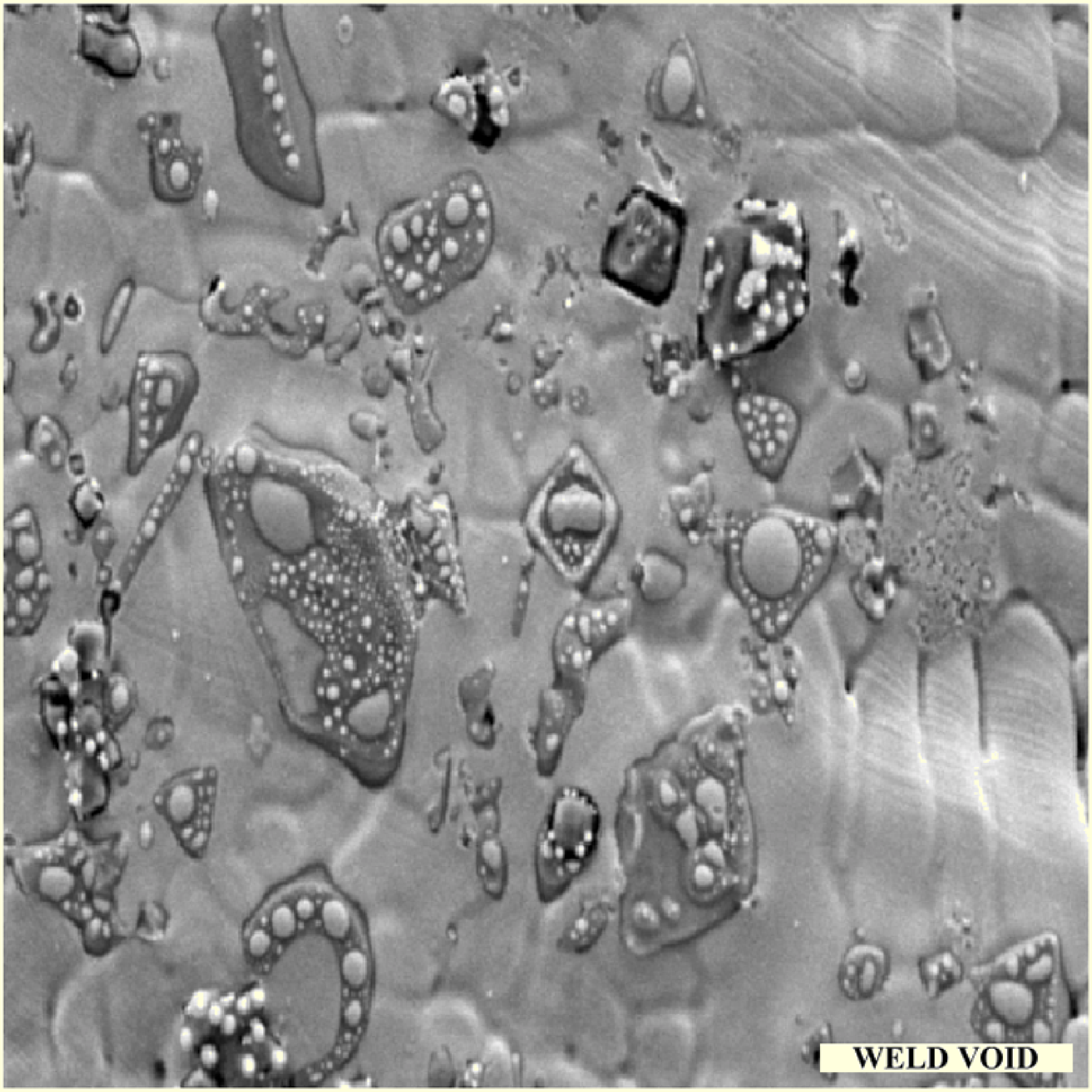
Woody's

PRACTICAL PRIMER

SEM IMAGING, X-RAY ANALYSIS, AND SPECIMEN PREPARATION



CHAPTER CONTENTS

1. CHARACTERISTICS & ATTRIBUTES OF SEMs
2. BASIC SEM OPTICS / CHAMBER
3. CHAMBER GEOMETRIES
4. SEM MAGNIFICATION
5. ELECTRON GUNS
6. SEM SIGNALS AND DETECTORS
7. DATA PRESENTATION / HARDCOPY
8. ELECTRON BEAM / SPECIMEN INTERACTION
9. OPERATING CONDITIONS - EFFECTS ON X-RAYS
10. COLLECTING SPECIMENS
11. SPECIMEN PREPARATION / MOUNTING
12. SPECIMEN COATING
13. COATING EFFECT ON BSE IMAGES
14. CHARGING ARTIFACTS AND PROBLEMS
15. COATING EFFECT ON X-RAY ANALYSIS
16. STEREO IMAGING (3-D)

1. CHARACTERISTICS & ATTRIBUTES OF SEM*s*

A scanning electron microscope (SEM) uses electrons instead of light to examine a specimen. Unlike visible light, electrons have no color. Also, specimens that are optically transparent are opaque in the SEM. Images from a SEM are produced in shades of gray and hard copy is usually a "black and white" photo. Digital imaging systems can, however, "pseudo-color" an image, usually by assigning a color to a particular gray level or range of gray levels.

When an electron beam strikes the specimen surface, many different interactions are possible. What occurs is a matter of statistics. Some electron energy is conducted away by the electrically grounded specimen. The rest of the electron energy leaves the specimen surface in various forms. The electrons may be "reflected" (backscattered), or lose much of their energy by interacting with an atom's electron shell (“bouncing around inside the specimen”). When this occurs, the electron shell(s) is raised to a higher, unstable, state. As the effected orbital electron decays to a stable state, the energy may be emitted in the form of "secondary" or auger electrons, x-rays, or visible light. Any of these may be collected to obtain information about a specimen.

SEMs raster (**scan**) an electron beam over an area of a specimen while synchronously rastering a viewing screen (CRT), much like a TV image is formed. The ratio of the sample scan size to the displayed scan size is the magnification. Various signals obtained from the specimen are used to modulate the brightness of the viewing/record CRT sweep. Photographic film is exposed with the “record” CRT that usually has higher resolution than the viewing CRT. Many newer systems implement digital imaging and storage.

SEM specimens are mounted to an X,Y,Z, rotate, and tilt translation stage to position the area of interest under the beam.

Specimens must be stable under vacuum, and be electrically conductive. *The "variable (chamber) pressure", or “environmental” SEM is an exception.*

Light microscopes produce images showing only the topography / morphology of a specimen. The most common mode of SEM imaging using secondary electrons (SE) acts similarly. SEMs can also produce images that are a function of sample composition using backscattered electron (BSE) or x-ray signals.

Typically, light microscopes are limited to less than 2,000x magnification, while SEM's magnification can range from less than 10x to more than 100,000x. The resolution of most SEMs limits *useful* magnification to about 50,000 times. The exact maximum will vary, depending on the instrument, it’s operating parameters, and the composition of the imaged surface.

The depth of focus available with a SEM is considerably greater than that of light microscopes. Even very rough, high relief specimens are usually in full focus. Although usually a positive attribute, this can be confusing at times, as surfaces may appear flatter than they really are.

It is easy to image in stereo using a SEM. Some systems are equipped to provide real time 3-D imaging, while others use stereo pair photos with a special viewer. Under controlled conditions, elevation measurements on a specimen can be made using stereo images. An example of a stereo pair is included at the end of this document.

If equipped with digital imaging, systems can do quantitative feature analysis. Examples are: particle counting, phase / inclusion counting, % area of features such as pores or inclusions, size distributions of particles / phases / pores, etc.

If equipped with x-ray analysis accessories (EDS/WDS), the SEM can analyze for constituent elements in specimens. The data may be qualitative or quantitative and can be presented in many formats. (graphical spectra, distribution maps, composition in weight percent, etc.)

VARIABLE PRESSURE SEMs (E-SEM, Environmental SEM)

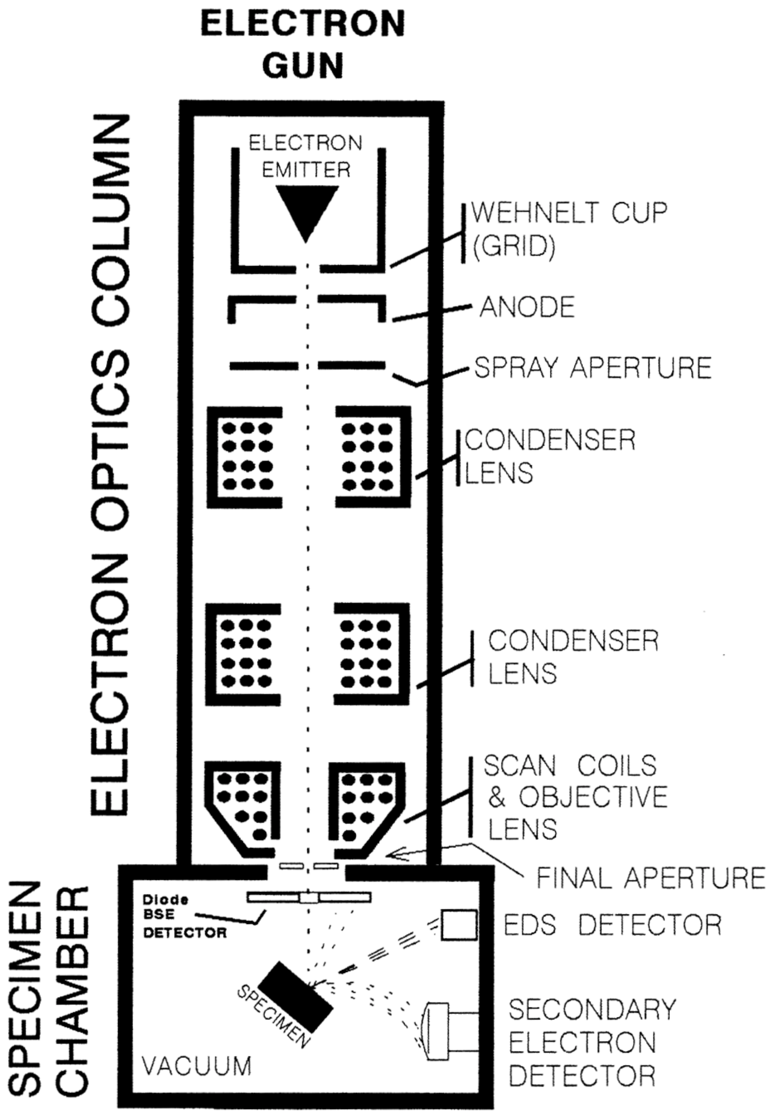
Historically all SEMs employed a high vacuum (Typ: 1×10^{-5} Torr or better) in the specimen chamber and optics column. This presented no problem for **conductive** specimen surfaces that could be “grounded”. In a vacuum, when an electron beam hits an **insulator**, there is no where for the electrons to go and a static charge builds. The repulsion from the static charge buildup can cause distortion of the beam sweep and resulting image. In extreme cases of charging, the beam may be “pushed” far enough to totally disrupt imaging. Very thin conductive coatings like gold or carbon can be applied to an insulating specimen to conduct away the charge. For most applications this is quite satisfactory. Some rough, convoluted, porous, or friable specimens may be difficult to coat with a good continuous conductive film. This will result in either general or localized areas of charging. Liquids or wet specimens were impossible to examine.

The variable pressure SEM uses differential vacuum pumping to maintain the electron gun at high vacuum while keeping the chamber pressure (specimen location) at a partial pressure of a gas or vapor. Higher pressure in the chamber has two benefits. The pressure can be set to minimize liquid/wet specimen evaporation, permitting some liquids and wet specimens to be examined. Also, since the partial chamber pressure can be employed to dissipate electron charge, insulators can be examined with no need for a conductive coating. There is, however, a disadvantage. Since higher pressure means more atoms in the path of the electrons, scatter occurs. Scatter in the electron beam will limit resolution and thus, useful magnification.

Many SEM manufacturers now offer microscopes that can be used at both high vacuum **or** as a variable pressure SEM.

2. ELECTRON OPTICS - SPECIMEN CHAMBER.

Illustrated is a simplified cross section of a typical SEM column.
Details of individual systems will vary.

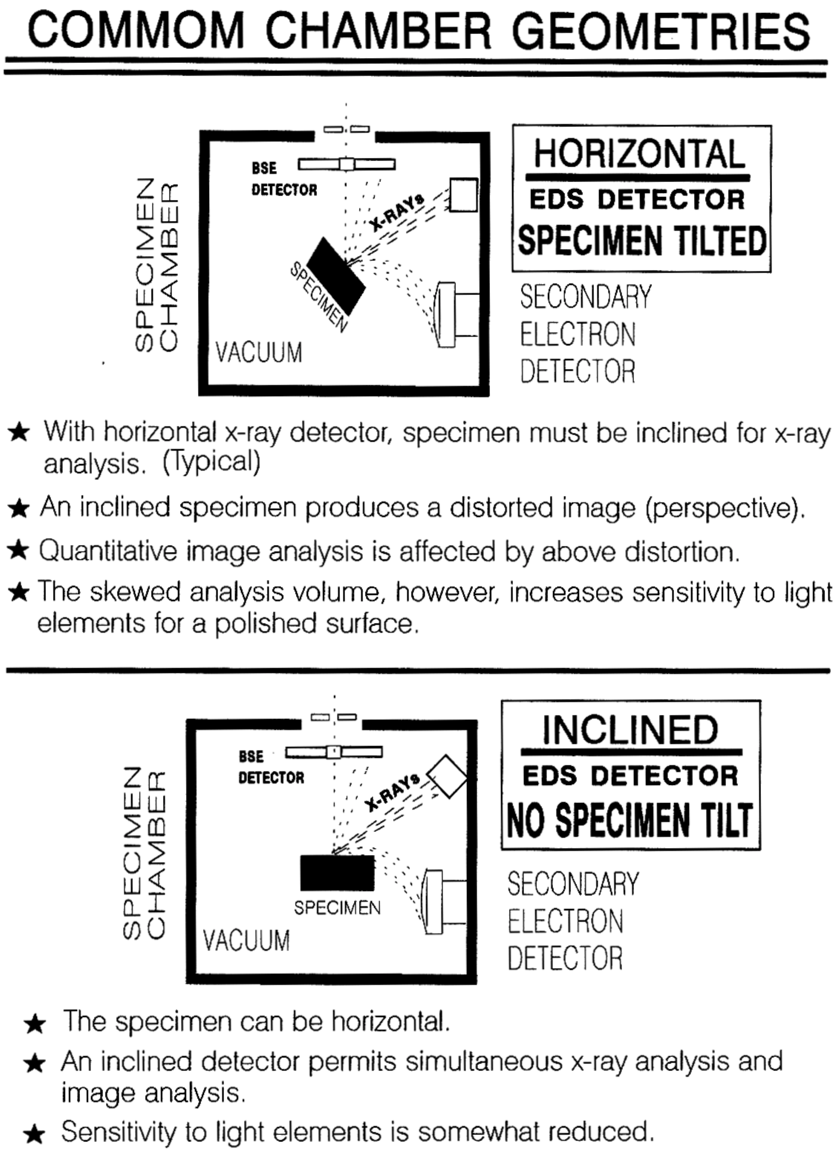


3. CHAMBERS

Of the two geometries, the inclined detector is used most on new systems.

Although either system performs well, the increased versatility offered by an inclined system is often the deciding factor.

Using an inclined system, it is possible to combine image and (x-ray) elemental analysis. For example, energy dispersive x-ray spectroscopy (EDS) could be performed automatically on a specified size range of particles in a field of view.



COMMOM CHAMBER GEOMETRIES

HORIZONTAL

EDS DETECTOR

SPECIMEN TILTED

- ★ With horizontal x-ray detector, specimen must be inclined for x-ray analysis. (Typical)
- ★ An inclined specimen produces a distorted image (perspective).
- ★ Quantitative image analysis is affected by above distortion.
- ★ The skewed analysis volume, however, increases sensitivity to light elements for a polished surface.

INCLINED

EDS DETECTOR

NO SPECIMEN TILT

- ★ The specimen can be horizontal.
- ★ An inclined detector permits simultaneous x-ray analysis and image analysis.
- ★ Sensitivity to light elements is somewhat reduced.

4. SEM MAGNIFICATION

SEM magnification is determined by the ratio of the distance scanned on a specimen to the area displayed for view.

The magnification range of the average SEM is from 10x to 50,000x. Most SEMs can be set to higher than 250,000x, but because of lack of resolution, the magnification is "hollow". That is, no more detail is available than if a lower magnification was used. Specialized SEMs, on appropriate specimens, can achieve resolution at the higher magnifications.

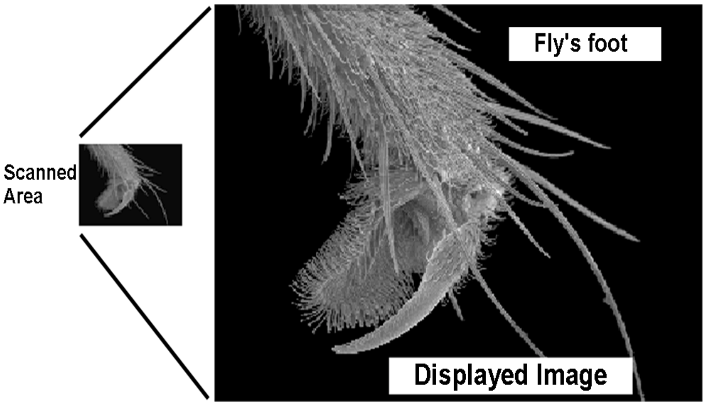
Magnification of most SEMs is calibrated to the hardcopy device, often 4 x 5 film. The real time viewing screen is typically larger than the "recording" CRT / 4 x 5 photo. Thus, the real time image is higher in magnification than indicated on the SEM. Micron marker bars can be displayed on the real time screen to determine feature sizes.

SEM magnification values are not inherently very accurate. Changes in operating conditions such as beam kV., condenser lens settings, working distance (distance from final lens to specimen), etc., can affect calibration. If it is important to know the exact magnification of an image, the SEM should be standardized (calibrated) at the same operating conditions used for imaging. A magnification correction coefficient can then be determined.

Errors in magnification can be +- 10% or more for some older analog systems. New technology digital systems are often more stable. For close tolerance measurements, special "critical line width" systems are available. Such systems are often used in the semiconductor industry.

The figure to the right is a hypothetical example of SEM magnification. In reality 8-10x is the minimum magnification of most systems.

EXAMPLE OF 5x MAGNIFICATION



Magnification = Ratio of scanned to displayed size.

5. ELECTRON GUNS

Every SEM has an electron gun, the source of the electron beam. Most electron sources used by SEMs are thermionic emitters, the gun assembly consists of a heated filament (cathode), wehnelt cup (grid), and an anode. A beam accelerating potential (high voltage) is applied between the filament and the anode. The potential can range from about 100 volts to over 30,000 volts. The electron beam current is stabilized to the desired value by a potential applied to the wehnelt cup ("grid" as in a radio vacuum tube). A sizable amount of current can be lost passing through small apertures in the column. The gun may emit as much as several hundred microamps (10^{-6} amps). Beam current at the specimen can range from only a few picoamps (10^{-12} amps) to several microamps (10^{-6} amps), depending on the gun type and operating conditions. In most practical applications, the beam size (diameter) will increase with an increase in total beam current. It follows that, all else being equal, high magnification resolution is easier to achieve at lower beam currents, while high beam currents reduce resolution and can limit useful magnification.

Beam brightness is the current density of the beam. It is a function of beam current versus its' cross-sectional area / angular divergence.

Specimen current is the beam current entering a specimen, minus the effective current re-emitted from the surface of the specimen as x-rays, secondary electrons, backscattered electrons, etc.

Note: Voltage is analogous to water pressure in a pipe and current is analogous to water delivery from a pipe in gallons per second.

FOUR TYPES OF ELECTRON GUNS

TUNGSTEN HAIRPIN

A tungsten hairpin is the oldest and most common electron emitter. It is a thermionic electron emitter, that is, it is heated like a light bulb filament to eject electrons. This emitter is durable and inexpensive. Contamination on the filament is not usually a problem and no special vacuum system is required. The emission current is quite stable which makes it a good choice for quantitative x-ray analysis. The (wide) electron energy bandwidth emitted and the large effective source size, however, make focusing the resultant beam to a small, bright spot difficult. This, in turn, makes high resolution, low accelerating voltage/low current imaging more difficult.

LaB_6 (lanthanum hexaboride) EMITTER

In the past 10 - 15 years this emitter has come into common usage where higher beam brightness is desired. The emission current from a LaB_6 gun is considerably higher than for tungsten. This emitter is also a thermionic device, but instead of a directly heated tungsten wire, a single crystal of lanthanum hexaboride is heated. Several tip geometries are available. Pointed tips are brighter while more blunt tips extend emission life. Current stability of LaB_6 emitters is reasonably good and is suitable for quantitative x-ray analysis work. This emitter is considerably more expensive that tungsten. Higher cost is mitigated by a much longer life, but purchase and operational costs are still somewhat higher than tungsten. Since LaB_6 systems are subject to loss of emission from contamination, the electron gun vacuum is isolated from the chamber vacuum and has it's own high vacuum pump.

FIELD EMITTERs (Cold Emission, non-thermionic)

A field emission gun is not typically a thermionic emitter, but relies on an extremely sharp tip to generate a very high electric field, thereby emitting electrons. A FE emitter is quite sensitive to contamination and must be kept at a very high, clean vacuum. Since only an atomic monolayer of contamination can seriously affect emission current, this gun is generally considered too unstable to perform highly accurate quantitative x-ray analysis. Like the LaB_6 gun, a separate vacuum environment is required, but cleanliness and vacuum requirements are even more critical. Even so, contamination must occasionally be cleaned from the surface by a process known as "flashing". Flashing is a process of cleaning the emitter by heating under high vacuum. FE systems are very expensive, but if treated well, exhibit long life. Since flashing will eventually blunt the tip and reduce electron emission, lifetime is primarily related to the system cleanliness. A FE gun produces a very small beam diameter and a high current density (high brightness). It also produces the most "monochromatic" beam (each electron in the beam is close to the same potential). For these reasons, it is the best choice for low accelerating potential, high resolution imaging.

THERMALLY ASSISTED FIELD EMITTER

A variation on the cold field emitter is the thermally assisted field emission gun. The principle of operation is quite similar to the cold emitter, except a special emitter tip is heated. One primary effect is to reduce contamination build-up on the tip that helps to stabilize emission current and reduce cleaning requirements. Manufacturers of these systems claim emission stability is sufficient to perform good quantitative x-ray analysis.

6. SEM SIGNALS AND DETECTORS

SECONDARY ELECTRONS (SE or SEI)

Secondary electrons are emitted from an atom when it is stimulated by the incident electron beam. Since they have low energy, and are easily absorbed by the specimen, they provide information from the (near) surface of a specimen. SE detectors can attract these low energy (~ 2-8 eV.) electrons - they do not necessarily travel in a straight line from the specimen to the detector. The relative tilt of the surface under the beam will affect production of secondary electrons. High tilt angles and micro-roughness yield more electrons than very smooth or flat surfaces. Since the SE production varies with incline, topography is well detected. More electrons collected from an area on the specimen produce a brighter area in the image. The most common detector is an "Everhart-Thornley" type that is a scintillator plate coupled to a photomultiplier tube. This detector is also sensitive to backscattered electrons, but is usually positioned to avoid them. A portion of the secondary electron signal collected by most systems is not related to the incline/roughness under the beam but are spawned by backscattered electron (below) interaction with the sample and chamber. Composition can also have an affect. For example, all else being equal, carbon will appear darker than a metal oxide.

Secondary Electrons (SE, SEI) are used to show topography and fine morphology, the image is most nearly analogous to that from a light microscope.

BACKSCATTERED ELECTRONS (BSE)

Backscattered Electrons are incident beam electrons that are elastically scattered (i.e. reflected) from the specimen. BSEs have a broad energy range. Depending on losses in the specimen, the energy can range from near 0 eV. to nearly that of the incident beam. This can be as high as 30 kV. on most SEMs. BSE signals from a high potential (voltage) beam can be quite penetrating and may image tens of nanometers or more into the specimen surface (see Effects of Coatings). BSE yield is a function of the BSE coefficient of the specimen material . Typically the coefficient is a function of average atomic number. Thus, higher atomic number (Z) material in a sample yields more electrons and image brightly. Conversely, lower atomic number areas in a specimen will be darker. Topography response may - or may not be suppressed, depending on the type of detector. BSE signals can be used to image grain structures also, using a property known as electron channeling. Since BSEs are higher energy, they tend to travel in straight lines. The detector is placed over the specimen with the incident beam passing through a hole in it.

There are two general types of BSE detectors in common usage:

Scintillator (**Robinson**) detector - A scintillator plate is placed over the specimen and the light generated is detected in proportion to the backscattered electrons. This detector is compact and sensitive to a wide range of electron energies. It tends to suppress any topography. A strong point is its sensitivity to low potential back scattered electrons and fast response time. This permits relatively fast scan BSE while using low beam kV.

Diode (solid-state, four quadrant) detector - This detector is comprised of four photovoltaic cells arranged in annular quadrants with a hole in the center for the incident beam. Each of the four quadrant polarities can be individually controlled. If all are summed, topography is suppressed and "Z" (atomic number) contrast is obtained. If certain quadrants are differenced, then "Z" is somewhat suppressed and topography contrast is enhanced. Typical diode detectors are more sensitive to electron energies above approximately 5 kV. This can restrict fast scan, low kV., high resolution imaging capability.

A third type of detector that can be used for either secondary or backscattered electrons is the Electron Channel Plate. This is a rather new detector which is versatile but rather fragile.

BackScattered Electrons (BSE) are used to show topography or, more often, changes in atomic number across a specimen.

BSEs can also be used to show grain structure in some polished specimens. Contrast is obtained by an "electron channeling" effect. Various orientations of crystals in a material will exhibit different backscattering coefficients, thus generating more or less image brightness. This response is weak and affected strongly by a poor surface polish or coating on a polished specimen. Channeling contrast closely resembles an etched (for grain structure) image obtained with a light microscope.

SACP

(Selected Area Channeling Pattern) is another, less common BSE mode. In this mode, the incident electron beam is not scanned, but focused to a point on the specimen. The incident angle of the beam is then "rocked" through an arc. The backscattered electrons will then produce a pattern (image) on the synchronously scanned viewing CRT that indicates the orientation of an individual crystal. This effect is similar to "Kikuchi" patterns of crystal orientation formed with transmission electron microscopes.

X-RAYs

When an incident electron (beam) interacts with the specimen, x-rays may be produced instead of SEs or BSEs. Two “types” of x-rays that may be produced are continuum (Bremsstrahlung) x-rays; a "background" of energies ranging from near 0 eV. to that of the incident electron beam. This "background" is generally undesirable “noise” on the desired spectrum, but can provide useful information about the x-ray absorption characteristics of a specimen. Characteristic x-rays are emitted when an incident electron has excited an orbital electron to a higher energy level and it decays to its stable state. X-rays produced by decay from these orbits (M,L, & K Shells) produce x-ray energy emissions unique to each element. Thus, by observing the x-ray energy spectrum emitted by a specimen, the constituent elements can be identified. X-ray analysis systems can be used to identify/quantify elements excited by the beam. Most SEMs allow the beam sweep to be modified for x-ray analysis. In the “full scan” mode, the full field of view produces the spectrum. Reduced area scans are often available to analyze only a small segment of the field of view. Very useful is the “spot mode” where all scan is removed and the beam can be positioned as a “spot” to any location in the field of view. Since they penetrate matter more easily, spatial resolution for the average x-ray is worse than SE or BSE. The x-ray **analysis volume** can be quite variable. It is a function of geometry, beam kV., and specimen composition.

Another very useful application of characteristic x-rays coupled with scan control is generating elemental distribution maps (dot maps). These x-ray produced images show the elemental distribution in a field of view. See the area “Hard copy” pages for an example.

There are two types of x-ray detector systems in general use, energy dispersive (EDS, EDX) and wavelength dispersive (WDS, WDX). WDS detectors are also used on electron microprobe analyzers (EMPA).

EDS

EDS systems detect and display the intensity of acquired x-rays as a function of their **energy**. A solid-state silicon or germanium detector converts x-rays to an electrical pulse. The amplitude of the pulse is proportional to the energy of the x-ray. An "energy to digital" converter (Analog to Digital Converter, ADC) digitizes the pulses. X-ray events (counts) and their energy are measured, stored in computer memory, sorted and then displayed as a graph of integrated counts vs. energy. Most detectors will acquire x-ray energies from a little less than 1 kV. to 30 kV (Detectability from sodium up...). Special detectors are available which will provide limited sensitivities down to 100-120 eV. (Boron, sometimes Be).

Examples of EDS spectra are shown under " GEOM/OPERATING CONDITIONS".

The chief advantage of EDS is speed. Spectra encompassing most elements can be acquired to qualitatively analyze a specimen in as little as 20-30 seconds. Accurate quantitative (especially for specimens with a complex composition or low concentrations) may require acquisition times of several minutes. In many cases, quantification is also quick, using a "standardless" analysis computer program. One serious disadvantage of EDS is displayed characteristic x-ray peaks are much wider than the true characteristic x-ray energy spread. A peak width of approximately 130 eV. is collected and displayed vs. an actual peak width of 2-3 eV. Poor resolution leads to peak overlaps (convolution) which can cause ambiguities in peak (element) identification and quantification. Most EDS systems are equipped with a peak deconvolution computer routine as part of the quantification program. These peak modeling programs mitigate identification and integration errors and make accurate quantification possible. Minimum detectable concentration (MDC) limits depend on a number of variables, but are usually in the range of 0.5 weight percent or lower.

WDS

WDS systems detect and display the intensity of acquired x-rays as a function of their **wavelength**. The detector consists of one or more x-ray diffraction crystals and a "gas amplification" x-ray detector. There are detector variations, but a gas amplification detector can be a Geiger-Muller tube or a Proportional Gas Flow detector, depending on the construction/application. When a x-ray from the specimen enters the WDS detector, is diffracted (bent) by a crystal (or special layered synthetic) and, if the degree of diffraction is correct, hits the detector, and the data sent to the computer. X-rays of other wavelengths are diffracted more or less and miss the detector. The diffraction angle is a function of the x-ray wavelength and the diffracting crystal selected. The relative positions of the diffracting crystal and x-ray detector are variable. As a crystal and detector are driven through their range (Bragg Angle) x-rays of different wavelengths strike the x-ray detector. The chief advantage of WDS is that very narrow peaks can be obtained (a few eV. wide vs. ~130 using EDS). The benefit is twofold. Peaks do not often overlap which eliminates identification uncertainties, and because the peaks are narrow, the background radiation under a peak is reduced, increasing effective sensitivity. Minimum detectable concentration of an element, like EDS, depends on a number of variables, but can be as low as several hundred PPM. WDS, however, is slow and tedious compared to EDS. In order to acquire a spectrum, crystal and detector must be physically stepped through their diffraction range while collecting x-rays. Furthermore, no single crystal covers the entire wavelength range for all elements. To detect all possible elements typically requires data acquisition with each of four different crystals. Many EMPAs are equipped with four WD detector systems to minimize analysis time. Quantification can be more accurate than EDS, but at a price. It is more time intensive; concentration standards for each element must be available, analyzed, and entered into the WDS computer prior to the unknown analysis.

USEFUL, BUT LESS COMMONLY USED SIGNALS & MODES

CATHODOLUMINESCENCE

Some compounds emit visible or near visible light when excited by the incident electron beam. SEMs can be equipped with special detectors to collect this signal. It is often used in identification of certain minerals or organics. The simple fact that a specimen exhibits cathodoluminescence can be an important clue about its composition. A full spectral analysis of emitted light wavelengths may yield more detailed information.

SPECIMEN CURRENT CONTRAST

Energy not emitted from the specimen surface in one form or another is conducted away through a ground connection to the specimen. This current can be used to used to generate an image that is roughly the inverse if a BSE image.

VOLTAGE CONTRAST

Secondary electrons can be used to generate an image based on the potential of the specimen under the beam. This technique is usually used in the semiconductor industry to observe the operation of integrated circuits in the SEM. If "beam blanking" is used (stroboscope effect), electrical signals can be seen moving through the chip. Undesirable voltage contrast can occur in the form of specimen charging. (See SEM SPECIMEN COATING section)

MAGNETIC CONTRAST

Secondary electrons may also be used to image changes in magnetic materials such as audio/video tape and computer disks.   Strong magnetic fields, however, can deflect the electron beam and disrupt imaging.

ELECTRON BEAM INDUCED CURRENT (EBIC)

This examination technique is used by the semiconductor industry. Electrical contact is made to various layers of a semiconductor device. Current **in** the specimen, induced by the incident electron beam, is measured and this data is used to form an image.

7. DATA PRESENTATION / HARDCOPY FORMATS

Images generated by secondary or backscattered electrons are normally viewed in real time on a display (CRT). Some SEMs use "slow scan" (low scan repetition rate) CRTs which have long persistence phosphor screens. Digital" SEMs can scan at standard television rates, store the image to computer memory, average multiple scans to enhance signal-to-noise ratio, and display the result to a CRT display. Many accessory EDS systems can also acquire digital images and have quantitative image analysis programs that can count particles, sort sizes, etc.

Hard copy from older SEMs is usually available in the form of 4 x 5 format instant film. Resolution of analog photos can be as high as 2000 lines in four inches if negatives are available. Digital systems can store images to a file or direct them to a printer. For storage, TIFF (xxxx.tif) files are often used. The JPEG (xxxx.jpg) file type can be used to compress images, saving storage space. Lossy compression (.jpg) is not recommended if image analysis will be performed or if the file will be re-saved. The usual resolution of digital images is from 512 x 512 to 4096 x 4096 pixels in 256 (8 bit) shades of gray (or more). The exact resolution will be a function of the system capability and your patience. Even with today’s fast computers, massive images can take a while to process! Printers for these images can reproduce shades of gray, halftone, or full color, and vary widely in resolution, image quality, size and cost. Direct digital images and scanned analog images were both used to generate this document.

These same hardcopy systems can also be used to present data from other SEM sources.

X-ray data (elemental composition) can be acquired from the entire field of view when examining a specimen. The result is a spectrum that represents the average composition, which can be printed and/or saved. Spectra can usually be saved as either raw data or as images.

A spectrum (spectral print-out) of integrated x-ray counts vs. energy is often used to present qualitative analysis data. Characteristic x-rays are displayed as vertical peaks across the horizontal energy axis. Extreme care must be exercised not to "mentally quantify" since corrections to peak heights during true quantitative analysis can be hundreds of percent. Experienced analysts can often crudely rank elemental abundance, based on past observations.

Sometimes it is desirable to analyze a feature within the current field of view. Scan rasters can be controlled to limit composition analysis to a selected area within the image, a series of spots along a line (line scan), or a single spot in the image. For each mode, this data can be printed or saved to a file. Depending on the analysis circumstances, standards may, or may not, be required for quantitative results.

A line scan is used to show the relative distribution of a certain element in a line across the imaged area. It can be acquired and stored by computer, or acquired on 4 x 5 film as a triple exposure. The image is acquired, then a line defining where the beam will sweep the specimen is stored. Next, a line graphing x-ray count rate vs. position is stored as the beam sweeps across the analysis line.

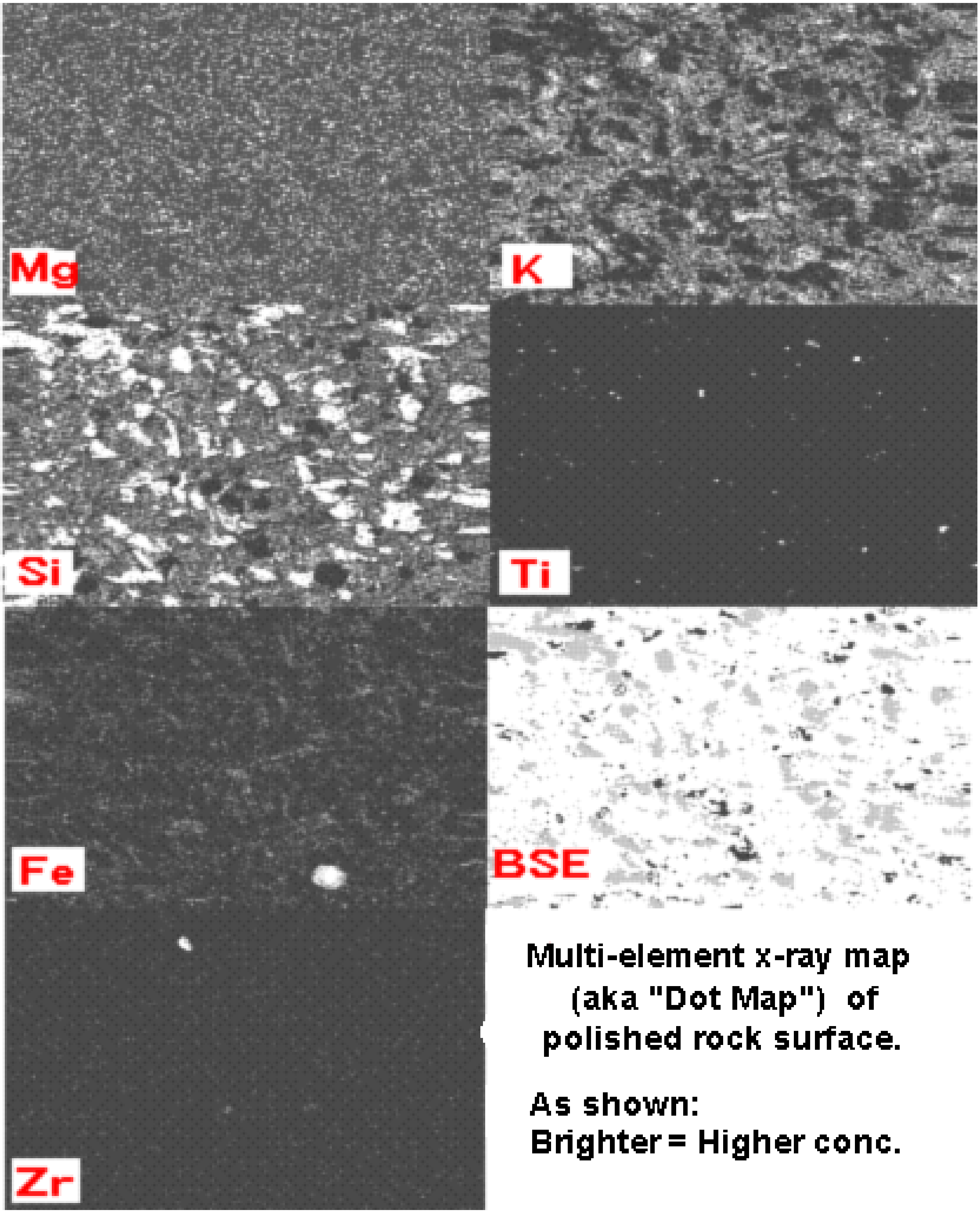
In the "spot" mode, the raster (sweep) is stopped and the beam directed to a desired analysis point within the field of view. The actual size of the analysis volume in spot mode is discussed elsewhere.

X-ray data can be used to form images.

X-ray mapping generates images of a field of view based on elemental composition. An area of a specimen is scanned as an array of single spots (i.e. pixels) while x-ray counts are collected for each. Areas of higher element concentration produce higher count rates that translate to higher pixel density/shading in the image. The data is presented as a halftone image for each element mapped.

Mapping is sometimes limited to a single element (at a time) if collected in an analog format on a SEM. With computerized EDS systems, however, many elements may be <u>mapped</u> simultaneously. In addition, software is available which can overlay pseudo-colored maps to make a color coded image of elemental distribution, quantify the percent area vs. composition, etc.

An example of a multi-element map is on the following page.



Multi-element x-ray map
(aka "Dot Map") of
polished rock surface.

As shown:
Brighter = Higher conc.

8. ELECTRON BEAM / SPECIMEN INTERACTION

SCATTER / RESOLUTION & SIGNAL PRODUCTION / ABSORPTION

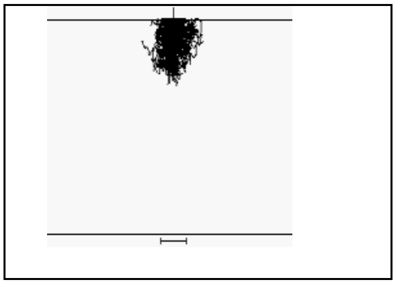
The electron beam diameter at its focal point on a specimen is a function of various settings on the SEM and the type of electron gun employed. It may be any diameter from a few angstroms (10^{-10} meters) to several microns (10^{-6} meters).

Resolution can never be better than the diameter of the beam. Because of electron scattering in the specimen, signals are usually generated from an area much greater than the diameter of the beam. The size and shape of this interaction volume is a function of beam energy (kV.), incident angle, and the "stopping power" of the specimen - for both the incident electrons and the generated signals. Stopping power of a material is related to its density and atomic number (Z).

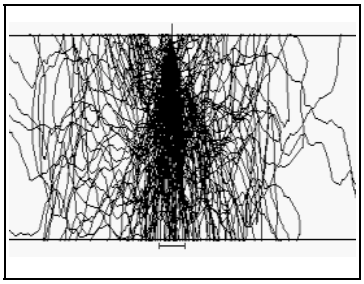
An incident electron entering the specimen may generate a secondary electron, a x-ray, or emerge as a backscattered electron. Other interactions occur, but are not typically used in the SEM. Which type of signal is generated by an electron is a matter of statistics.

A statistical computer program called "Monte Carlo" is available to simulate electron scatter in a specimen. The examples below are graphic outputs from three different runs. In each case, the electron beam enters normal to the surface at the top center.

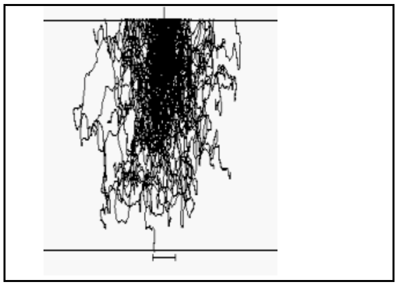
The marker, |----| , at the bottom of the image is one micron.



Aluminum, 5kV



Aluminum, 20 kV



Iron, 20 kV.

Since secondary electrons have very low energy (easily stopped), most originate from the immediate area around the beam. Backscattered electrons are those which make their way back to the surface and escape. Secondary electrons can also be generated by backscattered electrons as they exit the surface. X-rays are more penetrating than electrons and, if generated, may exit the surface well beyond the area shown in the above electron scatter models.

Like electrons, lower energy x-rays are more easily absorbed than are those of higher energy. It follows that better spatial (x-ray) resolution for x-ray imaging (maps) can be obtained by utilization of low kV. x-ray lines when possible.

Typically the best spatial resolution is achieved at low beam voltage and with high atomic number specimens. There are, however, trade-offs in the real world. As the beam voltage is lowered, it is more difficult to focus it to a small round spot and beam current is usually reduced. Another trade-off is that for maximum x-ray production efficiency, the electron beam should be 2 - 3 times the energy of an x-ray line to be examined. This is known as “over voltage”. Higher energy X-ray lines can be useful in the positive identification of an element. Unfortunately higher beam voltages mean larger excited volumes resulting in a loss of x-ray (spatial) resolution.

9. OPERATING CONDITIONS - EFFECTS ON X-RAYs

Both specimen geometry and operating conditions of the SEM can have profound effects on x-ray spectra. Effects are similar for EDS and WDS techniques. In the examples below, system geometry was varied by changing the tilt angle of a polished specimen surface. It is important to remember that on a rough surface such as a fracture surface, the instantaneous tilt angle may vary greatly. Although it is not impossible to estimate crude relative elemental concentrations from a x-ray spectrum, great care must be used to consider all variables. To attempt to quantify a spectrum simply by the ratio of peak heights can result in huge errors.

Changes in tilt angle will affect the average depth from which x-rays are emitted. A change in depth will change the absorption path length for the x-rays. Higher angles of tilt result in a shorter absorption path, permitting more x-rays to escape. Lower energy x-rays (typically lighter elements) are more affected than higher energy x-rays.

Quantitative x-ray analysis, except in very special cases, is performed ONLY on a well polished surface. Even for a polished surface, light elements present a difficult quantitative analysis situation since they produce very low energy x-rays whach are strongly adsorbed. Conductive coatings can add to the absorption path as well as adding their own x-ray peaks.

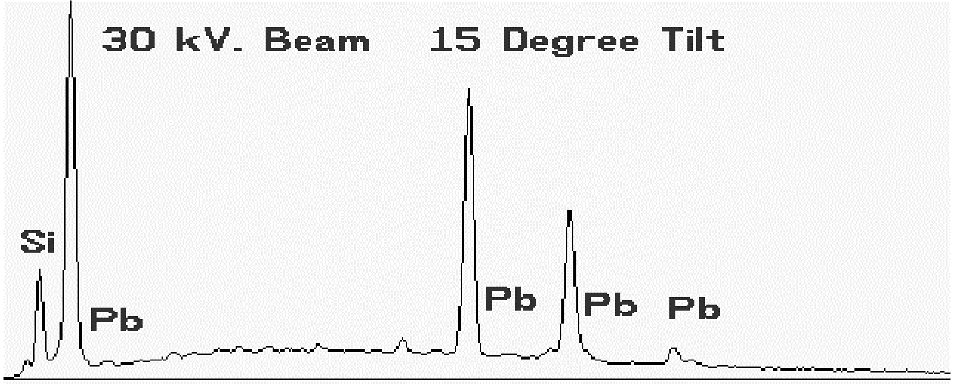
Change in incident beam energy (kV.) has two primary effects.

As the beam kV. is increased, so does it's penetration. This will increase the x-ray absorption path for a given tilt. More low energy x-rays which are generated - will be absorbed.

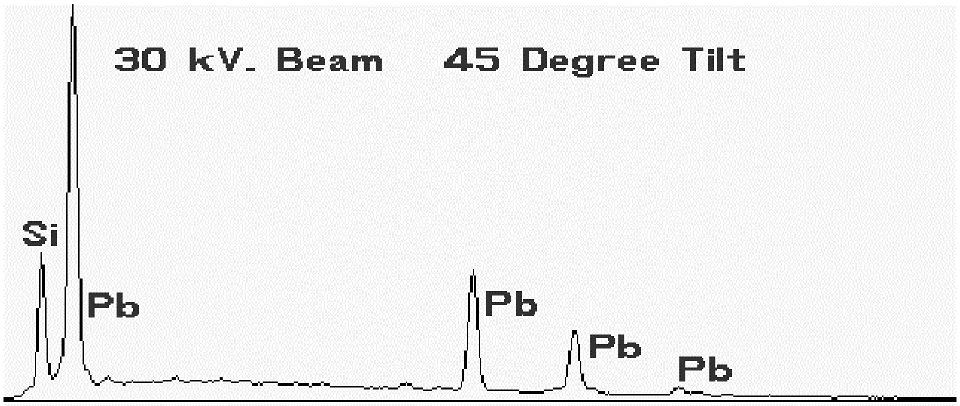
X-ray yield reduces as the energy of the x-rays approaches the energy of the beam. As the beam kV. is increased, the relative yield of higher energy x-rays will increase. The effect is to raise the spectral response at the high energy end of a spectrum**.**

Practical examples are illustrated on the following page.

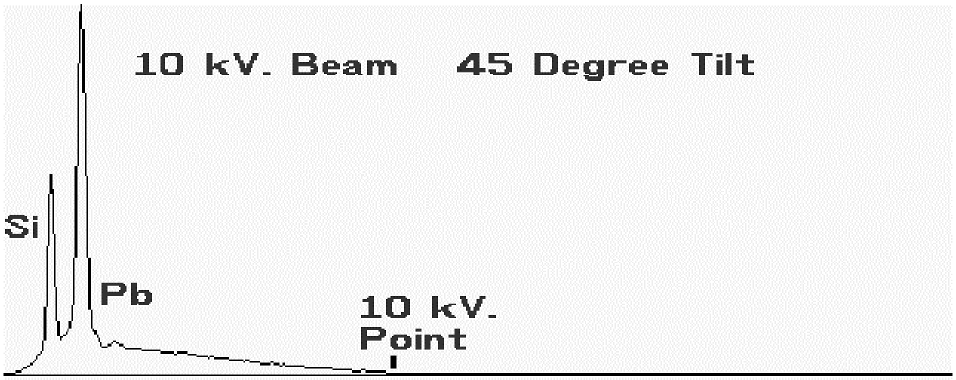
All spectra are normalized to make the tallest peak full scale.   Left to right scale is 0 - 20 kV.



At 30 kV. beam and 15 degrees tilt, lower energy x-rays are absorbed in the specimen and more high energy peaks are generated making the higher energy lead peaks appear taller. Note reduced low energy background.



When tilted to 45 degrees, the absorption path is shorter, increasing the low energy x-ray yield. The taller low energy peaks make the higher energy peaks appear smaller when normalized. Note increase in low energy background.



At 45 degrees tilt and only 10 kV. beam, the absorption of low energy x-rays is further reduced. Notice the apparent increase in Si relative to Pb at the left side.   The higher energy Pb peaks are greater than 10 kV. and are not generated.

SAMPLING TECHNIQUES & PRECAUTIONS

SEM specimens are diverse in type and source, but are typically solids. Gases and liquids are not usually candidates for SEM examination. Certain "environmental" SEMs can however, accept liquid or wet specimens. Specimens may be bulk material, fracture surfaces, powders, deposits or corrosion on surfaces, etc. They may be metals, ceramics, composites, plastics, biologicals, etc.

Whatever type of specimen is examined, it must usually meet three criteria:

- It must be electrically conductive or be coated with a conductive film.*
- It must be stable under an intense electron beam.
- It must be stable under a vacuum of about $1x10^{-5}$ Torr.*

Specimens that are volatile under the above conditions can contaminate the SEM internals.

* There are exceptions:

An "Environmental" SEM can operate at chamber pressures in the range of 20 Torr. This permits imaging of wet specimens and uncoated insulating specimens.

Imaging with low beam voltages (˜100 - 2000) can sometimes reduce beam damage and charging effects on insulators. For a given specimen composition, the beam voltage can be adjusted until the beam energy entering the specimen is equal to the energy leaving the surface (charge equilibrium). Field Emission gun SEMs are best suited to low kV. SE imaging.

The first rule in collecting any specimen is to preserve its integrity, introducing as few artifacts as possible.

This process should start the moment a need for a SEM examination is discovered.

SPECIMEN / SYSTEM COMPATIBILITY CONSIDERATIONS

SEM hardware and software must be considered when a specimen is generated...

SEMs have various chamber sizes.   The specimen must fit!

In general, specimens should be made as small as practical which will maximize positioning flexibility. Reduction of outgassing is another important reason to keep the specimen small. If the specimen is large and porous, long pump down times and poor vacuum can result.

SEMs may be configured with all possible accessories for full analysis capability: Quantitative imaging, EDS, WDS, etc. Others may have only basic SE imaging. Check capabilities of the SEM you have in mind to see if it will meet your analysis requirements.

PRACTICAL COLLECTING

The examination surface should not be touched if possible. Microstructure may be altered and contamination added to the surface. Never try to mate fracture surfaces, the surface detail **will** be damaged.

If the material to be examined is radioactive, toxic, etc., protective gear (gloves, goggles, etc.) must be used when collecting a sample. It is *always* a good idea to wear clean, lint-free or plastic gloves when a SEM specimen is collected. Contamination of the specimen from the hands is thus minimized. If gloves are used be sure they are *powder free* or it will look like a snowstorm hit your specimen!

Move the specimen to favorable, clean environment.... Store in a clean glass or poly bottle or poly bag. If a poly bag is used, avoid mashing the bag against the specimen during handling. Microstructure may be altered and poly embedded in the surface.

When specimens must be cut from bulk material, it is difficult to minimize contamination. If examination of surface chemistry is not required, a "dirty" specimen may be cleaned. It is important to **keep** the surface clean if x-ray analysis is required and surface chemistry may be altered by washing. Dry cutting and a poly "band-aid" over the area of interest can help minimize contamination.

If a specimen is dusty, a jet of high pressure gas *may* be sufficient to remove the contamination. This technique is effective on large, poorly adhered debris, but will **not** usually remove very small particles which present a small surface area to the blast of gas.

If a specimen must be more aggressively cleaned (ex: oily/dusty), select a solvent, acid, etc., least likely to affect the specimen. If it will not remove any deposits requiring analysis, an ultrasonic bath will greatly assist cleaning. Solvents should be selected to minimize washing away of pertinent material. For example, polar solvents like water/detergent clean well but will readily dissolve a chloride deposit. Ethyl, isopropyl, and other alcohols are less polar and also do well as drying aids when applied to water washed specimens. Acetone is an effective degreaser, but will attack many materials. A very good, but expensive solvent is trichlortriflorethane (Dupont FREON TF^{TM}) or a cousin. It is very aggressive against most oils but will not dissolve most metal salts or otherwise harm most materials. Freons are dense, have a low surface tension, dry quickly and are non-flammable. ...Ideal for an ultrasonic cleaner, but possibly hard on the ozone. More and more, its use is not permitted.

Always use caution when dealing with flammable materials! Many can be very hazardous if used in an ultrasonic cleaner.

Whenever possible, thoroughly and slowly air dry a porous or (metallographically) mounted specimen after wet preparation and/or cleaning. Immediate exposure to vacuum will hasten drying, but will cause any fluid trapped in pores or cracks to erupt to the surface. The fluid usually carries with it, small amounts of contaminants that are deposited on the specimen surface.

Other techniques can be used in cases involving surface analysis when the whole piece will not fit the SEM and it cannot be sectioned (cut).

DABBING

If loose surface debris (or powder) is to be analyzed, "dabbing" with pressure sensitive tape is often effective. In an emergency situation, common mylar office tape could be used. Protect the mastic/specimen from other contamination, but do NOT apply it to another surface to capture the specimen. SEMs cannot "see" through tape and the mastic is impervious to almost all solvents.

A much better approach is to acquire materials designed especially for SEM applications and make a "collection kit". A typical "kit" consists of a glass vial with stopper, and a SEM specimen mount (stub) with double face conductive tape applied to its surface. The vial stopper must be one that has a closed inside face. A hole is cut in this face to hold the stub in the stopper that will act like a handle. Double faced, carbon loaded tape for SEM use has a fairly low vapor pressure and is "spectrally pure". That is, if x-ray analysis is performed, the tape will contribute only carbon and oxygen x-rays. An example is shown to the right.

SCRAPING

Sometimes a deposit is adherent and must be scraped from a surface to collect a specimen. This is less desirable because sub-surface material is often collected along with the material of interest. The extra material in the specimen complicates analysis. NEVER use a scraper material which can interfere with the SEM analysis. For example: If tungsten is an element of concern in a specimen, do not use a tungsten carbide scraping tool. It is quite common to have chips or smears of the tool appear in a specimen when examined carefully. Scraping is useful only for elemental analysis of specimens. The material collected is severely deformed and usually will not yield any visual information.

REPLICAs

A replica is a (cast) impression of a surface of interest. Common materials used for making a replica are acetate film and silicone rubber. Conventional use of a replica is to examine surface features from an otherwise inaccessible location. Features are inverted in the casting and can be difficult to interpret. Most SEMs, however, can easily invert imaging video which makes the replica appear quite like the original surface. Most replicating material is a good insulator and a conductive coating must be applied.

Another use for replicas is collection of surface deposits. If a deposit is not tightly adhered, some will usually stick to the replica. The deposit can then be analyzed. As with the scrapper, always be sure the chemistry of the replicating material is compatible with the analysis requirements.

A nice side effect of replication is the specimen surface is sometimes cleaned. Unfortunately a rough surface can capture bits of plastic when the replica is removed. The plastic will charge if not removed or a conductive coating applied. Careful and repeated cleaning in acetone is usually effective in removing the remaining acetate.

11. SPECIMEN PREPARATION & MOUNTING

The diversity of SEM specimens is nearly infinite. They can be roughly divided into two families, materials and biologicals. In some cases, preparation and mounting techniques may over lap. In other aspects, techniques differ substantially. Material preparation methods will be discussed in detail. Biological specimen preparation techniques will be briefly covered.

SUPPLIES AND EQUIPMENT

MISC. TOOLS

Tweezers, hemostats, beakers, solvents, scalpels/hobby blades, scissors, diagonal cutters, heavy shears, dental picks, scribes, sample bags & vials, etc.

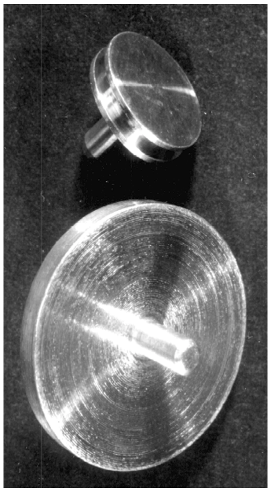
Metallographic preparation equipment such as saws, mounting presses, and grinding/polishing wheels are needed to prepare materials cross sections.

Microtomes (diamond knife) critical point dryers, cryo equipment and fixative/labeling chemicals are used to prepare biological specimens. On occasion, Microtomes are also being used in the materials arena.

A low power, stereo zoom, optical microscope (10x +) can be invaluable.

SPECIMEN MOUNTS (stubs)

Provide an interface between the specimen and the movable SEM stage (x,y,z,tilt, rotate). Most mounts are available in two or more "table" sizes. Typical are 0.5" and 1" diameters shown at the right. Many mounts have a bottom pin to engage the SEM stage. See figure to right.



CONDUCTIVE TAPES & PAINT

Shown to the right, are often used secure a specimen to the mount. There are two general types of tape; metallic, conductive, single face (adhesive) and a polymer, double face, conductive tape.

The metallic tape is usually copper or aluminum foil. This tape is used to "strap down" a specimen to the mount. Care must be taken not to cover the examination surface.

The double face tape, made conductive with carbon, is not an excellent conductor, but is quite sufficient for SEM work. Two faced tape and carbon "paint" are used "glue" specimens to a stub. Similar tapes and paints are available using other conducting materials such as silver, but carbon is cheaper and works well for most applications.

Carbon paint has another important use. If the specimen is a large insulator, such as a plastic metallographic mount, all but the area of interest can be painted to provide superior, uniform conductivity. If the area of interest is an insulator, a thin film coating (sputter, evaporate) must also be applied. No coating is required if the area is conductive and the paint contacts it. Painting areas can help "fix" dust and debris. This is especially helpful when dealing with radioactively contaminated or toxic specimens.

It can be handy to keep several “vintages” of carbon paint in use in the lab. As the (opened) bottles are used and time passes, they thicken, making available several viscosities.

Whenever possible, cure carbon paint in a clean, rough vacuum oven before loading into the SEM.

If it is convenient, or in cases where adhesives should not contact a specimen, it may be "clipped down". Clip type mounts may be purchased for most SEMs or can be custom fashioned from miniature copper alligator clips, stainless steel shim stock, etc.

Other preparation accessories may include a vacuum oven, ultrasonic cleaner, sputter coater, and a carbon evaporator.

BIOLOGICALS

Preparation of biological specimens can be complex and details are beyond the scope of this materials primer. Only a few aspects will be discussed.

A microtome can be used to section (slice) frozen or fixed biological specimens for either TEM or SEM examination. Critical point dryers or various solvents can be used to remove water from a specimen so that it is “fixed” and vacuum compatible (Sometimes unnecessary if a variable pressure SEM is used). Dehydration can change the character of a specimen and must be used with caution. Some SEMs are equipped with a cryogenic stage. If so, liquid nitrogen can be used to freeze tissue (lowering the vapor pressure) for direct viewing or, to facilitate fracturing for a cross section view. Biologicals are typically insulators and a conductive coating must be applied (except using VPSEM). A coating will also help protect the specimen from beam damage.

MATERIALS

LOCATING an AREA of INTEREST

Often there is a small specific area of interest on a large specimen that can be difficult to locate once in the SEM. Imagine trying to find a lost penny in a large gravel driveway at night with a small flashlight!

There are several techniques available to mark an area so that it may be quickly and positively located.

> SEM stage(x, y, z, tilt, rotate) motions are controlled either by micrometer drives or electric motors coupled to a joy stick, trackball, etc.. If the area of interest is known relative to an edge or large easily identifiable feature, the stage can simply be set to position over the area. This technique alone is most useful for large features.
>
> Small specific areas of a mounted and polished specimen may be marked using a microhardness indenter. A microhardness tester is equipped with an optical microscope and leaves tiny diamond or square impressions in the specimen surface.
>
> The easiest and often the most effective method is to use a fine permanent marker (ultrafine "Sharpie™") to simply draw a circle around the area of interest. The carbon based ink images well on most surfaces.

To mark outer surfaces of a polished "mount" specimen, see "POLISHED SECTIONS"

FRACTURE SURFACES

Measures should be taken to preserve a surface as soon as it is discovered.
Never fit two mating fractures together (a common problem). The fracture structure WILL be damaged.

Small, conductive fracture specimens may be examined with no more preparation than outlined in the section about collecting a specimen.

Very large fracture surfaces may have to be cut into suitably sized pieces to fit the SEM chamber. A cutting diagram is typically prepared prior to sectioning to locate the individual specimens in the large fracture.

Often fracture surfaces submitted for failure analysis have been exposed to environments which are degrading. Seldom does a "real world" specimen resemble it's pristine "textbook" (laboratory induced failure) cousin. Case history texts, however, provide both interesting and informative reading. Clues about the failure mode may be obscured by mechanical damage, corrosion or perhaps suspect contaminants may be washed away.

To avoid some of the above pitfalls, it is often useful to locate and examine fine crack tips. These can be easily located in polished section mounts using BSE. Another excellent approach is not to mount and polish, but to cut out the crack, minimizing "good" connecting metal. Then carefully "pry" or bend it open, exposing a fracture surface which will likely exhibit less mechanical and corrosion damage. At the same time, any material which may have contributed the failure is less likely to have been washed away and is available for analysis. **NOTE**: Beware of artifact material if the specimen has been dye penetrent tested.

For failure analysis of surfaces where environmental chemistry is suspected, an attempt should be made to examine the specimen surface "as failed". Preliminary imaging and detailed x-ray analysis work should be done at this time. After elemental data has been collected, the deposits can (we hope) be removed to improve the imaging quality of fracture surfaces. If aggressive solutions are used for cleaning, care must be taken not to mistake any cleaning damage for in-service damage.

POLISHED SECTIONS

A good, clean polished surface is essential for accurate quantitative x-ray analysis!
Because a surface looks good under an optical microscope does not always mean it is suitable for a critical SEM examination. No films must remain from grinding and polishing. Etching should be avoided if specimens are to be elementally analyzed. Not only will it roughen the surface, but may remove some element of interest.

Some quantitation software will model signals from irregular objects (like small particles or features) in an attempt to properly calculate concentrations. While certainly better than no capability, this modeling is usually approximate and accuracy may suffer.

If the examination requires x-ray analysis, preparation materials should, if possible, be chosen which differ from suspected deposits or contaminants. This will avoid mistaking any remaining preparation debris for components of the specimen.

Water is the usual fluid used for grinding and polishing. If any components of a specimen are water soluble (e.g.. chlorides, etc.) they may be washed away during preparation. If this is suspected, a specimen should be prepared without water, using lubricants such as decane/high purity kerosene and cleaned using a non-polar solvent. Store specimens under vacuum or in a desiccator since many of these compounds are hydroscopic.

To prepare polished cross-sections of materials such as metals and ceramics, specimens are often embedded in cold casting, or thermosetting resins. Casting resins are typically catalyst curing acrylics or epoxies. Thermosets are often mineral filled and are hot pressed with the specimen embedded. Standard sized molds or heated presses are used to make a small "hockey puck" containing the specimen. Specialized equipment is used to grind flat, and polish the specimen.

Sometimes the outside surface of a cross section (as a deposit layer for example) images nearly like the mount and it is difficult to locate the interface. To define the interface, the surface can be gold sputtered before it is mounted and polished. The thin gold boundary line is easily seen using BSE imaging.

In a similar fashion, some specimen surfaces may be both marked and **protected** by plating a relatively thick layer of metal. Either electroless or electroplate techniques may be used. Beware that plating solutions may radically alter deposit chemistry and should not be used if x-ray analysis is required.

PREPARING A MOUNT

The choice of mounting material depends on the character of the specimen. If a specimen may be damaged by pressure or heat, the proper choice would be a cold set resin. Vacuum outgassing of the resin and vacuum impregnation are recommended to fully infiltrate the specimen and avoid voids in the mount. Curing times range from less than an hour to overnight. A disadvantage of some cold mount resin is shrinkage that can produce small gaps between the mount material and the specimen. Gaps exhibit two potential problems when a mount is examined in the SEM. Unsupported specimen edges will round during grinding and polishing causing a loss of edge detail and surface deposits. Gaps can also accumulate polishing fluids and debris and are tough to clean. This can make a good polish difficult. Also, the fluid and debris can creep (out) over the polished surface after preparation. This is especially troublesome if the mount is placed under vacuum before it has completely dried.

Hot press mounting of a specimen with a thermosetting resin is relatively quick and produces a dense mount with good specimen/resin edge retention (reduced gap). Resins are often mineral loaded to increase hardness and reduce shrinkage (saves resin too!). Mineral loading is also used with some cold set resins. The main disadvantage of hot pressing is the potential for heat or pressure damage to a fragile specimen. Special equipment is also required.

SEM specimens must be conductive or coated. None of the mounting systems described are normally conductive. If analysis requirements prohibit a conductive coating and it is not practical to (conductive) paint around the specimen, an electrically conductive mount material should be used. Most use carbon, iron, or sometimes copper loading in the resin to obtain bulk conductivity. “Homemade” conductive resins can be made by adding a (rich mixture) metal dust to the conventional resins. Zinc dust is readily available and has proven to work well. Others may work well too - experiment.

Beware: Most metal dusts are either pyroforic or highly combustible and may be difficult to extinguish. Beware of metal dust/resin compatibility as well.

All of the conductive mount systems exhibit bulk conductivity but are not homogenous and still have “islands” of insulating resin. The interior of a conductive specimen may be examined with no charging problems. If the edge of a polished specimen must be examined and it is in close proximity to one of the resin islands, localized charging will occur. A conductive coating may then have to be applied.

Another conductive mounting material successfully used in a hot press is “Woods Metal” or a similar low melting point alloy. The major advantage is that the alloy is a fully conductive material with no charging islands. It can also provide some shielding for use with radioactive specimens. These alloys are, however, very soft and make polishing difficult.

FILTERING

SEM specimens can be collected by filtering solids from a liquid sample. If the solids form a thick deposit on the filter, almost any filter media is suitable. The entire filter, or a portion, can be easily mounted to a “stub” coated if indicated, and examined. Most filter material is open and rough, forming deep crevices. If the resulting layer is thin and composed of tiny particles, much of the specimen can be buried within the filter. For rare combinations of sample and filter, an option may be to dissolve the filter in an attempt to recover the sample.

Proper selection of filter type and material to optimize an examination is important. If the sample is large and abundant, fibrous filters are acceptable, but if x-ray analysis is required, care should be taken in the selection of the material in an attempt to minimize interfering x-ray lines. It is also advisable to obtain a virgin filter for use as a comparative reference (control specimen). Filters are available in various glasses, cellulose, and other organics. Try to use one that will not conflict with analysis goals. Some organic filter media is unstable in the SEM and care must be taken to minimize its beam exposure damage. Membrane filters can be quite useful since they have a flat surface with holes of a specified size. They are ideal for use when a SEM exam is anticipated. Their disadvantages are cost, low flow/high pressure drop, and they are fragile.

PARTICLE ANALYSIS MOUNTS

Particles may need to be dispersed on a mount for image/x-ray analysis. Some samples may be simply “dusted” onto a specimen mount covered with conductive carbon tape. Some vendors offer special tape with mastic dots in a tiny array. Another way to disperse particles is to suspend a small amount in a liquid, then apply a drop to a specimen mount and let dry. Control the mounted particle density by adjusting the concentration in the liquid. Larger particles can also be mounted, ground, and polished. Coat as needed.

12. SPECIMEN COATING

Specimens examined in a SEM are bombarded by an intense electron beam. If the specimen is a good conductor of heat and electricity, or a variable pressure SEM is used, no coating is usually required. A clean metal fracture is a typical example of a specimen that need not be coated.

If conductive specimens are mounted in an insulator (i.e. polished metallographic mount), a conductive coating may be required to connect the mounted specimen to "ground". When it is not necessary to observe edges, carbon paint can be used to coat the (insulating) mount, leaving the conductive area of interest uncoated.

If the specimen is an electrical insulator or subject to beam damage, a conductive coating must be applied to the surface. The goal is to provide a continuous discharge path ("ground") for electrons with a coating thin enough to avoid obscuring surface detail. Thickness of most coatings are in the range of several 10 of nanometers.

Uncoated insulating specimens will charge with electrons from the beam, disrupting imaging. The effects can range from apparent poor resolution to complete deflection of the incident beam. A strongly charged specimen can even act as an electron mirror, causing the internals of the SEM chamber to be imaged!

Specimens that are sensitive to beam damage (typically heat) may be protected to a degree by applying a coating that is a good thermal conductor, such as gold.

Often a conductive specimen may have accumulated particles of insulating dust/debris or non-conductive phases/inclusions. This can cause spot charging in an image. It is easy to identify such a problem from its characteristic appearance. In secondary imaging, the charged area will appear abnormally bright, surrounded by a dark halo. To avoid charging problems, the sample must be cleaned or coated, depending on the situation.

Sometimes, charging of non-conductive specimens may be minimized by using very low beam potentials (kV.) For some specimens, using beam energies between 100 eV. and 2 kV., an equilibrium can sometimes be achieved between incident beam input and signal output. This will result in a net zero charge accumulation. High resolution imaging at very low beam kV. is difficult and is only practical with specialized, state-of-the-art equipment. Often, x-ray analysis is not possible while imaging in this mode because most characteristic x-rays will not be generated due to the low beam voltage.

Some insulating specimens may be made conductive by infiltration with chemicals which modify their chemistry. This is technique is not in common use for materials science, but is often used for biological specimens.

COATING MATERIALS AND TECHNIQUES

There are a multitude of coatings materials and techniques. The most common are described.

SPUTTER COATING: (Low vacuum, Diode, D.C., R.F., Ion gun, "Magnetron")

The most common instrument used for coating SEM specimens is a low (rough) vacuum, “magnetron” sputter coater. The term "magnetron" should not be confused with the tube used in a microwave oven. It implies there is an annular magnet surrounding the sputter "target". The magnet deflects energy generated in the sputter coater which otherwise would cause excessive specimen heating. Although it is possible to sputter many materials, the most commonly used targets are pure gold or a gold-palladium alloy. These materials are easy to sputter and provide a thin, fine grained, electrically and thermally conductive coating. The metal layer also helps protect beam sensitive specimens, but can sometimes interfere with SEM analysis requirements.

Sputtered Au or Au/Pd are the most often used coatings for high magnification and high resolution imaging. It is not recommended when x-ray analysis is required. Instead, carbon coatings should be used.

EVAPORATIVE COATING: (Low vacuum, High vacuum)

Thermal/vacuum evaporation may be used to apply the same coatings materials as the sputter coater. This is not normally done, however, because the coating thickness is more difficult to control and the film grain size is usually larger. Since much thicker coatings can be applied quickly, however, "problem charging" specimens may benefit. Unfortunately, fine surface detail will possibly be obscured.

Carbon cannot be easily sputtered. Evaporative coating is most often used to apply a carbon film. It is usually the coating of choice when x-ray analysis or sensitive backscattered (atomic number) imaging is required because carbon least attenuates most x-ray and BSE signals.
High magnification secondary electron imaging is degraded by carbon, relative to a coating like gold. This results in a poor SE signal-to-noise ratio, but carbon x-rays do not overlap or significantly absorb most other (x-ray) lines.

13. COATING EFFECTS on BSE IMAGING

Poorly conducting specimens can be examined without coating by using a variable pressure SEM. Many labs may only have a high vacuum SEM, or for other reasons, choose a high vacuum mode. In order to examine insulating specimens in a high vacuum SEM (using operating conditions conducive to BSE and X-ray analysis) specimens must be coated with a conductive thin film.

Back Scattered Electron (BSE) image contrast is primarily a function of the average atomic number of an imaged area. This is particularly true for polished specimens where there is no topography to contribute to contrast. The *BSE coefficient* is the ratio of back scattered electrons to incident (beam) electrons. Higher atomic number elements have a higher BSE coefficient (i.e. are more “reflective”). A higher rate of detected BSEs will normally produce a brighter image response.

Higher atomic number (Z) compositions are brighter.

Lower atomic number materials are darker.

BSE imaging is invaluable when searching a specimen for variations in composition. An uncoated, polished, conductive specimen is ideal for most SEM procedures including BSE imaging. With such a specimen, variations in compositions can be seen using BSE imaging that cannot be detected by most Energy Dispersive Spectrometers (EDS).

The real world, however, is seldom that nice! Conductive coatings must be used which *will* influence the examination. The obvious goal is to use a coating that will minimize this influence. The coating of choice will depend on the examination goals. Considerations must be given to a number of variables including the secondary electron (SE) resolution requirements, BSE sensitivity, X-ray analysis requirements (for both interference lines and loss of detectability limits), etc. There are always exceptions, but in general, the thinner the layer and the lower the atomic number of the film, the better.

Electron penetration (and subsequent escape as a BSE) depends on the composition of the specimen (*and the coating*), beam incident angle and the beam potential. “Monte Carlo” computer programs are available to model the analysis volume generated. Under many operating conditions, subsurface composition variations can be observed using BSE imaging. The ability to subsurface image permits the specimen to be coated for conductivity and maintain composition contrast. There is unfortunately a trade-off. Any coating will reduce apparent sensitivity to composition changes. A coating material and method must be selected to provide conductance while minimizing sensitivity loss.
Beryllium may make the ideal film for many BSE exams, but is not practical because of its toxic properties. Carbon is the best practical material for most BSE and X-ray examinations. Since carbon is difficult to sputter, it is typically applied by evaporative coating.

For enhanced secondary electron imaging resolution, specimens are often sputter coated with gold, palladium, Au/Pd alloys or similar alloys. The thickness of these sputtered noble metal films must be minimized to maximize BSE sensitivity.

In an attempt to quantify the loss of BSE sensitivity with increasing gold film thickness, the following experiment was performed.

The specimen was a polished tool steel mount, imaged under nearly identical conditions for each test. There is only a small amount of unintended topographic relief resulting from hardness differences in the specimen (polishing artifact). This material has precipitates of several atomic numbers (Z), best seen in the “uncoated” condition, Figure 1. Composition contrast from the specimen is shown to reduce as the coating “Z” is raised and/or the coating thickness increased.

During the experiment, the absolute brightness was adjusted as necessary to yield usable images. As the gold coating thickened and became a more significant portion of the BSE *analysis volume*, the “apparent” average Z of the specimen increased. The brightness (dark level/DC level) adjustment was set for approximately the same specimen matrix gray level for each example. The contrast (BSE detector gain) and beam current were constants.

Figures on the following page show that thicker, higher "Z" coatings increasingly mask the underlying material.

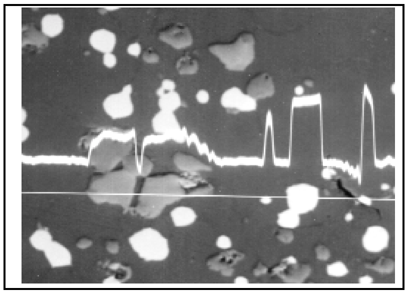
Similar problems occur with x-ray analysis (EDS, WDS) also. Not only can characteristic x-ray lines from coatings mask other elements, but detectability limits for elements in the specimen will be reduced as the coating increases. This is especially significant when analyzing for lighter elements.

Graphic results of the experiment are shown in Figures 1 - 4. Figure 1 is the uncoated specimen that is referenced as a 100% BSE response. The carbon coated surface is shown in Figure 2 and exhibits a response that is 89% of the uncoated surface. This thickness, applied using a “yarn” type evaporator is estimated to be in the realm of 100 nm. Figure 3 shows the reduced response from an indicated 4 nanometers of gold. It is only 30% as intense as the uncoated surface. In Figure 4, additional 8 nanometers of gold were applied for a total thickness of 12. At this thickness and beam potential, almost all composition contrast is lost. Nearly all BSEs are originating from within the coating rather than the specimen. The accuracy of the gold thickness is not established. A quartz crystal (oscillator) supplied as an accessory to the sputter coater was used to obtain the reported coating values.

The relative effects are valid, but unfortunately, the beam voltage used for this experiment is not available. The voltage was likely on the lower end (5-10 kV). At higher voltages (20-30 kV) the coating effect would be reduced as the analysis depth is increased and the coating signal becomes a smaller component of the total BSE signal.

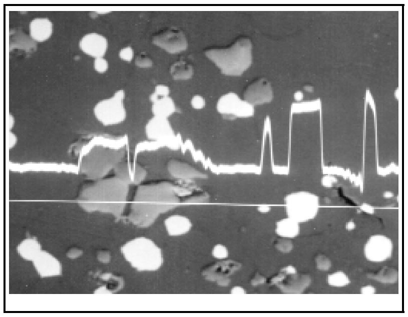
Description of images

- Matrix is lowest atomic number phase (darkest).
- Large phase is next higher Z. Small phase is highest (brightest).
- The left-to-right straight line through the image is the line of analysis.
- The stepped, wider line is a plot of the image brightness along the line of analysis.



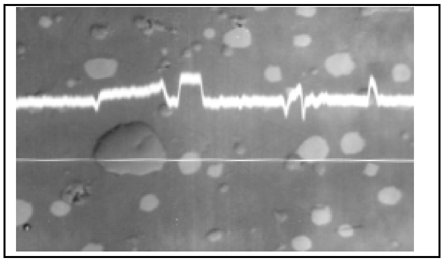
Uncoated surface, relative response is 100 %.

Fig.1



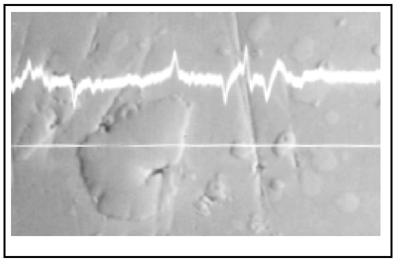
Carbon coated surface. Response: 89%
Applied using yarn evaporator.
…Thickness not determined.

Fig. 2



Gold sputter coated (~4nm). Response: 30%

Fig. 3



Gold sputter coated (~12nm). Response, Almost none. Most is topographic, not composition.

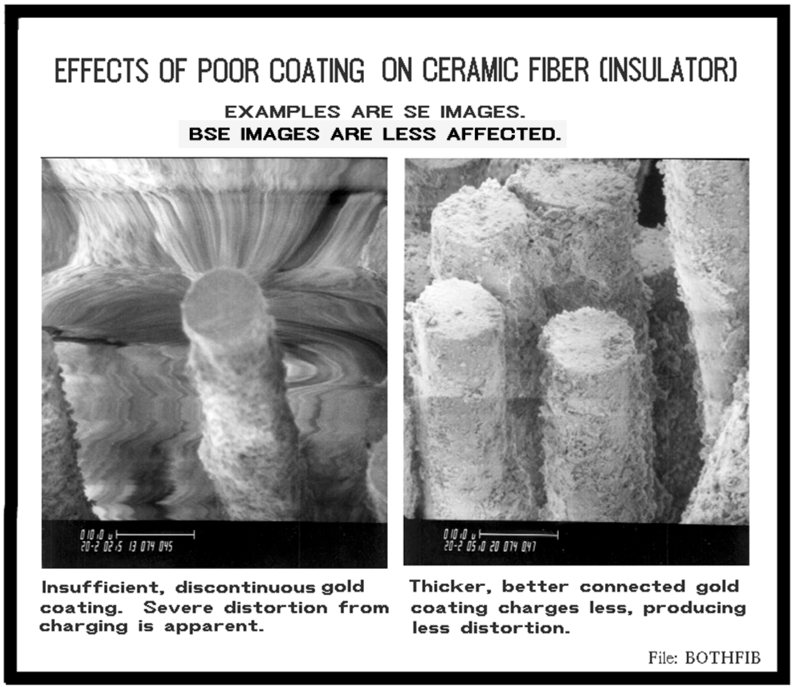
Fig. 4

14. CHARGING ARTIFACTS

Improper or insufficient coating can introduce imaging artifacts.

Charging can totally change an image. What you see is not necessarily what you have…

A quite common effect of a small piece of charging debris on an otherwise conductive surface is a very bright white response from the debris, ringed by a "too dark" zone. As the debris is charged, its field affects the relative potential in the immediate area around the charging particle, causing it to be dark.



EFFECTS OF POOR COATING ON CERAMIC FIBER (INSULATOR)

EXAMPLES ARE SE IMAGES.
BSE IMAGES ARE LESS AFFECTED.

Insufficient, discontinuous gold coating. Severe distortion from charging is apparent.

Thicker, better connected gold coating charges less, producing less distortion.

File: BOTHFIB

If the charging level is high enough, the electric field will deflect the beam away from the specimen altogether. For example, if an attempt is made to image an insulating sphere, it can charge, reflect the beam (electron mirror) and actually give a fish-eye view of the chamber internals.

HINT: If charging is not severe, but is disrupting SE imaging, try BSE imaging. Since most backscattered electrons are higher energy than secondary electrons, they are less affected by stray charges. Severe charging, however, will disrupt even BSE imaging.

Specimen charging can cause x-ray analysis errors.
As charging occurs, the potential level of the surface is no longer at "ground reference". The beam is referenced to ground. Therefore, the **effective** beam kV. is the beam potential minus the surface potential. This can be seen in a (graphic) EDS spectrum. The EDS "background" will normally taper to zero counts at the beam kV. For a charged surface, the background will taper to zero at a lower kV. than the beam. For example, if the beam is set to 20 kV. and the EDS background tapers to zero at 16 kV., the surface has charged to 4 kV. This condition will have three effects. First, as described above, the beam may deflect or wander making it difficult to keep it on the area of interest. Second, higher kV. x-ray peaks may be attenuated or disappear as the effective kV. (and thus, the over voltage margin) is reduced. Third, quantitative x-ray analysis software uses the beam voltage (among other things) to adjust concentration calculations. If the beam voltage variable is wrong, so is the quantitation.

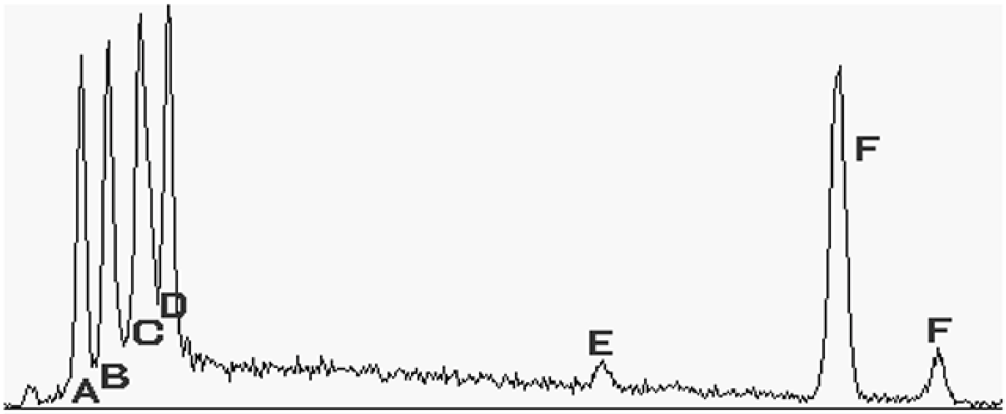
15. COATING EFFECT on X-RAY ANALYSIS

EDS: Energy Dispersive X-ray spectroscopy
WDS: Wavelength Dispersive X-ray spectroscopy

Most common coatings: CARBON, GOLD, GOLD/PALLADIUM

The effects of coatings are similar in both EDS and WDS x-ray analysis. There are two major categories of effects. First, like the BSE imaging effect, is a reduction in desired signal. Typically, thick high "Z" coatings will attenuate the x-ray response from the specimen compared to thin low "Z" coatings. This will raise the minimum detectable elemental concentration (MDC). Secondly, the coating may produce peaks that interfere with (convoluted with) the peaks produced by the specimen. While some software claims to be able to "de-convolute" peaks, nothing is better than an interference free spectrum! A spectrum free from artifacts speeds analysis and reduces error. If the specimen is to be quantitatively analyzed, the problem is further compounded. Both the attenuation and spectral contribution of the coating must be considered when calculating concentrations.

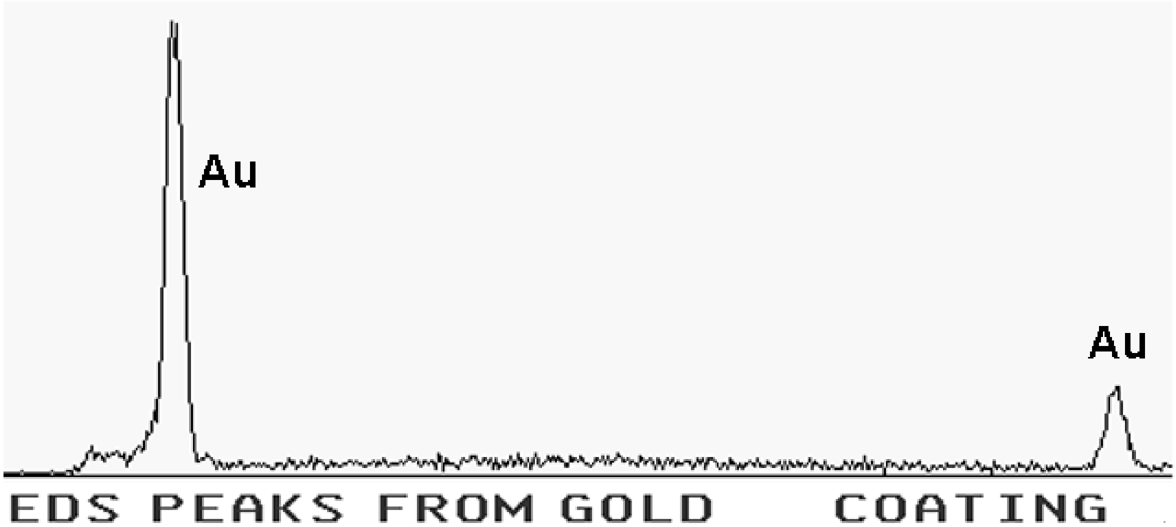
Carbon is typically the coating of choice for specimens requiring x-ray analysis. Although there are exceptions, it attenuates x-rays least and does not often produce interfering peaks.



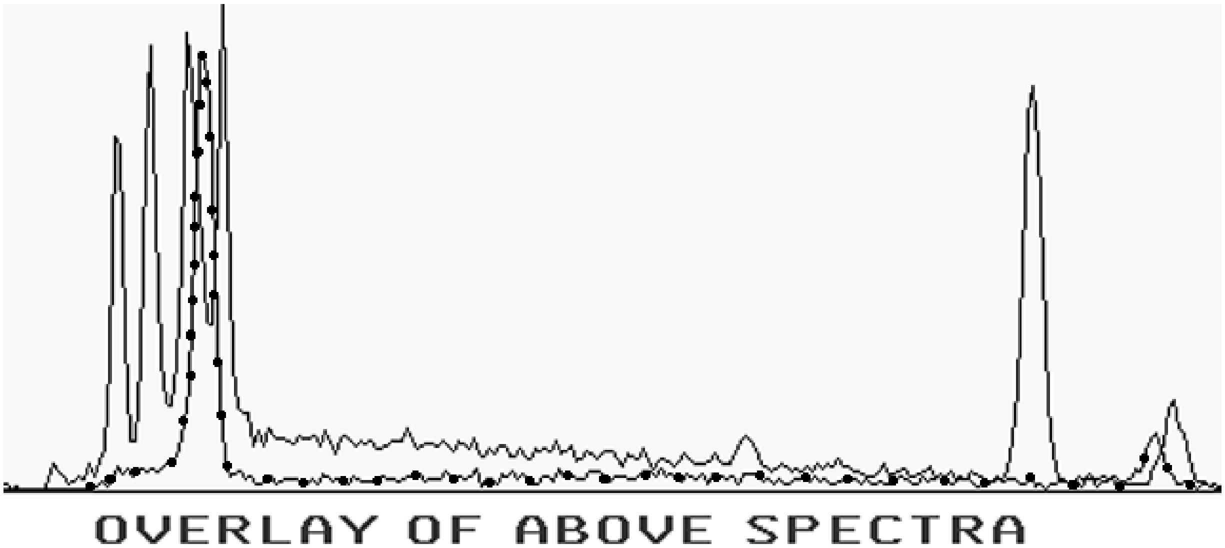
EDS PEAKS FROM COMMOM ELEMENTS
WHICH MAY OVERLAP GOLD

The above spectrum a composite, illustrating the peaks produced from several common elements. Multiple notations result from possible characteristic x-ray peak overlaps.

A: Al, Br **B**: Si, W **C**: P, Zr, Y **D**: S, Mo, Pb **E**: Fe **F**: Zn



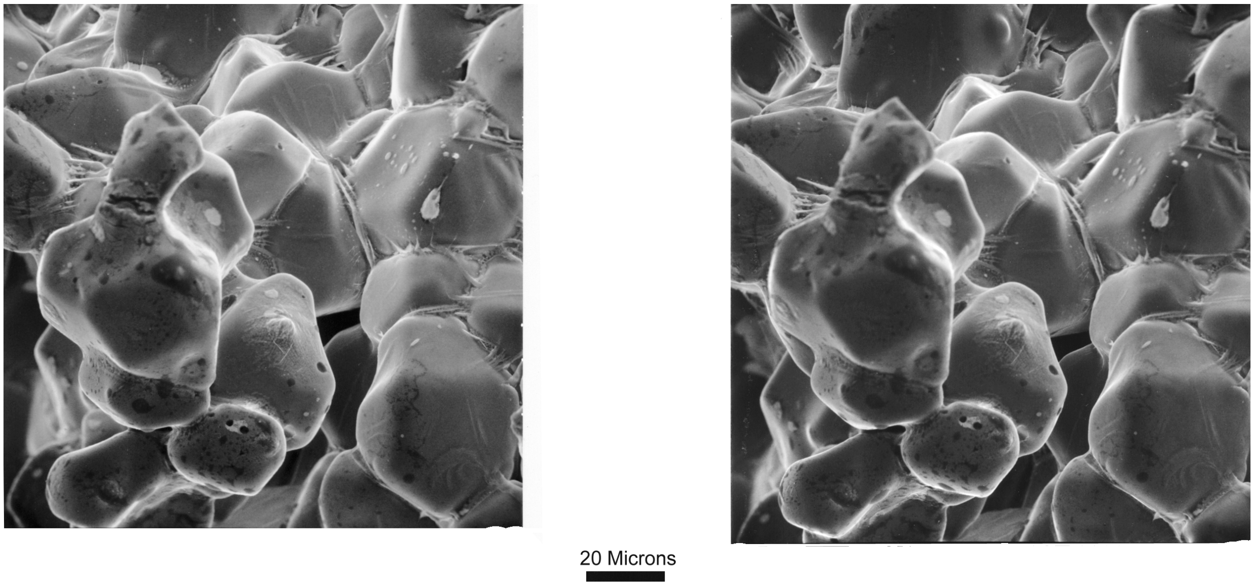
This spectrum is from pure gold. Carbon would produce a peak at the extreme left edge of the graph. It takes a special high performance, thin window EDS detector to acquire a carbon line.



When gold is added to the spectrum of many common elements, it becomes much more difficult to determine what elements may be present in a specimen.

16. STEREO PAIRs - 3-D Imaging

Stereo imaging can be a useful tool when trying to visualize topography. It is especially helpful when performing a failure analysis on a fracture surface. Stereos can also be used to calculate elevation changes in a field of view. The 3-D effect is typically observed using a special viewer (with hard copy) or on a computer display (with appropriate software).



Above is a stereo pair of a fracture surface. Two images were acquired from the same location. Between exposures, the surface was tilted about six degrees. This image presents a strong 3-D effect. Hint: The large grain to the left is a high spot and the black areas to the lower right are deep openings. The best effect is obtained using a viewing device made for the purpose, but some people are able to perceive some 3-D without a viewer. There is a technique, give it a try… If the picture spacing is not optimal for you cut them out and adjust the spacing. Use caution, it is possible to swap the images and invert the topography. Hold the images at a comfortable reading distance. Defocus your eyes and gaze “beyond” the images. Slightly cross your eyes (no kidding!). Then, at the same time, begin to focus and uncross your eyes. As you do so, you should “see” a third image between the two originals. Concentrate and focus on this center image. Good luck!

The specimen is an aluminum alloy fracture that resulted from overload while it was (for a few minutes) near its melting point in air.