SE<sub>3</sub>, 115–117
    - SE<sub>4</sub>, 115–117
    - yield, 112, 113
  - Secondary electron yield
    - directional dependence, 122
    - effect of atomic number, 120
    - effect of beam energy, 116, 118–120
    - effect of tilt, 120, 121
  - Shot noise, 85
  - Signal-to-noise ratio (SNR), 41, 52, 65, 82–85, 99, 112, 117, 136–140, 150, 151, 154, 165, 207, 208, 270, 336
  - Spatial resolution
    - airy disc, 129
    - effect of accelerating voltage, 137
    - effect of beam current, 135
    - effect of convergence angle, 135
    - effect of probe size, 135
    - limiting factors, 140
  - Specimen chamber, 4, 15, 16, 24, 56, 58–61, 63, 65, 69, 71–73, 76, 77, 101, 117, 166, 174, 182, 189, 190, 193, 202, 204, 207, 211, 213, 214, 267, 301, 302, 309, 320, 324
  - Specimen stage
    - cold stage, 58, 346
    - high precision stage, 58
    - hot stage, 13, 58
  - Spot size, 29, 36, 39, 40, 51, 52, 82, 110, 131, 134–136, 138, 139, 151, 158, 171, 175, 176, 182, 189, 192, 203
  - STEM-in-SEM
    - advantages, 208
    - applications, 209
    - drawbacks, 208
    - working principle, 208
  - Strengths of the SEM, 8, 11, 116
- ## T
- Thermionic emission electron gun, 20–22, 24
  - Through-the-lens detector, 65, 66, 138
  - Topographic contrast, 7, 77, 96, 97, 100, 108, 109, 111, 113–127
  - Transmission electron microscopes (TEM), 2–4, 6, 11, 12, 37, 80, 198, 201, 206, 208, 344
  - Tungsten filament
    - advantages, 27
    - beam current, 24, 26, 29
    - bias resistor, 24–25
    - drawbacks, 27
    - emission current, 19, 22, 24, 25, 27
    - false peak, 27, 58
    - filament heating current, 24, 26
    - material, 22–23
    - saturation, 26
    - service lifetime, 27
    - work function, 20–22, 27
    - working principle, 23–24

**U**

Useful magnification, 2, 5

**V**

Vacuum system

diffusion pump, 73, 74

ion getter pump, 73

rotary pump, 73, 74, 318

turbomolecular pump, 73

Vibrations, 23, 34, 36, 71, 138, 169, 170, 182, 205, 219, 334

**W**

Water chiller, 15, 74, 75, 178

Wavelength dispersive x-ray spectroscopy

analyzing crystals, 303

diffraction, 303, 304, 328

electron probe micro analyzer, 300

Johann geometry and Johansson geometry, 303

Rowland circle, 302, 303

WDS detector, 301–304

Wobbler, 160, 177

Working distances (WD), 6, 41, 42, 44, 52–55, 58, 65, 71, 99, 112, 135–137, 139, 142, 143, 154, 169, 174, 176, 177, 182, 185, 186, 192, 194, 197, 208, 218, 227, 272

**X**

X-rays

characteristics, 237

characteristic x-rays, 4, 78, 86, 237, 238, 241, 242, 244, 250, 251, 258, 261, 263, 265, 267, 273, 282, 284, 293, 298, 303

continuous x-rays, 236–238, 240, 241, 250, 251, 276, 278, 284, 285, 301

critical excitation energy, 244–247

critical ionization energy, 245, 254, 256–259, 262

cross section of inner-shell ionization, 246–247

depth distribution profile, 253–254

Duane-Hunt Limit, 239–241

excitation potential, 244–246

fluorescence yield, 236, 237, 246, 298

Kramer's law, 239

mass absorption coefficient, 256–262, 293

Moseley's law, 244

orbital transition, 241–249

production, 234–241

secondary electron fluorescence, 261–263

short-wavelength limit, 239

x-ray absorption, 256–261

x-ray absorption edge energy, 245

x-ray absorption edges, 245, 258, 259

x-ray range, 250–252

x-ray spatial resolution, 251–252