

Image Formation and Interpretation

SEM Imaging Process

- Electron gun generates high energy electrons which are focused into a fine beam, which is scanned across the surface of the specimen.
- Elastically and inelastic interactions of the beam electrons with the atoms of the specimen produce a wide variety of radiation products like backscattered electrons, secondary electrons, absorbed electrons, characteristic and continuum x-rays, etc.
- A sample of this radiation is collected by a detector, most commonly the Everhart—Thornley detector and the collected signal is amplified and displayed on the computer

Scanning Action

- It is usually accomplished by energizing electromagnetic coils arranged in sets consisting of two pairs, one pair each for deflection in the X and Y directions.
- The electromagnetic scanning coils are located in the bore of the final objective lens and are controlled by a scan generator.

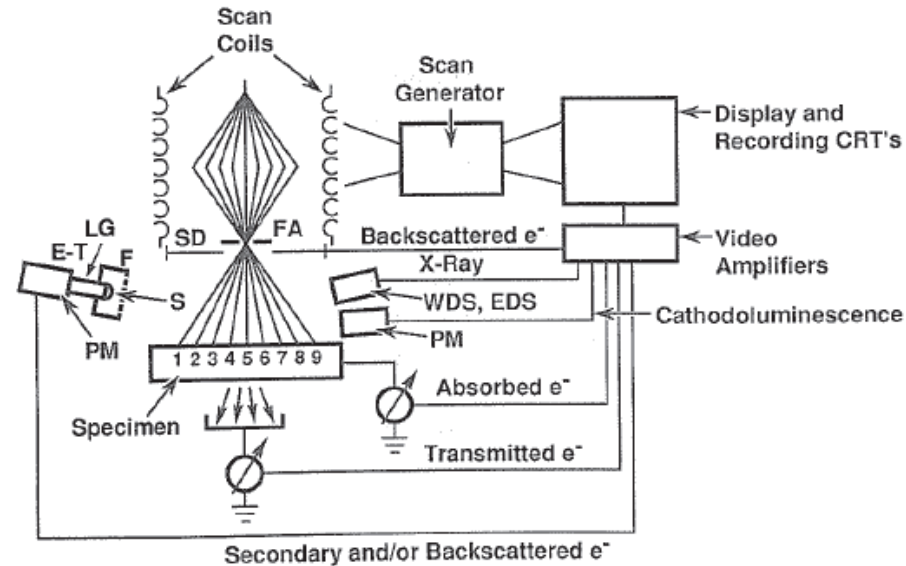


Image taken from Ref.1

- The strength of the current in the scan coils is altered as a function of time, so that the beam is moved through a sequence of positions (e.g. locations 1,2,3,4,etc.) on the specimen and each detector samples the electron-specimen interaction at a defined sequence of points.

Image Formation

- The SEM image is a 2D intensity map in the analog or digital domain. Each image pixel on the display corresponds to a point on the sample, which is proportional to the signal intensity captured by the detector at each specific point.
- In an analog scanning system, the beam is moved continuously; with a rapid scan along the X-axis (line scan) supplemented by a stepwise slow scan along the Y-axis at predefined number of lines.
- In digital scanning system, only discrete beam locations are allowed. The beam is positioned in a particular location remains there for a fixed time, called dwell time, and then it is moved to the next point.

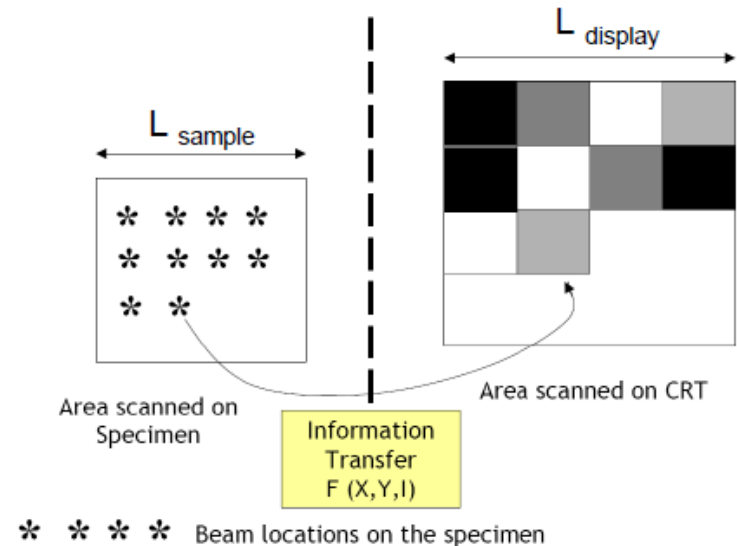


Image taken from Ref.1

Magnification

- It is achieved by scanning an area on the specimen, which is smaller than the display.
- Since the monitor length is fixed, increase or decrease in magnification is achieved by respectively reducing or increasing the length of the scan on the specimen
- It depends only on the excitation of the scan coils and not on the excitation of the objective lens, which determines the focus of the beam
- The magnification of the SEM image is changed by adjusting the length of the scan on the specimen (L_{sample}) for a constant length of scan on the monitor (L_{display}) (fig. in previous slide), which gives the linear magnification of the image (M)

$$\text{Magnification} = L_{\text{display}} / L_{\text{sample}}$$

Depth of Field/Focus (DOF)

- In the observation of a specimen with a substantial depth, if the focus is adjusted to the top side, the bottom side may be out of focus.
- In such a case, if the range between upper and lower image blur is large, it is said that “the DOF is large” . If blur is small, it is said that “the DOF is small”.

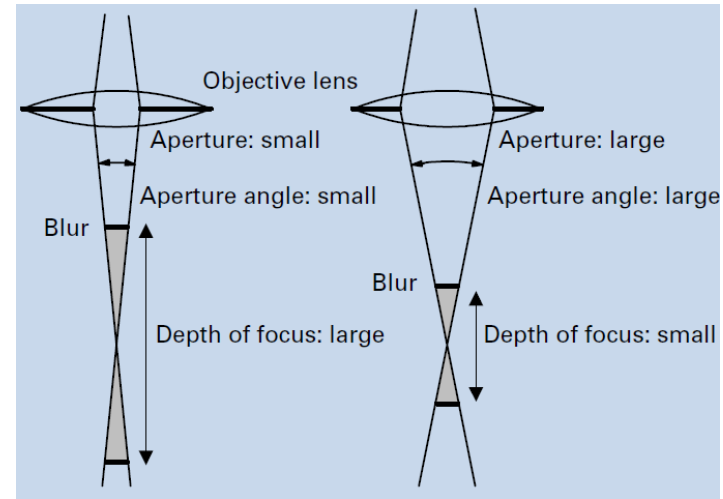


Image taken from SEM A to Z

- When the electron probe is considerably parallel (aperture angle is small), the image stays in focus even if the focus is changed by a large amount. If electron probe is substantially angular (aperture angle is large), the image goes out of focus even if the focus is only slightly changed.
- A practical expression for the DOF is given by $D = 0.2/\alpha M$. α is the beam divergence (semi-cone angle), and M is the magnification.

Sources of distortion

Projection Distortion: Gnomonic Projection

- The flat object placed perpendicular to the optic axis. The image is constructed by a scan that diverges from a rocking point. For a scan generated by rocking about a point, the SEM image is geometrically equivalent to a projection obtained by extending the beam vector to intersect a plane perpendicular to the optic axis of the instrument.

Projection Distortion: Image Foreshortening

- When the planar specimen is tilted around an axis parallel to the image horizontal, the projection situation changes. The projection of the scan line on the projection plane intercepts the expected length of specimen parallel to the tilt axis, but perpendicular to the tilt axis and greater length of the specimen is projected into the same length of scan.
- Magnification is therefore lower perpendicular to the tilt axis (more distance on the specimen is mapped) compared to parallel to the tilt axis. This effect is referred to as "foreshortening."
- *Dynamic Focus Correction:* When a flat surface is highly tilted and is of such a large size that some portion of it extends out of the depth of field of the SEM image, dynamic focus can restore focus to these areas. It is achieved by adjusting the strength of the final objective lens as a function of the scan position perpendicular to the tilt axis so as to bring the plane of optimum focus to coincide with the surface at all working distances in the image field.

Sources of distortion

Scan Distortion(Pathological Defects):

- The image may contain unexpected distortions due to pathological defects in the scan. It arise from improper adjustment of the scan, typically because the two orthogonal scans are produced by different circuits which may not be adjusted to exactly the same value of deflection.
- It may also increase with the scan speed, due to hysteresis in the scan coils.
- The scan pattern may also become nonlinear, particularly at the edges of the image, even at high magnifications.

Moiré Effects:

- When the specimen itself has features with a periodic structure, such as a grid, the superposition of the periodicities of the specimen and the image can lead to the formation of moiré fringes.
- Moiré fringes are the interference patterns between gratings of similar period. When the specimen has a periodic structure, moiré patterns may appear in SEM images and should not be confused with real features of the specimen.

Detectors

Principles of detection:

- When a primary electron (PE) beam hits a sample, certain electron beam interaction processes occur. Secondary electrons (SE) and backscattered electrons (BSE) are then generated.
- Specific types of detectors are able to detect the SE and BSE, and the detector signals can be used to create images and produce information about properties of the sample.
- SE's are ejected from the outer atomic shell of the sample (<50eV).
- BSE's are generated by elastic scattering in a much deeper range of the interaction volume and carry depth information (>50eV).

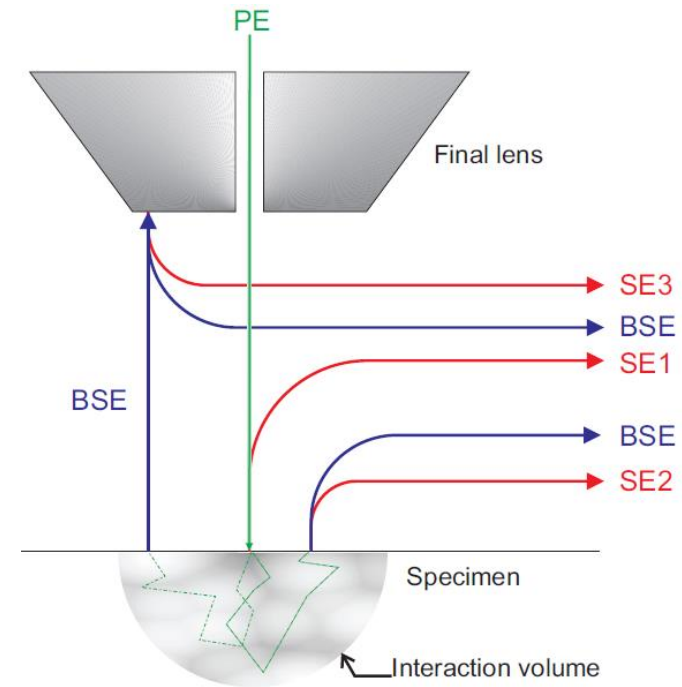
Detectors

The 3 main categories of SE's are:

- *SE1* electrons are generated and leaves the surface of the specimen directly at the spot where the PE beam impacts on the specimen surface.
- *SE2* electrons are generated after multiple scattering inside the interaction volume, and leave the sample at a greater distance from the primary beam's impact point.
- *SE3* electrons are generated by BSE colliding with chamber walls or the lens system

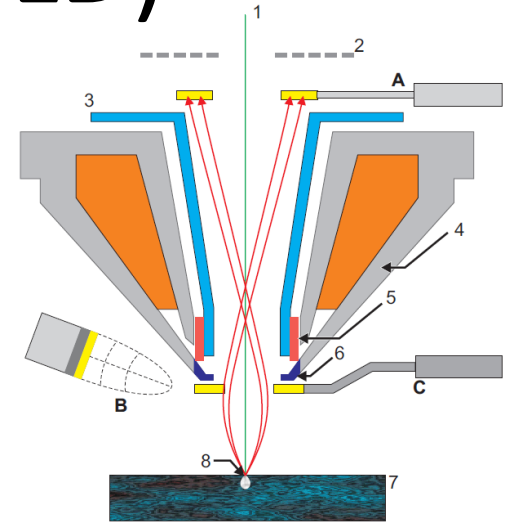
Types of Detectors

- Inlens detector
- Everhart-Thornley (E-T) - SE detector
- Backscattered electron (BSE) detector
- Energy dispersive X-ray spectrometer (EDS) detector




In-lens detector (ILD)

- To map the actual surface of a sample, SE1 type should be detected, because they are the only electrons generated at the primary beam's impact point.
- Also SE1 are generated near the upper region of the interaction volume and therefore only they provide direct information on the sample's surface.
- SE1 electrons can be detected very efficiently by the in-lens detector, since it is located in the beam path and is combined with the electrostatic/magnetic lens.



- | | | | |
|----|--------------------|----|--------------------|
| 1. | PE beam | 7. | Sample |
| 2. | Filtering grid | 8. | Interaction volume |
| 3. | Beam booster | A. | In-lens detector |
| 4. | Magnetic lens | B. | ET SE detector |
| 5. | Scan coils | C. | BSE detector |
| 6. | Electrostatic lens | | |

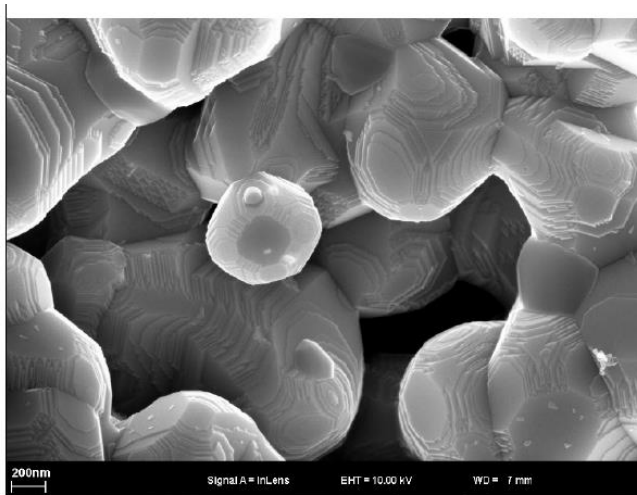
Image taken from Ref.3  Secondary electron paths

- SE electrons are absorbed, re-accelerated and focused through the electromagnetic field to the in-lens detector, where they hit a scintillator and generate photons of light.
- These photons of light are guided out of the beam path through a light guide and transferred to a photomultiplier.
- Photomultiplier multiplies the photons and outputs a signal to generate a image.

In-lens detector (ILD)

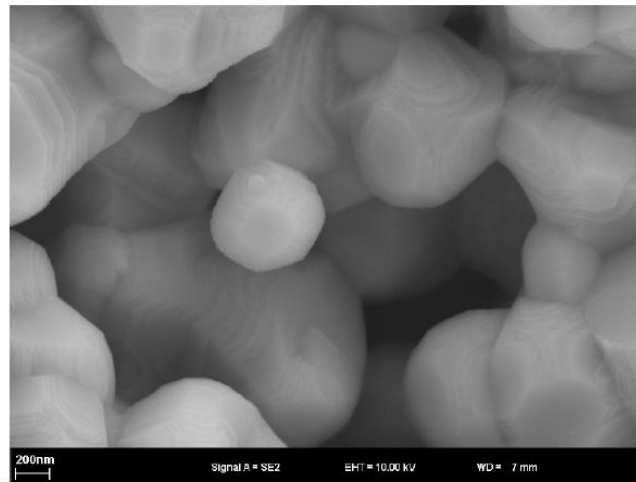
Benefits:

- High detection efficiency at very low voltages, pure detection of SE.
- It provides an ideal tool to map the surface of a sample.
- Even at high voltages, images produced from ILD include more surface information than ET-SE detector (Figure).
- Signals from ILD is generated entirely through detection of SE1 and SE1 electrons. It do not include signals by BSE or SE3 electrons.



In-lens detector

Clear edge effect with good imaging of the surface structures



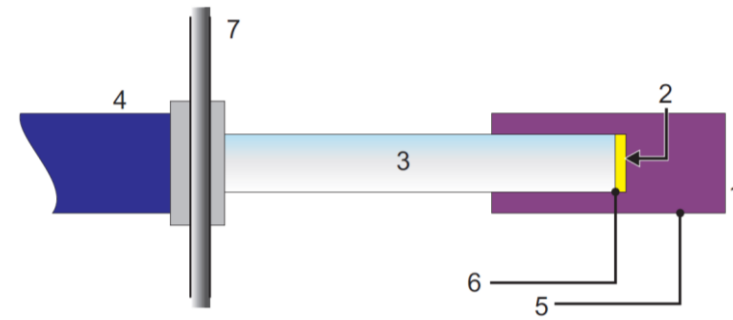
ET-SE detector

Little surface information

Image taken from Ref.3

Everhart-Thornley (E-T) detector

- It is mounted on the wall of the specimen chamber and it views the specimen laterally.
- SE moving towards the detector are absorbed by the collector and are directed towards scintillator.



- | | | | |
|----|-----------------|----|----------------------------|
| 1. | Collector | 5. | Collector bias |
| 2. | Scintillator | 6. | Scintillator voltage |
| 3. | Light pipe | 7. | Inside of specimen chamber |
| 4. | Photomultiplier | | |

Image taken from Ref.3

- When the electrons hit the scintillator layer, photons are generated inside the scintillator. These photons are directed out of the vacuum system through a light pipe and are transferred to the photomultiplier. The photomultiplier multiplies the flashes of light and outputs a signal that can be used for imaging.
- Detector can be used at very high voltage range. Also it is very good when used for imaging at long WD.
- Surfaces tilted towards the detector provide more surface detail with brighter edges than the samples tilted away from detector.

Backscattered Electron Detector

- It is located below the final lens and views the specimen from above. This position offers very large solid angle for the detection of BSE.
- It can be used to image various types of specimen (crystal orientation; magnetic contrast type II and so on), its main application is the display of material contrast (compositional differences).
- Its performance w.r.t contrast is based on the backscattering coefficient, which increases with increasing atomic number. A higher backscattering coefficient results in an increase in the number of BSE generated by the PE beam, which are then made available for detection.
- If different phases exist on the specimen those with higher atomic number display higher brightness than those with a small atomic number.
- It is usually located in the chamber and are moved into a parked position. When needed, they are moved to active position in an arrangement called “push-pull”.

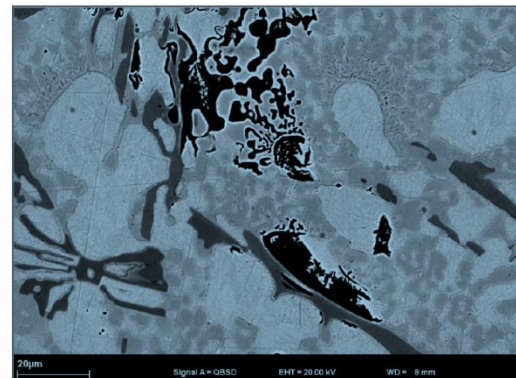
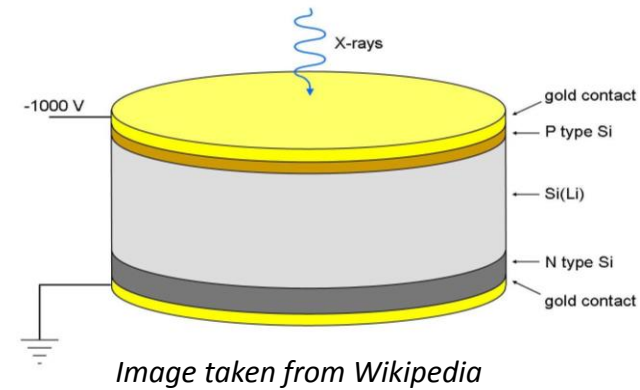


Image taken from Ref.3

Energy Dispersive X-ray Spectrometer Detector

- It has a thin silicon crystal doped with lithium so that the central intrinsic region is sandwiched between a thin p-type layer and thin n-type layer.
- Thin gold electrodes are deposited on the both crystal surface and potential is applied to make the p-n junction reversed biased.
- When an x-ray photon hits the detector, it produces a large number of electrons and holes in the Si(Li) region. Electrons are then accelerated to the positive side and holes to the negative side.
- Thus a pulse of current is generated. The pulse current is proportional to the x-ray energy.
- In operation, each current pulse is analyzed to determine the energy of the incident photon.
- The count for photons within a small energy range is stored in one channel of a multichannel analyzer.
- The X-ray signal is accumulated for a predetermined period of time and the X-ray counts accumulated in the multichannel analyzer constitutes an X-ray spectrum from the specimen.



Contrast

- It is a measure of the real information in the signal and related to the properties of the specimen (shape, composition, etc.) that we wish to determine.
- Defined according to the equation, $C = (S_2 - S_1)/S_2$, $S_2 > S_1$, $0 \leq C \leq 1$. Where S_1 and S_2 represent the signals detected at any two arbitrarily chosen points of interest in the scan raster.
- It can be influenced by a complex mix of the characteristics of the beam-specimen interaction, properties of the specimen, nature of the signal carriers, and position, size, and response of the detector.
- In general, the contrast components can be measured in three different ways:
 - *Number component*: It refers to the contrast that arises simply as a result of different numbers of electrons leaving the specimen at different beam locations in response to changes in the specimen characteristics at those locations.
 - *Trajectory component*: It refers to contrast effects resulting from the paths the electrons travel after leaving the specimen.
 - *Energy component*: It arises when the contrast is carried by a certain portion of the BSE energy distribution. Typically, the high-energy BSE's are the most useful for imaging contrast mechanisms.

Compositional (Atomic Number) contrast

- It arises because the intensity of the signal generated from areas with different composition is proportional to the difference in the average atomic number of the respective areas.
- The most efficient way to image compositional contrast is by using BSE's because of the nearly monotonic increase of the BSE coefficient (η) with atomic number
- Regions of high average atomic number will appear bright relative to regions of low atomic number.
- If we use a detector sensitive to the number of BSE's, then the detected signal is proportional to the backscatter coefficient.
- The contrast between 2 regions can be calculated as the difference between the backscatter coefficients (η_1 and η_2). $C = (\eta_2 - \eta_1) / \eta_2$.
- Elements separated by one unit of atomic number produce low contrast; e.g., Al and Si yield contrast of only 6.7%. For pairs widely separated in atomic number, the contrast is much larger; e.g., Al and Au produce contrast of 69%.

Topographic contrast

- It includes all effects by which the shape and morphology of the specimen can be imaged.
- It arises because the number and trajectories of BSE and the SE depend on the angle of incidence between the beam and the specimen's surface.
- The angle of incidence varies because of the local inclination of the specimen.
- At each point the beam strikes, the number of BSE and SE detected gives direct information on the inclination of the specimen.
- The topographic contrast actually observed depends on the detector used and its placement relative to the specimen and on the exact contribution BSE and SE detected.

Digital Image Enhancement

- Image enhancement involves a collection of techniques that are used to improve the visual appearance of an image to a form which is better suited for human interpretation. The most frequently used technique is given below.

Gray level histogram modifications

- The gray level histogram of an image is a chart, listing all of the gray levels that are used in the image on the horizontal axis and indicating the number of pixels having each level on the vertical axis.
- Gray level images usually consist of 256 levels of gray, so, the horizontal axis of the histogram runs from 0 to 255. The vertical axis varies in scale depending on the number of pixels in the image and the distribution of the gray level values.
- The technique is based on remapping the gray levels within an image by applying a transformation function. A common linear transformation function is called autoscaling.

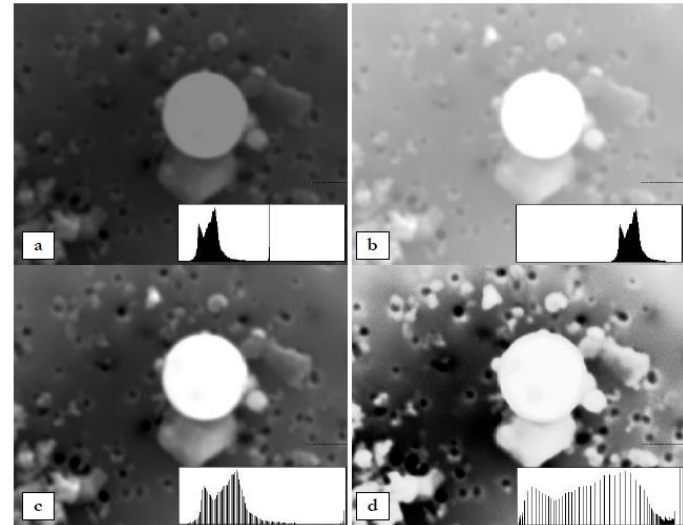


Fig. 1.4.1. a) original image; b) the same image after brightness correction; c) results of autoscaling of the gray level histogram; d) image after histogram linearization.

Image taken from Ref.1

Specimen Preparation for SEM

Specimen Preparation - Metals

- A specimen of suitable size must be removed from the sample.
- It is important not to damage the specimen during the sawing operation.
- The surface to be prepared for micro-structural analysis is polished using a graded sequence of abrasive materials.
- The first step involves coarse grinding using an abrasive 60-180 grit. Then a sequence of finer abrasives is used.
- Grinding steps are usually carried out wet to prevent heating of the specimen and to promote a better surface finish.
- After the specimen has been ground to a 600-mesh finish, the sample must be polished to produce a flat, scratch-free surface.
- Polishing or lapping the sample with abrasives having particle sizes starting with 30 μm for coarse polishing and proceeding to 1 μm or smaller for final polishing accomplishes this objective.

Specimen Preparation - Ceramics

- Ceramic samples are generally insulators and will require conductive coating for good imaging.
- Preparation of ceramic samples is in many ways similar to the preparation for metals. Diamond pastes are commonly used for polishing of ceramics.
- Coating with carbon or other metal films should allow imaging and microanalysis to be performed. For x-ray analysis carbon is coated.

Common steps for both Metals and Ceramics

- Samples must be thoroughly degreased and dried to eliminate any outgassing from organic contamination and water.
- Samples can be cleaned ultrasonically using solvent like acetone and methanol. When cleaned by volatile solvent, samples can be blown dry using a compressed gas.
- After cleaned by water, samples should be dried completely using oven or hot plate. Surface dusts are removed using above processes, and they can also be removed by blowing a compressed gas.

Specimen Preparation - Semiconductors

- It is desirable to understand the structure of an IC's or semiconductors, also important to understand and characterize the electrical performance of the device.
- Voltage-contrast and charge collection microscopy may require special surface preparation to be performed.
- Voltage contrast is a useful technique for the study of active microelectronic circuits. Voltage differences in the metallization on the IC result in a change in contrast observed as a function of the voltage level. Accurate voltage contrast imaging is generally performed on devices that have had the passivation removed.
- The signals used in the charge collection mode are the currents or voltages induced in the specimen by the electron beam. The most important type of signal in charge collection mode is electron-beam induced current (EBIC). EBIC is used to study Schottky barriers as well as diffused and ion-implanted p-n junctions. The examination of p-n junctions in devices requires that the device to be free of passivation.
- When EBIC is used to study bulk semiconductor materials without p-n junctions, a Schottky barrier contact must be applied to the surface of the sample. For p-type material, evaporated Al or Ti layers and for n -type material are Au-Pd and Au of about 100 nm thickness is deposited.

FIB Sample Preparation for SEM

- The major difference is that instead of electrons as in the SEM, FIB uses a beam of positively charged ions.
- FIB are extremely useful for both material science and semiconductor specimen preparation because of their unique capabilities. They include:
- The ability to make a precise cut or cross section with the ion beam. The cross section may be less than $1\mu\text{m}$ to as large as $100\text{-}200\mu\text{m}$ in width and $10\text{-}30\mu\text{m}$ in depth. The cross section may be located with an accuracy of better than 20 nm
- Immediate imaging, using the ion beam with a resolution of 5 nm , with either SE's or secondary ions. Excellent channeling contrast of crystalline materials can be obtained using either secondary electrons or ions generated by the FIB.
- The possibility of injecting several different gases at the ion beam impact point to deposit both conductive and insulating materials, or to selectively etch the cross section or the sample surface.

Sample Preparation - Particles

- Proper preparation of particles requires that the particles be well-dispersed, secure, electrically conductive, and attached to a substrate that does not interfere with x-ray microanalysis.
- If particle size is small (<100nm):
 - Add small amount of powder in solvent
 - Ultrasonicate to disperse particles well
 - Place a small drop of suspension on flat surface
 - Coat to prevent charging
- If particle size is large (>1 μ m):
 - Sprinkle powder on carbon or copper tape
 - Blow off excess with compressed air
 - Coat particles

Sample Preparation - Polymers

- Beam / radiation damage is a large problem
 - Aromatic compounds more stable than aliphatic
 - Hole formation, surface cracking most evident
 - Prepare samples accordingly (sputter coat, Evaporation)
- Simple preparation
 - Isolate surface / fracture surface
 - Sputter coat
 - Image
- Polishing - for internal structures, etc
 - use lubricated sawing and polishing with low pressure. Ends up with low contrast
- Thin sections
 - Cut with microtome
 - Mount piece to advancing arm, Advancement techniques: Thermal, Mechanical, Piezoelectric.
 - Advancing arm comes down on to knife edge, Knife materials: Glass, Diamond, sapphire, WC, Steel

Metal coating

- Coating the sample with a small amount of conductive material to carry away the charging electrons.
 - coating materials: metals (Au, Au/Pd, Pt, Cr); carbon
 - keep the material as thin as possible and keep the coating layer particles as small as possible in order to accurately image the sample surface
 - conductive materials give a high SE yield - more SE signal, better images.

Method for producing a thin layer of metal :

- **Sputtering:** bombard a metal target with an ionized gas, knock metal atoms off the surface of the target and let them settle on the sample surface
- **Evaporation:** heat the metal or carbon in a vacuum chamber until it evaporates and settles on the sample surface

Polishing of Polymers

- Hard-to-section materials such as glass or carbon-fiber-filled composites, tire cords, and large samples have been polished.
- Polishing specimens can produce artifacts at each stage of preparation. Initial cutting can cause formation of micro-cracks, easily interpreted as voids or cracks in the specimen.
- Wet grinding is done with abrasive papers ranging from 120 to 600 grit just long enough to remove the scratches from the previous grinding.
- Grinding should be done with gentle pressure. Cleaning the specimen is important between stages so as not to contaminate the polishing cloth.
- Fine polishing is conducted using graded alumina suspensions in water, chromium oxide slurries, or diamond paste suspended in oils.
- It is best done on a rotating wheel with a layer of fluid on the surface of the polishing cloth using manual or automatic wheels.

Microtomy of Polymers

- It is a sectioning method which allows the cutting of extremely thin sections of material.
- The 3 forms of microtomy are useful for polymers which are *simple peel-back methods, thin sectioning, and ultrathin sectioning*.
- Methods are useful for direct examination of the bulk of fibers and films and for the formation of flat surfaces for x-ray microanalysis of embedded specimens or sections of larger samples.
- In peel-back method the sample is held onto a glass slide or SEM stub with an adhesive or tape, and a scalpel or razor used to nick the surface. Fine forceps are used to peel back the cut either near the central or surface part of the specimen.
- Pieces of molded or extruded plastics are trimmed using small saws and razor blades. Glass knives are best used to prepare flat block faces.
- Thin sections ($>5\mu\text{m}$) are prepared with the aid of a microtome device. Semi-thin ($0.2\text{-}3\mu\text{m}$) as well as ultrathin ($<0.2\mu\text{m}$) sections are prepared with the aid of a ultramicrotomes device

Staining of Polymers

- Most polymers, in common with biological materials, are composed of low-atomic-number elements and thus exhibit little variation in electron density.
- Staining involves the incorporation of high-atomic-number atoms into the polymer to increase the density and thus enhance contrast.
- Stains may be applied directly to the specimen, to block faces produced by microtomy, or to the sections themselves.
- Most of the stains applied to polymers are positive stains. In positive staining, the region of interest is stained dark by either a chemical interaction or selective physical absorption.
- In negative staining the shape of small particles mounted on smooth substrates is shown by staining the regions surrounding the particles rather than the particles themselves. It is often applied to latex or emulsion materials.
- It can be used to enhance contrast for SEM, generally with BSE imaging, to reveal details in multiphase polymers and in composites with inorganic fillers.
- Most commonly used stains are osmium tetroxide and ruthenium tetroxide.

Etching of Polymers

- It is a preparation method that potentially enhances the information available in a polymer, although the methods are not reproducible and the resulting images may be difficult to interpret.
- The three general categories of etching are *dissolution*, *chemical attack*, and *bombardment* with charged particles as in plasma and ion beam etching.
- *Dissolution* implies the removal of whole molecules of a material as it is dissolved. Solvent extraction tends to be incomplete and material is often left on the surface.
- In *chemical etching* there is chemical attack on molecules and removal of fragments. For instance, acid treatments can selectively oxidize one phase in a multiphase material, aiding contrast.
- Charged species activated by high voltages or in radiofrequency plasma are also used to etch away material differentially on the surface of specimens. Plasma and ion beams are employed to bombard or sputter and remove surface atoms and molecules

Image Interpretation and Artifacts

- Selection of preparation methods for microscopy study is the nature of the potential artifacts formed.
- Preparation of specimens from the bulk generally involves deformation of the specimen, for example, peeling, fracturing, sectioning, and etching.
- Peeling results in fibrillation, which is a function of the material and the force used.
- Sections and sectioned block faces exhibit knife marks and other textures associated with the preparation.
- Chemical etching removes polymer material, often re-depositing it on the surface and creating new structures unrelated to the material.
- Ion etching can result in heating and melting of the specimen, and formation of cone-like structures is often observed.
- Plasma etching is less likely to form spurious structures, although if there is oxygen present, there can be surface oxidation.
- Application of metal or carbon coatings to polymers can cause formation of grain-like textures or melt or otherwise damage the specimen surface

Charging and related issues

- When the beam interacts with the sample part of the incident electrons are emitted back, but larger fraction remains in the specimen as the beam electrons lose their initial energy and are captured by the specimen.
- This charge flows to ground if the specimen is conductor and a suitable connection exists to conduct away the charges. If the ground path is broken, even conducting specimen quickly accumulates charge and its surface potential rises.
- Charging is more often observed whenever a specimen or portion of it is an insulator. The charges injected by the beam cannot readily flow to ground. The resulting accumulation of charge is a complex, dynamic phenomenon. The specimen is in a continually changing state of surface potential due to the accumulation and discharge of electrons.
- Charging manifests itself in images in a variety of ways. When local charging alters the surface potential, the field lines of the detector potential, which exist around the sample are disrupted, and collection of SE is greatly altered.

Charging and related issues

- A contrast mechanism develops known as voltage contrast in which the potential distribution across the surface is imaged. Some areas appear extremely bright because they are negative relative to the E-T detector and enhance SE collection. Other areas appear black because they charge positively and suppress the collection of SE.
- The difficulty arises because the contrast due to surface potential becomes so large that it overwhelms the contrast from the true features of the sample.
- Effects due to charging can be minimized by coating the sample with conductive film, by utilizing rapid scanning, by imaging with BSE, and/or low accelerating voltage.

References

- Scanning Electron Microscopy and X-Ray Microanalysis, 3rd edition 2003, Joseph I. Goldstein.
- Electron Microscopy of Polymers, Springer 2008, Goerg H. Michler
- Sigma FESEM User Manual, Carl Zeiss.