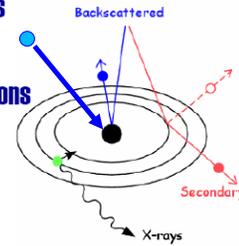


Scanning Electron Microscopy (SEM)

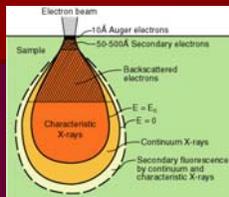
- What is it?
- How does it work?
- Interpreting images
- Samples

sample-primary beam interaction

- **Secondary electrons**
 - topography
- **Back scatter electrons**
 - compositional
- **X-rays**
 - chemistry



Interaction Volume



Depth of penetration increases as the accelerating voltage of the primary beam is increased. It will also be deeper when the sample composition is of a lower density and/or is of a relatively low average atomic number.

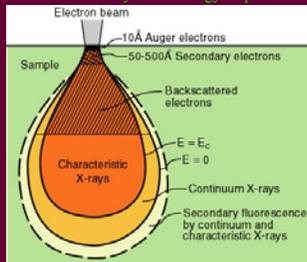
- SE, BSE, and X-ray are produced throughout the interaction volume provided the beam electrons still have enough energy to generate it.
- The SE signal is readily absorbed and therefore can only be detected relatively close to the surface (i.e. less than 10 nm).
- The BSE signal is of higher energy and able to escape from a more moderate depth within the sample.
- The x-ray signal can escape from a greater depth, although the x-ray signal absorption is actually variable depending upon its energy.

Beam Interactions

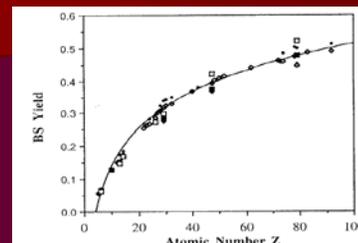
- The *secondary electron* (SE) signal derives its contrast primarily from the topography of the sample. For the most part, areas facing the detector tend to be slightly brighter than the areas facing away from the SE detector, and holes or depressions tend to be very dark while edges and highly tilted surfaces are bright. These electrons are of a very low energy and very easily influenced by voltage fields.
- The *Backscattered electron* (BSE) signal is caused by the elastic collision of a primary beam electron with a nucleus within the sample. Because these collisions are more likely when the nuclei are large (i.e. when the atomic number is large), the BSE signal is said to display atomic number contrast or "phase" contrast. Higher atomic number phases produce more backscattering and are correspondingly brighter when viewed with the BSE detector.
- *X-ray signals* are typically produced when a primary beam electron causes the ejection of an inner shell electron from the sample. An outer shell electron takes its place but gives off an x ray whose energy can be related to its nuclear mass and the difference in energies of the electron orbitals involved.

sample-electron beam interaction

1. Secondary electrons for the best resolution of surface topographical features.
2. Backscattered electrons give contrast based on atomic number to resolve microscopic composition variations, and topographical information.
3. Qualitative and quantitative chemical analysis: an energy dispersive x-ray spectrometer



Backscattered electrons



- Elastic scattering can deflect > 90°
- The Backscattered Electron yield is $\eta = N(\text{BS}) / N(\text{incident})$
- η increases with Z and incident angle
- η does not depend much on energy

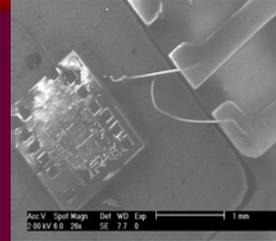
Backscattered electron Image (BSE)



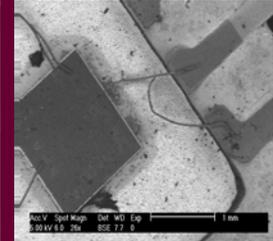
BSE image of Al-Cu alloy

<http://mse.istatc.edu/microscopy/backscat2.html>

SE vs. BSE Images



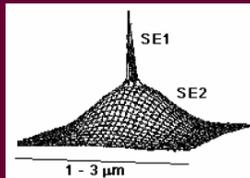
SE -- Edge effect, charge sensitive, very little Z contrast.



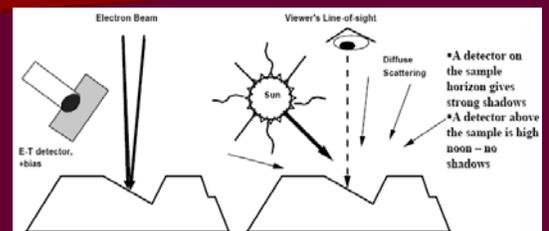
BSE -- Z contrast dominates, no edge effect, no charging seen.

Beam Interaction Effects

- The SE1 signal comes from an area 1-5 nm in diameter – it therefore contains high resolution detail on a scale comparable with the probe size
- The SE2 signal comes from an area that can be up to a few microns in diameter at high energies (the emission area of the BSE) - it is therefore low resolution detail and essentially is a copy of the backscatter image



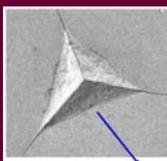
Contrast effects



Which detector to use?

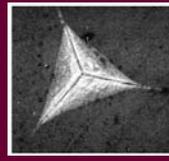
Lower Detector

- The detector position therefore affects the image appearance
- The lower (ET) detector views the sample from one side and so the face looking away from the detector is shadowed

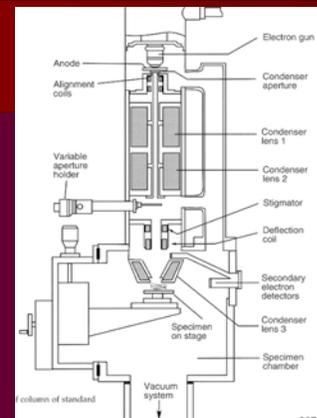


Upper detector

- The upper (through the lens) detector views the sample from above
- The SE collection is now symmetrical and so all faces of the indent are equally visible. They are brighter than the flat surface because of topographic contrast.



SEM



Generating incident electrons

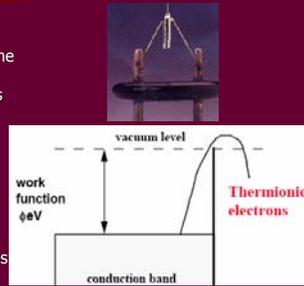
There are two methods for generating the primary electrons:

Thermionic emitter

- Boil electrons over the top of the energy barrier. $T \sim 2800K$
- The current density J_c depends on the temperature and the cathode work function ϕ - Richardson's equation.....

$$J_c = AT^2 \exp(-e\phi/kT)$$

- Cheap to use, modest vacuum required (W only)
- Can also use LaB6 which has a better performance but requires a higher quality vacuum



Generating incident electrons

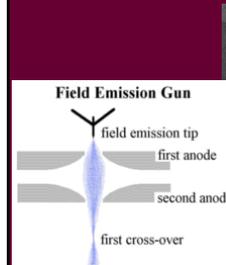
There are two methods for generating the primary electrons:

Field emission

- Electrons tunnel out from the metal because of the high field
- The field is obtained by using a sharp tip (100nm) and a high voltage

$$J_c = AF^2/\phi * \exp(-B\phi^{1.5}/F)$$

- Note: the emission is Temperature independent
- Needs UHV but gives long life and high performance



Typical Emitter Gun

Emitter brightness

At 20keV typical brightness values (units - A/cm2/str)

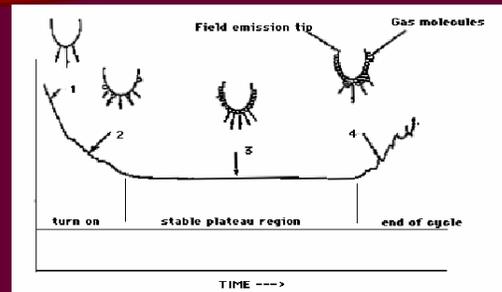
- W hairpin 10^5
- LaB6 10^6
- FEGs 10^8
- nano-FEG 10^{10}

Source Size

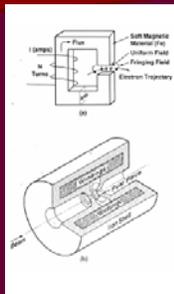
- Tungsten hairpin -50μm diameter
- LaB6 - 5μm
- Schottky FEG - 25nm
- Cold FEG - 5nm
- Nano-FEG - 0.5nm

The physical size of the tip is not necessarily the same as the source size

Life cycle of a Field Emission Tip



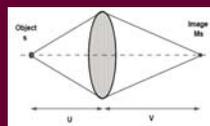
From Emitter to Lens



- A lens forms an Image of an Object
- Electron optical Lenses use magnetic or electrostatic fields
- The focal length f can be changed by varying the lens excitation (current or potential)

$$1/U + 1/V = 1/f$$

$$M = V/U$$



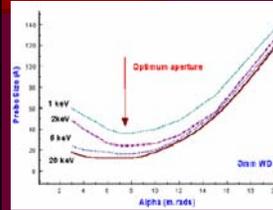
Column operations

- The SEM operator can vary the alignment of the beam using mechanical or electronic methods
 - Mechanical alignment can involve physically changing the aperture or beam path
 - Electronic alignment of the beam path can be done by
 - Varying the field at each electromagnetic lens
 - Varying the stigmation of each lens

Maximizing Resolution

- Spot Size
- Beam current
- Working Distance
- Use of a cold finger
- Limitations

Spot Size

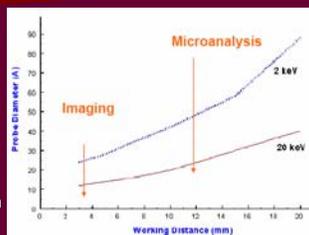


Variation of probe size with energy and beam convergence

- The spot size depends on the beam energy, WD, and the final aperture α
- Performance also improves with higher beam energies
- On many SEMs the aperture size is set automatically by the mode control

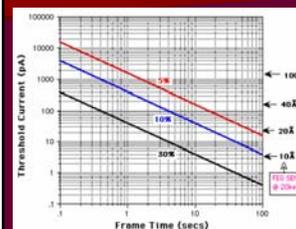
Working Distance

- *Working distance* is the most important user controlled parameter - shorter WD means lower aberrations, more current, and more efficient detection
- Always use the smallest WD that is possible for a given specimen and mode of operation. For imaging WD ~ 3 mm, microanalysis WD = 12mm
- Note also that the image resolution is almost independent of the beam energy in a state-of-the-art SEM



Resolution vs Working Distance

Beam Current

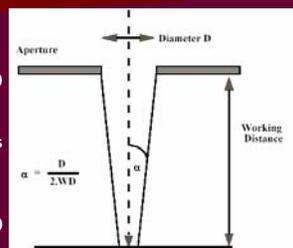


The Rose Criterion Nomogram

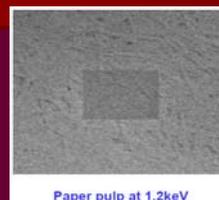
- A minimum beam current I_b - the Threshold Current - is required to image a given contrast level
- Typical SEM image contrast levels are 3-10% on most samples so high currents or slow scan times are needed
- Improving signal contrast lowers the required I_b and so improves the resolution
- Increase I_b by raising the tip emission current from 10 μ A to 20 or 30 μ A if necessary

Apertures

- The final aperture defines α which is usually ~ 1 - 20 mrad (i.e. smaller than 1/2 degree)
- When α is small the depth of field is high, the resolution is good, but the beam current is low
- When α is big - we get high current, but a big spot size and poor Depth of Field (DoF)



Charging Samples



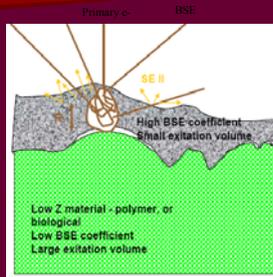
Paper pulp at 1.2keV



Paper pulp at 2.5keV

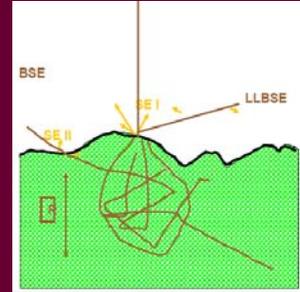
- If the scan square is dark compared to the background then the sample is charging positive or there is something on the surface
- If the scan square is brighter than the background then the sample is charging negative

The wrong way to coat



- Coatings can be **THICK** or **THIN**, particulate or smooth
- Coating are usually metal layers of a high Z material such as **Cr, Ta, W, Pt, Au**
- With a **THICK (20-50nm)** coating the beam interaction occurs mainly within the coating Layer
- The SE-signal is then comprised of SE2 (i.e. converted BS electrons)
- The topographic resolution is limited by the thickness of the metal coat and the SE II range

The right way to coat



- Use a **THIN** film
- The beam interaction is now mainly in sample
- The SE-signal is SE I from the coating
- There is very little SE II from the metal layer., and little or no signal contribution from specimen itself because SE produced beneath the metal layer cannot leave the specimen
- Topographic resolution is now limited by thickness of the metal coat and the diameter of the electron beam.

Energy-dispersive X-ray Spectroscopy (EDS or EDX)

- What is it?
- How does it work?
- Interpreting spectra

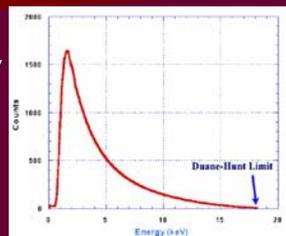
X-Ray microanalysis

- The fluorescent production of X-rays by electrons is one of the most important interactions available in the SEM because it permits elemental identification and quantitative analysis to be performed
- Elements can be detected at high sensitivity and with a spatial resolution on the order of a few hundred nanometers.
- X-rays are photons *and have an energy E and a wavelength λ* but have zero mass and no charge.

$$E = \frac{hc}{\lambda} \longrightarrow \lambda(\text{nm}) = \frac{1.226}{E(\text{keV})}$$

X-Rays

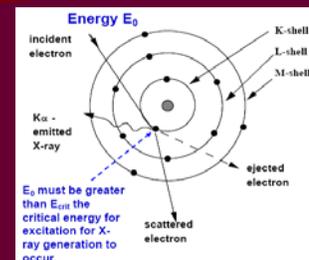
- X-rays come in two different forms- continuum X-ray and characteristic X-ray
- Continuum X-rays** are produced by the slowing down of the beam electrons in the Coulomb field of the sample atoms
- This gives a continuous band of radiation from zero energy up to the energy of the incident beam
- The **Duane-Hunt limit** is the point where the continuum intensity goes to zero and identifies the actual electron landing energy



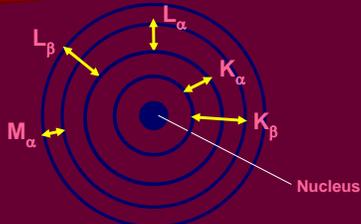
Characteristic X-rays

- Characteristic X-rays are formed by ionization of inner shell electrons. The inner shell electron is ejected and an outer shell electron replaces it. The energy difference can be released as an X-ray
- E_{crit} is the binding energy of the electron and is higher than the emitted X-ray energy

For example:
Cu K α line is 8.0 keV,
 $E_{\text{crit}} = 9\text{keV}$



Bohr Model of the atom (a simplified view) --- where X-rays come from



Real life spectra are more complex because there are multiple orbitals (esp. for the L, M and N orbitals). L-series spectra in EDS can have 6 or 7 peaks. Also, *two elements can have X-rays lines of the same energy (overlap)*, but not all of the lines in both cases will be the same

Moseley's Law



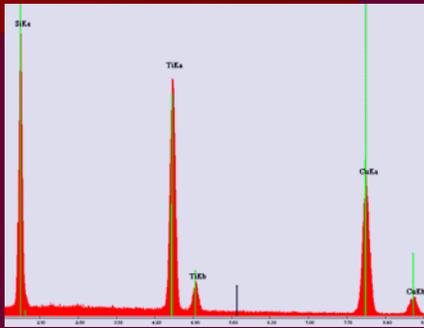
Moseley in his laboratory in 1911.
Killed Dardanelles campaign 1918.

- Moseley showed that the energies of the characteristic X-ray lines are unique to the atom which emits them.

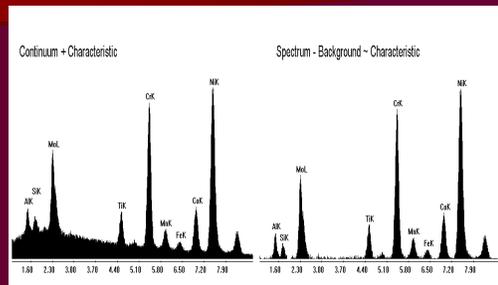
$$E_Z = k_j(Z-1)^2$$

- So if a line is detected that element must be present
- Moseley's Law is the basis of microanalysis using beams of electrons, photon, or nuclear particles

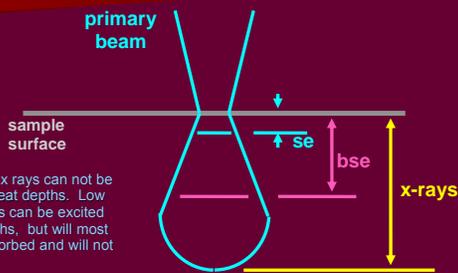
K-series Peaks – variation with energy



Background Artifacts



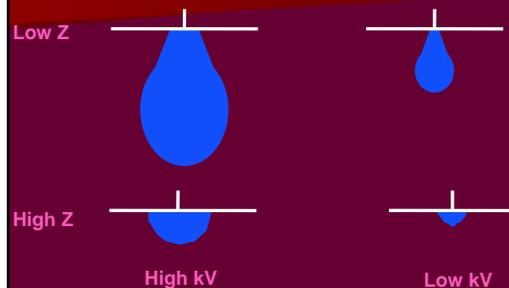
Interaction Volume Regions



High energy x rays can not be excited at great depths. Low energy x rays can be excited at great depths, but will most likely be absorbed and will not escape.

This diagram is somewhat misleading. High-energy and low-energy x rays behave very differently (just like e-).

X-Ray Spatial Resolution

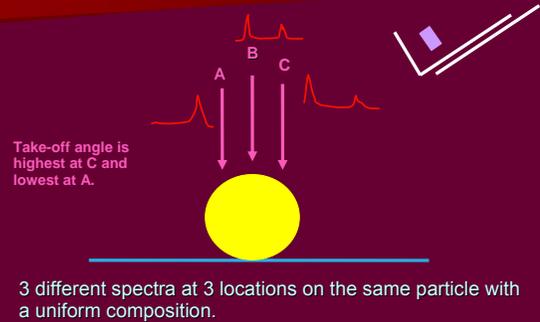


Spot size does not determine the resolution but kV and Z are more significant.

Directionality of Signals

- **SE Signal** - attracted to positive voltage on wire mesh network in front of detector.
- **BSE Signal** - Detector is arranged to collect signals from a large, symmetrical area.
- **X-ray Signal** - most directional of all signals, only one detector with no way to influence the trajectory of x-rays

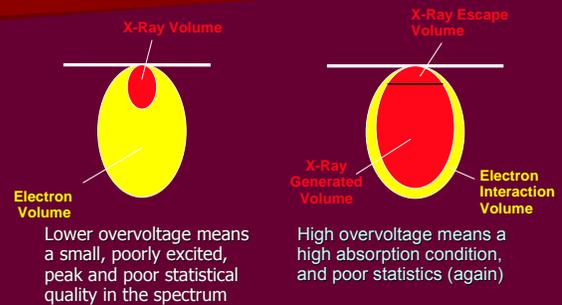
Topography and Take-off Angle Effects



Accelerating Voltage

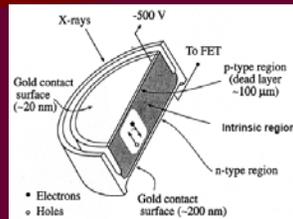
- Overvoltage = Voltage/Peak Energy
- Typically, the accelerating voltage should be 2x the highest energy line and no more than 10 to 20 times the lowest energy line of interest.

Why does the overvoltage matter?



EDS Instrumentation -

- Energy Dispersive Spectrometers measure photon energy, E. They were first developed in the 1940s for Nuclear Spectroscopy and first used in an SEM in 1968. They are fast, convenient, and give good sensitivity but have limitations in their energy resolution
- The detector is a diode which converts photons to electron-hole pairs. Usually the diode is a Si P-I-N device, but Ge diodes are also used
- The X-ray photon produces electron-hole pairs in the intrinsic region. The charge generated is proportional to the X-ray energy
- The conversion of an X-ray to a charge occurs very quickly so many X-rays can be measured each second



Schematic of a typical Si P-I-N detector

EDS Instrumentation & Signal Detection

- X-Ray Detectors
- The Detector Efficiency
- Geometrical Efficiency
- Signal Processing
- The Signal Processor
- Energy Resolution
- Collimation

References:

Note: This presentation is an amalgamation of course presented by the following:

Dr. David Joy, Hitachi SEM training course

Dr. Bob Anderhalt and Lara Swenson,
EDAX/Ametek EDS training course