
Scanning Electron Microscopy and Energy Dispersive X-ray Analysis

Basic On-line Short Course

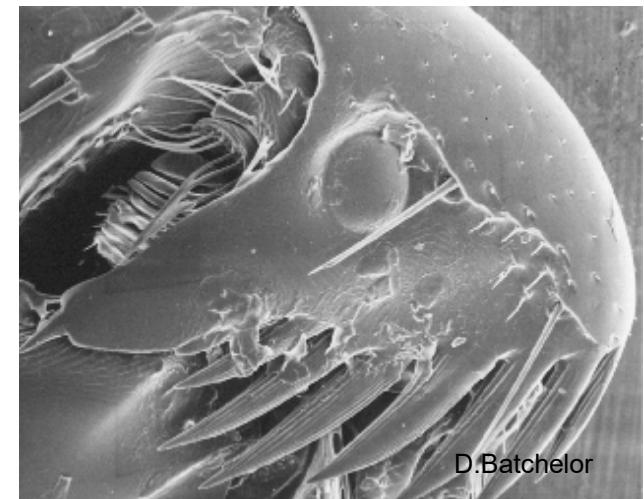
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Outline

- Goal for the course
- General comments on instrumentation
- What is an SEM and why do we care
 - More to microscopy than photons...
 - Scanning vs. traditional microscope techniques
- Electron Optics
- General Introduction to SEM operation
- Electron beam-sample interactions, signals and detectors
 - Elastic Scattering → Backscattered Electrons
 - Inelastic Scattering → Secondary Electrons and X-rays

Virtual SEM Lab

- Beam parameters and how to choose them
 - Practical SEM rules
- Magnification and Resolution
- Scan rates, contrast, and counting
- Charging and coating
- Beam damage, contamination, and artifacts
- How to make good SEM images



SEM micrograph of a cat flea @ 350X
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Goal for the one day SEM short course

- To obtain a basic understanding of SEM in general
 - What is an Scanning Electron Microscope
 - How do we make an electron beam
 - How do we control the beam
 - What happens when the beam strikes a sample
 - How do we form an image from the result of the beam striking the sample
 - What is a signal detector and how do detectors work
 - What are SEM parameters and what they mean
- To learn the basics of SEM operation (hands-on course)
 - How to mount and load a sample
 - How to set up the SEM for data collection, i.e., how to choose operating parameters
 - How to collect high quality images, i.e., how to focus, correct astigmatism, set the signal gain and offset (contrast and brightness), choose dwell time, and pixel resolution
 - If you understand the basics of SEM operation, then learning any new SEM is easy!
- It is assumed that you know nothing about SEM
 - It is assumed that you passed freshman physics...

AIF Overview www.aif.ncsu.edu

- Originally founded as part of Engineering Research Services in 1923
 - The longest continuously operating research facility in the UNC system
 - Other two parts of ERS are/were a soil testing lab in Asheville and the NCSU machine shop (in Burlington labs)
- Center for materials characterization and analysis specializing in microscopy and micro/nano analysis
- AIF provides a central resource available to all with cost shared by many
 - Specialized staff for assistance with instrument operation, experimental design and data interpretation
 - Shared access to capital intensive analytical instruments
 - Individual training for instrument operation and data interpretation
- AIF operational support from use fees, research projects and COE

Analytical Capabilities

- Scanning Electron Microscopy (SEM)
- Transmission Electron Microscopy (TEM)
- Scanned Probe Microscopy (SPM)
- Dual Beam Focused Ion Beam System (FIB)
- Time of Flight Secondary Ion Mass Spectrometry (TOF-SIMS)
- X-Ray Photoelectron Spectroscopy (XPS or ESCA)
- X-ray Diffraction (XRD)
- CT or X-ray Tomography (image the inside of a sample!)
- Metallographic SEM, TEM, and SIMS sample preparation
- Bio-sample prep and imaging
- Optical Microscopy
- Microtome
- Oxygen plasma etching

Note on Instrumentation

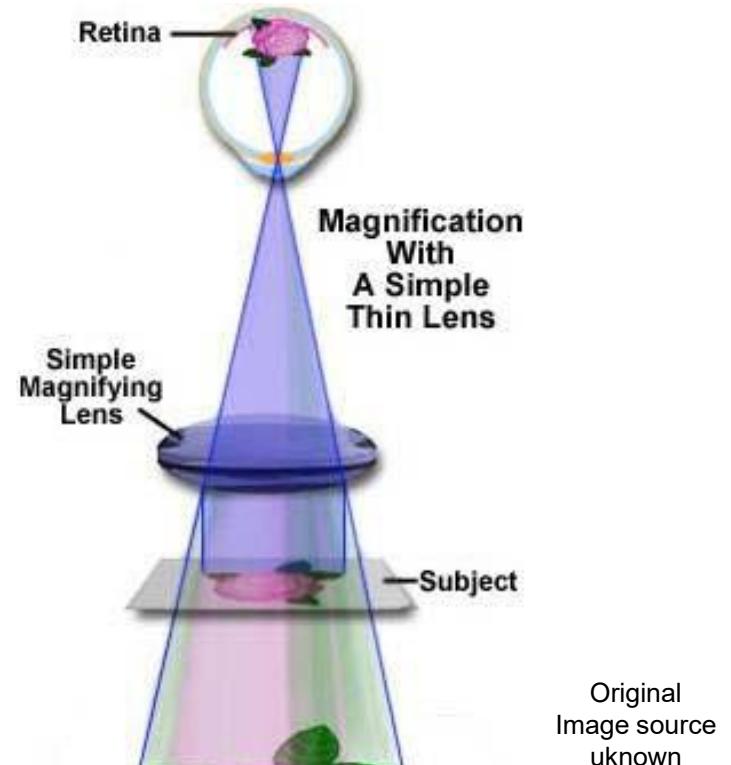
- Tools only do one thing
 - If all you have is a hammer, everything looks like a nail...
- Instruments have adjustable parameters and can be configured to do different things
- Instruments require tuning to achieve the desired results
 - Whether scientific or musical!
 - Tools do not typically have adjustable parameters!
- Understanding how an instrument works and interacts with a sample is critical to understanding and correctly interpreting the results – true for *any* scientific instrument
- All of the operating parameters affect the collected data!
- Understanding how to choose an instrument's parameters is the difference between a instrumentalist and someone who uses a tool

Why Use Electrons for Microscopy?

- Microscope resolution (far field) is diffraction limited
 - Limit is about $\frac{1}{2}$ of the wavelength of the incident radiation
 - Far field means that the optics and sample are separated by more than the distance of a single wavelength
- Photons have a longer wavelength than electrons
 - Average wavelength of visible light $\sim 550\text{nm}$ (green)
 - Visible light microscopes limited to $\sim 250\text{nm}$ resolution
 - Can be improved using scanning (confocal and near field) and fluorescence techniques, but these are whole topics...
 - Note: many current technologies use sub-100nm structures...
 - Examples: Semiconductor devices, Ag nanoparticles used as anti-microbial agent on textiles, advanced Li-ion batteries, etc.
- Wavelength of electrons typically used in electron microscopy $<10\text{pm}$
 - Diffraction limit of EM resolution $<5\text{pm}$ (10^5 better than visible photons!)
 - The diameter of a C atom in a graphite lattice is 170pm...
 - Actual resolution limits are generally defined by the initial spot size, lenses, lens aberrations (limit $\sim 0.5\text{nm}$ for high end SEMs)

Traditional Microscopy

- Traditional optical microscopy **projects** an image onto a detector (e.g., an observer's eye or camera or imaging screen) using a lens set
 - Recall thin lens optics from Freshman Physics
 - With the advent of inexpensive electronics, digital cameras have commonly replaced the observer's eye and the image is observed on a monitor, usually via a computer interface
- The **entire** image is **projected** simultaneously
 - Almost all photon optical systems work in this manner
 - Eyes
 - Photon Optical Microscope
 - Binoculars/Telescope
 - Electrons can be used for traditional microscopy too! (TEM)



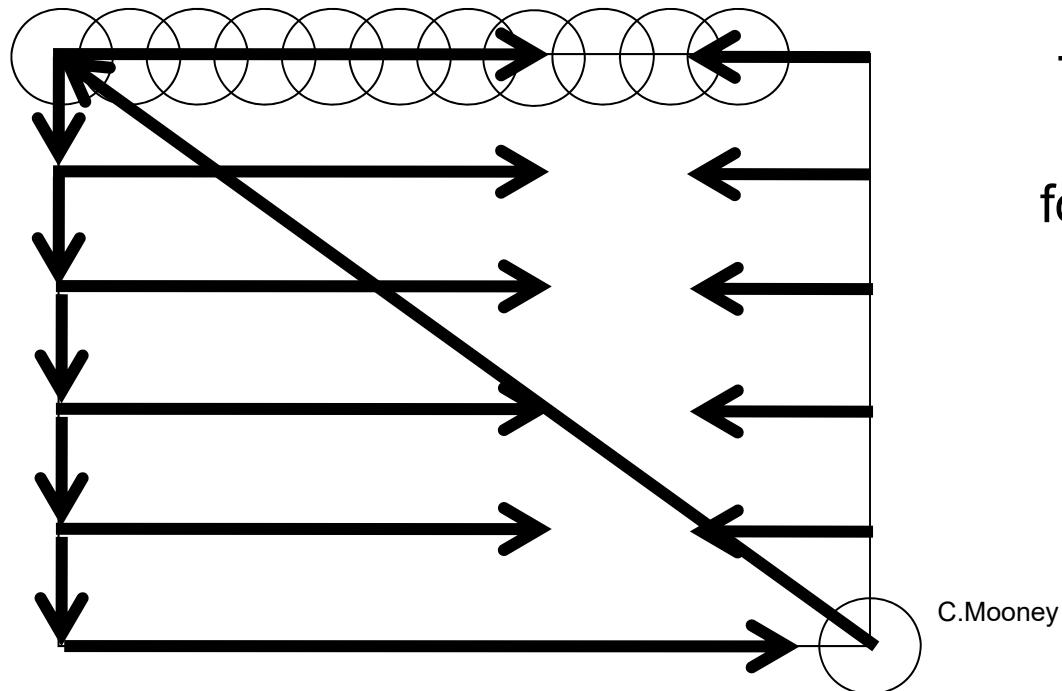
Original
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Projected image formed by lenses in optical microscope

Scanning Microscopy

- Scanning microscopes **do not** project an image on to a detector
- The image is formed by scanning a probe over the sample **one pixel at a time**
 - A pixel is a picture element in a digital array
- Scanned systems are common:
 - TV, whether analog or digital
 - Fax
- Possible to perform scanning fast enough to give the appearance of a projected image
 - Television is a good example of a scanning system that operates faster than human eyes work

The Scanning in Scanning Microscopy



Typical digital raster pattern for image formation in scanning microscopy shown schematically

In the SEM an electron beam is scanned across the sample

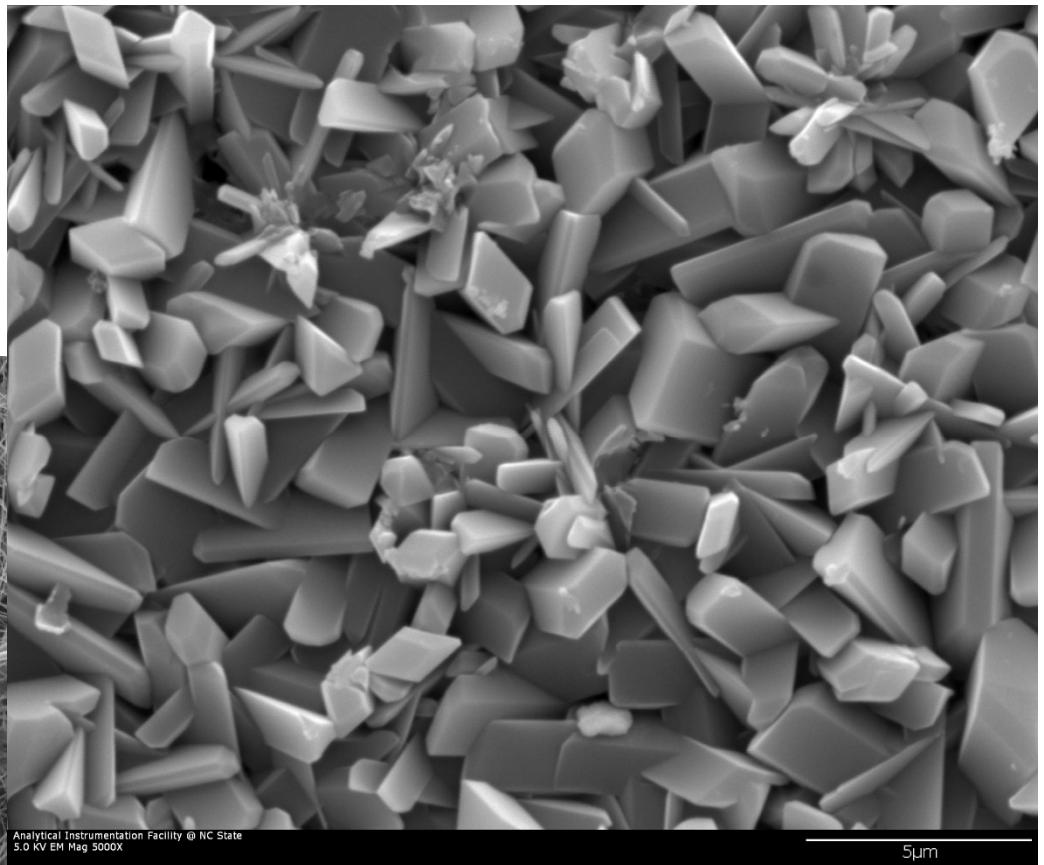
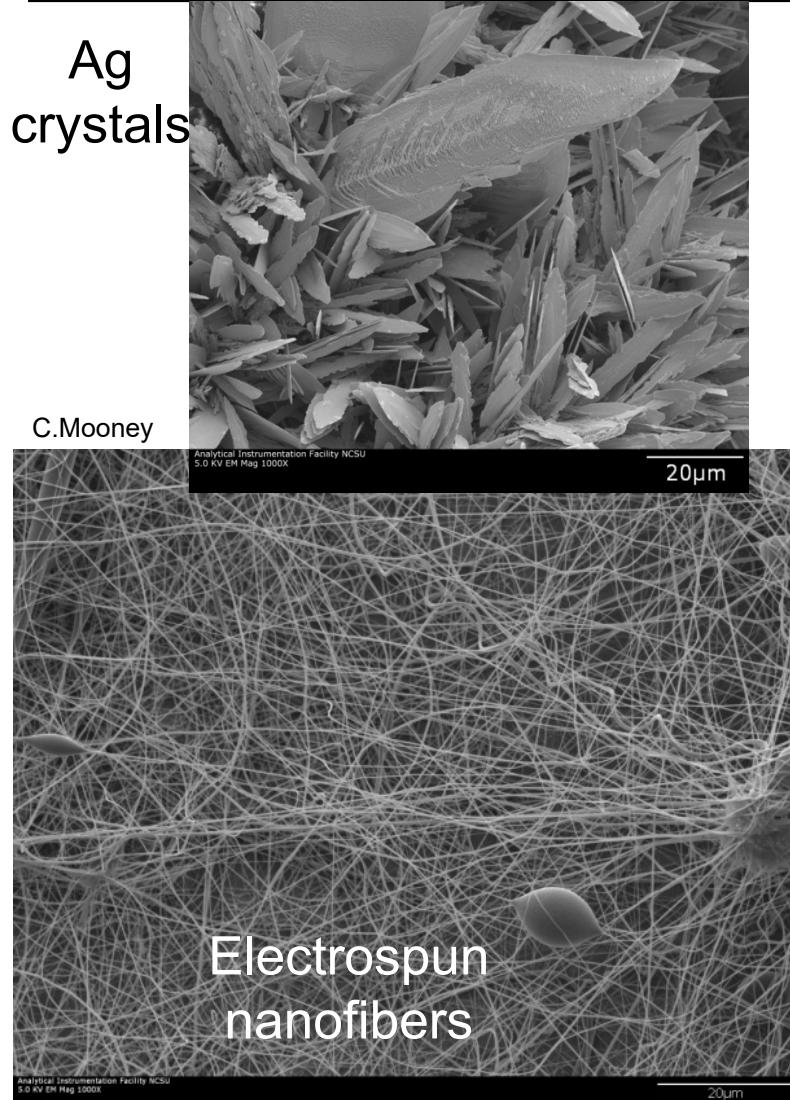
- Probe is first placed at point X₁Y₁
- Data is collected with a detector (at X₁Y₁)
- Data is displayed at point X₁Y₁ on monitor
- Repeat to point X_nY_m to build an image

General Definition of Scanning Microscopy

Scanning a probe over a sample and mapping probe position vs. sample-probe interaction where the displayed image is larger than the scanned region.

- Scanning Electron Microscopy scans an electron probe over a small region of a sample
- Atomic Force Microscopy (AFM) scans a small physical probe
- Focused Ion Beam (FIB) scans an ion probe

A few example SEM images...



Manganese Phosphate protective coating on steel.

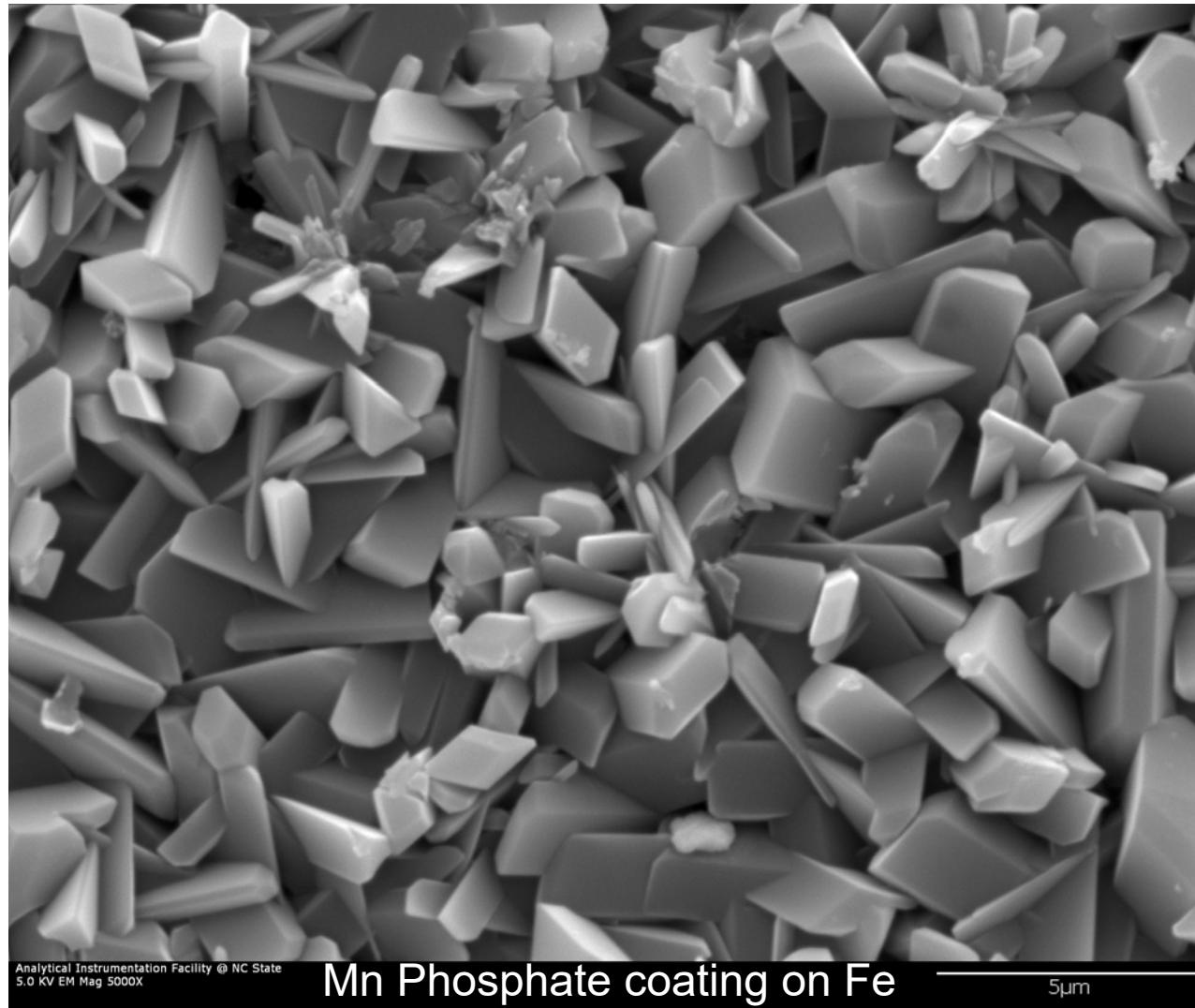
Scanning Microscopy Characteristics

- Optics do not form the image!
- Optics form the probe (SEM = electron beam)!
 - Optics = source, lenses, and apertures
 - Probe characteristics are determined by the optics parameters
 - Electron probe characteristics include beam energy, current, spot size, and convergence angle
 - The probe is aka the beam or the spot (terms used interchangably)
- Magnification is a function of how far the beam is moved in the raster pattern not optics!
 - Magnification is a function of scan size
 - Reduce scan size => increase magnification
 - Resolution is a function of optics parameters
 - Smaller probe = higher (potential) resolution
 - Note that magnification and resolution are independent!

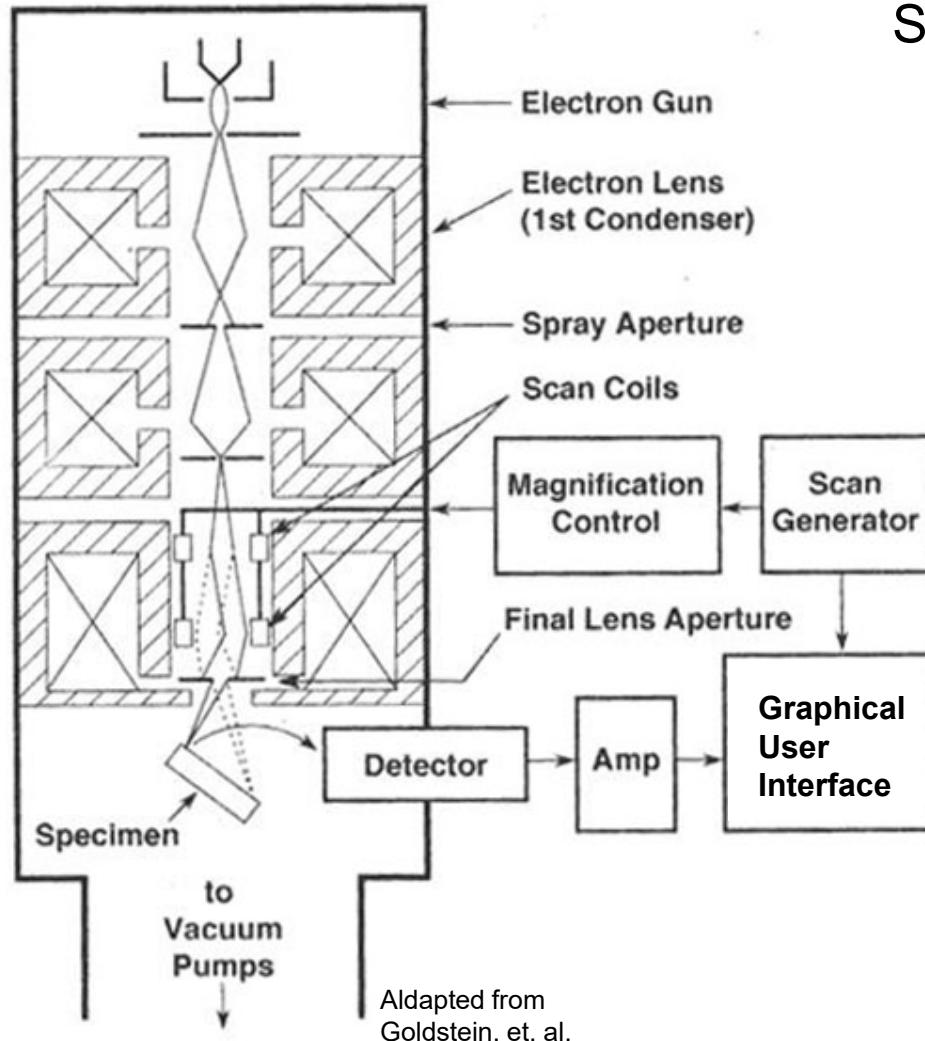
SEM in General

- Scan a small, round electron probe over the sample
 - The smaller the probe the better the potential resolution
 - Optics form the probe
 - The probe is also known as the spot or the beam
 - Those terms may be used interchangeably
- At each point in the scan, collect data with a detector
 - Multiple kinds of imaging data, the most common are secondary electrons
 - Unless the signal is cathodoluminescence, there is no color!
 - There may be energy information about the emitted electrons
- Display the data such that a map of probe position vs. detector output is shown
 - If the displayed map is larger than the scanned area, the system is a microscope!

SEM Example Image



SEM Schematic

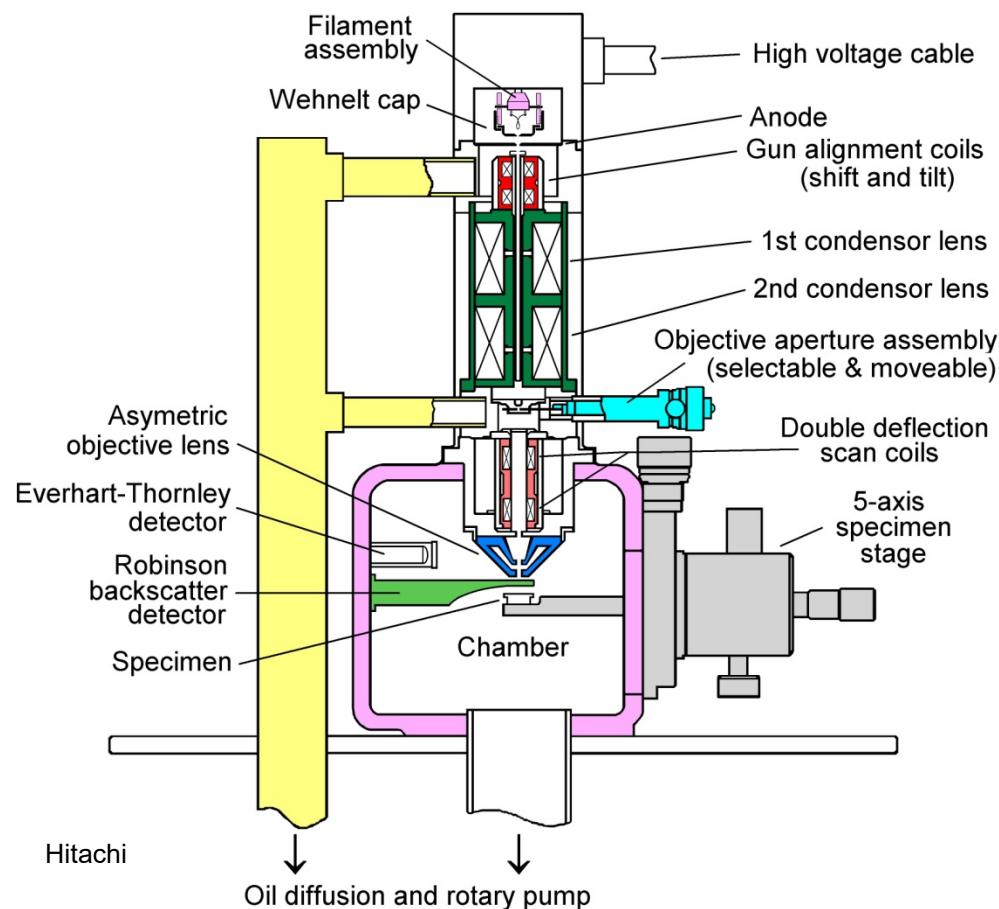


SEM Parts:

- Electron gun
 - Source of electrons
- Condenser lens(es)
 - Spot size/current control
- Objective lens
 - Focus control
- Objective aperture
 - Helps control depth of field
- Scanning coils
 - Magnification control
- Stigmator coils
- Scan generator
- Detector(s)
- Graphical User Interface (GUI)
 - Typically a computer control interface
 - Displays images and other data

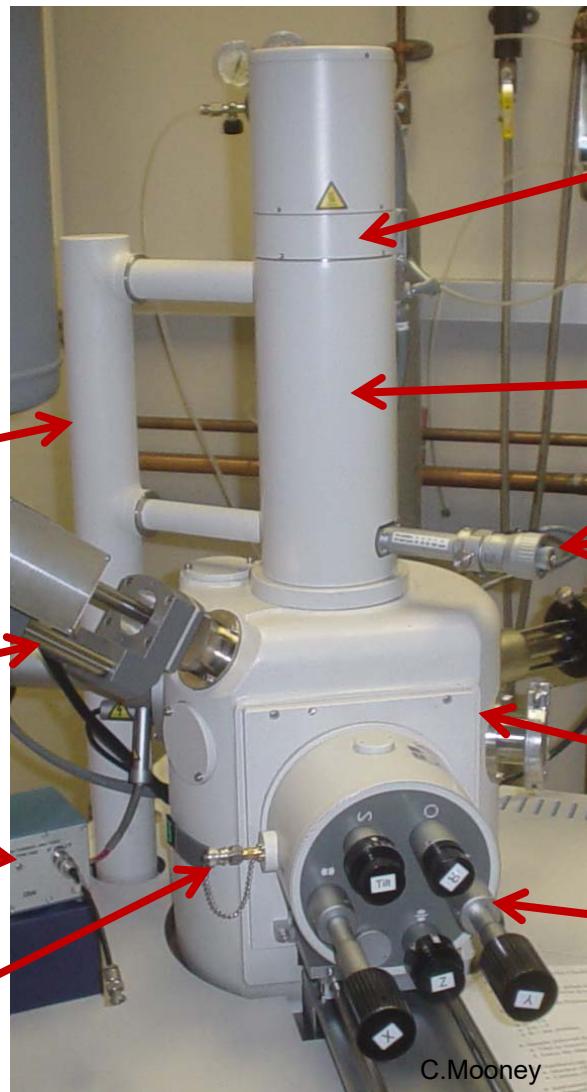
Typical SEM Diagram and Photo

Diagram and photo of the AIF Hitachi S-3200N SEM



SEM Labeled Photo

Photo showing the location of principle components of the AIF Hitachi S-3200N SEM



Vacuum pipe

X-ray detector

Current Amplifier

Stage Electrical
Connection

Electron Gun

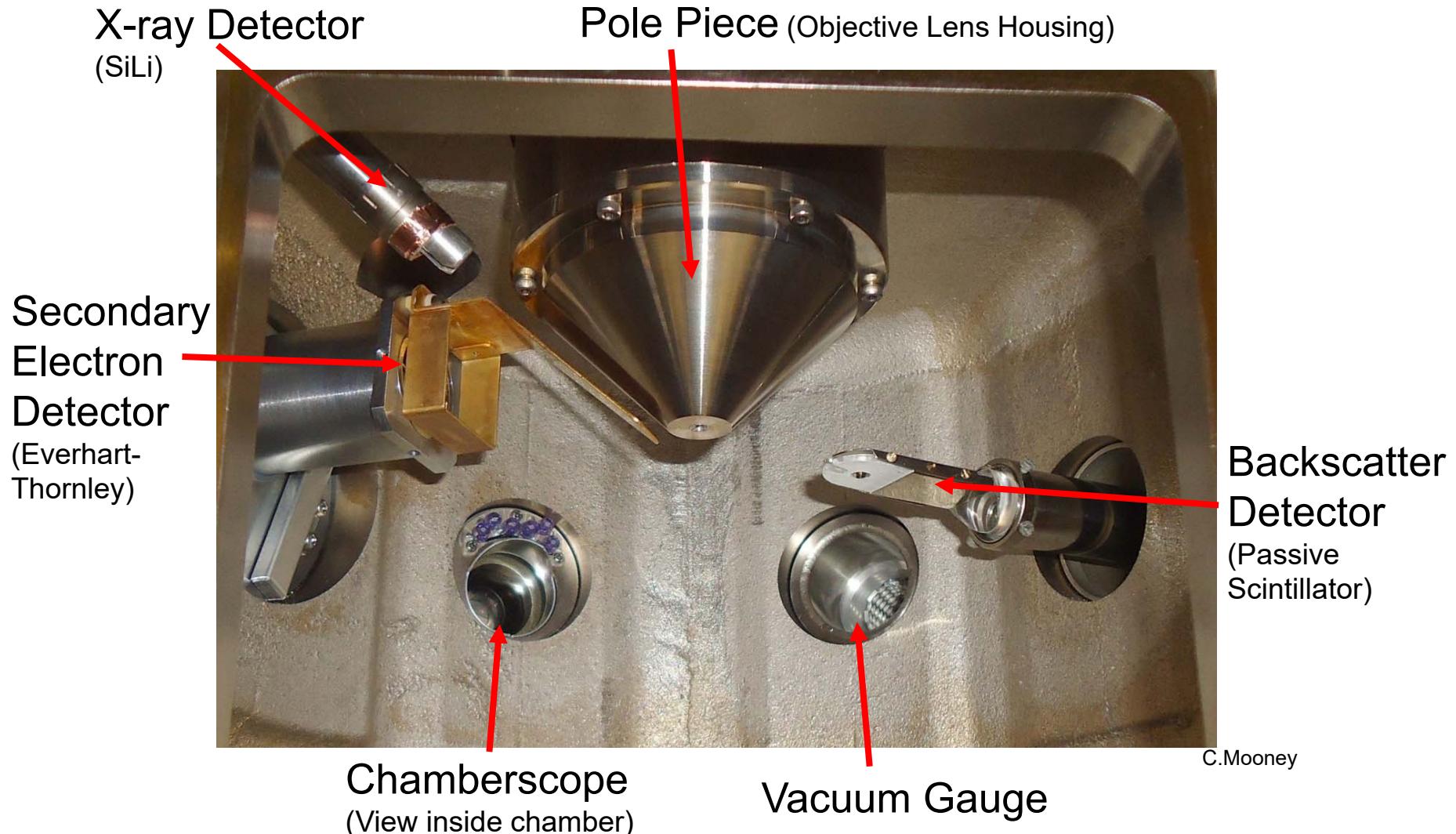
Condenser Lens

Objective Aperture
Holder

Main Chamber

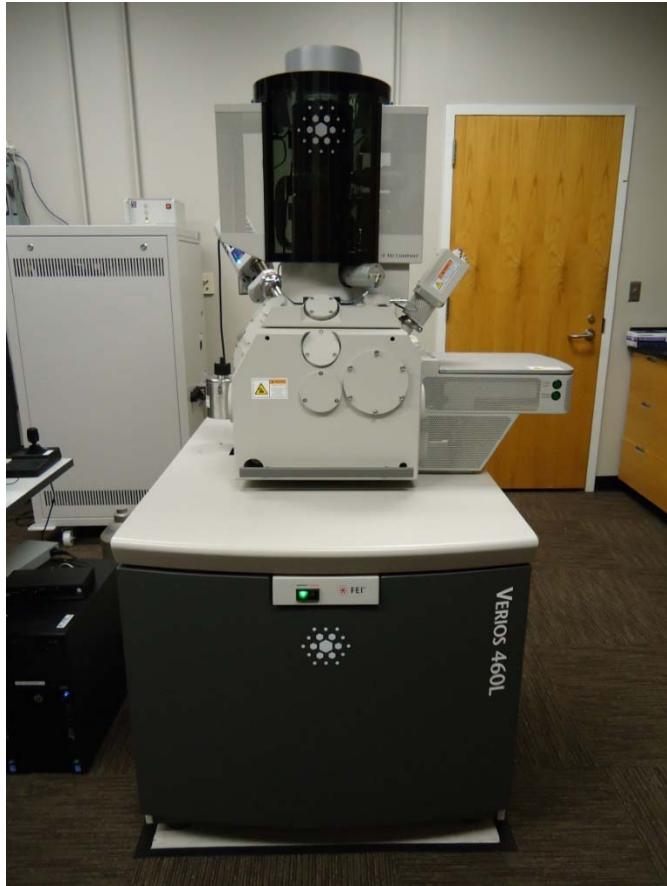
Stage Controls

SEM Inside the Main Chamber



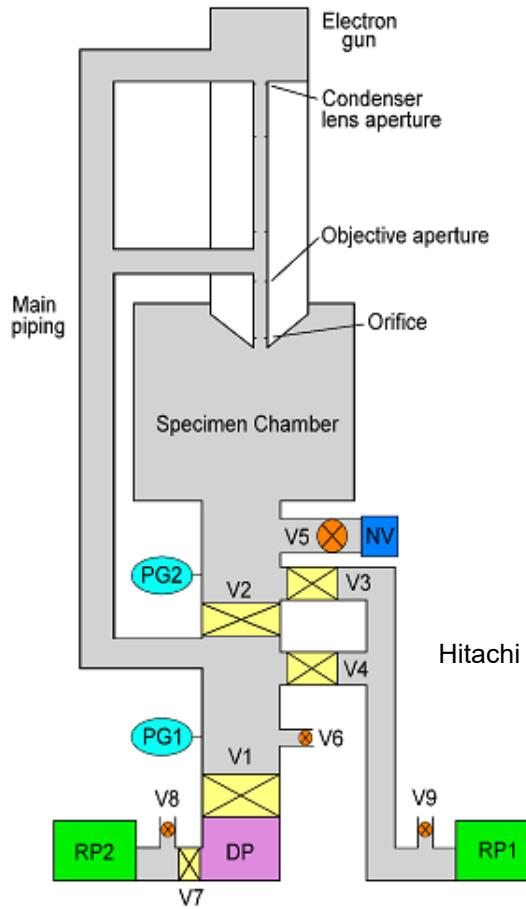
FEI Verios 460L

- Photo of Verios unconventional SEM



Vacuum Requirements for SEM

- Why do we need vacuum?
 1. Electron Mean free path
 2. Source lifetime
- Electron Mean free path
 - The mean free path of a particle is the distance that the particle can travel in a medium without a scattering event
 - Mean free path of an electron in air is approximately 100nm
 - Mean free path of an electron in a 10^{-4} torr vacuum is approximately 1m!
- Electron source
 - W filament functions well at 10^{-4} torr
 - Field emission requires 10^{-10} torr
- What is a Torr? (Not an SI unit)
 - Convenient way to express vacuum, not an SI unit
 - Named in honor of Evangelista Torricelli inventor of the barometer
 - Originally 1 atm = 760 mm Hg = 760 Torr at 0C and sea level
 - Now defined: 1 atm = 101.325 Pa
→ 1 Torr = 133.3 Pa



Vacuum schematic for
S-3200N SEM

Electron Optics

- Electrons optics consists of the parts required to form an electron beam
 - We ultimately want a small round probe of electrons on the sample
- Electrons optics generally consist of the following:
 - Electron gun (it shoots electrons down the column!)
 - Condenser Lens (set)
 - Objective Lens
 - Objective Aperture
 - Astigmatism Correction Coil Set, aka Stigmator Coils

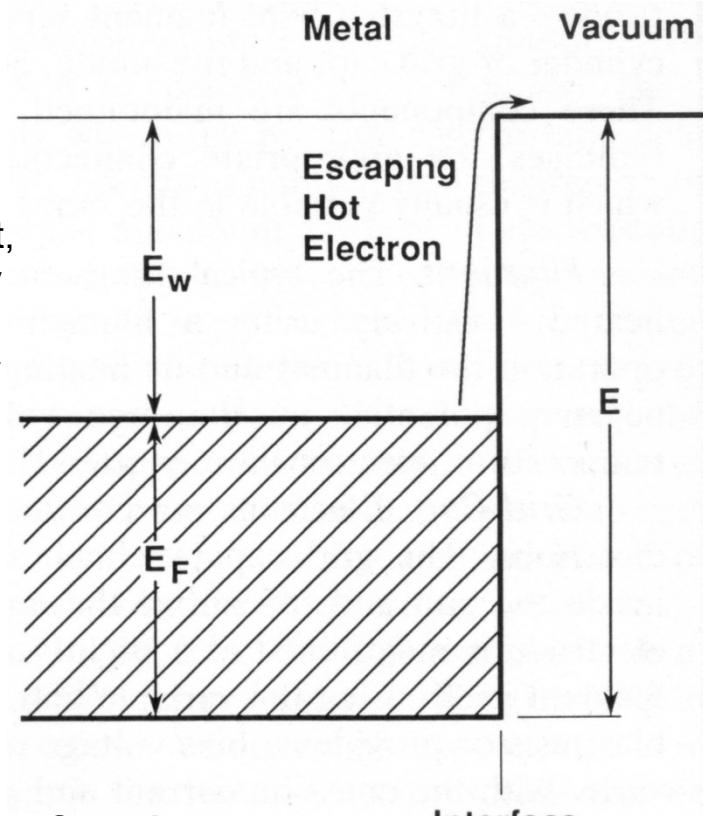
Electron Gun types

- Two main types of electron guns
 - Thermionic
 - Field Emission
- Why is the electron gun called an electron gun?
 - It shoots (accelerates) electrons down the column!
 - Anything that fires projectiles is a gun
 - Electrons are very, very small projectiles!
- Thermionic electron guns are simple and have a long history
 - Heat a cathode filament (~2700K) until electrons boil off the surface, then accelerate them down the electron optical column
- FE electron guns are capable of very high resolution – high end FESEM resolution is on the order of 0.5nm!
 - An intense electric field (10^9 V/m) is used to allow electrons to quantum mechanically tunnel out of the emitter tip and then accelerate
 - Suspect 0.5 nm is a fundamental resolution limit
- Typical acceleration voltages for SEMs are 0.5 – 30 kV
 - Most electrons in an SEM are accelerated to near relativistic velocities

Thermionic Emission Electron Gun

Thermionic Emission: Use thermal energy to overcome a cathode's work function (E_w) and release electrons.

- Most common electron source in SEMs
- >125 year history ← AKA the Edison effect, originally reported by Guthrie in 1873 and described in 1884 by Edison
- Simple operation
- Low cost
- Minimal Vacuum requirements
 - 10^{-4} Torr
- Heat material until electrons boil off the surface!

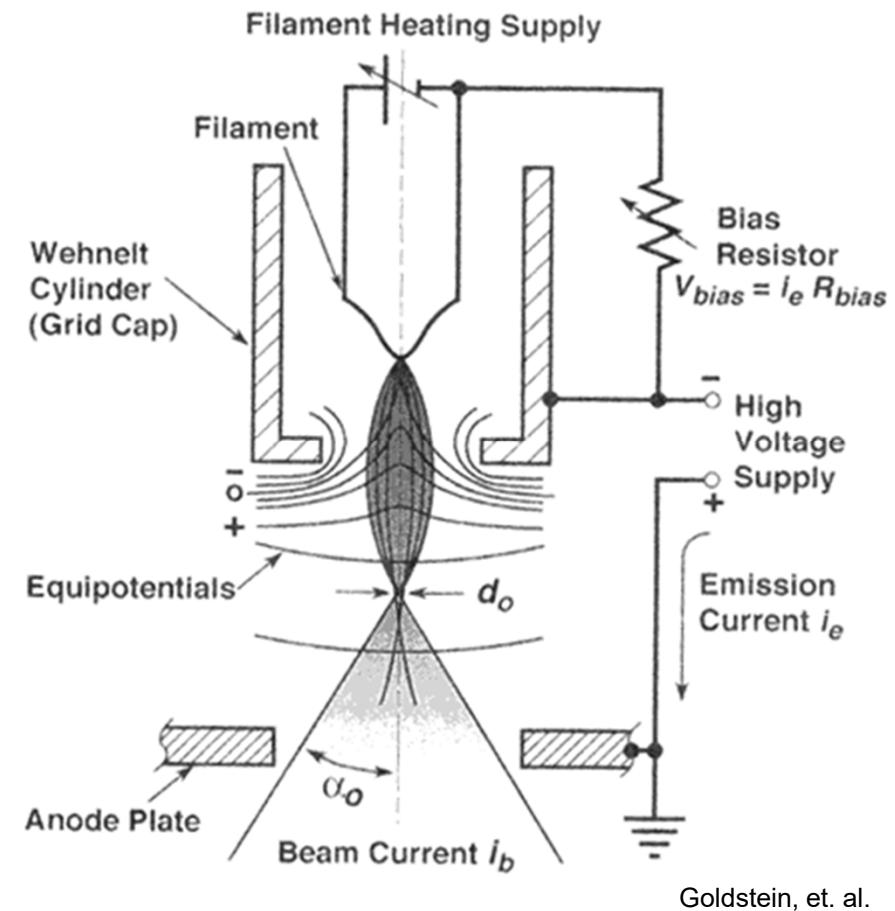


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Thermionic Electron Gun

Components of a Thermionic Electron Gun:

- Filament (plus heating supply)
 - Source of electrons
 - Electrons escape surface via thermal energy
- Wehnelt (plus bias resistor)
 - Simple electrostatic lens
 - Forms first crossover
 - Also inhibits emission from filament not at apex
- Anode (plus HV supply)
 - Accelerates electrons



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Physical Thermionic Electron Gun

Electron Gun

- Filament



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- Wehnelt Cap



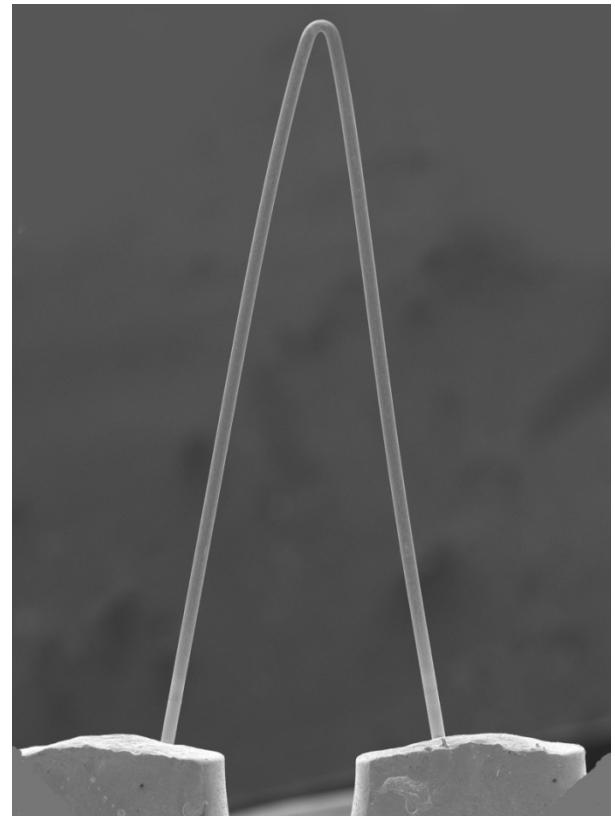
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- Anode (flat, conductive plate with a hole in the center)



Tungsten Filament (Cathode)

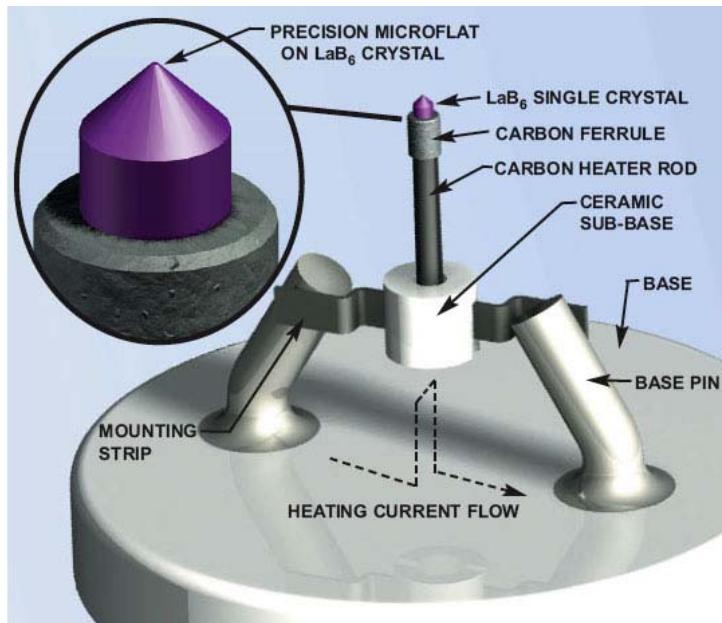
- Typical properties of W cathode:
- ~100 µm diameter W wire
- Directly heated (resistive)
- Emission Area \approx 100x150 µm
 - Not a round, point-like source!
 - To make a microscope, we need the electron probe as small as possible and round!
- Typical Operational Conditions
 - $T(\text{operation}) = 2700\text{K}$



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LaB₆ or CeB₆ Cathode

- LaB₆ or CeB₆ can also be used to make a cathode
- The primary advantage is source lifetime: > 1000 hours
- The primary disadvantage is that higher vacuum is required and the source is not easy to change in the field, which makes the microscope more expensive, so these are not as common

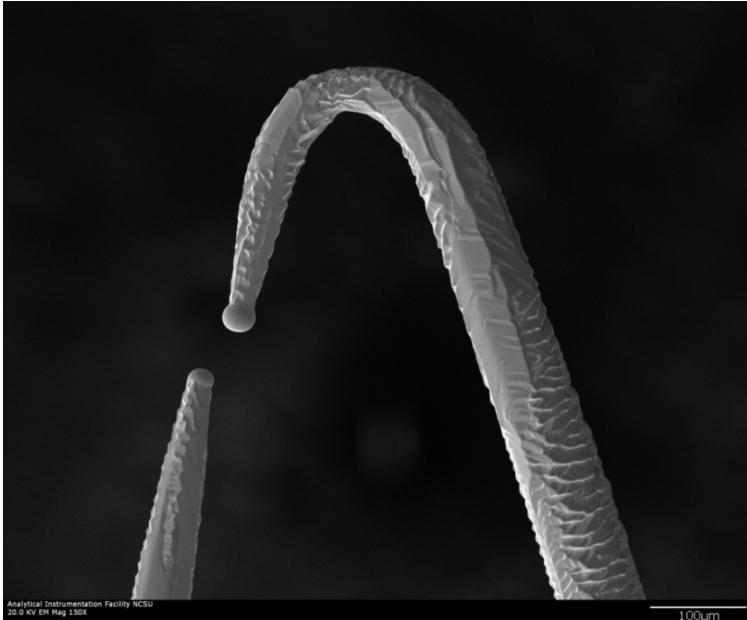


Ted Pella, Inc.



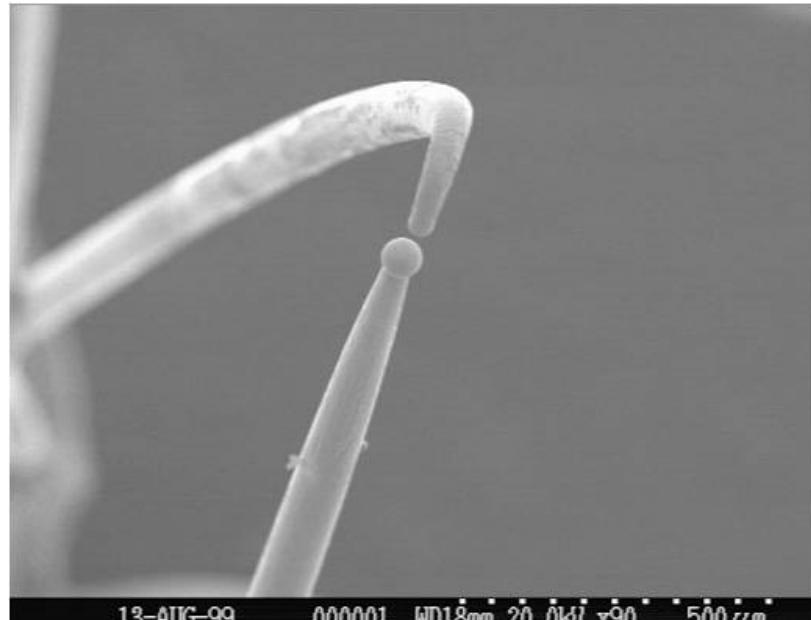
Advanced Ceramic Materials Corporation

Filament Failures



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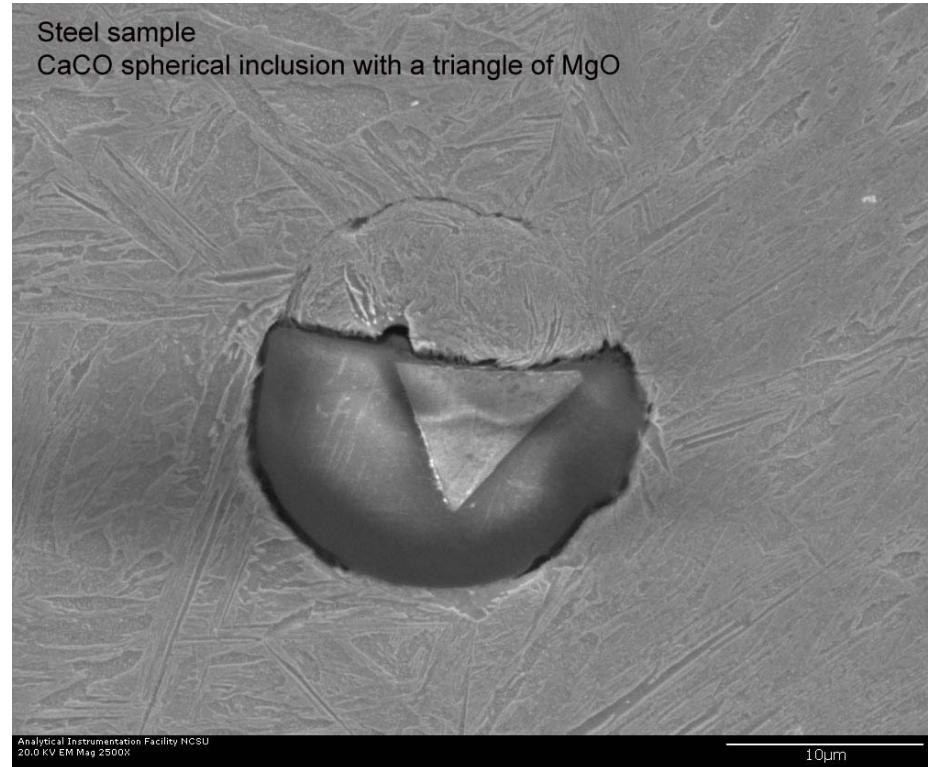
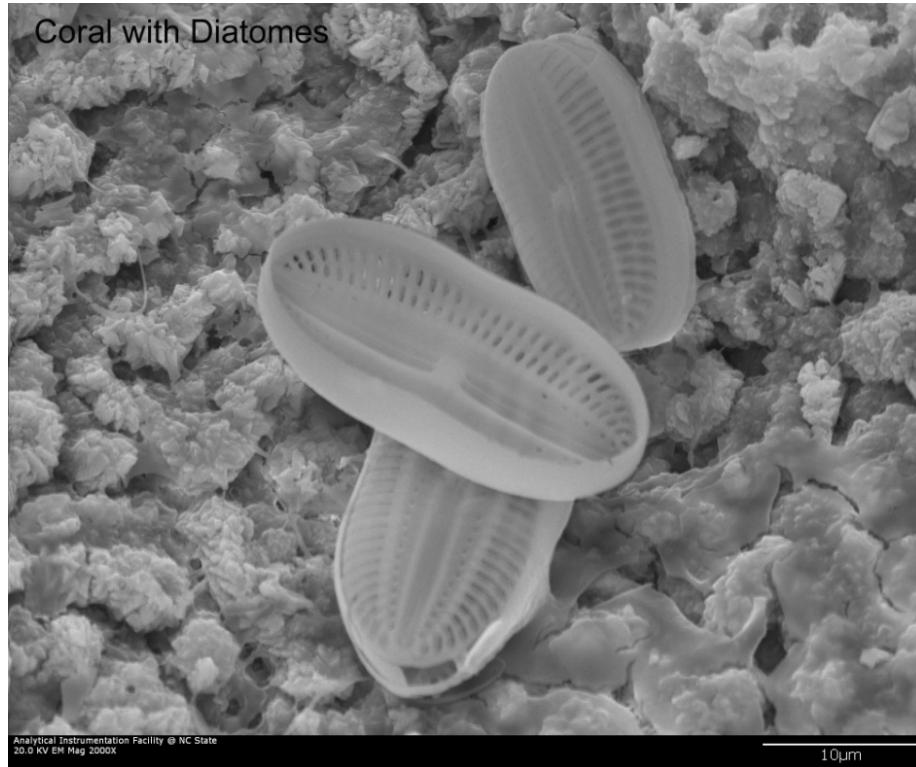
Analytical Instrumentation Facility NCSU
20.0 KV EM Mag 150X



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- Filaments do fail and must be replaced regularly
- A filament that failed due to normal use is shown on the left – note that the material is faceted (typical lifetime ~40-250 hours)
- A filament that failed due to overheating is shown on the right – note that the material is thinned but no faceting is observed
- Note that both failures were hot (clue: little melted regions at the break)

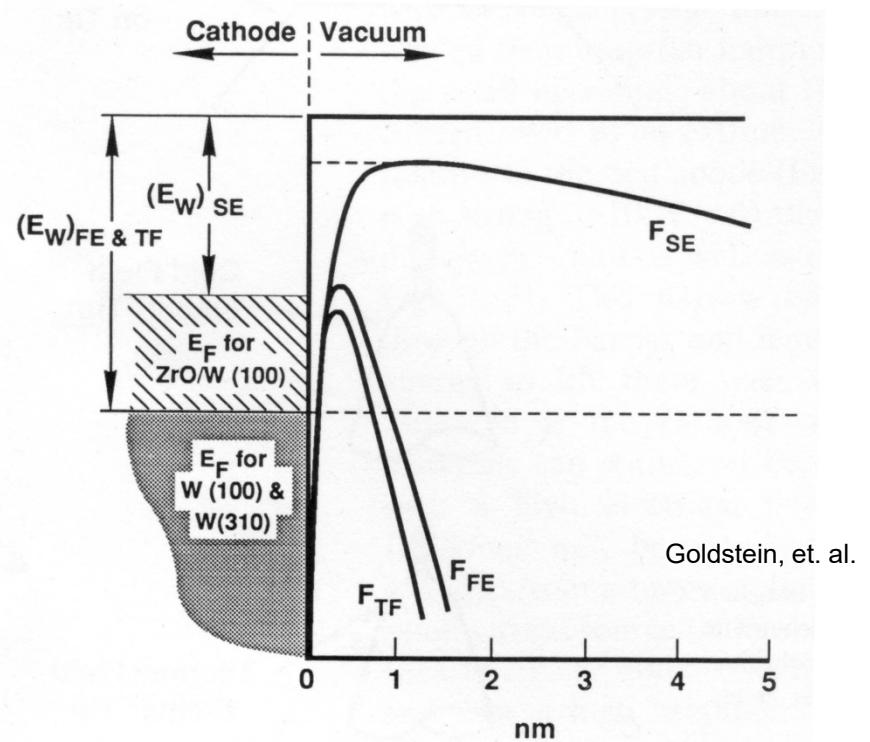
SEM Images from a Thermionic SEM



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Field Emission Electron Source

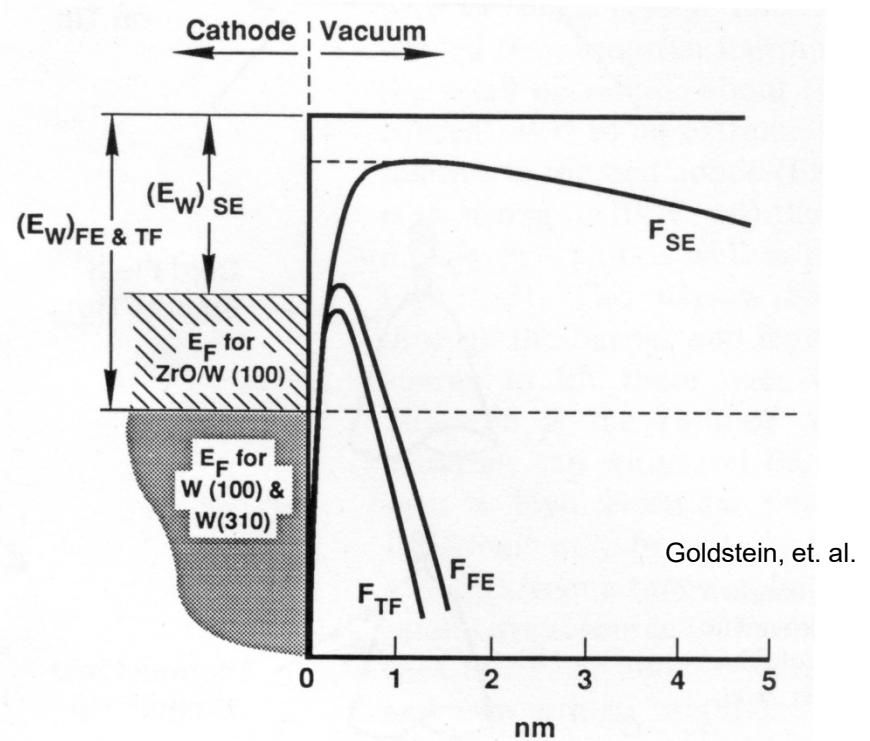
- High electric field is applied to a sharp needle shaped emitter
- The sharp needle concentrates the electric field
- In some cases, the source is also heated to reduce the field requirements
 - Thermal FE
 - Schottky FE
- The field at the tip becomes so strong ($>10^7$ V/cm) that the potential energy barrier is narrowed allowing electrons to tunnel out of the tip material
- Electrons tunnel out of a so-called virtual source that is very small ($\sim 10\text{nm}$)



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Energy diagram for field emission sources. Shown are cold FE (F_{FE}), thermal FE (F_{TF}), and Schottky FE (F_{SE}) emitters

Schottky Emitters

- Schottky emitters are a special case
- Schottky emitter is really a field assisted thermal emitter
 - Look at the energy diagram!
 - SE curve suggests distances that are too great for quantum mechanical tunneling
 - The design of Schottkey electron guns is another clue that these are field assisted thermal emitters: They require a suppressor cap (similar to a Wehnelt) to suppress emission everywhere but the apex of the tip
- Most refer to them as “FE”
 - Everyone knows that field emission SEMs have the best resolution!!!
 - Also, the behavior of a Schottky emitter is more like a FE in that the electrons are emitted from a very small virtual source, hence the improved resolution

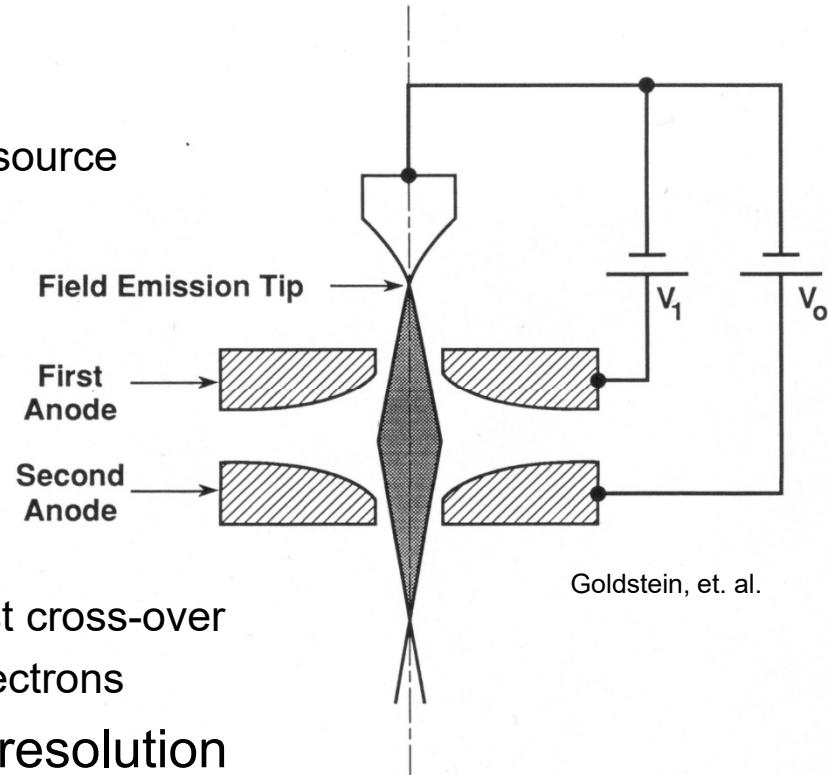


Goldstein, et. al.
Energy diagram for field emission sources. Shown are cold FE (F_{FE}), thermal FE (F_{TF}), and Schottky FE (F_{SE}) emitters

Field Emission Electron Gun

Components of a (cold) Field Emission Electron Gun:

- Emitter
 - Typically W wire etched to a sharp tip
 - Electrons tunnel out from a tiny virtual source
- First Anode
 - Creates intense electric field that allows electrons to quantum mechanically tunnel outside the tip
- Second Anode
 - Works with first anode to create the first cross-over
 - Works with first anode to accelerate electrons
- FESEMs are capable of **very** high resolution
 - Limit appears to be about 0.5 nm
 - The initial source is very small (nm scale), so very small focused electron beams are possible



Goldstein, et. al.

Example Cold FE Emitter

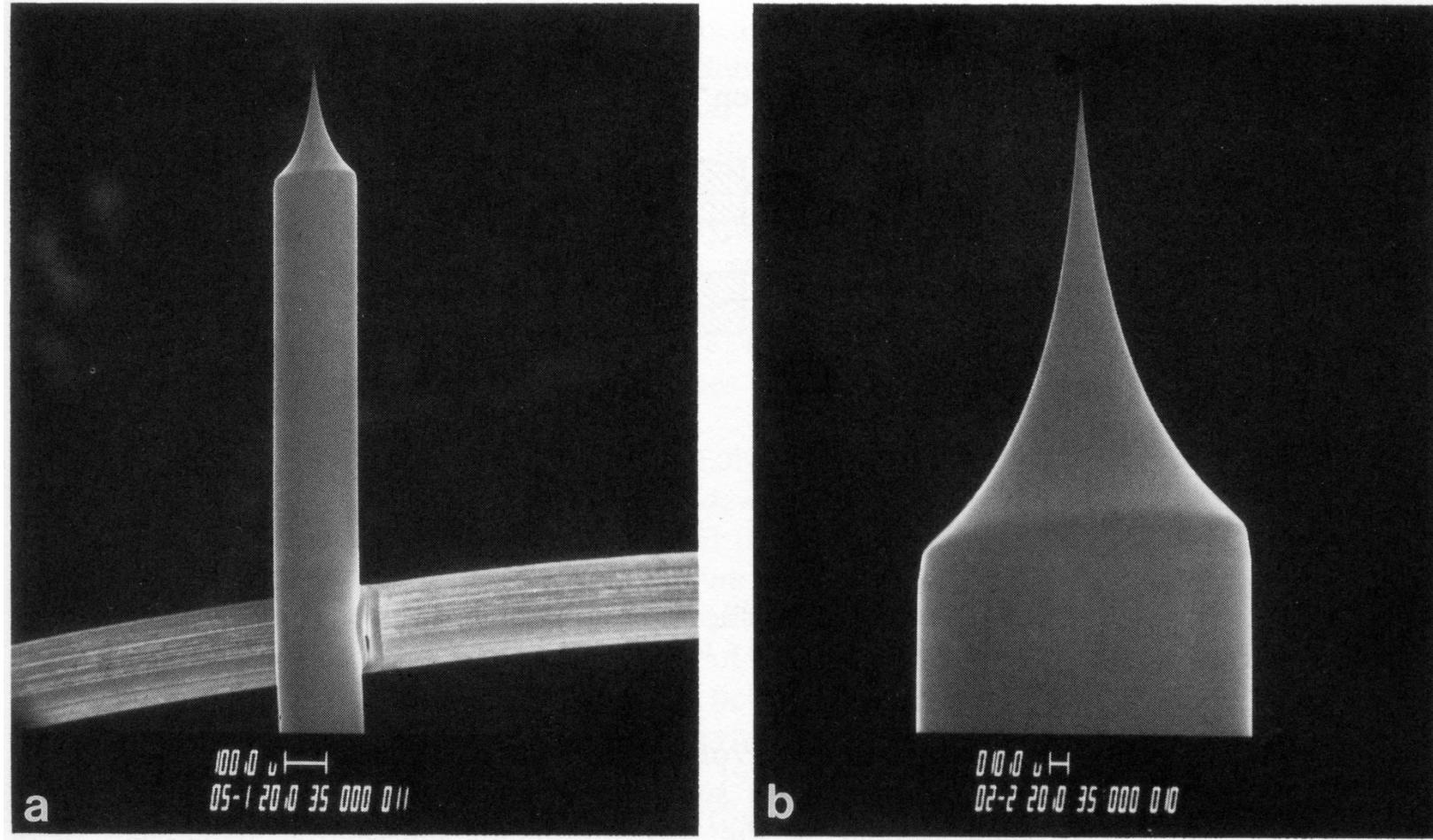
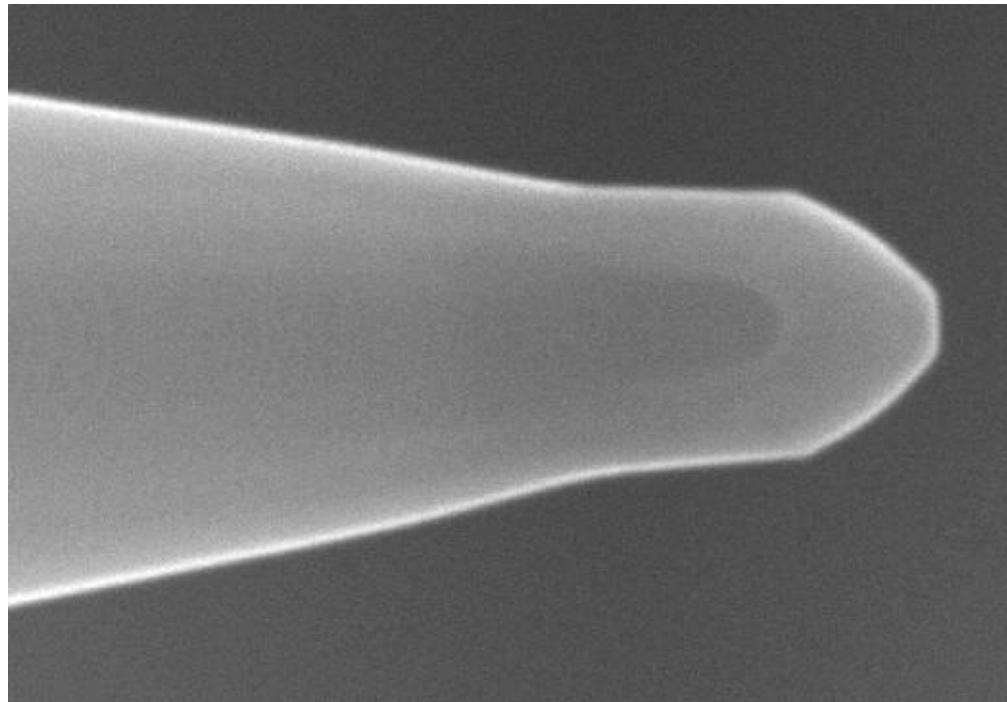


Image source unknown

SEM micrographs of a cold FE emitter tip

Schottky Emitter Tip



Schottky emitter tip has a different morphology from a cold field emitter. The tip is typically fabricated from W with a ZrO well (actually a coating or ZrO).

Schottky emitter with suppressor cap assembly

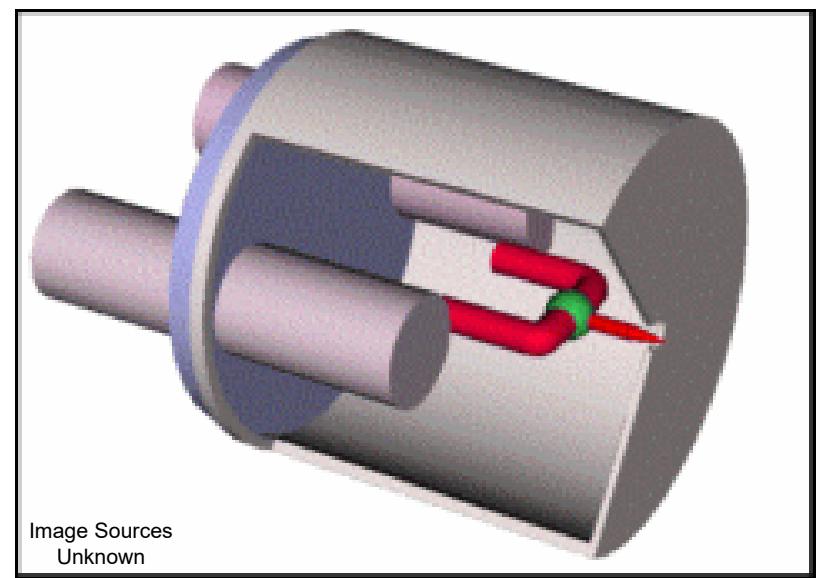


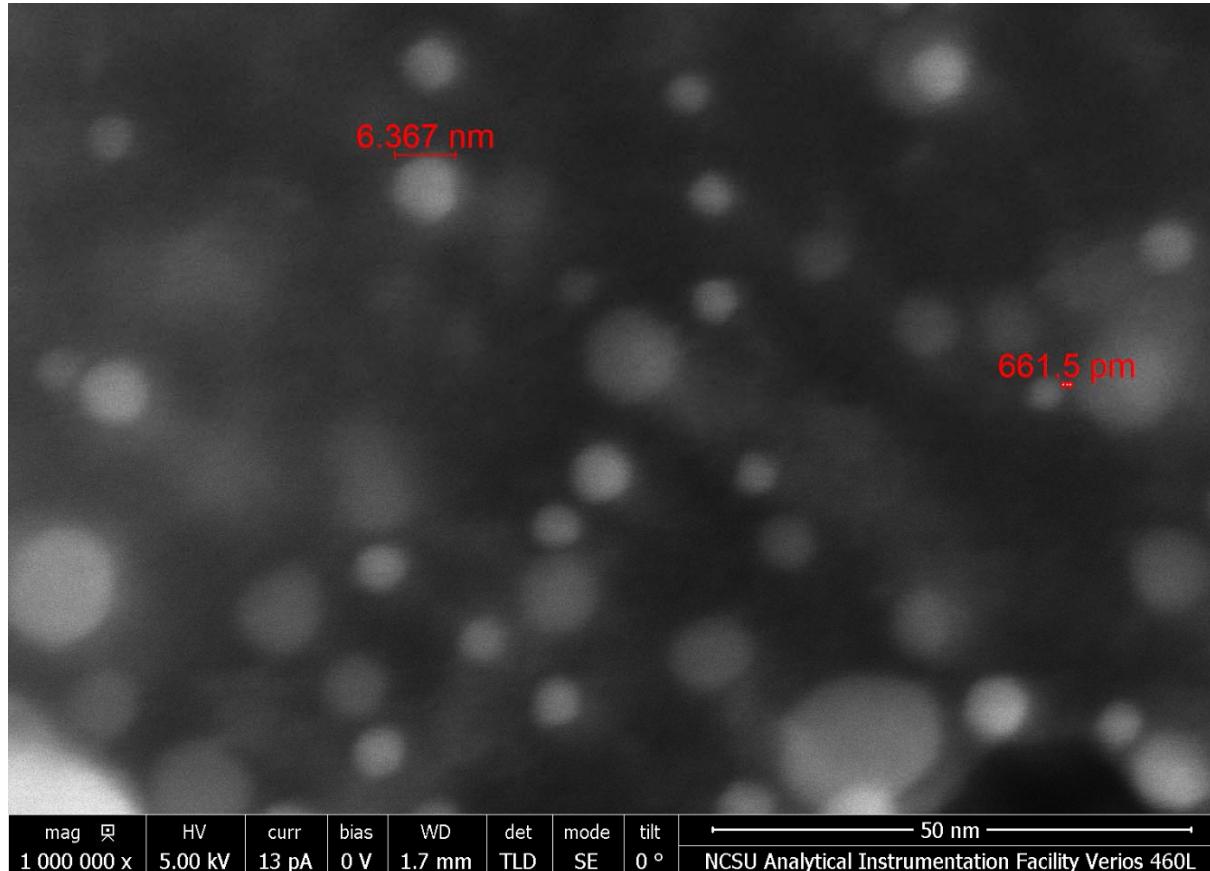
Image Sources
Unknown

The fact that a Schottkey electron gun requires a suppressor cap suggests that this is a field assisted thermal emitter!

Field Emission Electron Source

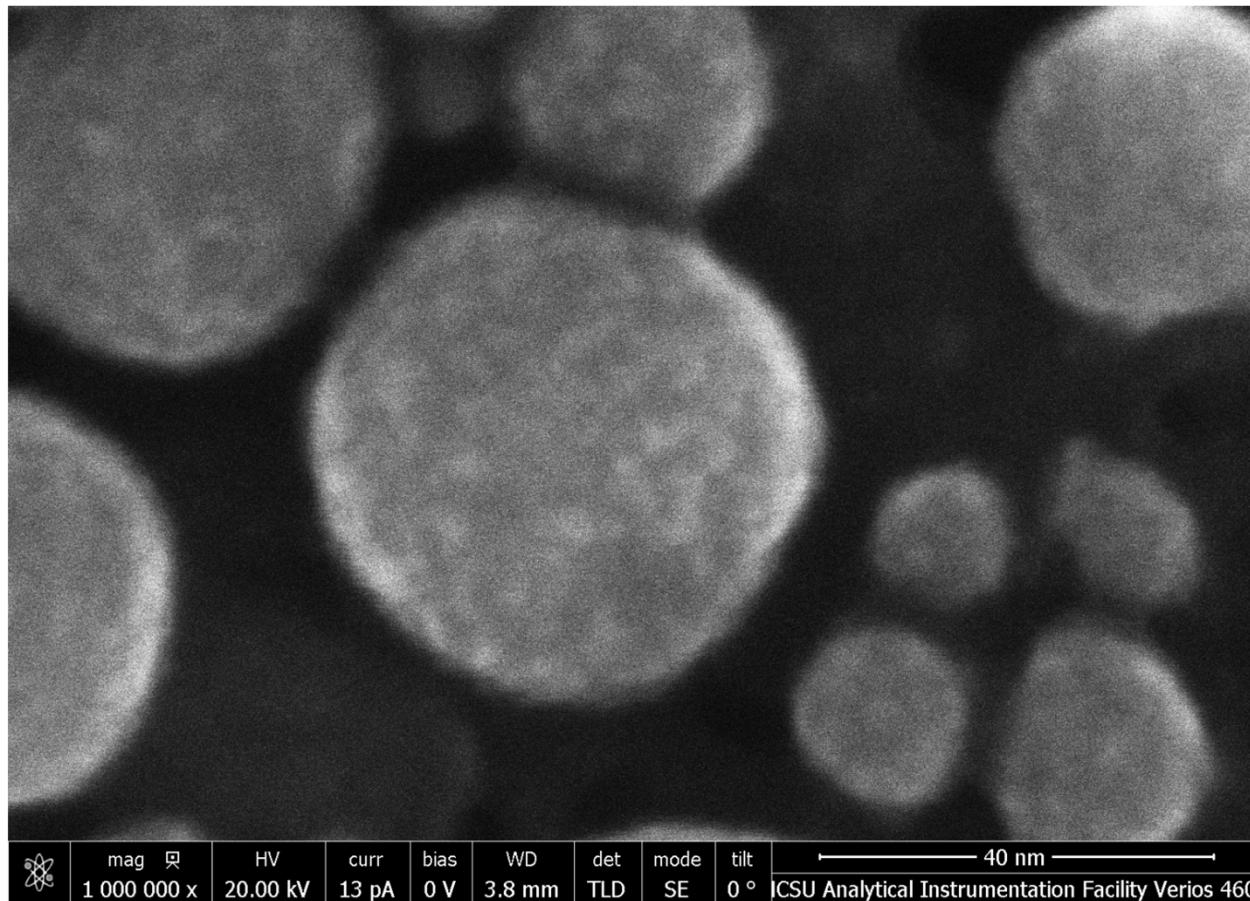
- Field emission is much harder than thermal emission
 - Requires much better vacuum
 - Harder to build a reliable electron gun
 - Geometry has to be very precise
 - Can't change the emitter in the field – usually change the whole electron gun assembly (it's easy to change a thermal emitter)
- Why do we go through the trouble?
 - The electrons in the beam come from a very small, “virtual source” that is on the order of a few nm for cold FE and <50 nm for Schottkey emitters (recall thermal source size $\sim 100 \times 150 \text{ }\mu\text{m}!$)
 - This makes it easier (possible?) to do high resolution
 - Also, FE and Schottkey guns have very high “Brightness.” This means that more electrons per unit area are emitted. As the beam in a thermal emitter is made smaller and smaller, the number of electrons in the beam becomes so small that no usable signal can be obtained from the sample. Also means current density can be high with a FE-SEM.

1,000,000X SEM Image



Au on C resolution standard image collected with AIF's FEI Verios 460L SEM. Au particles are bright. A 0.6nm space between particles is resolved. The Verios has a Schottkey emitter and is an immersion lens type SEM.

1,000,000X SEM Image



mag 1 000 000 x HV 20.00 kV curr 13 pA bias 0 V WD 3.8 mm det TLD mode SE tilt 0 ° ICSU Analytical Instrumentation Facility Verios 460L

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Sn on C resolution standard image collected with AIF's FEI Verios 460L SEM. Sn spheroids are bright. Local oxides on the surface of the Sn can be observed.

Why aren't all SEMs FE?

- Cost
 - Thermionic SEM ~\$150k, low cost examples can be less than \$100k
 - FE SEM start at \$500k, high end models can be over \$1M
- Why the difference? FE-SEMs require more expensive equipment
 - Ultra-High Vacuum (UHV) required for FE electron Gun (UHV = \$\$\$)
 - Additional equipment required
 - Much quieter, higher end electronics are required
 - The whole vacuum system is usually more complex
 - Often, the lens temperature must be constant (cooled with high end chiller)
 - Typically fitted with a complex vibration isolation system
 - Additional maintenance required
 - Once yearly bake (vacuum) and either gun conditioning (tip sharpness) or gun replacement
- Many samples simply don't require the higher resolution/magnification
 - Thermionic SEMs work very well to ~50kX
 - Most SEM images are <10kX

Electron Lenses

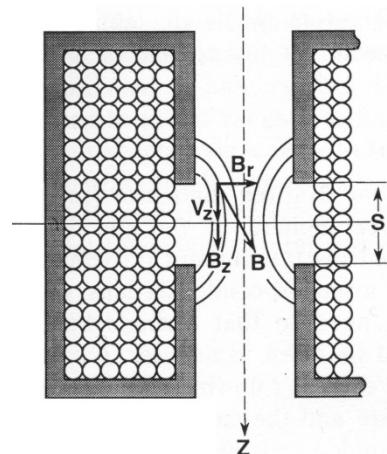
- Usually electromagnetic – current passed through a coil creates a magnetic field that affects the path of the electron
- Physical lens is typically a Fe housing with windings of Cu wire that is rotationally symmetric
- Follows the Lorentz Force Law $\vec{F} = -e(\vec{v} \times \vec{B})$
 - -e is the charge of an electron, v is the velocity vector, and B is the magnetic field vector
 - Electron lenses are always convergent
 - Electrons that are not exactly in the center of the column spiral down the column
 - The length of the column is insufficient for a full rotation
- Electrostatic lenses are possible, but the geometry is more complex

Electron Lenses

- Electromagnetic lens:

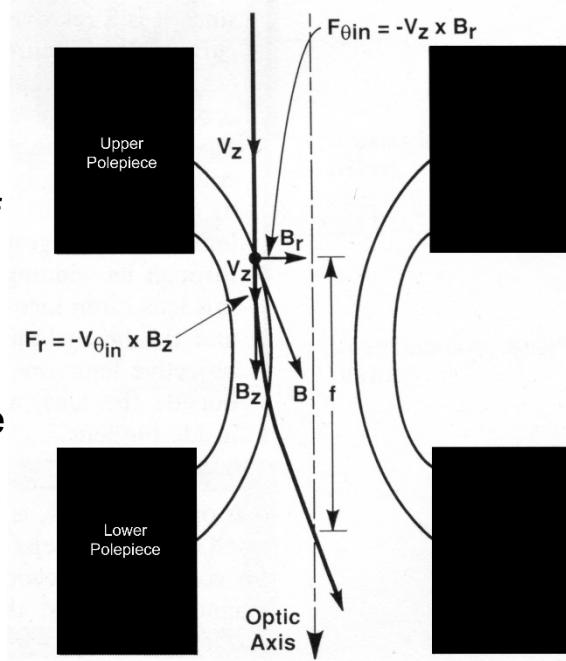
An Fe housing that contains windings of Cu wire

- Pass current through the wire to make a B-field
- The B-field emanates from the gap in the housing
- Lensing action happens in the B-field, i.e., in the gap!
- The beam moves through the bore of the lens (bore = hole through the middle of the lens housing!)
- Lenses are typically rotationally symmetric



Above: Schematic cross-section of an electron lens showing windings and magnetic field lines

Right: Schematic of electron lens action showing B-field lines and path of an electron through the lens



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Electron Lenses – Chromatic Aberrations

- Lorentz Force Law

$$\vec{F} = -e(\vec{v} \times \vec{B})$$

- We want all the electrons to have the same energy, which is expressed as velocity, however, there are often small differences in energy between emitted electrons
- A delta in the velocity will result in:

$$\Delta\vec{F} = -e(\Delta\vec{v} \times \vec{B})$$

- Electrons with different velocities get focused to different places, which is like not being in focus!
- This is called a chromatic aberration, or Ca

Electron Lenses – Spherical Aberrations

- Lorentz Force Law

$$\vec{F} = -e(\vec{v} \times \vec{B})$$

- The B-field varies radially across the bore of the lens, resulting in a delta in the B-field (in addition to any potential errors in the windings)
- A delta in the B-field will result in:

$$\Delta\vec{F} = -e(\vec{v} \times \Delta\vec{B})$$

- Electrons going through different parts of the lens will get focused to different places, which is like not being in focus!
- This is called a spherical aberration or Cs

Electron Lenses – Aberrations

- In general, we typically have both chromatic and spherical aberrations present:

$$\Delta \vec{F} = -e(\Delta \vec{v} \times \Delta \vec{B})$$

- SEM designers lie awake at night trying to figure out how to minimize these aberrations
- Both can be corrected
- Neither is generally corrected in a conventional SEM
- Some SEMs have Δv correctors that reduce the Δ in the v
 - AKA a beam monochromator
 - Verios 460L has a monochromator
- Some TEMs have Cs correctors
 - Titan TEM has a Cs corrector
 - Cost and SEM resolution limits have kept these off of SEMs thus far

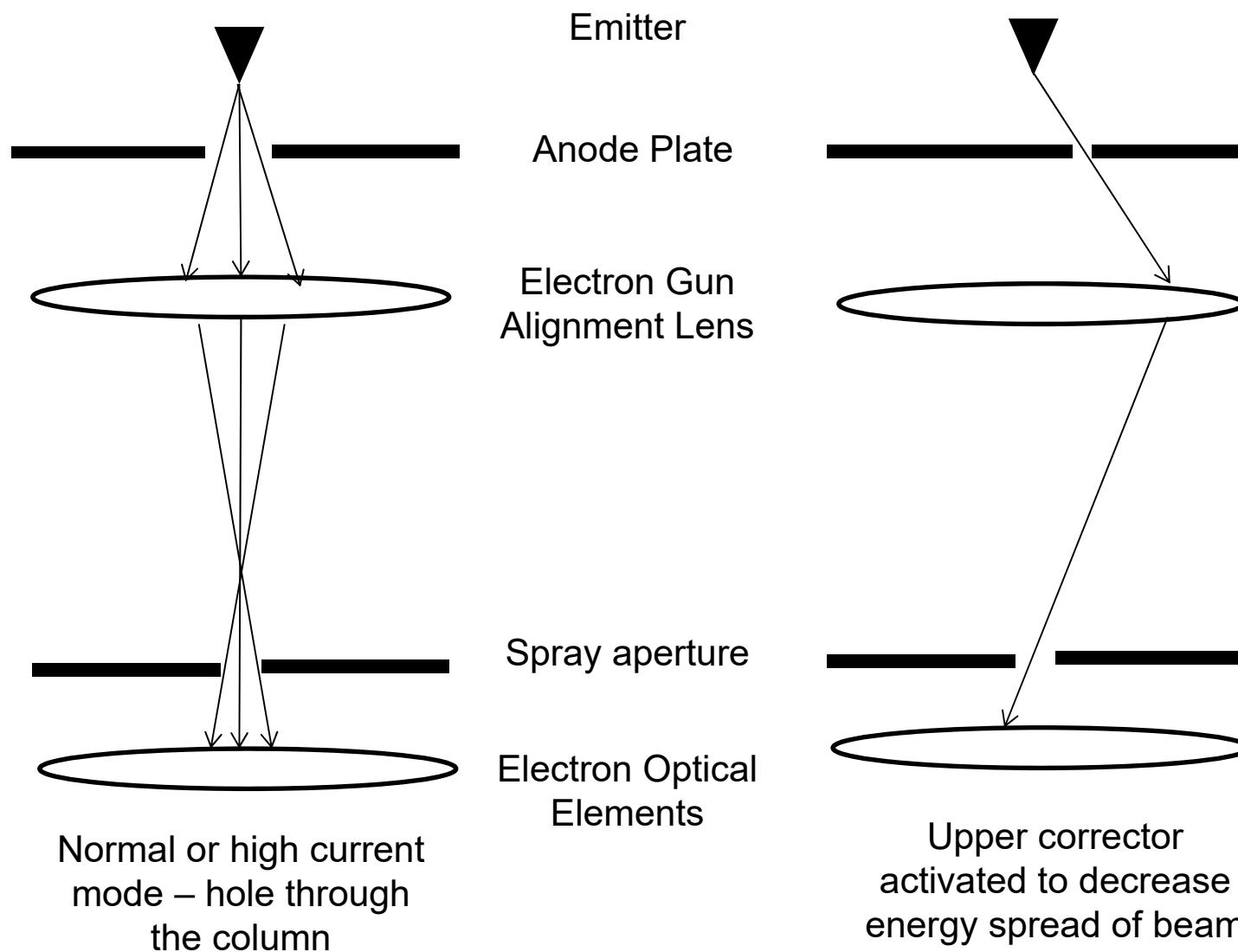
Monochromator to reduce Ca

- Some UHR-SEMs are equipped with a so-called beam monochromator
 - Verios 460L this is called the Upper Corrector
- The upper corrector is a slit type monochromator that limits the energy spread in the beam
 - A monochromator only allows a single wavelength/energy to pass
 - An energetic electron's wavelength is so short that there is still an energy spread, but it is reduced by about a factor of 10 in current designs
- Beam energy is a function of exit angle from the emitter
 - Energy typically varies by +/- 1eV for a Schottkey emitter
 - At low voltage this creates issues with respect to Ca (chromatic aberration)
 - Chromatic aberrations are caused by different energy electrons being focused to different places by the same strength B-field

Upper Corrector – Verios 460L

- The upper corrector (UC) works by electronically blocking the hole in the anode plate that the electrons would normally go through, forcing those electrons through a small slit that is off to the side
 - The slit is an order of magnitude smaller than the hole in the anode plate
- The electrons that pass through the slit have a smaller energy spread than those that go through the main hole in the anode plate
- This reduces the energy spread in the beam by approximately an order of magnitude
 - ± 1 eV uncorrected
 - $\sim \pm 0.15$ eV corrected
- The downsides:
 - Beam current is variable depending on alignment
 - Only so much current can be forced through the little slit: Max = 25 pA
- At low voltage/current, use it if you got it...

Upper Corrector Schematic



Electron Lenses

- Typically two sets of lenses:
 - Condenser lens (set)
 - Objective lens
- Condenser lens set
 - *Demagnifies* initial probe spot
 - Controls beam current
 - Usually there are two condenser lenses that work together
- Objective lens
 - Focuses probe on the sample, i.e., forces the probe into a small round spot at the surface of the sample

SEM Condenser Lens

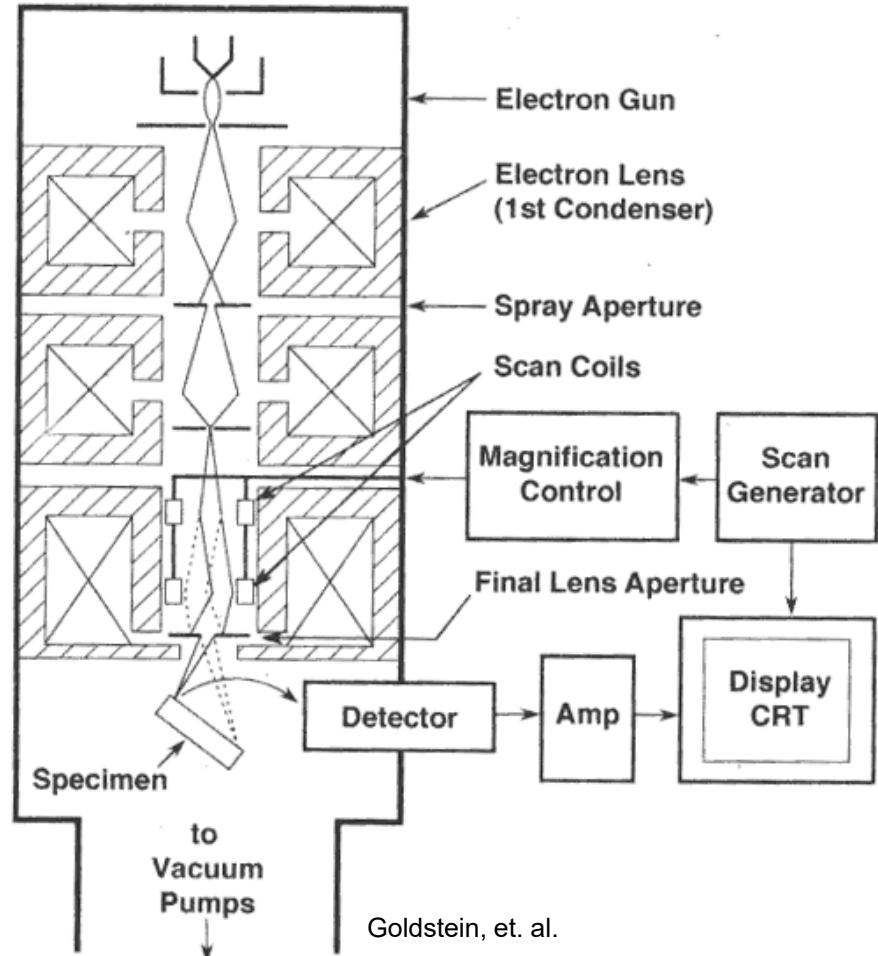
- The primary role for the condenser lens system is to demagnify the initial cross-over
 - Demagnify? After all, microscopes are supposed to *magnify* things...
- SEM: scan a small, round electron probe over the sample
- The emission area for a thermionic SEM is ~100x150um, which would lead to very poor resolution
- The initial electron spot (from the emission area) must be demagnified to make the probe on the sample as small as possible
 - Not only small but **round**
 - Non-round = distorted image

SEM Condenser Lens

- Condenser Lens
 - Usually two individual lenses linked together using one control
- Control labeled as:
 - Beam Current (most common)
 - Spot size
 - CL or Condenser

More lens current results in **less** beam current and a **smaller** spot size!

- Changing the beam current control changes the lens current in the condenser lenses!
 - Lens current = current passing through the lens coil



SEM Condenser Lens

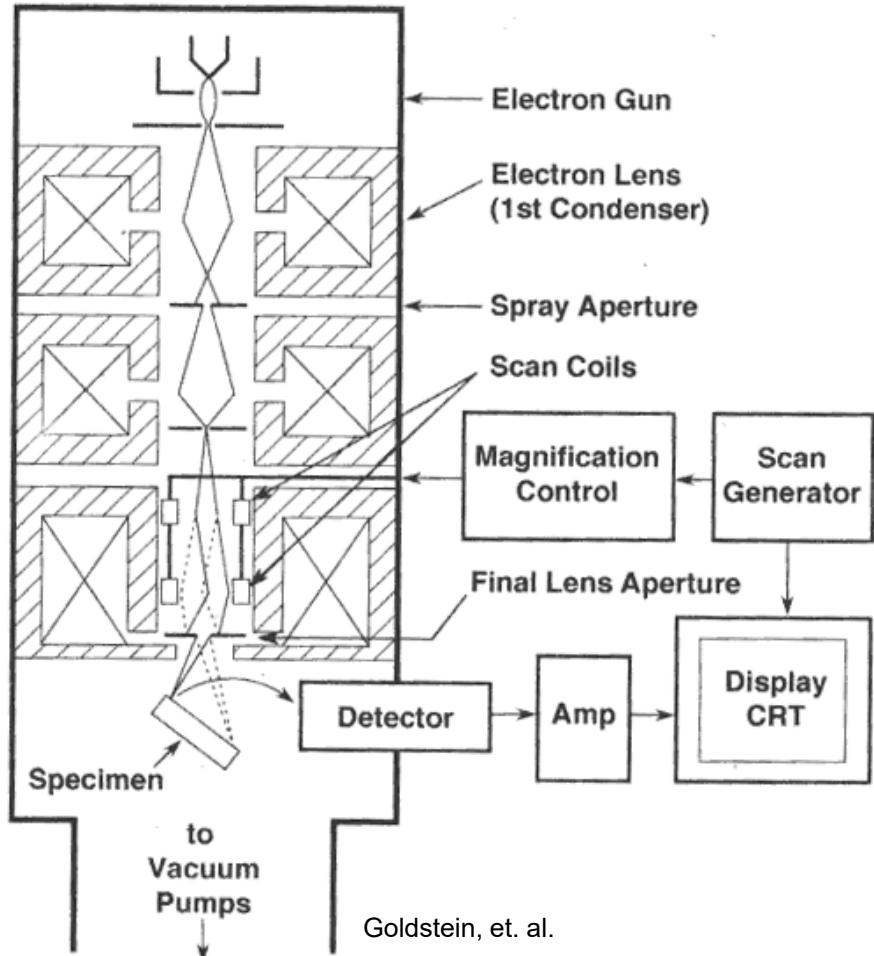
CL system does two things:

1. Demagnifies the initial spot

- Done to improve resolution
- What we want!

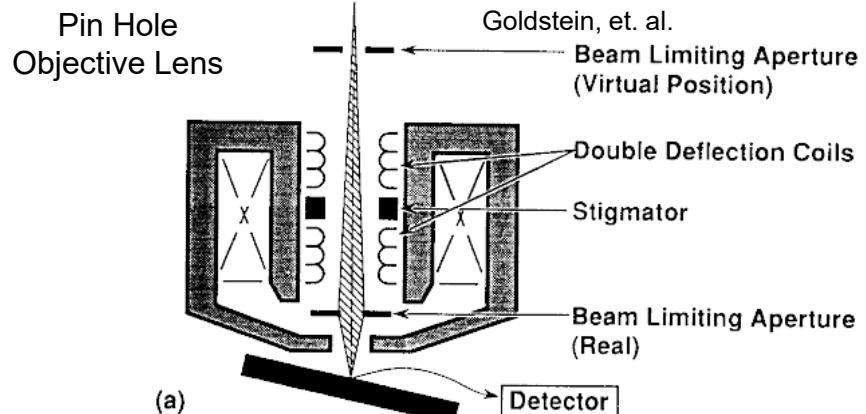
2. Limits beam current as electrons that strike the inside of the column are lost

- As the lens strength is increased, the cross-over from that lens moves up in the column
- Since the column has a finite diameter, the geometry means that some electrons will strike the inside of the column
- Those that strike column parts go to ground and are lost
- Electrons in the beam striking the inside of the column and being lost to ground will reduce the current in the electron beam



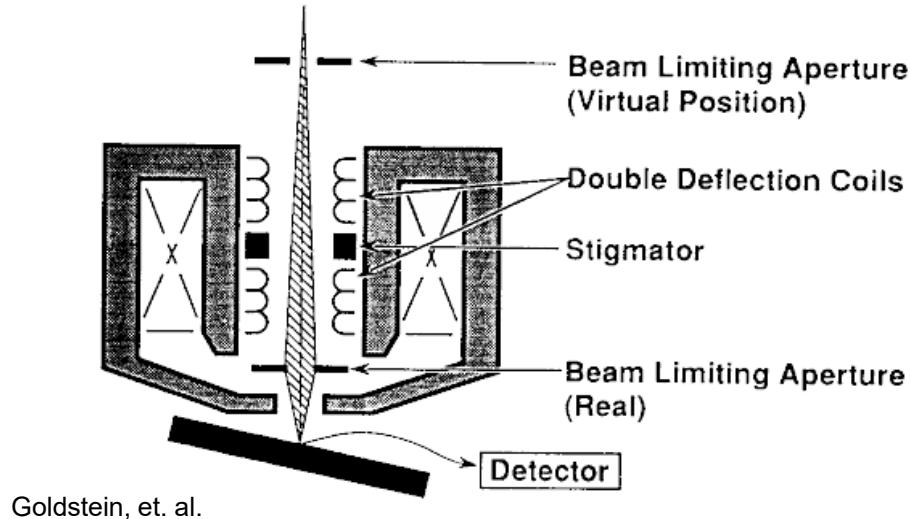
Objective Lens

- Final Lens
 - AKA the pole piece or pole tip
- Probe Forming Lens
- Control labeled as **Focus**
- Changing the focus control changes the lens current in the objective lens!
- Must allow physical space for :
 - Scan coils
 - Stigmator coils
- There are several major types
 - Pin hole (most common)
 - Immersion
 - In-lens
- Conical lens shape (except In-lens!)
 - Focal length 3 – 60 mm
 - Allows for large samples and tilt



Objective Lens – Pin-hole lens type

C. Mooney



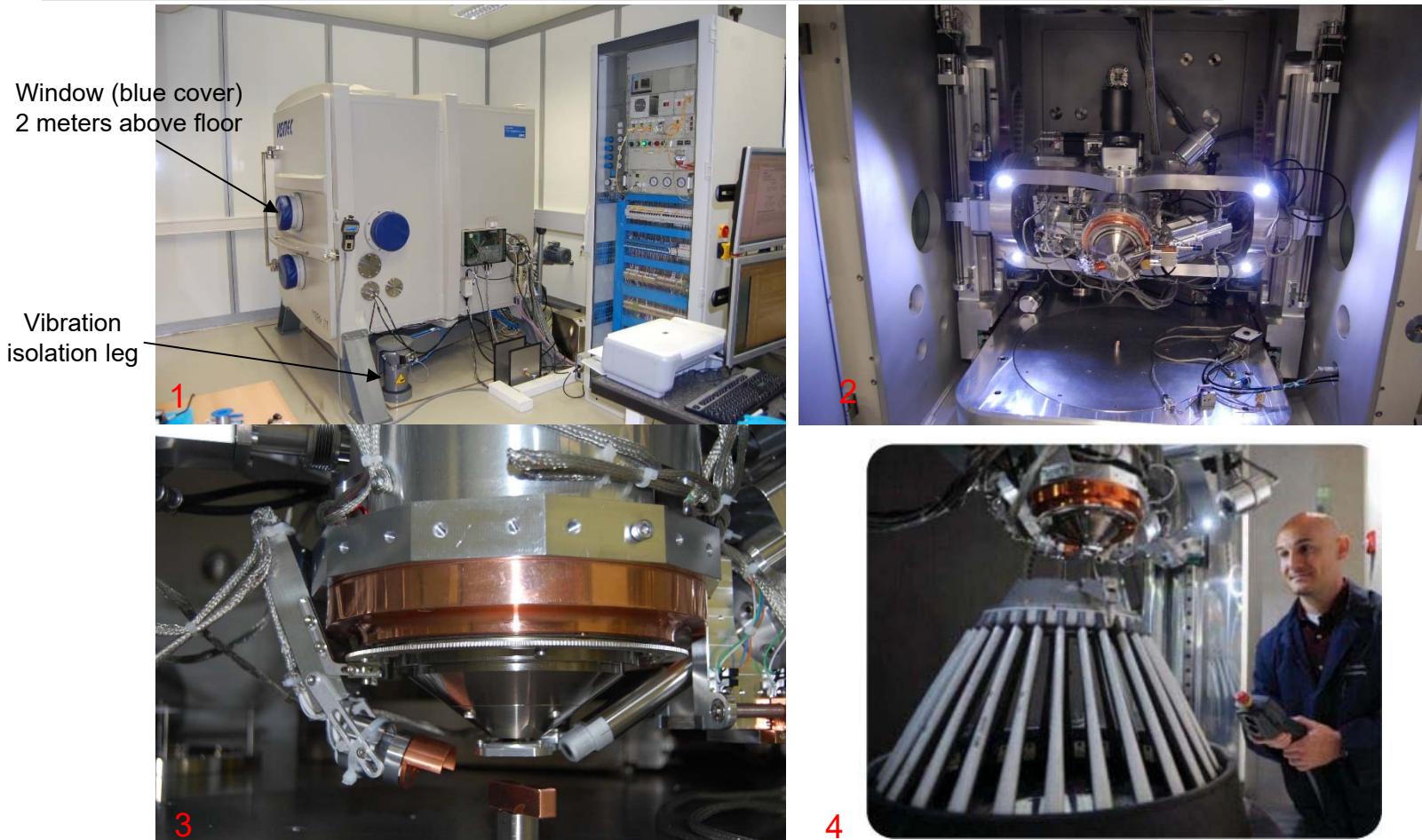
Goldstein, et. al.



Objective lens, aka the pole piece, inside the sample chamber of the VPSEM

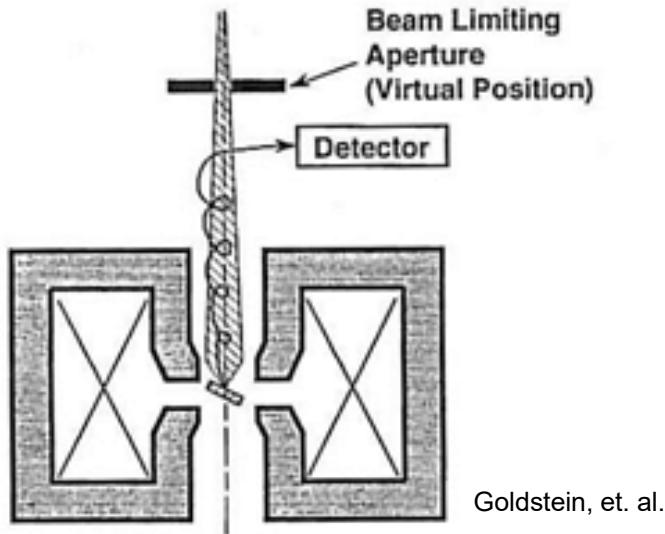
- Schematic of typical pinhole lens
 - Most common type of objective lens
- Most SEMs have this type of objective lens
- Lens is at the bottom of the electron-optical column, typically inside a vacuum chamber that also houses a sample stage and detector(s)
- Sample sizes are limited by the size of the chamber and stage
 - There are SEMs with chambers large enough that one can literally walk in!

Giant Sample SEM



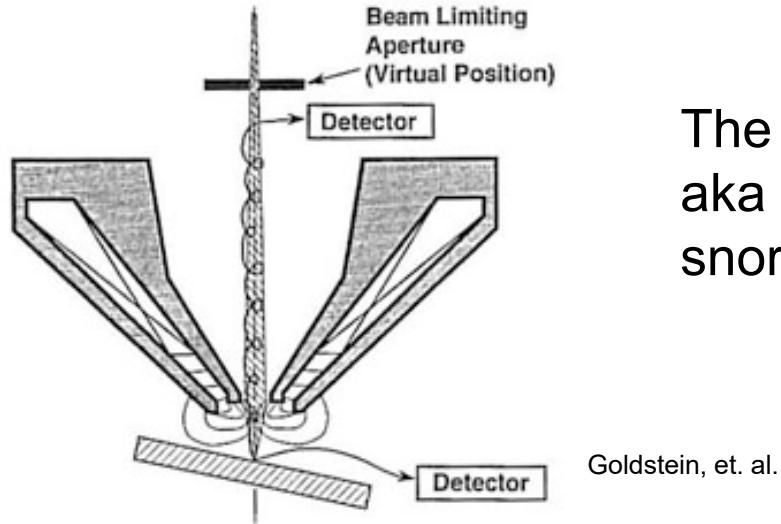
Mira Large chamber SEM. 1 Instrument. 2 Pole piece on movable rack. 3 Detail of pole piece showing detectors (SE, BSE, and EDS). 4 Preparing to observe part of an airplane

Objective Lens – In lens type



- Schematic of typical in-lens design SEM sample chamber
- Sample is placed inside the lens using a TEM style sample holder on a TEM grid (3mm diameter)
- Sample sizes are severely limited (3x5x1 mm is a “bulk” sample for an in-lens SEM)
- The electron detector is located inside the column above the lens
- Electrons emitted from the sample spiral up the column to the detector

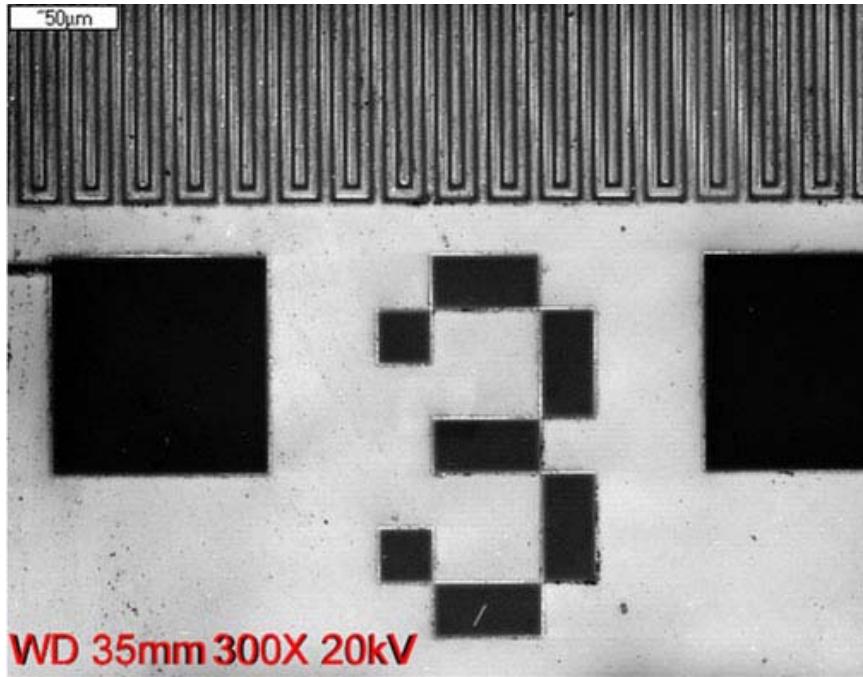
Objective Lens – Immersion lens type



The immersion lens is aka the semi-in lens or snorkel lens

- Schematic of typical immersion design SEM
- Sample is held below the lens but is immersed in a magnetic field emanating from the lens
 - With an electromagnetic immersion lens, ferromagnetic samples cannot be observed
 - Some newly designed SEMs are going toward electrostatic immersion lenses
- Bulk samples can be observed
 - Sample thickness may be more limited than a pinhole lens SEM
- The electron detectors are usually located both inside the column and in the chamber

Image Rotation vs. focal length change



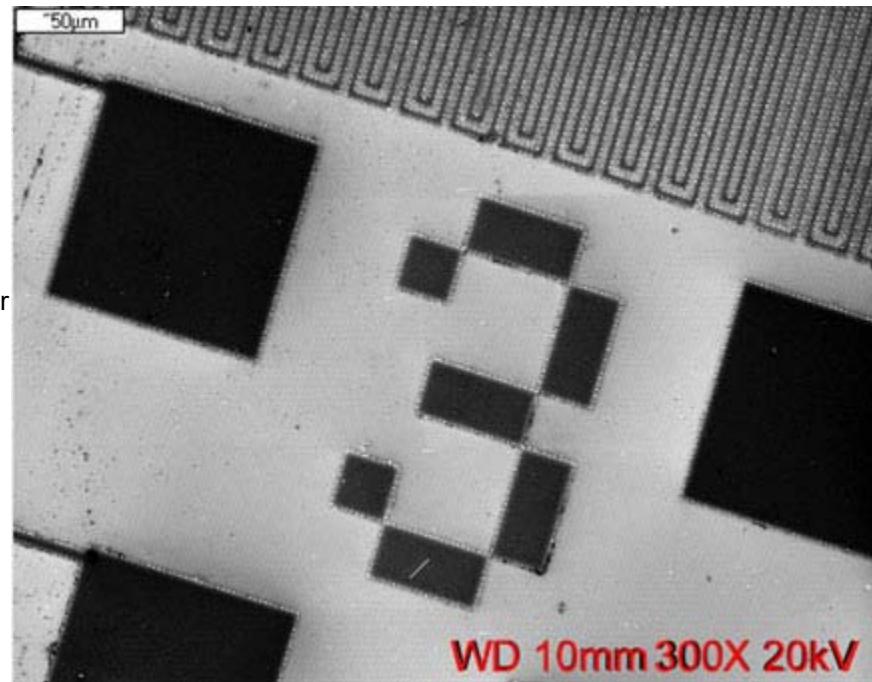
(Slight moire effect here)

D.Batchelor

Recall:

Lorentz Force Law: $\vec{F} = -e(\vec{v} \times \vec{B})$

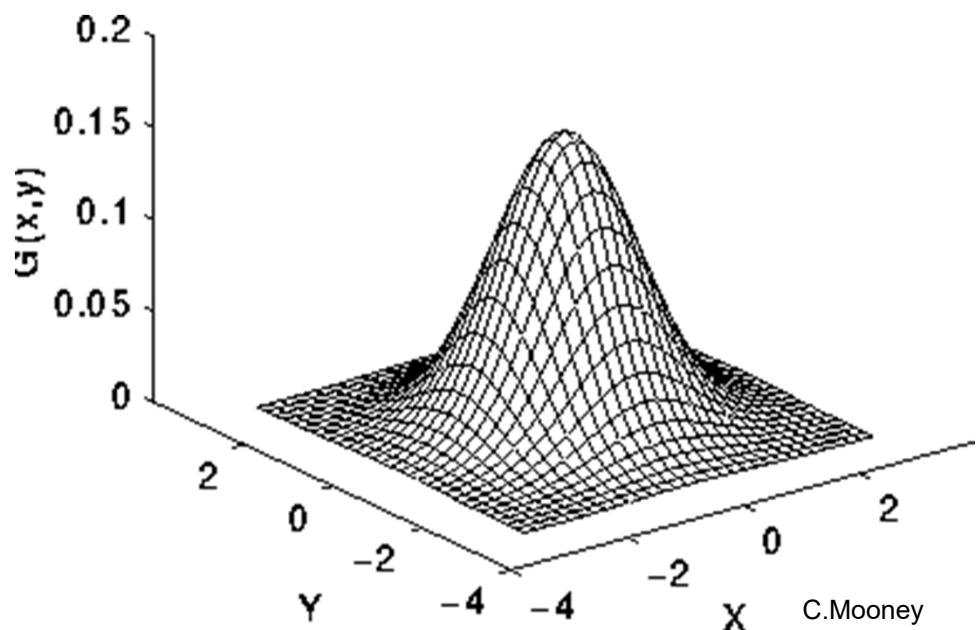
- The cross-product means that the electrons are spiraling down the column – change focus and the image rotates!



NC STATE UNIVERSITY

Objective Aperture

- Consider that the electron probe is Gaussian in nature
 - There will be a wide distribution of electrons, some of which will be far away from the center of the probe
- The aperture limits the far off-axis electrons
 - These are the electrons that run with scissors...
 - On axis electrons play well with others...



General Gaussian Distribution Plot

Electron probe has this shape at the sample surface

The so-called “tails” of the beam are the far off-axis electrons.

Objective Aperture

- The objective aperture serves several purposes:
 - Remove off-axis electrons (reduce aberrations)
 - Increase the potential depth of field (geometry effect)
 - Increase the potential resolution (reduce aberrations)
- The objective aperture is generally changeable, that is, there is an aperture strip that allows for different objective apertures depending on the needs of the sample.
- In general:
 - Larger apertures are used for X-ray analysis (need current!)
 - Smaller apertures are used for imaging (better geometry, reduce Ca)
 - Small apertures will limit current and signal – requires longer counting
 - More electrons in = more signal out!

Electron Optics Primary Goal

Point of the electron optics is to place a small, round electron probe on the sample!

The smaller the spot, the higher the potential resolution!

Electron Optics – Putting it together

1. Cathode supplies electrons
2. Wehnelt (#1 Anode in the FE) forms first spot
3. Anode (#2 in the FE) accelerates electrons
 - Typically from 1 – 30 keV
4. Condenser lens demagnifies initial spot
 - Also limits beam current at the sample
5. Objective lens focuses final spot on sample (note stigmator coils...)
6. Objective Aperture removes off-axis electrons

We still haven't formed an image!

7. Images are formed by scanning spot over sample and collecting signal with respect to XY position

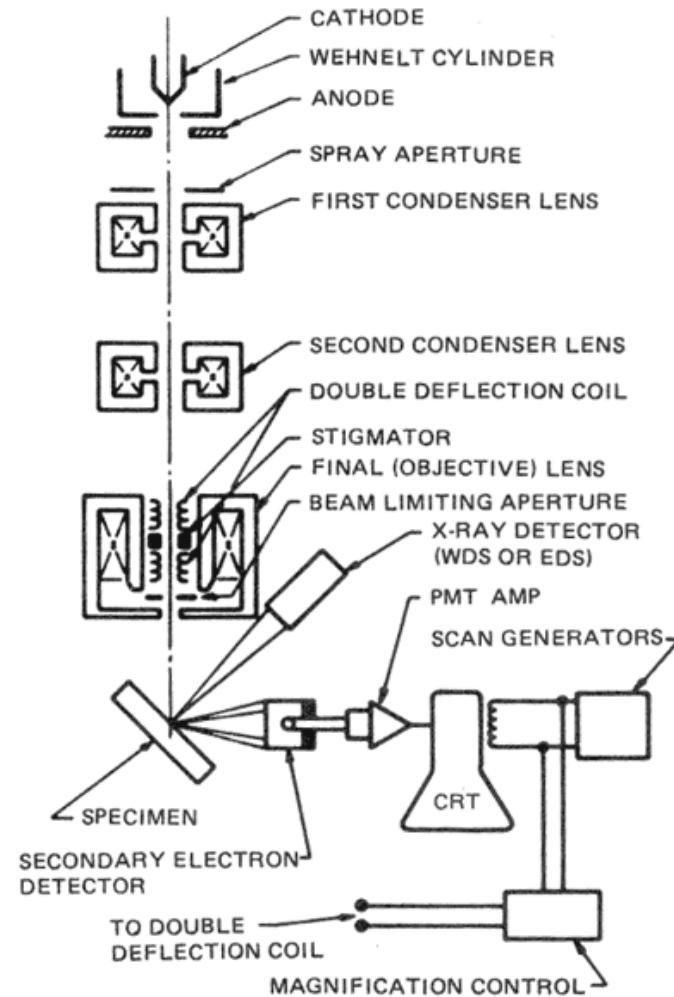
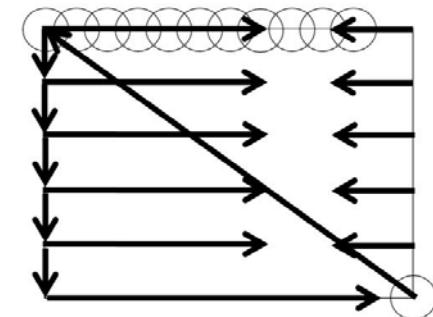


Image source unknown

Image formation in the SEM

- To construct a map (image) of the sample, we scan the beam over the region of interest
- Beam Deflection coils (electromagnets!) are used to move the beam across a specimen, usually in a raster pattern.
 - Place beam at point X1Y1, collect signal
 - Display signal intensity at point X1Y1 on monitor
 - Repeat through point XnYm to build an image
- For microscopy, the size of the pattern on the screen is larger than the size of the pattern on the specimen.

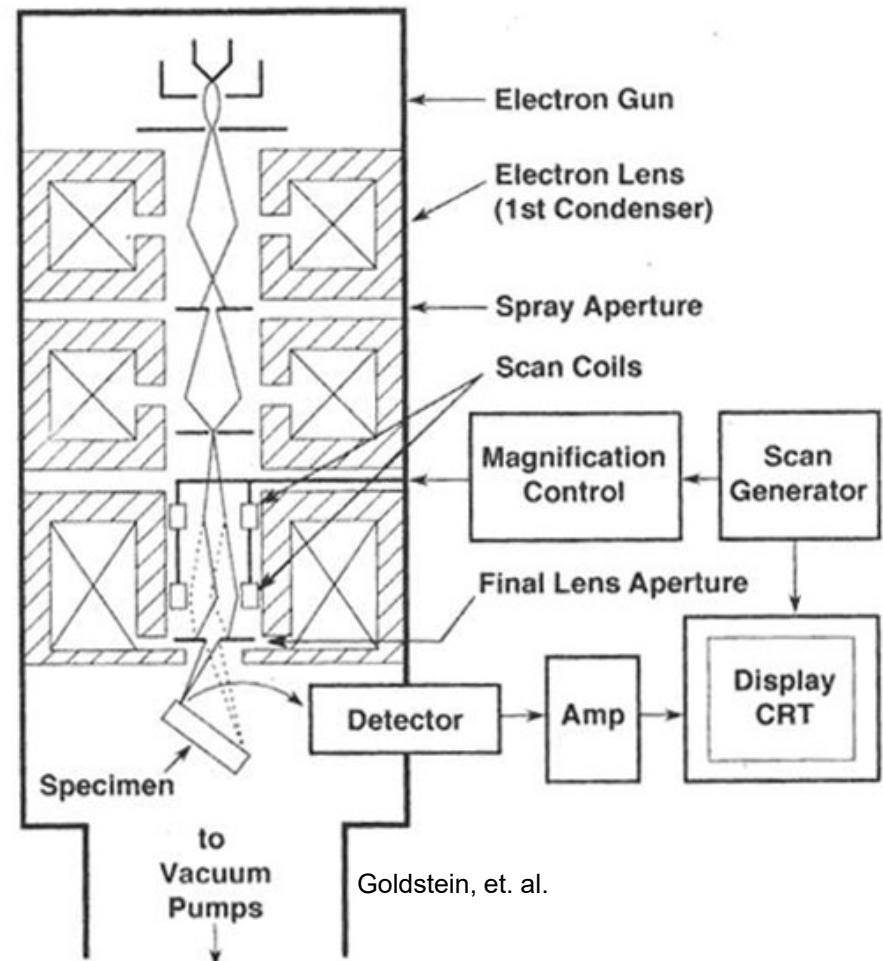


Deflection Coils

- Typically, a double deflection design
- Scan the beam over the sample
- The larger the scan, the lower the magnification

More scan coil current results in *more* beam deflection and a *larger* scan size!

- The magnification control changes the current through the scan coils!



Focus

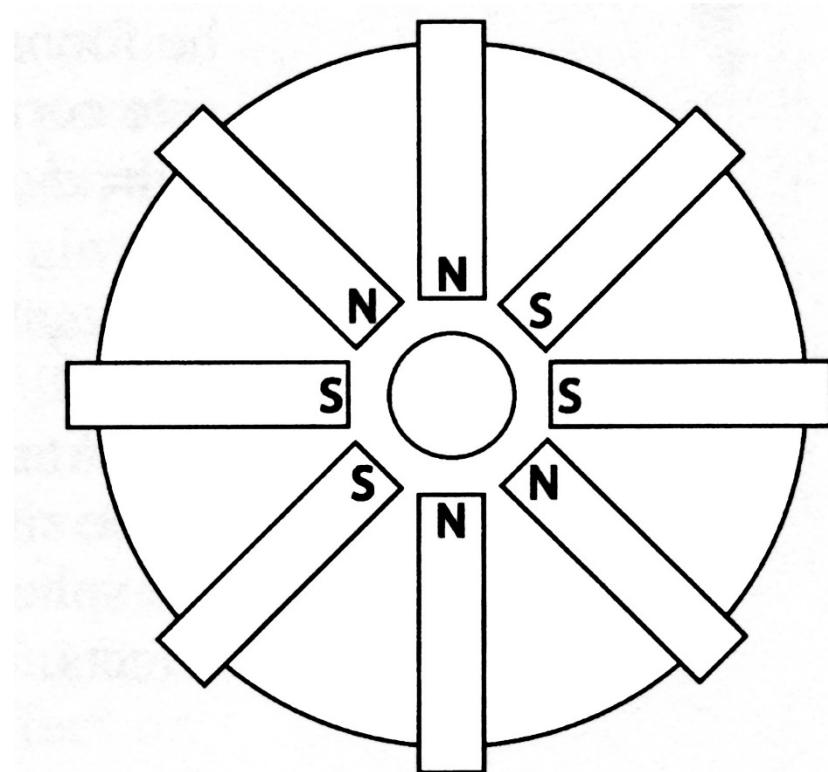
- Focus should be an easy concept
- If the image has sharp, crisp edges and is detailed, then it is focused
- In practice, to focus, one has to see the image out of focus
- That is, blurry features are brought into focus by going through focus to observe where the features are crisp and sharp
 - Can't sneak up on focus!
 - Must boldly go past in order to observe where it is the best!
- Focus is complicated by astigmatism in the beam

Astigmatism

- Stigmatism definition: The condition of an optical system (such as a lens or mirror) in which rays of light (from a point source) converge in a single focal point
- In an SEM, astigmatism means that the beam is not round
- Initial Spot formed by the Wehnelt cap in a thermionic SEM is generally not round
 - Emission Area $\approx 100 \times 150 \mu\text{m}$
 - ***Not a round, point-like source!***
 - The non-roundness continues through the optics to the sample
- In a FESEM the initial spot is theoretically symmetric, but non-roundness is introduced by imperfections in the anode plates and lenses and continues to the sample
- In both cases, astigmatism correction is critical for high quality images

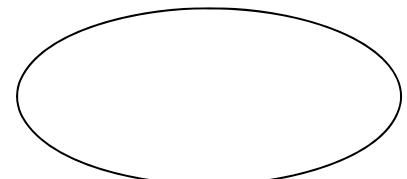
Octopole for Astigmatism Correction

- Typical design for an octopole astigmatism correction unit
 - Four N-S pole pairs
 - Each can create a B-field that can push on the electron beam
- The octopole creates an **asymmetric** magnetic field to correct astigmatism in the electron beam
- The beam passes through the center of the octopole

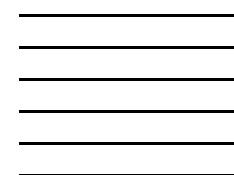
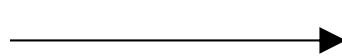


Astigmatism

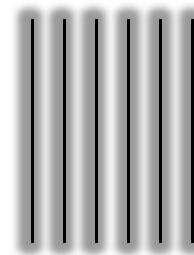
- At focus, an astigmatic beam will produce a fuzzy image that appears to be out of focus
- During over or under focus conditions (i.e., focus above or below the sample) the projection of the beam can appear elliptical
 - Result is different resolution in different directions!
 - By changing between over and under focus conditions, the astigmatism can be recognized and corrected



Elliptical Beam

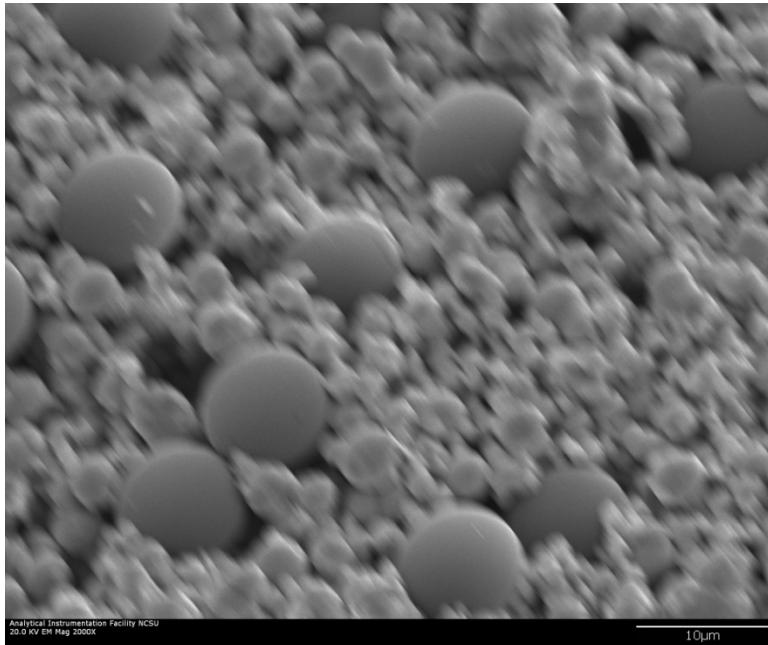


Sharp
Features

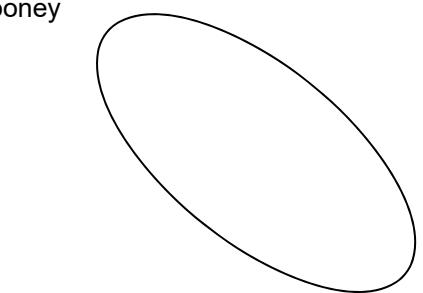


Blurred
Features

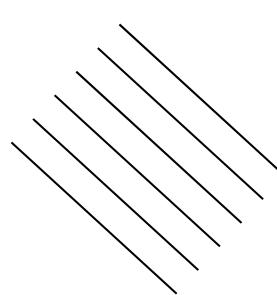
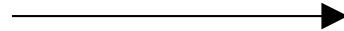
Astigmatism Correction



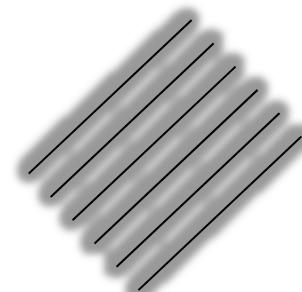
C.Mooney



Elliptical Beam



Sharp Features

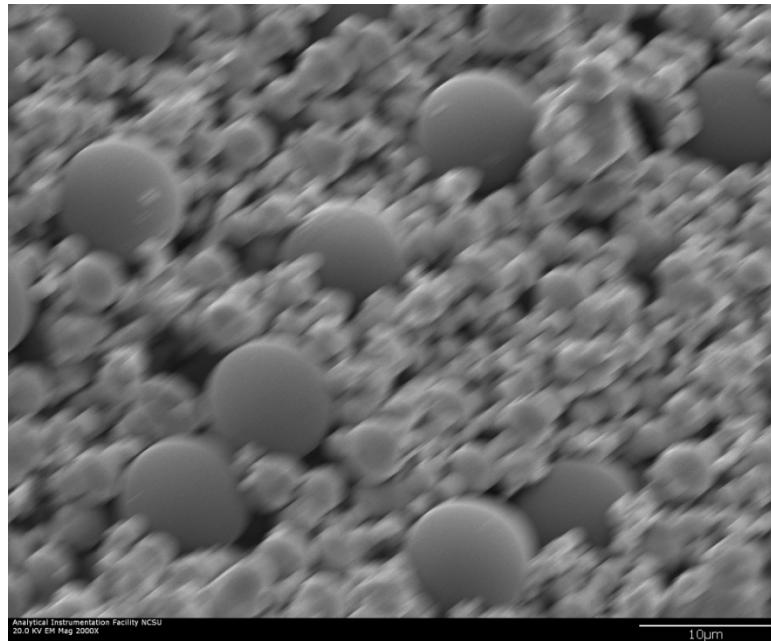


Blurred Features

To correct for astigmatism, the focus is changed from over focus (focus above the surface) to under focus (focus below the surface). Due to the cross product nature of electron optical lenses, the beam rotates causing features to appear to be sharp in one direction and not in the other.

Over focus Condition shown.

Astigmatism Correction

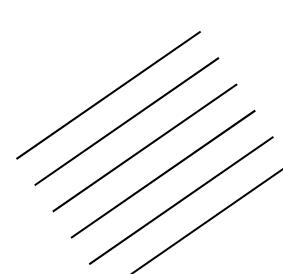
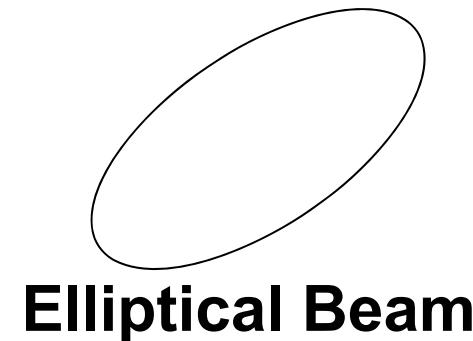


C.Mooney

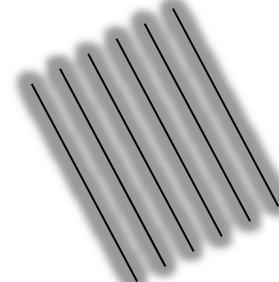
With an over or under focus condition, the features will appear to be stretched. At the just focused position, no apparent stretching will occur. At this point, the stigmator controls are adjusted to bring features into focus.

Note that this is easiest with round or irregular features!

Under focus condition shown.

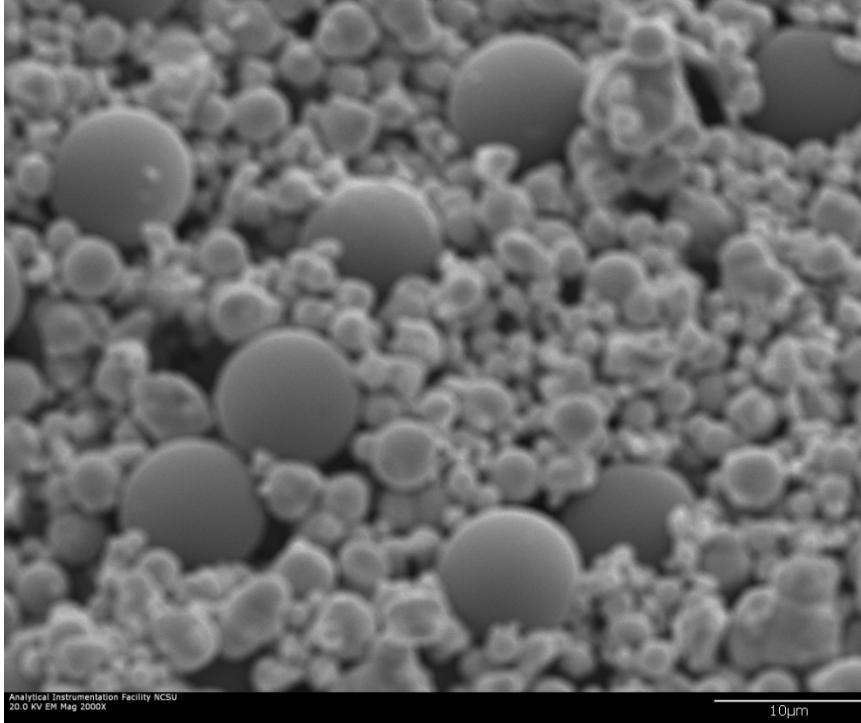


Sharp
Features

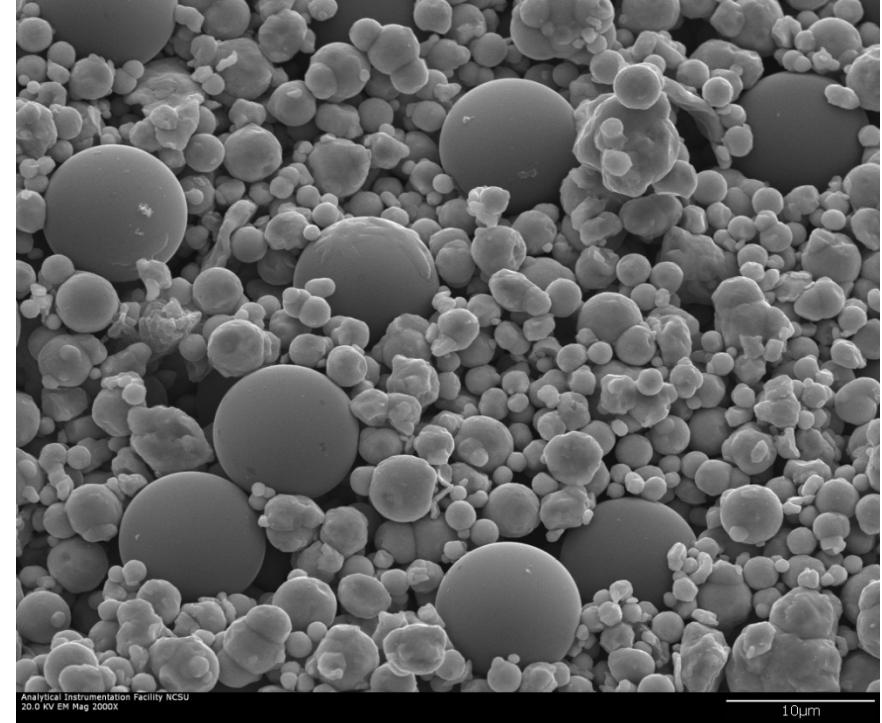


Blurred
Features

Astigmatism Correction



Good focus, bad stigmation. The image appears blurry but not stretched or distorted. Adjust stigmator controls now...



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Good focus and stigmation
Features are crisp and clear

***Correct astigmatism is critical
for high quality images***

Electron Optics Primary Goal

- ***Point of the electron optics is to place a small, round electron probe on the sample!***
 - Electron Optics includes lenses, apertures, and astigmatism correction coils
 - The electron probe can be called the spot
- The smaller the spot, the higher the potential resolution!

Electron Optics – repulsion effect

- It is well known that like charges repulse each other
 - Electrons should push each other away
- Do we need to worry about electrons in the column repulsing each other?
- Most of the time and especially for high resolution applications, **No!**
- In an SEM, due to the relatively low currents and near relativistic velocities of the electrons, on average there is ONE electron in the column at any given time
 - At very high currents this can become a problem, but as the current goes up, so does the beam size due to the nature of the optics

Electrons in the Sample

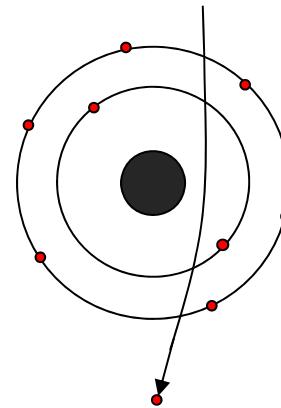
- It is possible to maintain control of a primary electron while it is in the column
- Once the electrons in the beam strike the sample all control is lost
- Electrons in the sample behave probabilistically, i.e., there are finite probabilities that one of a variety of different interactions will occur
- If we inject enough electrons into the sample, we can measure the results of the various interactions by collecting a measureable number of signal particles
 - A single ejected particle is typically not enough signal to measure
 - A large number of particles need to strike the detector

Electron Beam-Sample Interactions

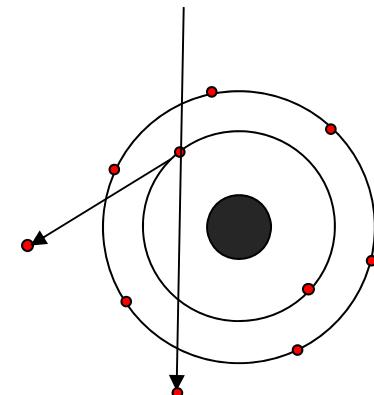
Interactions between primary electrons and the sample can be separated into two broad categories:

- Elastic scattering
 - Trajectory of the electron is affected
 - Energy of the electron is not (significantly) affected
 - Example: Backscattered Electrons

- Inelastic scattering
 - Trajectory of the electron is not (significantly) affected
 - Energy of the electron is transferred to an atom in the sample
 - Examples: Secondary electrons and X-rays



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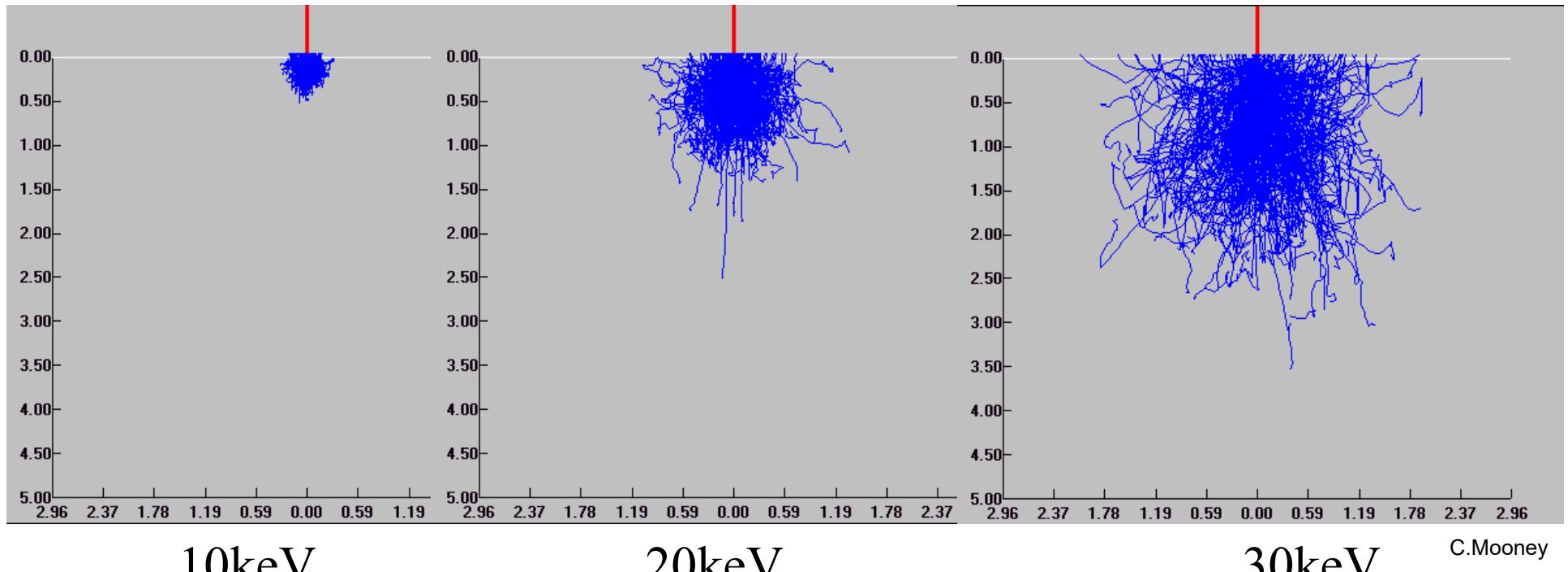
Interaction Volume

- Electrons do not stop at the surface – they can penetrate microns into a sample and generate signals throughout the interaction volume
- The dimensions of the interaction volume depend on:
 - Primary electron energy
 - Atomic weight
 - Density
- Interaction volume is generally determined via Monte Carlo simulations

Monte Carlo Simulations - General

- Monte Carlo simulations solve deterministic problems using a probabilistic analog
 - Essentially, we roll a set of dice that is weighted to the probabilities we are trying to measure
 - Enough dice rolls gives us a probabilistic simulation of a complex problem
 - Difficult to execute without computers
- Dice roll 1: How far into the sample is the first interaction (electron energy)?
- Dice roll 2: What is the interaction (elastic or inelastic)?
- Dice roll 3: What is the result of the interaction (secondary electron, x-ray, etc.)?
- Dice roll 4: How much energy does the electron lose (based on interaction)?
- Dice roll 5: What direction does the electron then travel (based on interaction)?
- Repeat until Energy = 0 (or below a threshold)
- Invented in the 1940s by Stanislaw Ulam while working on nuclear weapons development at Los Alamos Scientific Laboratory
 - Because it was part of the Manhattan project and secret, it required a code name
 - Ulam's uncle would borrow money to gamble at the Monte Carlo casino
 - Since the simulation is a series of dice rolls...

Interaction Volume as a function of Energy



10keV

20keV

30keV

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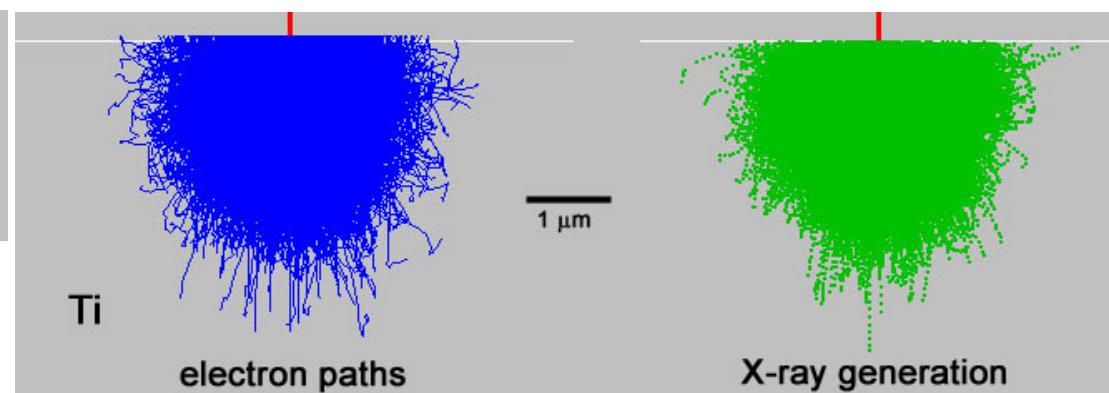
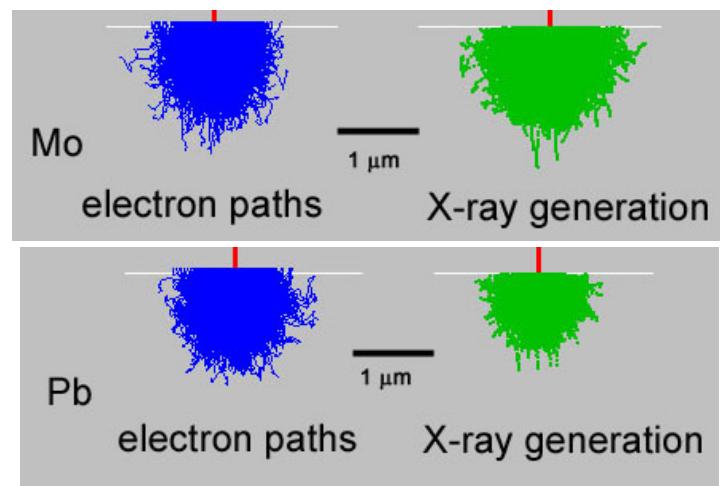
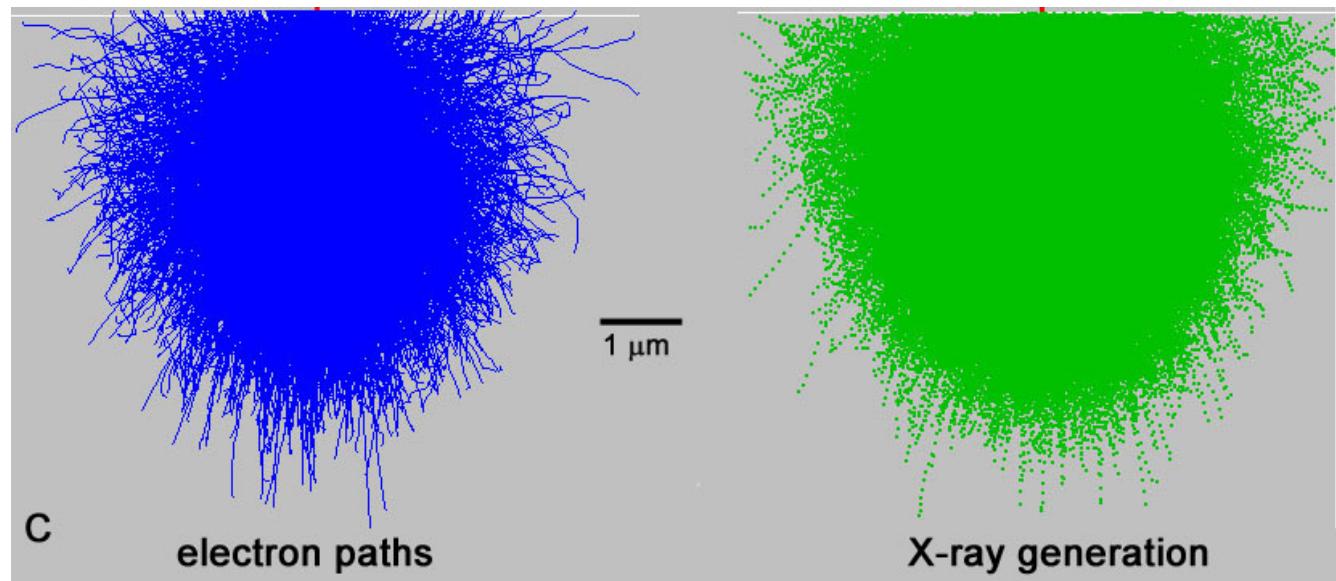
- Monte Carlo simulations to determine interaction volume as a function of beam energy for an Fe sample at different beam energies
 - Monte Carlo simulations take into account all of the possible interactions to give a visual representation of the interaction volume
- As beam energy increases, so does the interaction volume, typically by the energy to the ~ 1.7 power

Interaction Volume as function of Material

Beam energy 20keV
for all simulations

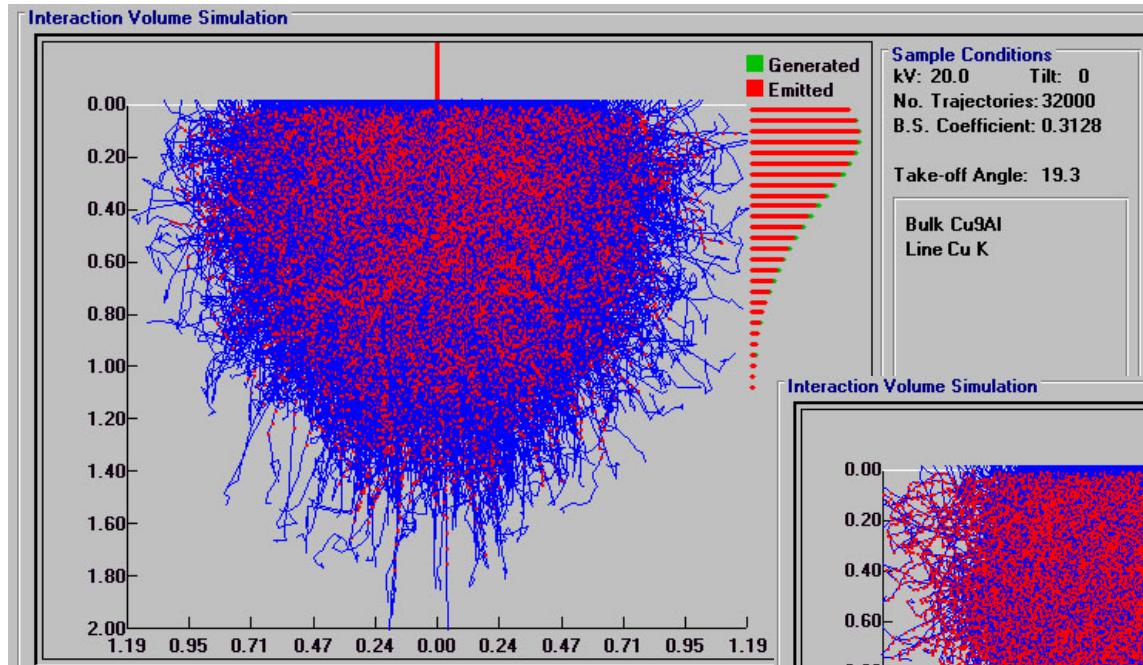
The scale marker bar
is constant for all
simulations

In general, as Z
increases, interaction
volume decreases



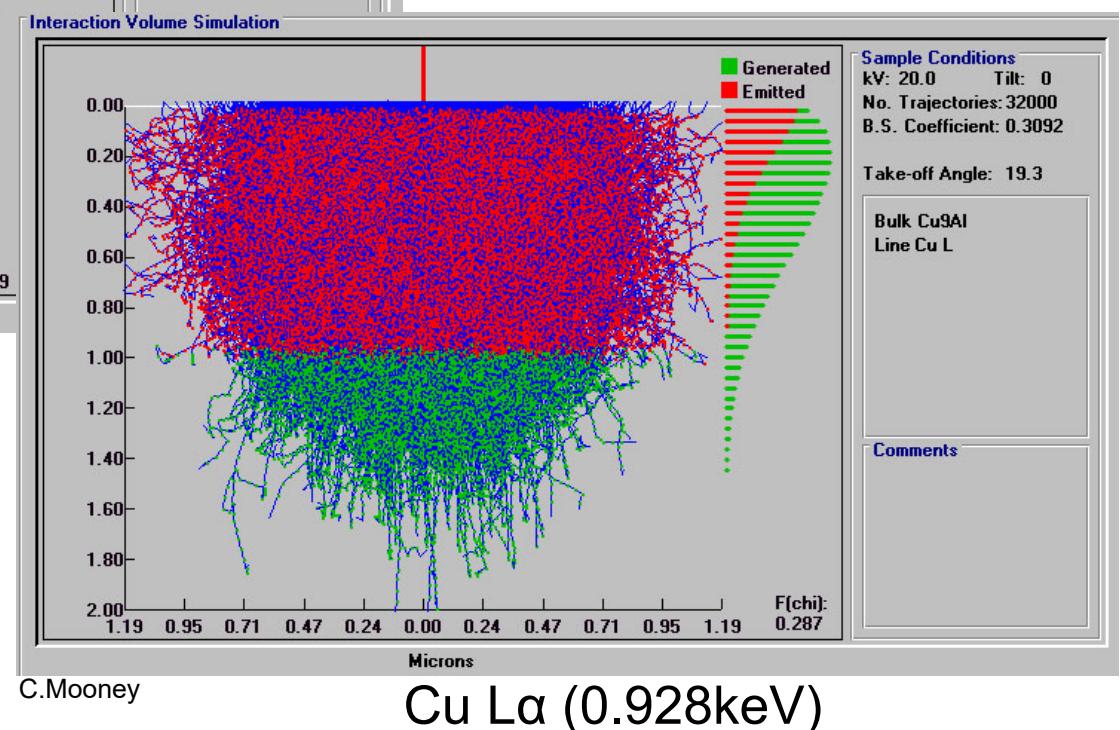
C.Mooney

Generated and Emitted X-ray Volume



More energetic particles will escape from greater depths in the sample

Generated (green) and emitted (red) X-rays in a Cu/Al (90/10) alloy from a 20keV beam
C.Mooney



Interaction volume

- Note from the preceding slides that most electrons are forward scattered into the sample
- Most signal particles are trapped inside the sample and do not escape!
- Only those signal particles that impinge the detector will be detected!

Analysis in the SEM – General

- Traditional SEM at high energy is a bulk technique
 - Micro-scale bulk, but still bulk
 - Think interaction volume – beam electrons have to interact with the sample, this interaction take volume, higher energy = bigger volume
 - At high energy, much of the interaction is below the surface in a volume
- X-ray analysis in the SEM is really a (micro) bulk technique
 - It takes energy to generate X-rays
 - Most SEM-EDS is done at high energy
 - We are collecting X-rays from an X-ray generation volume that is typically on the order of microns in diameter instead of the surface of the sample – micro scale bulk
- SEM is really a micro to sub-micro technique
 - We can observe nano-scale features spatially
 - Features smaller than ~ 10 nm are more satisfying with a S/TEM
 - Elemental analysis is really micro-scale due to the interaction volume

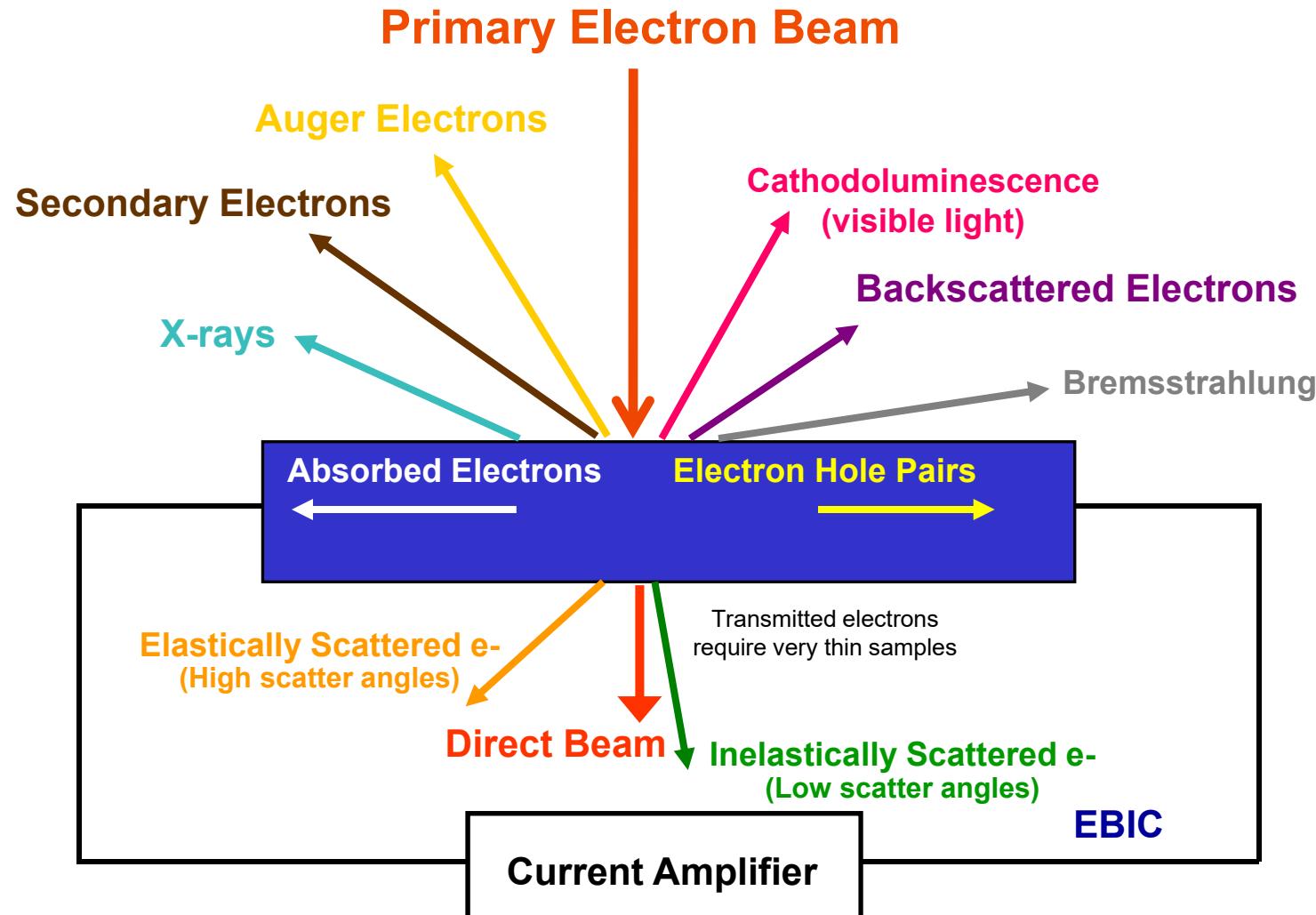
What is a Signal?

- In general: A signal is a response from the sample to an input
 - Imagine an experiment on a bear (highly recommended that you do not attempt this!)
 - Input is what you do to the bear, e.g., poke the bear with a stick
 - The signal is the bear's reaction, which will probably not be good
- In the case of the SEM, a primary beam electron is the input, it impinges and then interacts with the sample
 - Poke the sample with an electron beam
 - The signal is what the sample sends back
- Electron-sample interactions produce:
 - Secondary particles that may be ejected from the sample
 - The impinging primary electron can be ejected from the sample

SEM Signals – General

- Signals in the SEM are particles that are ejected from the sample that can be detected!
 - Electrons and photons are typical signal particles
 - Not all signal particles are detectable – have to impinge the detector
- Ejected particles that we detect usually need to move in the direction of the detector!
 - Big detector = large solid angle of collection → more signal!
 - Exception: Secondary electrons, specimen current
- Usually, signal particles are ejected opposite the direction of the primary beam, assuming the beam is normal to the sample
 - Exception: Primary electrons can be forward scattered through a tilted sample (70 degrees) to provide crystallographic information (EBSD)

Signals available from SEM



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Commonly Used Signals in an SEM

- **Secondary Electrons**
 - **Backscattered Electrons**
 - **X-rays**
 - Sample Current
 - Cathodoluminescence
 - Electron Backscatter Diffraction (EBSD)
 - Really forward scattered electrons
 - STEM – transmission through the sample
- Signals in bold will be covered in this presentation
- All of these capabilities are available in AIF SEMs

Signal Detectors

First we must ask: What do we want from a particle detector?

- Only detect one type of particle
 - Avoids cross-talk, i.e., don't want an X-ray to create signal in any detectors but the X-ray detector
- Minimize the number of signal particles required for a measureable response (i.e., high sensitivity)
 - All detectors have noise
 - Need enough signal to overcome the noise
 - Ideally, the signal is high enough that the noise can be ignored
- Subtend a large solid angle of collection
 - Maximize the surface area of the detector to maximize signal collection

Signal Detectors

- Fortunately, most detectors are signal specific
 - Only measure if a signal particle has stuck the detector or not
- BSE detectors are generally insensitive to SEs and X-rays
 - Exception is from a sample that has a high negative bias applied as this will accelerate the SE to BSE energy levels
- X-ray detectors are sensitive to high energy electrons
 - Fortunately, this is easy to deal with as electrons can be filtered out using a magnetic field that does not affect the X-rays

All is not bliss:

- SE detector will also pick up any BSEs that happen to strike the detector
 - Fortunately, the BSE contribution to the SE image is usually small

Signal Emission Coefficient

- The emission coefficient is defined as the number of particles that are emitted from the sample divided by the number of primary electrons that strike the sample
- Each signal will have its own emission coefficient
- In general,

$$(EC) = n_{ep} / n_b$$

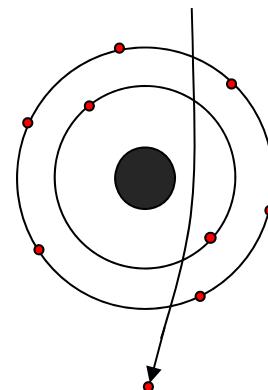
Where n_{ep} = number of emitted particles, n_b = number of primary particles
In an SEM n_b = number of primary electrons.

Emission Coefficient

- Why do we care about the emission coefficient?
- The images we create are maps of emission coefficient vs. spatial position!
 - The emission coefficient we observe is determined by the detector we choose to use
 - SE emission, SE detector, etc.
- That is, the image is a map of the number of electrons/X-rays emitted from different locations
 - No information about height!
 - Map BSEs, SEs, and X-rays, depending on the detector

Backscattered Electrons

- Elastic scattering of primary incident electrons causes some to exit the sample in the general direction of the primary beam
 - Ejected from the sample surface by Rutherford Backscattering
 - Usually multiple scattering processes take place before the electron is scattered out of the sample
 - Average scatter angle is < 5 degrees



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- The backscattered electron coefficient is denoted as η
- Energy is in the same range as the energy of the electron beam
 - Most BSEs have $\sim 80\%$ of the energy in the beam

Backscattered Electron Emission Coefficient

- The backscattered electron emission coefficient is a measure of how many electrons are scattered back out of the sample

$$\eta = n_{\text{BSE}}/n_b$$

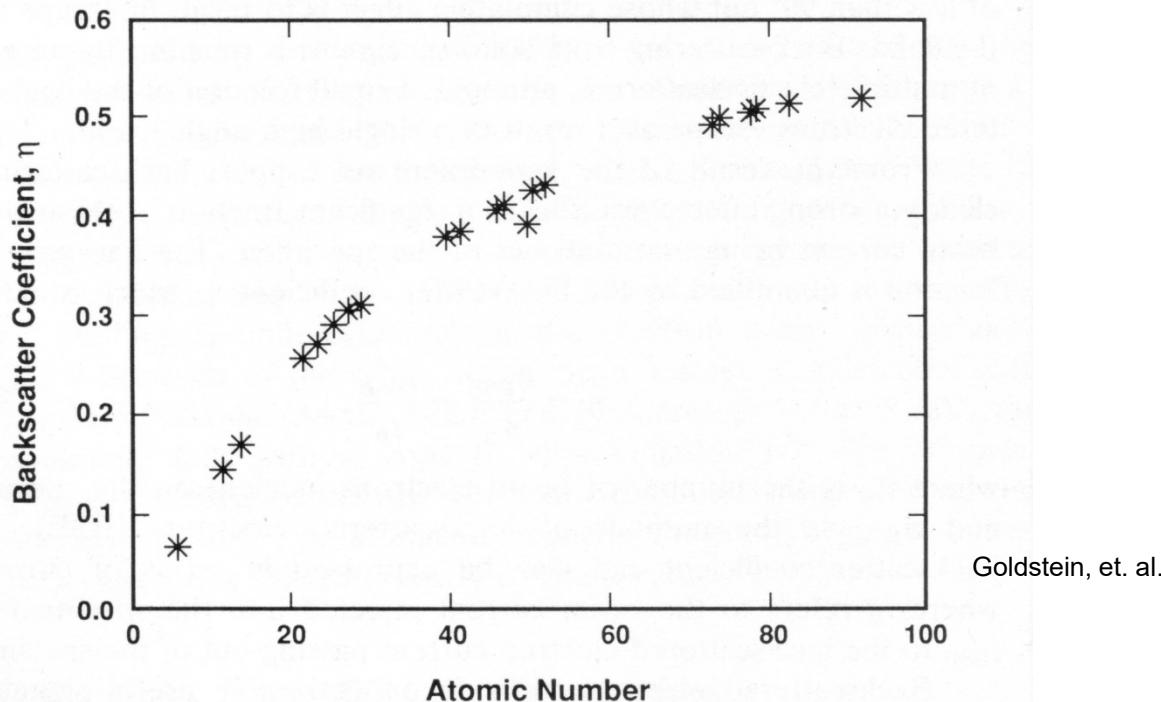
where, n_{BSE} is the number of backscattered electrons, n_b is the number of beam electrons

- To measure η , express as a function of current ($i = n/t$)

$$\eta = i_{\text{BSE}}/i_b$$

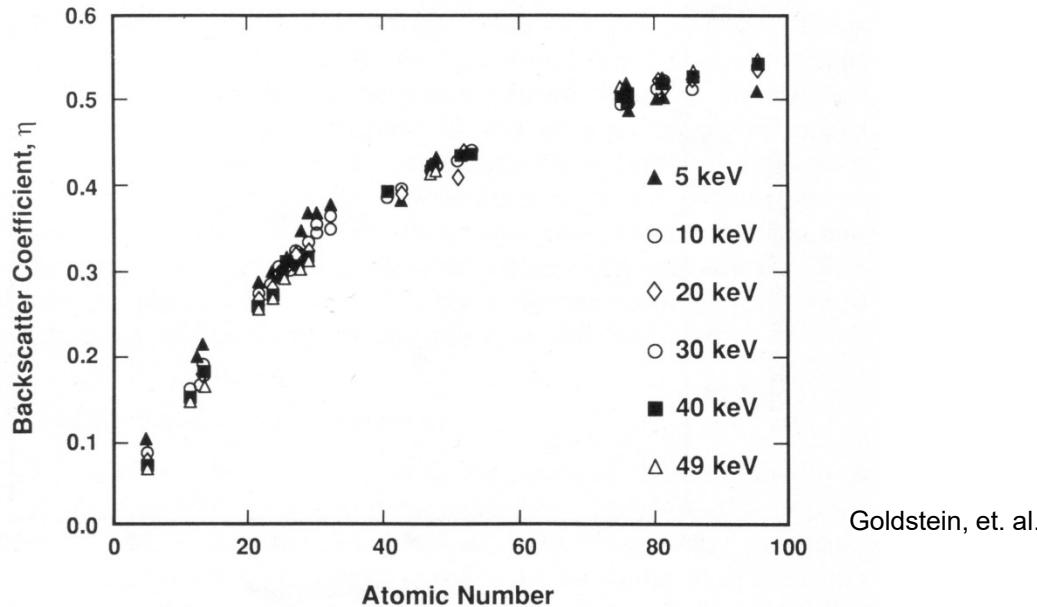
where, i_{BSE} is the backscattered current, and i_b is the beam current

η Dependence on Atomic Number



- There is a general dependence of the backscatter coefficient with increasing atomic number (Z)
- This provides atomic number contrast or Z contrast when using BSEs as the imaging signal
- The slope of η is initially very steep and falls off as Z increases

η Dependence on Beam Energy



- Since the interaction volume increases dramatically with beam energy, it might be reasonable to expect that η would also be a function of beam energy
- This is not found to be the case
- There is a small and not necessarily regular change in η with beam energy, usually less than 10%

Compositional Contrast

- Since η increases with increasing (average) atomic number, the primary contrast mechanism when observing BSEs is **compositional contrast**
- As the average atomic number increases, the number of BSEs increases, which makes high atomic number materials appear to be bright in a BSE image
 - A BSE image will not reveal what materials are there, but will reveal the distribution of high and low atomic number materials
 - If the materials in a sample are already known, reasonable guesses can be made about the distribution
 - It is often desirable to couple this data with X-ray (EDS) data

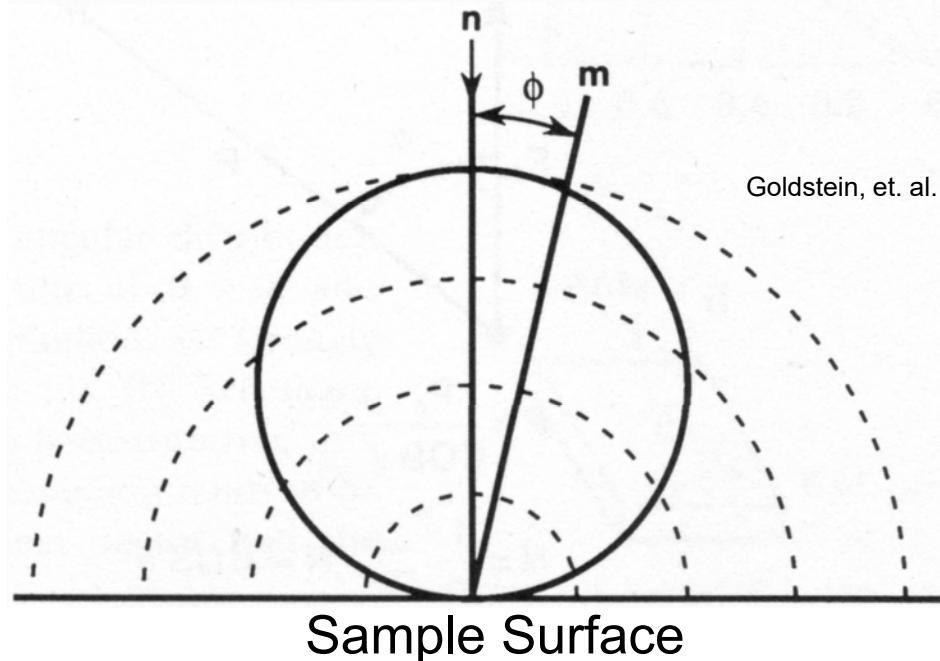
Angular Distribution of η

- For an incident electron beam following the vector n , the angular distribution of backscattered electrons ejected along the vector m is given by:

$$\eta(\Phi) = \eta_n \cos(\Phi)$$

where η_n is the value measured along the vector normal to the surface

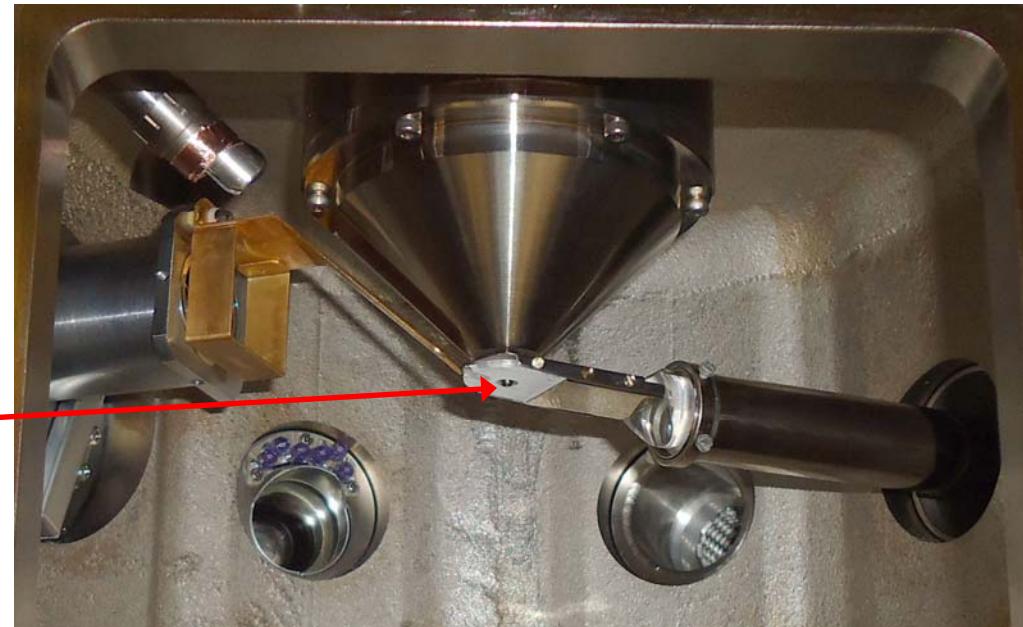
- For 0 tilt, the cosine distribution is angularly symmetric about the vector n
- Note that this suggests that most of the BSEs are scattered back in the direction of the primary beam!
 - This is generally a product of many scattering events → average scatter angle is ~2 degrees



Electron detection – BSEs

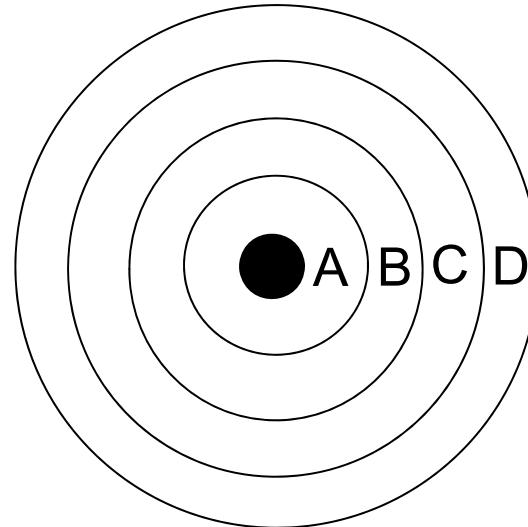
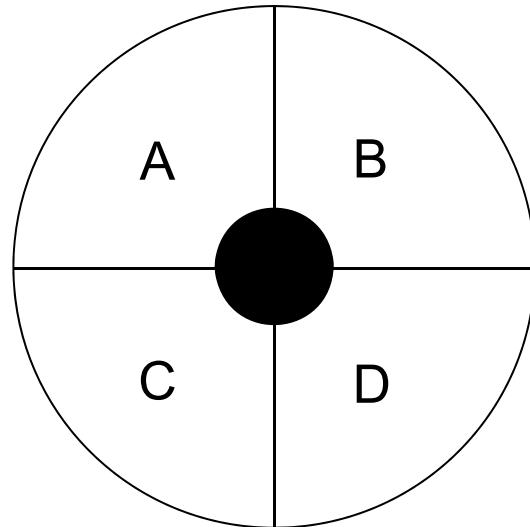
Robinson passive
scintillator backscatter
electron detector

Shown in the inserted
position



- The two most common BSEDs are solid state and passive scintillator
- Solid state detector forms electron-hole pairs when struck by a high energy electron – sweep apart the pairs to generate a current
- The passive scintillator works much like an ET detector with no bias on the scintillator itself (hence, passive – more on the ET detector coming!)

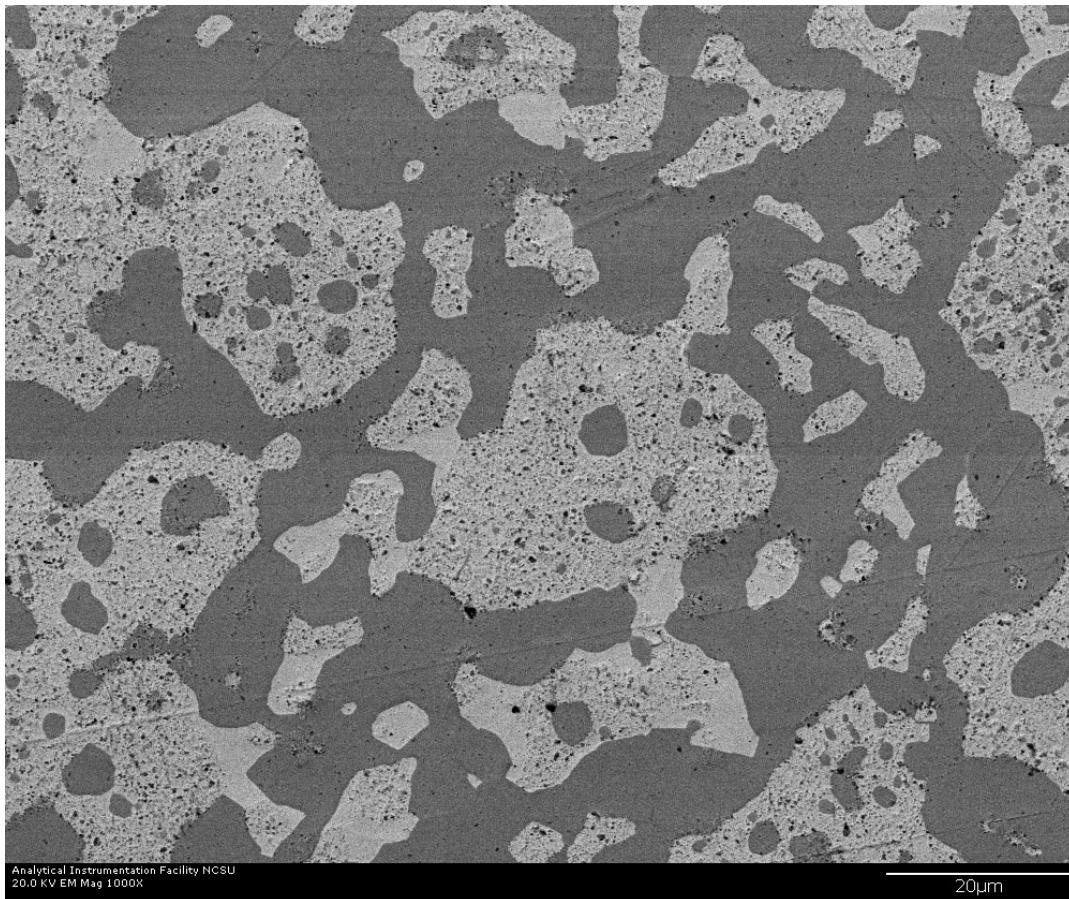
BSE Detector Geometry



Solid State BSED geometry: Four quadrant design on left, Four concentric rings design on right.

- For Compositional contrast, sum signals from all quadrants/rings
 - $A + B + C + D$
- Topography, quad design: Subtract top from bottom or side from side
 - $(A + B) - (C + D)$, plus permutations
- The concentric ring design allows one to observe BSEs with different take-off angles, which can be beneficial for stepped samples

SEM Compositional image

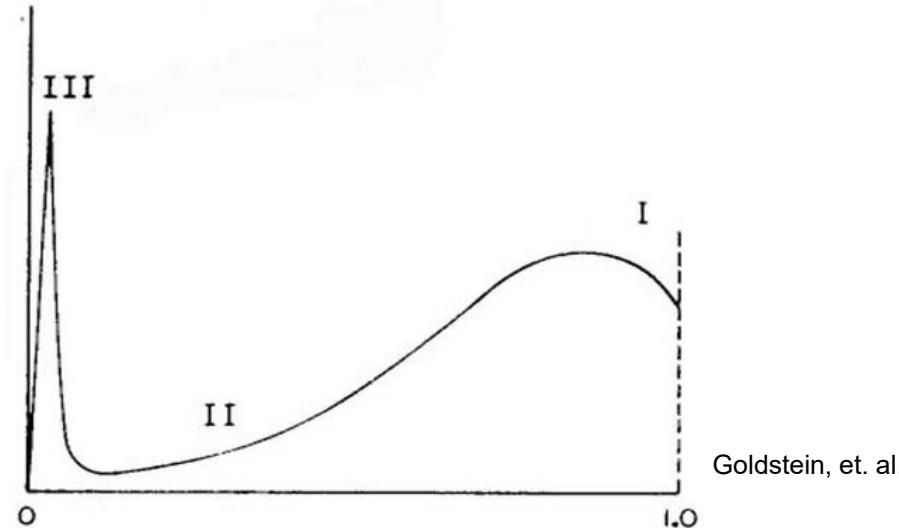


Backscattered SEM image of an PbSn alloy showing contrast based on the atomic number. The brighter areas are Pb-rich. Dark spots are embedded polishing media (SiO₂).

Electron Emission from a Sample

Plot of total electron emission vs. normalized energy

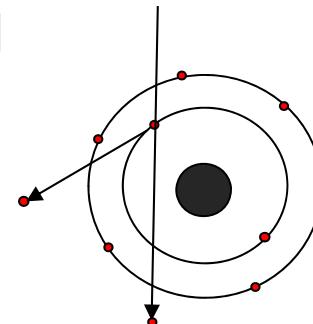
Energy = 1 is the energy of the incident electron beam



- Region I is the high energy BSE peak
 - Most BSEs have an energy close to that of the incident beam
- Region II is the low energy BSE tail
 - Should fall off gently to zero if BSEs were the only emitted electrons
- Region III is SE emission
 - Region is exaggerated for clarity – the peak is very narrow

Secondary Electrons

- Inelastic scattering of the primary incident electron can result in an electron from the sample being ejected

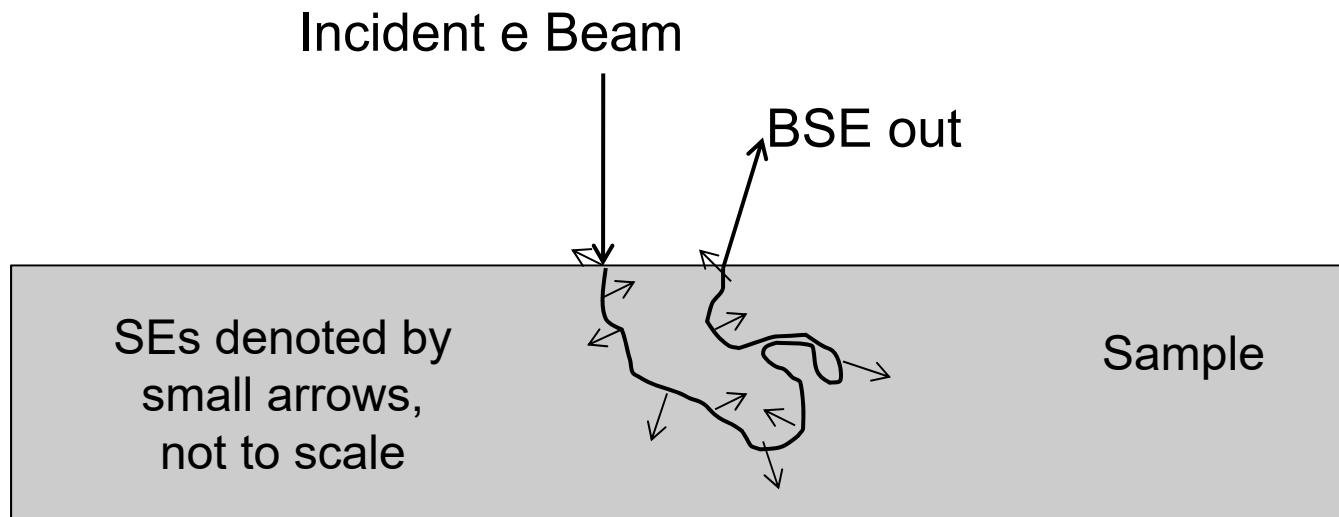


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- Since this is not the primary electron from the beam it is called a **Secondary Electron**
- SEs are **defined** as electrons that escape the sample with < 50 eV of energy
 - Energy peak in the 3-5 eV range
 - This is a definition of convenience
 - Some BSEs have <50eV
 - There are also high energy SEs (it takes a spin resolved detector to tell the difference)

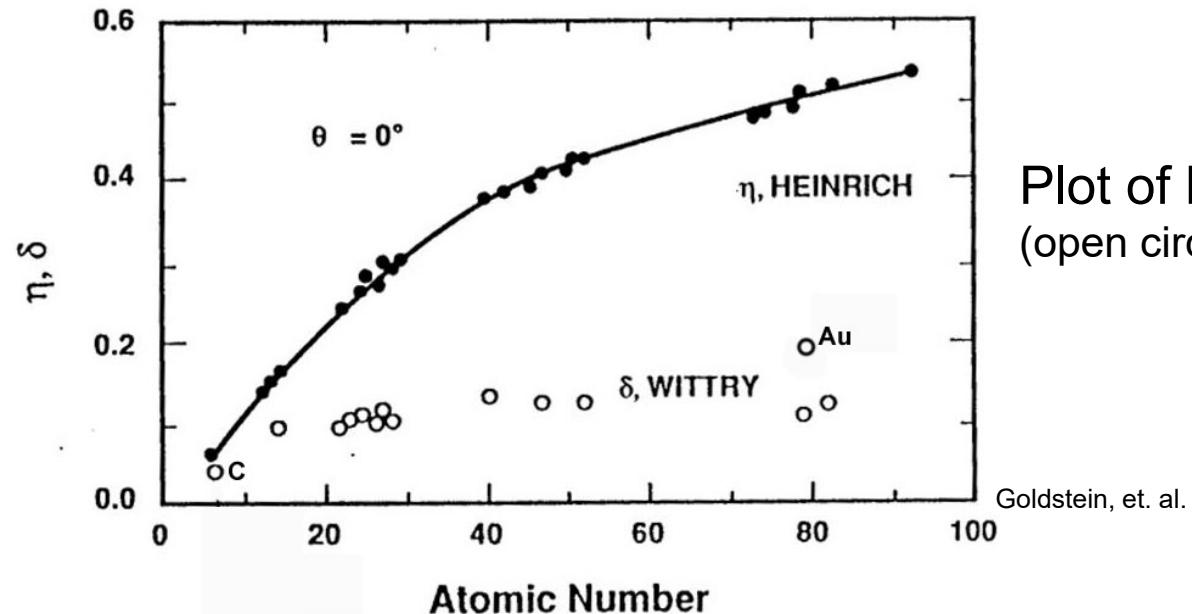
SE Production

- Secondary Electrons are produced all along the path of the incident electron
- Most do not escape from the interior of the sample!
- The secondary electron emission coefficient is generally denoted as δ



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SE emission is Constant with Z



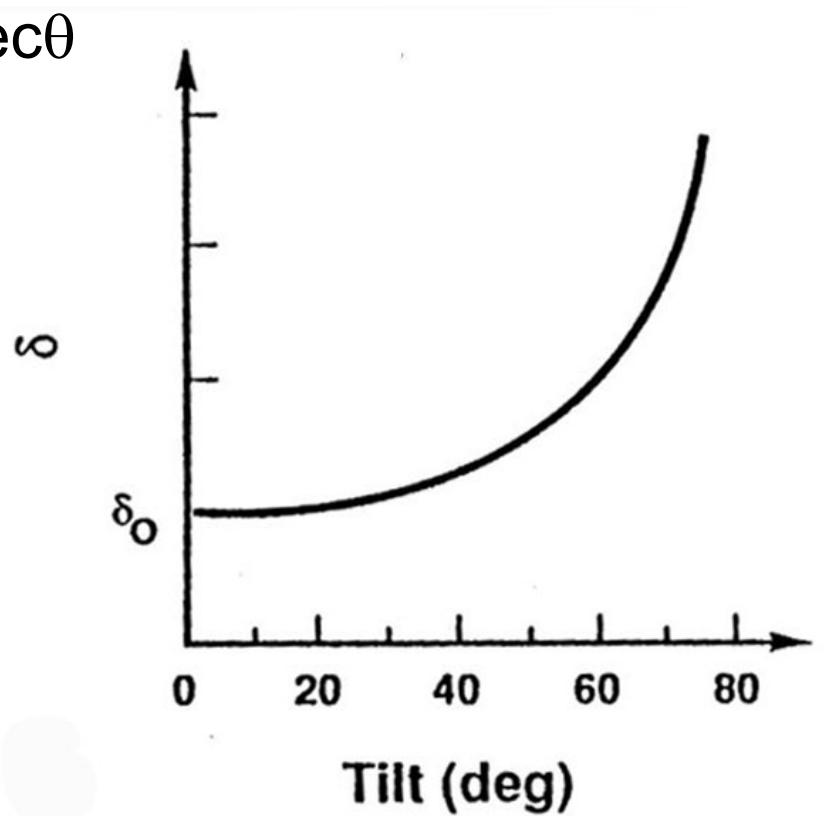
Plot of BSE and SE yield vs. Z
(open circles = SE, solid = BSE)

- Secondary electron yield on a flat polished surface is found to be almost constant when plotted against atomic number
 - This is unlike the Z dependence of BSEs
- Note:
 - C has a particularly low δ, η
 - Au has a particularly high δ, η
 - Au islands on C is best for a resolution standard!

SE Emission Varies with Sample Tilt

- Secondary electron emission is experimentally found to (mostly) follow a secant function as the sample is tilted:

$$\delta(\theta) = \delta_0 \sec\theta$$

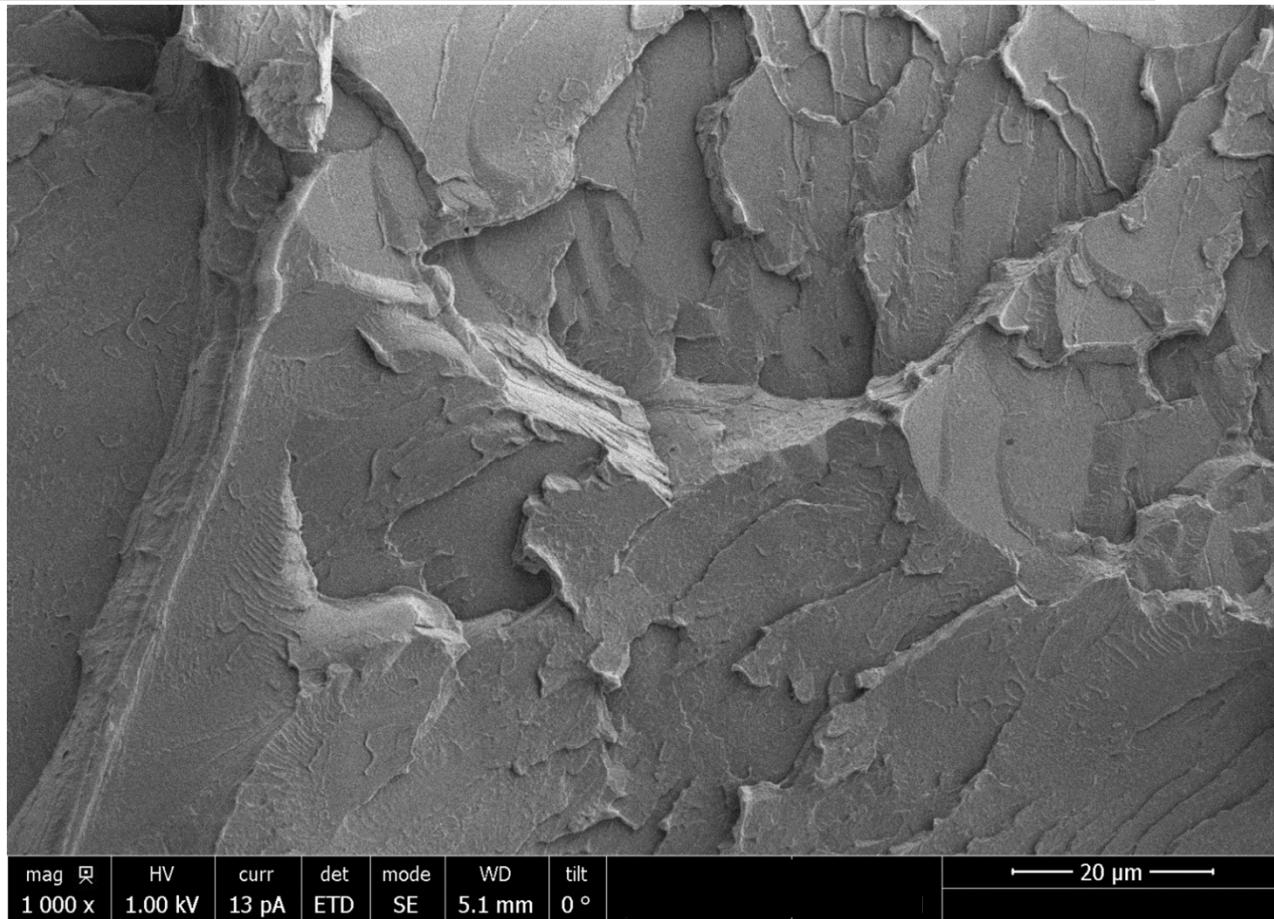


Goldstein, et. al.
from Kanter (1961)

Topographic Contrast

- The most common use of the SEM is to visualize the morphology of a sample
- This visualization relies on topographic contrast
- The SE emission coefficient for samples that are not perfectly flat is a function of the angle of incidence between the beam and the sample surface
- Since the beam can essentially be considered to be parallel over the scanned area, the angle of incidence will change only because of the local sample topography!

Topographic Contrast with an ETD



ETD image of a polymer fracture surface

- Note: ETD appears to be illumination source and perspective is from electron source
- Morphology is easily observed even with no knowledge of SEM

SE Sampling Depth

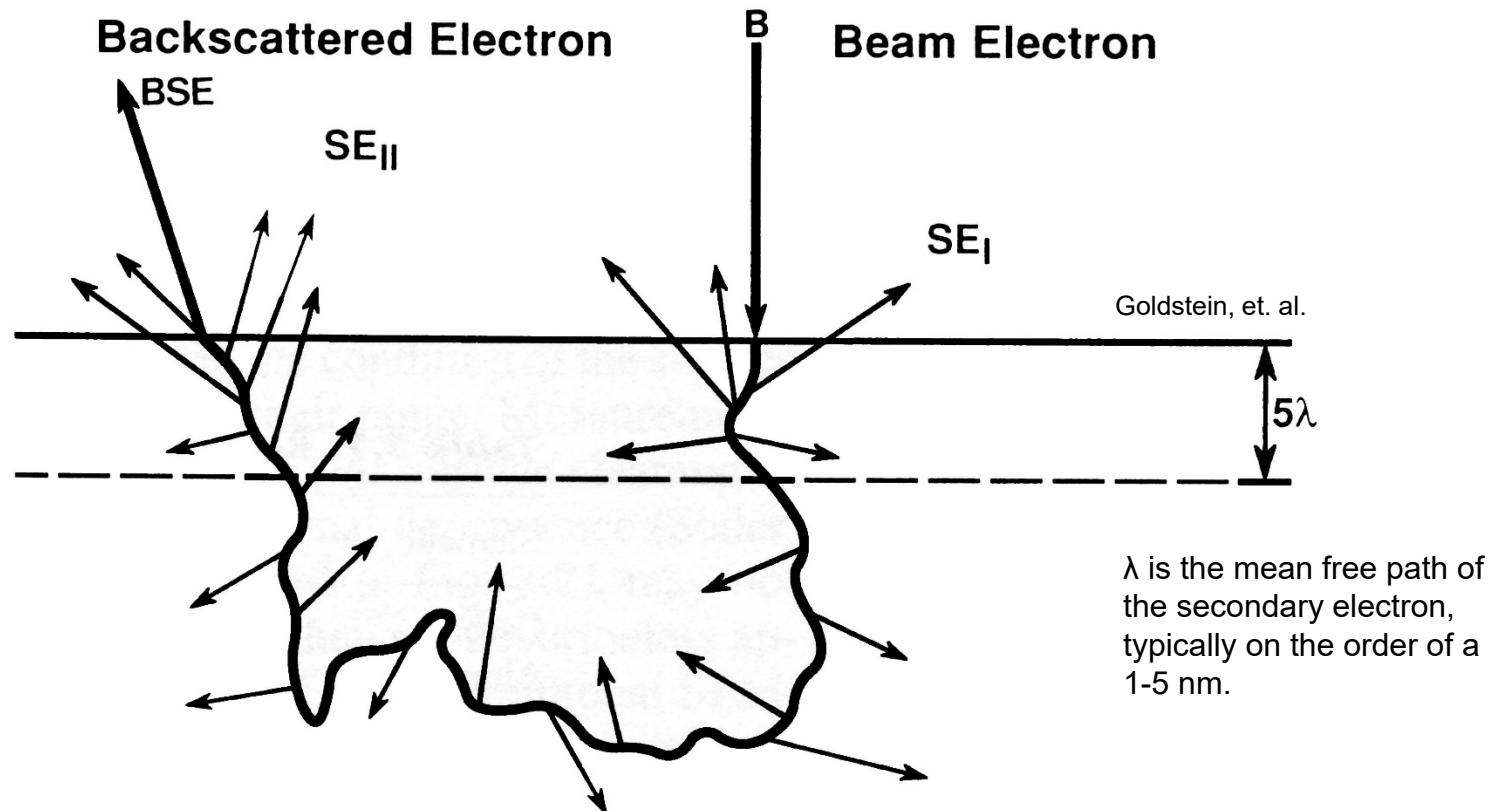
- Secondary electrons have a very shallow sampling depth
 - Maximum escape depth is on the order of 20nm
- The shallow sampling depth is a function of
 - SE's low kinetic energy
 - SEs lose energy quickly in the sample due to inelastic scattering
 - Inelastic scattering has a high cross section for low-energy electrons
- A shallow sampling depth implies that SEs can contain fine detail information about the surface

SE Types

Observable SE's can be formed by:

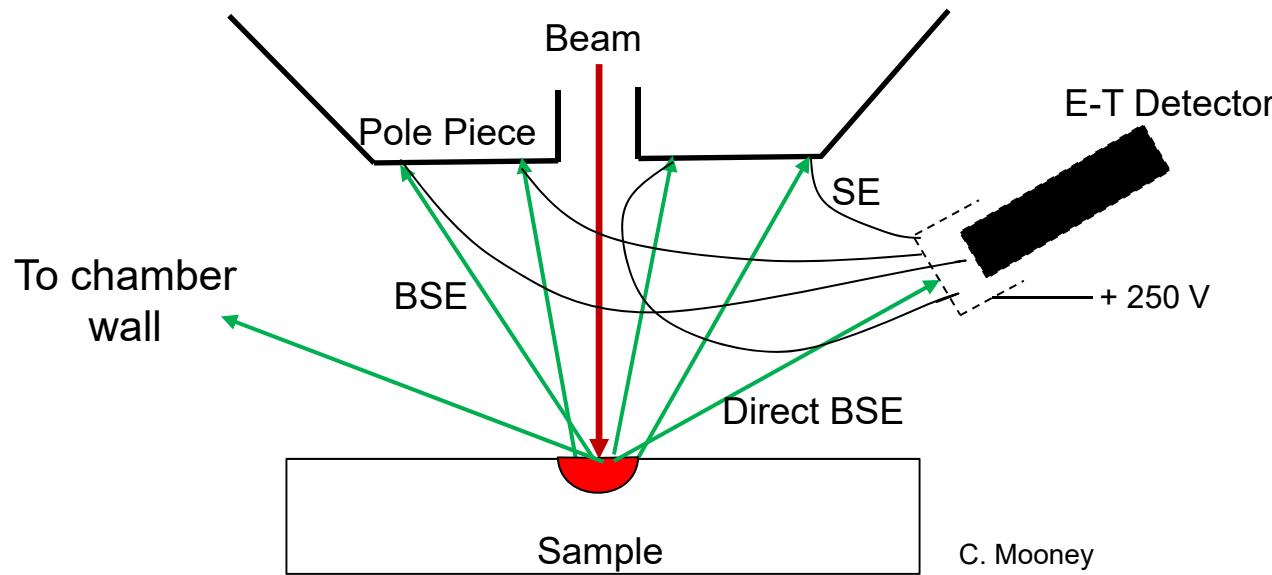
- Incident beam electrons as they enter the sample
 - SE Type I or SEI
- Backscattered electrons as they exit the sample
 - SE Type II or SEII
- Backscattered electrons striking parts inside the chamber
 - SE Type III or SEIII
- Incident electrons striking column parts (aperture, pole piece, etc.) and producing SEs prior to impinging the sample
 - SE Type IV or SEIV
 - These contain no sample information!

Origins of type I and II SEs



Schematic showing origin of type I and II secondary electrons

Source of type III SE

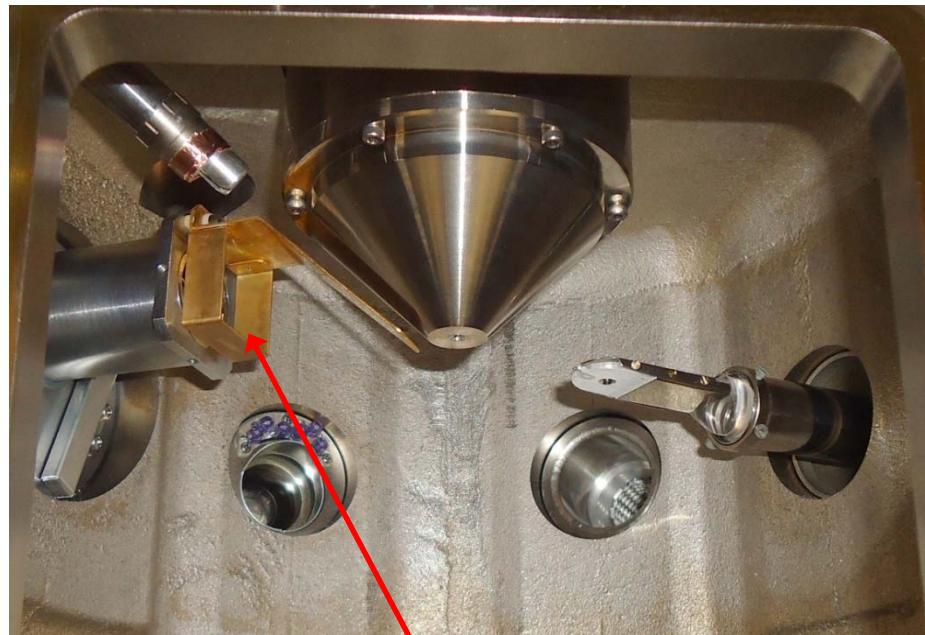


- Type III SEs come from BSEs that are emitted from the sample and strike parts inside the instrument producing SEs that are then detected
- Type III SEs can carry information from the sample since the BSEs that produced them carried information from the sample
- In most SEMs, the pole piece is polished and the rough chamber walls are far away at shallow angles (few SE IIIs)

Electron Detection – Secondary Electrons

Everhart-Thornley detector is the most common SE detector

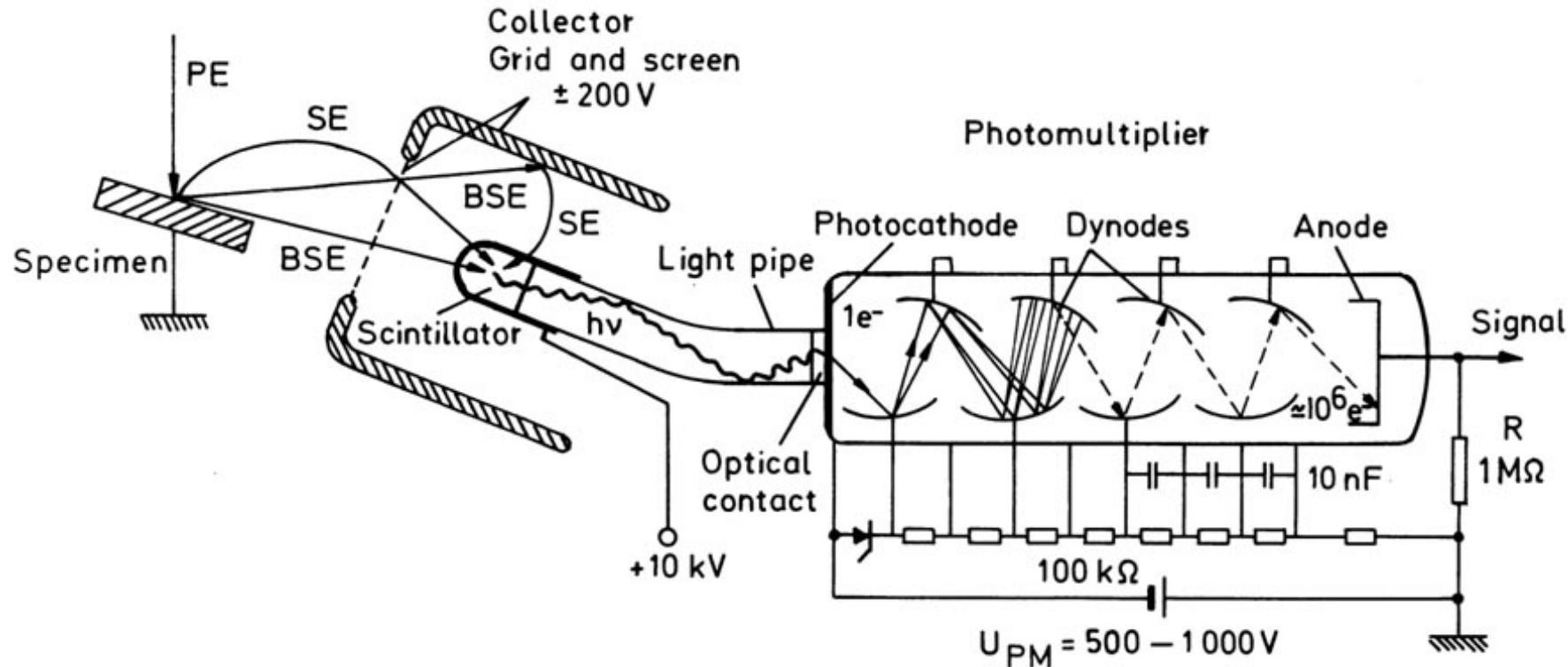
ET detector amplifies the signal by converting electrons to photons and then amplifying (typically by about 10^6) the photon signal with a photomultiplier tube



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ET Secondary Electron Detector

Everhart-Thornley Detector



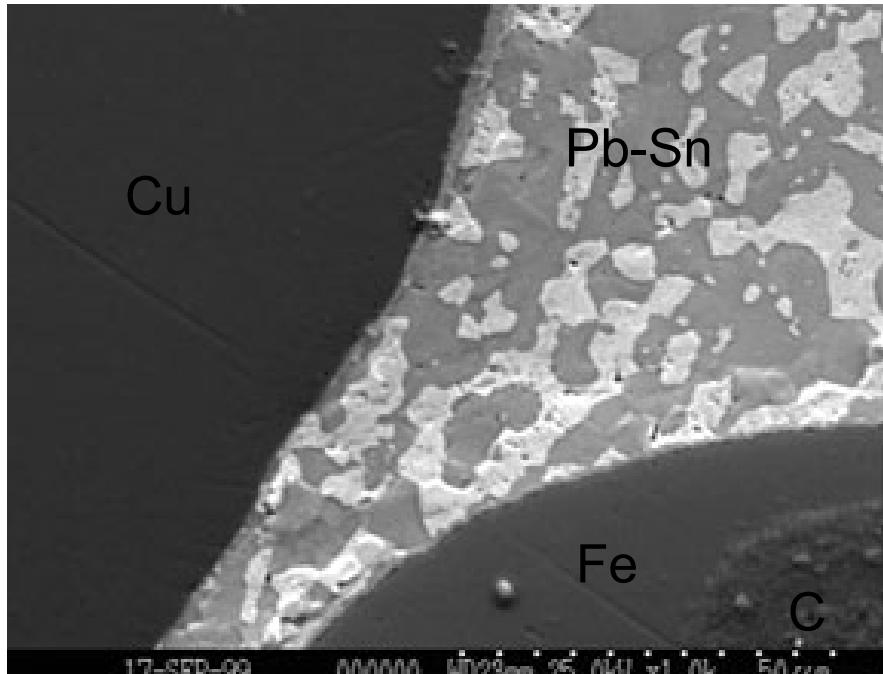
- Collector screen is typically biased to +200V
 - Attracts low energy electrons
- Scintillator is typically biased to +10kV
 - Accelerates the low energy electrons to enough energy to fire a photon
- Photon enters photomultiplier tube where it is amplified by a factor of $\sim 10^6$

Goldstein, et. al.

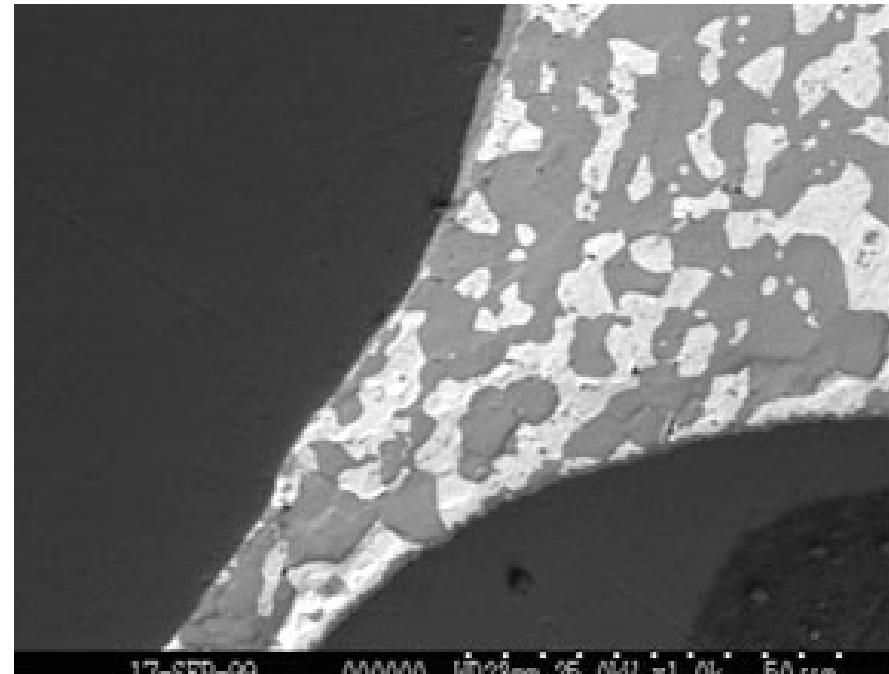
SEM Image Interpretation

- SE images are generally easy to interpret
 - Topographical features stand out in SE images
 - Human eyes/brains know how to interpret the images into topographical features
 - SE images will often look like grayscale visible light images – similar to black and white photography!
 - Artists are known to false color SE images...(see MicroAngela)
 - Note: SE detectors do collect a component of the BSE signal and will have some compositional contrast
- BSE images show compositional contrast with a directional component
 - Considerable topography can be observed in BSE images
 - Similar to a photograph taken with long shadows
 - Bright features point toward the BSE detector

SE and BSE images



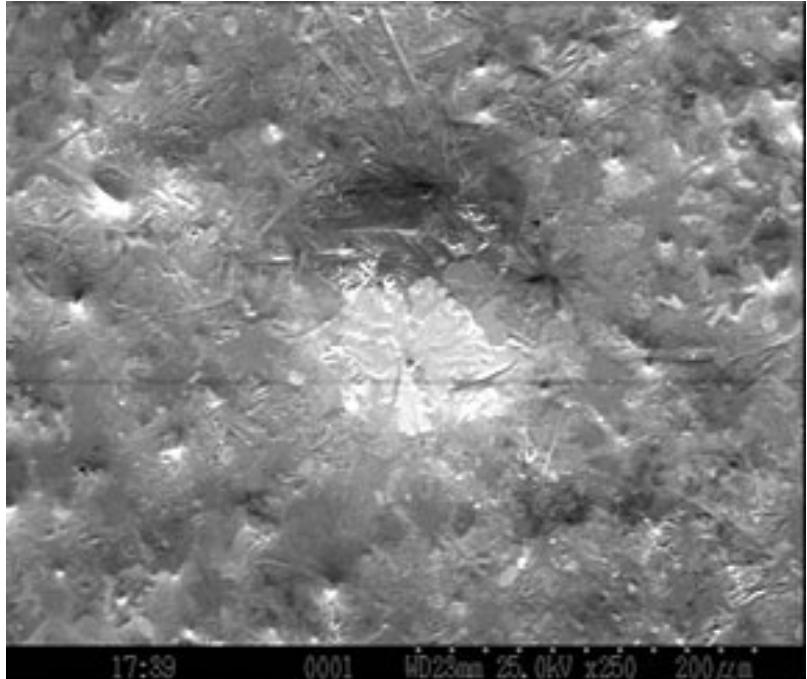
“SE” image of PbSn solder (with some
BSE content due to use of ET
detector)



BSE image of PbSn solder

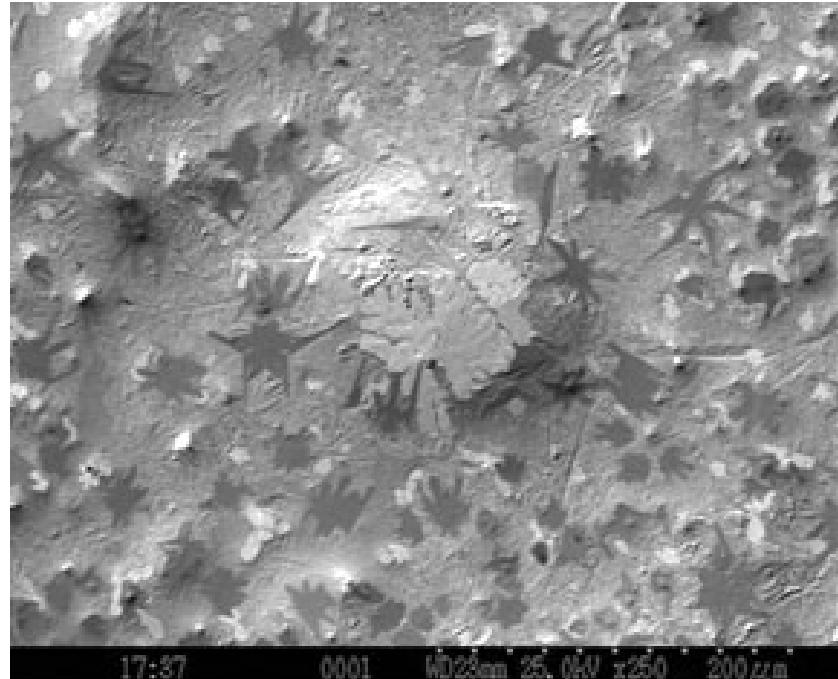
Notice that in the SE image that there is clear topographic contrast while the BSE image appears more flat. The BSE image will have a topographic component due to sample-detector geometry.

SE and BSE Images of Pb/Sn/Ag



R.Garcia

Secondary Electron Image,
Intensity is proportional to SE's
detected (with a bit of BSE mixed
in) leading to topographic contrast



Backscattered Electron Image.
Intensity displayed is proportional
to BSE's detected, i.e., Atomic
Number contrast. Ag is the
lightest element and is visible as
dark star-like structures

SEM Image Interpretation – SE images

- SE images show topographic contrast with a compositional component
 - Topographical contrast is due to the change in SE emission because of the angle between the beam and local sample topography
 - Compositional component is due direct BSEs striking the detector and minor variations in SE emission with material
- SE images are generally easy to interpret
- Topographical features stand out in SE images
 - Human eyes and brains know how to interpret the images into topographical features
- SE images will often look like grayscale visible light images – similar to black and white photography
 - Artists are known to false color SE images (Google MicroAngela)
- Note: SE detectors do collect a component of the BSE signal and will have some compositional contrast

SEM Image Interpretation – BSE images

- BSE images show compositional contrast with a directional component
 - Compositional contrast is due to backscatter cross-section increasing with increasing atomic number
 - Topographical component is due to BSEs being emitted toward and away from the detector – BSE detectors are line of sight only
 - Shadows indicate areas where the sample is pointing away from the BSE detector
- Considerable topography can be observed in BSE images
 - Similar to a photograph taken with long shadows
 - Bright features point toward the BSE detector
 - Some solid state detectors are divided into sections which can be added or subtracted from one another to enhance topographical contrast

Applied Sample Bias

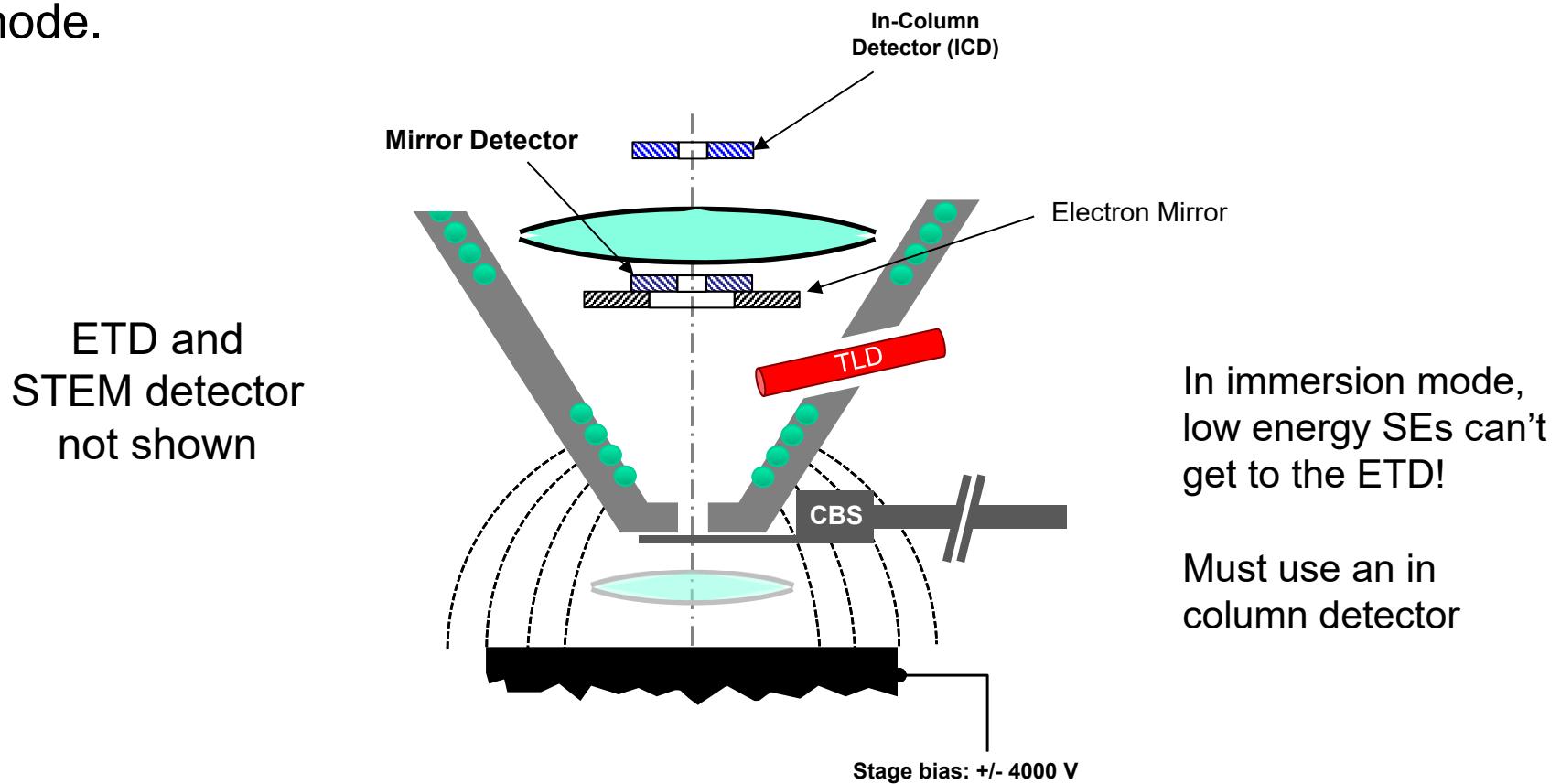
- Some SEMs allow a negative voltage to be applied to the sample
- This decelerates electrons before they strike the sample
 - Can help with charging
- A negative voltage will accelerate any electrons that leave the sample
 - Also forces those electrons closer to the optic axis of the column
- High enough bias can accelerate a SE to the point that it can be detected by the BSED
 - The SE has no compositional contrast
- Be careful when interpreting images when a high bias is applied to a sample!

In Column Detectors

- It is possible to place an electron detector in the column
- Under some conditions, it is possible to force SEs back up the optic axis of the column where it can be collected by a detector inside the electron-optical column
 - When using in-lens or immersion lens SEMs
 - If a high negative bias is applied to the sample
- Detectors in the column can be a low energy or high energy electron detectors
 - Low energy detectors pick up SEs
 - High energy detectors pick up BSEs and accelerated SEs
 - If a high enough negative bias is applied to the sample, an SE can be accelerated to enough energy to be detected by a BSED
 - Recall: An accelerated SE does not contain compositional information!

Verios 460L Electron Detectors

Verios 460L is a fairly typical Ultra-high resolution SEM equipped with several in column detectors and the ability to bias the stage. The Through the Lens detector (TLD) is the SE detector used in immersion mode.



Next: the Lab! (Almost)

- Almost time to virtually sit in front of an SEM and start learning how to make nice images
- First, we shall take a look at general SEM operation and the specific instrument we will be using and find out what to expect
- Once we are in the lab, instrument operation will be demonstrated and then each student will drive the microscope

SEM Operation

- All SEMs work pretty much the same way
- Your favorite deity is in the details!
- That is, all SEMs have similar controls, but the graphical user interface (GUI), the alignment procedure, and sample loading will vary from one instrument to the next, even within the same manufacturer!
- That said, an SEM is an SEM is an SEM
 - If you can drive one, all you need to do to move to another is learn the GUI and alignment procedures
 - Also have to learn how to load the sample
 - Every instrument I have ever used has a height gauge for the sample
 - It is bad form to break the microscope while loading the sample!

SEM Operation

In a nutshell:

- Load the sample and wait until the appropriate vacuum level is achieved
- Choose operating parameters, i.e. accelerating voltage, beam current, aperture size, and working distance
- Align the electron-optical column
 - Start at the electron gun and work toward the sample
- Perform final focus and astigmatism adjustments
- Set signal offset and gain (brightness and contrast)
- Take a pretty picture!
 - Or, collect spectral information

SEM Electron Optical Alignment

It is critical to understand how to align the electron optical column in order to get the best results with any SEM

In general, the SEM electron-optical column will need to be aligned at the start of each session. The instrument may already be aligned, but it is always a good idea to check and correct any issues that are found.

General Procedure: Start at the electron gun and work toward the sample, aligning the optics along the way.

Basic SEM Alignment

There are typically two basic types of alignments that are performed depending upon what the user observes in the GUI:

1. Turn low signal images into high signal images
 - Make the image as bright as possible with the alignment control at hand
 - Examples include aligning the electron gun and coarse alignment of the objective aperture
 - In some SEMs this is done by placing a bright spot on an alignment mark (FEI, now ThermoFisher)
2. Make shifting images stop moving
 - If the operator observes that the GUI starts wobbling the image, i.e., taking the image in and out of focus, then the goal is to adjust the system to stop the image from shifting
 - Examples include fine alignment of the objective aperture and astigmatism coil alignment.



Break

Lab starts in 60 minutes!

Virtual SEM Short Course

Afternoon Lecture

Beam Parameters, in Theory

Note: In theory there is no difference between theory and practice...

There is a very limited list of theoretical electron beam parameters

1. Energy (how fast are the e^- going)
2. Current (how many e^- strike the sample per unit time)
3. Spot size (e^- illumination area on the sample)
4. Convergence Angle (how fast does the beam converge)

A more detailed discussion of theoretical beam parameters is beyond the scope of this short introduction – there are a large number of texts from which this information can be learned, including the reference at the end.

Beam Parameters, in Practice

In practice, we have the following controls that allow the operator to set the electron beam parameters

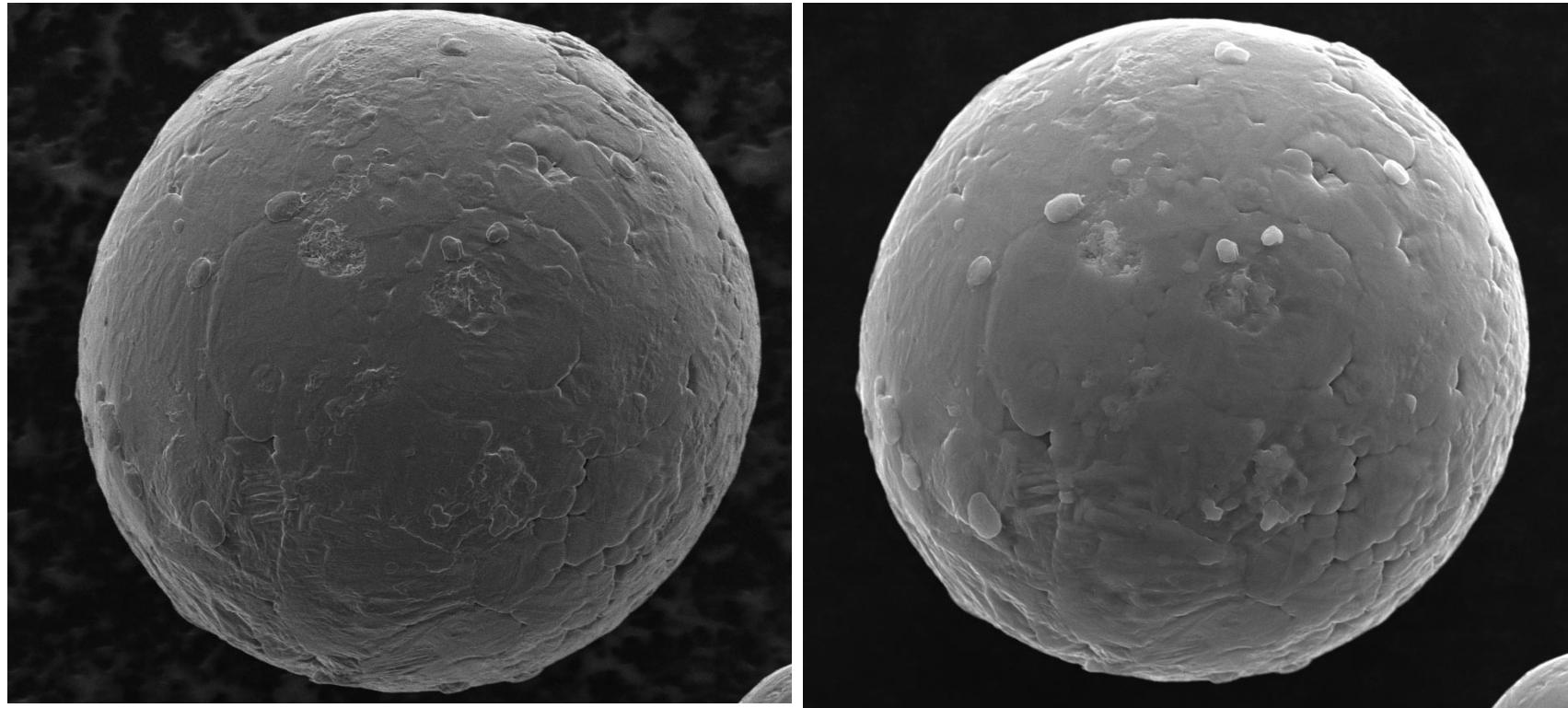
1. Accelerating Voltage
2. Condenser Lens excitation
3. Objective Aperture Size
4. Working Distance (focal length of objective lens)

Understanding how these parameters control the beam at the sample surface is critical to extracting all the performance possible from an SEM

Accelerating voltage

- Accelerating Voltage ↔ Beam Energy (1 – 30 kV typical)
 - Voltage is kV, energy is keV (please do not confuse energy and potential)
 - A 20 kV accelerating voltage will produce a beam with 20 keV electrons
- As seen in the Monte Carlo interaction volume simulations, as the beam energy increases, the depth of penetration into the sample increases
- High energy electrons (20 keV +)
 - Deep penetrating (20 kV beam → ~6 um in C)
 - Can observe sub-surface objects, lose surface detail
- Low energy electrons (5 keV or less)
 - Shallow penetration, interactions close to the surface
 - Can observe more surface detail
- X-ray analysis: Beam energy 3X X-ray energy maximizes X-ray production efficiency
 - Start with 20 kV for analyzing unknowns

Beam Energy Effect: Conventional SEM



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- Ti-6Al-4V alloy sphere used in industrial 3D printing of metal parts
- 5kV image (Champion Data) on left, 30kV image (nice) on right
- Note the increase in surface detail in the 5kV image
- Note edge effects in the 30kV image
- SE image: a 20 keV BSE image could show composition distribution

Beam Energy Effect: High Resolution SEM

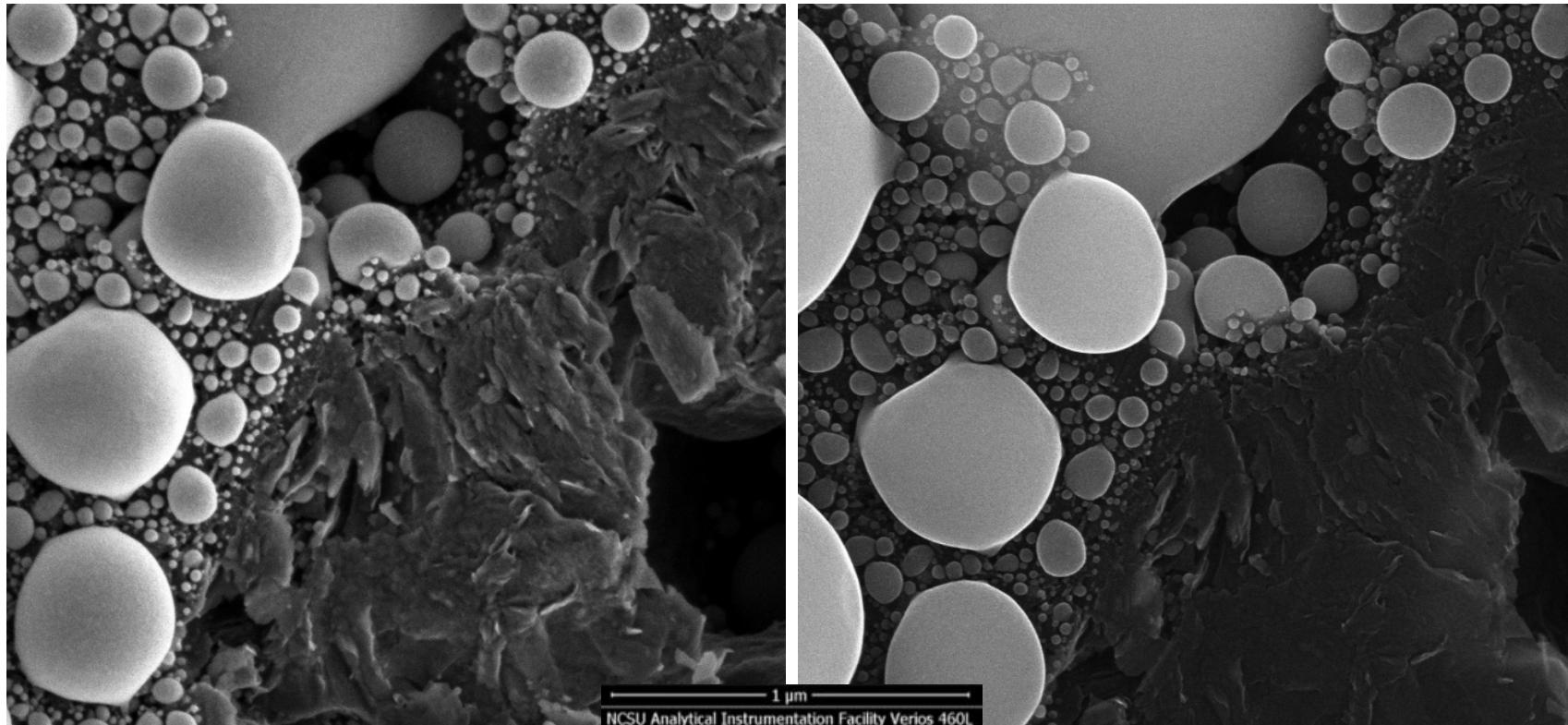
Theory:

- As the accelerating voltage increases, the wavelength of the electrons decrease
 - Smaller wavelength means higher potential resolution
 - It is also easier to control high energy electrons in magnetic field
- In general, higher accelerating voltages are used for high resolution applications in a conventional SEM

Practice:

- In practice, observed resolution at 5 keV is usually about the same as at 20 keV with a modern conventional SEM
- Modern UHR-SEMs make very high resolution with very low energy
- At low voltages (5 kV or less) more surface detail will be visible
- Resolution in a modern UHR-SEM is not spot size limited but interaction volume limited, i.e., very small spot size with a low voltage

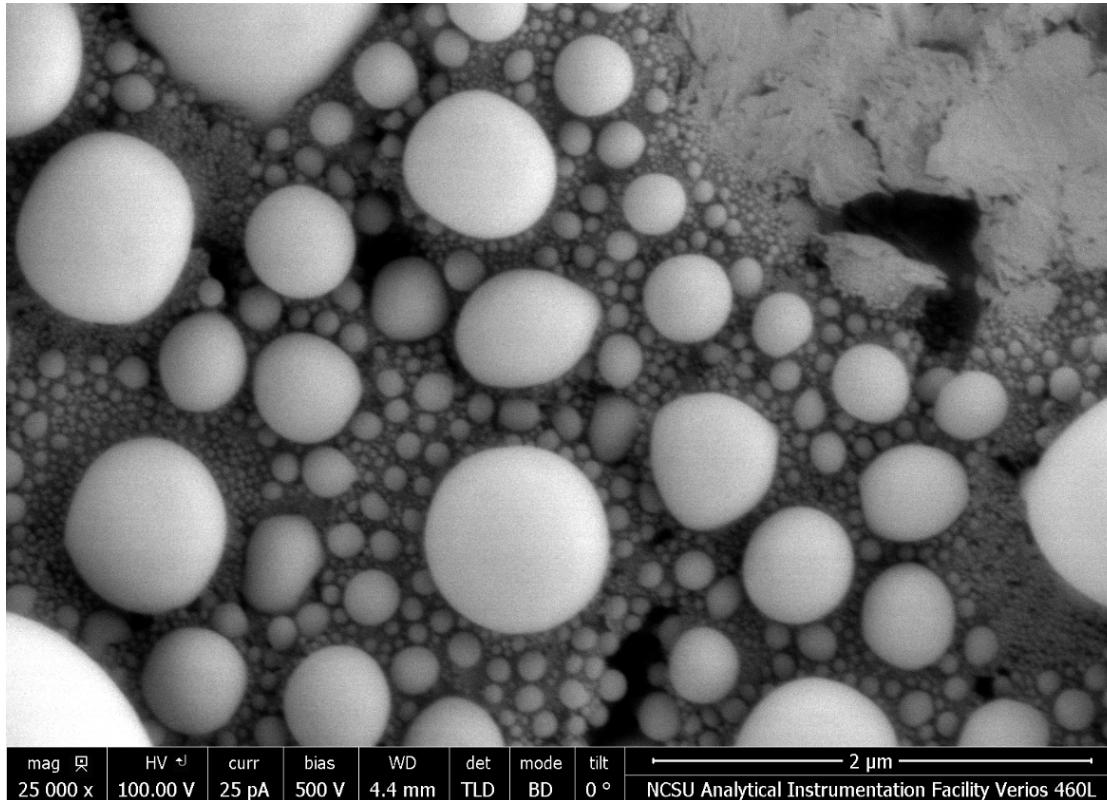
Beam Energy Effect: UHR-SEM



1 kV on left (Champion Data), 20 kV on right. 50 kX. Other conditions: 25 pA beam current, WD = 4.2 mm, upper corrector and stage bias (500V) for 1 kV image. Data collected from a modern UHR-SEM.

- Note the increase in surface detail with no loss in resolution in the 1 kV image
- A modern UHR-SEM is capable of very high resolution at very low voltages (by SEM standards)

Low Beam Energy – 100 eV



100 eV!

Champion Data

Sample:
Sn spheres
on C

C.Mooney

mag	HV	curr	bias	WD	det	mode	tilt	2 μm
25 000 x	100.00 V	25 pA	500 V	4.4 mm	TLD	BD	0 °	NCSU Analytical Instrumentation Facility Verios 460L

At very low energies, the interaction volume is so small that there is very little compositional contrast – the BSE energy can be in the same range as SE energies and the contrast between high and low atomic number materials will decrease

SEM Rules: Beam Energy

- High energy electrons (20 keV +)
 - Deep penetrating
 - Less surface detail (less interaction close to the surface)
 - Can observe sub-surface features
 - Some samples look better at high voltage, e.g., etched grain structure in metals
 - X-ray analysis – it takes energy to produce X-rays, choose beam energy to be 2 – 3 times the highest X-ray energy one desires to measure
- Low energy electrons (5 keV or less)
 - Reduces charging, especially below 2 keV
 - Shallow penetration, interactions close to the surface
 - Can observe more surface detail
 - Less beam damage
 - Resolution falls off as the beam energy is decreased
- Mid-range Energies (6 keV – 19 keV)
 - Need a compelling reason... use your brain
 - EBSD: 10 keV gives good signal and minimizes interaction volume

Practical SEM: Beam Energy

Conventional SEM, unless there is a compelling reason to do otherwise:

- 20 keV for high energy imaging and X-ray analysis
- 5 keV for low energy imaging (surface detail maximized)
- The fact that these two choices work for 90+ % of all samples doesn't mean that there aren't 10 % that need something different

UHR-SEM, unless there is a compelling reason to do otherwise:

- 20 keV for high energy imaging and X-ray analysis
- 2 keV for low energy imaging
 - 2 keV gives more surface detail than 5 and still looks like a traditional SEM image
 - Low energies can make for funny looking images with odd contrast
- 500 eV (+/-) is for insulators without a coating
- Experiment! Try different things to see what works the best!

Condenser Lens

- The more highly excited the condenser lens, the lower the beam current and the smaller the spot size
 - Recall, control is usually labeled Beam Current
 - Usually, a dimensionless number – measure with a Farady cup
- High resolution imaging requires a small spot size
 - To improve resolution, reduce the beam current
- Low magnification images usually look better with a larger spot size
 - High currents increase signal and reduce data collection times
- X-ray analysis (EDS) generally works better with a relatively high current
 - EDS resolution is a function of beam energy and not spot size (recall Monte Carlo simulations) so having a small spot is not advantageous for collecting X-rays

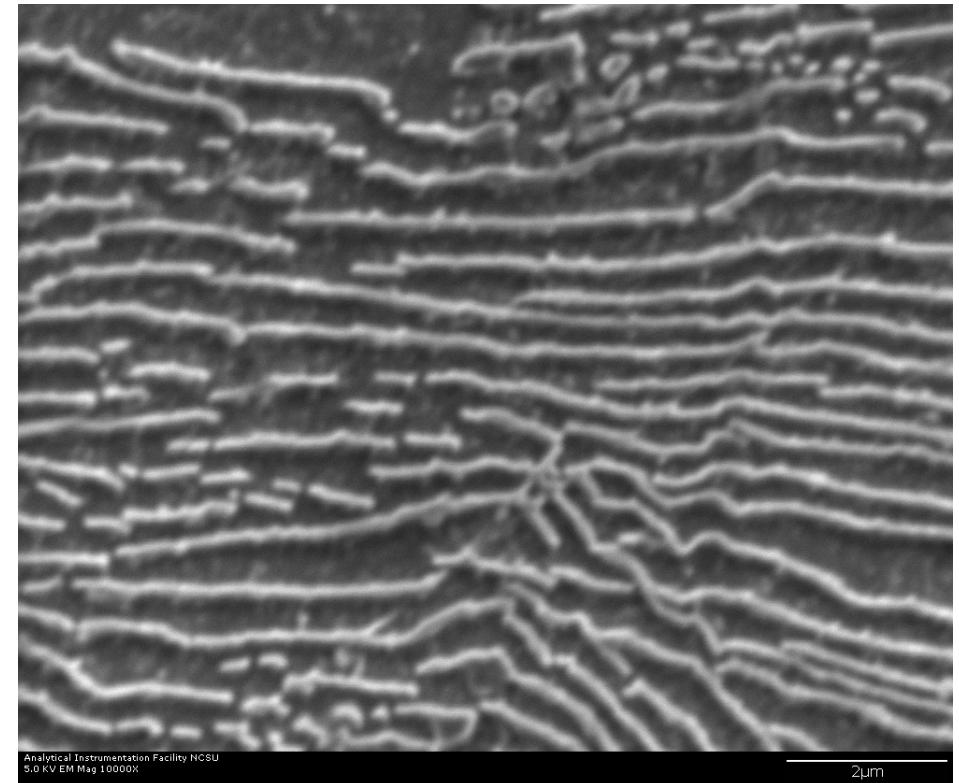
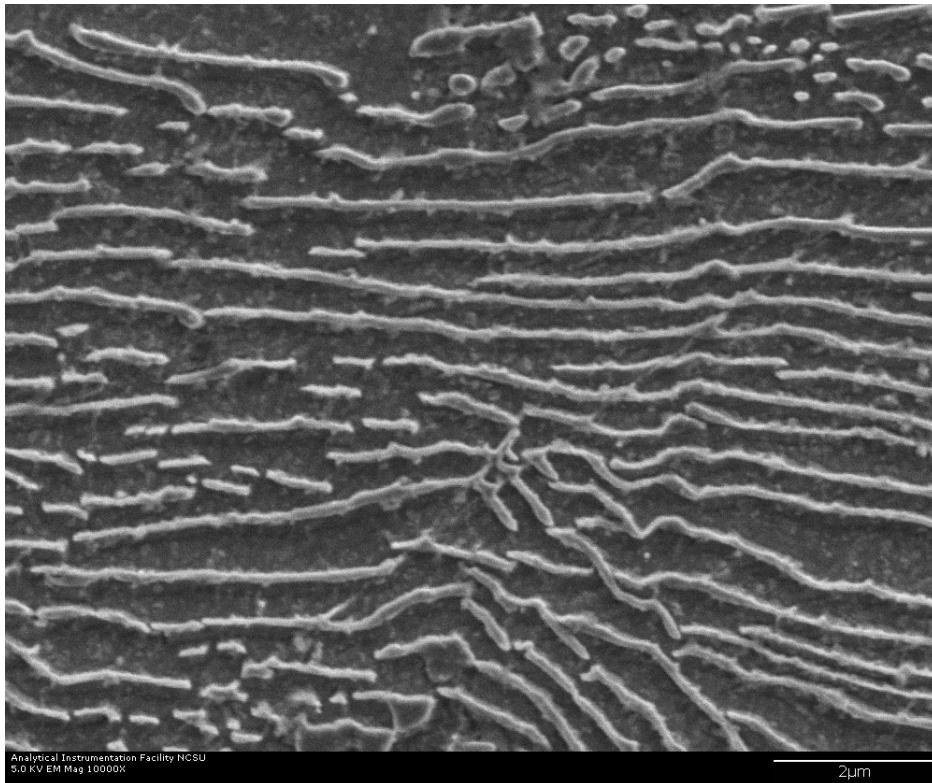
Practical SEM: Beam Current

- The control for the condenser lens is usually labeled Beam Current or simply Current
- To make the potential resolution better, choose a smaller beam current
 - **SEM rule: smaller current => smaller spot size**
- Often the beam current control is a dimensionless number that is related to the excitation of the condenser lens
 - To know the beam current, use a Faraday cup and a current meter
 - Faraday cup is a type of electron trap that allows for accurate beam current measurements at the sample
- Some instruments have a current setting with a dimension in amps
 - These instruments use a combination of the CL and an objective aperture to control the current

Resolution: Beam Current Effect

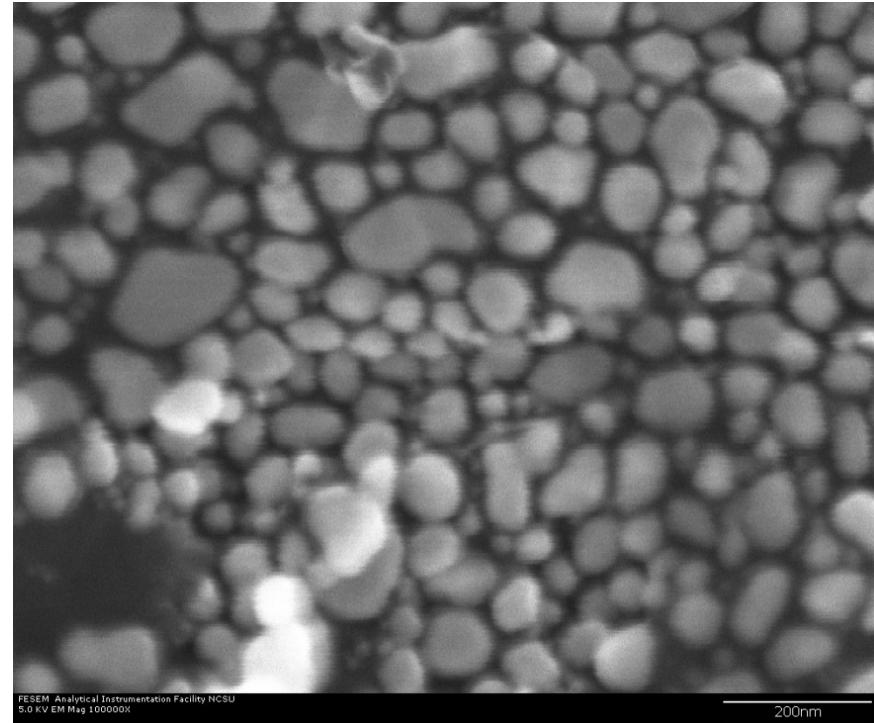
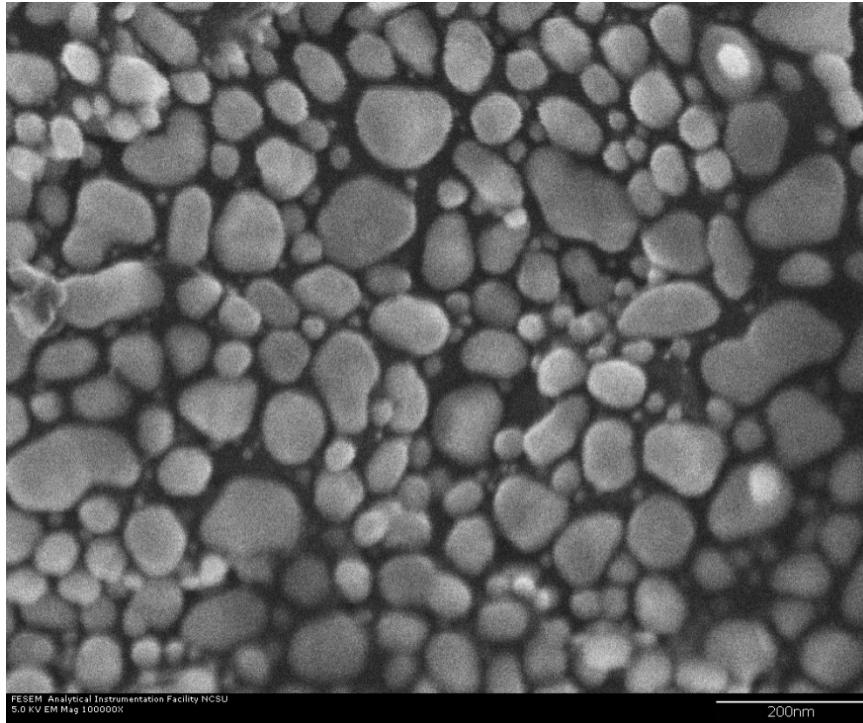
- The condenser lens or beam current is the dominant setting for changing the resolution of the SEM
- Small beam currents = small spot size
 - Small spot size is needed for good resolution

Condenser lens effect on resolution



- Pearlite sample
- All imaging conditions kept constant except for condenser lens
- Image on left: CL 70% excited = low beam current, small spot size
- Image on right: CL 30% excited = high beam current, large spot size

Beam Current Effect: Conventional FESEM



CL = 80% excited on left (Champion Data), CL = 20% excited on right. Other conditions: 20keV beam, WD = 5mm, OA = 30um, 100kX SEM magnification. Data collected from an FE-SEM (JEOL JSM-6400F).

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- The CL does not affect the image as much in the FESEM as it does in the thermionic VPSEM (the initial FESEM source is very small)
- That is, at the 10 kX mag of the previous set of images, this effect would not be very noticeable

Working Distance

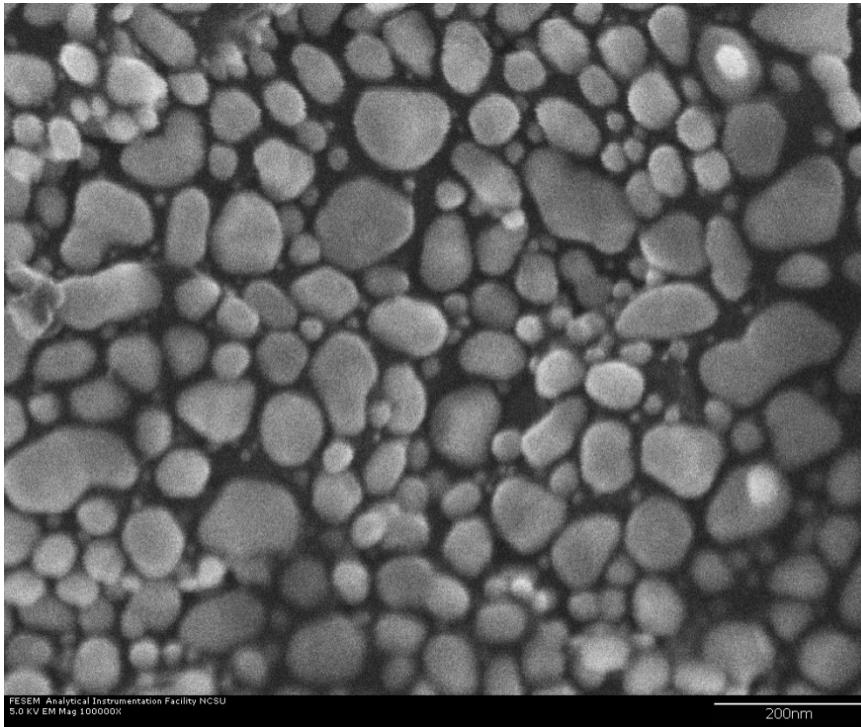
- Electron optical lenses work most efficiently when they are highly excited (convergent and demagnifying)
- This means that for high resolution applications, a highly excited objective lens (short working distance) and a highly excited condenser lens (small spot) will give the best images
- For high depth of field imaging, a long working distance is desirable
- Some detectors may call for a specific working distance for best efficiency (EDS!)
- Typical working distance range is 4 mm to 30 mm
 - When in doubt, pick 10 – 15 mm to start

Practical SEM: Working Distance

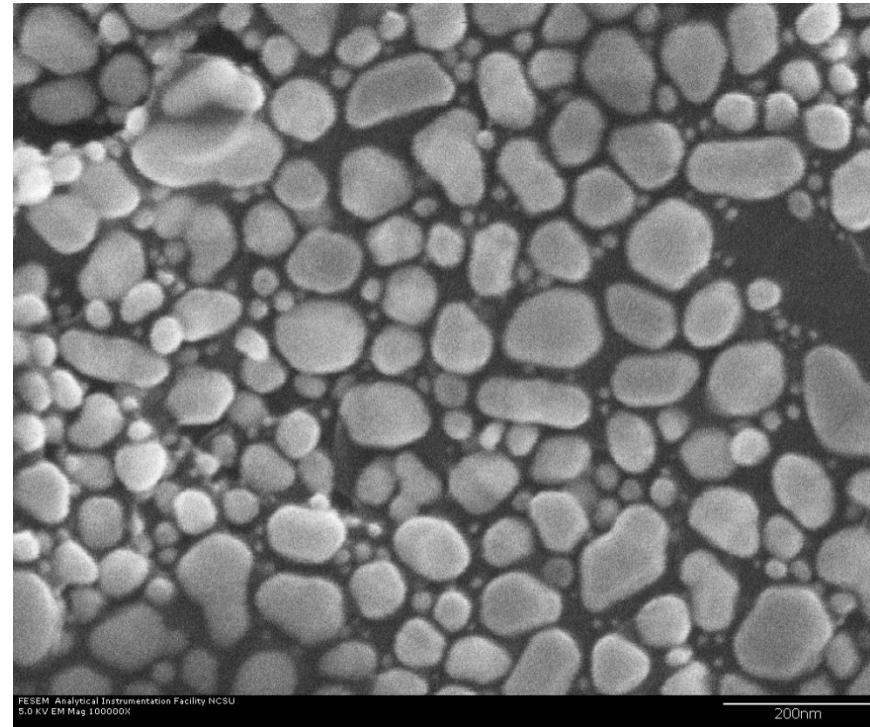
Choose working distance based on what you are trying to observe:

- Short WD for high resolution
 - Conventional SEM: 10 mm or less
 - UHR-SEM: 4 mm or less
- Long WD for high depth of field
 - Conventional SEM: 20 mm or more
 - UHR-SEM: 8 mm or more (max is only about 12, so not much dof)
- Mid-range WD for most routine imaging
 - Conventional SEM: 15 mm (+/-)
 - UHR-SEM: 4 – 6 mm (high resolution mode 7.5 mm max)
- EDS will have a specific WD for best results
 - Varies according to the geometry of the instrument, VPSEM = 15 mm, Verios = 5.2 mm
- If you don't know:
 - Conventional SEM: Choose 15mm, which is also typically the correct WD for EDS!
 - UHR-SEM: 4 – 6 mm WD will typically give good results

WD Effect: Conventional FESEM



FESEM Analytical Instrumentation Facility NCSU
5.0 kV EM Mag 100000X



FESEM Analytical Instrumentation Facility NCSU
5.0 kV EM Mag 100000X

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5mm on left, 20mm on right (both Champion Data). Other conditions: 20keV beam, CL 80% excited, OA = 30um, 100kX SEM magnification. Data collected from JSM-6400F.

- The image on the right is softer due to a larger beam diameter at a longer WD (effect is subtle, 100 kX mag!)
- Note that the short WD image has depth of field issues

Practical SEM: Working Distance

- **Working Distance displayed on the data bar is really the focal length of the objective lens!**
 - Focus control
- **The physical working distance is the distance between the bottom of the pole piece (which houses the objective lens) and the sample!**
 - Z-stage control
- **If the image is in focus, then the focal length of the objective lens is equal to the physical working distance!**
 - True for all microscopes (or telescopes or binoculars or cameras or anything that uses lenses no matter the type of lens) that work in the far field

Practical SEM: Working Distance

- There are two controls for working distance on an SEM:
 - One is labeled “Focus” (look for this on the knobset of most SEMs)
 - The other is the Z-stage control
- When the operator changes the focus control, the current through the objective lens changes, which changes the focal length of the objective lens
- When the operator changes the Z-stage control, the height of the stage changes
- The SEM can be focused using either of these controls
 - Usually done with the focus control (finer control)
- To achieve a specific working distance: Adjust the focus knob (at low mag, focus is scaled with magnification) until the desired WD is displayed in the image data bar, then adjust the Z-stage until the image is in focus

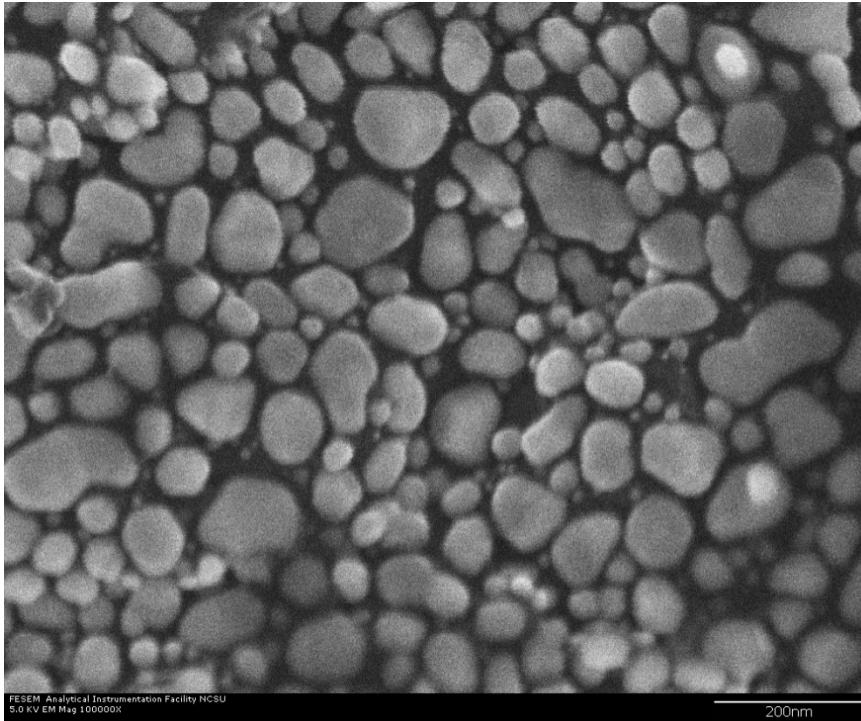
Objective Aperture

- The effect of the OA is generally more subtle than either the beam energy or the condenser lens
 - Good images can be taken with no OA at all!

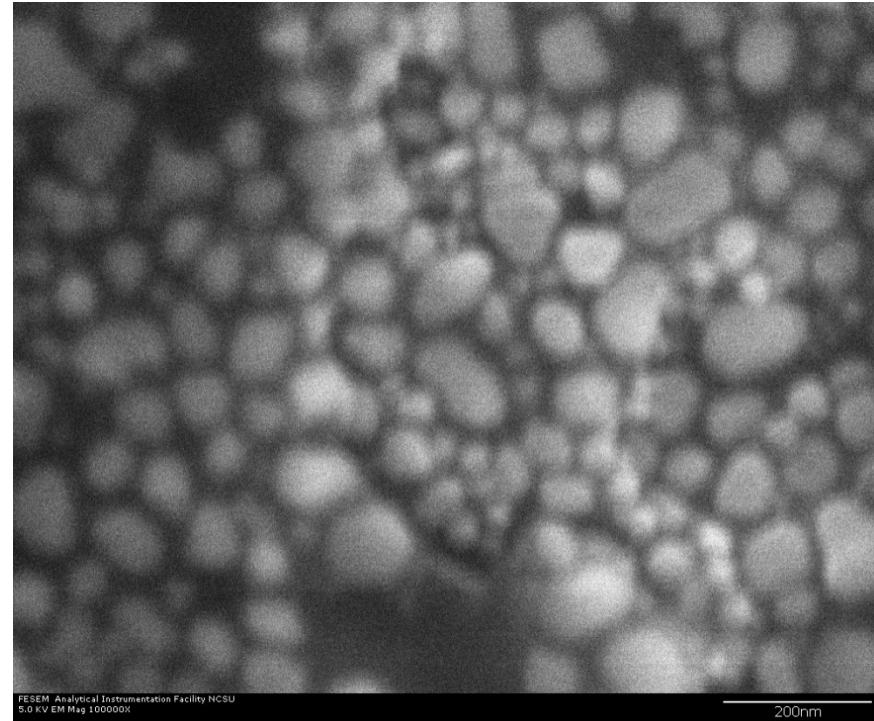
In general:

- Use a small OA for high resolution or high depth of field imaging
- Use a large or no OA for EDS
- Use no OA when changing accelerating voltage or condenser lens

Objective Aperture Effect



FESEM Analytical Instrumentation Facility NCSU
5.0 KV EM Mag 100000X



FESEM Analytical Instrumentation Facility NCSU
5.0 KV EM Mag 100000X

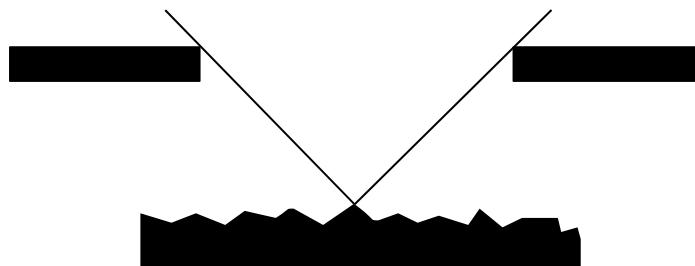
200nm
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30um (Champion Data) on left, 150um on right. Other conditions: 20keV beam, CL 80% excited, WD = 10mm, 100kX SEM magnification. Data collected from JSM-6400F.

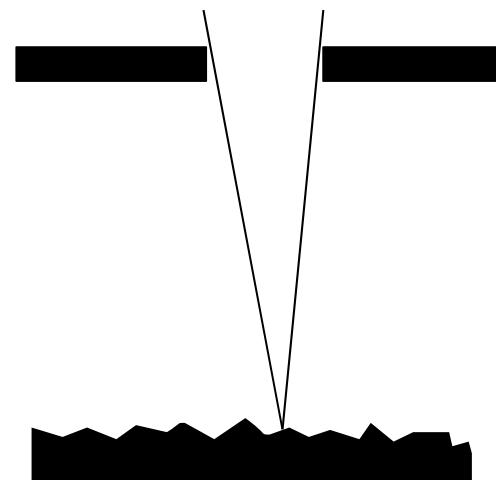
- In a thermionic SEM, the OA effect will not be so great as in the FESEM

Depth of Field

- Depth of Field is the distance over which objects appear to be in focus
- The things that one does for high depth of field are the same for photon and electron optics
- Increasing DOF is achieved through geometric considerations



Low depth of field above
High depth of field to right
(Highly exaggerated)

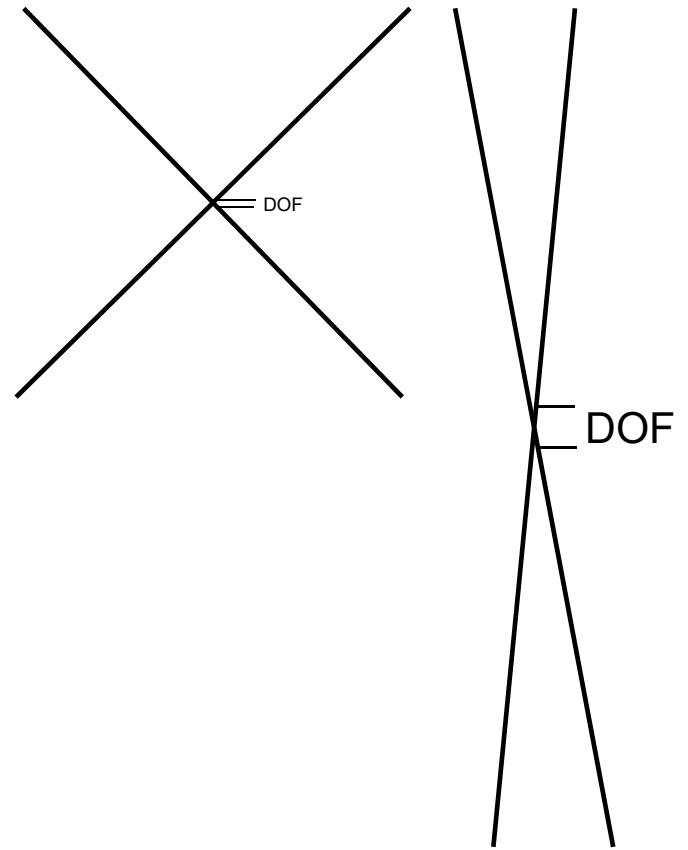


High Depth of Field

- High Depth of Field is achieved decreasing the convergence angle of the beam
- The convergence angle of the beam is decreased by:
 - Decreasing the objective aperture size
 - Increasing the working distance
- The more parallel the beam, the higher the potential depth of field
- **SEM Rule: High Depth of Field requires a long working distance and a small objective aperture**
 - Assuming a selectable OA
 - Maximize WD to maximize depth of field

Disk of Least Confusion

- The disk of least confusion is when the spot is the smallest
 - Aberrations, astigmatism, and focus issues mean the beam is not perfectly focused to a infinitesimally small spot
- High depth of field conditions will extend the disk of least confusion as far as possible in Z-space
- Low magnification increases the disk of least confusion in Z-space
- Small Aperture and long working distance (right) give the highest possible depth of field

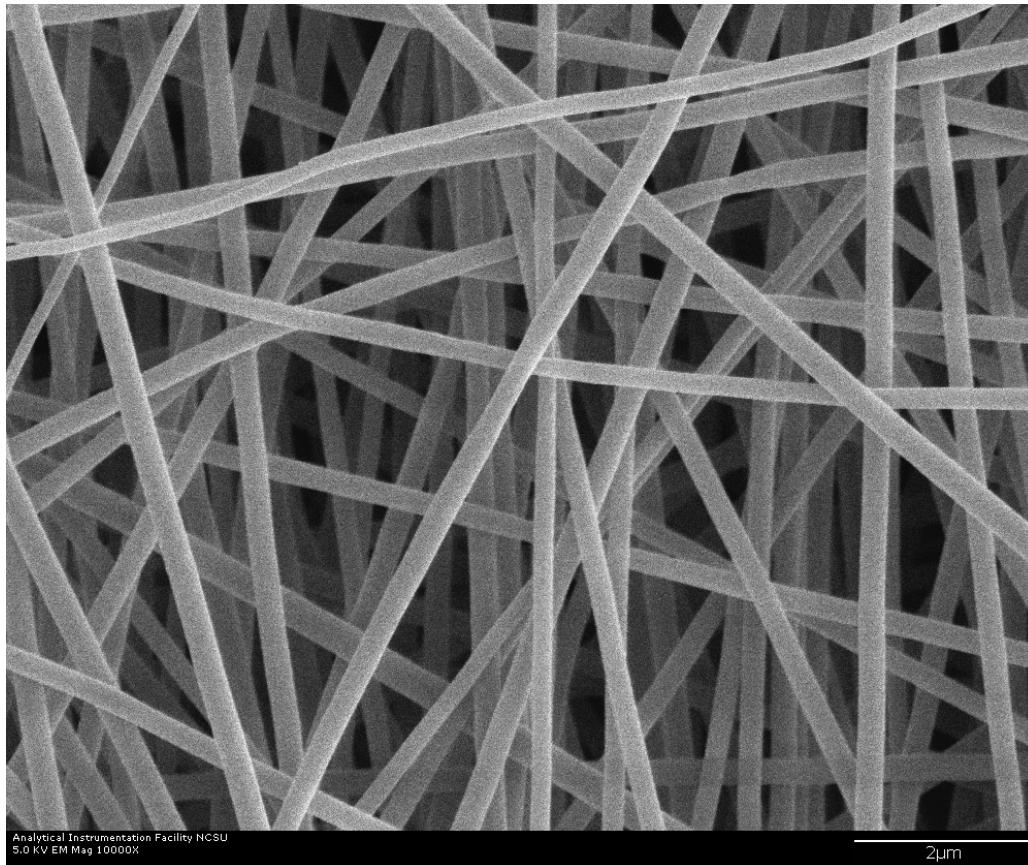


Simple geometric schematic of increasing depth of field.
Low depth of field condition on left.
High depth of field condition on right

High Depth of Field

- The apparent DOF can be increased by using a large spot size and a low magnification, however, these conditions will not work for high resolution imaging
- There is no good way to make both high resolution and high DOF images, that is, as the resolution (spot size) of the beam decreases and the magnification increases, the apparent DOF will decrease
 - This is a common problem with visible light microscopes
 - High depth of field is part of the reason that SEMs were developed!

High Depth of Field



- High depth of field image of an electrospun nanofiber sample
- The image appears in focus at least 10 fiber diameters into the sample
- Image collected with: 5 kV beam, 30 um objective aperture, 60% condenser lens, and a 20 mm working distance using a JEOL JSM-6400F conventional FESEM

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Electrospun Nanofibers
Champion Data

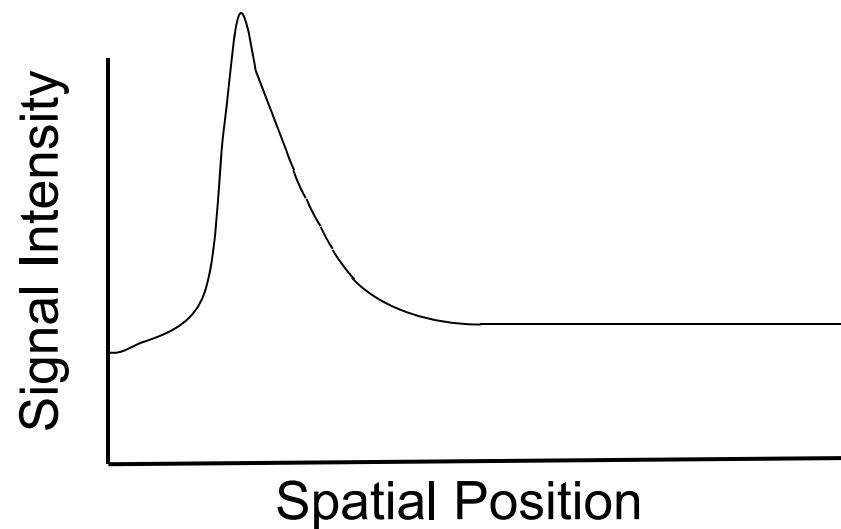
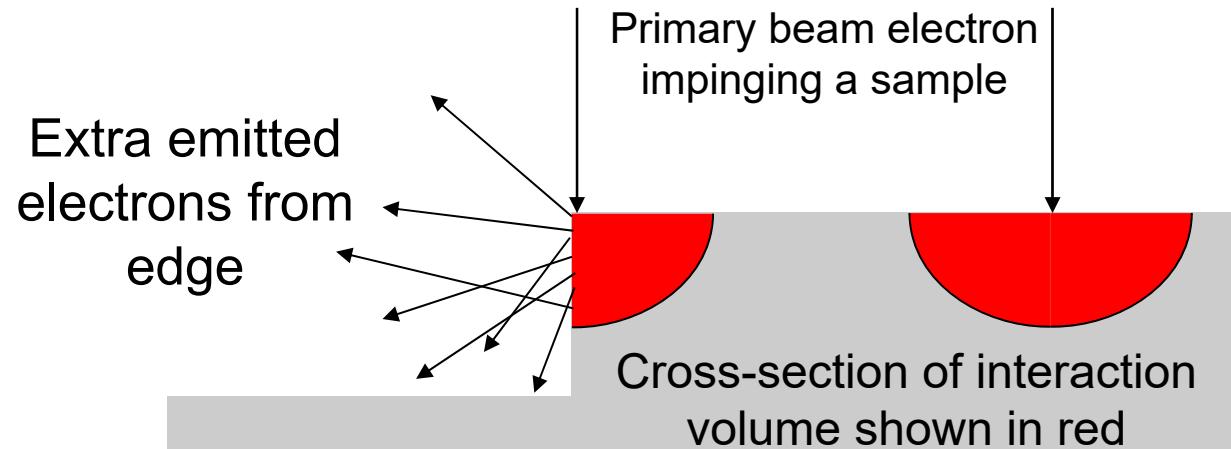
Verios 460L UHR-SEM Apertures

- The Verios automatically chooses the aperture based on the beam current chosen
 - The user does not have any control over the aperture choice
- What does this mean?
- Less work for the operator!
- The UHR-SEM is not designed for high depth of field
- For high resolution, choose a small current!

Edge Effects

- As the beam approaches an edge, the interaction volume will extend out of the sample
 - The interaction volume is really in the sample
 - Forward scattered electrons and secondary electrons are emitted from the edge of the sample into free space where they can be detected
- Since more electrons are scattering out of the edge than out of the sample surface normal to the beam, the edge appears bright
- Edge effects highlight edges
 - Photons do this too!
 - Human eye/brain knows how to interpret edge effects from the way that visible light photons behave at edges
 - This is why SEM images look so nice and are so easy to understand!
 - Most humans can interpret an SE image without any knowledge of what an SEM is

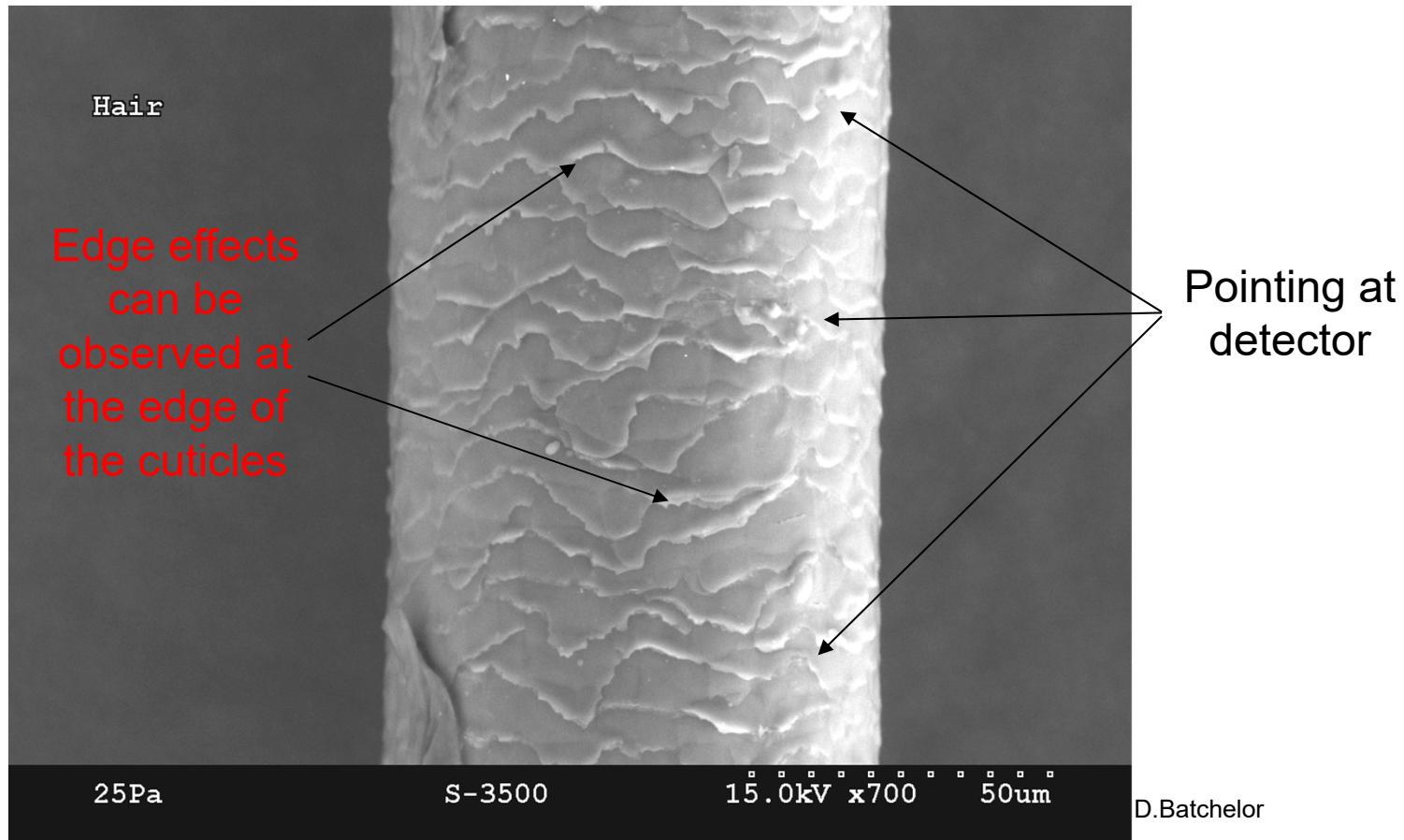
Edge Effect Schematic



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Signal increases at the edge due to increased electron emission

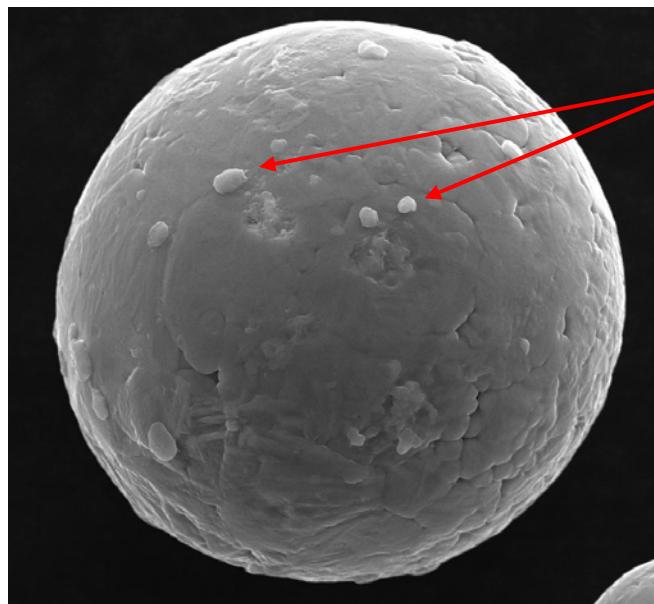
Edge Effects



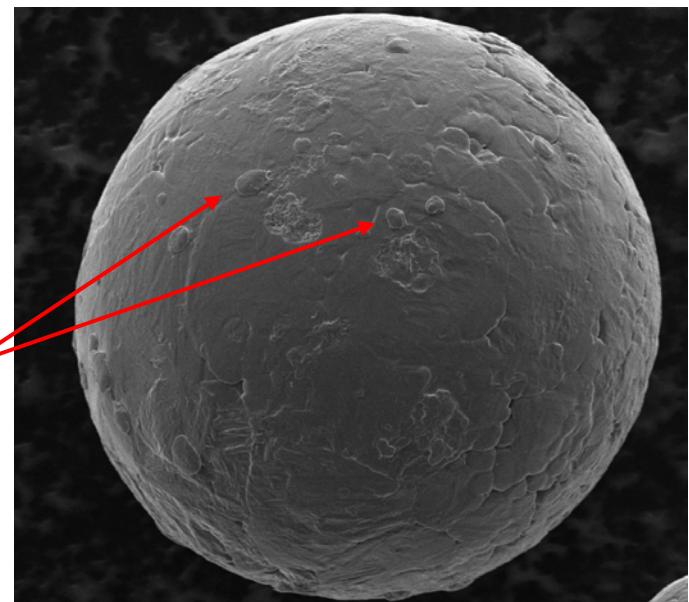
Note: The side of the fiber facing the detector is brightest and the edges of the scales are also bright

Edge Effect Issues

- If a feature is smaller than the interaction volume, then the whole of the feature becomes an edge effect and the whole feature will be brighter than it should be
- This is a problem for nanoscale particles and features
- Use low energy electrons to reduce edge effects



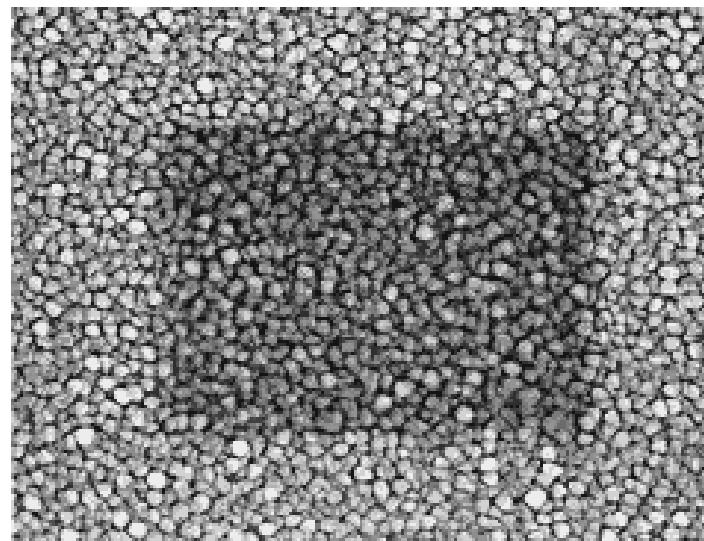
30 kV: All edge effect



5 kV: Edge effect only at edge for a more natural appearance

Beam Damage

- Beam Damage can take on many forms: contamination growth, heating (melting or degradation), etc.
- Easy to tell if beam damage has occurred by observing a lower magnification image
- Beam damage can be reduced by reducing beam energy and/or beam current and/or dose (total number of electrons that strike the sample)



R.Garcia

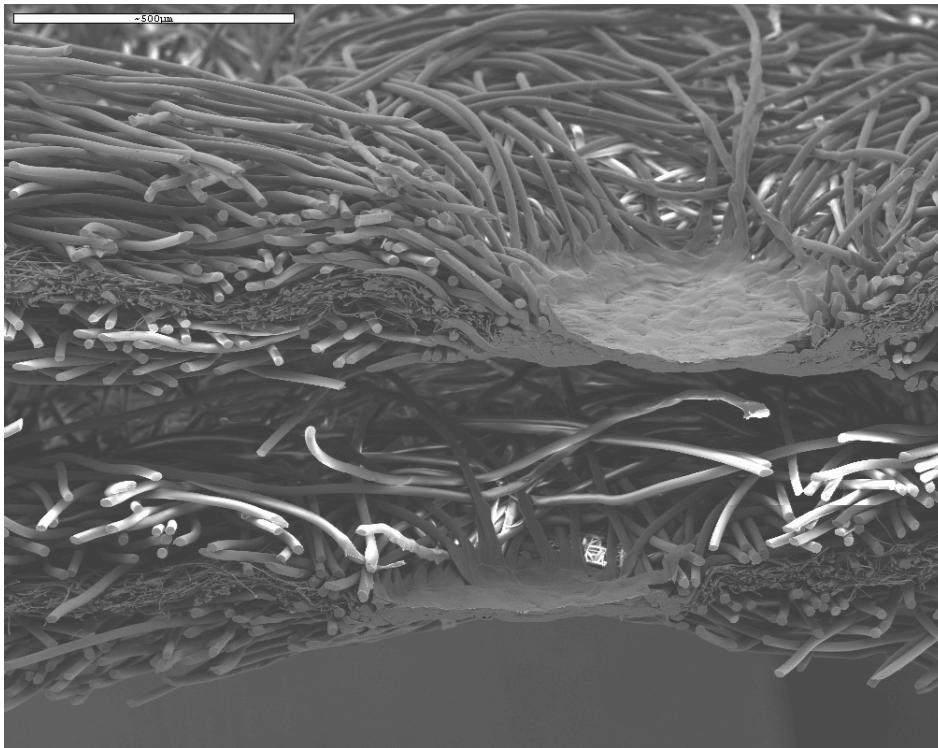
ITO (Indium Tin oxide) sample

- Image taken after leaving the beam in place for a long time (10 or so minutes)
- The dark area in the center of the image has been damaged and has a layer of carbonaceous contamination
- Note that cracking can be observed around the scan box (heat effect)
- Note how the contaminated area has less contrast
- Contaminated areas will also have less detail

Charging

- Charging occurs when the number of electrons going in a sample is different from the number of electrons exiting the sample. The electrons exiting can be via sample current as well as SE and BSE's that exit the sample.
- Charge balance is generally achieved by flowing electrons in or out of the specimen directly. This is typically done via the ground path, which is also used for specimen current imaging. Note that insulators typically do not flow charge very well!
- Charging can be overcome in several different ways:
 - Coating with a conductor (Au/Pd 60/40 is a good choice, ~5nm full coat)
 - Reducing the beam energy until the charge balance point is reached
 - Tilting the sample until charge balance is reached
 - Operating the instrument in charge reduction (variable pressure) mode

Charging Example



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- Not enough coating on a non-woven fabric
 - ~15 nm coating (micron marker is 100 um!)
 - 50 nm is a heavy coating – within measurement error at <1kX!
 - Accepted measurement errors for SEM are +/- 5%
- Areas that are both excessively light or dark indicate charging

Charging is Time Dependent

- Charging phenomenon are time dependent
- This is due to scanning the beam
- Charge is injected at a point, then the beam moves on and the stored charge can bleed off
 - When the beam returns, charging in the interaction volume begins again
- Change the dwell time, change the charging artifacts

Schematic of an RC charge-discharge cycle. In the SEM, the charge and discharge rates may not be equal. This behavior is similar to that of a simple RC circuit.

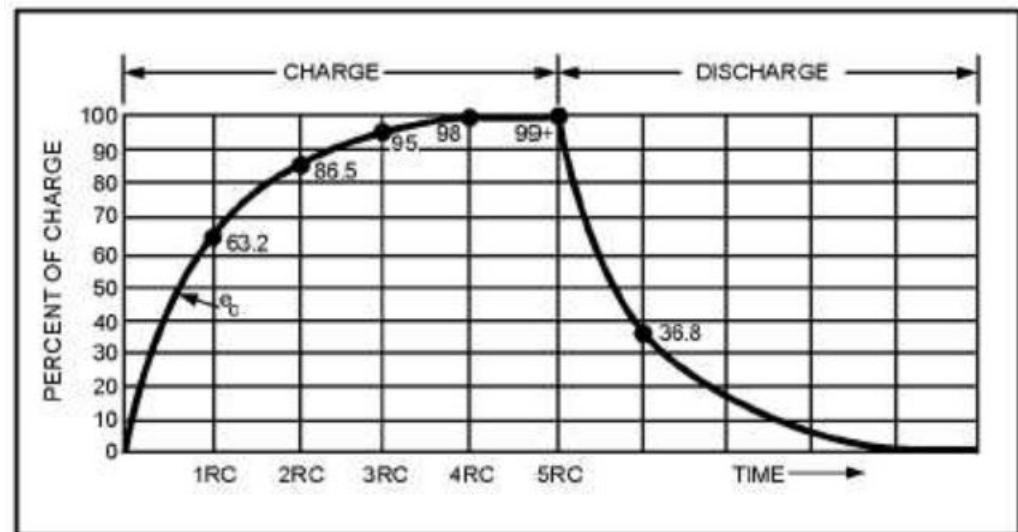


Image source unknown

Charging Example

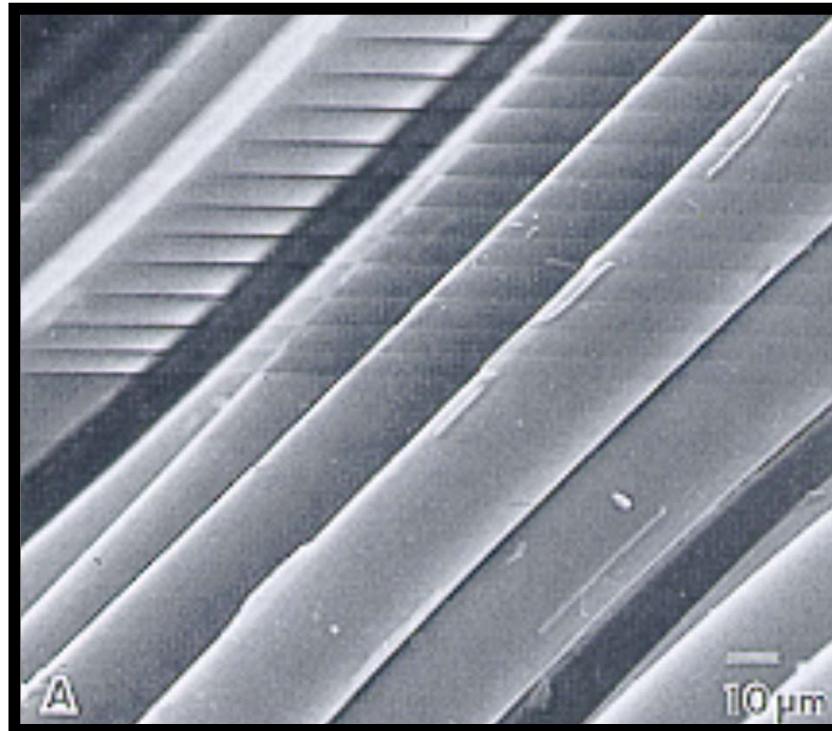
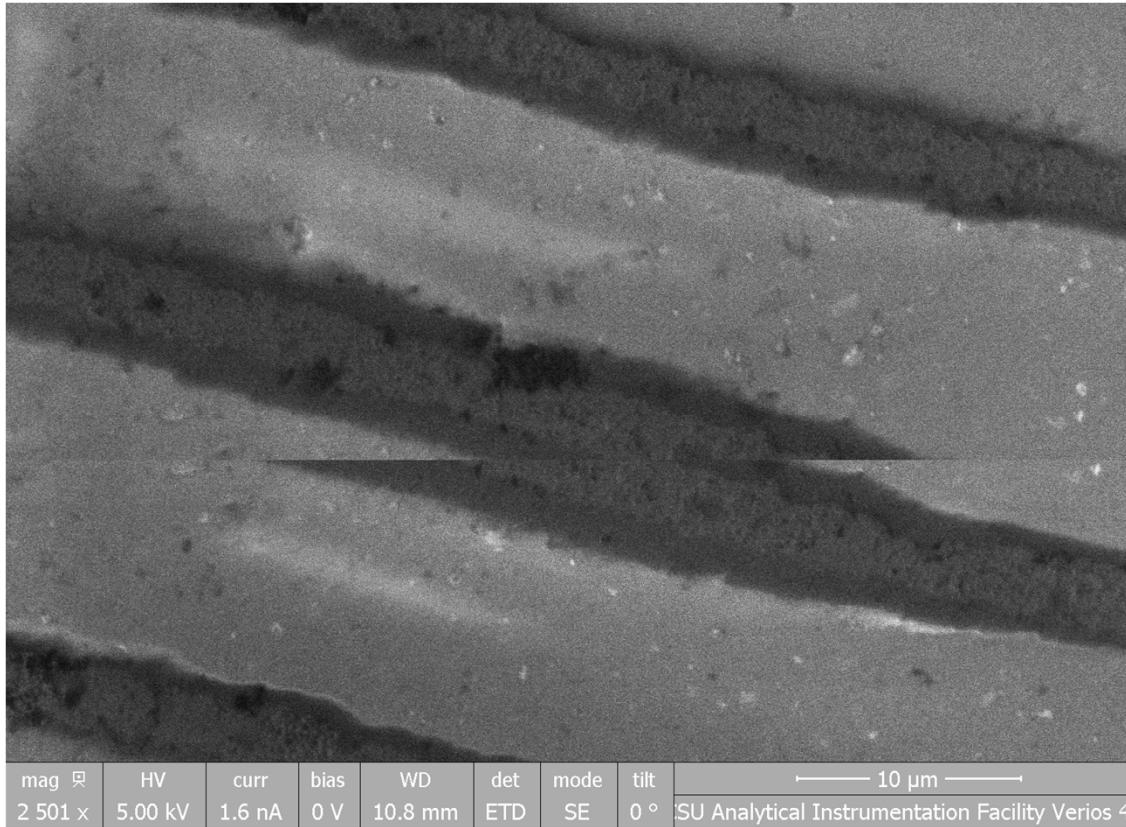


Image source
unknown

- Streaks are where there has been a charge-discharge cycle
- Likely some insulating debris just off screen on the upper left
 - SEMs overscan due to the non-linear and hysteretic nature of the scan coils
 - Beam dwells out of view on left before beginning the next scan
- In general, any imaging irregularity observed in the fast scan direction should be considered an artifact unless proven otherwise

Charging Example



mag	2 501 x	HV	5.00 kV	curr	1.6 nA	bias	0 V	WD	10.8 mm	det	ETD	mode	SE	tilt	0 °	— 10 μm —
-----	---------	----	---------	------	--------	------	-----	----	---------	-----	-----	------	----	------	-----	-----------

SU Analytical Instrumentation Facility Verios 46

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- Patterned insulator
- Movie shows charge affecting SE emission
 - Detrimental to morphology measurements
- Sample isn't moving! Charge is pushing the beam around

How can we deal with charging?

There are several ways to deal with charging:

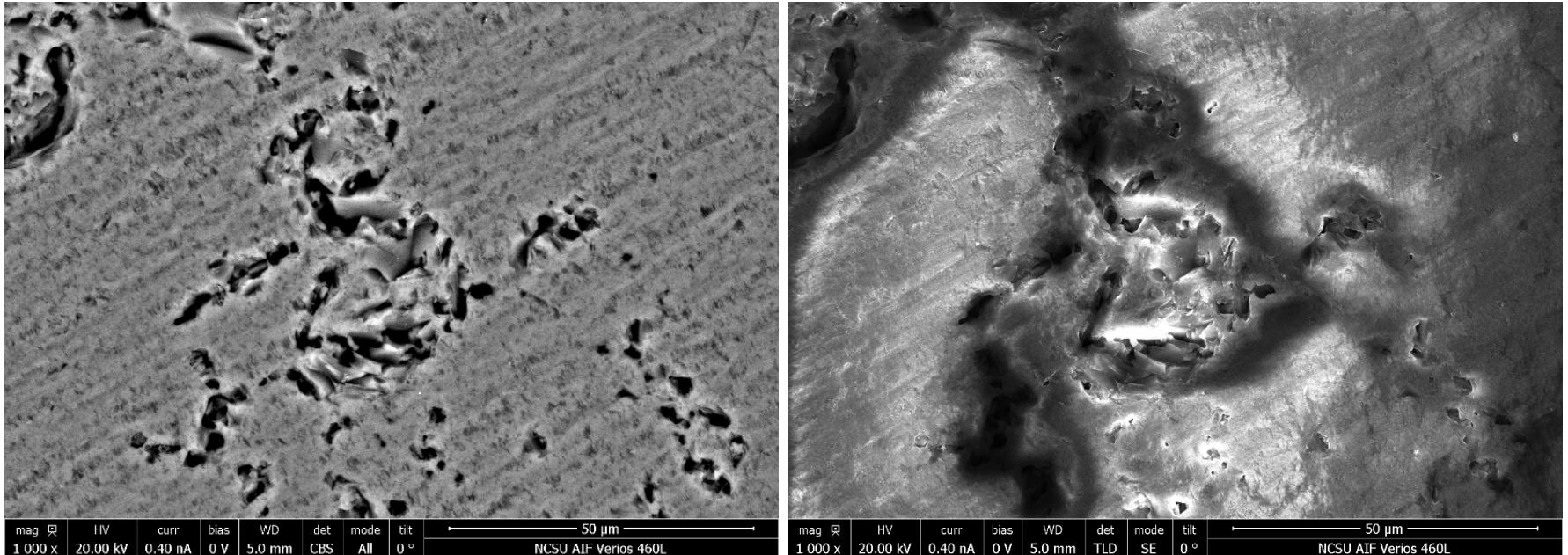
- Choose to image only conductive samples
- Ignore it
- Coat insulators with a conductive coating
 - Possible: Stain soft or bio samples with conductive salt solution
- Tilt insulating samples until charge balance
- Image insulators at the charge balance point
- Operate in variable pressure mode
- Operate in environmental mode

Please note that variable pressure and environmental modes are two different things!

Practical SEM: Ignore the charging

- Some insulators (mostly ceramics and other polycrystalline materials) can be imaged without a conductive coating at high voltage using the BSED
- Insulators of this type do not seem to build up very high surface charge
 - That is, these samples will charge but not to the point that high energy ejected electrons are affected
- High energy BSEs are not affected as much by charging as low energy SEs
 - BSE image is nice
 - SE image not so much
- How can you tell which insulators will work this way?
 - Experiment with imaging conditions
 - Generally speaking, if the sample gets charged up it can be discharged by bringing it out of vacuum for a bit

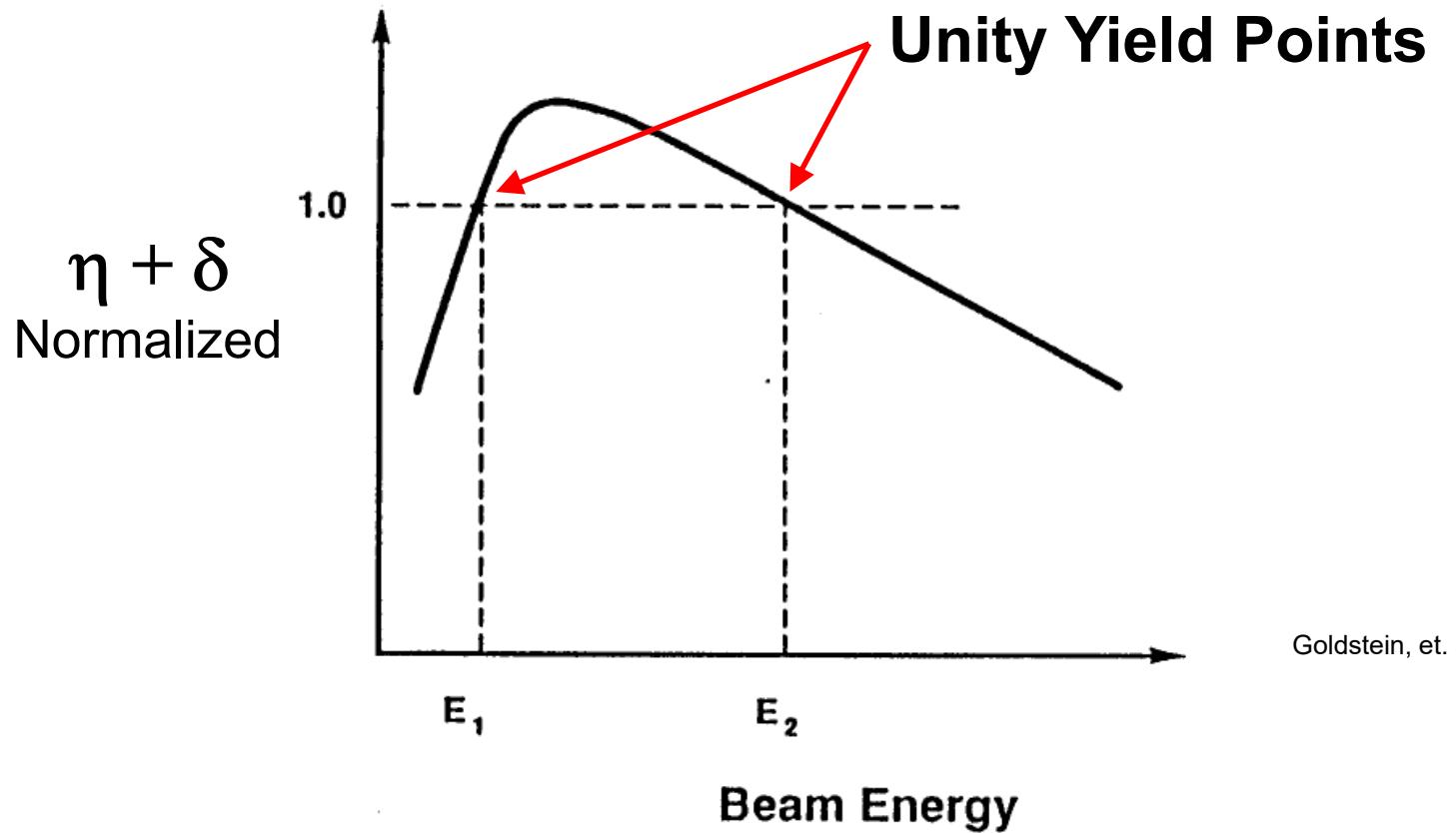
Charging Example



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- Ceramic sample imaged at 20 kV with 400 pA of current
 - High voltage, moderate current conditions
- BSE image on the left, SE image on the right
- BSE image is nice
- SE image shows charging

Total e⁻ Yield vs. Beam Energy



- E1 and E2 energies depend on material, typical E2 ~ 1.5 keV
- Traditionally, SEMs operated at > 10 keV, well above E2

Achieving Charge Balance with an Insulator

- If the beam energy is much greater than E_2 , then the system is unstable because charge is injected deep into the sample and is difficult to remove
- If the beam energy is close to E_2 , the system tends to stabilize in a charge balance condition – there is a natural feedback loop that pushes the system to charge balance
 - If the energy is slightly less than E_2 a slight positive charge will develop, which will accelerate the electrons, thus increasing the energy in the beam and trending toward charge balance
 - If the energy is slightly more than E_2 a slight negative charge will develop, decelerating the primary electrons – trend toward charge balance
 - E_2 tends to be on the order of 0.5 kV – 5 kV for most insulators
- If the incident beam energy is off slightly from E_1 , then the system will not trend toward charge balance due to the slope of the curve being positive

Upper Crossover Energy

Table 3.7. Upper Crossover Energy for Various Materials (Normal Beam Incidence)

Material	E_2 (keV)	Reference
Kapton	0.4	Joy (1988)
Electron resist	0.55–0.70	Joy (1987)
Nylon	1.18	Joy (1988)
5% PB7/Nylon	1.40	Krause <i>et al.</i> (1982, 1987)
Acetal	1.65	Vaz (1986)
PVC	1.65	Vaz (1986)
Teflon	1.82	Vaz and Krause, (1986)
Glass passivation	2.0	Joy (1987),
GaAs	2.6	Joy (1987)
Quartz	3.0	Joy (1987)
Alumina	4.2	Joy (1988) Goldstein, et. al.

Minimizing Deposited Charge

- We want to minimize any deposited charge
- How do we do this?
 1. Low Voltage
 2. Low Current
 3. Scan fast
- Low voltage gets us close to charge balance
- Low current minimizes the number of electrons injected
- Scanning fast minimizes the number of electrons injected at any particular location in the scan
 - Charging is time dependent
 - If we allow any charge to bleed off before we inject more, we can reach charge equilibrium (if not balance) and make a good image
 - Equilibrium, in this case, means that the sample has reached a stable charge state
 - At equilibrium, it is usually possible to make reasonable images

Insulators: Low Voltage Imaging

- Before we go farther, we have to ask:
“What is our goal?”
- Usually, the goal is a pretty (SE) image of a region of interest of the sample
 - Want our image to be drift and distortion free
 - Want our image to have to be pretty and have rich contrast
 - Want our image to be scientifically valid
- If we are looking to do compositional contrast with a BSED or elemental X-ray analysis, we may be out of luck...

Limitations of Low Voltage (<1 kV) SEM

Compositional Contrast

- BSED usually only detects electrons with > 1keV of energy
 - Most BSEDs work best at 5 keV or more
- Compositional contrast will likely not work well under low voltage conditions
 - Unless the BSED cut off energy is ~50 eV

X-ray analysis

- It takes energy to excite X-rays
 - e.g., can't excite a 5.8 keV Mn Ka X-ray with a 500 eV electron
- EDS energy resolution is poor (> 100 eV)
- Low voltage X-rays have many overlaps
 - If the overlaps are not a problem, then this can be done

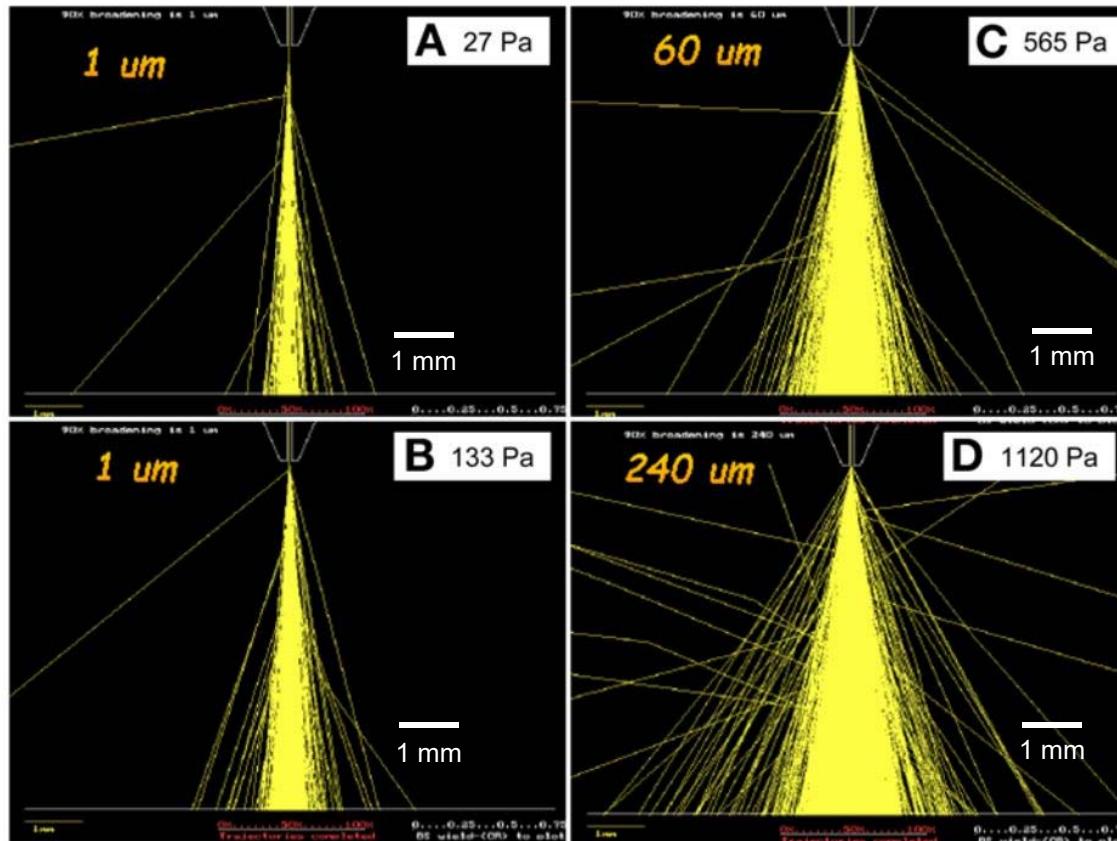
Imaging Insulators – Variable Pressure

- By bleeding a little gas into the sample chamber, charging can be reduced
- Instead of having high vacuum in the chamber and the column, the chamber has a reduced vacuum
 - A pressure limiting aperture is placed between the chamber and column
 - The vacuum system is more complex to have high vacuum in the column and low vacuum in the chamber
 - Chamber vacuums are on the order of 5-300 Pa
 - Higher gas pressures cancel more charge at the expense of a scattered beam (worse resolution)
- As soon as the beam enters the chamber, it is scattered by the gas in the chamber
 - Scattering increases with increasing working distance

Imaging Insulators – Variable Pressure

- Some of the gas molecules will be positively ionized by high energy primary and BSEs in the chamber
- Charge is cancelled by:
 - Positively ionized gas is attracted to negative charge on the sample
 - Low energy SEs are attracted to positive charge on the sample
- Cannot use the ET secondary electron detector
 - ET detectors typically use a 10kV bias – arcing may occur damaging the detector and other expensive SEM parts
- **Coated samples imaged under normal high vacuum conditions make prettier images!** (Usually)
 - This rule only applies to samples that would be imaged in variable pressure mode
 - Variable pressure mode does not work well for high resolution applications
 - What to do? EDS/BSE uncoated, then coat for high quality SE images

Beam Scattering



Griffin, Brendan J, Methods in molecular biology (Clifton, N.J.), 2007, Volume 369

Scattering of a 10 keV beam by water vapor with a 6-mm working distance and for chamber gas pressures of (A) 27 Pa (0.2 torr), (B) 133 Pa (1.0 torr), (C) 565 Pa (4.0 torr), and (D) 1120 Pa (8 torr). Bar = 1 mm.

Magnification in the SEM

- The **magnification** of the specimen image is the ratio of the displayed image on the viewing screen to the size of the pattern on the specimen, or

$$M = A_{\text{display}} / A_{\text{sample}}$$

- Since the screen area is constant, higher magnifications are obtained by reducing the size of the raster on the specimen
 - Note with digital systems, the display size may not be constant...

Magnification in the SEM

Magnification of the specimen image is obtained by beam deflection and not by lens action!

- Changing magnification does not change the optics!
 - If the image is in focus at high magnification, then it will be in focus at low magnification
 - In general, one should focus at higher magnification than is desired for the image and then reduce magnification to the desired magnification
- Nomenclature:
 - Increase mag = Zoom in
 - Decrease mag = Zoom out

Note on Magnification in the SEM

- Traditionally, SEM images were recorded on Polaroid film
 - Film size: 5"x4"
 - Everyone used the same size film
 - Magnification in one lab equals magnification in another...
- With the advent of digital imaging, image display sizes are no longer fixed – Compound this with the use of projectors for lectures...
- SEM magnification still uses the traditional definition
 - Some instruments allow the user to define the display size
- That is, SEM magnification numbers assume a 5"x4" display → most of the time the displayed magnification is wrong!
- It is not all bad: the micron marker scales with the image!
- It would be smarter to define the width of the horizontal field of view
 - Then we would not need a micron marker and we would not need to define magnification based on an arbitrary display size

Hollow Magnification

General definition of hollow magnification:

Hollow magnification: Increasing magnification leads to no gain in information and perhaps the loss of information.

- Hollow magnification exists in many venues...
- Consider a film of dance
 - Too often, the director and camera operator want close-ups that are too close
 - Information about motion the dancer is performing can be easily lost
- This definition covers situations where the resolution is sample dependent, i.e., where the spot size is small enough for higher magnification but no additional gain in information can be achieved due to sample limitations.

Microscopist's Trap

The Microscopist's Trap is a corollary to hollow magnification.

The Microscopist's Trap is to look at a very small area with high magnification and claim that is what the whole of the sample looks like.

In practice, it is very smart to collect a series of images from low to high magnification to show that the high magnification images are representative of the sample and not some special case that is not representative of the whole.

Magnification and Resolution

- Magnification and Resolution are two different things that are completely independent of each other
 - Magnification = displayed area/scanned area
 - Resolution is the ability to show a feature clearly and with detail
 - Resolution requires contrast between two features
 - Magnification without resolution is meaningless
- Resolution measurements are typically done using a Au on C resolution standard
 - Au islands evaporated onto a C puck
 - Au on C is chosen because this system gives the highest contrast
 - Most samples will not have the contrast level that Au on C has
- Resolution is generally sample limited!

General Resolution Considerations

- In general, resolution in a scanning microscope is **SAMPLE LIMITED!**
- Most of the time, the limit of what a high end, high resolution scanning microscope can do is defined by the sample!
 - This is an electron-sample interaction limit
- Resolution standards exist because they provide the highest possible resolution
- If this is in doubt, it is always possible to put in a resolution standard to prove what resolution the instrument is capable of for the conditions chosen

Resolution Limiting Factors

- Spot size limited resolution:
 - Where the size of the beam is greater than the size of the feature or the pixel
- Pixel limited resolution:
 - Where the size of the beam is smaller than the size of the pixel but the pixel is larger than the feature of interest
 - Also note that it takes more than one pixel to resolve a feature!
- Interaction volume limited resolution:
 - Where the size of the beam is smaller than the size of the feature, but the energy of the beam is great enough that minimal interactions occur within the feature

SEM Resolution

- Spot size is often the limiting factor
 - It is easy to observe the effects of increasing probe size
 - As the spot size increases, the observed resolution will decrease
 - This is true for ANY scanning microscope!
- Pixel resolution is very important
 - The more pixels in the image, the higher the potential resolution
 - Pixel resolution is selectable with (most) digital imaging systems
- Interaction volume limited resolution is trickier to define
 - This is what creates the idea of a fundamental resolution limit for SEM
 - That is, we are injecting electrons into the sample and usually expecting to measure either an electron or X-ray that comes back out of the sample (opposite the direction of the primary electron!)

Pixel limited Resolution

- Pixel resolution is easy to define and understand
- We now live in a digital world, with respect to imaging
- As pixel densities increase, the image gains resolution
 - Think old school 640x480 TV vs. a modern 4k (3840 x 2160) monitor
 - A 4k monitor has ~6X the resolution of the 640x480

In general:

- The more pixels in the image, the higher the potential resolution
- The more pixels in an image, the longer it will take to acquire
- Pixel resolution is selectable with (most) digital imaging systems
- A single pixel is not enough to spatially resolve a feature

Pixel limited Resolution

- A single pixel is not enough to spatially resolve a feature!
- A good rule of thumb is that a minimum of 10 pixels are needed to claim that a feature has been spatially resolved.
- A very small pixel is not helpful if the beam is very large...

How to Choose Image Pixel Resolution

What is the image to be used for?

- Publication in a journal or on a poster
 - On the order of 1000x1000 pixels should be enough
 - Journals usually shrink images to small sizes, so the image is likely to be compressed negating the need for high pixel resolution
 - Full pixel resolution will be good for a poster (generally printed with large images)
- General research, i.e. reduction of parameter space or a quick and dirty look at the sample – not to be published
 - Relatively low pixel resolution is OK
- Artwork
 - High pixel resolution is good
 - How high depends on the application and the amount of time required to collect the image

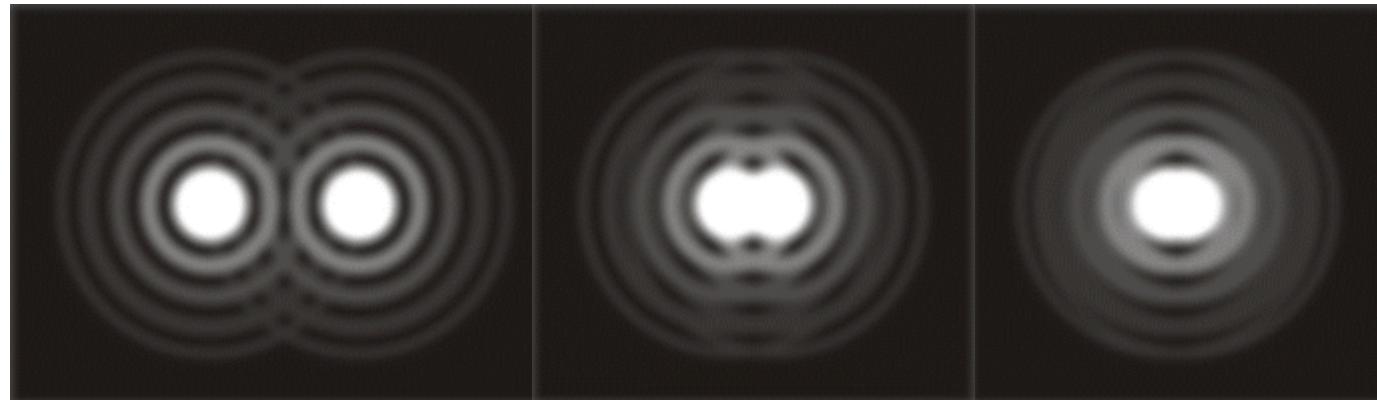
Practical SEM: Image Pixel Resolution

- In general, on the order of 1500 x 1000 pixels (1.5 Mega pixels) is enough resolution for most purposes
- Standard resolutions tend to be something like
 - 1024 x 768
 - 1536 x 1024
 - 2048 x 1536 ...
- Any of these will work for most data collection purposes
 - We typically use either 1024 x 768 or 2048 x 1536 with the VPSEM and 1536 x 1024 with the Verios.

Spot Size Limited Resolution

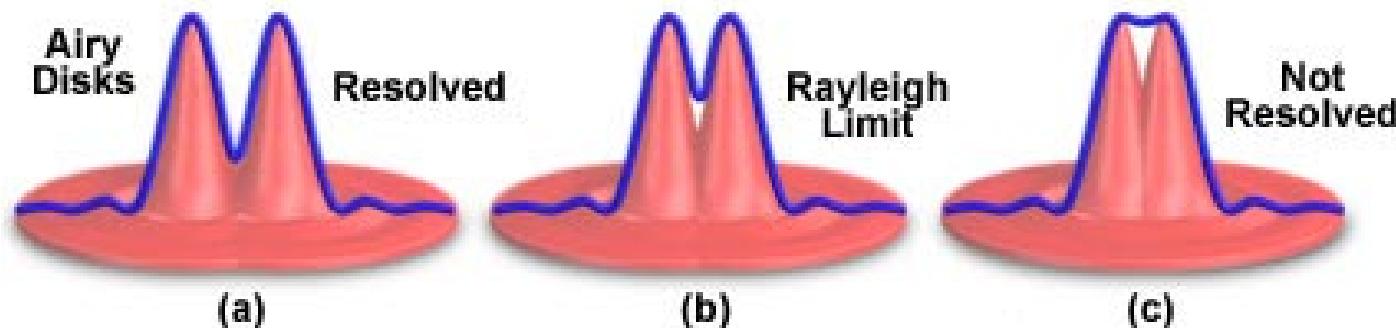
- The best physical definition for spot size limited resolution comes from photon optics and is generally referred to as the Rayleigh criterion
 - This ignores the interaction volume because the interaction volume for photons is very shallow relative to achievable spatial resolution
 - There is an interaction volume for photons! It is known as the skin depth and is on the order of a few nm
- The Rayleigh criterion is based on the diffraction limit and is the minimum distance between two Airy disks that can be observed
 - An Airy disk is the central part of a diffraction pattern created by a circular aperture
- The Rayleigh criterion definition is when the maximum of the two disks overlaps the first minimum

Resolution – Rayleigh Criterion



Randy Culp

Airy Disk Separation and the Rayleigh Criterion



Olympus Microscopy

- The system is considered not resolved when the maximum of the first disk overlaps the first minimum of the second disk.
- This was first defined by Lord Rayleigh

Lord Rayleigh, F.R.S. (1879). "Investigations in optics, with special reference to the spectroscope." *Philosophical Magazine*. 5. 8 (49): 261–274

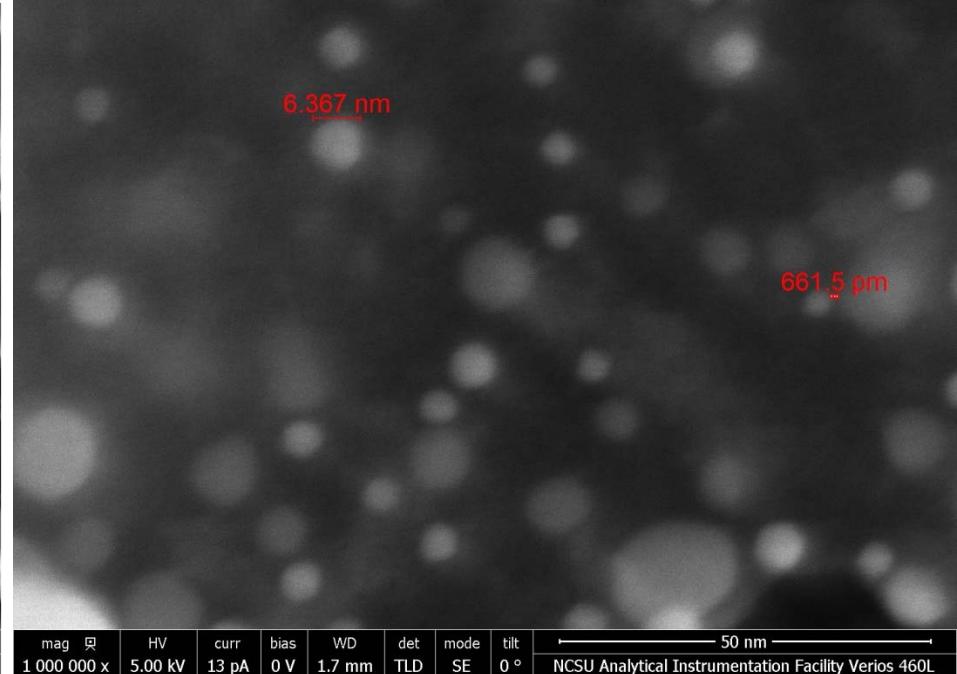
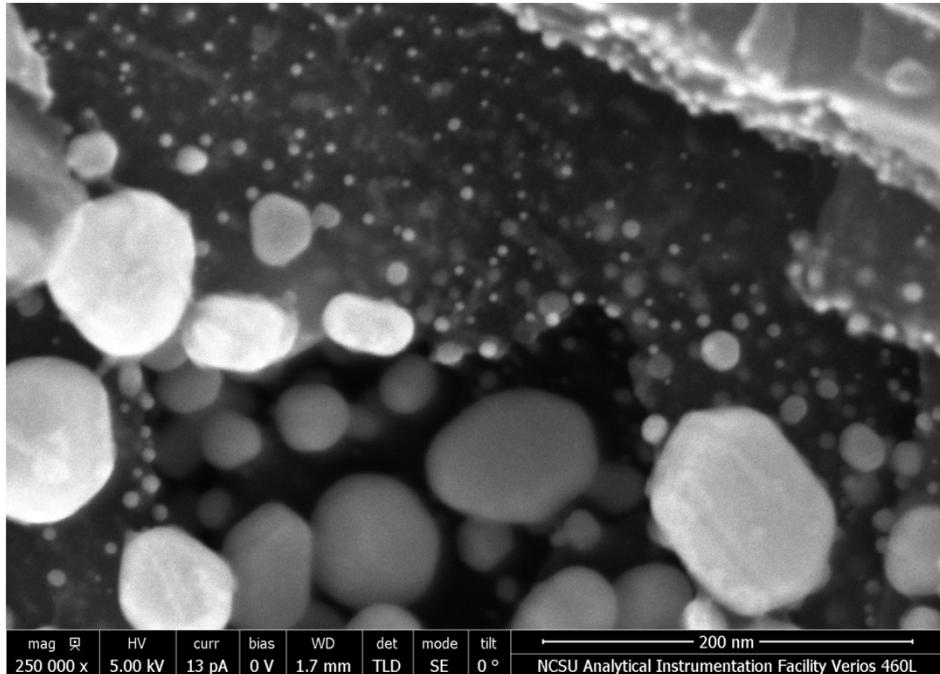
Chuck Criterion Resolution

- In the SEM, we aren't creating Airy disks
- The electron beam has a Gaussian distribution and appears as an Airy disk without the diffraction rings
 - The electron spot is not diffraction limited!
 - We don't have to worry about the diffraction rings
- This also means that we can't use the definition proposed by Lord Rayleigh

Chuck Criterion for spot size limited resolution:

- When the Gaussian distributions that define the diameter of the beam overlap by less than the full width at half maximum, then we have spot size limited resolution

Au on C Resolution Standard



C.Mooney

- Au on C resolution images taken at 250kX and 1MX in the AIF FEI Verios 460L SEM
- Measuring the smallest distance resolvable between Au grains approximates the Chuck criteria

Fundamental SEM Resolution Limit

Know the following:

- With all other things being equal, as the energy of the beam increases, the spot size will decrease
- Electrons form an interaction volume in the sample
- BSEs most likely go back in the direction of the beam
- The average scattering angle for a pure elastic scattering event is ~2 degrees
- SEs are formed all along the path of the BSE
- High end SEMs have the same resolution at low voltage (~2kV) as they do at high voltage (30kV)
- Operated in STEM mode (beam passes through a very thin sample with a detector below the sample), one can achieve slightly higher resolution than by viewing a bulk sample
- The limit of what a high end SEM can do has remained at 0.5 nm for >15 years! (It is easier now!)

Fundamental SEM Resolution Limit

The information on the preceding slide suggests a fundamental resolution limit for SEM of ~ 0.5 nm

- Consider that the average separation between atoms in solids is on the order of 1 – 3 Angstroms (0.1 nm)
 - On the order of 100 atoms in the average 0.5 nm volume of solid material
 - If the primary electron is scattered in the same direction at the average scatter angle, then it will take 90 or so scattering events to make a 180 degree turn
 - Not all primary electrons will be scattered this way, so this is not a statistical average
- We are injecting electrons into the sample and expecting to measure signal coming out of the sample
- It takes volume for the electron to interact with the solid and produce signal that can be measured!

Fundamental SEM Resolution Limit

What the fundamental resolution limit means:

- High end SEMs do not have spot size limited resolution
- The resolution is limited by the electron-sample interaction
- The fundamental limit is a function of the electron-sample interaction volume

Prediction for the future of SEM

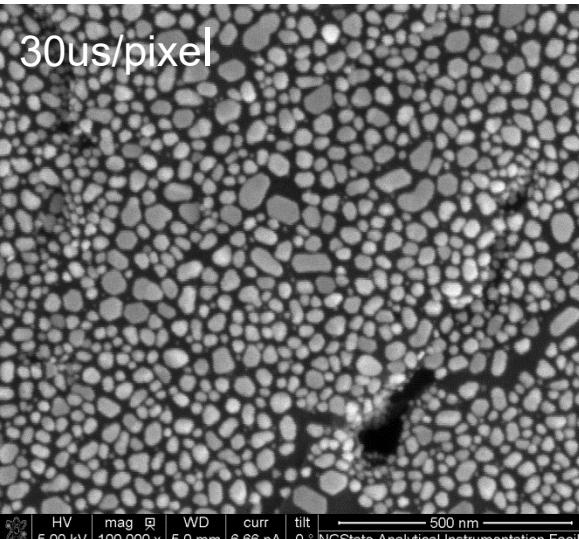
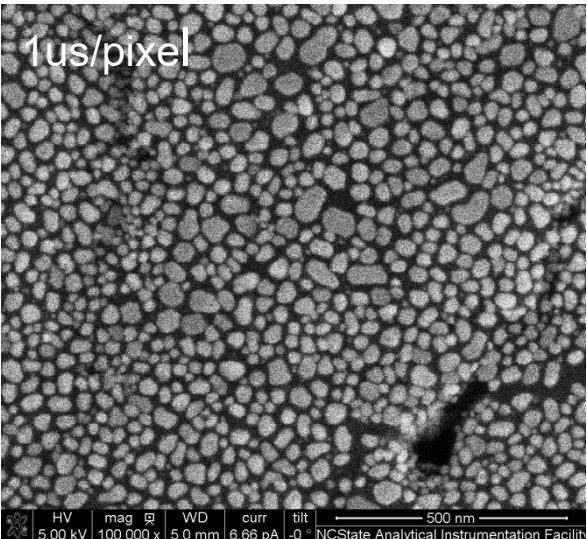
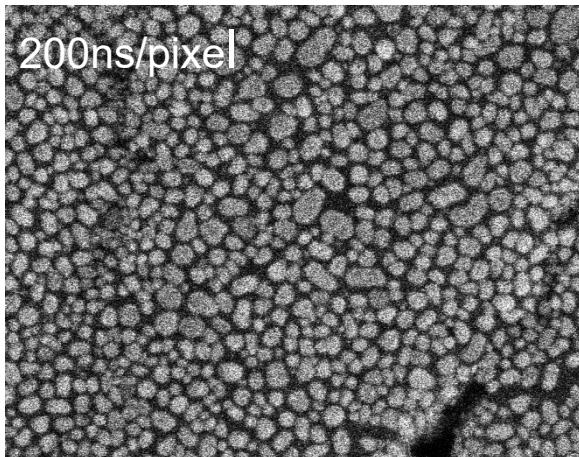
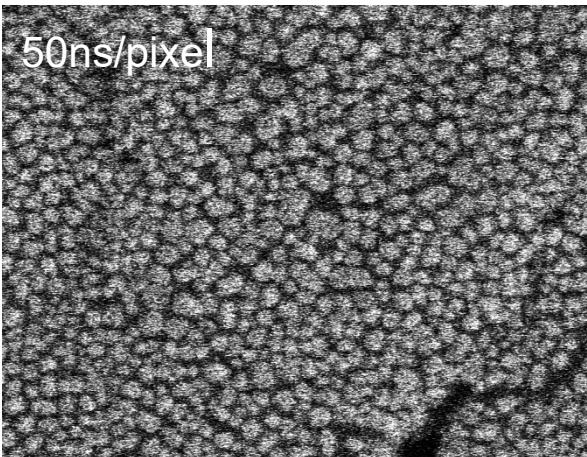
- Resolution will not get any better than 0.5nm
- It will get easier to achieve 0.5nm resolution
- 0.5nm resolution will happen at lower voltages as the optics improve
- Cs correctors will not become common on SEMs
 - Cs correctors allow for smaller, more precisely defined beam sizes and are now used on TEMs
 - Exception might be for extreme performance at very low voltages

Practical SEM: Dwell Time

Assuming a conductive or coated sample:

- ETD images are publication quality with ~30us per pixel of dwell time
- BSE images can take longer
- Actual dwell time will depend on beam conditions (current and voltage) and detector efficiency
- How to decide for sure – look at the image!
 - If the image is noisy, increase the dwell time
 - If the image is taking a very long time to collect and is not noisy, decrease the dwell time

Au on C: different dwell times



Au on C
resolution
standard

Note:
Image quality
and **resolution**

C.Mooney

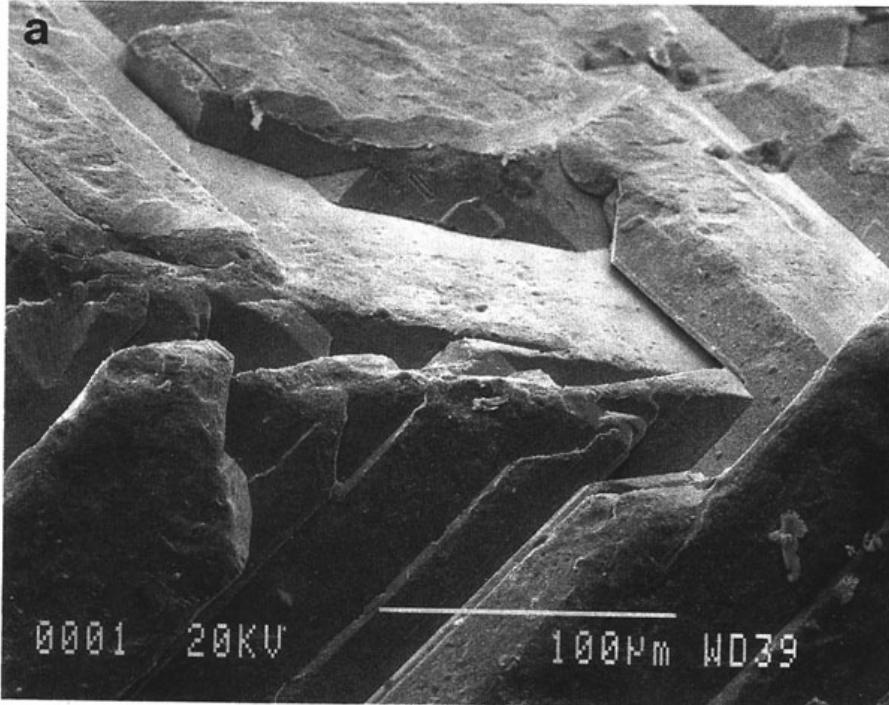
Image Appearance: General

In general in the SEM:

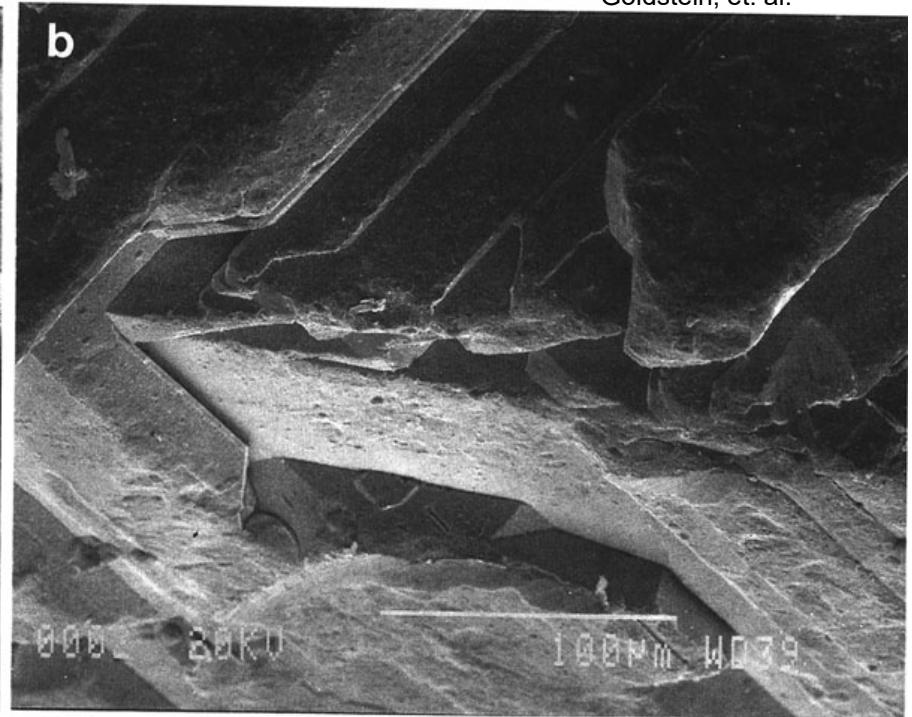
- The illumination source appears to be the detector position
- The view angle appears to be from the electron source, i.e., down the column
- Consider the geometry of the system to make images the most aesthetically pleasing
 - System geometry = beam-sample-detector geometry

Detector Position

Goldstein, et. al.



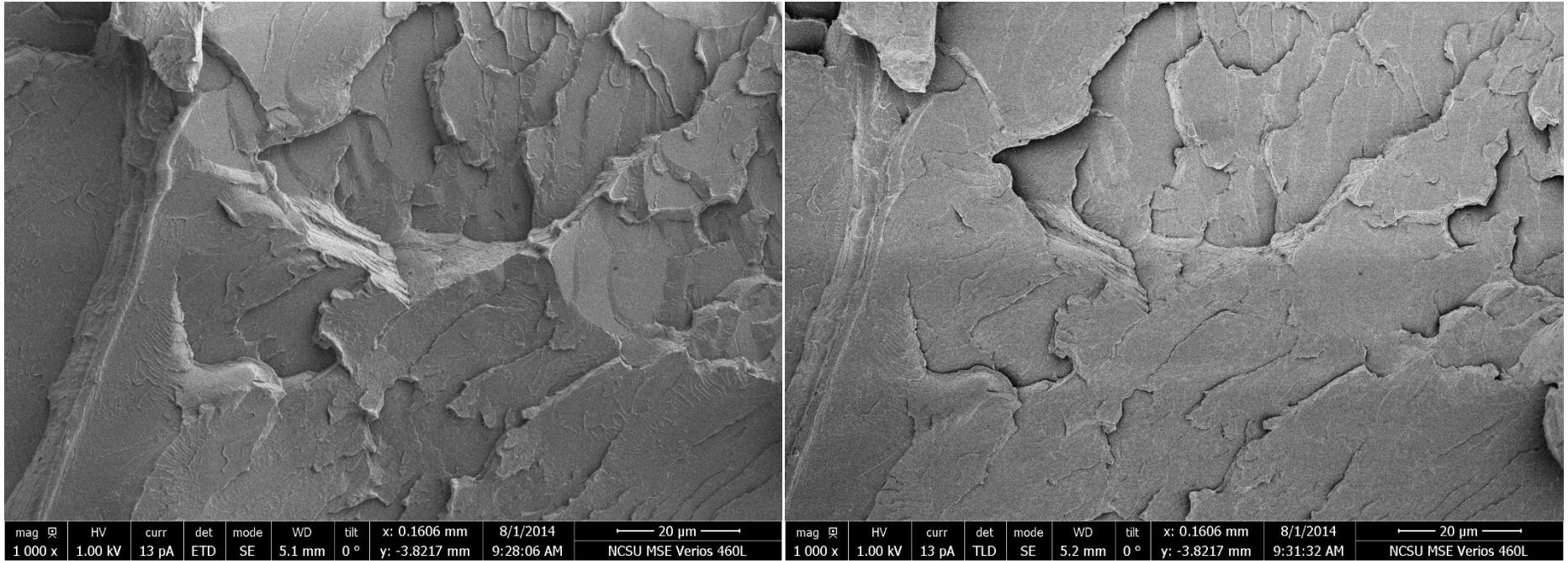
Champion Data: Shadows look natural



Not a bad image, but shadows are unnatural

- Same sample region, different imaging orientations
- The detector is up and to the right in both images
- Orienting the sample such that the shadows appear to be low in the image will appear more natural

Detector Choice



ETD image (left, Champion Data) and TLD (right) image of a polymer fracture surface

- Both SE images were collected using the same conditions. The only difference is the choice of detector
- The ETD image looks much nicer! This is due to the beam-sample-detector geometry.
- The ETD image appears to be taken from above with illumination from the upper right (ETDs are usually in the upper right relative to the image)
- TLD image looks flat and unnatural

Magnification Choice

- It is smart to use the same magnification sets when collecting data
 - This way, data from different samples can be directly compared!
- It is also smart to pick nice round numbers for magnification sets
 - It is easy to collect the same magnification sets if the numbers are easy to remember
 - 100, 250, 500, 1000, 2500, 5000, ...
 - Many conventional SEMs will not allow odd magnifications
 - The Verios (and presumably other FEI/ThermoFischer ‘scopes) will adjust the magnification if the focus is adjusted with the knobset (but not if focus is done with the mouse) – be careful that the mag is what you want it to be after focusing
- The exception is when an object should fill the field of view and nice round number does not work

Aesthetics

- Humans are aesthetically driven creatures
- That is, we like to look at things that are pretty!
 - The corollary is that we are fascinated by things that are ugly
- The effective microscopist will not only generate high quality scientific data but will also generate pretty images

Image Aesthetics

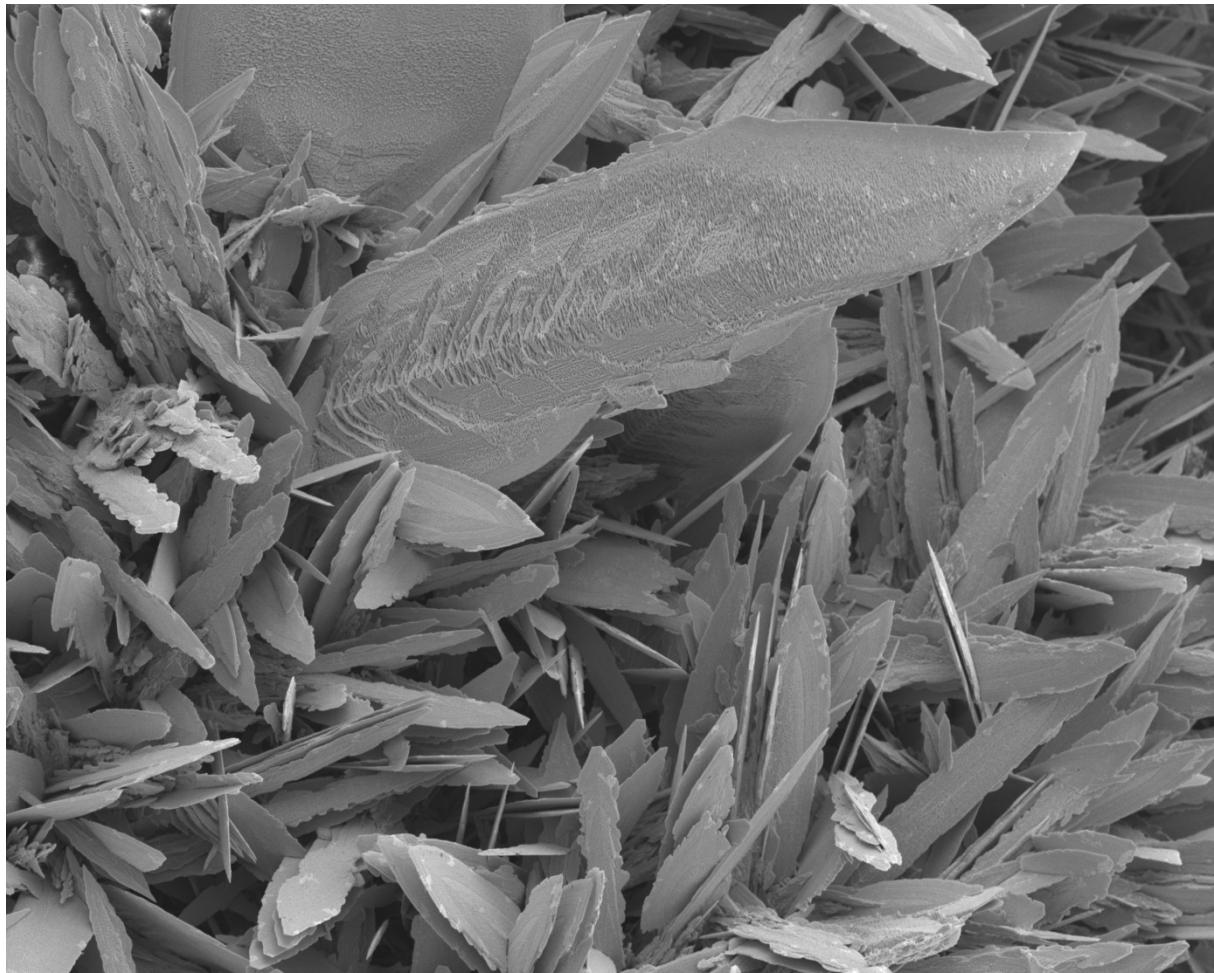
To create aesthetically pleasing images, we must consider what humans like to see

- Shadows should be low
 - The sun is above our heads and generally casts shadows down
- Objects should reach up
 - Trees and other tall plants, mountains, etc., reach up to the sky
 - Things reaching down at humans are scary!
- The primary feature of interest should be just above the vertical center and even around the horizontal center
- The image should be bright but not oversaturated
 - Humans are diurnal (daytime) creatures and like bright images (the dark of night is scary!)
 - Saturated images lose information

Champion Data

- Champion data is not only scientifically valid but aesthetically pleasing
 - Humans like to look at things that are pretty
- Now that we have seen the effects of the various instrument controls, how do we make champion data?
 1. Operator needs to understand beam-sample-detector geometry on data appearance
 2. Choose conditions appropriate for the data to be collected
 - Beam energy, Beam current, Working distance, Objective aperture
 3. Choose the correct detector
 4. Have a clean sample!
 5. Collect a high S/N image with sufficient pixel density

SEM Imaging Done Right...



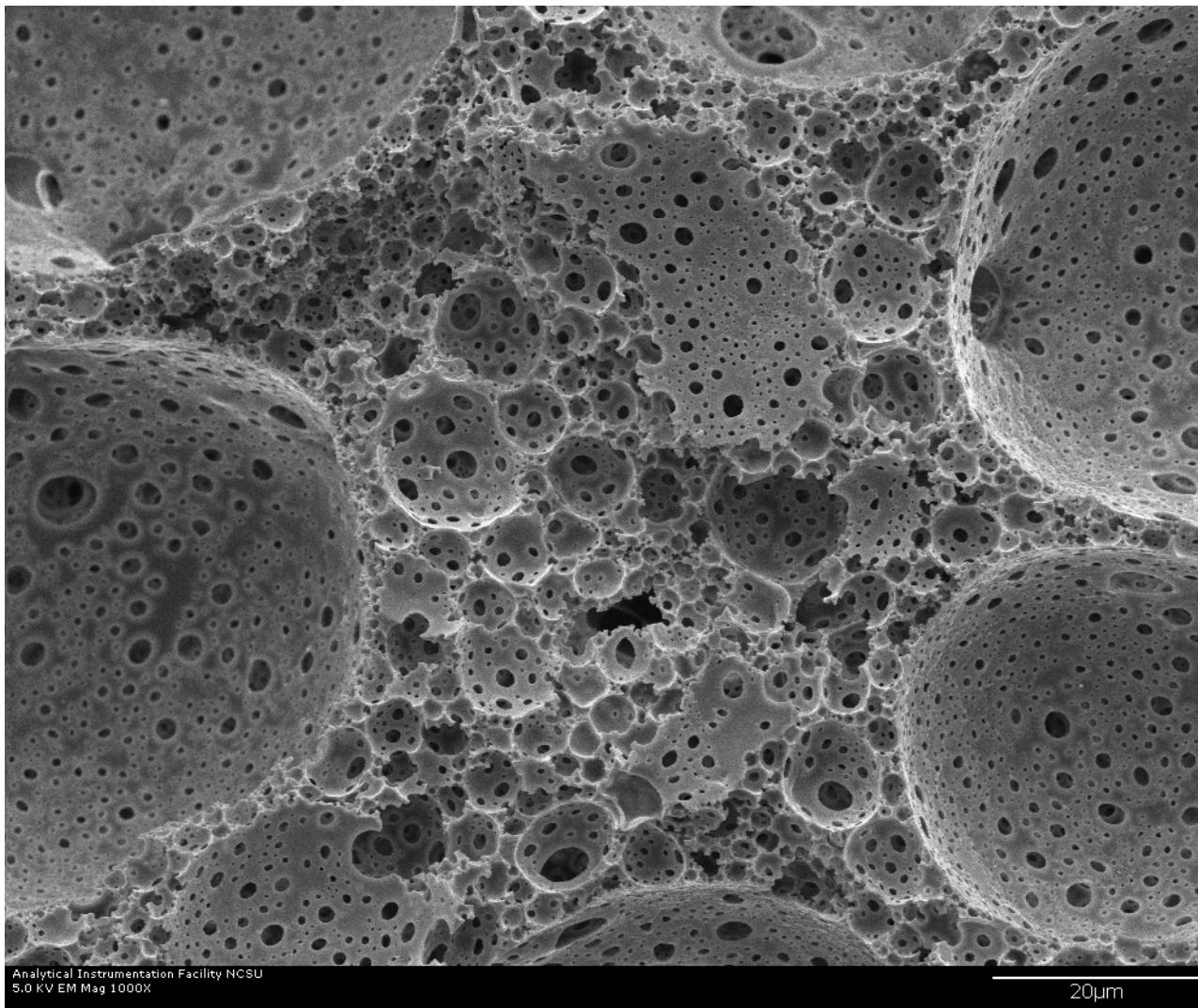
Analytical Instrumentation Facility NCSU
5.0 KV EM Mag 1000X

20 μ m

C.Mooney

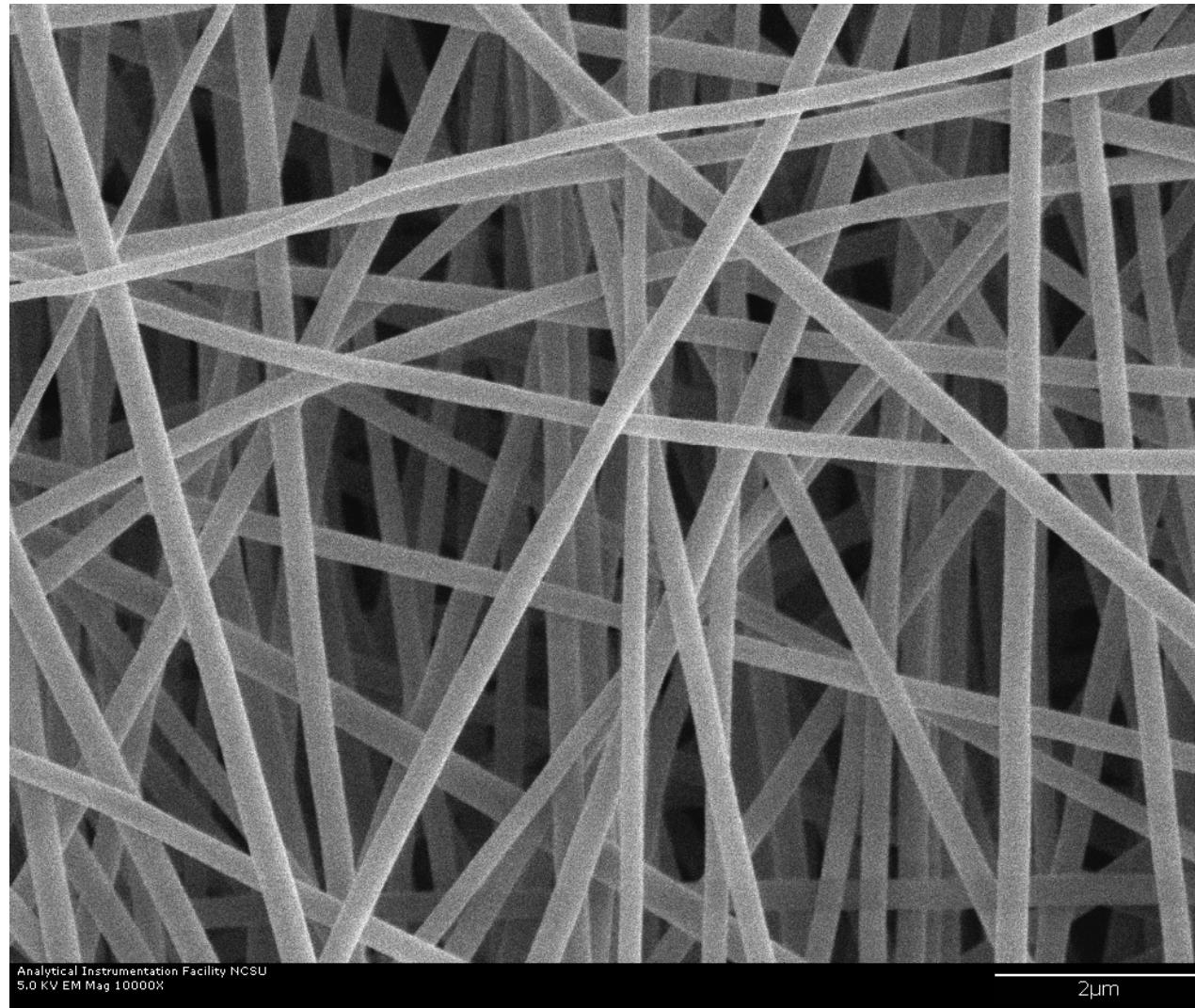
SEM Imaging Done Right...

Aluminum
Foam



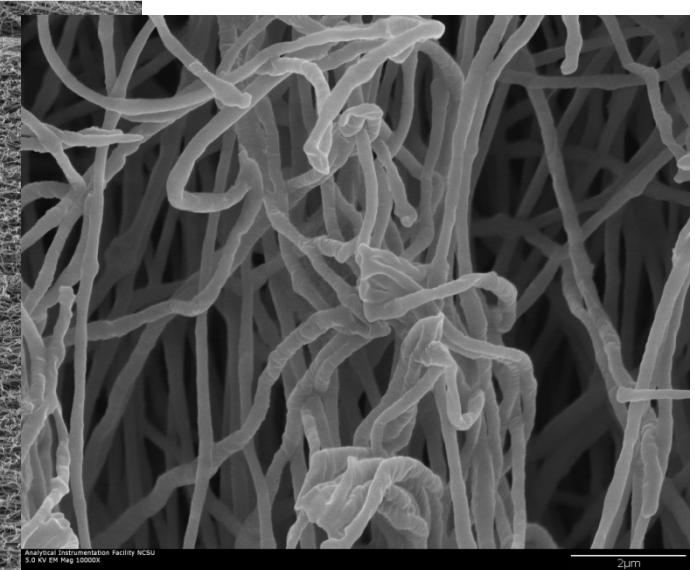
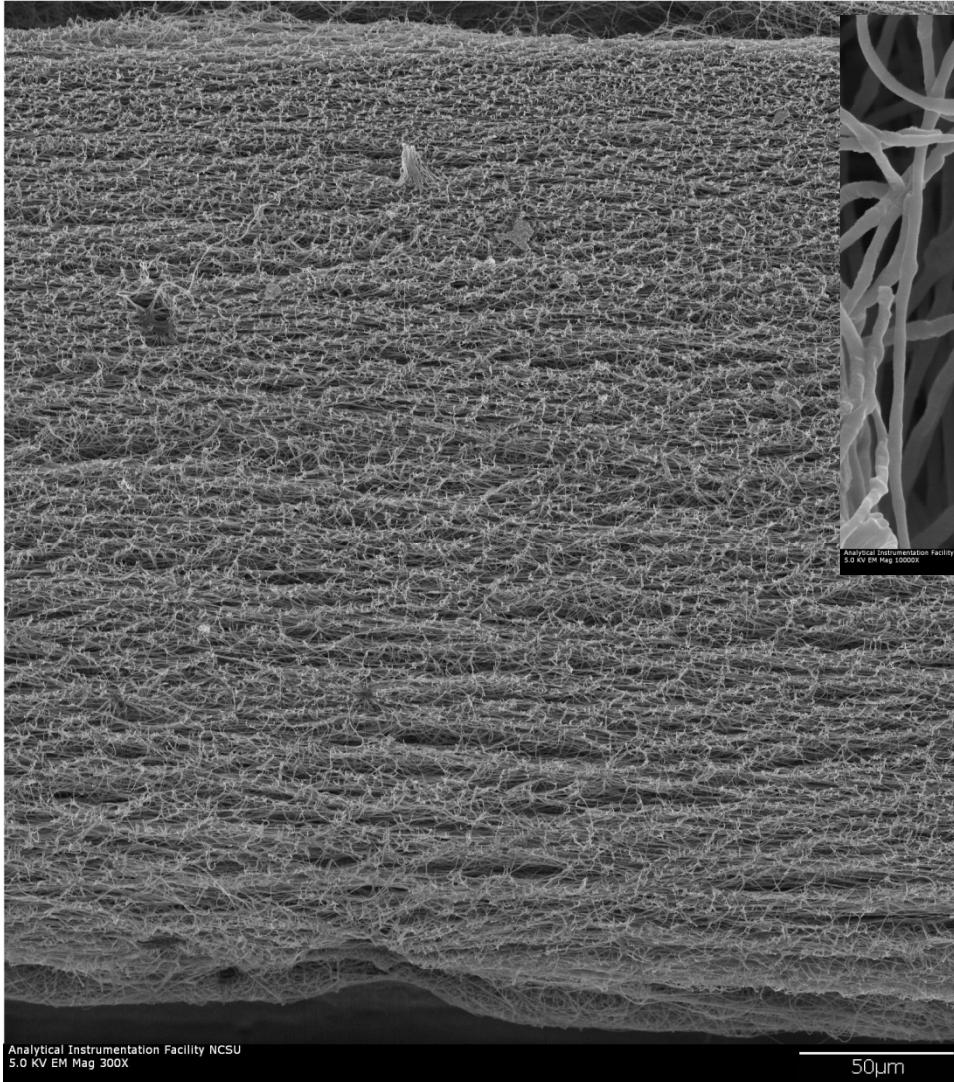
SEM Imaging Done Right...

Electrospun
Nanofibers



SEM Imaging Done Right...

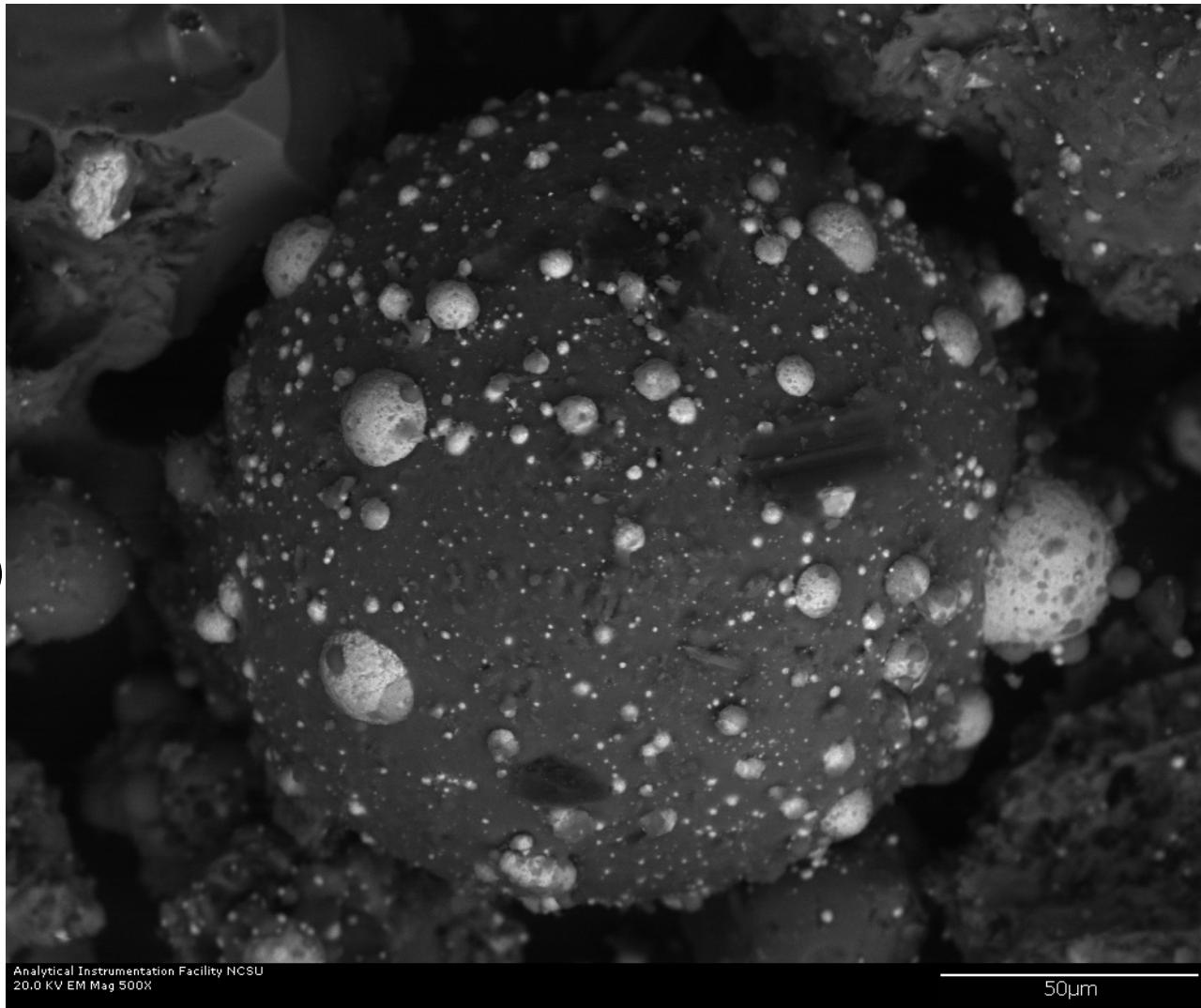
Cross-section
of thick
electrospun
nanofiber mat



High Magnification
detail

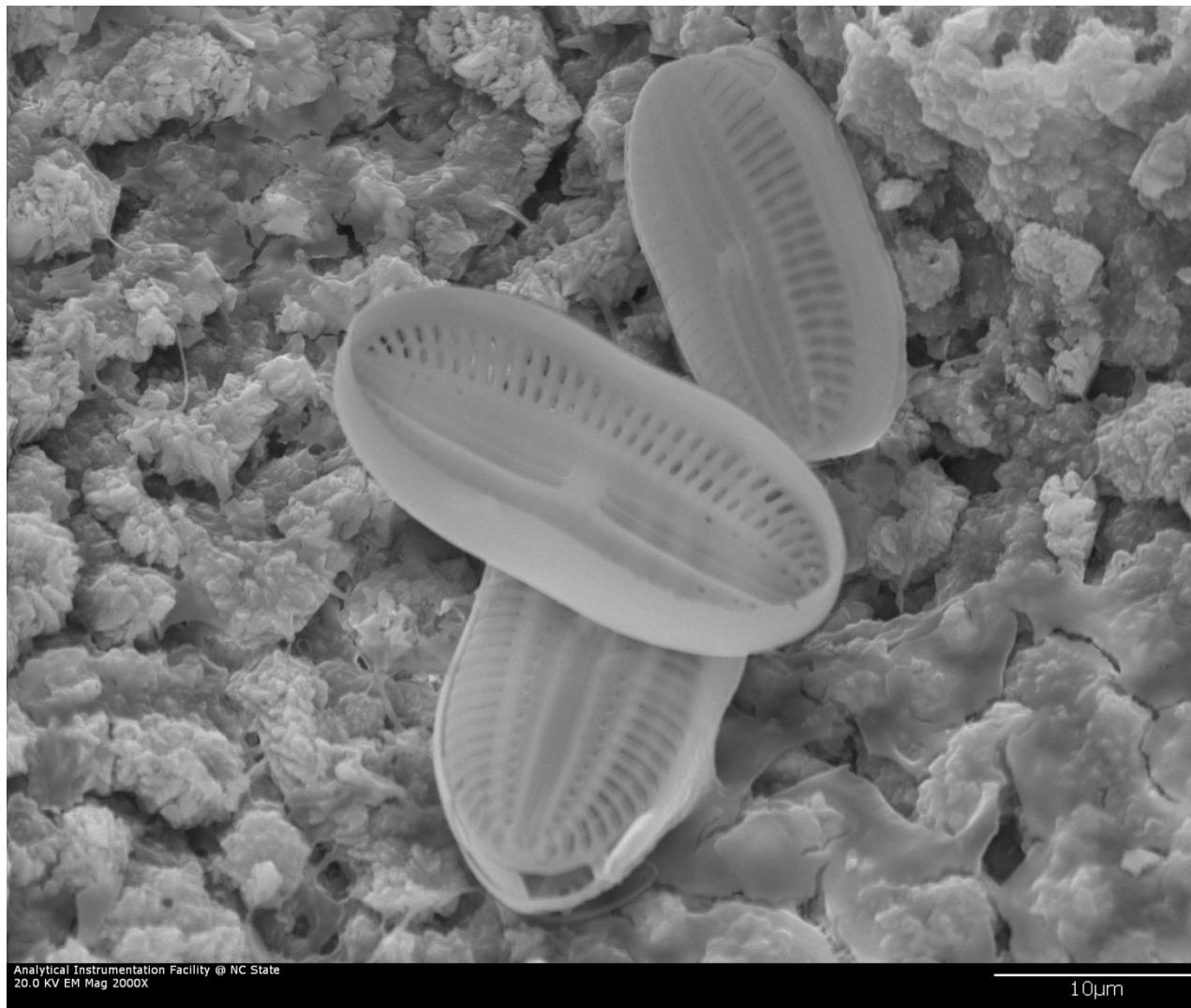
SEM Imaging Done Right...

Carbonaceous
sphere with
metallic
spheroidal
inclusions
(BSE detector)



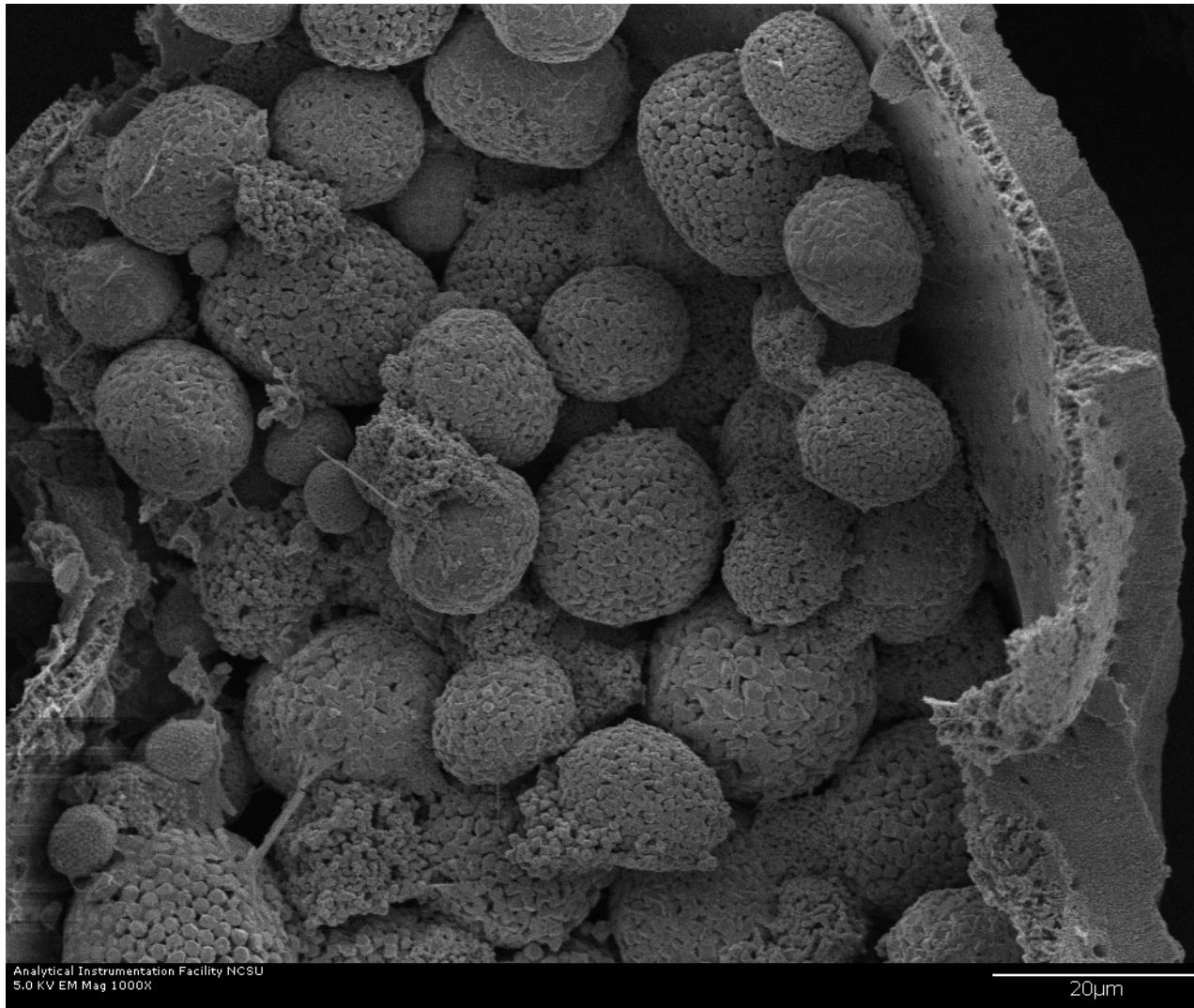
SEM Imaging Done Right...

Diatomes
on Coral



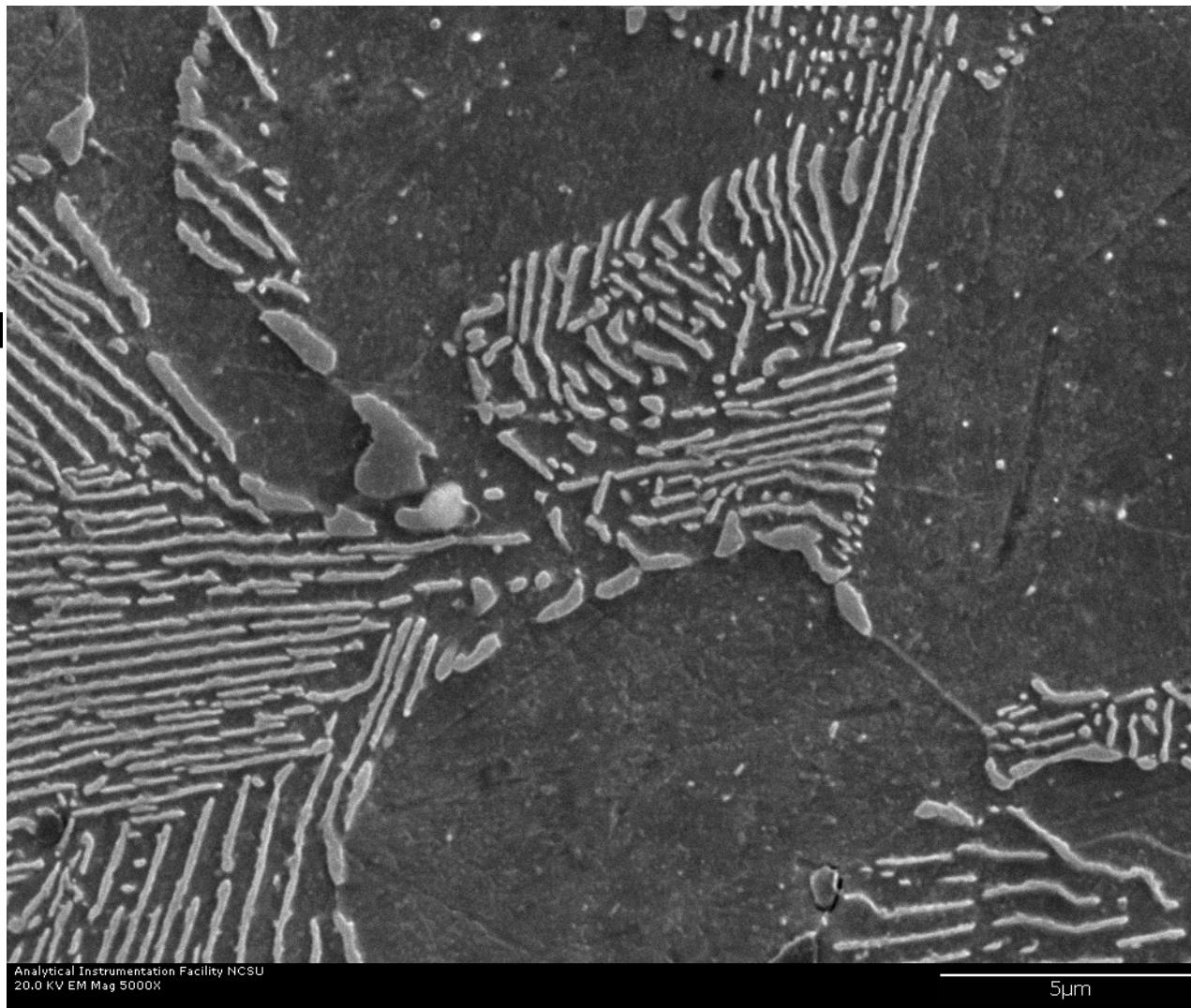
SEM Imaging Done Right...

Fossilized
Diatomes



SEM Imaging Done Right...

Polished
and etched
pearlite



Only a Taste of SEM...

For More Information see:

J.Goldstein, et. al., Scanning Electron Microscopy and X-ray Microanalysis, New York, Plenum, 2003



X-ray Analysis in the SEM

X-ray microanalysis

- Why do we care about X-rays in the SEM?
 - X-rays can be used to identify elements in the sample
 - If we are clever, we can learn a great deal about the sample's composition by collecting the X-rays already inside the SEM
- If we impinge a sample with an electron beam of sufficient energy, X-rays are naturally produced
 - If we are already imaging a sample with high energy electrons, then the X-rays are already there waiting to be collected, i.e., free data!
 - Not really free, the detector adds a cost and the time to collect X-rays adds cost
- X-rays are a desirable signal
 - Compliment BSED compositional contrast with elemental composition!
 - X-rays can be used to make a compositional map of a sample!

X-ray microanalysis

- X-ray microanalysis is used for:
 - 1.) Qualitative Analysis
 - 2.) Standardless Quantitative Analysis
 - 3.) X-ray based elemental mapping
and sometimes, if you absolutely have to know with great confidence
 - 4.) Quantitative Analysis with Standards
- More than ½ of SEM's have X-ray detectors!
 - IMNSHO no SEM is complete without an X-ray detector
 - Only exception might be an SEM in biology

X-rays: Physics Review

- X-rays are photons that are longer in wavelength than UV and shorter than gamma rays
 - The range is not set in stone and varies according to author
 - The only photon wavelength range that is agreed upon is the visible light range
 - X-ray wavelength range is in the nm scale: 10 nm to about 10 pm
 - X-ray Energy range is typically in keV: 100 eV to about 100 keV
 - X-ray frequency range: 10^{16} Hz to about 10^{19} Hz
 - Energy and wavelength have an inverse relationship
 - Photons: $E = h\nu = hc/\lambda \rightarrow \lambda = hc/E$
 - Electrons: $E = p^2/2m \rightarrow \lambda = h/\sqrt{2mE}$
- where: λ is wavelength, ν is frequency, E is energy, p is momentum, m is mass, c is speed of light, and h is Planck's constant

X-rays in the SEM

X-rays are produced in the SEM by high energy electrons interacting with the sample.

Two types of X-rays produced:

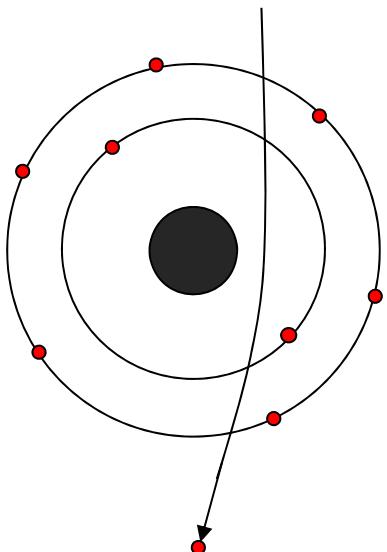
- Bremsstrahlung
 - German for Braking Radiation
- Characteristic X-rays
 - Allow identification of elemental species

X-ray Spectrum

- An X-ray spectrum can be used to determine the elemental species of a sample illuminated by a high energy electron beam
 - X-rays are produced wherever the electron beam strikes
 - A point or wide area probe can be used to create a spectrum
 - A scanned probe can be used to create an X-ray map of the sample
- An X-ray spectrum is data in the form of an XY plot with the Y axis being the number of X-ray counts and the X axis being the X-ray energy
- X-ray spectra contain both bremsstrahlung and characteristic X-ray data
- Bremsstrahlung is the background
- Characteristic X-rays form the peaks in the spectra and identify elemental species

Bremsstrahlung

- Bremsstrahlung is German for braking radiation
- As high energy electrons are decelerated in the Coulombic field of the atoms of the sample, they shed energy in the form of X-ray photons
 - The primary beam electron is being decelerated as it moves through the Coulombic field of an atom



C. Mooney

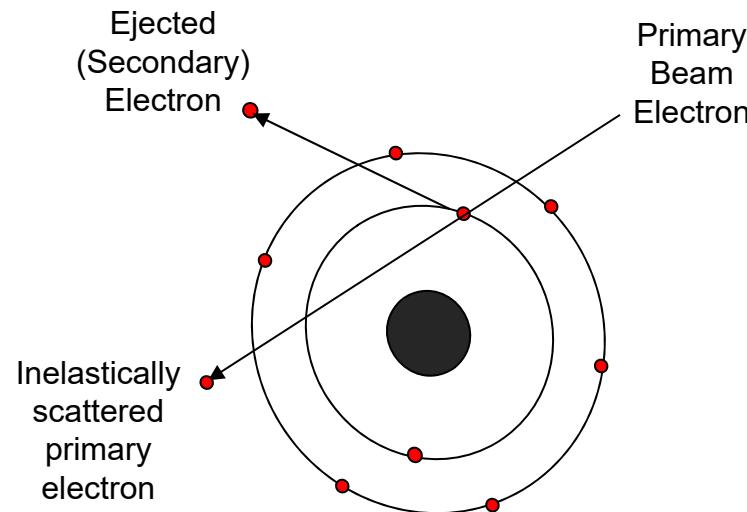
Bremsstrahlung

- Bremsstrahlung are not characteristic of any of the elemental species in the sample
- Bremsstrahlung X-rays form a continuum of energy from zero to the energy of the beam
 - Many bremsstrahlung counts at low energy
 - Very few bremsstrahlung counts at the beam energy
- Bremsstrahlung is not to be confused with noise!
 - Noise in an X-ray spectrum are extraneous counts that do not come from detected X-rays
 - Bremsstrahlung X-rays are real X-rays

Characteristic X-rays

- Characteristic X-rays are produced by inelastic electron scattering events producing inner shell ionization
 - When an inner shell electron is ejected from the atom, the atom becomes ionized
 - Ionized atoms are in an excited state
 - De-excitation of the ion, i.e., another electron moving into the hole in inner shell electron space, can create X-rays that are characteristic of the atomic species

Schematic of an inelastic scattering event



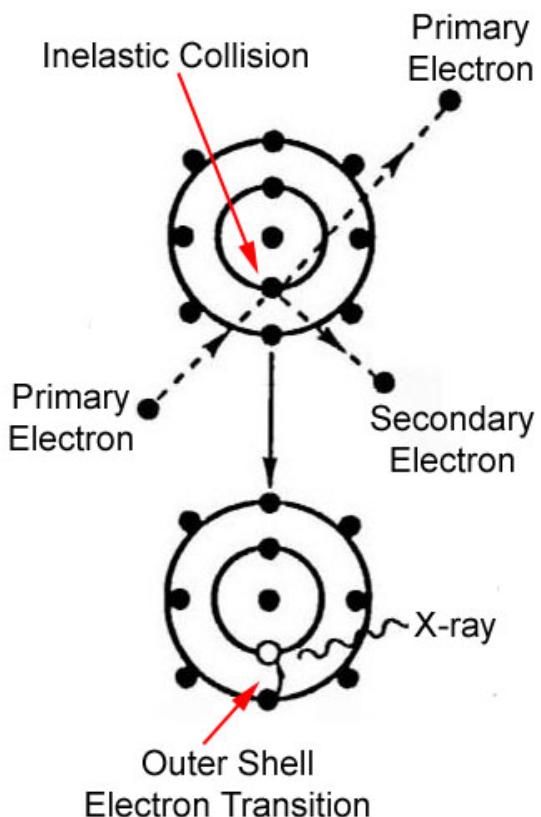
C. Mooney

Inner Shell Ionization

- The transition from one shell to another involves a change in energy
- Since orbital electrons must have specific energies, the transitioning electron must give up energy equal to the difference in energy between its outer shell position and the inner shell position
- The energy released from the atom can manifest itself either in the form of a characteristic (X-ray) photon or an ejected characteristic (Auger) electron

Characteristic X-rays

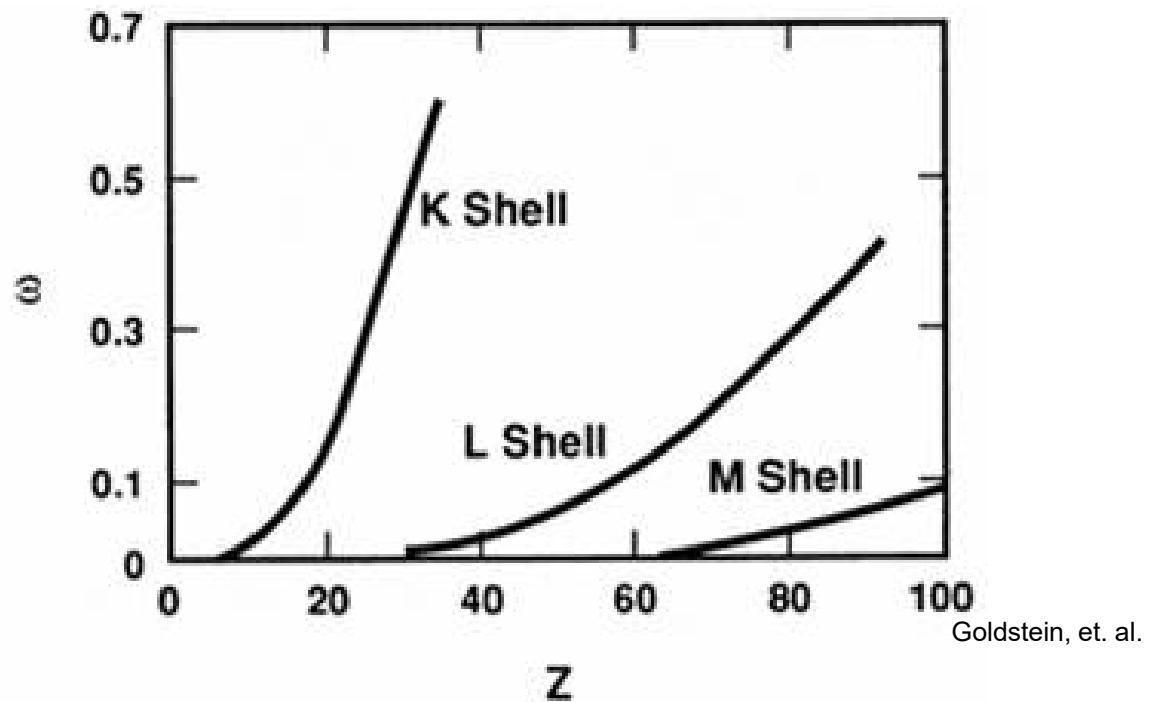
- A primary beam electron ejects (inelastic scattering) an inner shell electron from an atom in the sample
- An outer shell electrons transitions to the inner shell position, releasing an X-ray photon with energy equal to the transition energy (the energy difference between the inner shell electron and the outer shell electron)
- The characteristic X-ray photon is emitted by the atom
 - Characteristic because the energy is equal to the transition energy and can be used to identify the element



Adapted from
Goldstein, et. al.

Fluorescence (X-ray) Yield

$$\omega = \frac{\text{# of X-rays produced}}{\text{# of ionization events}}$$



Plot of fluorescence yield, ω , vs. atomic number

- Fluorescence (X-ray) yield + Auger yield = 1
 - An ionization event will result in either an X-ray or an Auger electron
 - Auger signal is usually higher than the X-ray signal!
 - Why aren't there more Auger instruments out there? Cost.

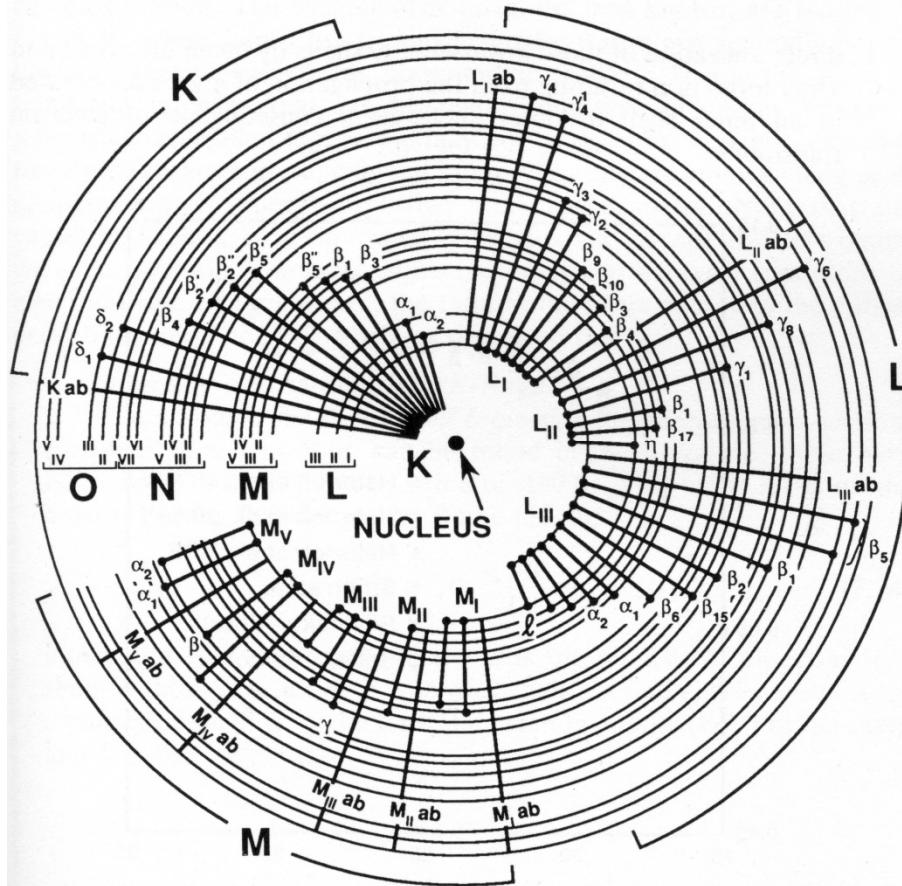
Siegbahn Notation

- Siegbahn notation is typically used in X-ray spectroscopy
 - Named for Manne Siegbahn, Swedish Physicist, 1924 Nobel in Physics
- This notation uses lower case Greek letters to designate X-rays from a particular shell transition in the general order of observed intensity: α , β , γ , etc.
- The X-ray line with the most probable transition is the α
- The X-ray line with the most probable transition will also be the highest peak in the family!
 - X-ray families are K, L, and M
- Not all transitions are assigned Greek letters
 - Example: Li and Ln are often observed in an X-ray spectrum

Seigbahn Notation

- For the K-shell only $K\alpha$ and $K\beta$ are possible (at SEM energies! $K\gamma$ X-rays possible)
 - Shells can be further divided into subshells, so $K\alpha_1$, $K\alpha_2$, $K\beta_1$, $K\beta_2$, ..., $K\beta_5$, etc., are possible
 - Many transitions are close enough in energy that they cannot be resolved by EDS
 - For standard SEM X-ray analysis (EDS) the $K\alpha_1$ and $K\alpha_2$ are typically right on top of each other and unresolvable – we only see a $K\alpha$ (having two X-rays close together may change the shape of the peak!)
 - The same is generally true for the $K\beta$ series
- There are multiple L-shell and M-shell transitions
 - At high X-ray energies (5 keV +), the various lines are typically resolvable
 - At low X-ray energies (< 5 keV), the various lines are not typically resolvable

Comprehensive Energy Level Diagram



Goldstein, et. al.

Comprehensive X-ray Energy Level Diagram showing all electron transitions that lead to K, L, and M series X-rays

Critical Ionization Energies

- Critical Ionization Energy is the energy transfer required to form a particular X-ray
- Every transition has its own particular critical ionization energy
- Also called absorption edge
 - K edge
 - L edge
 - M edge

Edge	Absorption Edge (keV)	X-ray energy (keV)	Edge	Absorption Edge (keV)	X-ray energy (keV)
K	69.525	59.305	K	8.328	7.477
L	12.100	8.396	L	1.008	0.851
M	2.820	1.774	M	--	--

Absorption Edge and X-ray
Energies for W (74)

Absorption Edge and X-ray
Energies for Ni (28)

Energy Dispersive X-ray Spectroscopy

- Energy dispersive spectroscopy collects all of the X-rays at once and sorts them by energy
- An X-ray strikes a Si detector and creates a charge pulse that is proportional to the energy of the X-ray
- The energy sorting process is accomplished through the electronic pulse processor
 - The design of the electronics is very important to the operation of the spectrometer
- The pulse processing induces significant artifacts on the collected spectrum
 - Most of the appearance of the spectrum is an artifact!

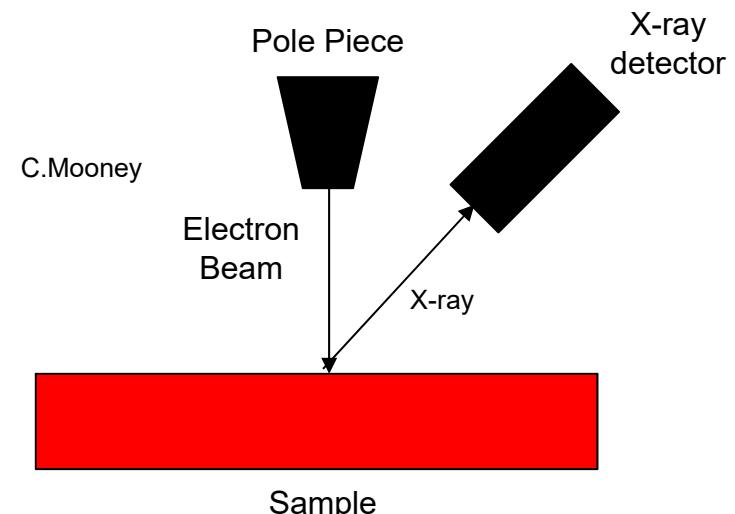
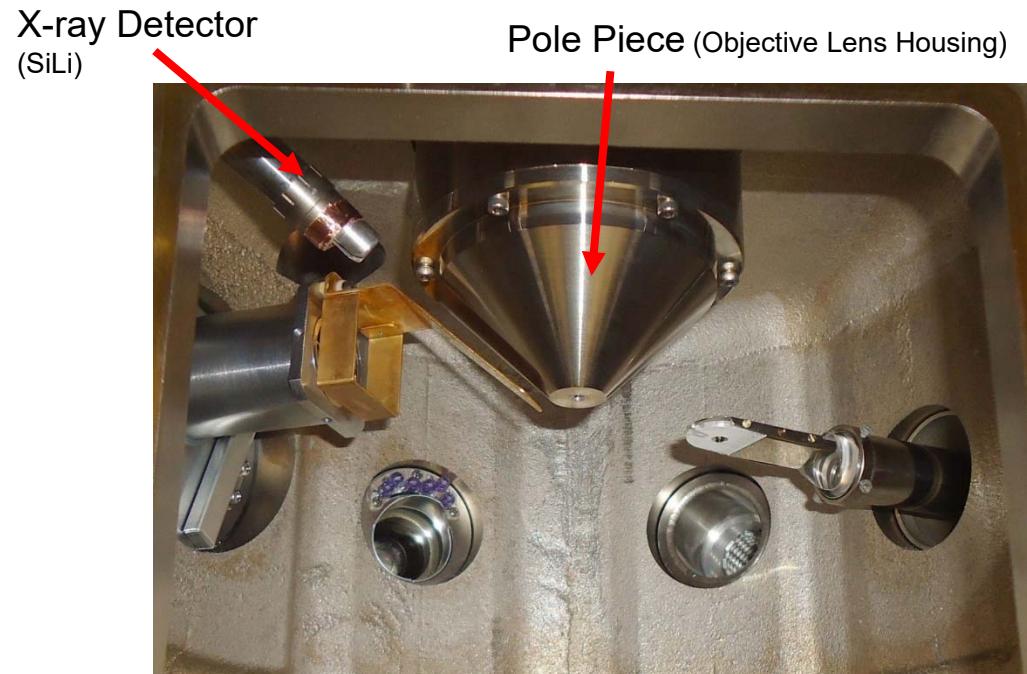
Energy Dispersive X-ray Spectroscopy

- EDS has poor energy resolution, ~130 at the Mn K α (5.9 keV) line
- Low energy X-ray analysis has issues
 - Oxford instruments is pushing low energy EDS now
 - Many overlaps making it difficult for use with many materials

If the spectrum is mostly artifact, why do so many SEMs (>50%) have EDS detectors?

- There is a single detector that detects the X-rays
 - WDS = multiple crystals, usually multiple detectors
 - EDS has much lower overall cost
- All the X-rays are collected at once (faster! – time is money!)
- Determining the unknown contaminant is relatively quick and easy

EDS System



- Typical EDS detectors are inserted at an angle and come in next to the pole piece
- This reduces the chances of crashing the stage or sample into the detector



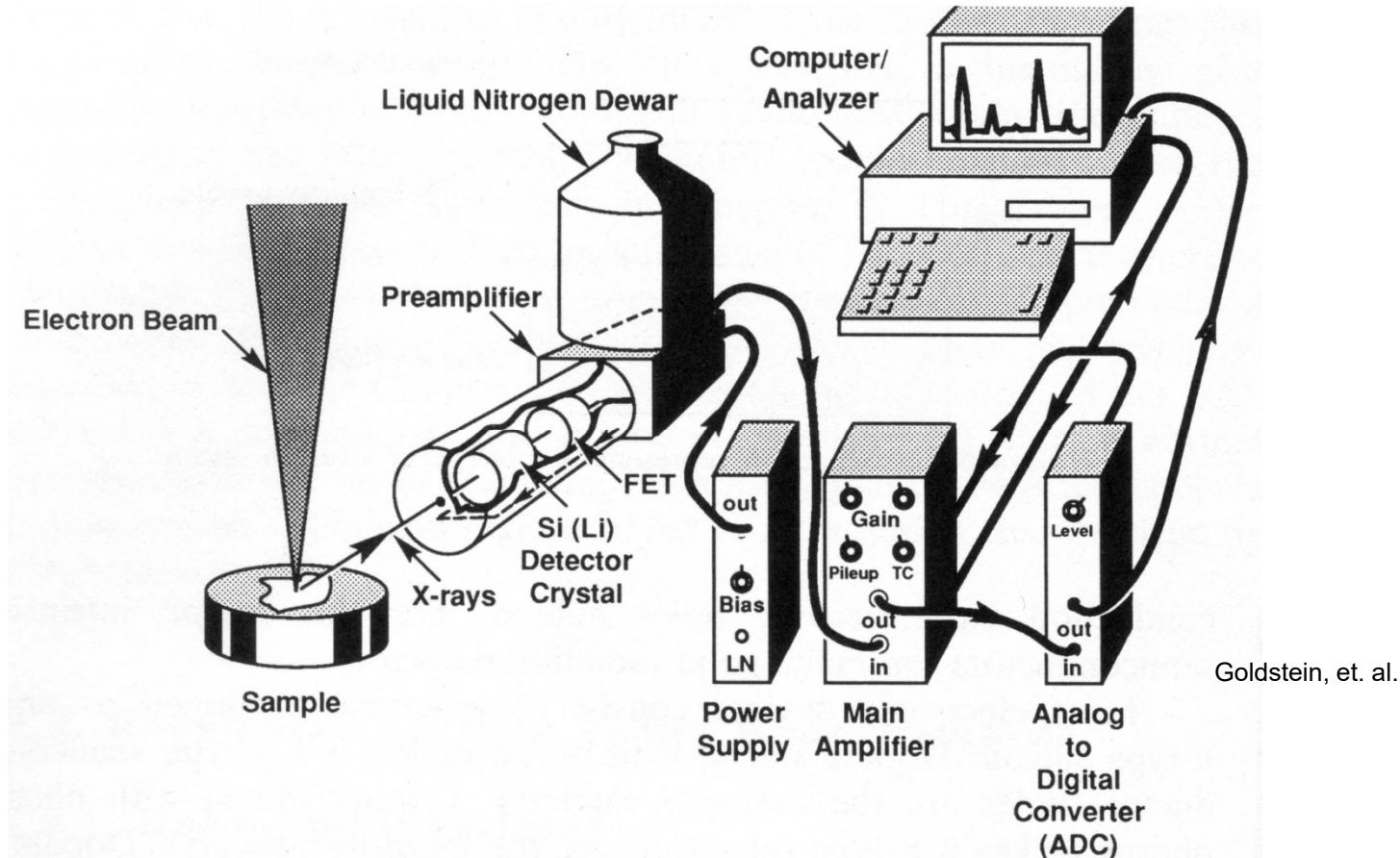
X-ray Detection with an EDS detector

1. X-rays from the sample pass through a protective window and strike the detector crystal
 - Why a window? The detector is cooled: Window protects the detector from contamination collecting on the cold surface
 - Old school detectors had Be windows – absorbed energies below 1 keV, so no elements below Na ($K\alpha = 1.04$ keV) could be detected
 - Modern detectors use ultrathin polymer windows that absorb little energy, although they will absorb Li X-rays
 - Beware: Some polymer windows have an absorption edge close to the N $K\alpha$ X-ray (0.392 keV) and will absorb most, if not all N X-rays while letting Be, B, and C (0.108, 0.185, 0.277 keV) through
2. Absorption of each X-ray by the detector provides the energy required to boost electrons into the conduction band, leaving holes behind
 - The electron-hole pairs are charge carriers
 - 3.86 eV required to promote an electron into the conduction band from Si
 - X-ray energy is proportional to the number of charge carriers produced!

X-ray Detection with a EDS detector

3. An applied electric field sweeps the electron-hole pairs apart to form a charge pulse
 - SiLi detectors used a thin Au contact on the front face of the detector
 - SDDs use microfabricated rings of conductor on the back of the detector with a large cathode contact on the front
4. The charge pulse is converted to a voltage pulse by a charge to voltage converter (or preamplifier)
 - Field effect transistor (FET) preamp
 - Why convert? A voltage pulse is easier to transmit down a wire to the rest of the electronics than a current pulse (no worries about current loses)
 - Always remember: Resistance isn't futile. Resistance is voltage divided by current.
5. The voltage pulse is then processed and shaped by a pulse processor to form the spectrum
 - Voltage peak is proportional to X-ray energy
 - Modern pulse processors are digital

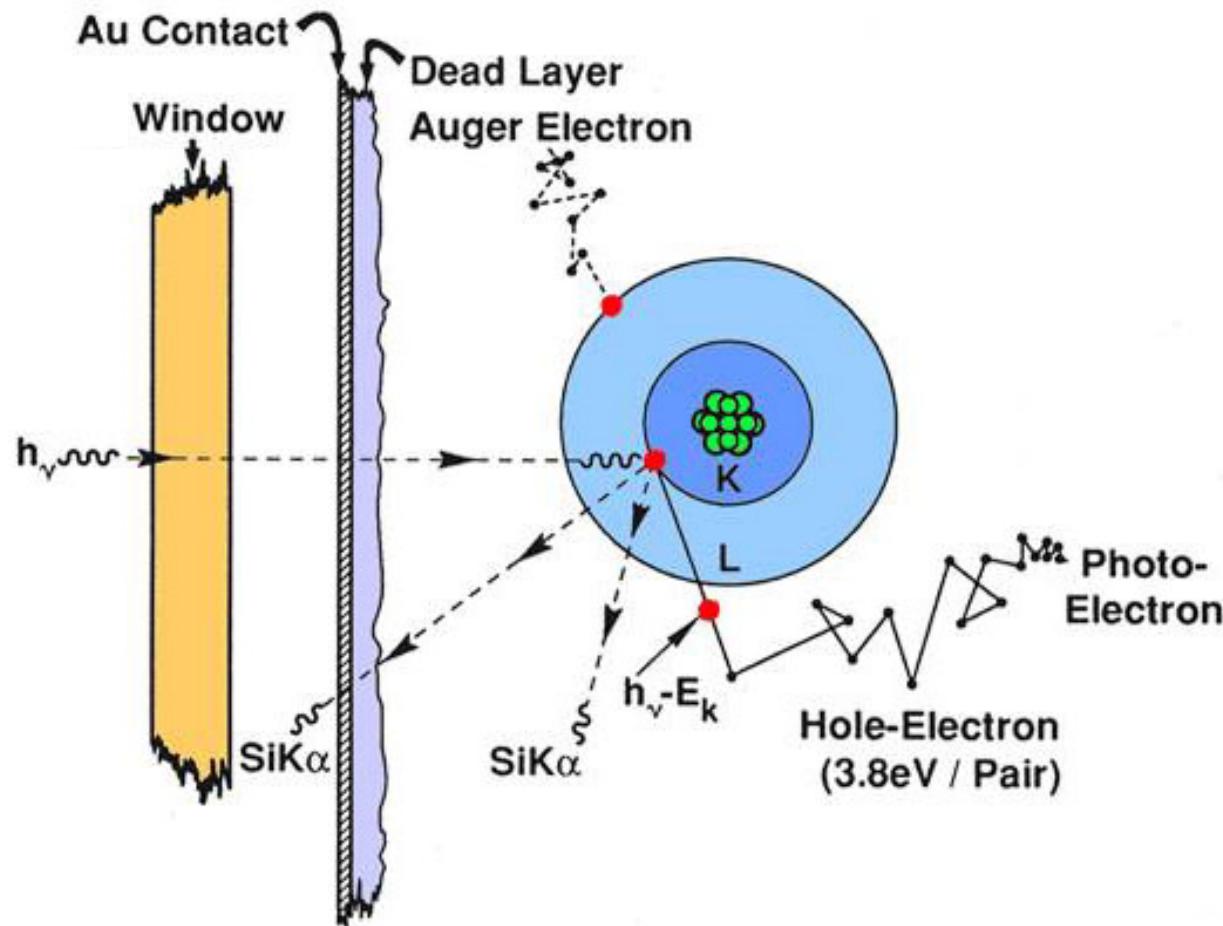
EDS Spectrometer



Goldstein, et. al.

Schematic of a typical EDS system using a SiLi detector

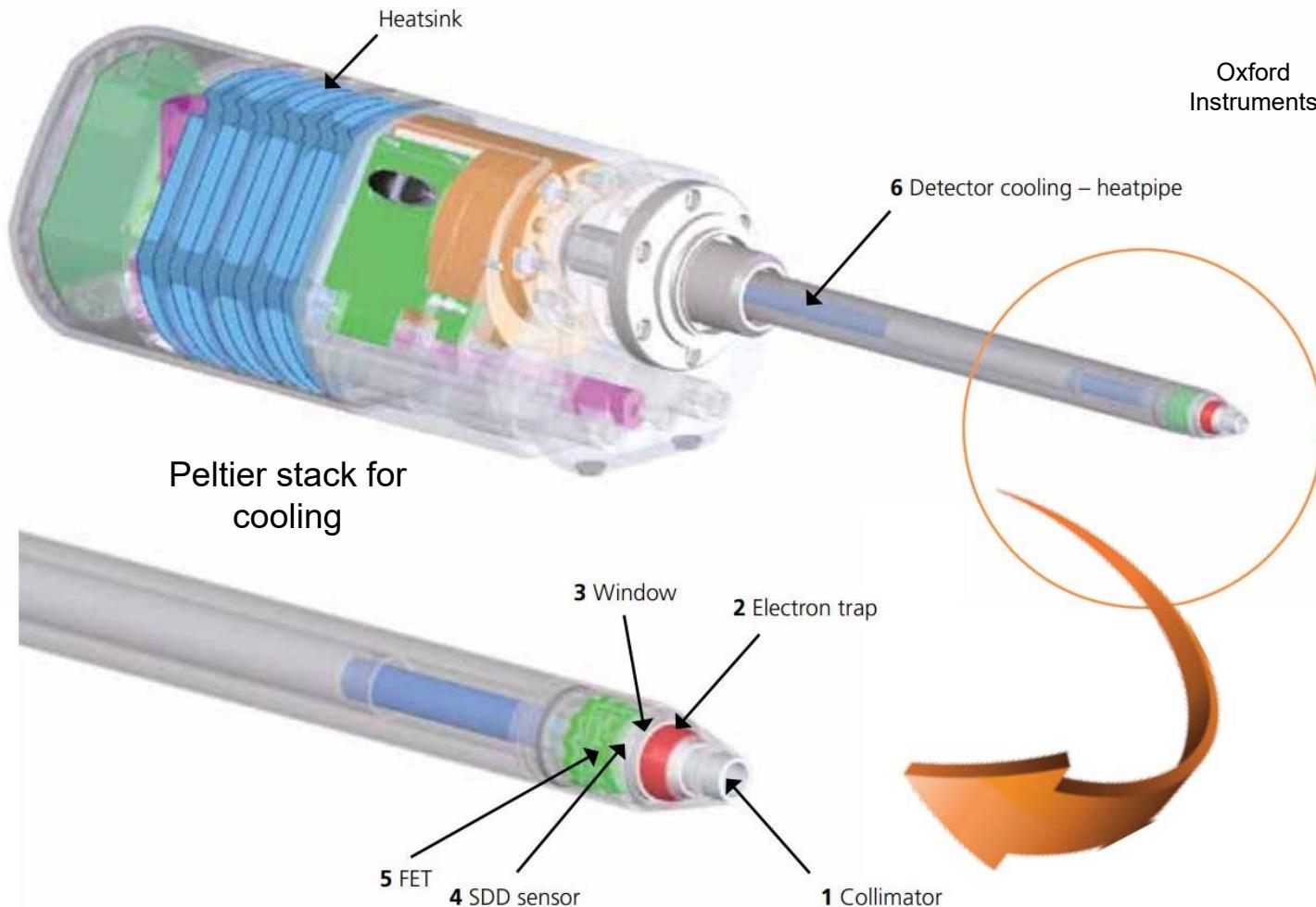
X-ray detection process



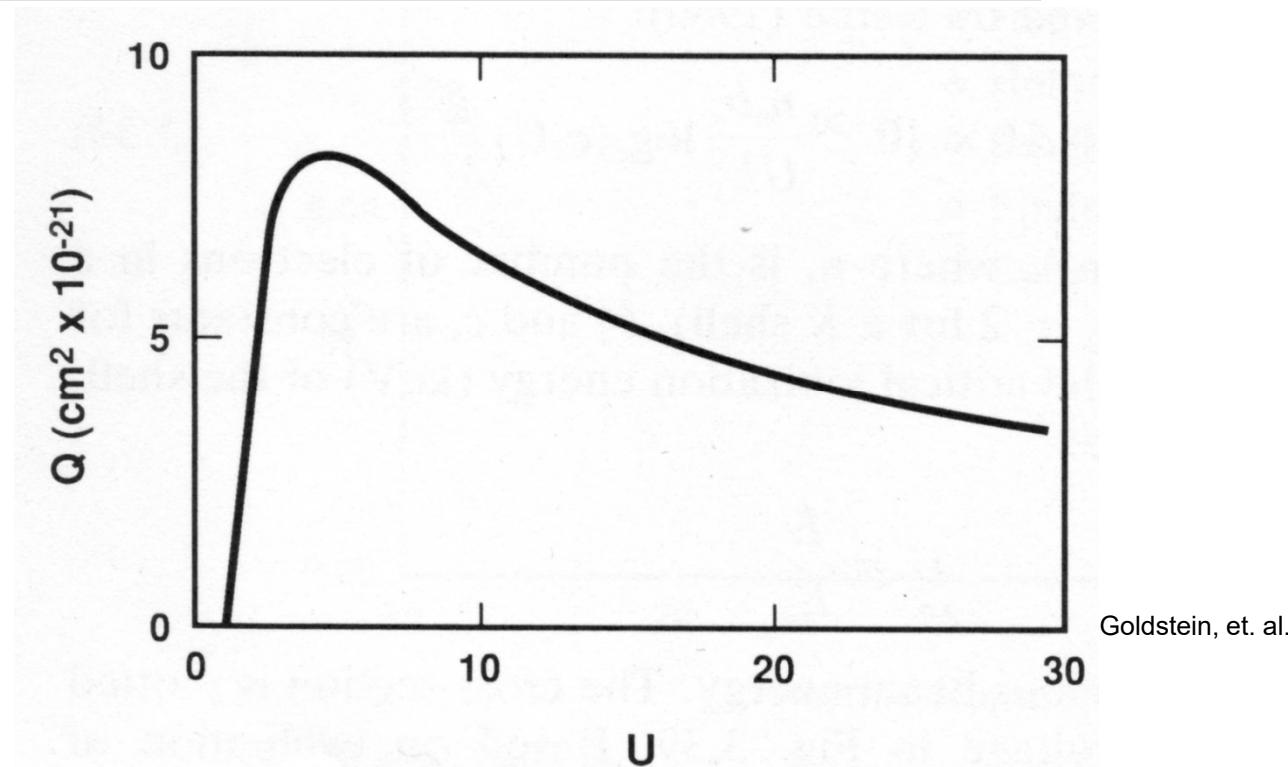
Goldstein, et. al.

Schematic of the X-ray detection process
SiLi detector shown

Modern SDD EDS Detector



Cross Section for inner-shell ionization



Plot of cross-section for inner shell ionization as a function of overvoltage U , where $U = E/E_c$. This plot suggests that the optimum energy for producing the most photons is approximately three times the critical ionization energy or $U \sim 3$ to 5 . As $U > 5$, the cross section begins to diminish. Note that the cross section is very small for $E \sim E_c$.

Weights of X-ray Lines

- While there are many different transitions that can produce X-rays, the probability of those transitions varies considerably
- This relative probability difference will manifest as differing count rates while collecting EDS data
 - Higher peaks = higher transition probability
- This gives rise to the relative weights, or probabilities, of a transition
- The weights vary in a complex fashion by atomic number
 - The weights of K lines are well known
 - The weights of L and M lines are not so well known*

*EDS instrument manufacturers know, but they may not necessarily publish all their hard won data in a publicly available database.

Weights of X-ray Lines

- A table of approximate weights of X-ray lines is shown below
- These are only approximate, but can be useful for interpreting X-ray data
 - K lines are well known and the displayed relative peak heights should match the actual peaks
 - L and M lines are not so well known and displayed peak heights are usually based on the approximation below

Family Approximate Interfamily Weights

K

$$K\alpha = 1 \quad K\beta = 0.1$$

L

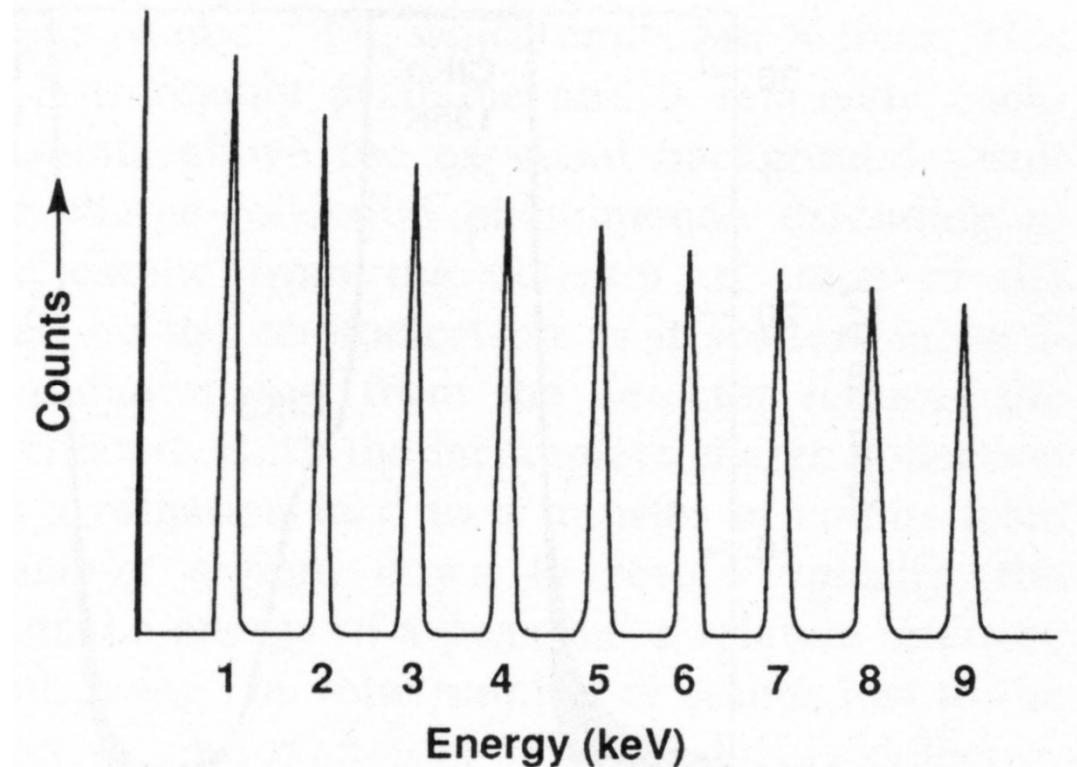
$$L\alpha = 1 \quad L\beta_1 = 0.7 \quad L\beta_2 = 0.2 \quad L\gamma_1 = 0.08 \quad L\gamma_2 = 0.03$$

$$L\gamma_3 = 0.03 \quad Ll = 0.04 \quad L\eta = 0.01$$

M

$$M\alpha = 1 \quad M\beta = 0.6 \quad M\zeta = 0.06 \quad M\gamma = 0.05 \quad M_{II}N_{IV} = 0.01$$

FWHM and amplitude versus Energy



Goldstein, et. al.

- EDS energy resolution is not constant with energy
- Peaks at higher energy will be broader and with lower peak intensity
(can't integrate area to quantify!)

Qualitative vs. Quantitative X-ray Analysis

- Qualitative analysis only measures what elements are in the sample
 - No attempt is made to know how much
 - Often this is all that is needed
- Quantitative analysis is trying to determine how much of each element is there based on the measured spectrum
 - There are multiple correction factors
 - Best if one uses standards (but this can be painful)
 - Quick and easy to use standard-less
 - So quick and easy it is often not used correctly

Qualitative analysis

- Define:
 - Concentration ranges of interest
 - Major constituents > 10 wt%
 - Minor constituents 1-10 wt%
 - Trace constituents < 1 wt%
 - Detection Limit constituents < 0.5 wt % (a subset of trace)
- Major constituents will have the tallest peaks (most counts) because more X-rays are generated from more material!
- Minor constituents will not have very tall peaks, but the peaks should be easily observable
- Trace constituents will have small peaks that are observable above background
- Detection limit constituents will have very subtle peaks that are just barely above the background (and may only appear after collecting many counts)

Basics of Qualitative Analysis

- Collect high quality data
 - Garbage in, garbage out
 - Think about the required overvoltage
 - 20 keV is a good place to start when in doubt
 - Think about the magnitude of the interaction volume
- Pay attention to the dead time and count rate
 - Keep it below 30%
 - 20% is usually the best compromise between waiting (dead time) and data collection
 - Typical count rate for a 10 mm² SiLi detector with 20% dead time is on the order of 2000 counts per second (cps)
 - Don't know the typical count rate for a 50 mm² SDD with 20% dead time because we never get there – stop turning up current when the count rates hit ~ 10 – 15 kcps (don't want to damage the sample with high current!)

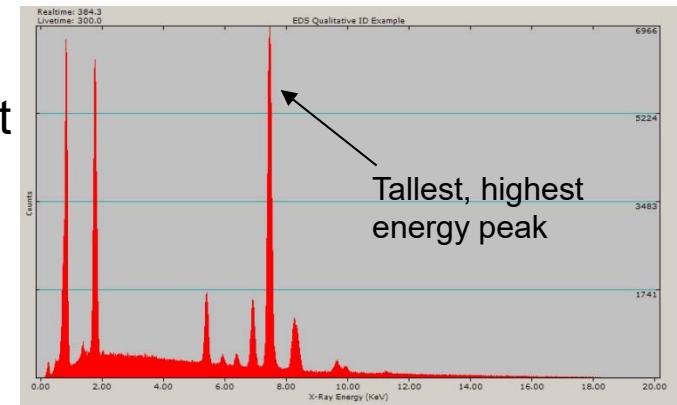
A Method for Qualitative Analysis

Most important:

- Account for ALL observed peaks
- If there is a peak, something caused it (probably not a new and unknown element)
- Consider Si escape peaks and sum peaks
 - Sum peaks can be from two or more elements at once
 - Escape peaks are observed at 1.74 keV below a major peak
 - Don't overdrive the detector (too much dead time)
- Know your pulse processor and software!
 - Different pulse processors seem to have different issues
 - Older Oxford systems seemed to favor sum peaks over Si escape peaks
 - Newer Oxford systems seem to identify low counts at 1.5 keV to be Br instead of Al (overlap) – this goes away as counts accumulate
 - Al is much more common than Br
 - Br has a K α peak at 11.2 keV

A Method for Qualitative Analysis

1. Start with the tallest peak
 - a. If two peaks have equal numbers of counts, start with the highest energy peak
 - b. Label **ALL** peaks associated with that element
 - c. Take care in identifying potential overlaps



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2. Move to the next tallest, high energy, repeat steps 1a – 1c
3. Repeat until **ALL** peaks are accounted for
 - If there is a peak, something caused it
 - Account for any sum or escape peaks

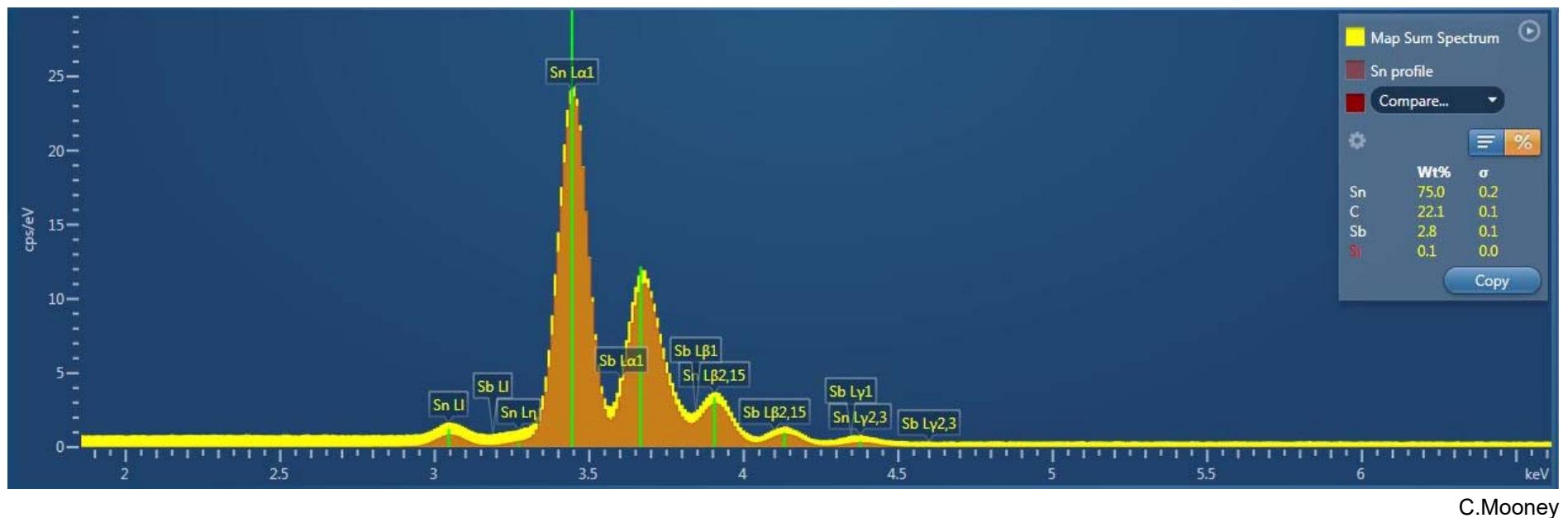
A Method for Qualitative Analysis

- Account for all peaks associated with a particular element before moving on to the next peak
- Check for overlaps with other elements
- If there is an overlap, what other peaks should one observe for each of the elements that has an overlap with the peak in question
- Usually high energy peaks have a lower energy counterpart
 - Example: If one observes major constituent Cu peaks at $K\alpha = 8.04 \text{ keV}$ and $K\beta = 8.90 \text{ keV}$, then one should also observe a set of (unresolved) Cu L peaks at 0.93, 0.95, 1.02, and 0.81 keV

EDS Peak Matching

- Modern EDS software allows for peak matching, i.e., the shapes of known elemental peaks can be shown in the software and matched to the unknown spectrum
 - This is better than mere line markers
 - Some elements with overlaps have different peak shapes, e.g., Mo and S can be identified based on peak shape even though there is a significant overlap
- Peak matching also aids in:
 - Identifying elements when there is an overlap in a complex sample
 - Determining if there is a minor or trace amount of material when there is an overlap, e.g., the Cr K β and Mn K α

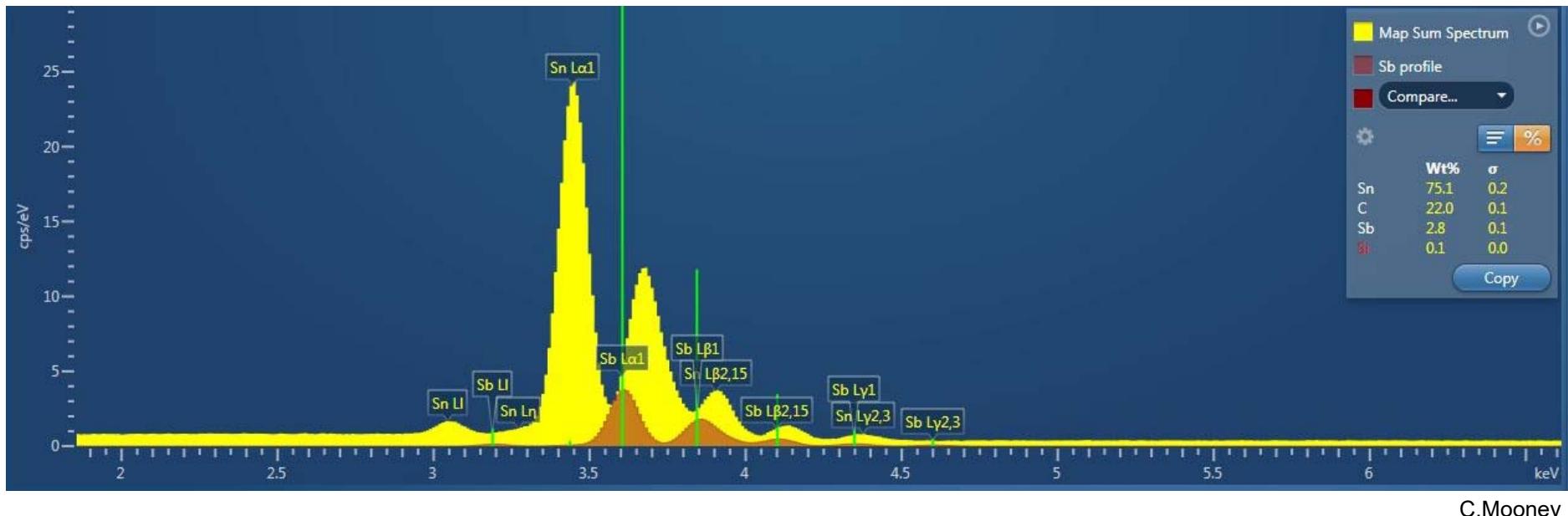
EDS Peak Matching



Detail of a spectrum of a Sn on C standard showing the Sn L family

- The auto ID thinks there is a trace of Sb (which is possible) in the Sn
- The peaks perfectly match the Sn
 - The L β 2 peak is a little too tall, which makes the system suspect an Sb overlap
 - Sn LI is too tall because of the background shelf

EDS Peak Matching



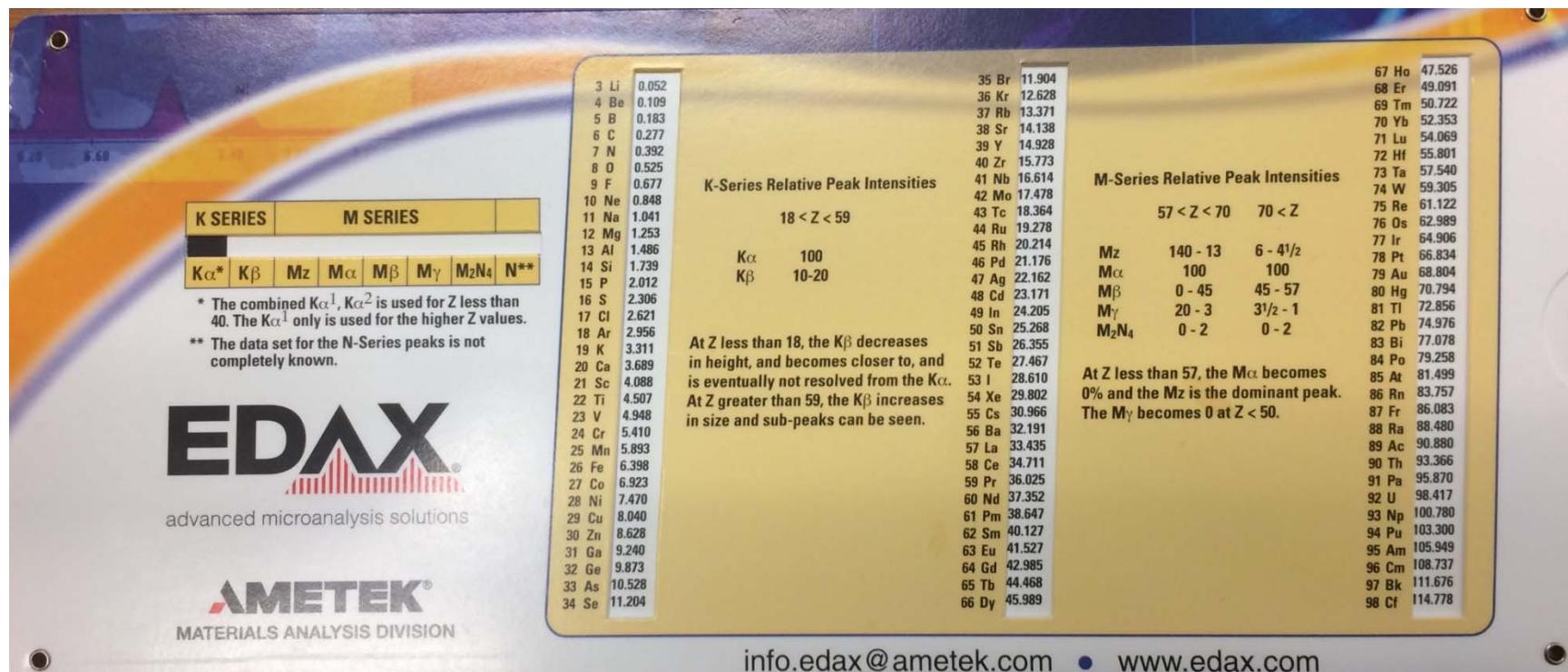
Detail of a spectrum of a Sn on C standard showing the Sb L family

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- The auto ID thinks there is a trace of Sb (which is possible) in the Sn
- The Sb peaks do not match up well
- If the trough between the Sn L β 1 and Sn L β 2 did not match the Sn L family and if there was a tiny peak between the Sn LI and Sn L α 1, then this might be believable
- As it is, the operator is unconvinced of more than a trace of Sb

Old School EDS Analysis

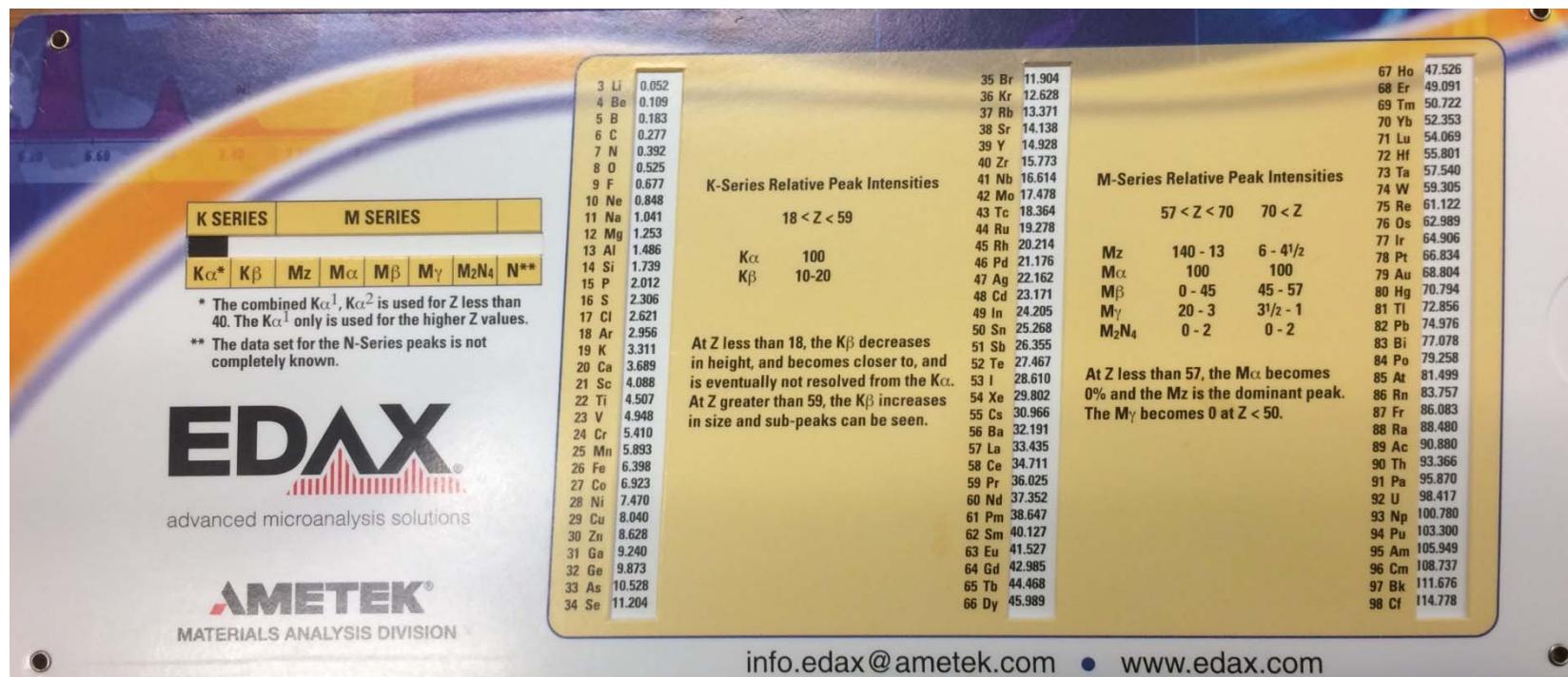
- Old school EDS analysis was done with either a table of X-ray energies or an EDS slide rule



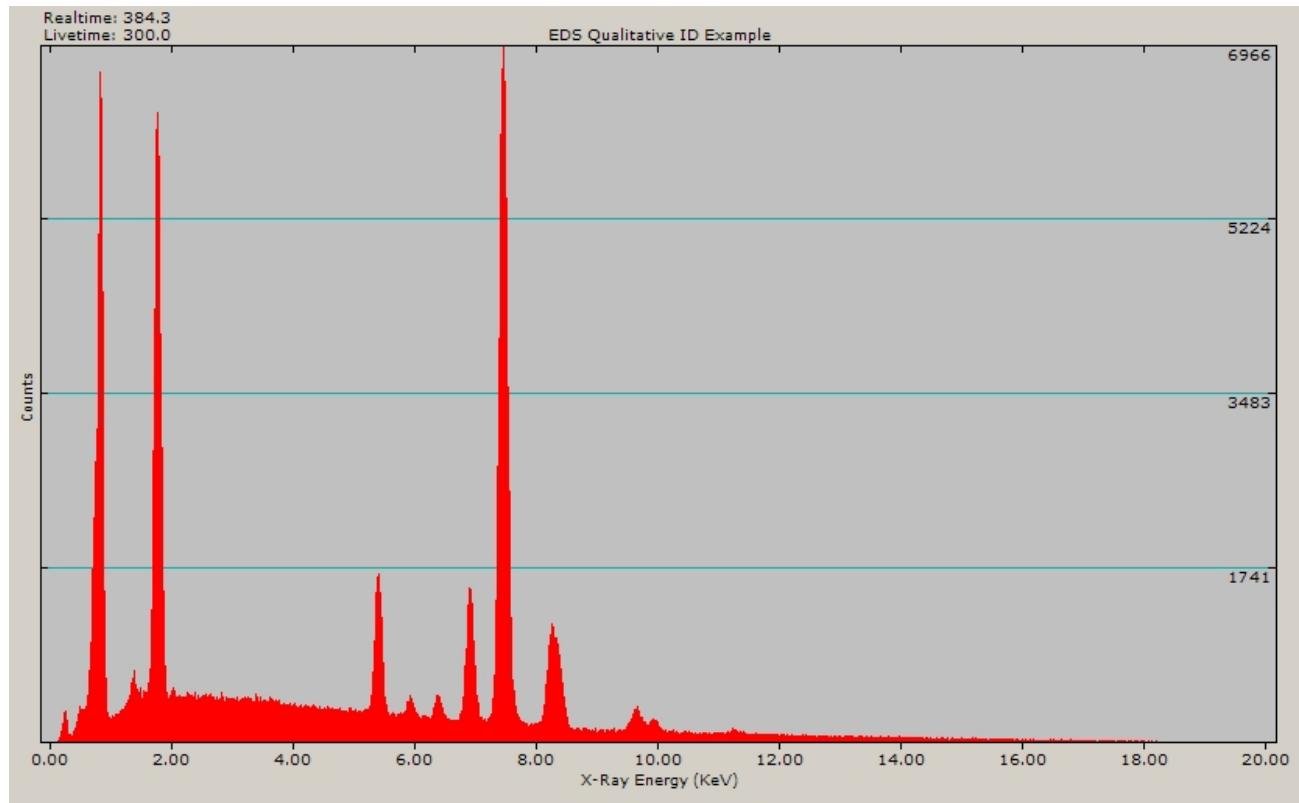
EDS energy slide rule. $K\alpha$ energies shown.

Old School EDS Analysis

- Using a slide rule or table is still the smart way to learn qualitative analysis
- ID the spectrum yourself until you feel comfortable
- Never trust the software!
- Trust your brain!

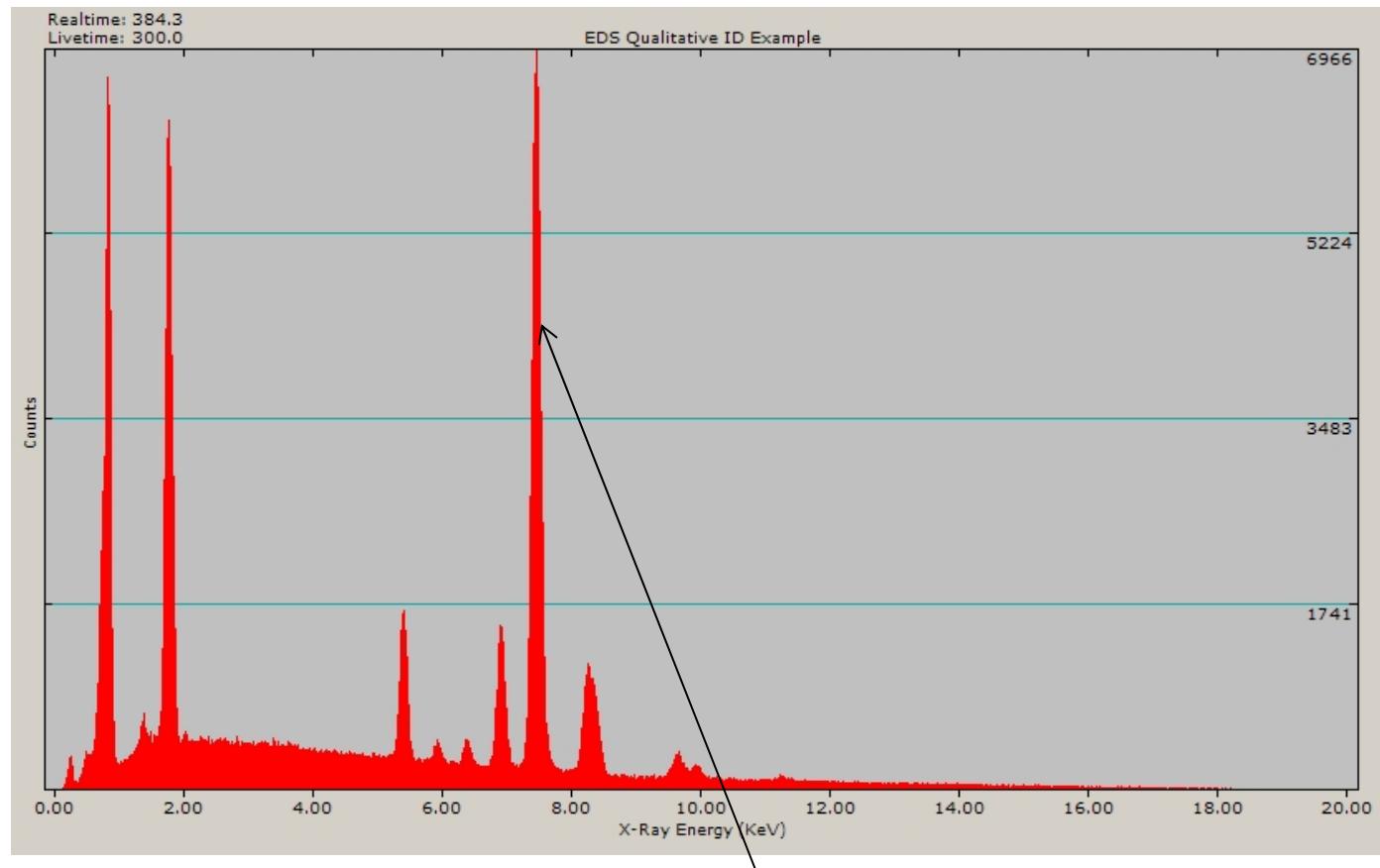


EDS Qualitative Analysis Example



- Example of an unknown metallic sample for analysis
- EDS spectrum collected at 30keV
- Step by step analysis using an X-ray energy slide rule (and X-ray line markers in the software!)

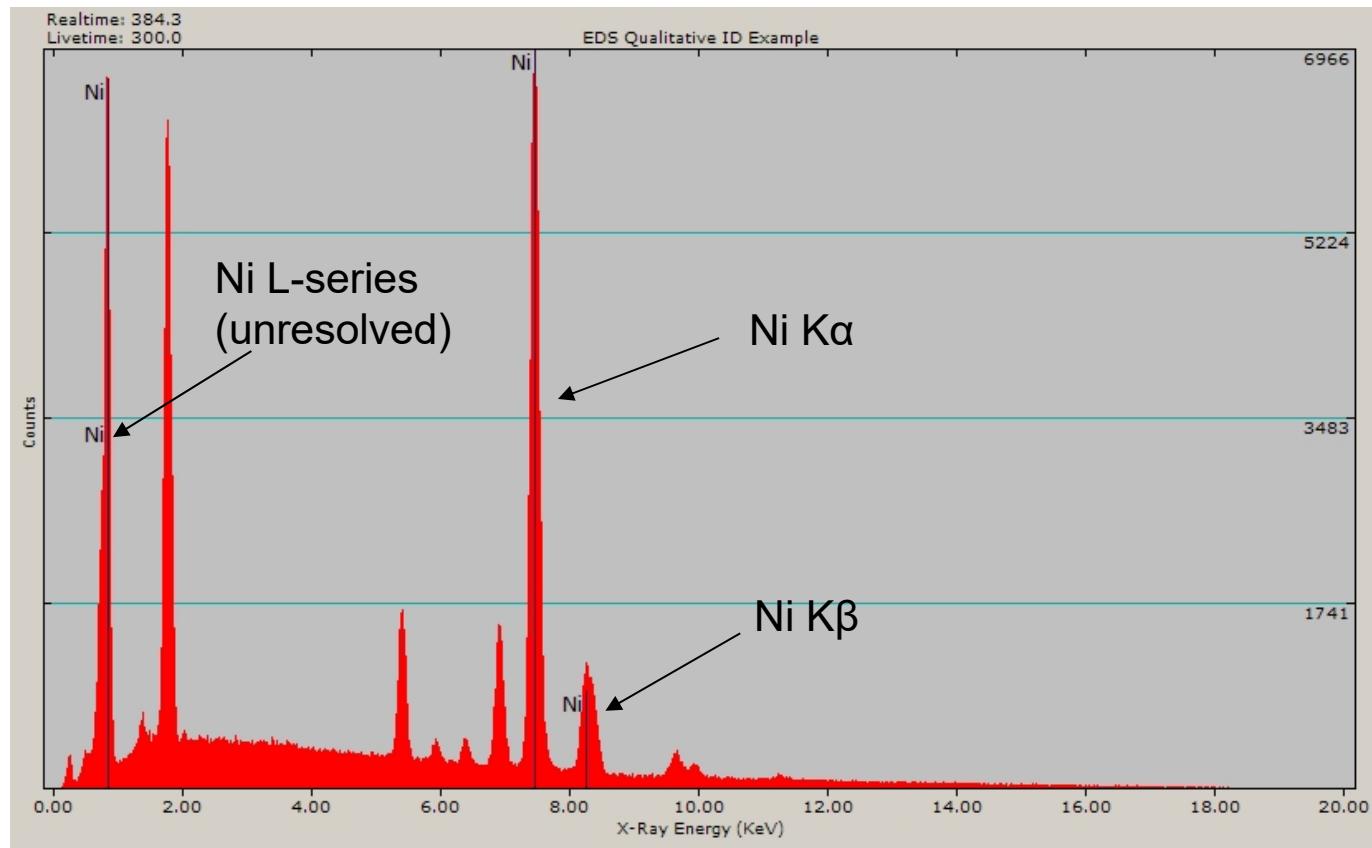
EDS Qualitative Analysis Example



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- Step 1: Identify the tallest, highest energy peak
- Energy ~ 7.5 keV = Ni K α peak

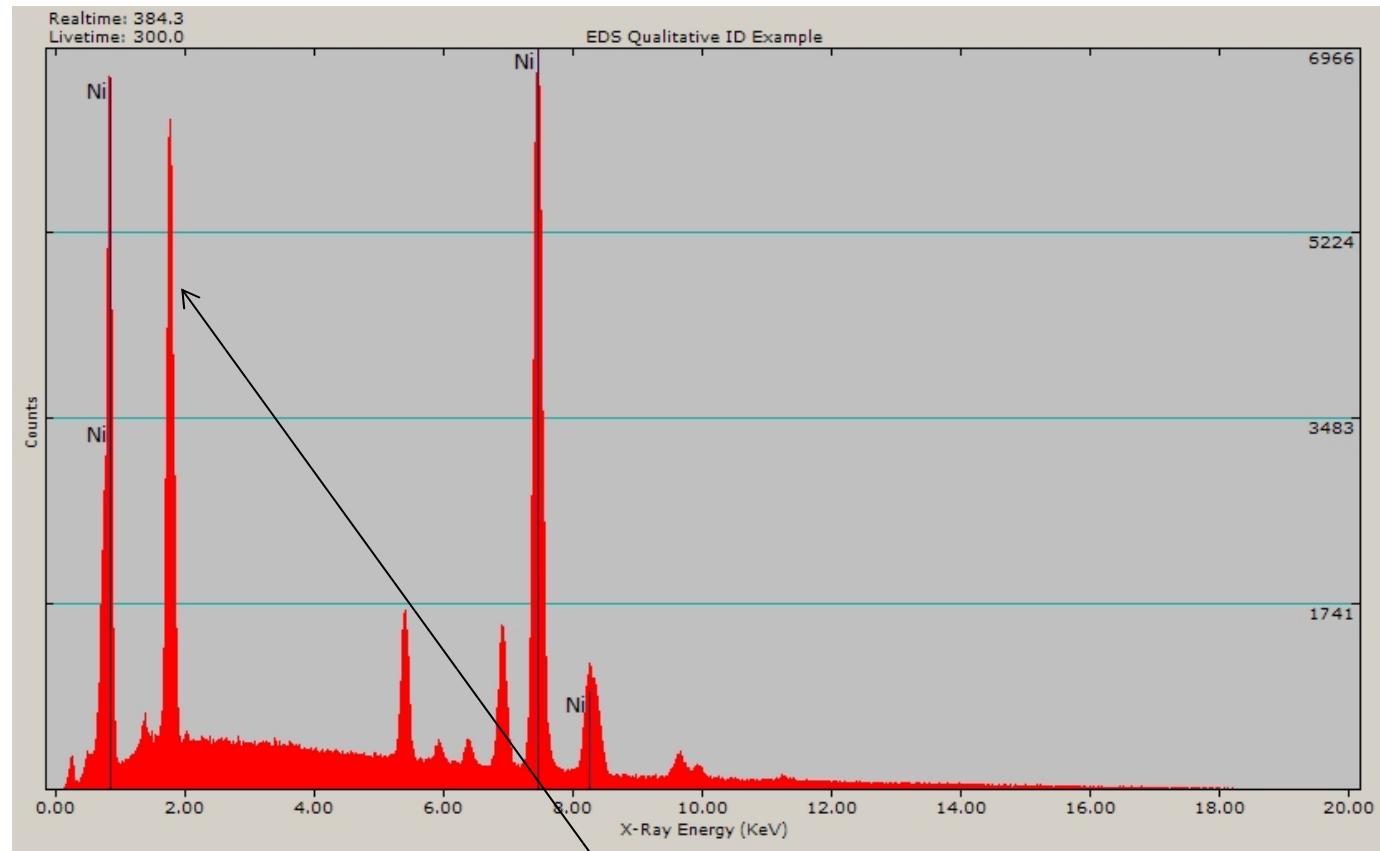
EDS Qualitative Analysis Example



- Step 1 continued: Mark ALL Ni peaks
 - K α , K β , and the L-series
 - Notice that the Ni K β peak looks a little wide and tall and offset

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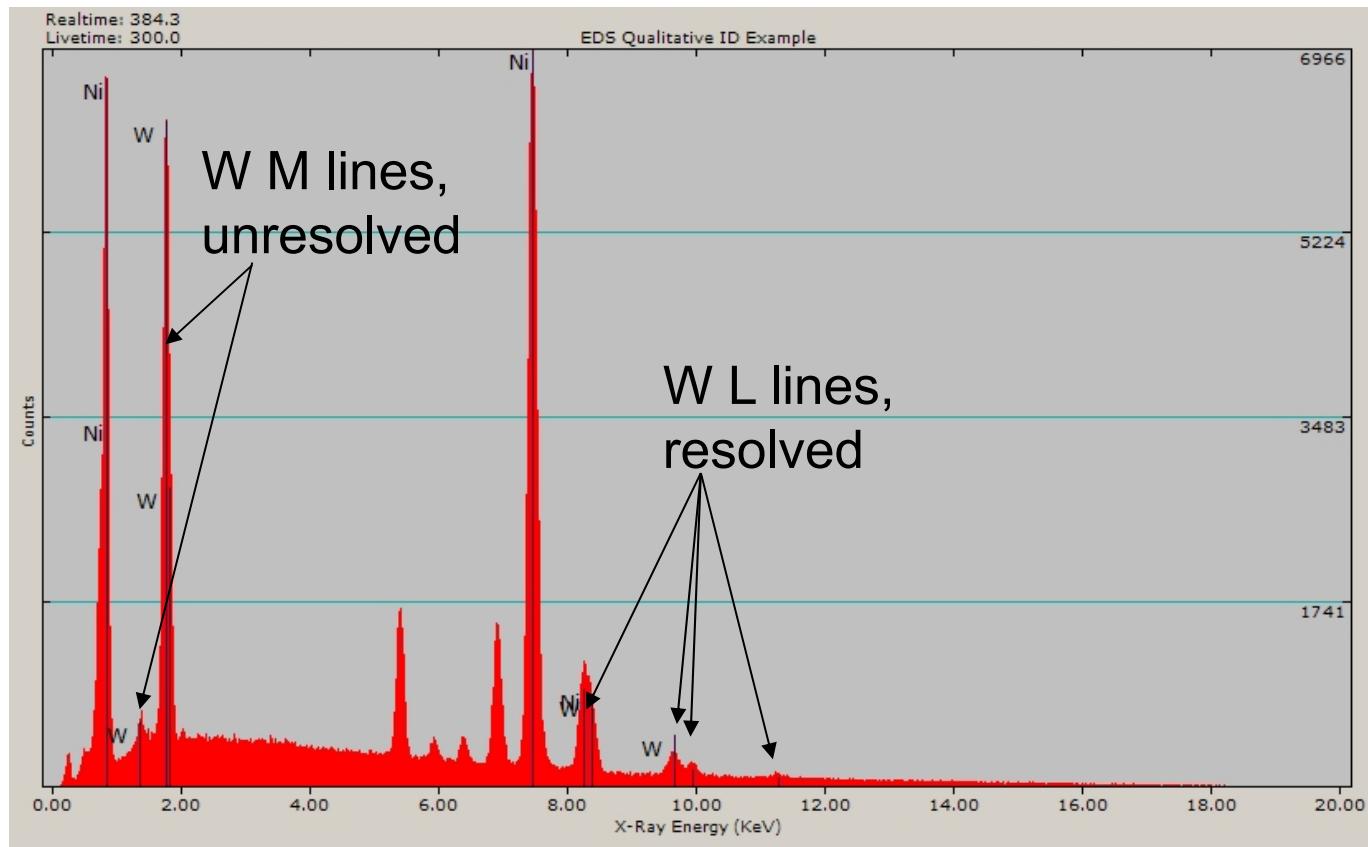
EDS Qualitative Analysis Example



- Step 2: Identify the next tallest, highest energy, unmarked peak
- Energy ~ 1.7 keV = W M α peak

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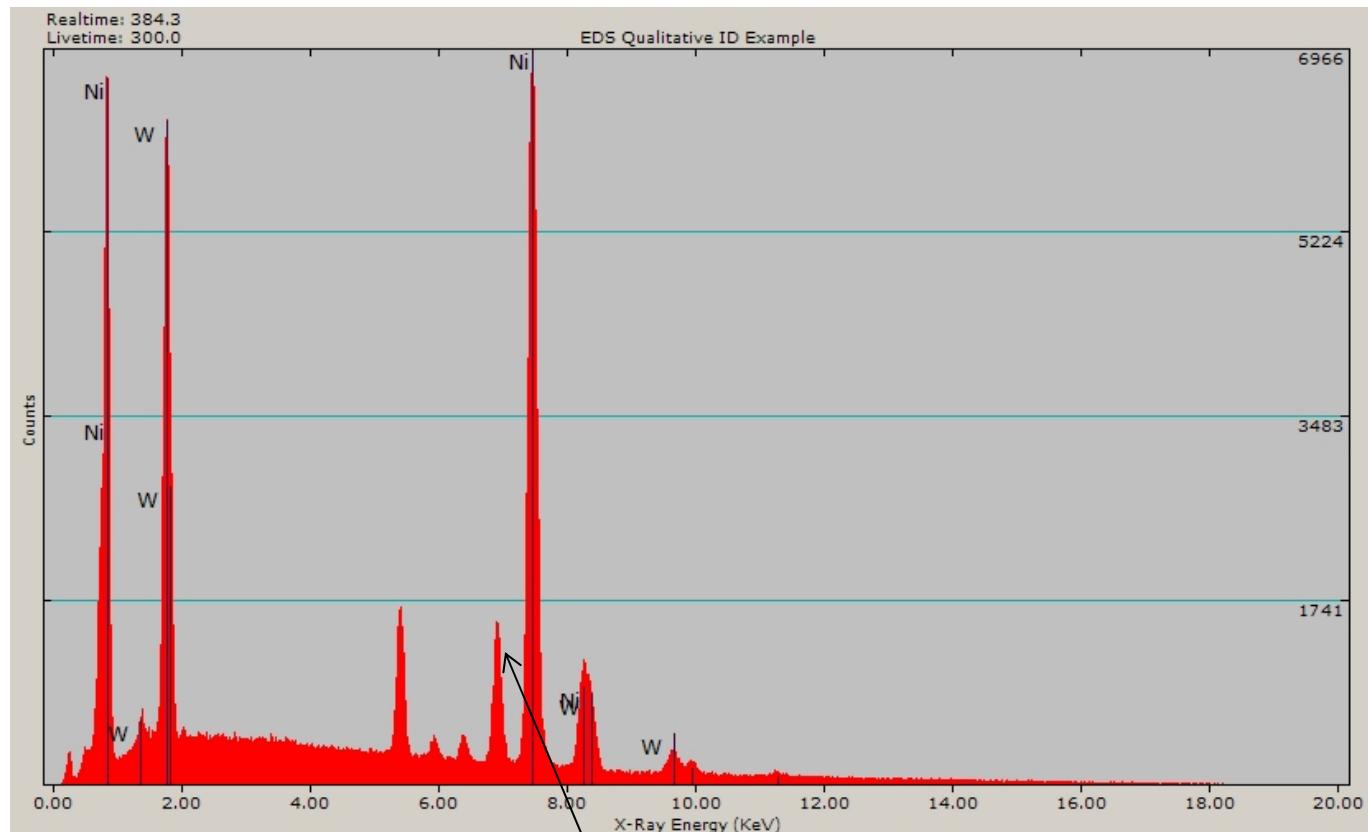
EDS Qualitative Analysis Example



- Step 2 continued: Mark ALL W peaks
- Note how the Ni K β peak has an overlap with the W L α peak
- Now multiple peaks have been identified – one peak id leads to others

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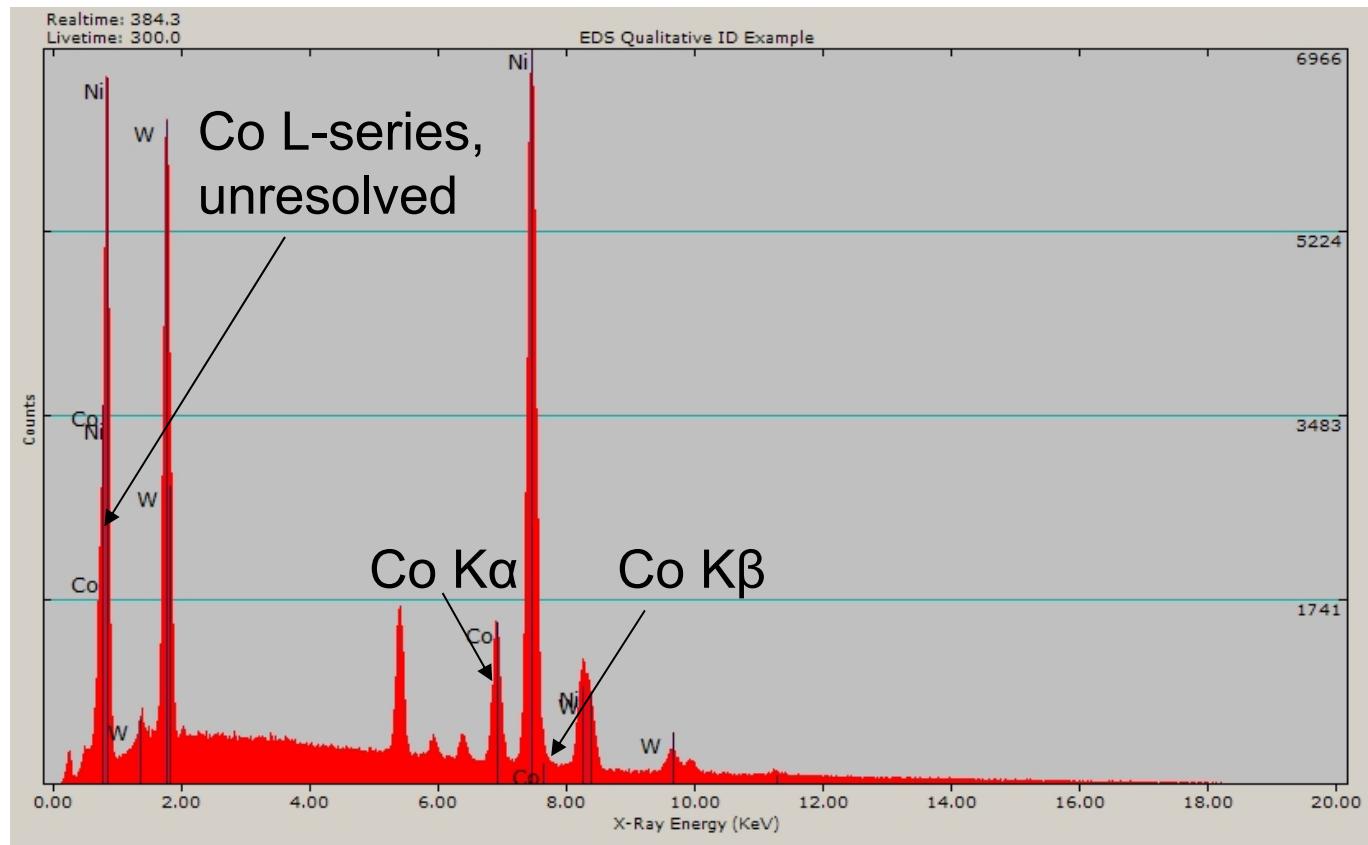
EDS Qualitative Analysis Example



- Step 3: Identify the next tallest, highest energy peak
- Energy ~ 6.9 keV = Co K α

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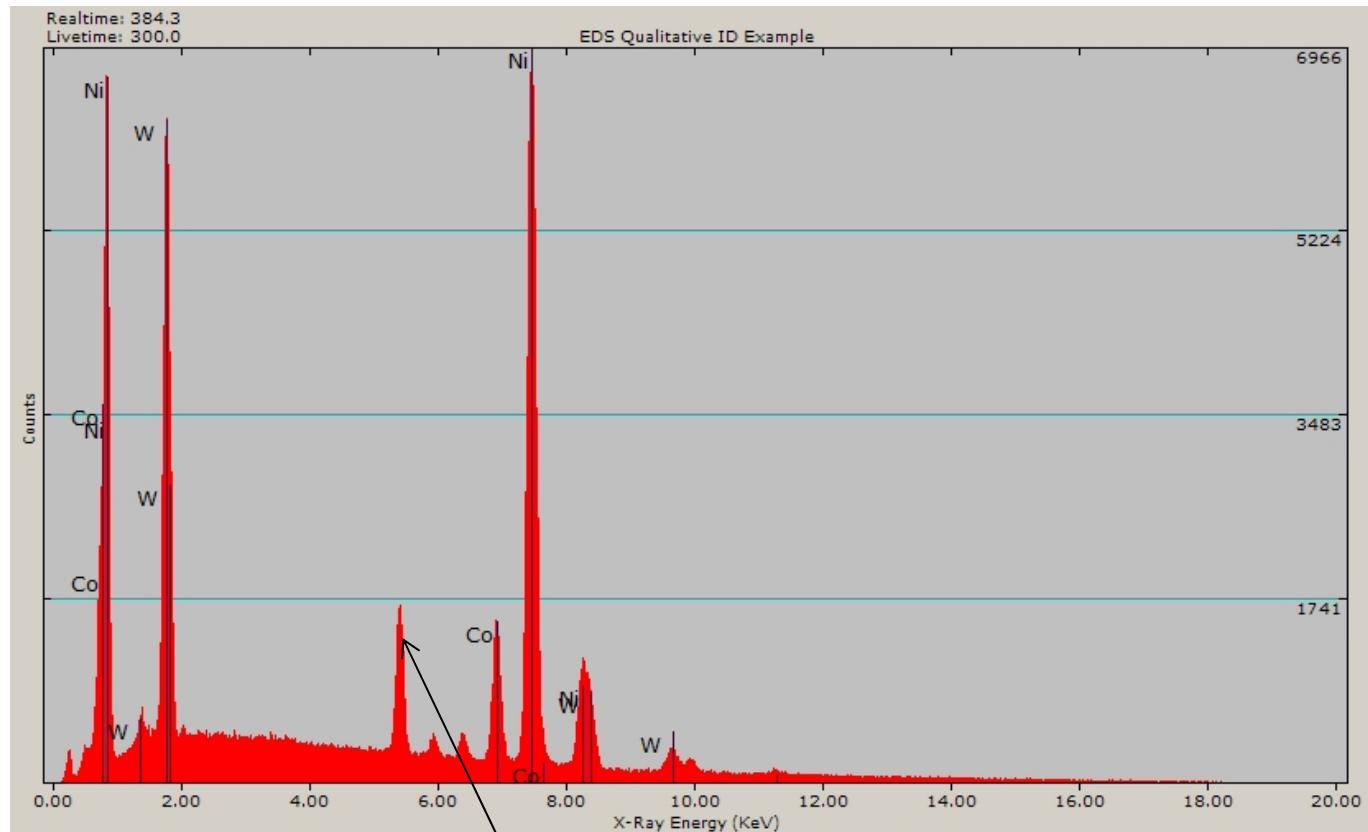
EDS Qualitative Analysis Example



- Step 3 continued: Mark ALL Co peaks
 - Note how many peaks are cleared with three elements
 - Notice the overlaps at low energy and Ni K α peak shape (asymmetric)

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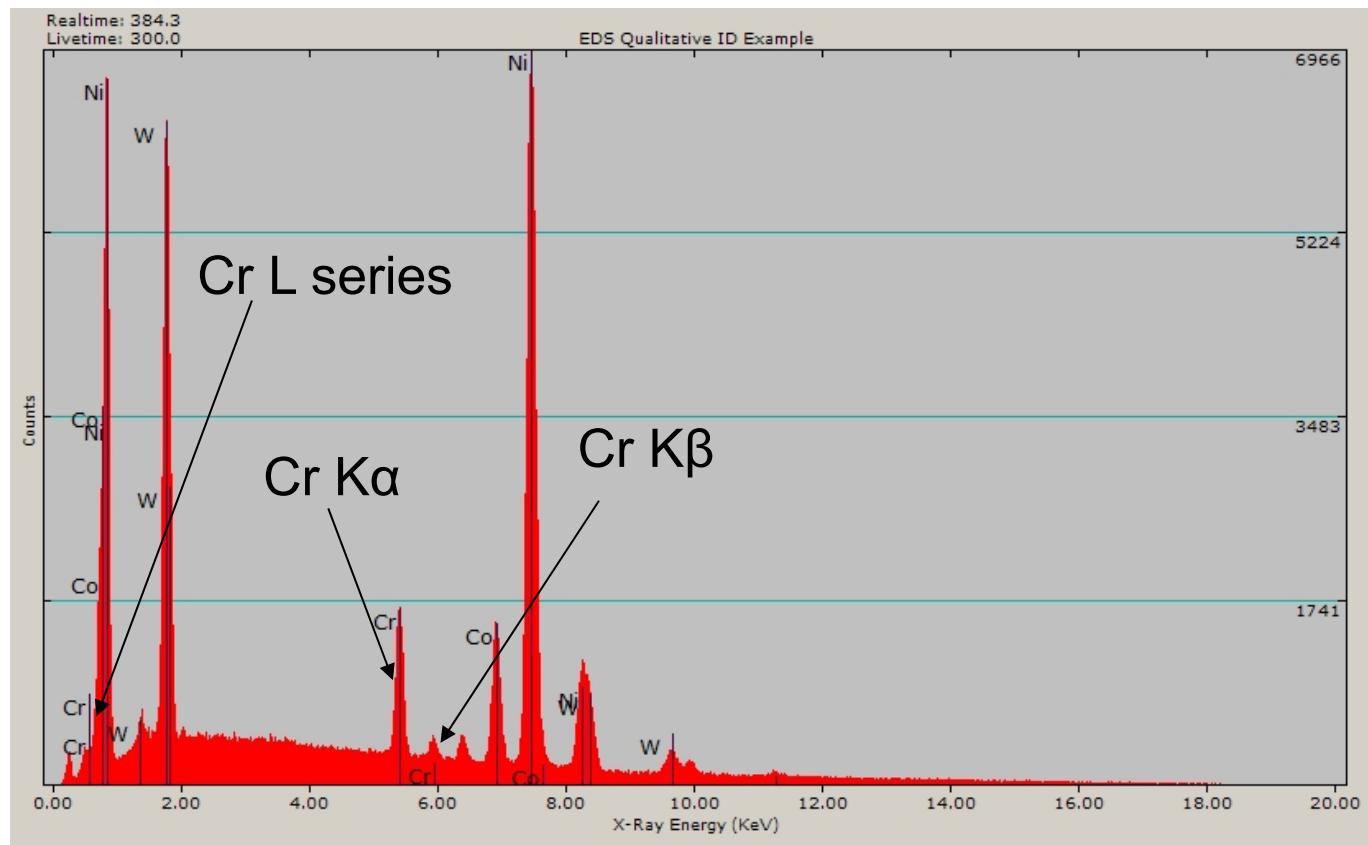
EDS Qualitative Analysis Example



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- Step 4: Identify tallest, highest energy, unmarked peak
- Energy \sim 5.4 keV = Cr K α

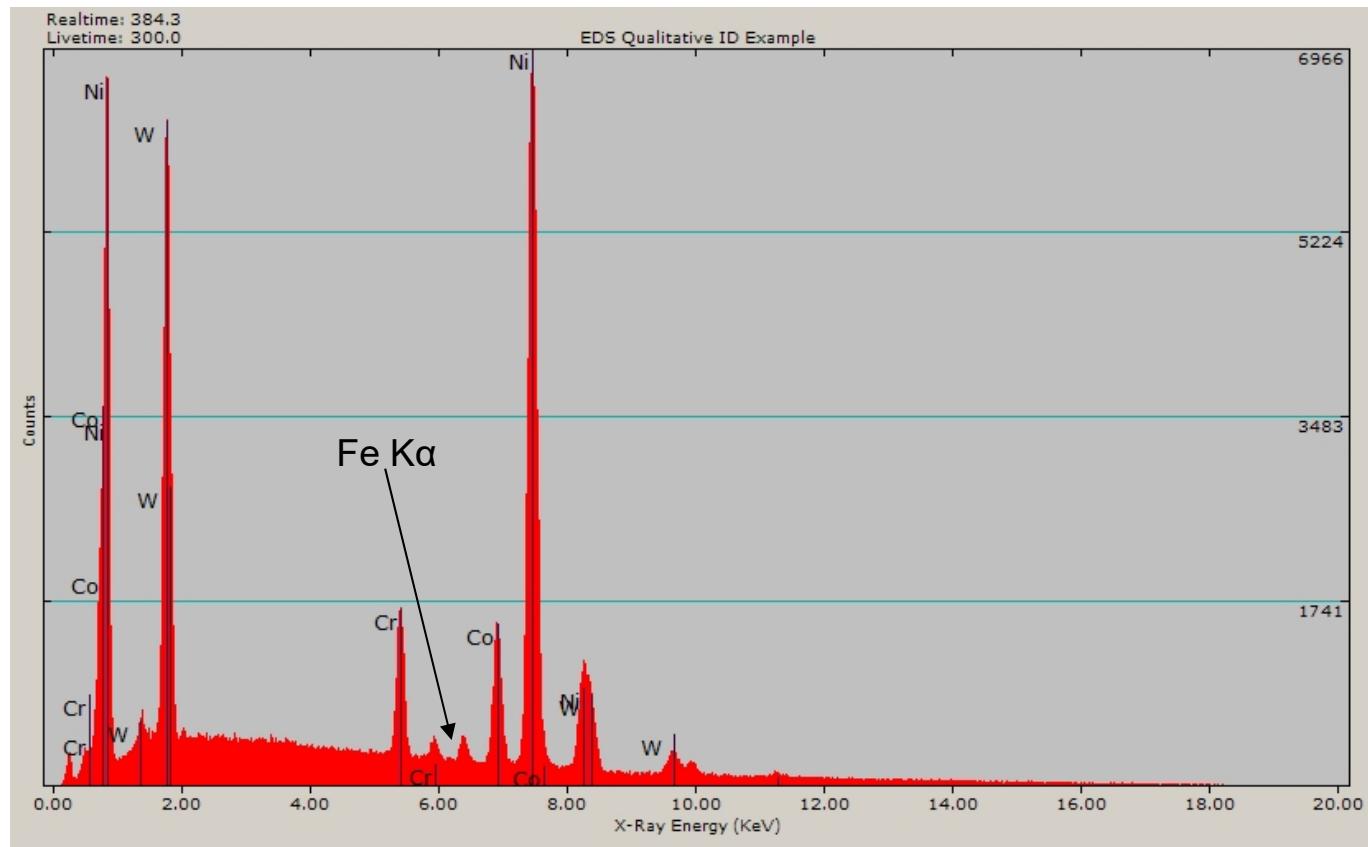
EDS Qualitative Analysis Example



- Step 4 continued: Mark ALL Cr peaks

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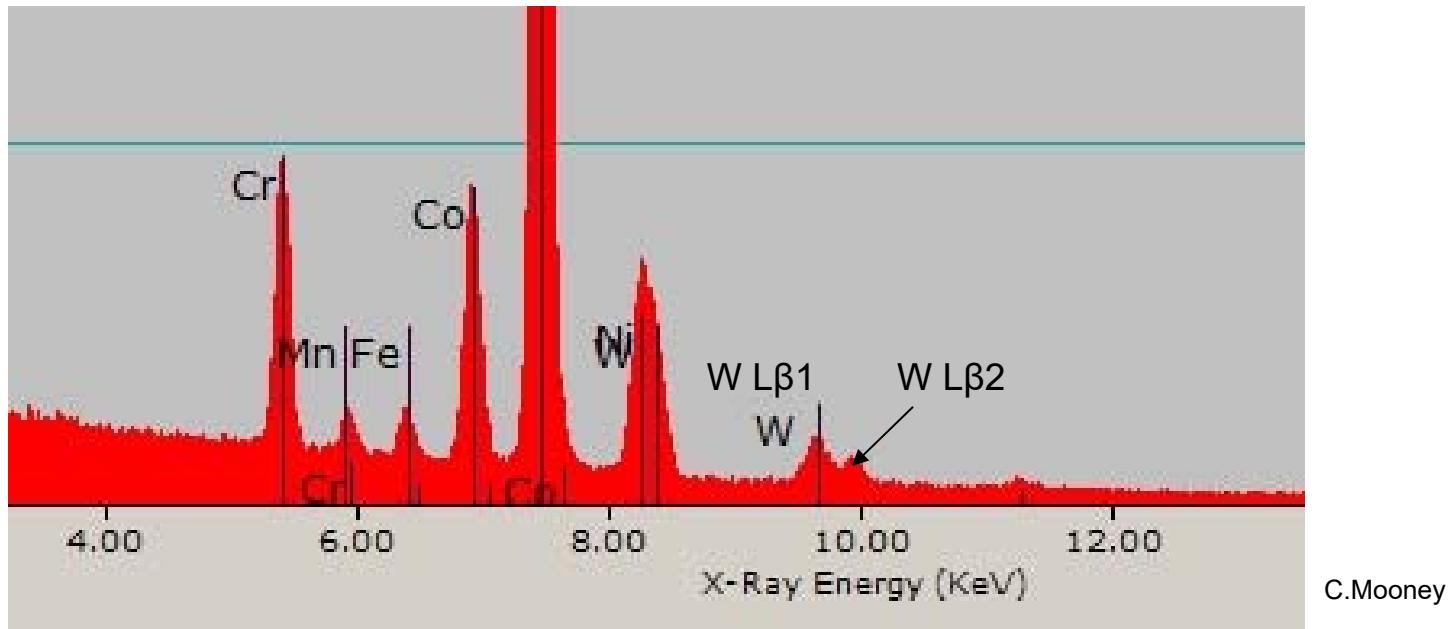
EDS Qualitative Analysis Example



- Step 5: Identify remaining peak
- Energy ~ 6.0 keV = Fe K α
- Mark all Fe peaks

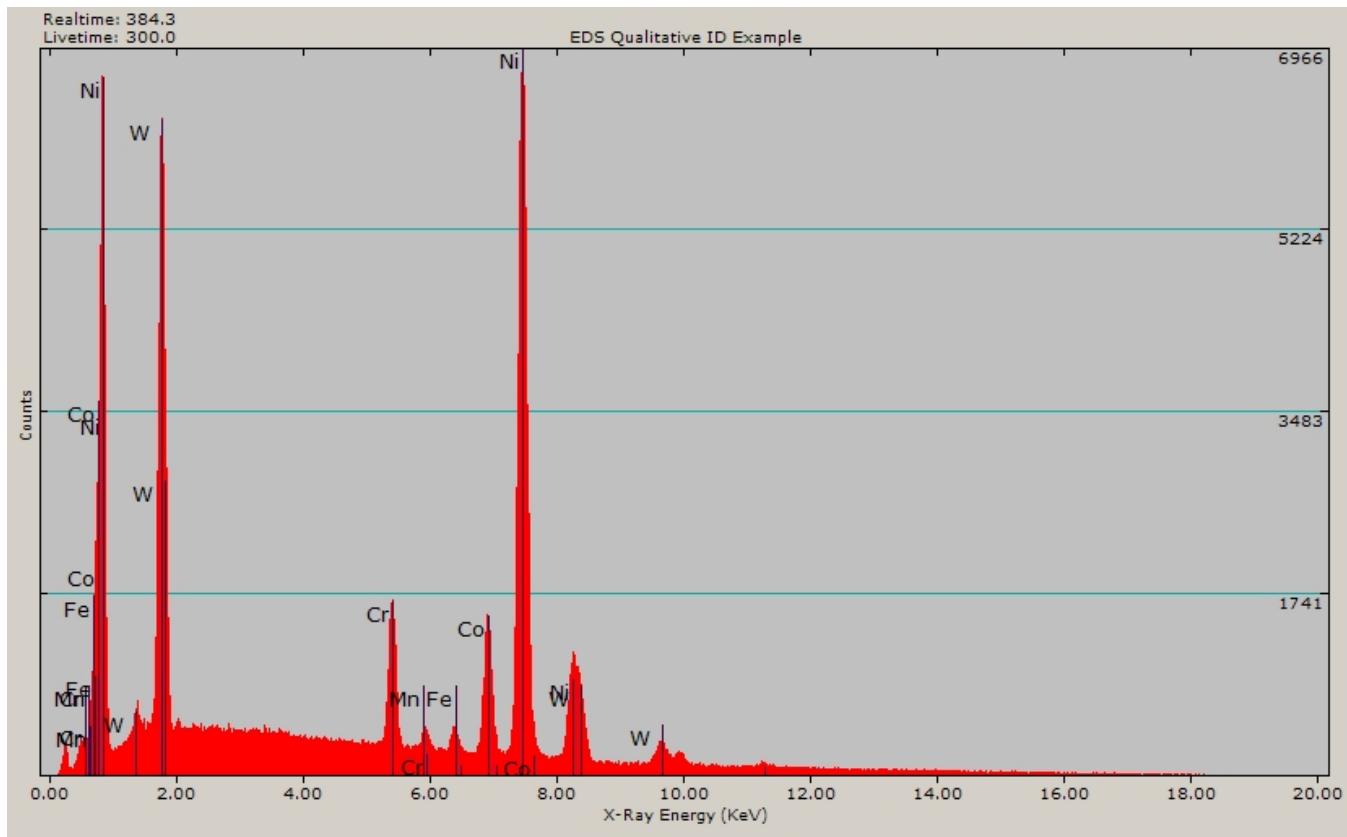
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EDS Qualitative Analysis Example



- Look for discrepancies
- Cr K β can't account for the peak height and position
- Mn K α has an overlap, so there is some Mn in the sample too!
- Notice that we can observe the noise in the spectrum when we expand the Y (count) axis

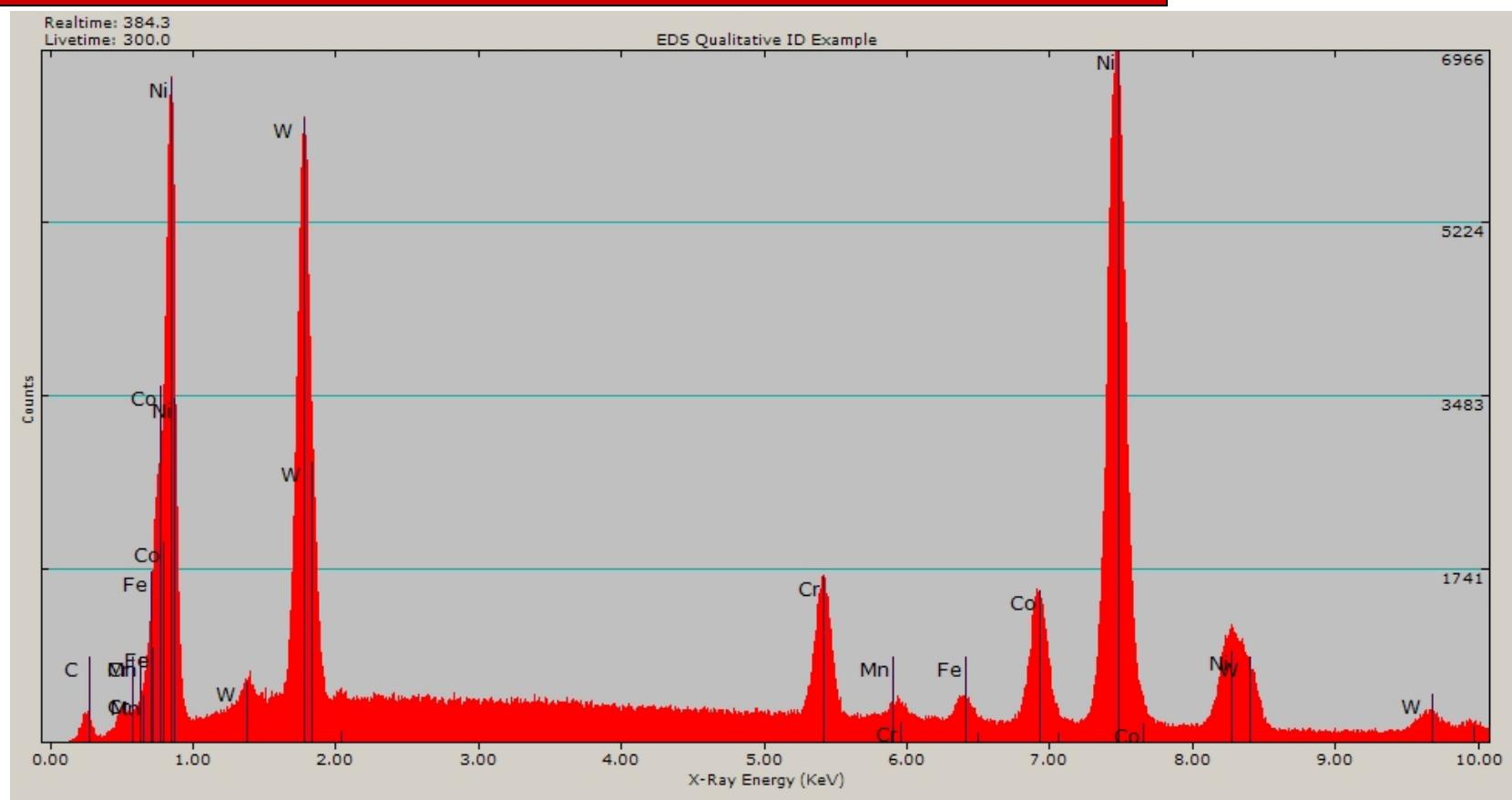
EDS Qualitative Analysis Example



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- Finished! All peaks are accounted for!
- The unknown alloy is composed of Ni, W, Co, Cr, Fe, and Mn

EDS Qualitative Analysis Example



- Detail of low energy portion of spectrum
- Note how Ni, Co, Cr, Mn, and Fe all have peaks in the 0.75 keV range
- Low energy EDS has many overlaps (C is from the mount!)

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EDS – Practical Qualitative Analysis

- The preceding discussion presumed unassisted analysis using a slide rule or X-ray energy table to determine the X-ray line positions
- In practice, when using EDS software, each manufacturer of EDS equipment will have their own method
- In most cases there are two methods: assisted and manual
 - Computer assisted mode is AKA automatic ID
 - AutoID can be turned off and the user can ID the peaks manually
 - This is recommended for new users
- Don't panic – a new user does not need a slide rule!
- Most software has a function that will allow the user to advance through the periodic table displaying the peaks for each element
 - Highly recommended: Figure it out for yourself when you first start!

EDS – Practical Qualitative Analysis

- Back in the day, computer assisted ID was not so good
 - The operator was encouraged to figure it out for themselves
- New systems are good enough that the auto ID function works pretty well
- There are some things to notice:
 - Overlaps confuse the computer more than they confuse you
 - The software generally won't ID an escape or sum peak correctly
 - Some software tends to make odd choices
 - Example: Current Oxford AZtec systems seem to want to auto ID small amounts of Al as Br
 - Al K α = 1.486 keV
 - Br L α 1 = 1.480 keV
 - Seems odd since Al is much more common than Br

EDS Artifacts

- Sum peaks
 - If more than one X-ray strikes the detector at the same time, we get a peak at the sum of the two X-rays
 - This can be avoided by not overdriving the detector and not generally a problem with modern detectors
- Escape peaks
 - If the incoming X-ray has enough energy to cause the Si detector to fluoresce ($> 1.9 \text{ keV}$), then Si X-rays will be formed inside the detector
 - Most of these Si X-rays will be absorbed and counted as the original X-ray
 - If some of these X-rays escape before being absorbed, then we lose the energy of a Si X-ray and an escape peak can appear in the spectrum

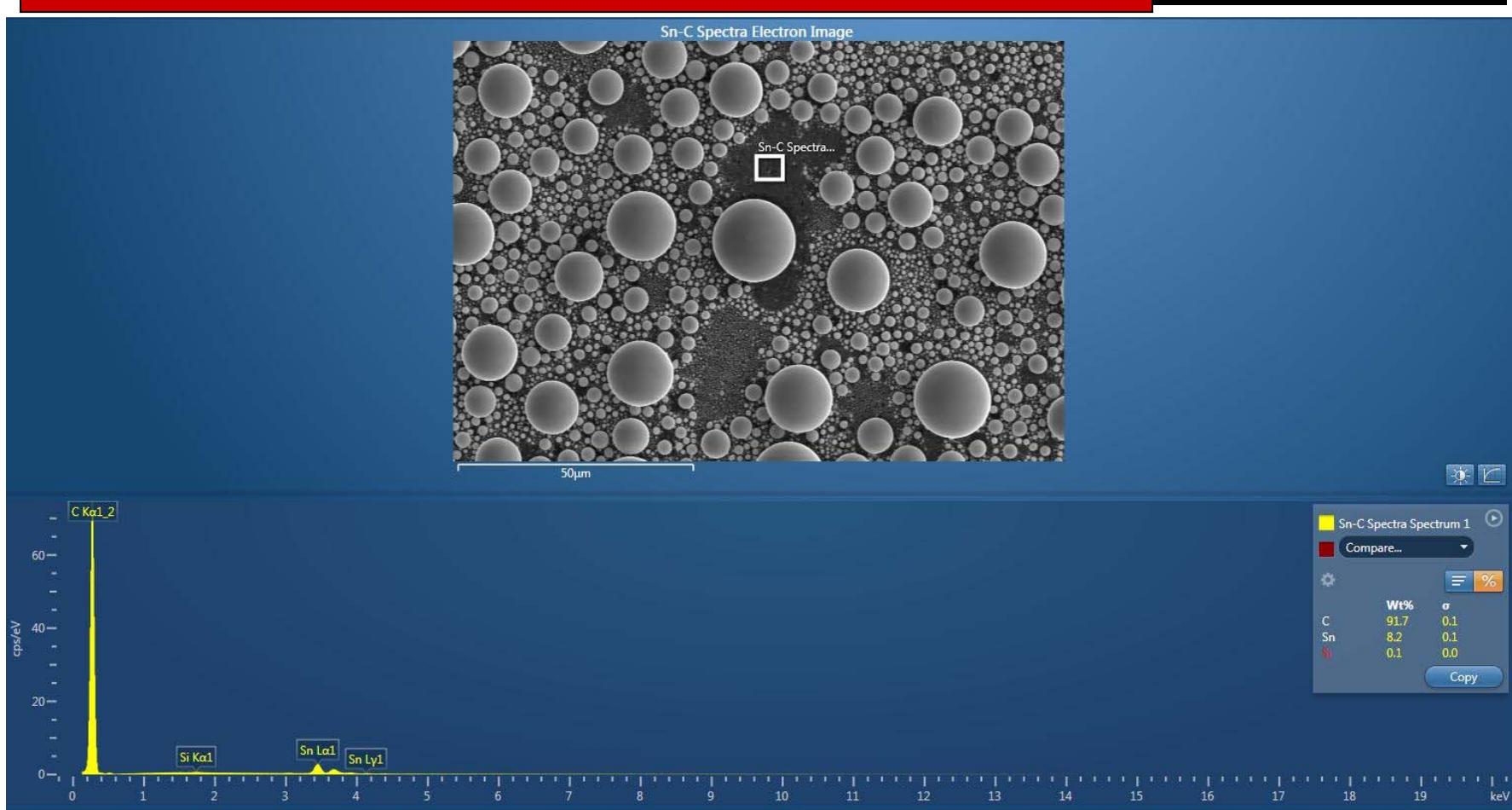
X-ray Mapping

- X-rays can be used to determine the distribution of elements near the sample surface in the X-ray generation volume
- This can be done in a variety of ways:
 - Spot probes of various locations on the sample
 - Area probes of various locations on the sample
 - X-ray mapping along a line, aka, a linescan
 - X-ray map of an area of the sample

X-ray Point Probes – Practical SEM

- Don't trust them to be where you think they are, especially if you are choosing the position based on a collected image
- Any charging of the sample will cause the charge state of the sample to change, which will result in a shift in the point that the next electron strikes
 - This will appear as drift in an image
 - This will cause the X-rays that are generated to come from a different point than was chosen by the operator
- Much smarter to choose an area that appears representative
 - Any drift effects will be minimized
- To illuminate very small features with the beam, zoom to very high magnification (often with a reduced area scan box) such that only the feature of interest (or a small part of the feature) is observed on the SEM display – appearance will be solid contrast
- If the contrast changes, adjust the image shift to keep the feature in the field of view

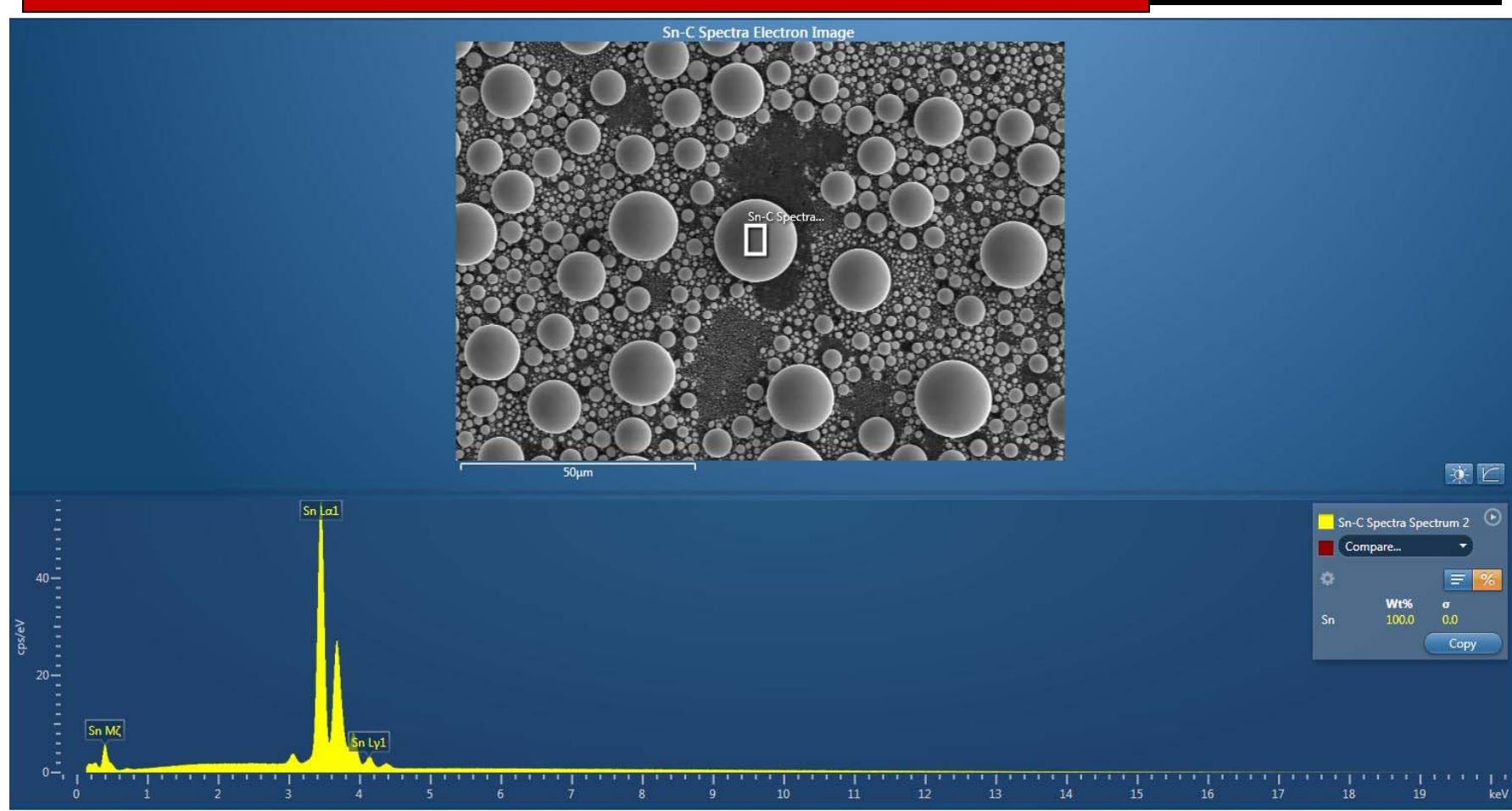
X-ray Area Scans



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- X-ray area scan of a Sn-C standard showing a C rich region
- Notice that the system is picking up 0.1% Si (don't believe it!)

X-ray Area Scans

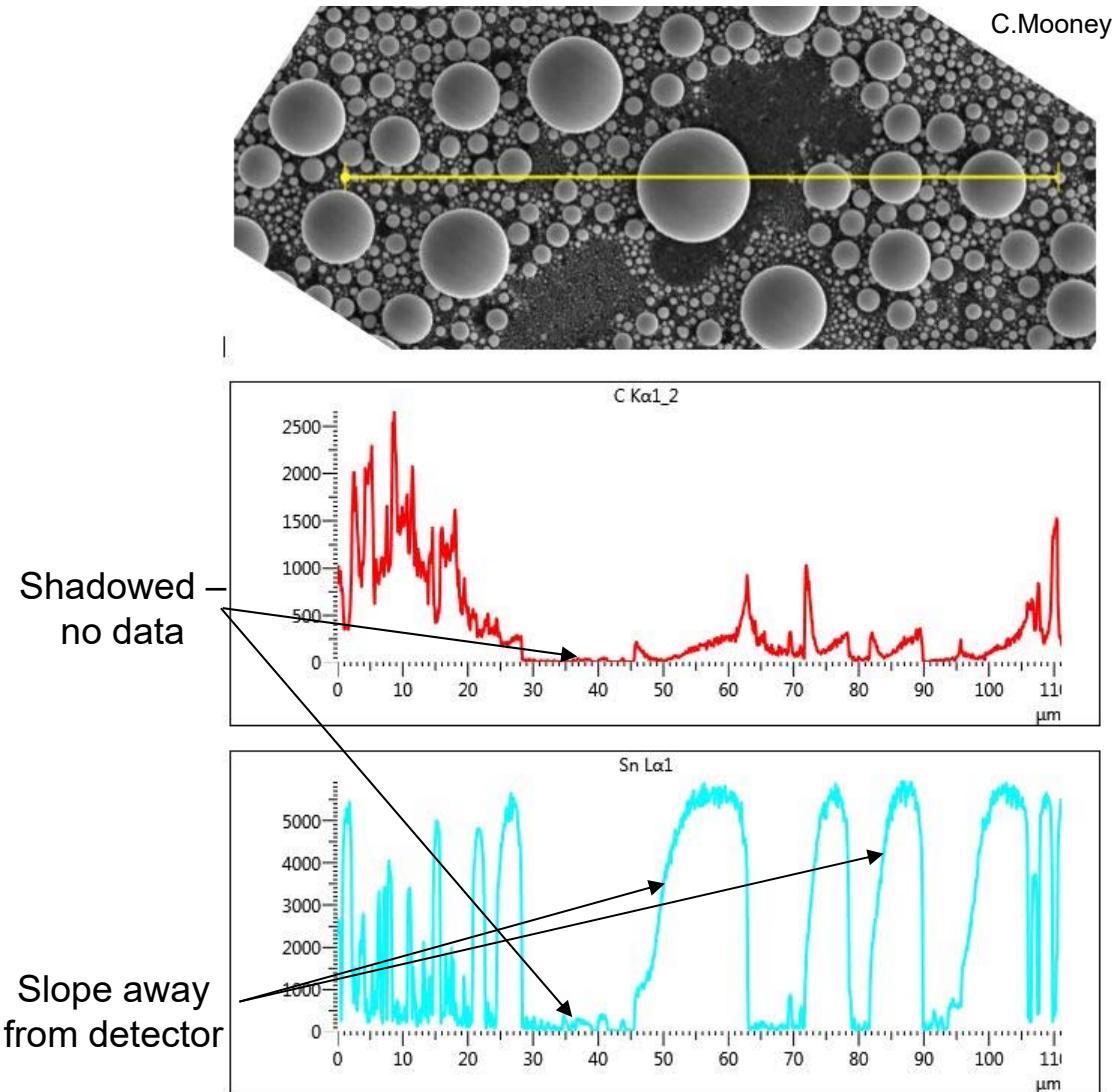


- X-ray area scan of a Sn-C standard showing a Sn rich region
- This scan convinces that there is no Sb in the Sn

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X-ray Line Scan

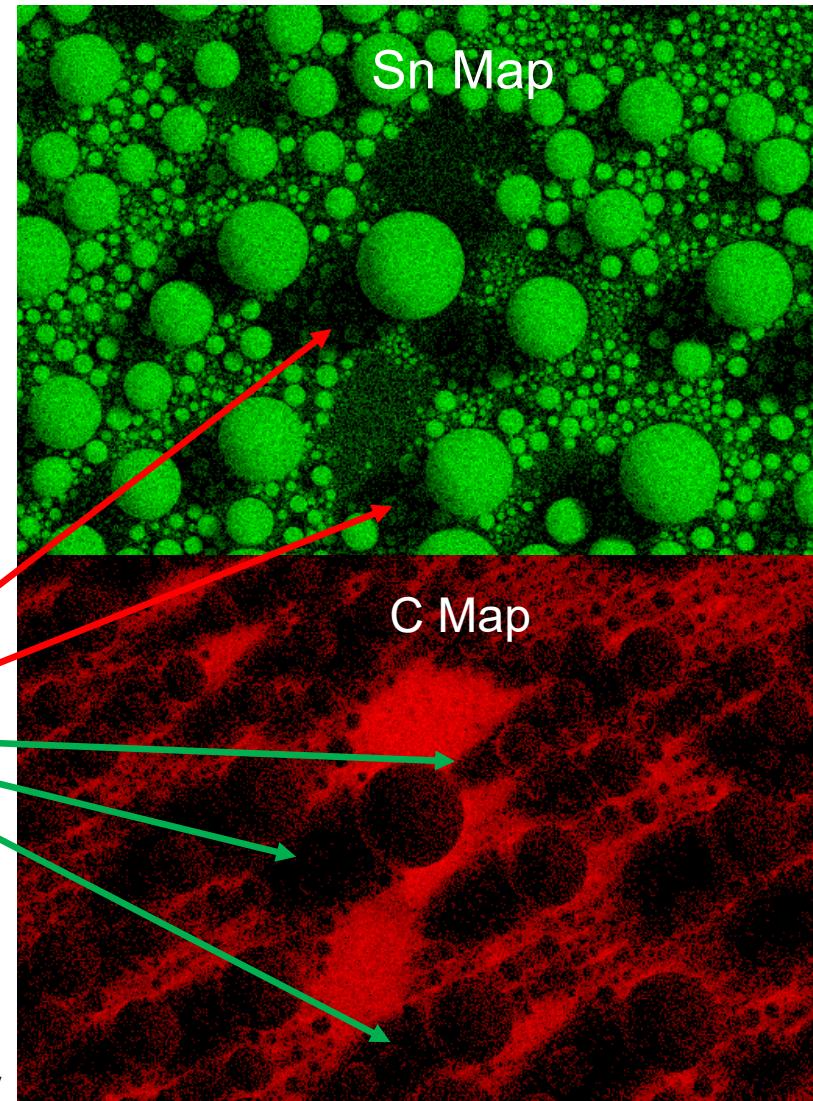
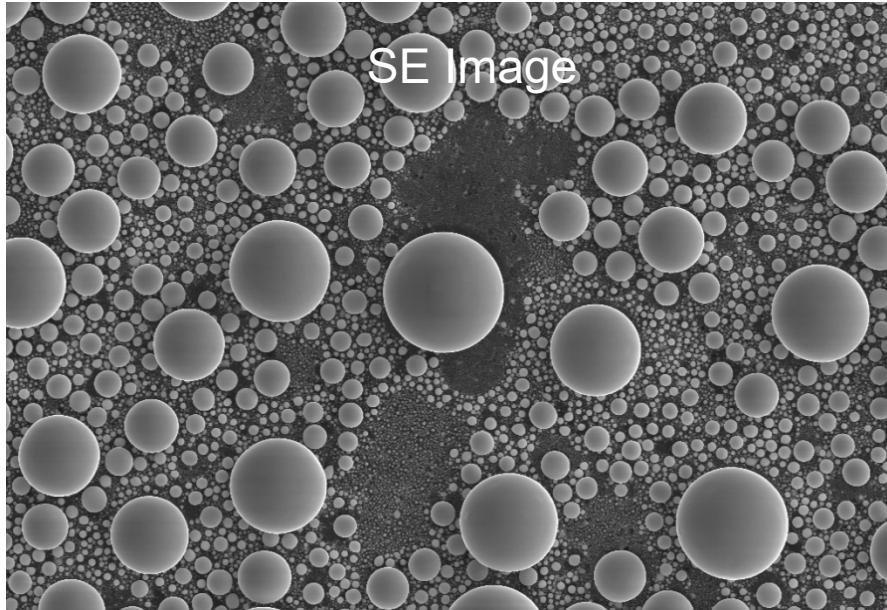
- Instead of an area scan, a line scan can be generated
- This was smart when detectors were slow and mapping took a long time, but mapping with an SDD is faster than line scans used to be with a SiLi detector
- Note that shadowing effects give the X-ray data a slope away from the detector



X-ray Mapping

- X-rays can be used for mapping
- Scan the beam as in normal imaging and use X-rays as the signal
- Most systems collect a spectrum at each point in the scan
 - Typically the system will integrate multiple fast scans together
 - Integrates the spectrum at each point in the map
- Don't generally display each spectrum
- Choose the element one wants to map and pull that elemental data from the spectral data to generate a map
- Possible to generate a map for each elemental species
- Map data takes time to collect
 - SDD equipped SEMs should be able to generate a reasonable map in less than five minutes
 - Increasing current will decrease the map collection time
 - Increasing current will not degrade X-ray spatial resolution
 - EDS spatial resolution is a function of beam energy (X-ray generation volume)
 - EDS map spatial resolution

X-ray Map



Notice incomplete X-ray data

Why? Shadowing from a shallow angle X-ray detector in a UHR SEM with a short working distance. Shadows point away from the detector.

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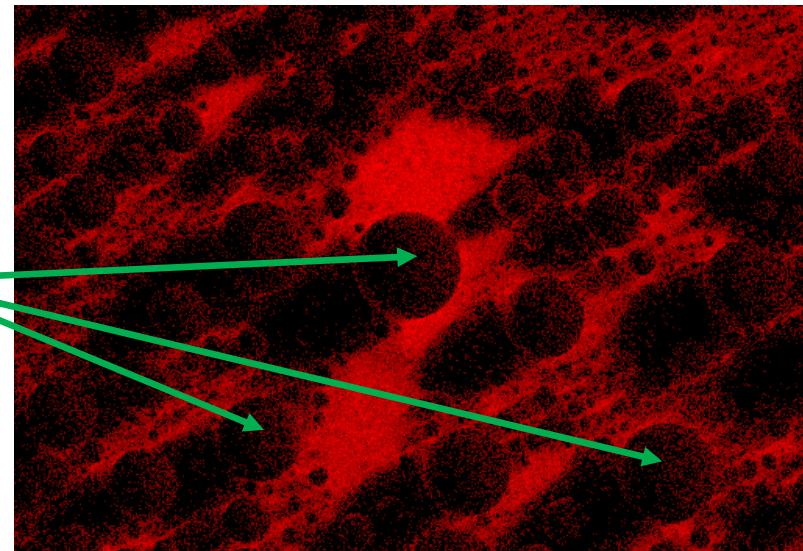
Dangers of X-ray Mapping

- Background!
 - If the software is not effective at removing background and the P/B ratio is not very high, then the background may appear everywhere

No C here!

Only background!

Background can be from
bremsstrahlung or from forward
scattered electrons from the Sn
sphere striking the C



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- Overlaps!
 - If there is an energy overlap, the map will likely be wrong unless the two elements with an X-ray overlap actually have a physical overlap

Quantitative EDS

- Quantitative EDS is very complicated
 - More than integrating the area under the peak
- Modern standardless quantitative analysis is pretty good
 - To claim an absolute, one should use standards
 - For comparisons between samples collected under identical conditions, standardless quant is fine
- If you want to get high quality quantitative results, make sure you do further reading and really understand the issues and how quant analysis works
- Beware those who simply press the quantify button and hope for the best...

Supplemental Information

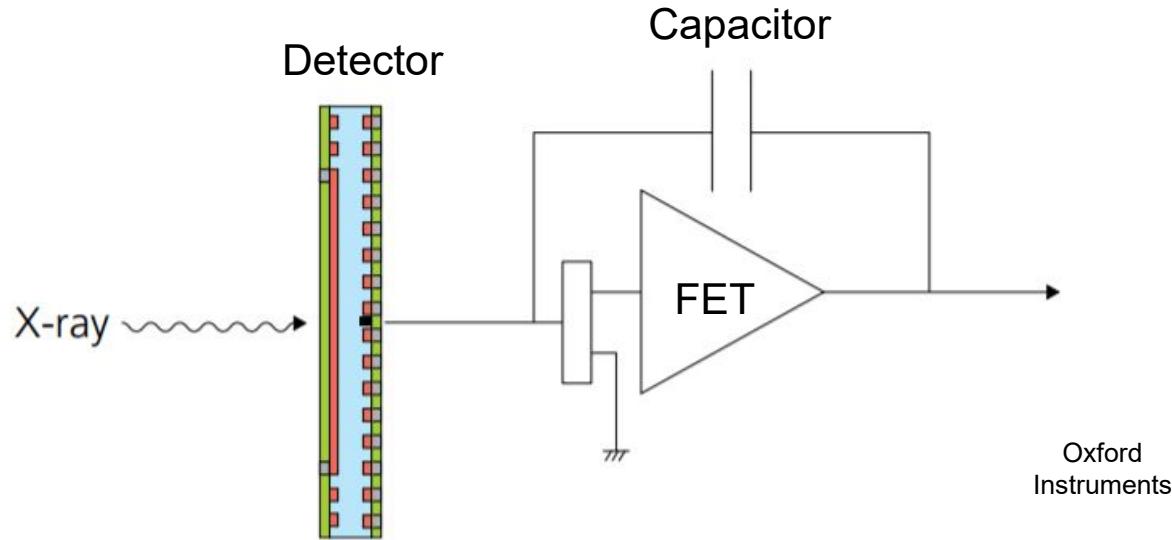
EDS – Electronics

- After the X-ray is converted to electron-hole pairs, the free charge carriers are then swept out of the detector by the applied bias
 - SDDs do this very quickly

Now that we have swept apart the electron-hole pairs, what do we do with them?

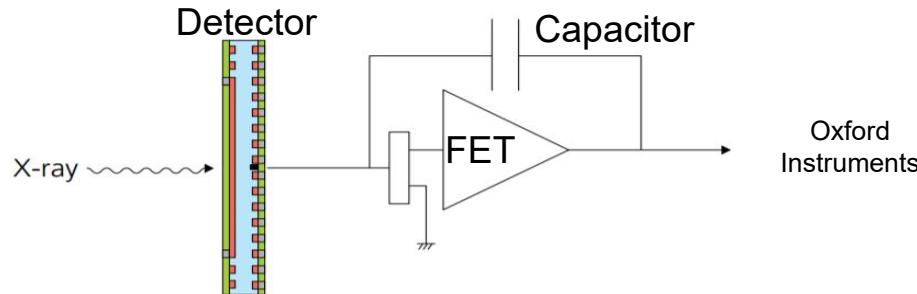
- First, we convert the charge to a bias (voltage)
- Then the voltage pulse is then sent to a pulse processor to measure the X-ray energy based on the original collected charge
 - All of the spectral dispersion is done electronically with EDS
 - WDS uses a crystal to separate the peaks
- The output of the pulse processor is an X-ray count of a particular energy that is filed into a spectrum
 - Recall the spectrum is counts vs. energy
- Lastly, the spectrum is displayed on a computer

EDS Pre-amplifier



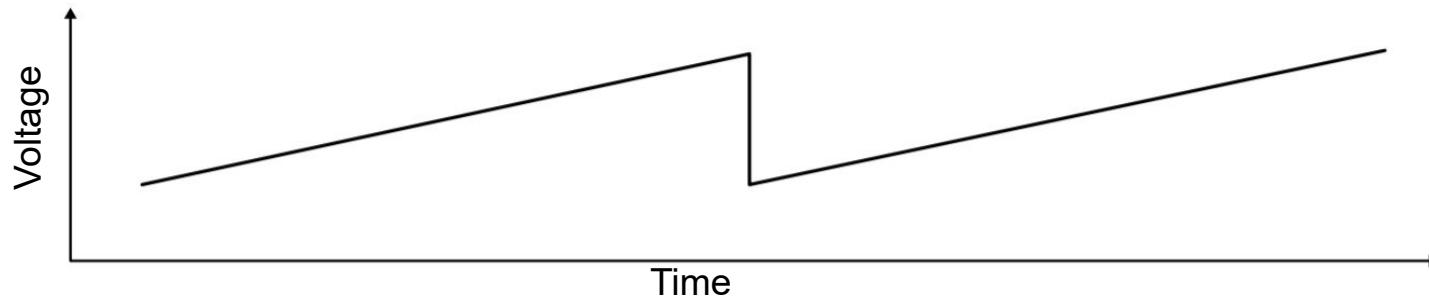
- The first stage of the pre-amp is a low noise field effect transistor, or FET, which is cooled and placed immediately behind the detector
 - The FET is often in contact with the detector or integrated with the detector
- Charge is stored in the capacitor and converted to a voltage by the FET, so the output voltage increases by steps equal the amount of charge collected

EDS Pre-amplifier



- There are two sources of charge:
 1. The X-ray induced charge we want to measure
 2. Leakage current from the sensor material
- The leakage current will appear as an increase in voltage over time with a constant slope
- The X-ray induced charge will appear as sharp, discrete steps superimposed on the slope of the leakage current
 - The height of the step will correspond to an X-ray energy
- At some point, the capacitor becomes saturated with charge and has to be discharged, resetting the FET
 - This can be hundreds of X-ray detection events

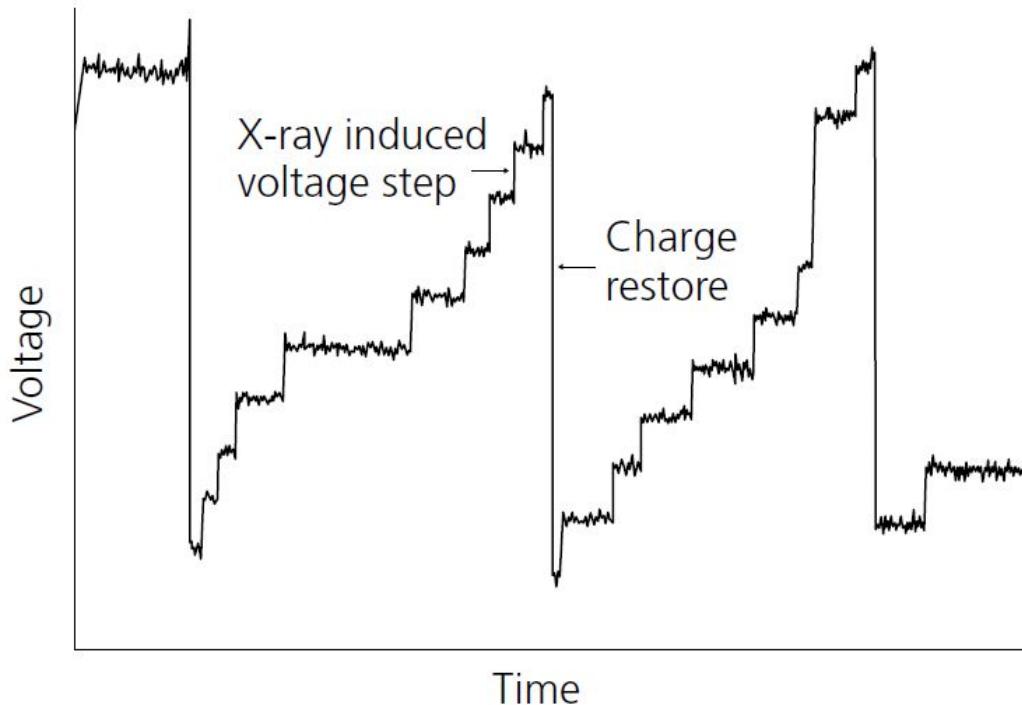
Leakage Current



Schematic of leakage current response with no incident X-rays. The charge from an X-ray would appear as a step on the voltage ramp.

- Leakage current results from the bias applied to the detector
- This gives rise to a slope on the voltage coming out of the detector upon which lie the discrete steps defining individual X-rays
- Fortunately, the leakage current slope is constant
- SiLi detectors require a long process time and cooling with LN₂ reduces leakage current noise
- SDDs do not require a long process time
 - The leakage current slope can be steeper while maintaining the same energy resolution

Charge to Voltage



- Output of the pre-amplifier (voltage) vs. time showing X-ray induced charge steps that are converted to voltage by the FET
 - The leakage current slope is not shown
- When the capacitor is saturated, it is discharged to reset
- The discharge process is part of dead time

Pulse Processor

- The primary job of the pulse processor is to accurately measure the energy of the incoming X-ray photon and then give it a digital count in the spectrum
 - Energy is proportional to charge
 - Charge is proportional to voltage
 - Measure the height of the voltage pulse with the pulse processor

The pulse processor has to be able to do the following:

- Accurately sort a wide range of energies from ~ 100 eV – 30 keV
 - Place a count of energy in the right place on the spectrum
- Remove noise in the voltage ramp of the pre-amp
 - Average the noise signal over time
- Remove the leakage current slope
- Differentiate between events that happen very close in time
 - Avoid pulse pile up

Pulse Shaping

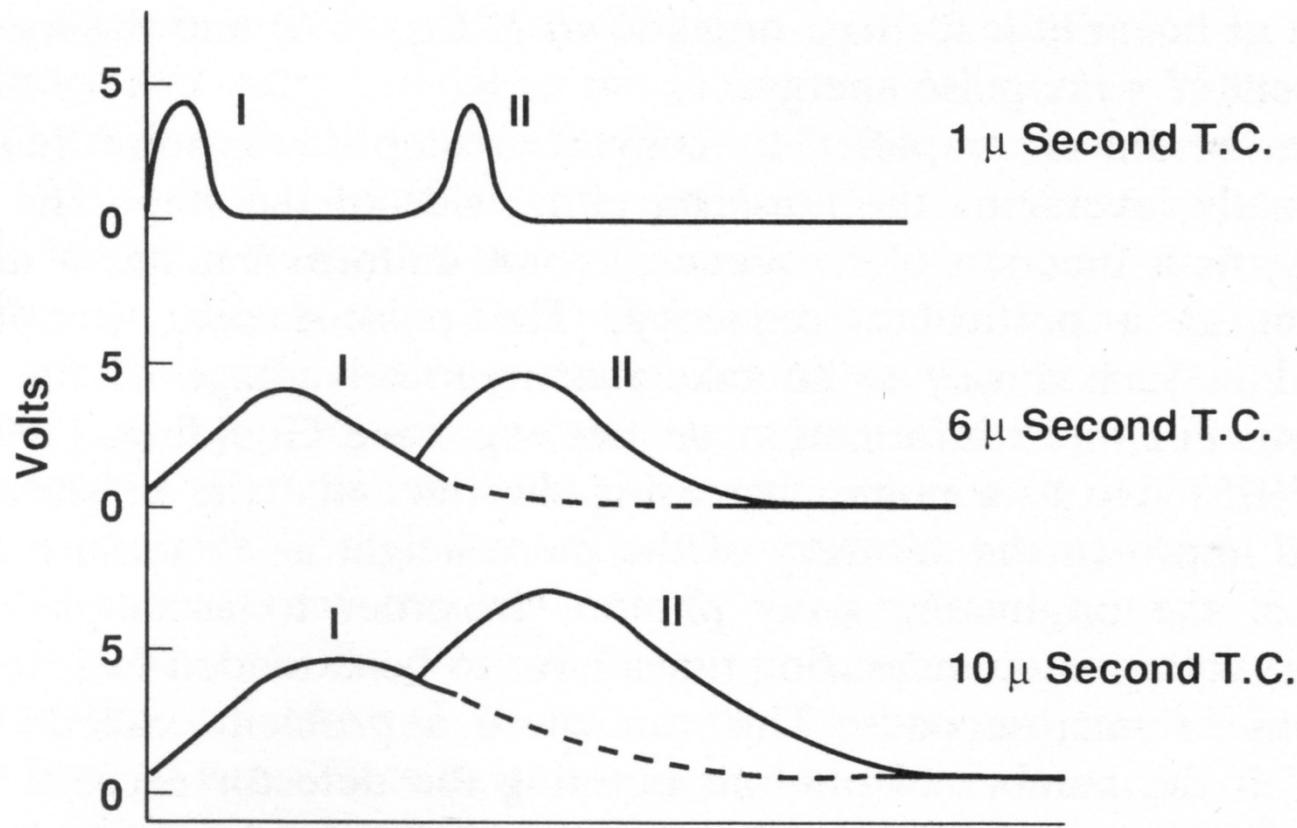
From the charge to voltage converter we go to pulse shaping.

- If we want to average noise we would like to have a long time to process the voltage pulse
 - To get a time average of the noise baseline
- But doing this requires that we do not allow another X-ray produced pulse to enter the amplifier
 - That is, we must shut down for some period of time
- If we do not shut down we risk pulse pile-up
- The shut down time is generally referred to as the Dead Time
 - Dead time is *all* shut down time
 - Live time is data collection time
 - Clock time – Dead time = Live time

Pulse Pile-up

- Pulse pile-up happens when two voltage pulses happen close together
 - This can be the result of two X-rays striking the detector very close in time and creating two charge pulses
 - The charge pulses are converted to voltage pulses
- If the voltage has not returned to baseline before the second voltage pulse arrives, the pulses start to pile-up on top of one another
- That is, the second pulse starts at a higher voltage than baseline and the output of the circuit will indicate a higher energy X-ray than was collected!

Pulse Pile-up



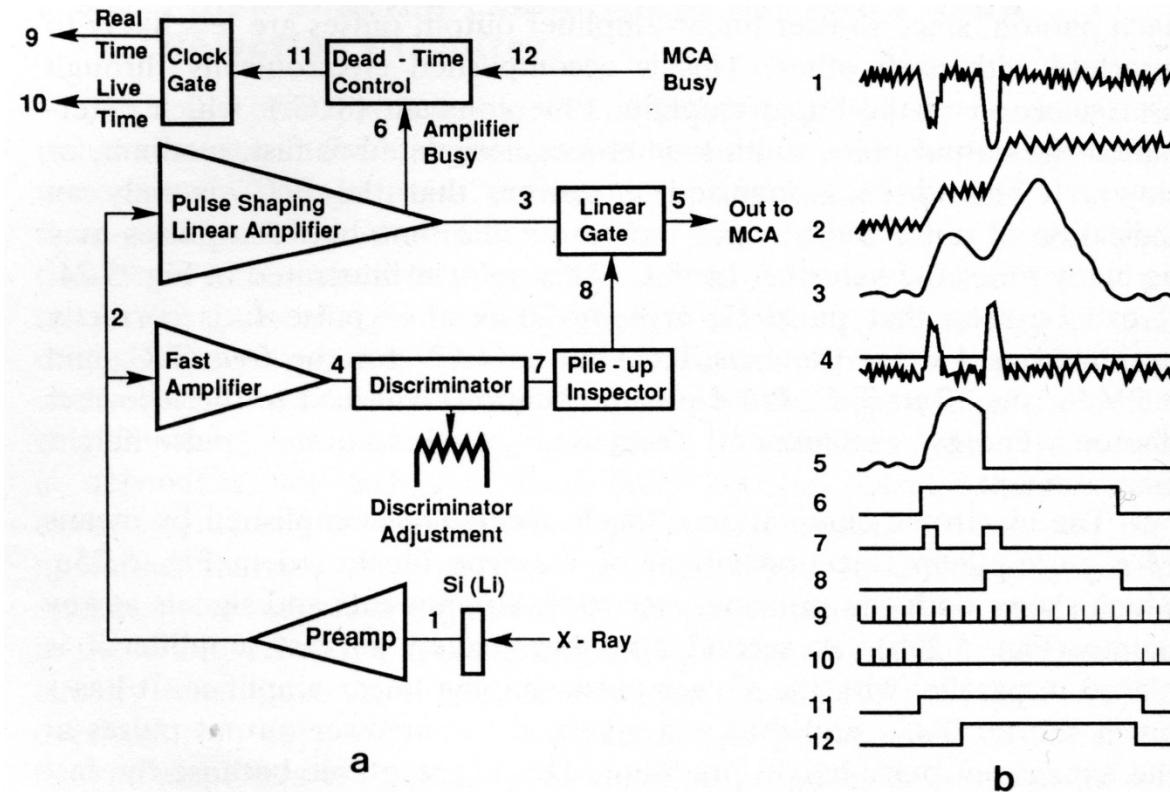
Pulse shapes for different main amplifier time constants

Amplifiers

How do we achieve both long averaging and prevent pulse pileup?
We use two amplifiers!

- Fast Amplifier
 - Detects when the next charge pulse (X-ray) is detected
 - If it is too soon then reject the new charge pulse
- Slow Amplifier
 - User chooses the time constant, 1 to 100 μ sec typical
 - Very linear, pulse shaping amplifier
- We must then keep up with amount of dead time (as a % of clock time)
 - Too much dead time is inefficient
 - Too little dead time is inefficient
 - ~ 20 % dead time is usually a good tradeoff

Amplifier



Goldstein, et. al.

Pulse Processor Amplifier Schematic

- a. Schematic, b. Waveforms at key points in the circuit
- Point 3 shows pulse pile up in the slow amplifier
 - Point 4 shows noise in the fast amplifier
 - 5 is the output to the DAC in the computer (max height = X-ray energy)

Edison Effect

- In the 1880s, Thomas Edison was trying to perfect the lightbulb
- He was trying to figure out what sort of filament to use
 - Carbonized bamboo was a candidate
 - The problem was that after a few hours, the bulb turned black
- An assistant noticed that C seemed to be coming from the end of the filament attached to the power supply, flying through the vacuum to settle on the inside surface of the bulb
 - Further determined that the C carried a charge!
- Made some special bulbs with a third electrode that could be connected to a current meter
- If the third electrode was biased negative relative to the filament, no current was measured
- If the third electrode was biased positive relative to the filament, current would flow
- Basis for modern electronics
 - Vacuum tubes!

Rutherford Backscattering

- In 1909 Ernest Rutherford was trying to understand how atoms work
- One of the atomic models of the day, the plum pudding model, was proposed by Lord Kelvin and developed by JJ Thompson
 - Thompson discovered the electron and that electrons were part of all atoms
 - Thompson proposed that an atom was a sphere of positive charge throughout which the electrons were distributed, like plums in a pudding
- Rutherford had Hans Gieger and Ernest Marsden point a beam of alpha particles (He^{2+}) at a thin gold foil
- If the plum pudding model is correct, the beam should pass directly through the foil
- A measurable fraction of the particles were deflected through $>90^\circ$, some going back in the direction from whence they came!
 - Rutherford: "It was as if one fired a 15 inch shell at a piece of tissue and it came back and hit you."
- This implied that atoms were composed of tiny spheres of positive charge separated by mostly empty space!
- Thus Rutherford rejected Thompson's model of the atom!