Scanning Electron Microscopy and Energy Dispersive X-ray Analysis

Basic Short Course

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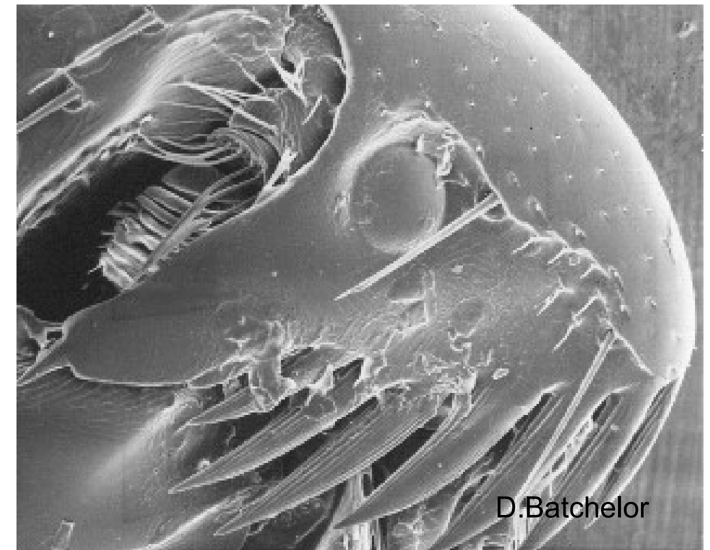
Outline

- 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
- Electron beam-sample interactions
- Signals and detectors
- Beam parameters and how to choose them
  - Practical SEM rules
- Practical SEM issues
  - Magnification and Resolution
  - Dwell time
  - Charging
  - Beam Damage
  - Aesthetics
- Energy Dispersive X-ray Spectroscopy



SEM micrograph of a cat flea @ 350X

Goal for the one day SEM short course

- General goal: Be happy, treat others well, live long, and prosper
- 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 observe the basics of SEM operation
- To practice the basics of SEM operation, if you sign up for hands-on time
- It is assumed that you know nothing about SEM
  - It is assumed that you passed freshman physics...

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 ½ 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 ~ 550nm (green)
  - Visible light microscopes limited to ~250nm resolution
    - Can be improved using scanning (confocal and near field) and flourescence 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 <10pm
  - Diffraction limit of EM resolution <5pm (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 ~0.5nm for high end SEMs)

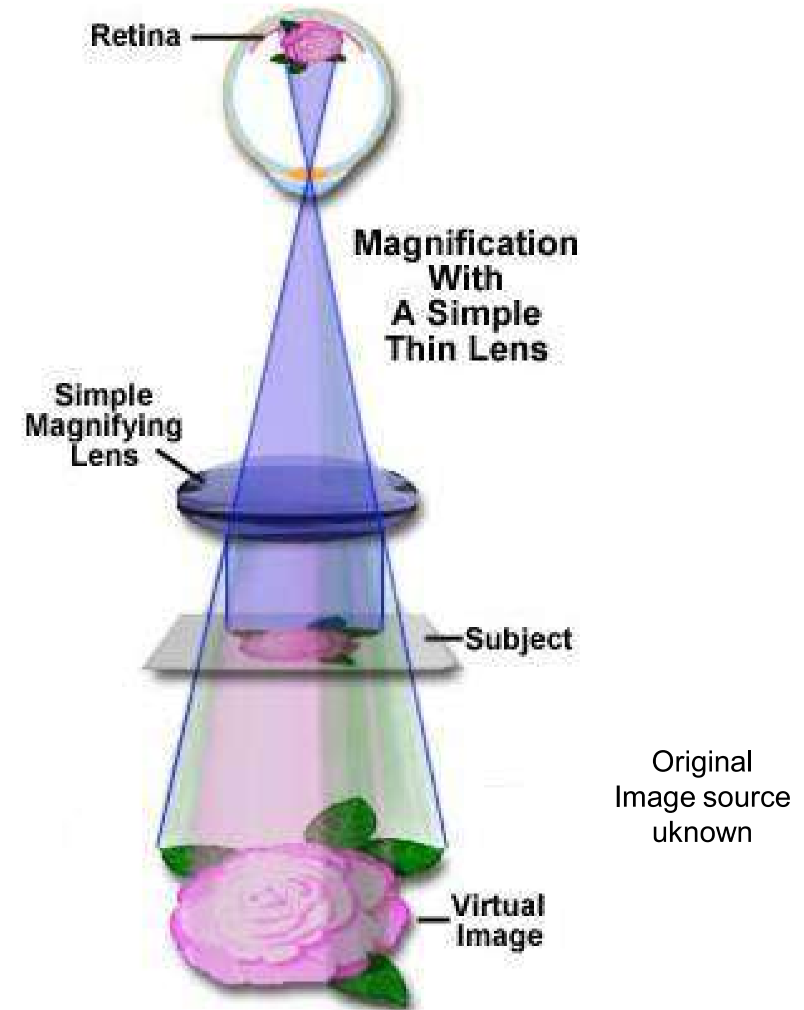
SEM general analysis scales

- Scanning electron microscopy, in general, is a micro-scale technique
- Nanometer scale features can be observed spatially
- Elemental analysis is on the micrometer scale
- Must worry about what is behind the feature being analyzed because the electrons don't just stop at the surface of the sample
- Nice thing about SEM is that bulk samples can be observed, often with little sample preparation
- Downside is that SEM analysis is a bulk analysis technique (micro-scale bulk)

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)



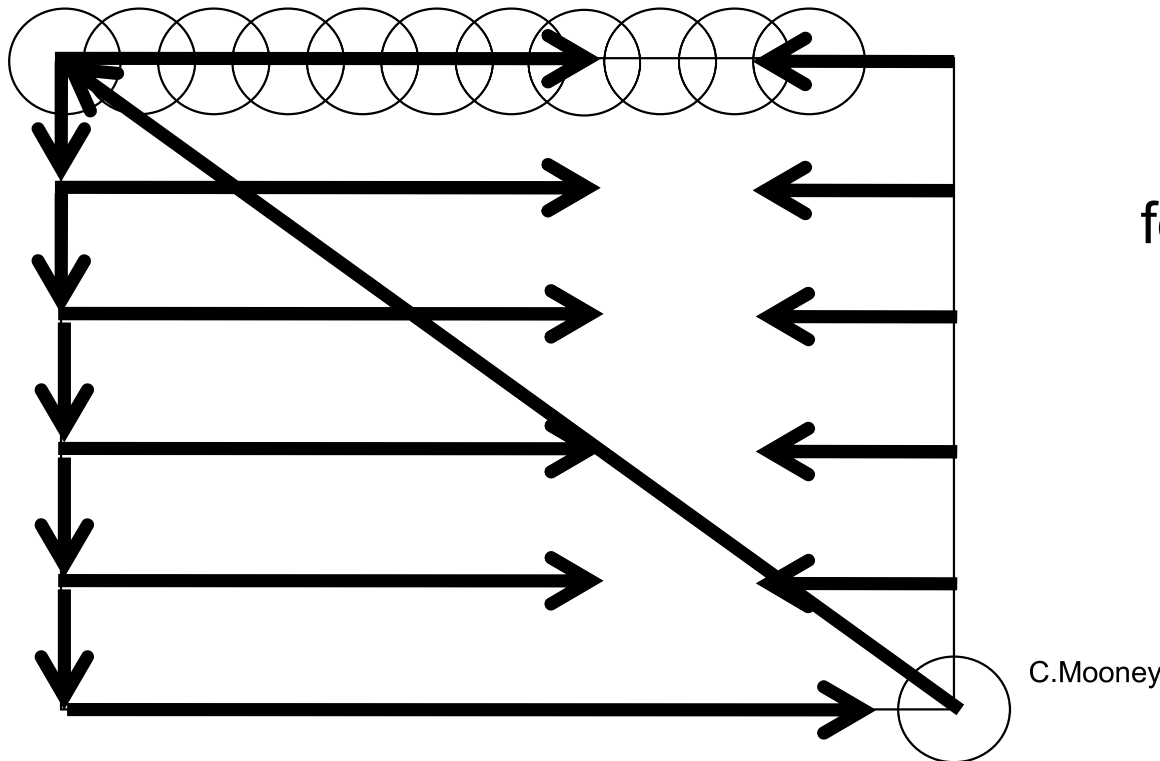
Original Image source uknown

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 XnYm 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…

Ag crystals

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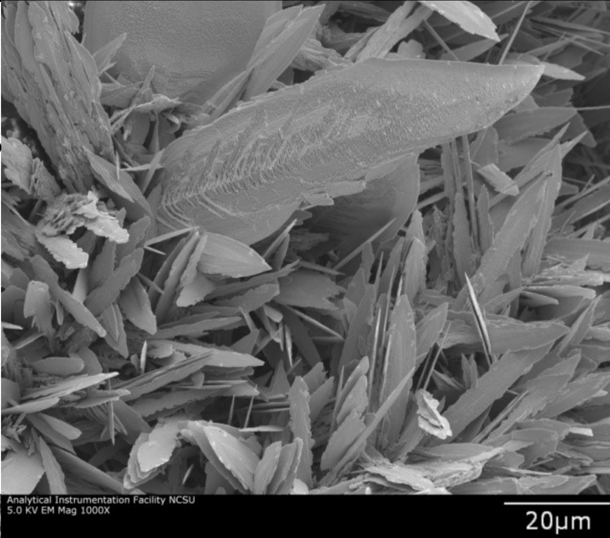
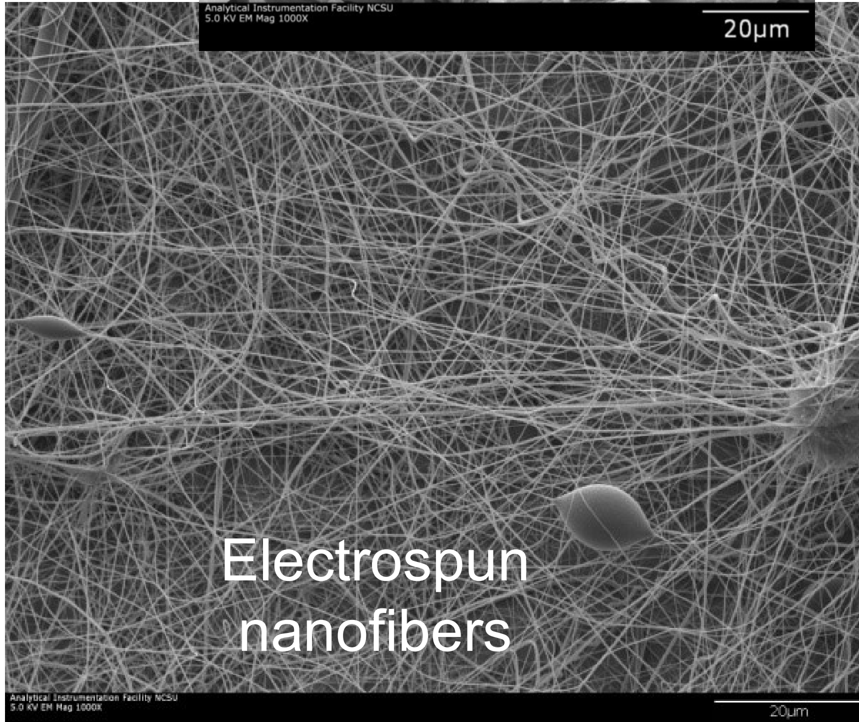
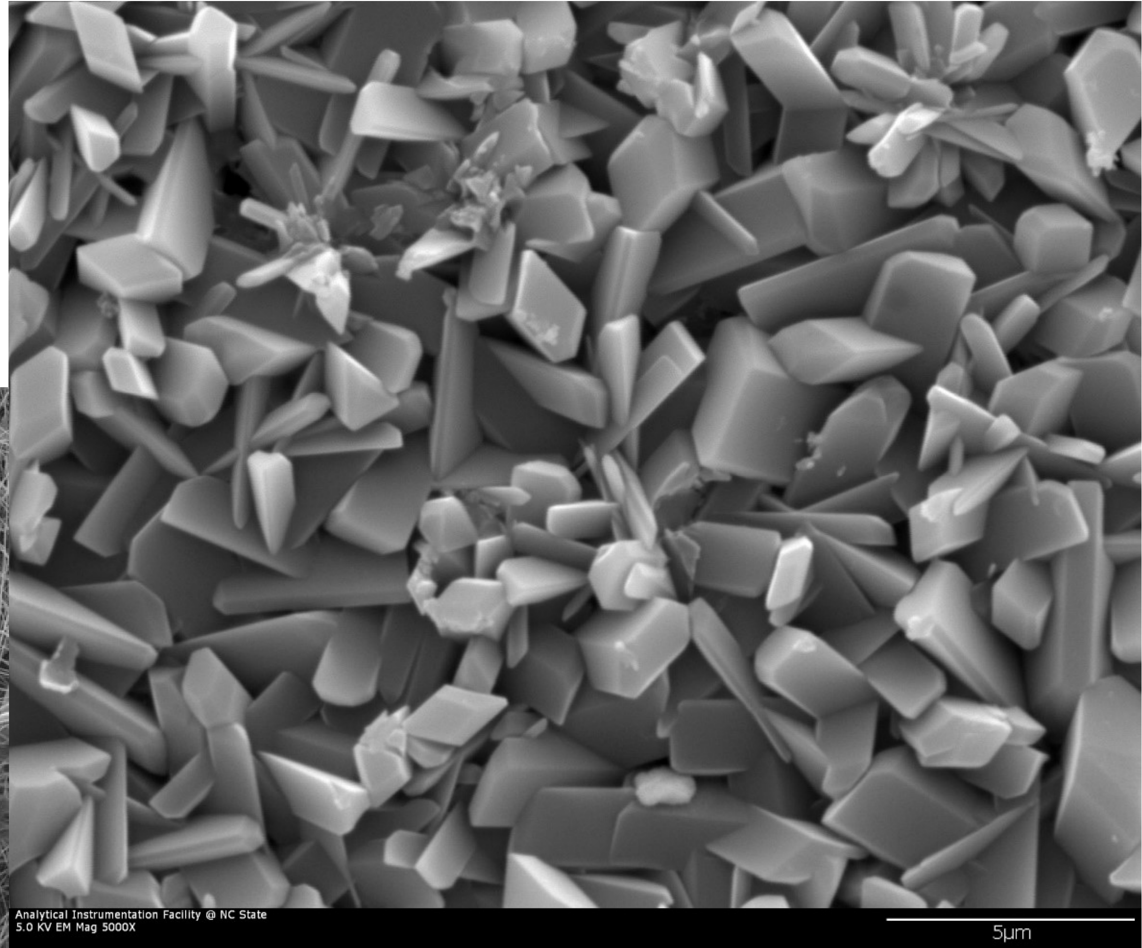






Manganese Phosphate protective coating on steel.

Scanning Microscopy Characteristics

- Optics do not form the image!

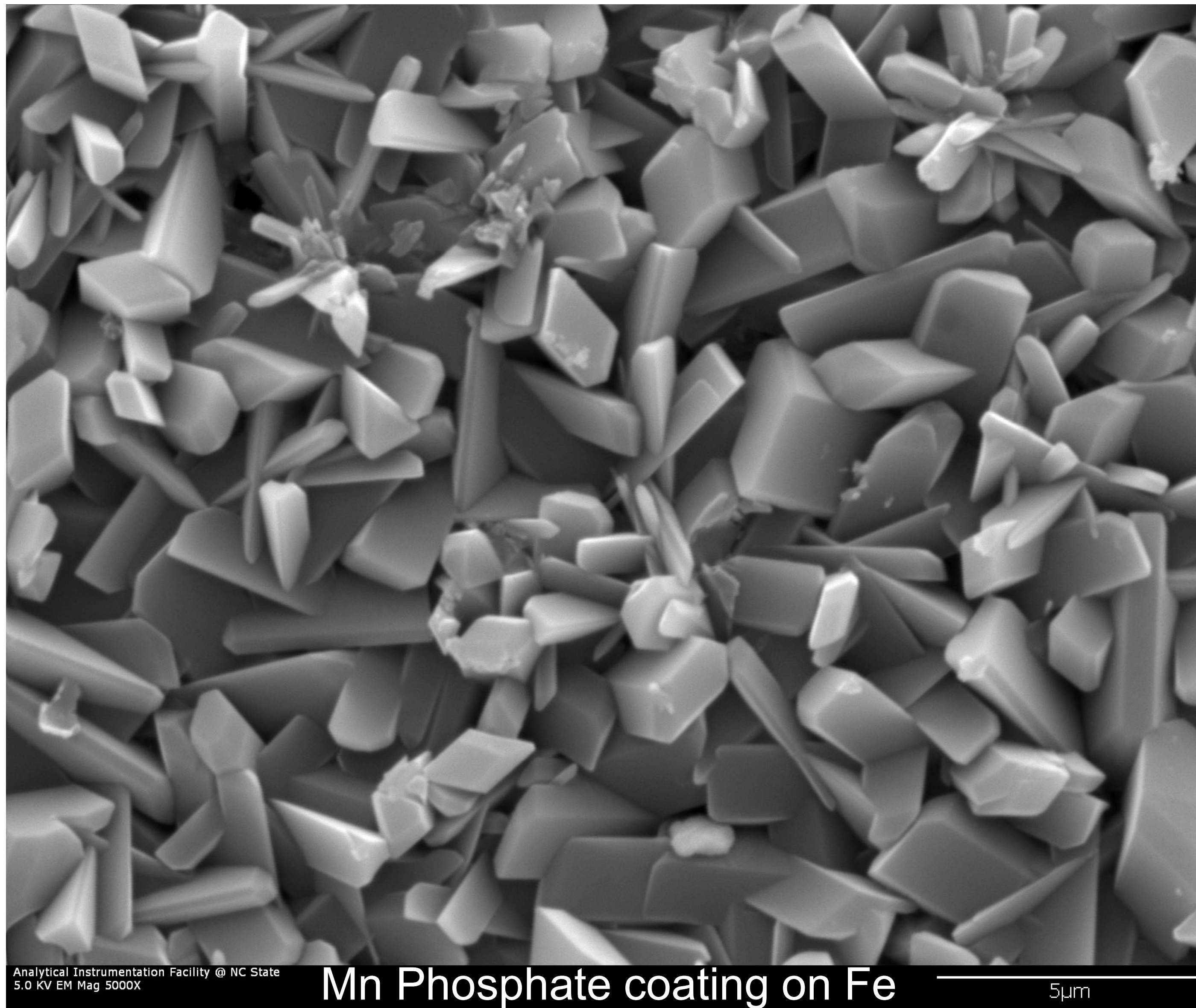
- Optics form the probe! (SEM probe = electron beam)
  - Optics = source, lenses, apertures, and correctors
  - Probe characteristics are determined by the optics parameters
  - Optics parameters include beam energy, current, spot size, and convergence angle
  - The probe is aka the beam or the spot (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!

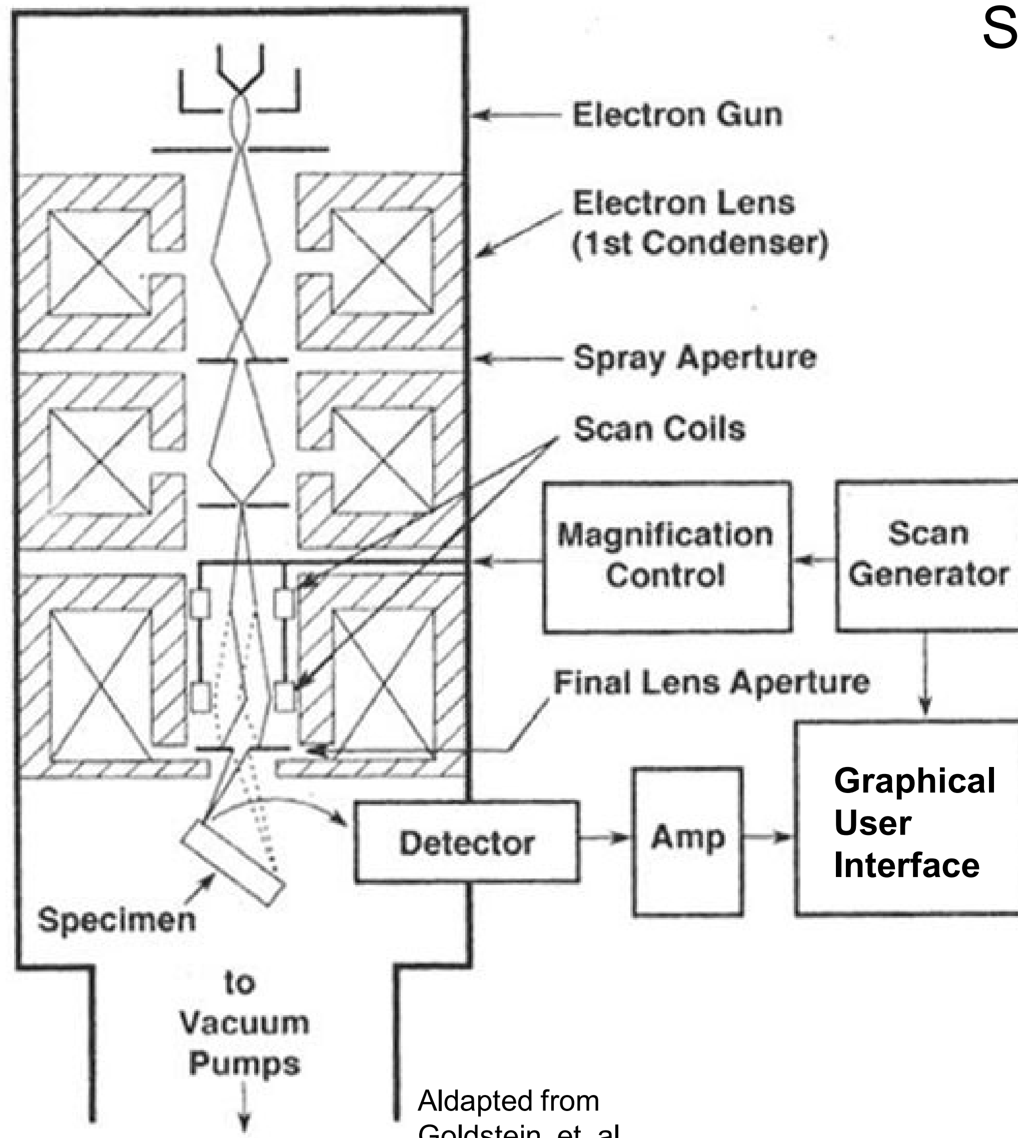
SEM in General

- 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 cathodolumenscence, 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



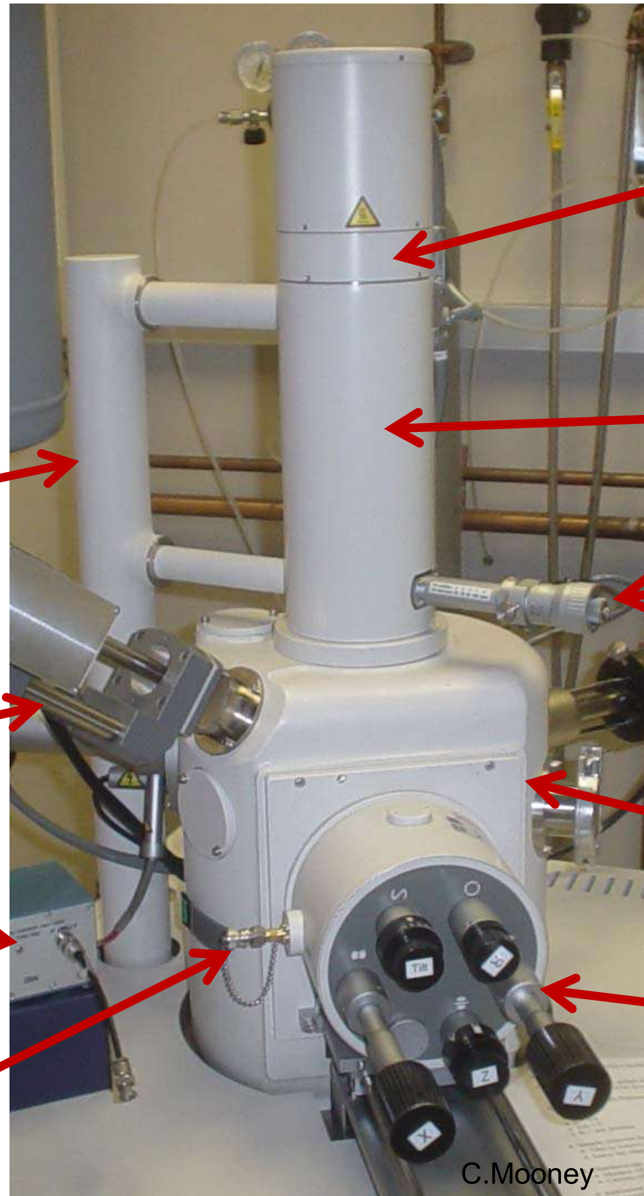
Aldapted from Goldstein, et. al.

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

SEM Labeled Photo

Photo showing the location of principle components of a prototypical conventional SEM



Electron Gun

Condenser Lens

Vacuum pipe

Objective Aperture Holder

X-ray detector

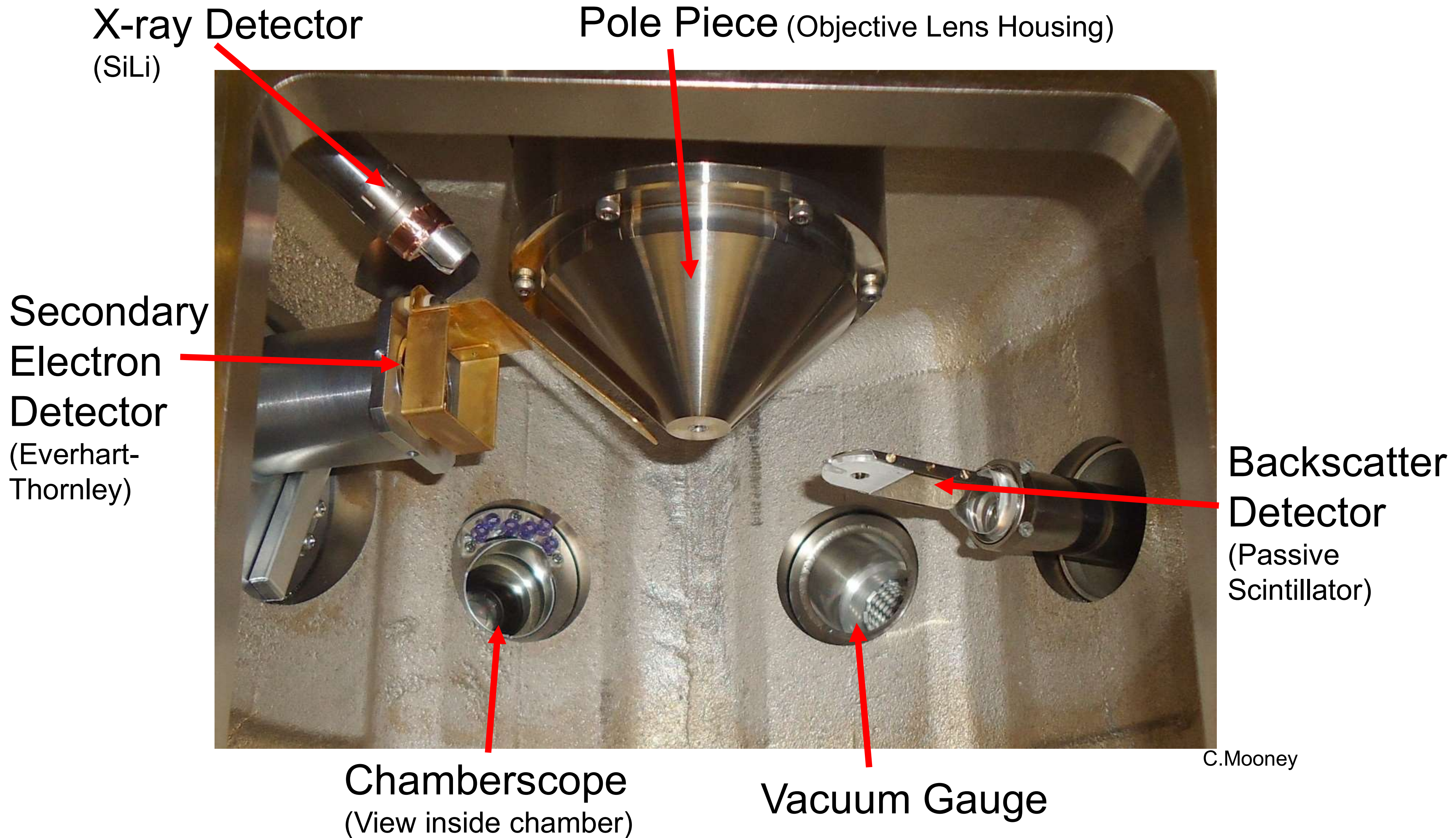
Main Chamber

Current Amplifier

Stage Controls

Stage Electrical Connection

SEM Inside the Main Chamber



SEM Labeled Photo

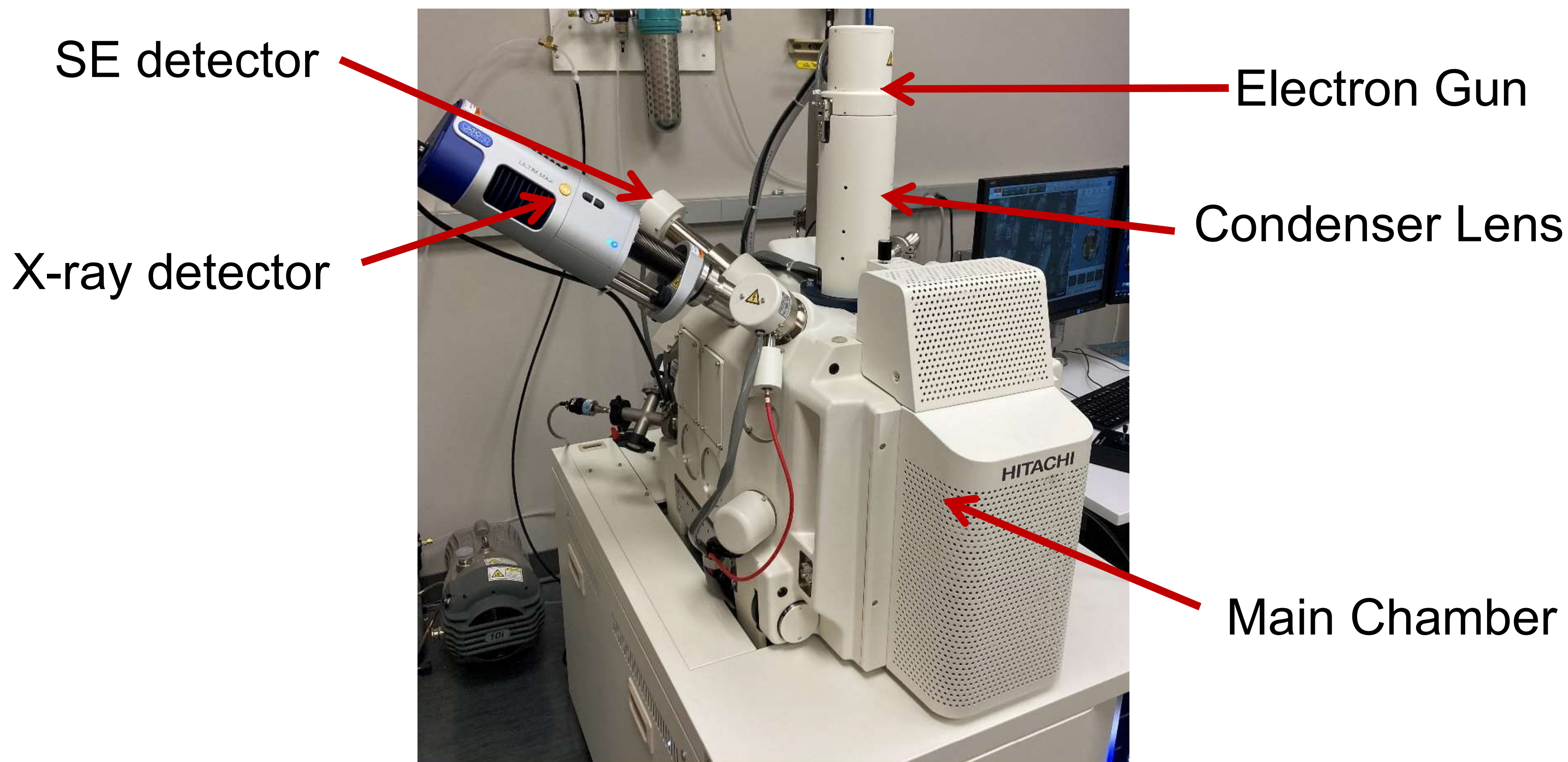


Photo showing the location of principle components of AIF's Hitachi SU3900 SEM

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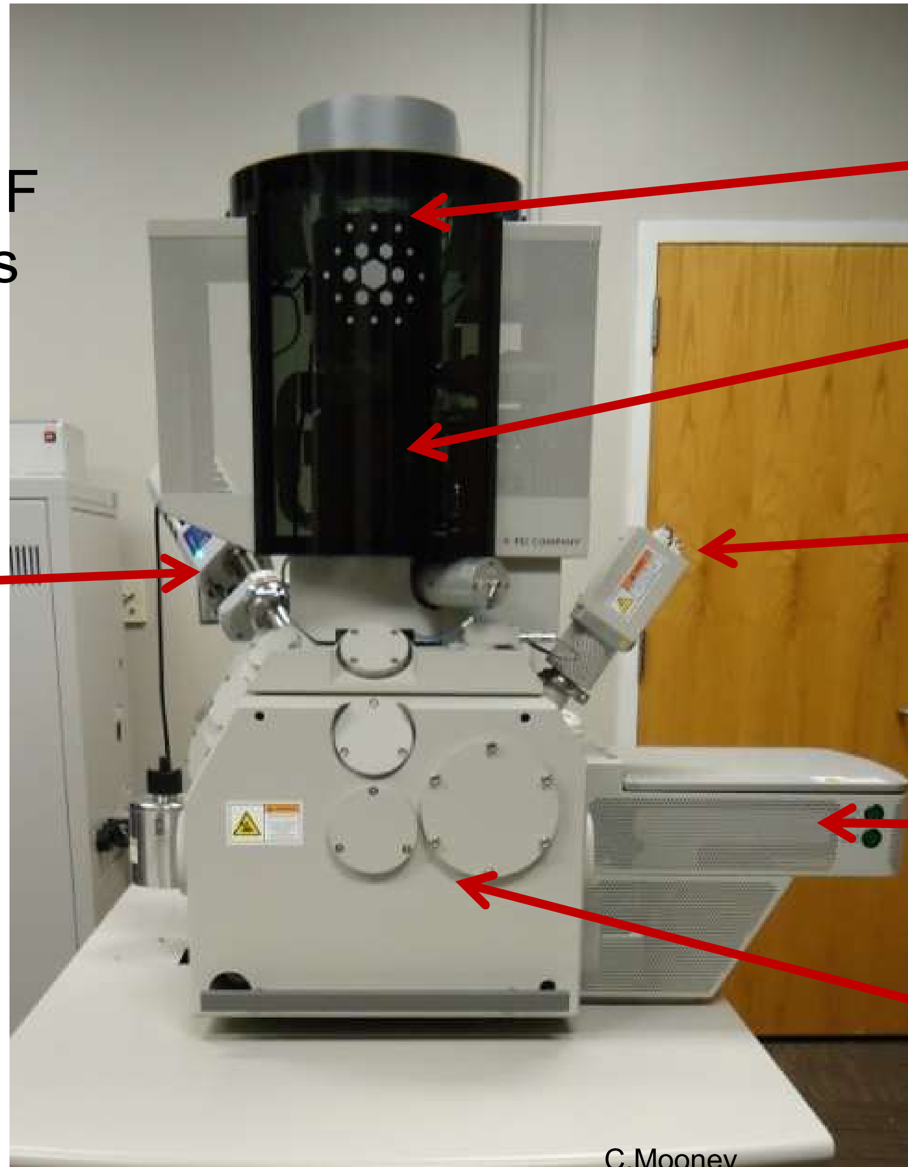
SU3900 Inside Main Chamber

Verios 460L SEM Labeled Photo

Photo showing the location of principle components of the AIF ThermoFisher Verios 460L SEM

X-ray detector



Electron Gun
(Schottky)

Condenser Lens

Oxygen Plasma Generator
(sample cleaning)

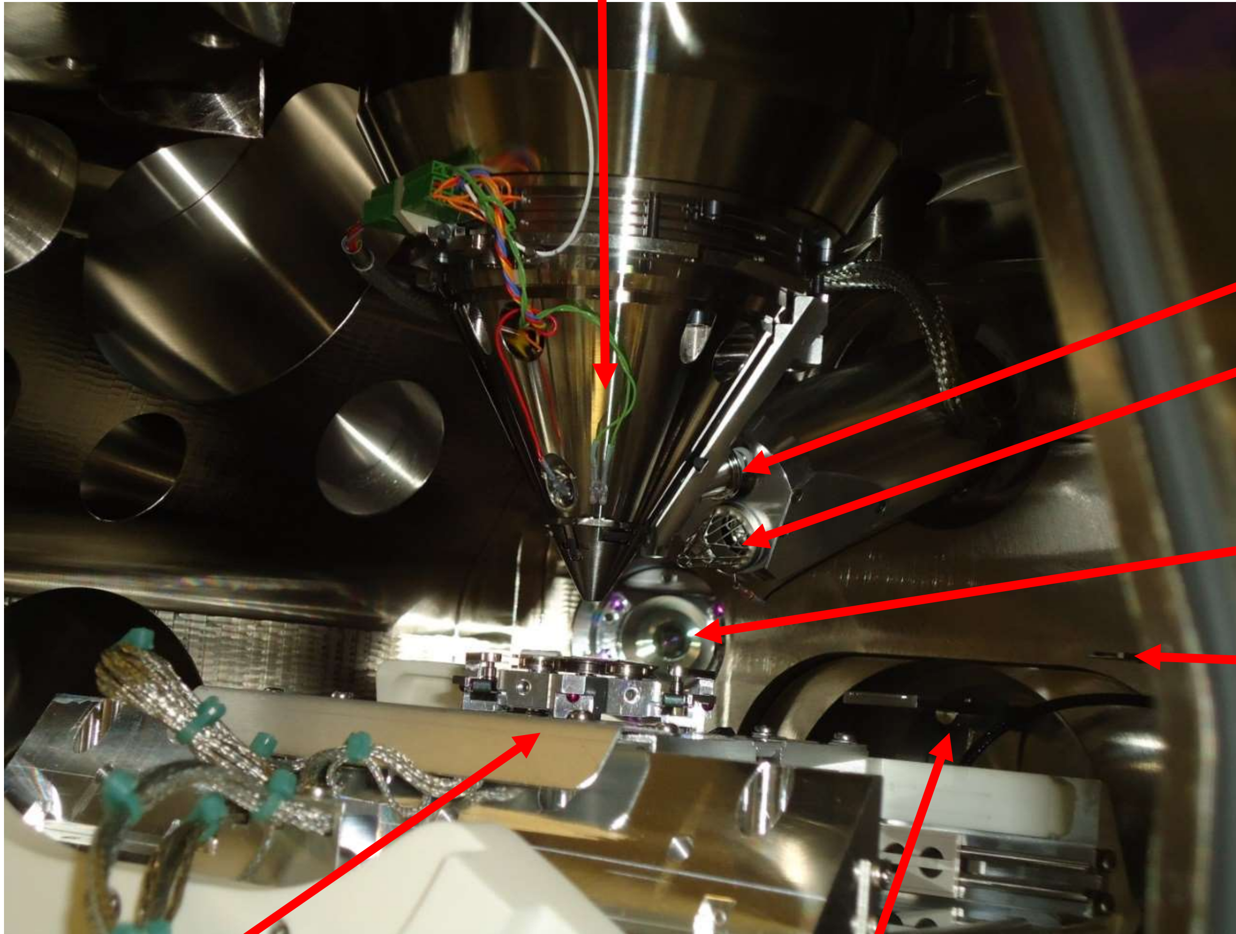
Load-lock Chamber
(Load samples)

Main Chamber

Verios 460L Inside the Main Chamber

X-ray Detector
(behind pole piece)

Pole Piece (Objective Lens Housing)



Secondary Electron Detector
Through the Lens
Everhart-Thornley

Chamberscope
(View inside chamber)

Backscatter Detector
(solid state, concentric)

Stage (w/shuttle)

STEM detector

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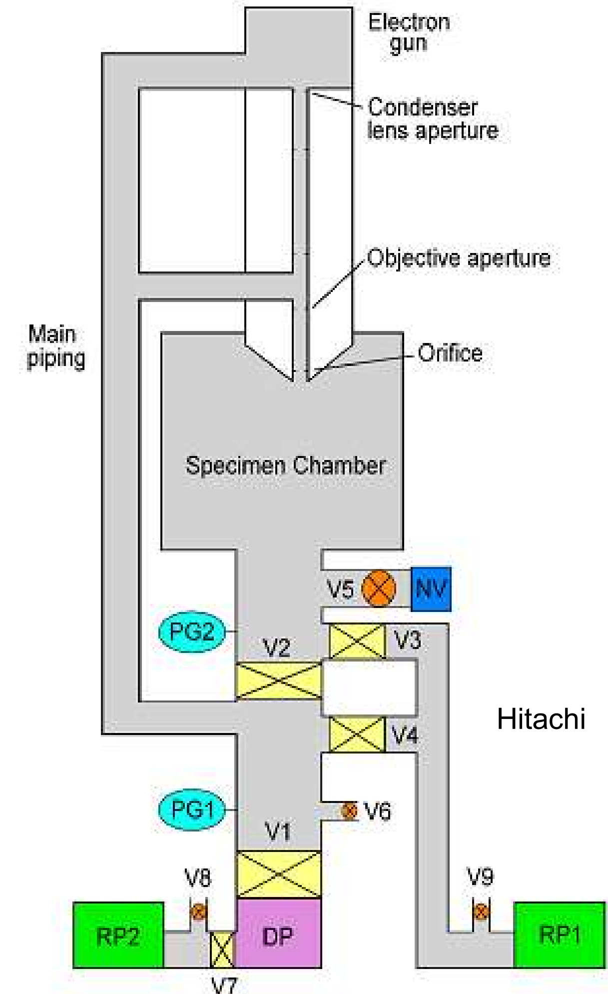
Vacuum Requirements for SEM

- 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

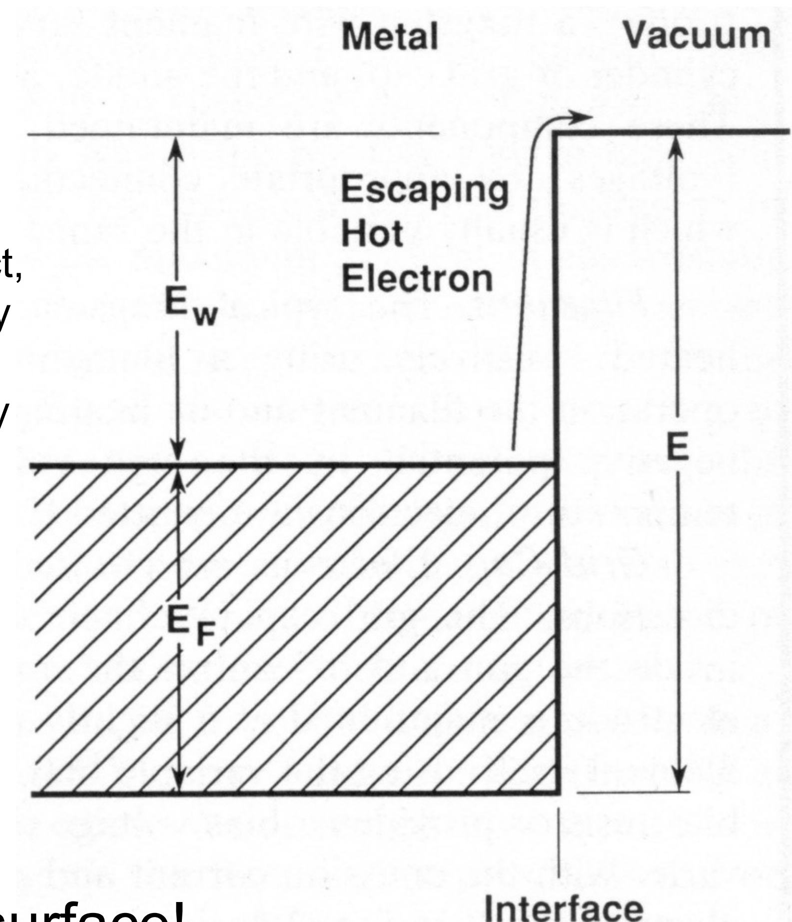
Electron Gun types

- 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 relativisitic 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



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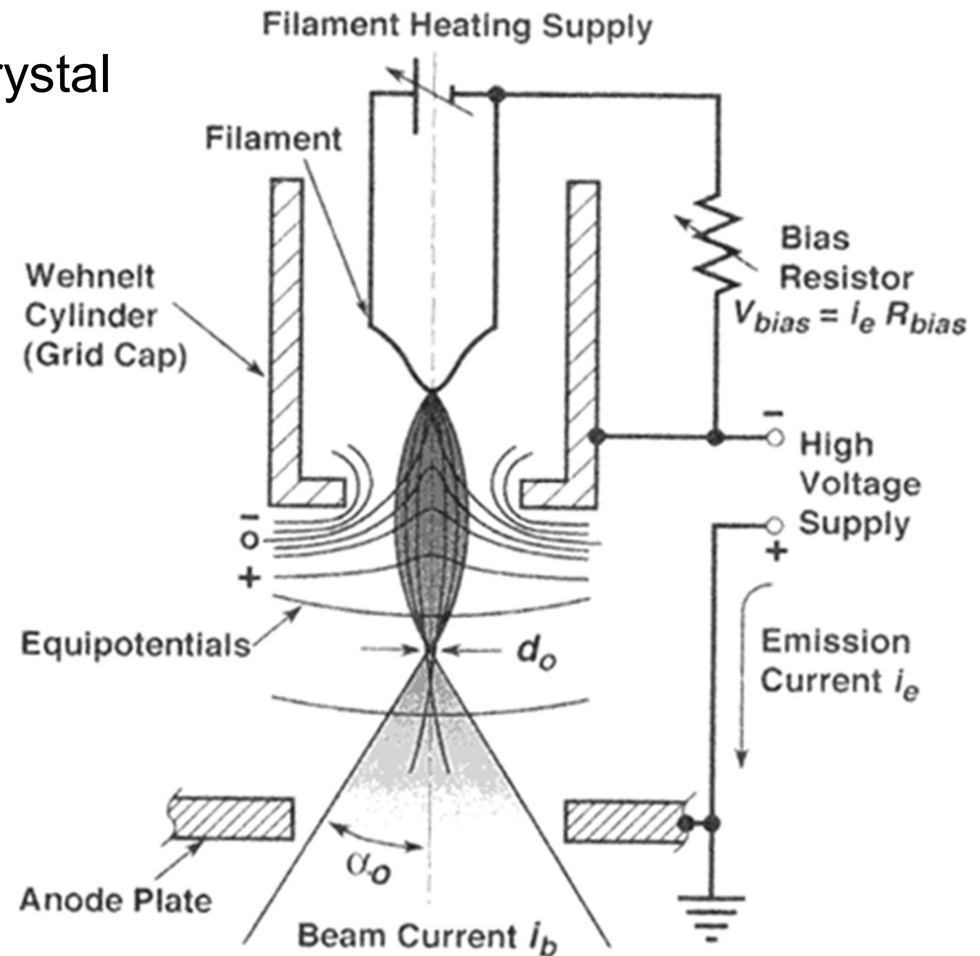
- Heat material until electrons boil off the surface!

Thermionic Electron Gun

Components of a Thermionic Electron Gun:

- Filament (plus heating supply)
  - Typically W wire or a LaB6/CeB6 crystal
  - 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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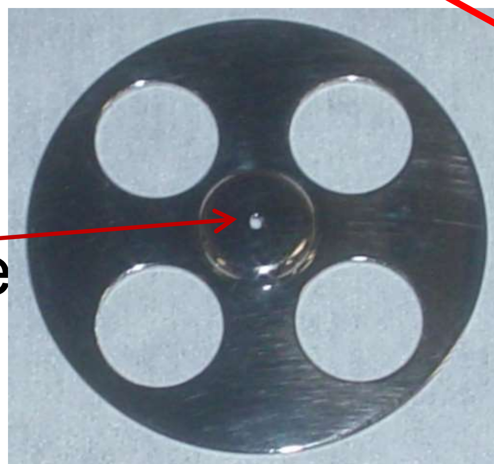
- Wehnelt Cap

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



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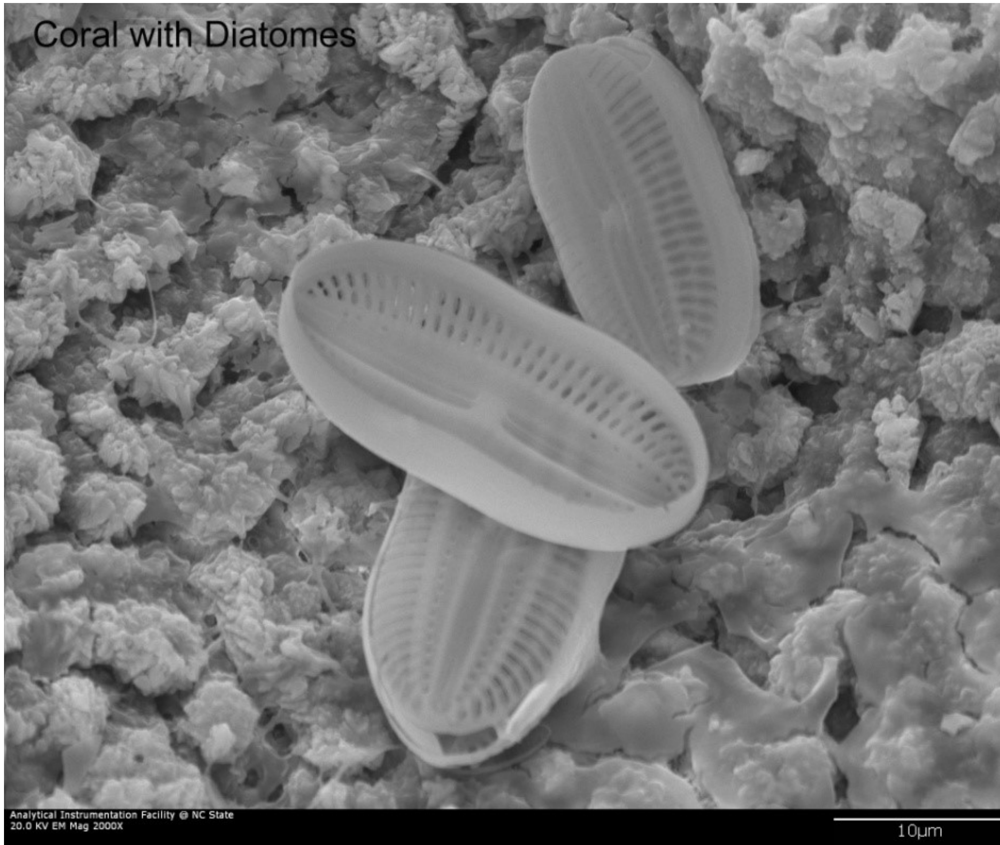
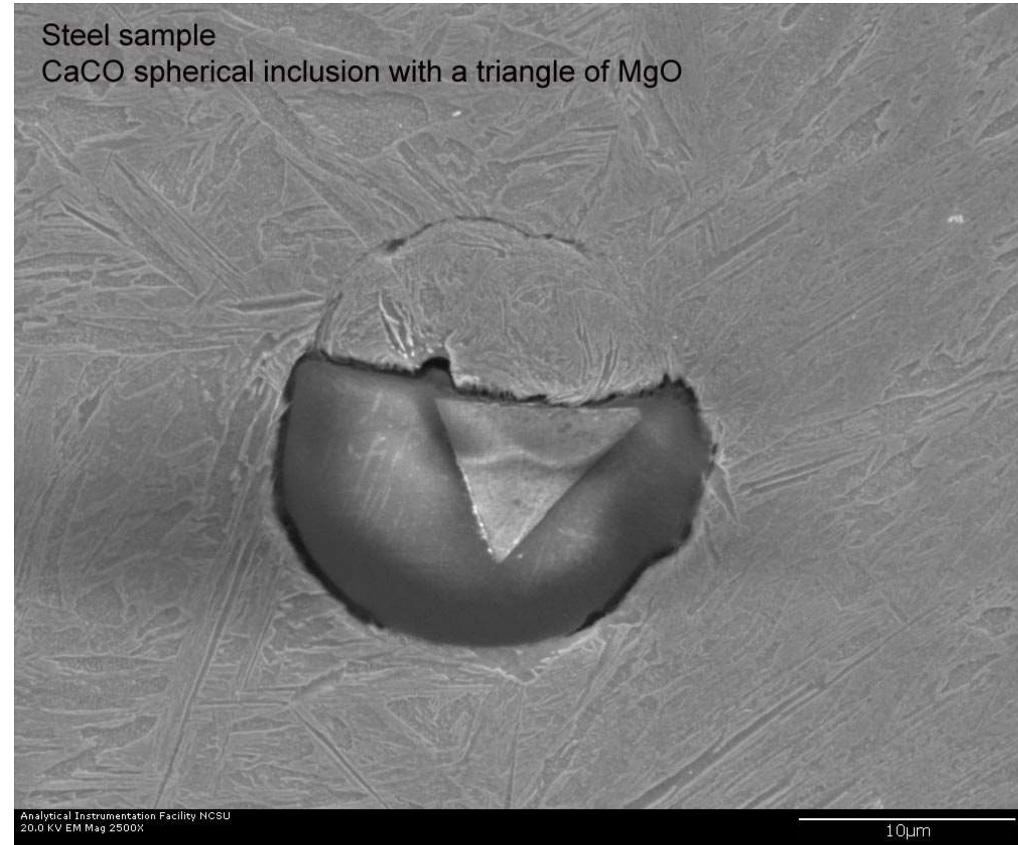
SEM Images from a Thermionic SEM





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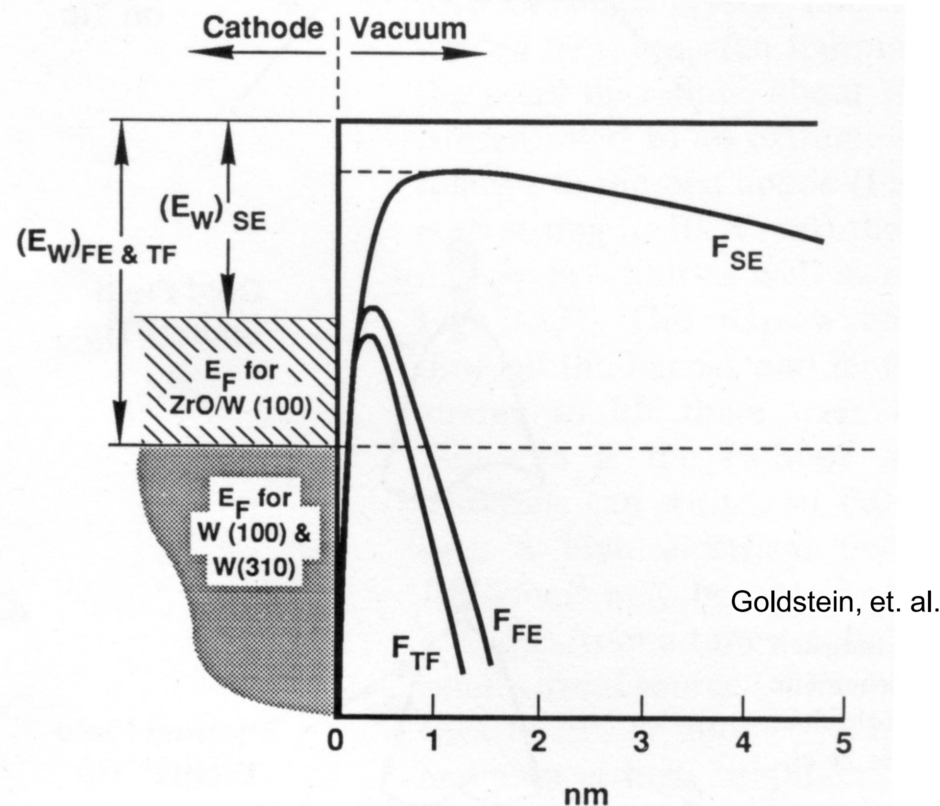
Field Emission Electron Source

- 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 (~10nm)



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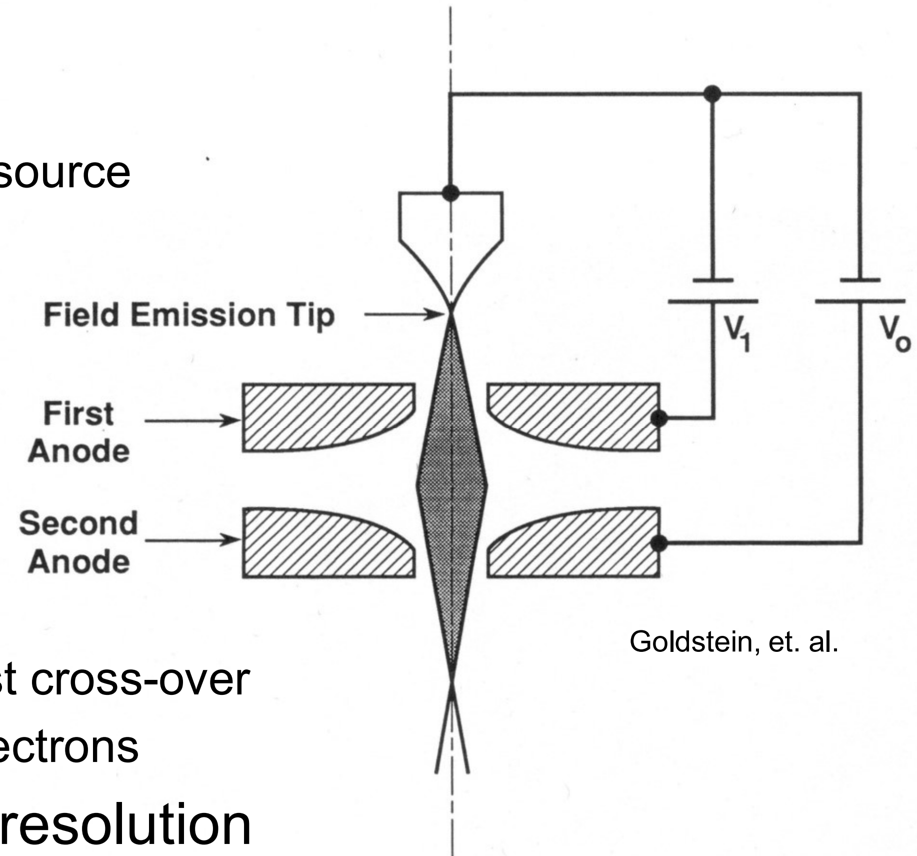
Energy diagram for field emission sources. Shown are cold FE (F_{FE}), thermal FE (F_{TF}), and Schottky FE (F_{SE}) emitters

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



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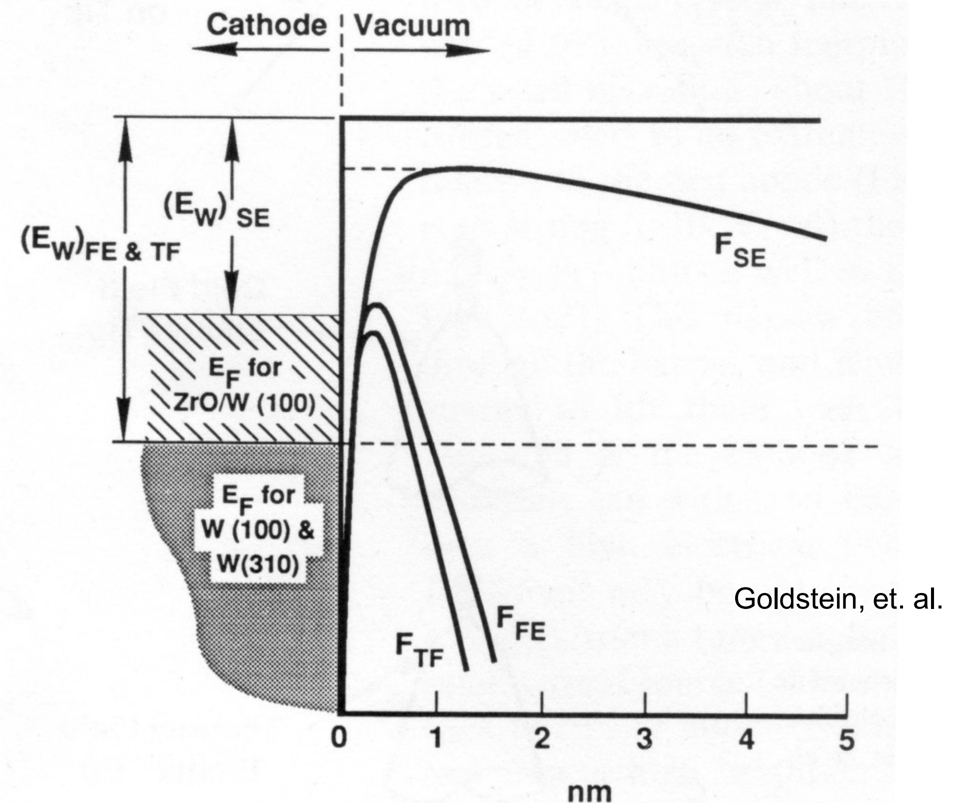
Schottky Emitters

- Schottky emitters are a special case
- Schottky emitter is really a field assisted thermal emitter
  - Look at the energy diagram!
  - Quantum Mechanical tunneling takes place over barriers < 3 nm!
  - SE curve suggests distances that are too great for quantum mechanical tunneling
  - The design of Schottky 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



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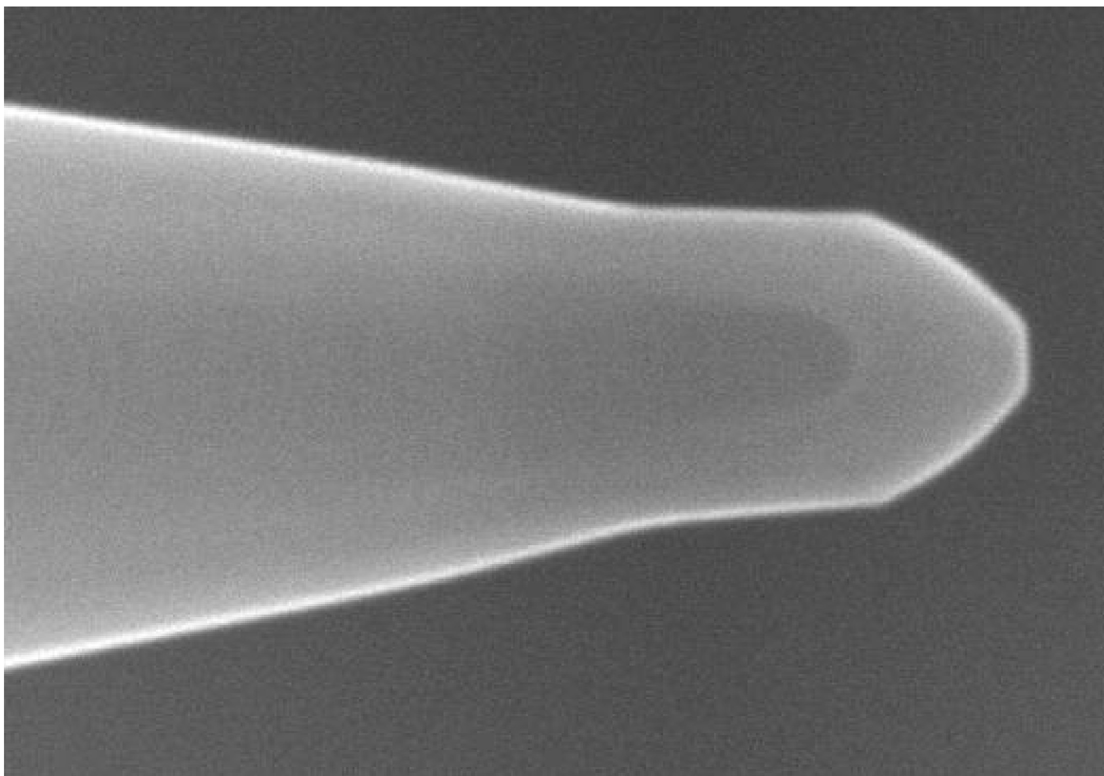
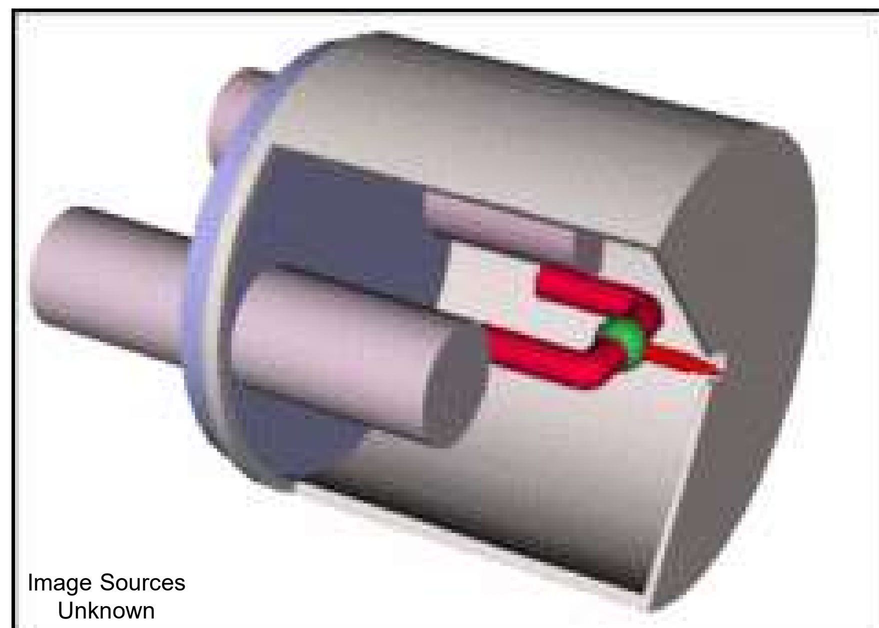
Energy diagram for field emission sources. Shown are cold FE (F_{FE}), thermal FE (F_{TF}), and Schottky FE (F_{SE}) emitters

Schottky 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 of ZrO).

Schottky emitter with suppressor cap assembly



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 ~ 100 x 150 **um**!)
  - 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.

Note on FE SEMs

- In theory, a cold FE source will give the best resolution
- In practice, Schottky sources are most commonly used in a FESEM

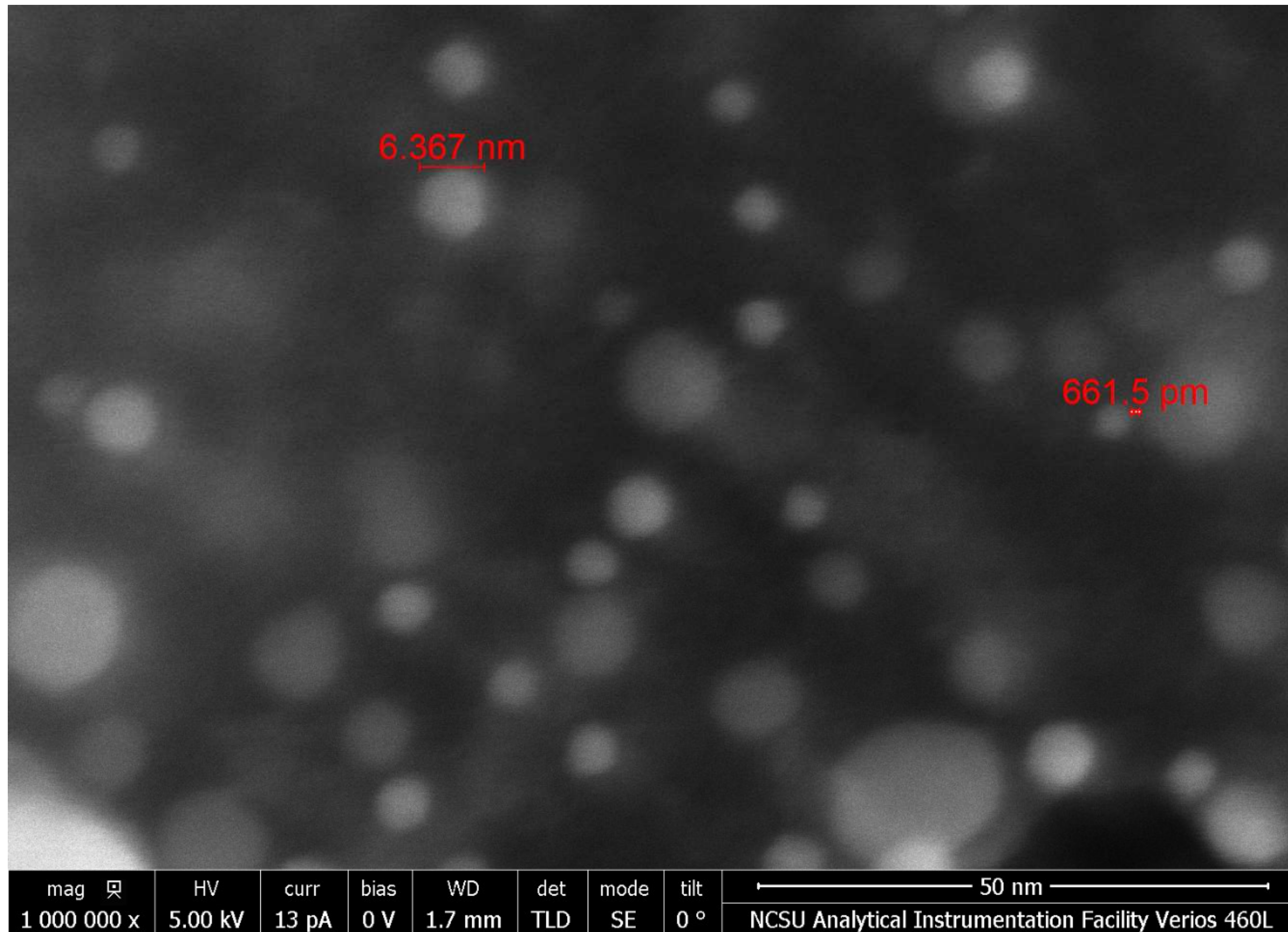
- Why?

- A Schottky source can produce more actual current than a cold or thermal FE source

    and

- There is a fundamental limit for SEM resolution and having a probe smaller than the resolution limit doesn't buy anything

1,000,000X SEM Image



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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.

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
  - Looks like a steel donut with some wires poking out
- Follows the Lorentz Force Law

Lorentz Force Law

- Generally expressed as:

$$\vec{F} = q(\vec{E} + \vec{v} \, x \, \vec{B})$$

- $q(E + v)$ Is the electric force
- qB Is the magnetic force

- In the absence of an electric field, we can ignore the electric field (E) component

- In the case of a single electron (q = -e) moving with velocity $\vec{v}$ through a magnetic field $\vec{B}$ with no electric field, we get...

Electron Lenses

- Follow the (simplified) 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

- The cross product means:
  - 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

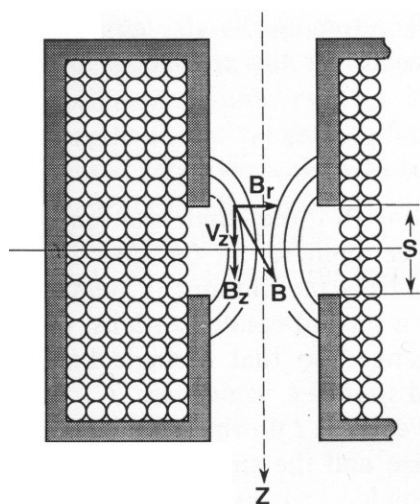
- Electrostatic lenses are possible, but the geometry is more complex

Electron Lenses

- Electromagnetic lens:

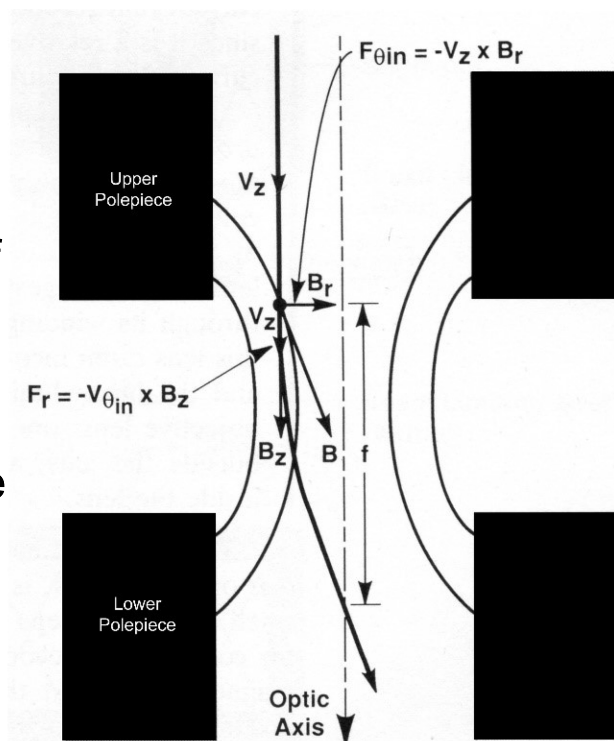
  An Fe housing that contains windings of Cu wire

  – 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:

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

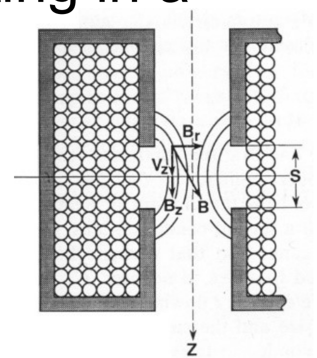
- Electrons with different velocities get focused to different places, which is like not being in focus!

- 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 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 Schottky 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

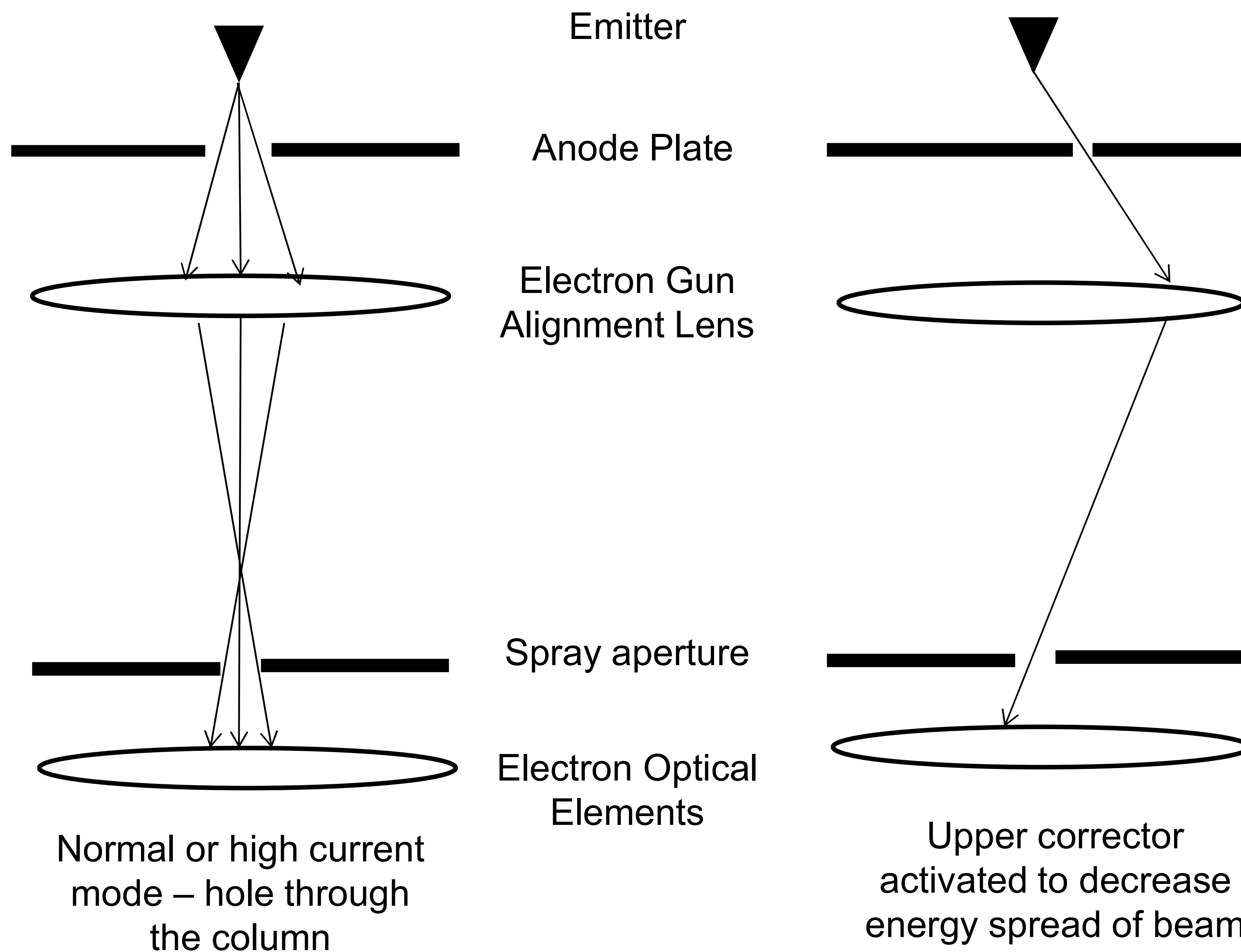
- The electrons that pass through the slit have a smaller energy spread than those that go through the main hole in the anode plate

- This reduces the energy spread in the beam by approximately an order of magnitude
  - +/- 1 eV uncorrected
  - ~ +/- 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

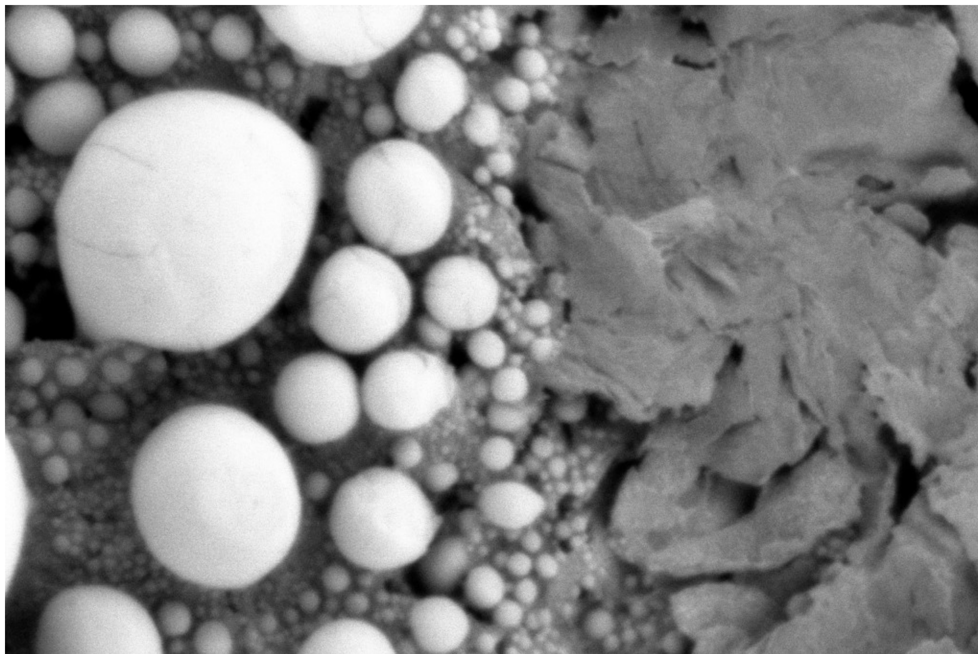
- At low voltage/current, use it if you got it…

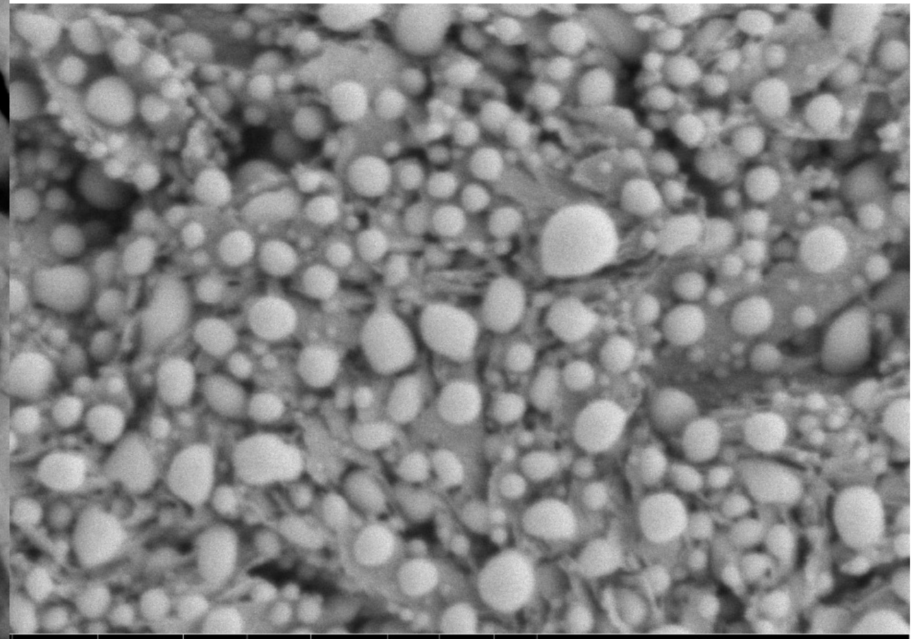
Upper Corrector Schematic



Upper Corrector – Verios 460L

- At low voltage/current, use it if you got it…
- At high energy, it doesn't make enough difference to be measureable
  - Consider a 20 keV beam with an energy spread of +/- 1eV
  - The difference between a 19,999 eV electron and a 20,001 eV electron is pretty small
  - Consider the same difference for a 100 eV beam... (2% difference)





CMooney

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

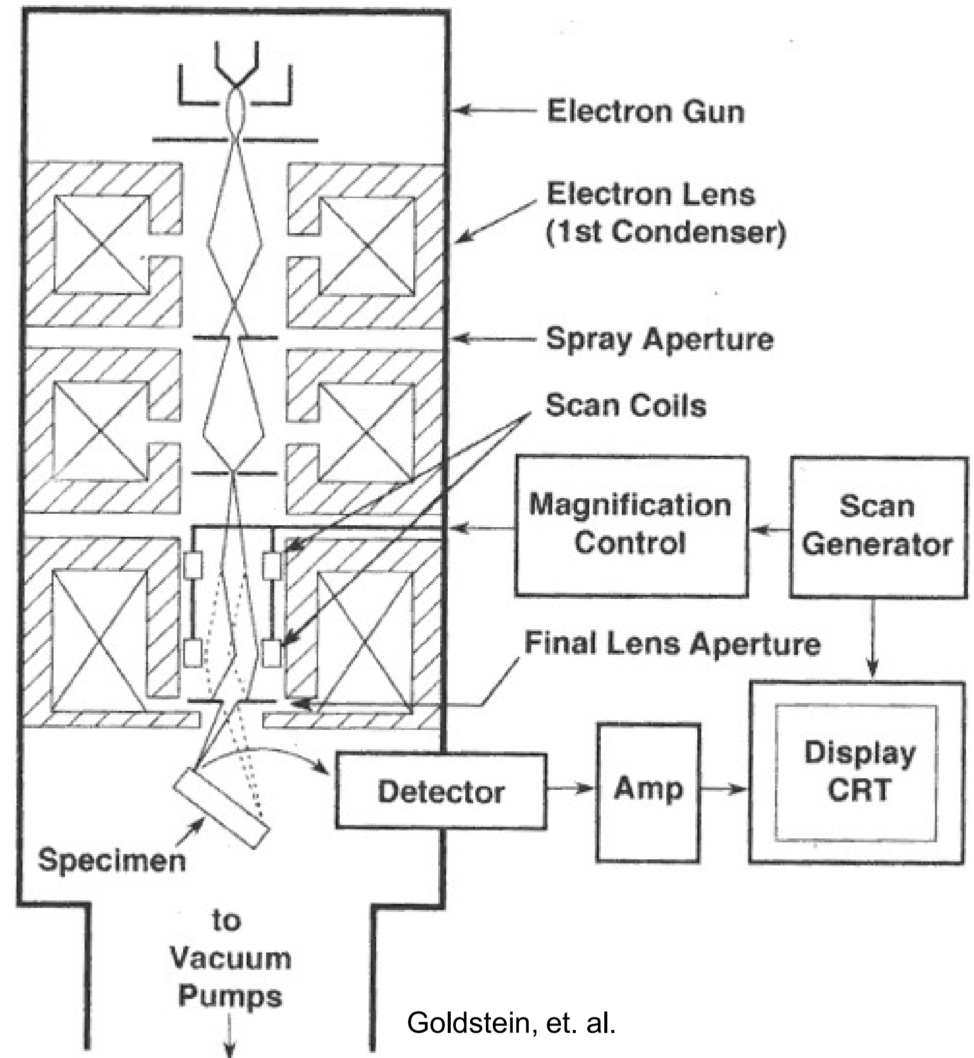
- Objective lens
  - Focuses probe on the sample, i.e., forces the probe into a small round spot at the surface of the sample

SEM Condenser Lens

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



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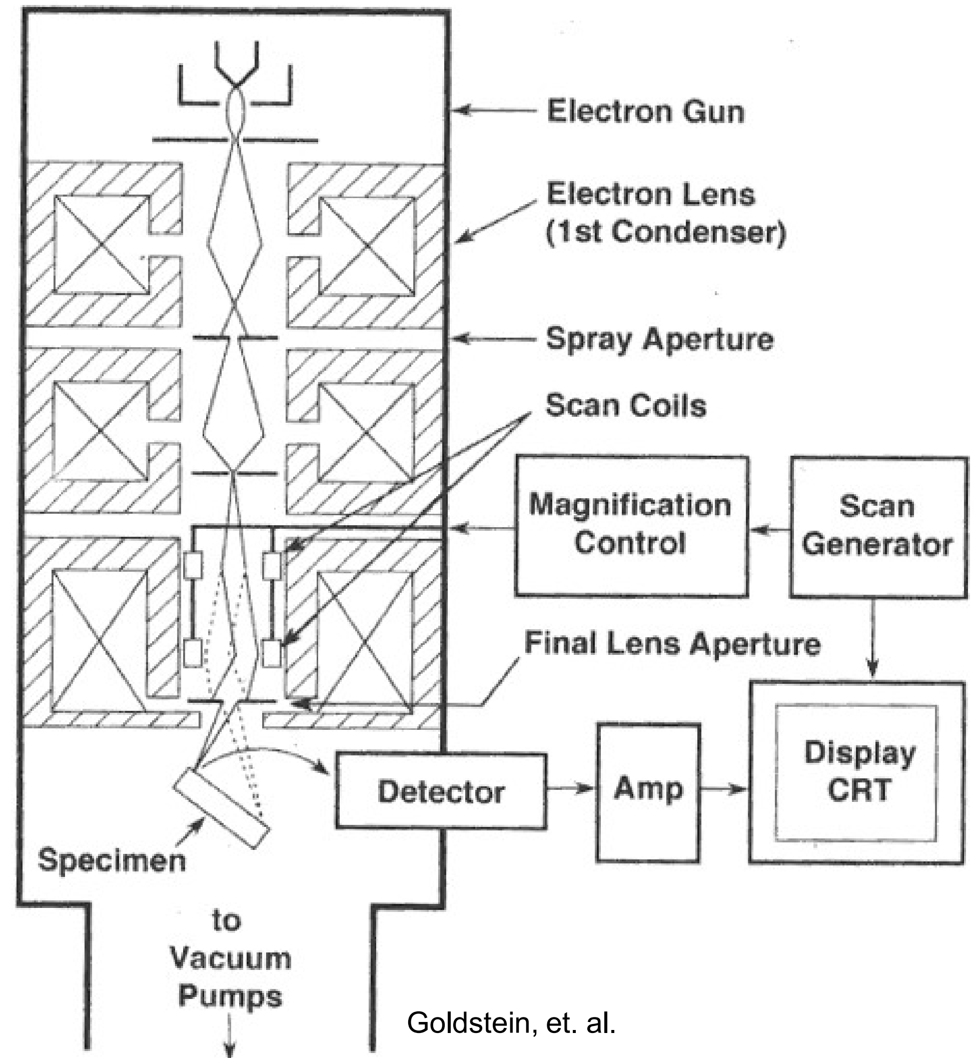
SEM Condenser Lens

CL system does two things:

1. Demagnifies the initial spot
   - Done to improve resolution
   - What we want!

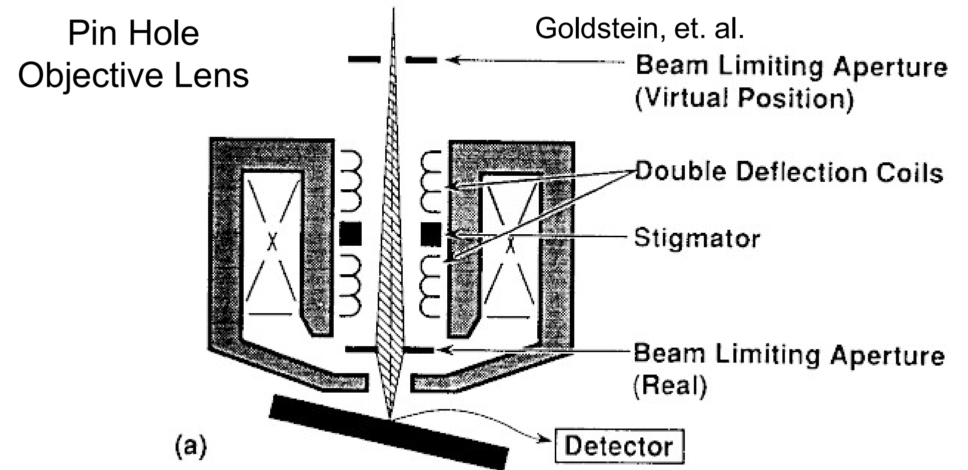
2. Limits beam current as electrons that strike the inside of the column are lost
   - As the lens strength is increased, the cross-over from that lens moves up in the column
   - Since the column has a finite diameter, the geometry means that some electrons will strike the inside of the column
   - 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



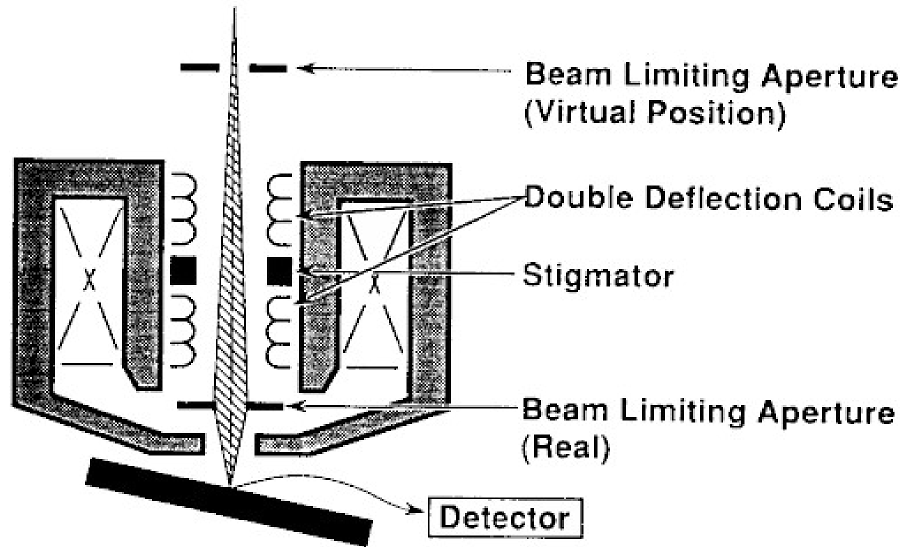
Goldstein, et. al.

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



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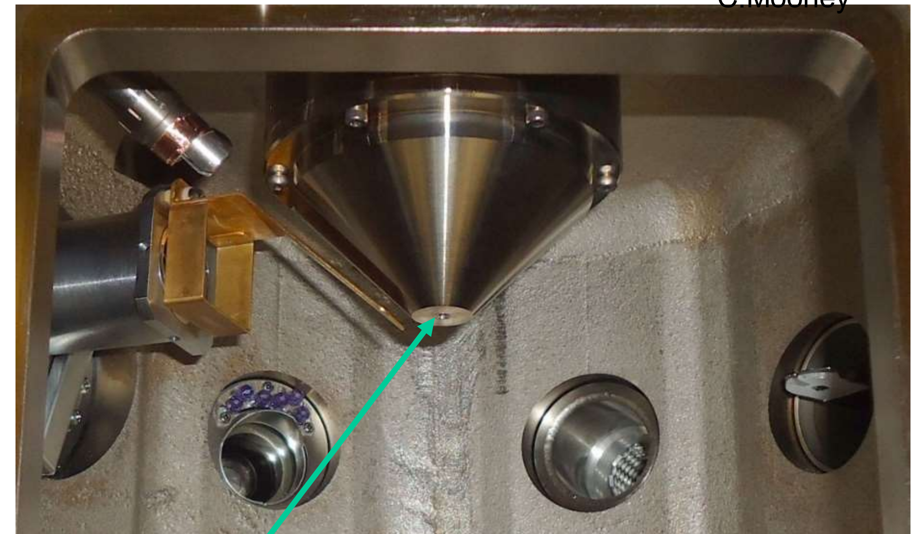


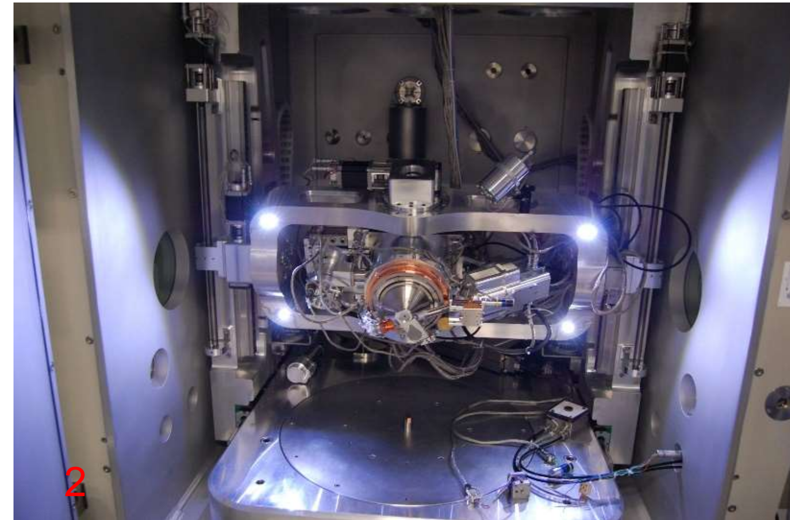
Objective lens, aka the pole piece, inside the sample chamber of the VPSEM

- Schematic of typical pinhole lens
  - Most common type of objective lens
- Conventional (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!

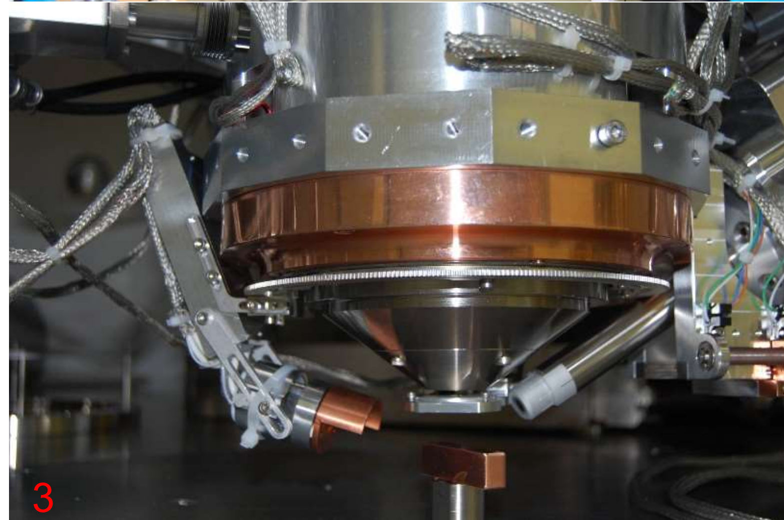
Giant Sample SEM

Window (blue cover)
2 meters above floor

Vibration
isolation leg



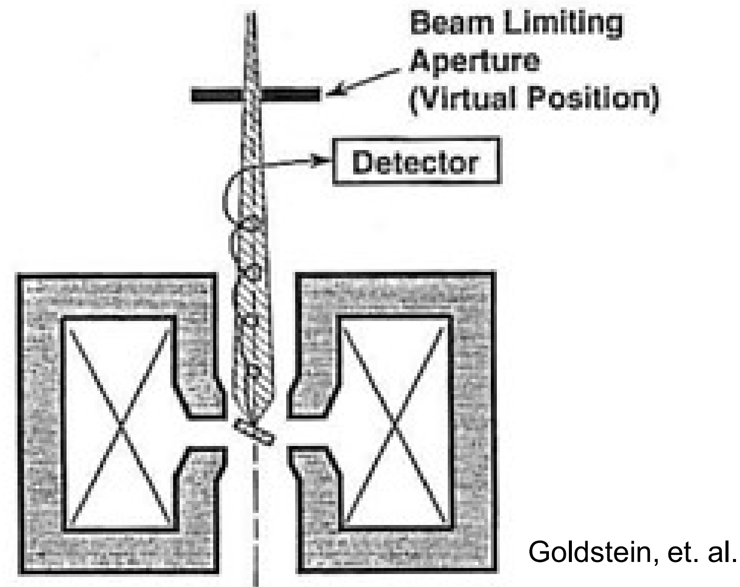
Visitec
Microtechnik,
USAF Tinker
Air Base



4

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



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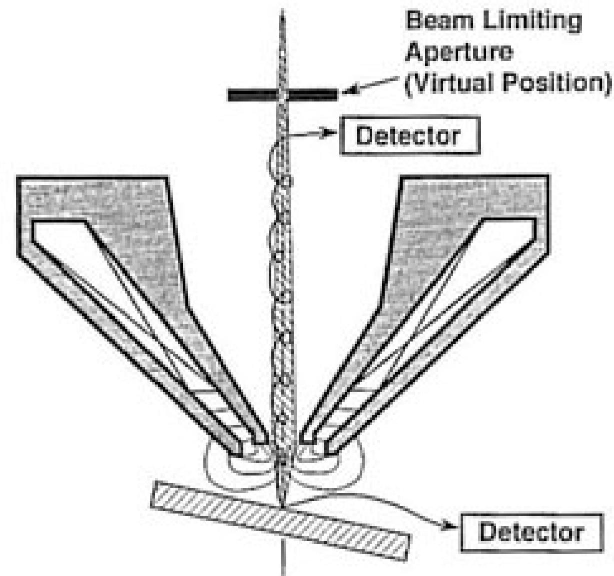
In lens SEMs have the highest possible potential resolution with current models claiming 0.4 nm

- 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

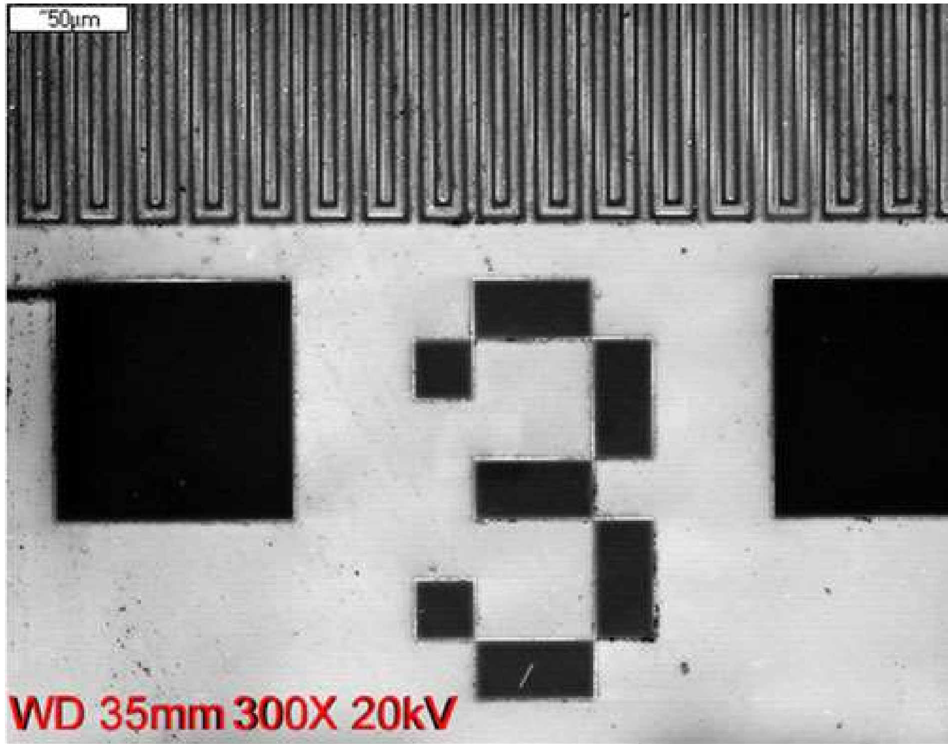


The immersion lens is aka the semi-in lens or snorkel lens

Goldstein, et. al.

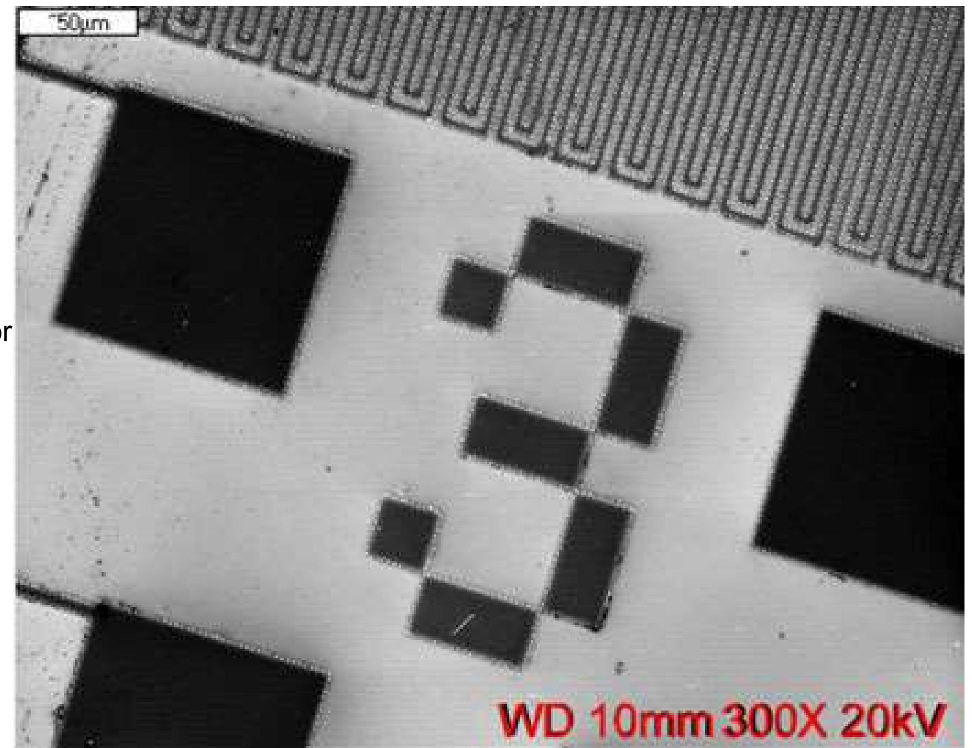
- Schematic of typical immersion design SEM
- Sample is held below the lens but is immersed in a magnetic field emanating from the lens
  - With an electromagnetic immersion lens, ferromagnetic samples cannot be observed
  - UHR-SEMs are available with electrostatic or hybrid lenses that allow imaging magnetic samples
- 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



D.Batchelor

(Slight moire effect here)



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!

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

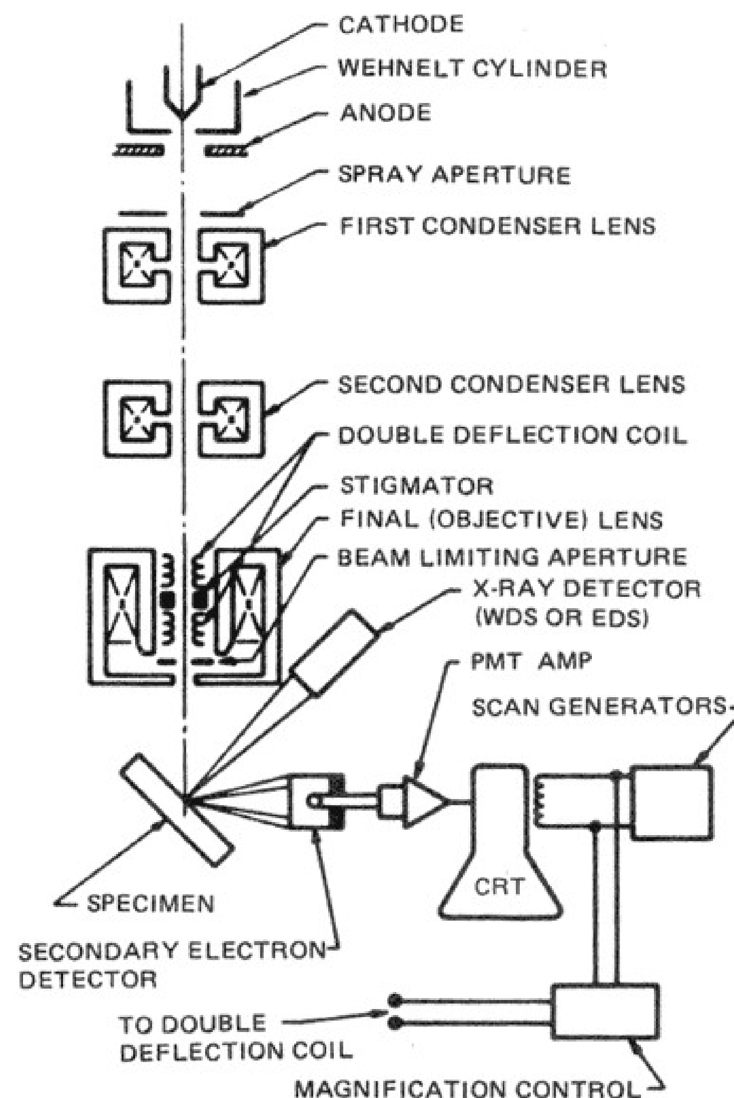


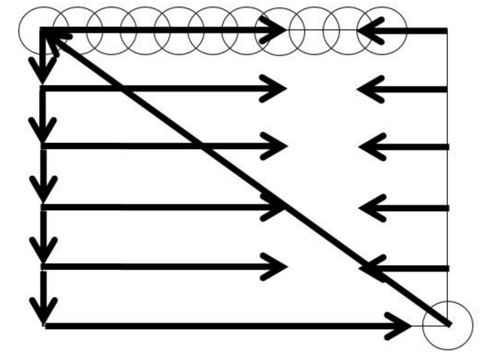
Image source unknown

Heavy Duty Mathematics

- Not really...
- When one sits down and works through some of the theoretical aspects of electron optics, one finds:
- High energy electrons have a shorter wavelength and are therefore capable of higher resolution than low energy electrons
- High energy electrons are easier to control in the column than low energy electrons
- Small spot sizes are easy with high energy electrons
- Result: In a conventional SEM, high energy produces better resolution and (often) prettier images
  - Modern conventional SEMs are very good down to ~ 5 keV
  - Unconventional SEMs perform well at low energy
    - Verios resolution spec: 0.5 nm at 2 – 30 keV, 0.75 nm at 1 keV, 1 nm resolution at 500 eV
    - Quanta FIB (conventional FESEM): 1.2 nm at 30 keV, 2.9 nm at 1 keV
    - SU-3900 VPSEM spec: 3 nm at 30 keV, 15 nm at 1 keV, 4 nm at 30 keV in VP mode

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.

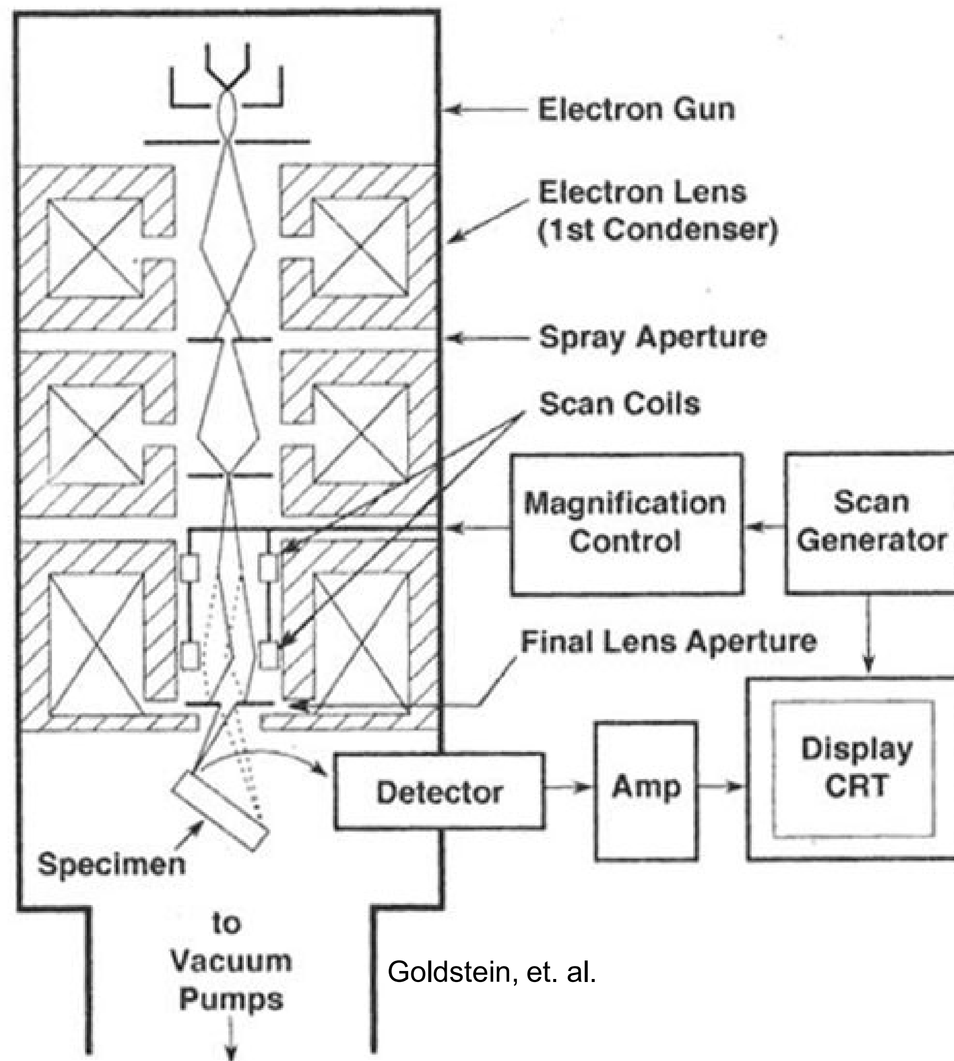
Deflection Coils

- 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!



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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

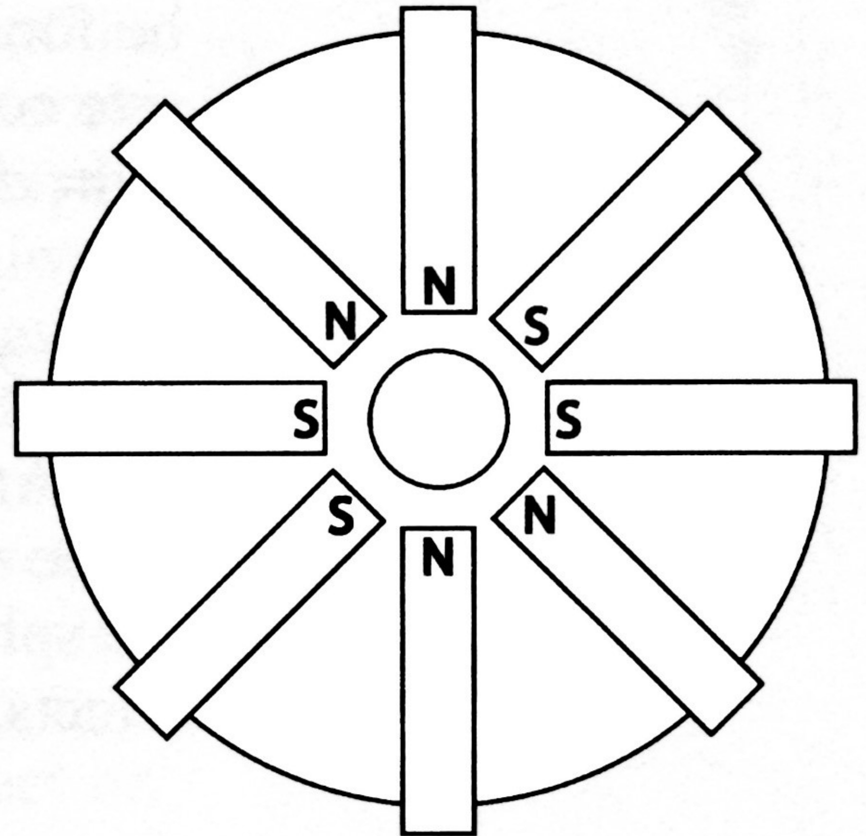
- Stigmatism definition: The condition of an optical system (such as a lens or mirror) in which rays of light (from a point source) converge in a single focal point
- In an SEM, astigmatism means that the beam is not round
- Initial Spot formed by the Wehnelt cap in a thermionic SEM is generally not round
  - Emission Area ≈ 100x150 μm
    - ***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 can be 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

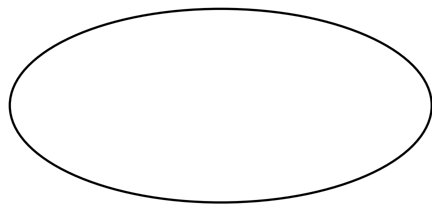
- The octopole creates an ***asymetric*** magnetic field to correct astigmatism in the electron beam

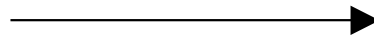
- The beam passes through the center of the octopole

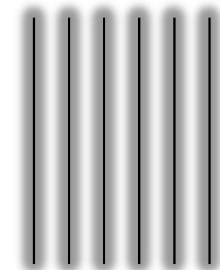


Astigmatism

- 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





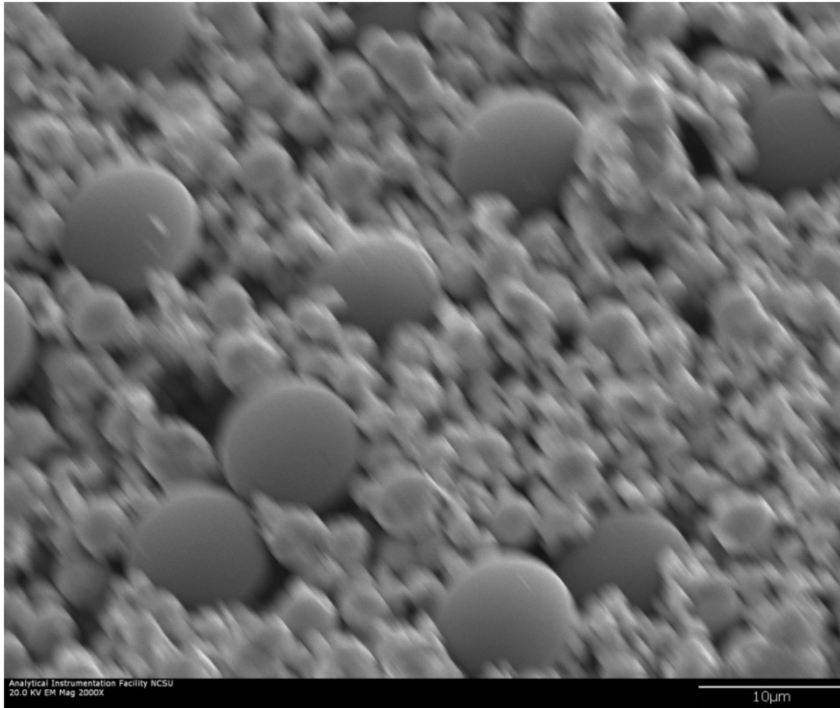


Elliptical Beam

Sharp Features

Blurred Features

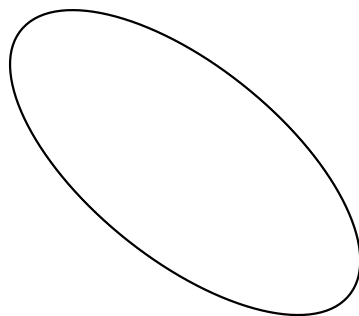
Astigmatism Correction



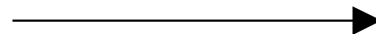
C.Mooney

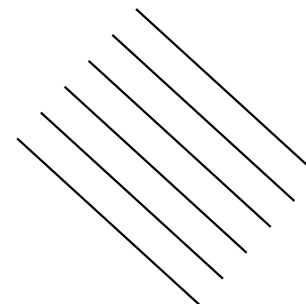
To correct for astigmatism, the focus is changed from over focus (focus above the surface) to under focus (focus below the surface). Due to the cross product nature of electron optical lenses, the beam rotates causing features to appear to be sharp in one direction and not in the other.

Over focus Condition shown.

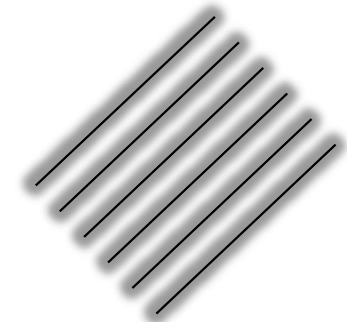


Elliptical Beam



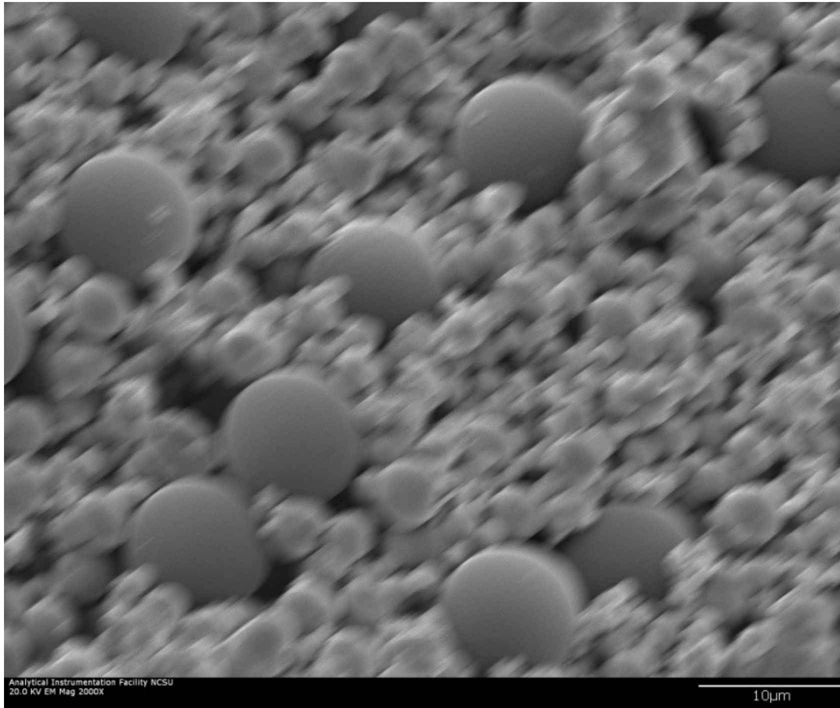


Sharp Features



Blurred Features

Astigmatism Correction

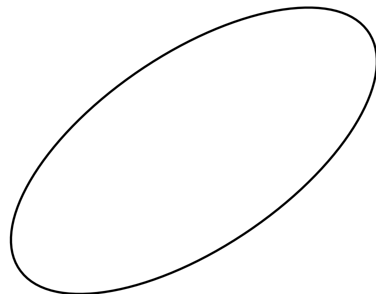


C.Mooney

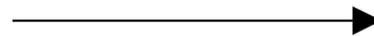
With an over or under focus condition, the features will appear to be stretched. At the just focused position, no apparent stretching will occur. At this point, the stigmator controls are adjusted to bring features into focus.

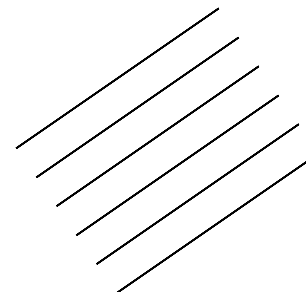
Note that this is easiest with round or irregular features!

Under focus condition shown.

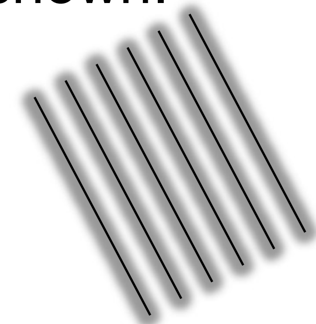


Elliptical Beam



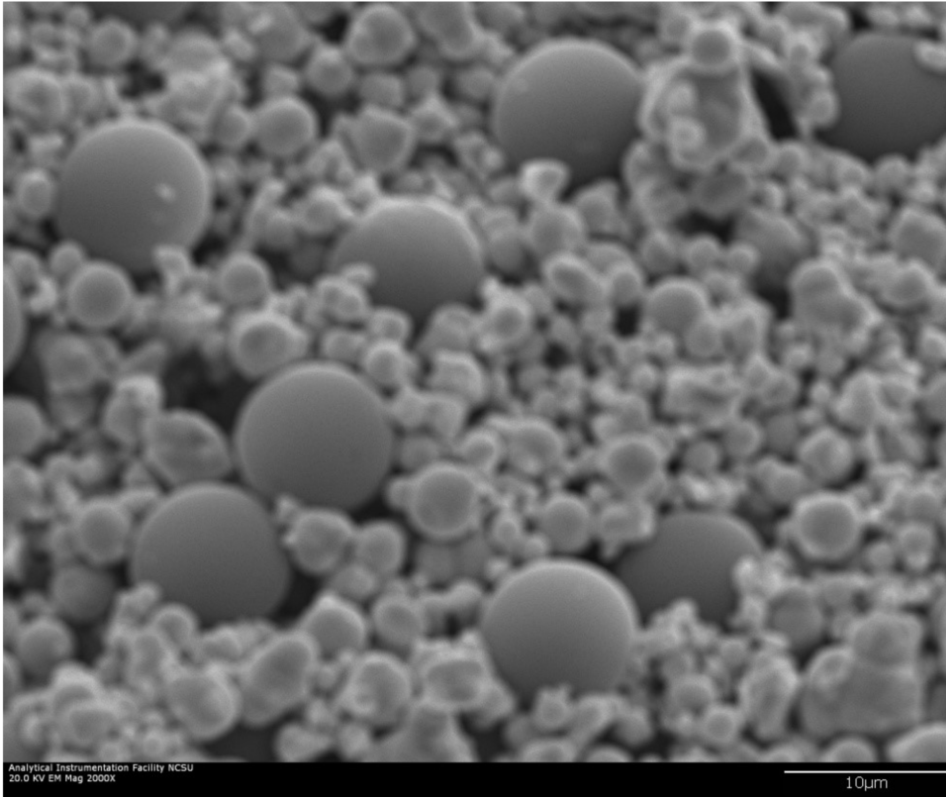


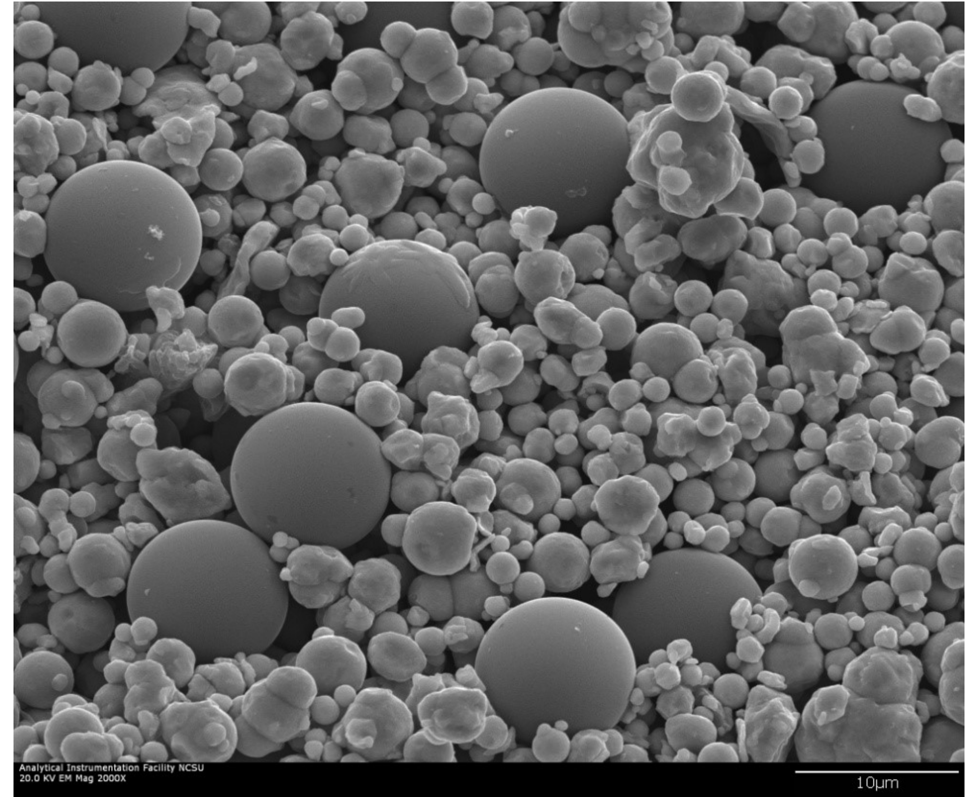
Sharp Features



Blurred Features

Astigmatism Correction





C.Mooney

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

Good focus and stigmation
Features are crisp and clear

Correct astigmatism is critical for high quality images

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

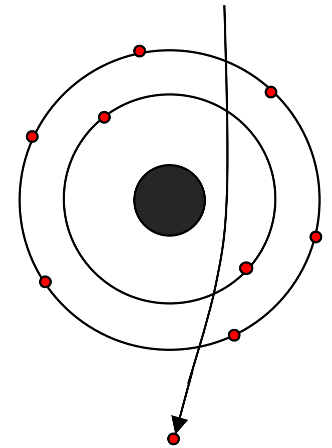
Electrons in the Sample

- 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

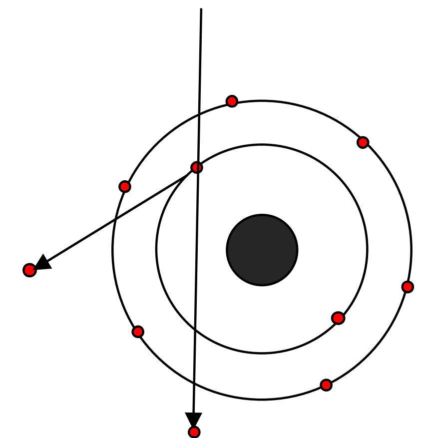
Interactions between primary electrons and the sample can be separated into two broad catagories:

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



C. Mooney

- 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



Interaction Volume

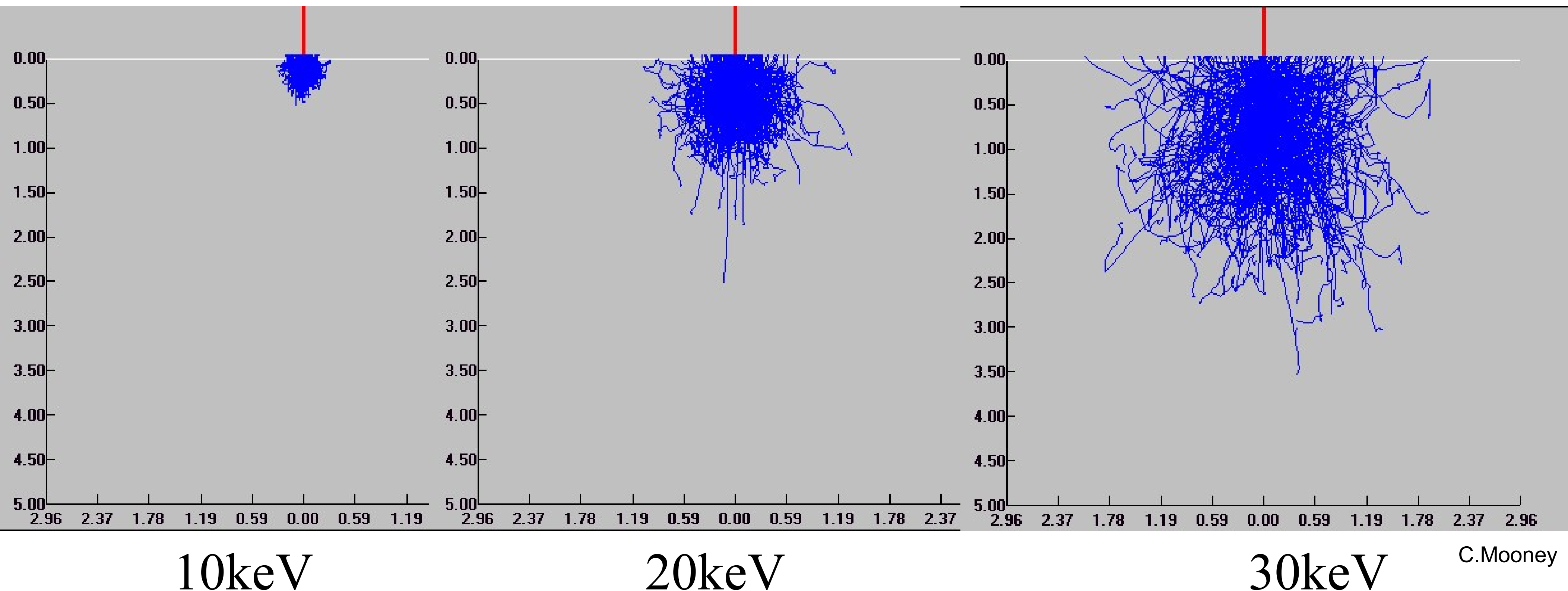
- Electrons do not stop at the surface – they can penetrate microns into a sample and generate signals throughout the interaction volume
- The dimensions of the interaction volume depend on:
  - Primary electron energy
  - Atomic weight
  - Density
- Interaction volume is generally determined via Monte 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 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 does the electron travel before there is an 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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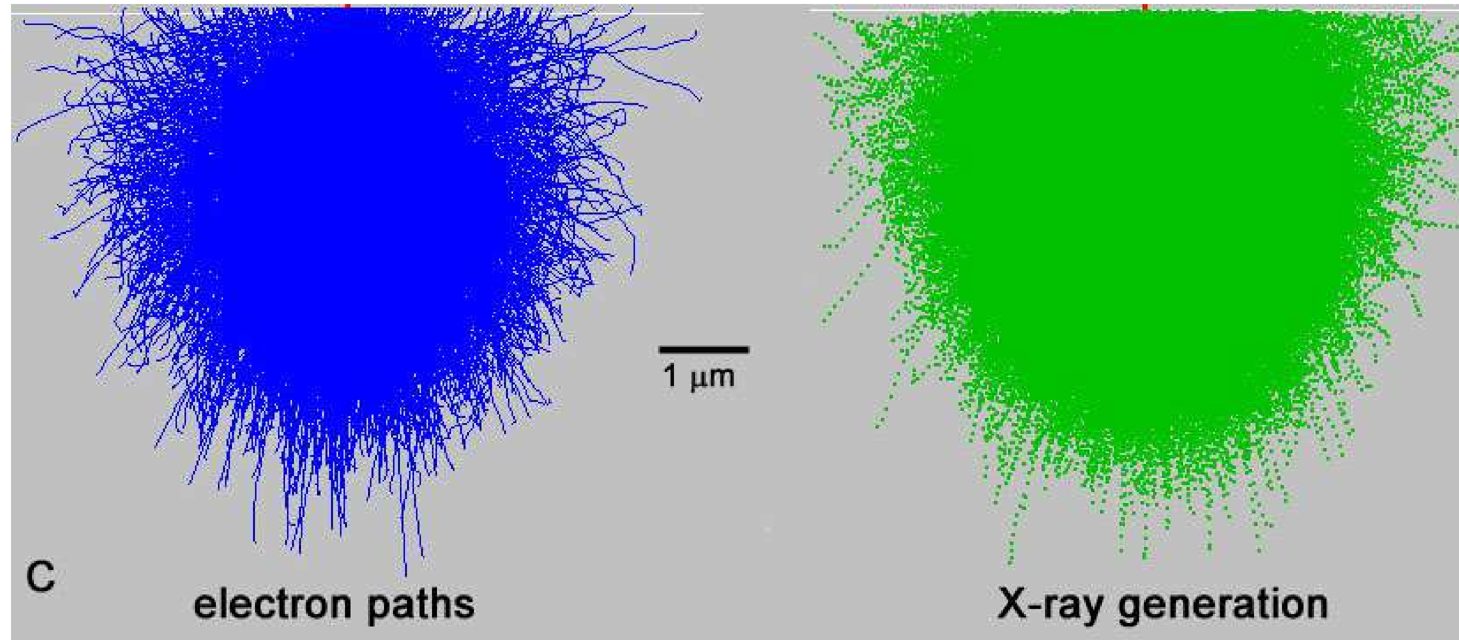
- Monte Carlo simulations to determine interaction volume as a function of beam energy for an Fe sample at different beam energies
  - Monte Carlo simulations take into account all of the possible interactions to give a visual representation of the interaction volume
- As beam energy increases, so does the interaction volume, typically by the energy to the ~1.7 power

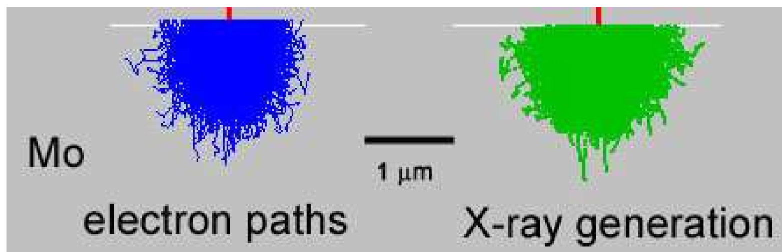
Interaction Volume as function of Material

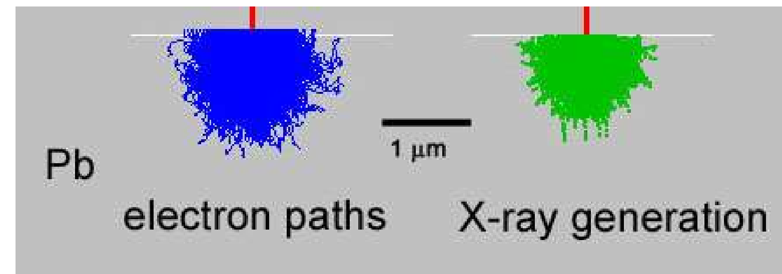
Beam energy 20keV for all simulations

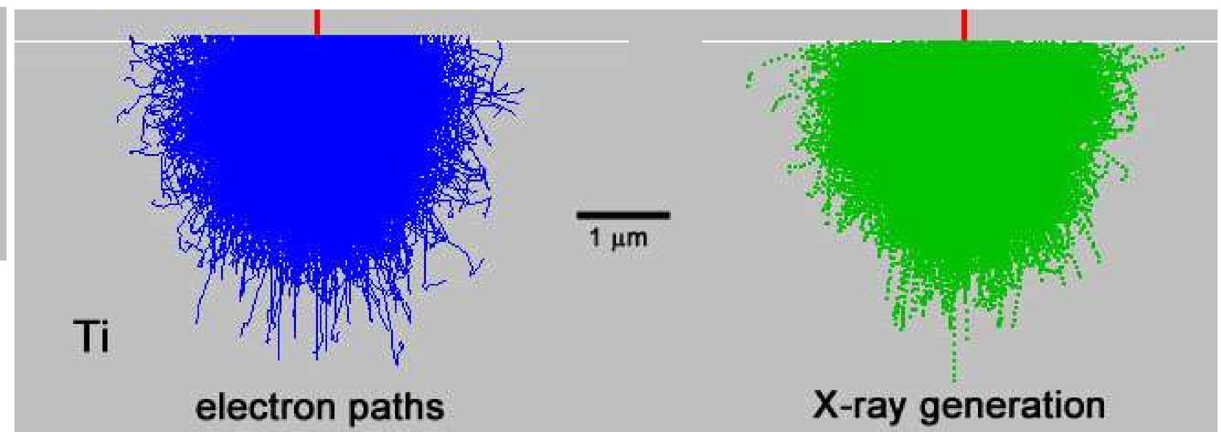
The scale marker bar is constant for all simulations

In general, as Z increases, interaction volume decreases









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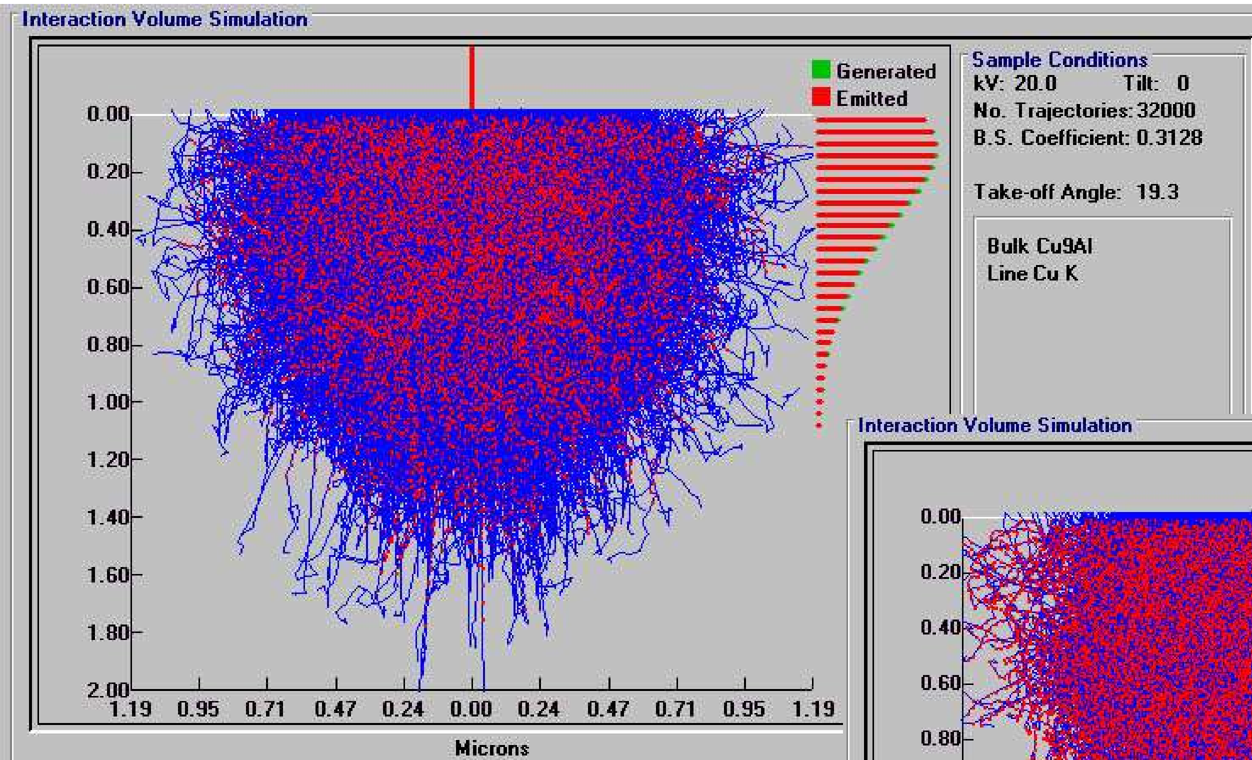
Generated and Emitted X-ray Volume



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

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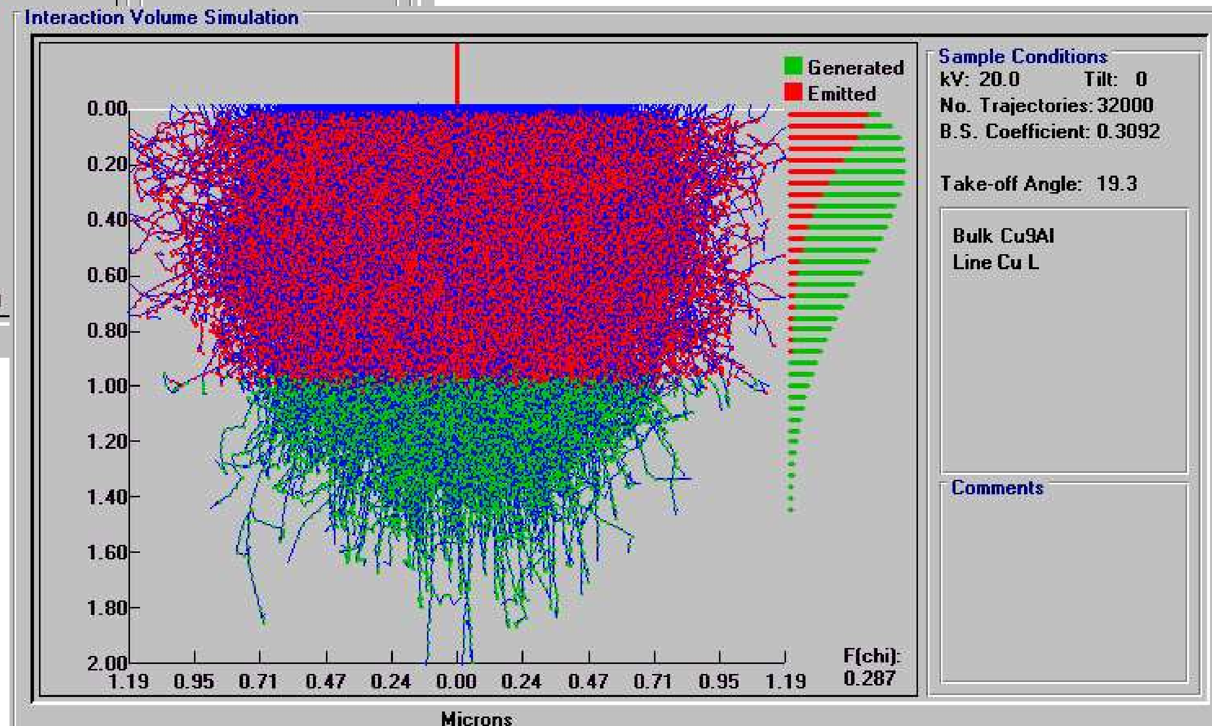
Cu Kα (8.04keV)



More energetic particles will escape from greater depths in the sample

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Cu Lα (0.928keV)

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 takes 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 the sample's reaction

- 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

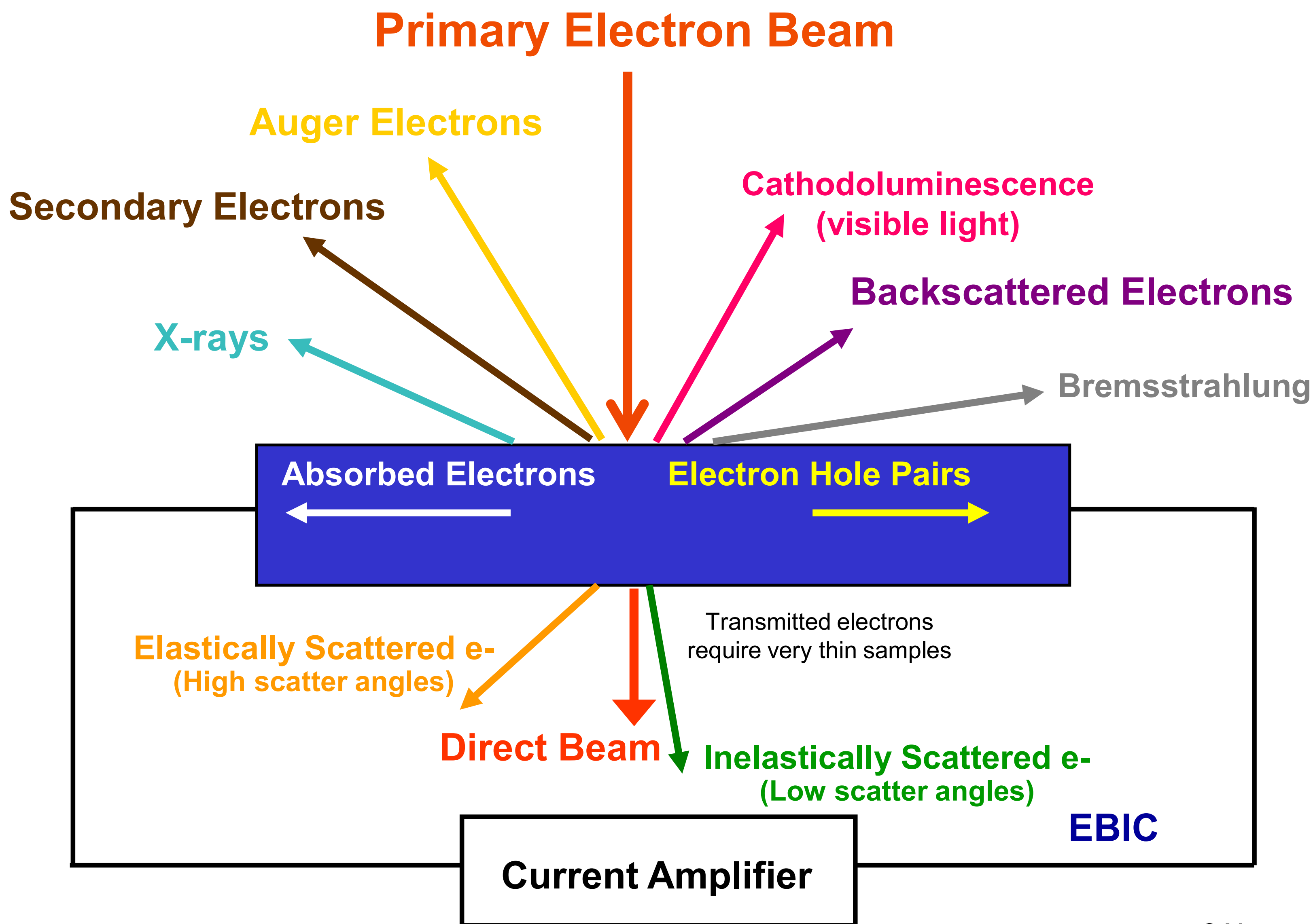
- Signals in the SEM are particles that are ejected from the sample that can be detected!
  - Electrons and photons are typical signal particles
  - Not all signal particles are detectable – have to impinge the detector

- Ejected particles that we detect usually need to move in the direction of the detector!
  - Exceptions: Secondary electrons, specimen current

- Wide area detector = large solid angle of collection → more signal!

- 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,

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

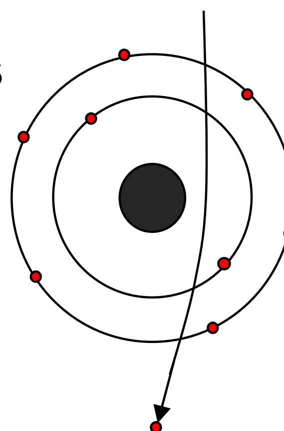
Where n_{ep} = number of emitted particles, n_b = number of primary particles
In an SEM n_b = number of primary electrons.

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 detector measures SE emission, 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, X-rays, etc., 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 ~ 2-3 degrees



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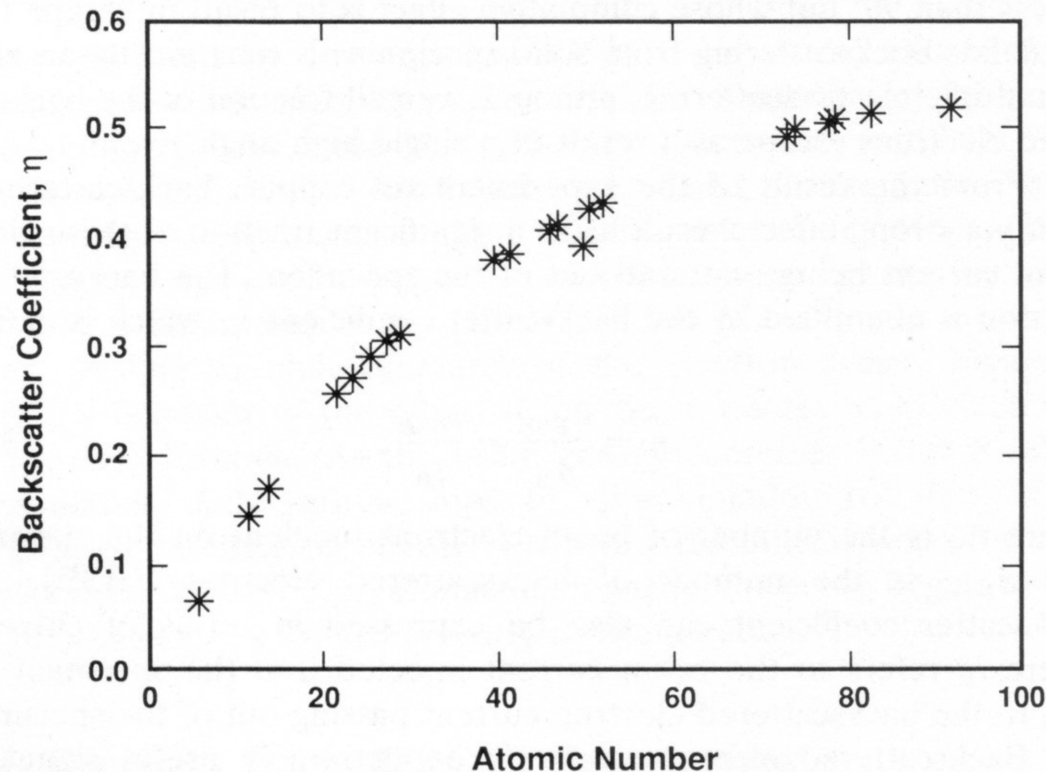
- The backscattered electron coefficient is denoted as η

- Energy is in the same range as the energy of the electron beam
  - Most BSEs have ~ 80 % of the energy of the the beam

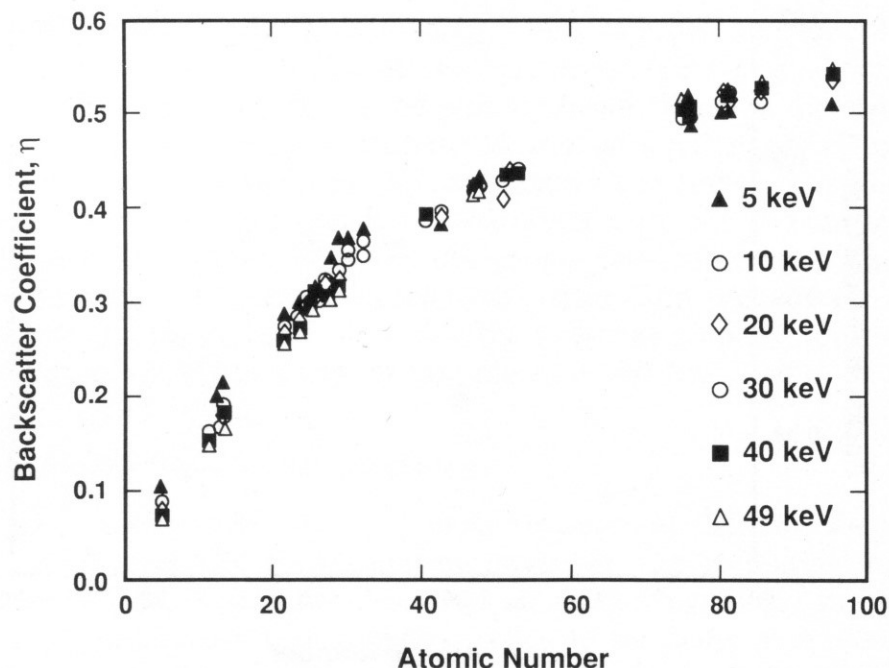
η Dependence on Atomic Number



Goldstein, et. al.

- 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



Goldstein, et. al.

- 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
- The trend of η increasing with energy holds even though the details differ

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

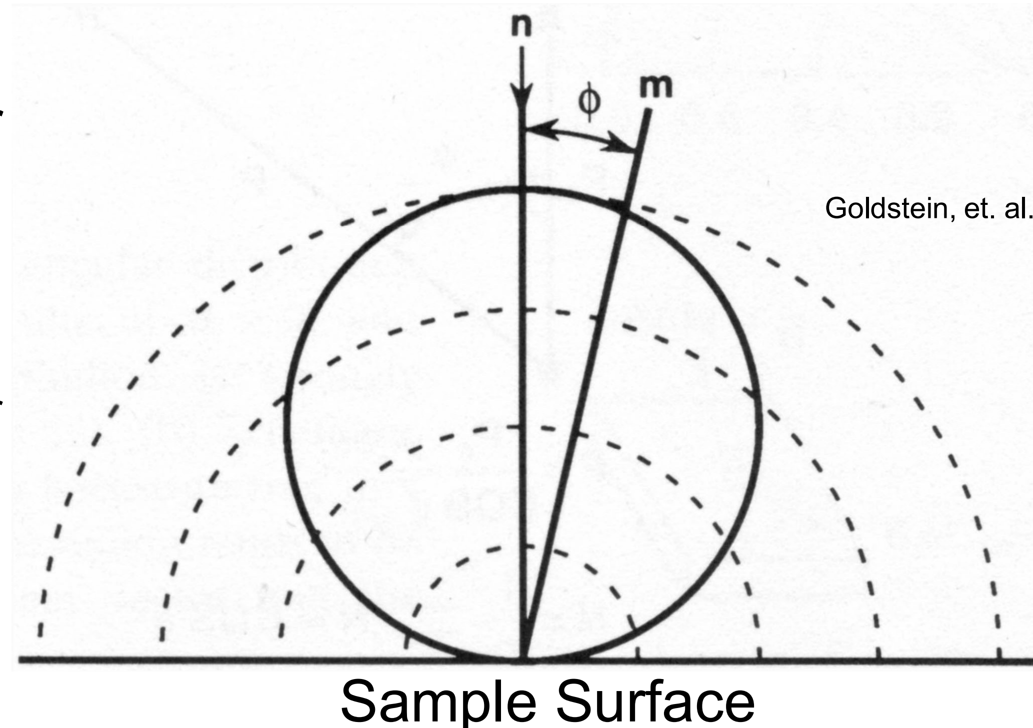
Angular Distribution of η

- 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

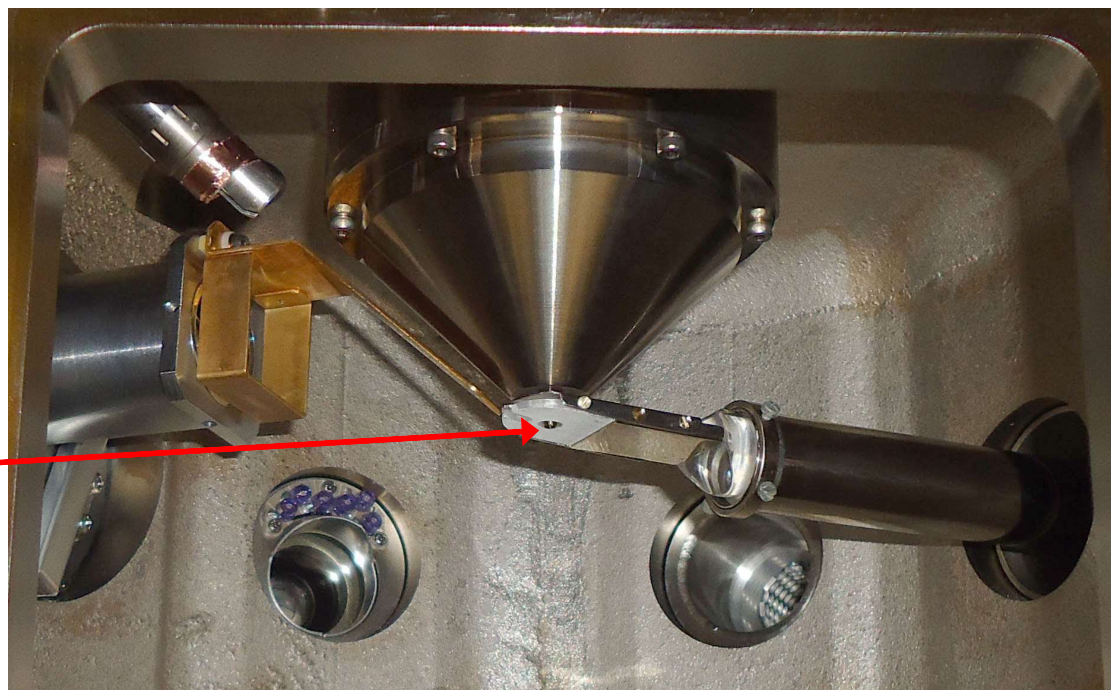


Sample Surface

Electron detection – BSEs

Robinson passive scintillator backscatter electron detector

Shown in the inserted position



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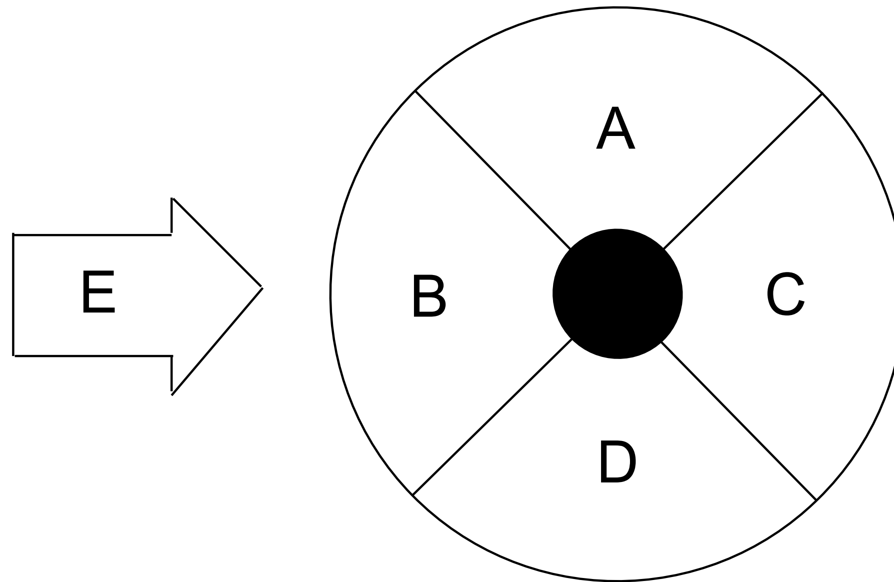
- Since most BSEs go back in the direction of the beam, the BSED is generally placed under the pole piece
- Most BSE detectors are insertable – retract when not in use
- The two most common BSEDs are solid state and passive scintillator
- Passive scintillators work like an ETD with no collector grid or applied bias, the entire detector area is a scintillator

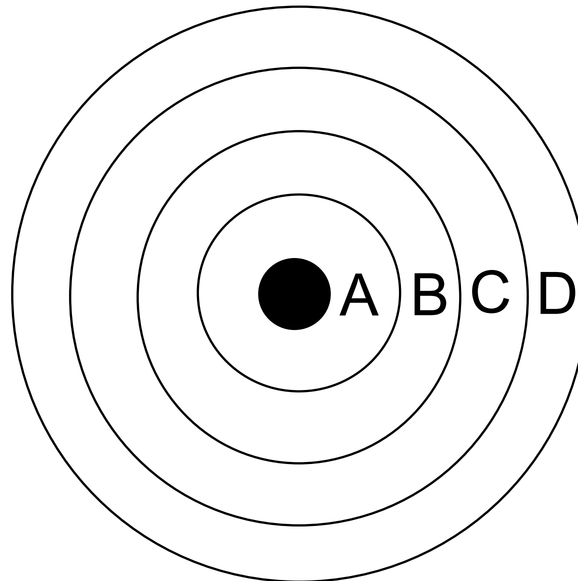
SU3900 BSE Detector Geometry



SU3900 BSED geometry: Five segments with a four quadrant design plus an offset element

- For Compositional contrast, sum signals from the quadrants
  - A + B + C + D
- Topography: Ignore C and add E
  - There are some other ways to enhance topography, which we will go over in the lab

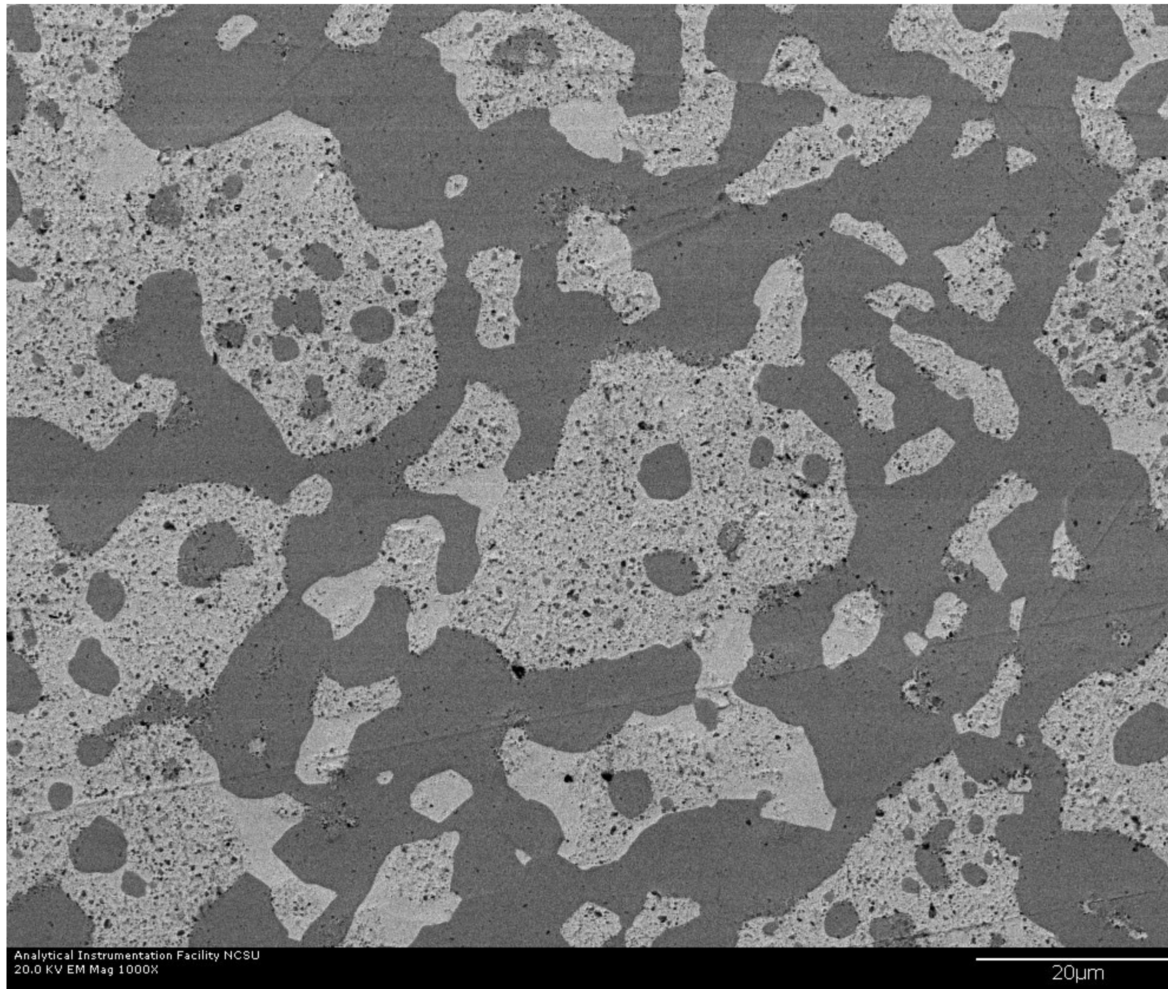
Verios BSE Detector Geometry



Verios Solid State BSED geometry: Four concentric rings

- The concentric ring design allows one to observe BSEs with different take-off angles, which can be beneficial for stepped samples
- Also, if the sample is insulating and charging, adjusting which rings are being displayed can help with charging effects

SEM Compositional image



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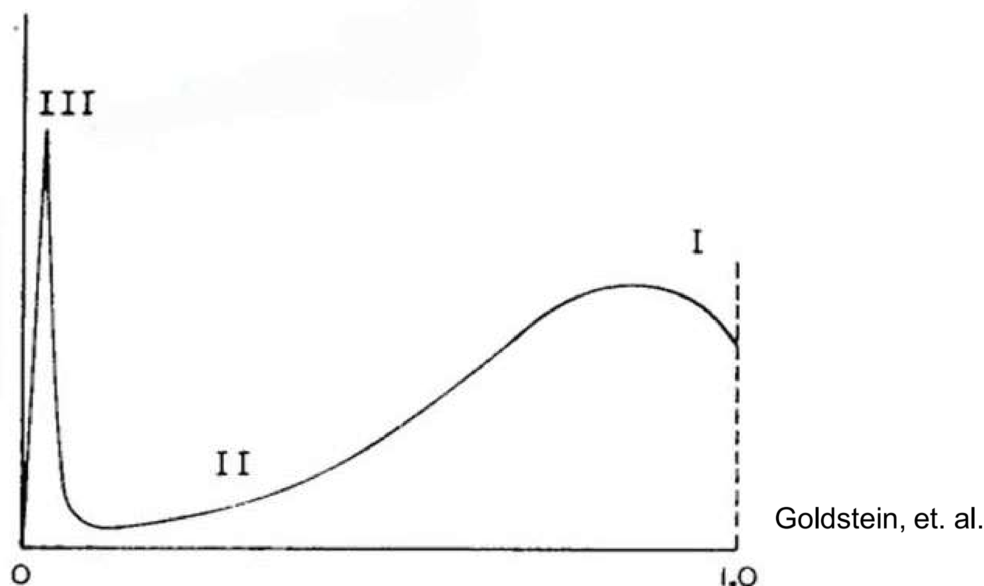
Backscattered SEM image of an PbSn alloy showing contrast based on the atomic number. The brighter areas are Pb-rich. Dark spots are embedded polishing media (SiO_2).

Electron Emission from a Sample

Plot of total electron emission vs. normalized energy

Energy = 1 is the energy of the incident electron beam

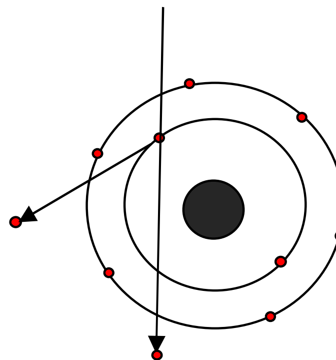


Goldstein, et. al.

- 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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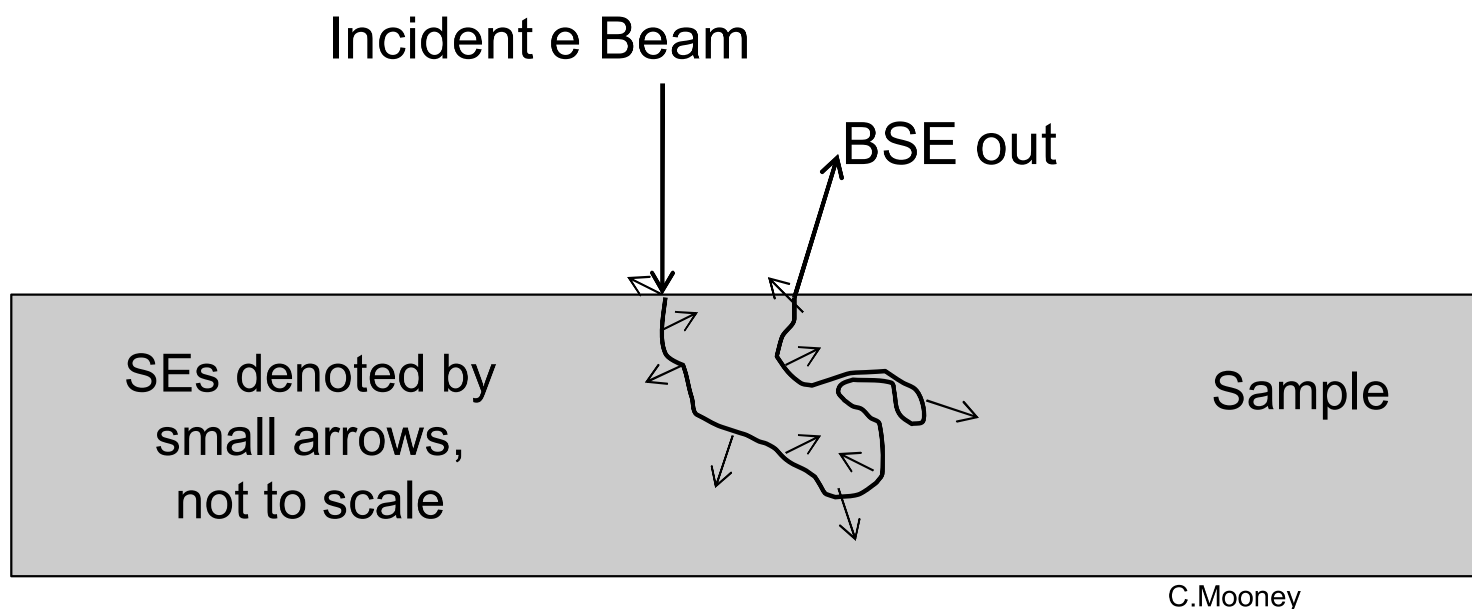
- Since this is not the primary electron from the beam it is called a **Secondary Electron**

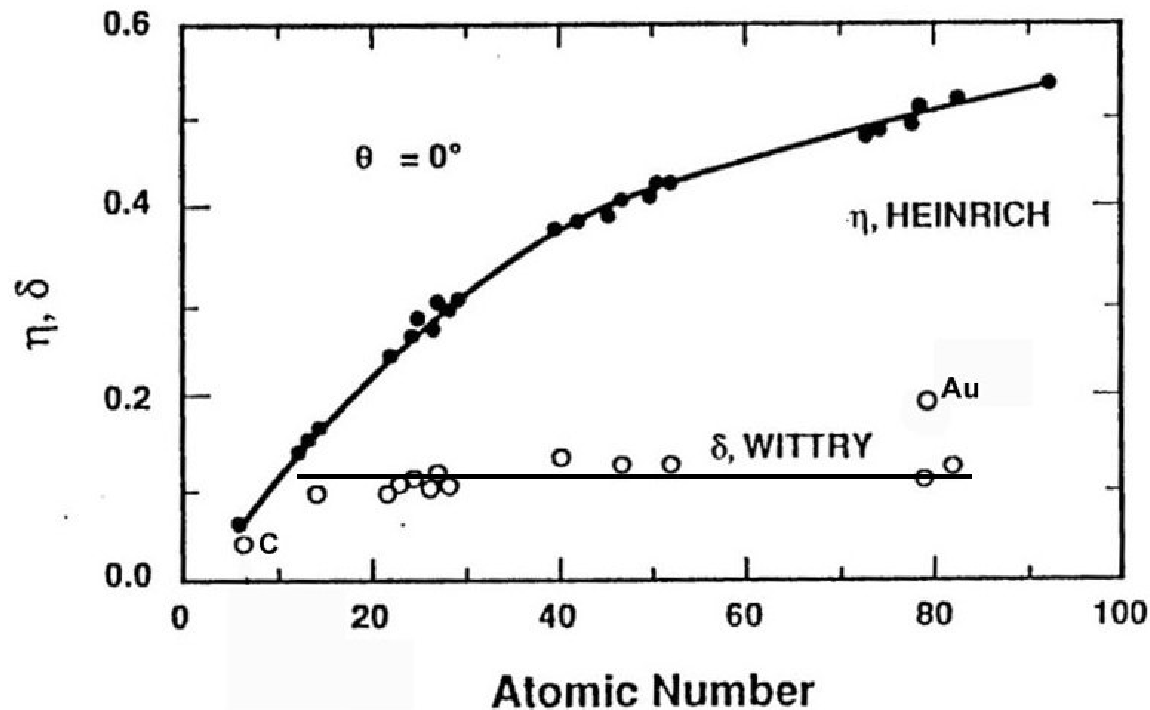
- SEs are ***defined*** as electrons that escape the sample with < 50 eV of energy
  - Energy peak in the 3-5 eV range
  - This is a definition of convenience
  - Some BSEs have <50eV
  - There are also high energy SEs (it takes a spin resolved detector to tell the difference)

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



SE emission is Constant with Z



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

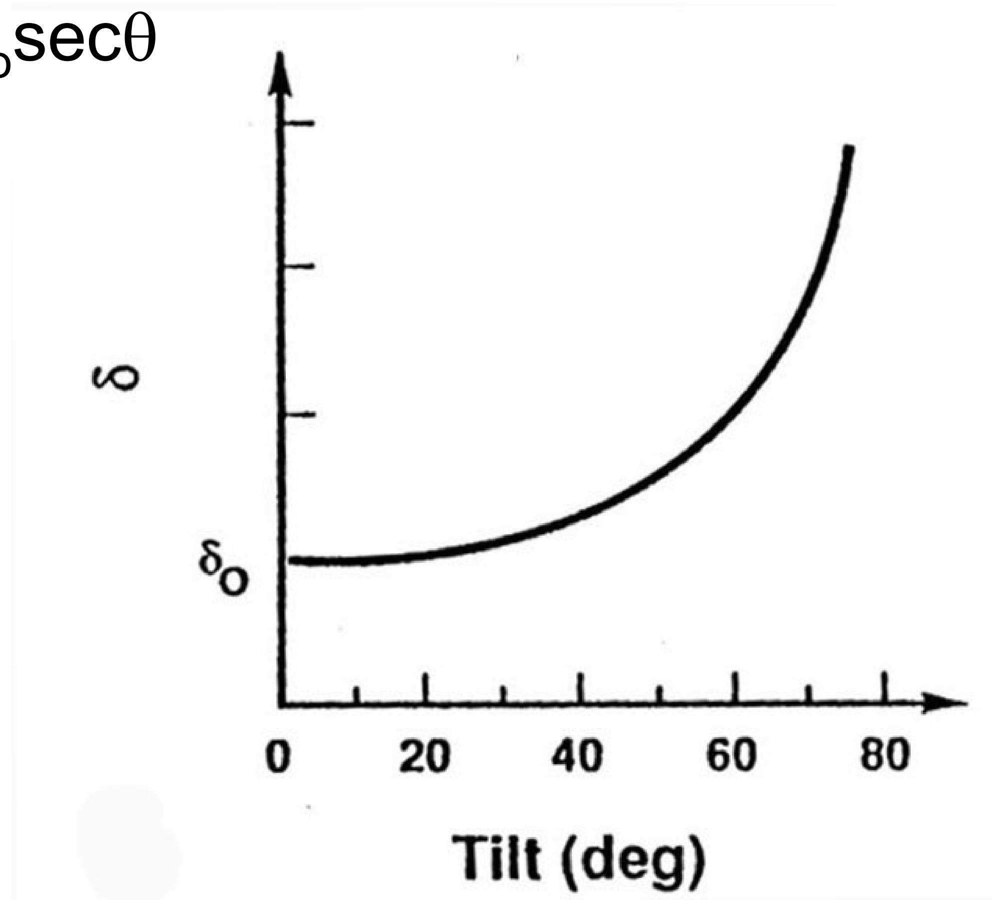
Goldstein, et. al.

- 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:

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

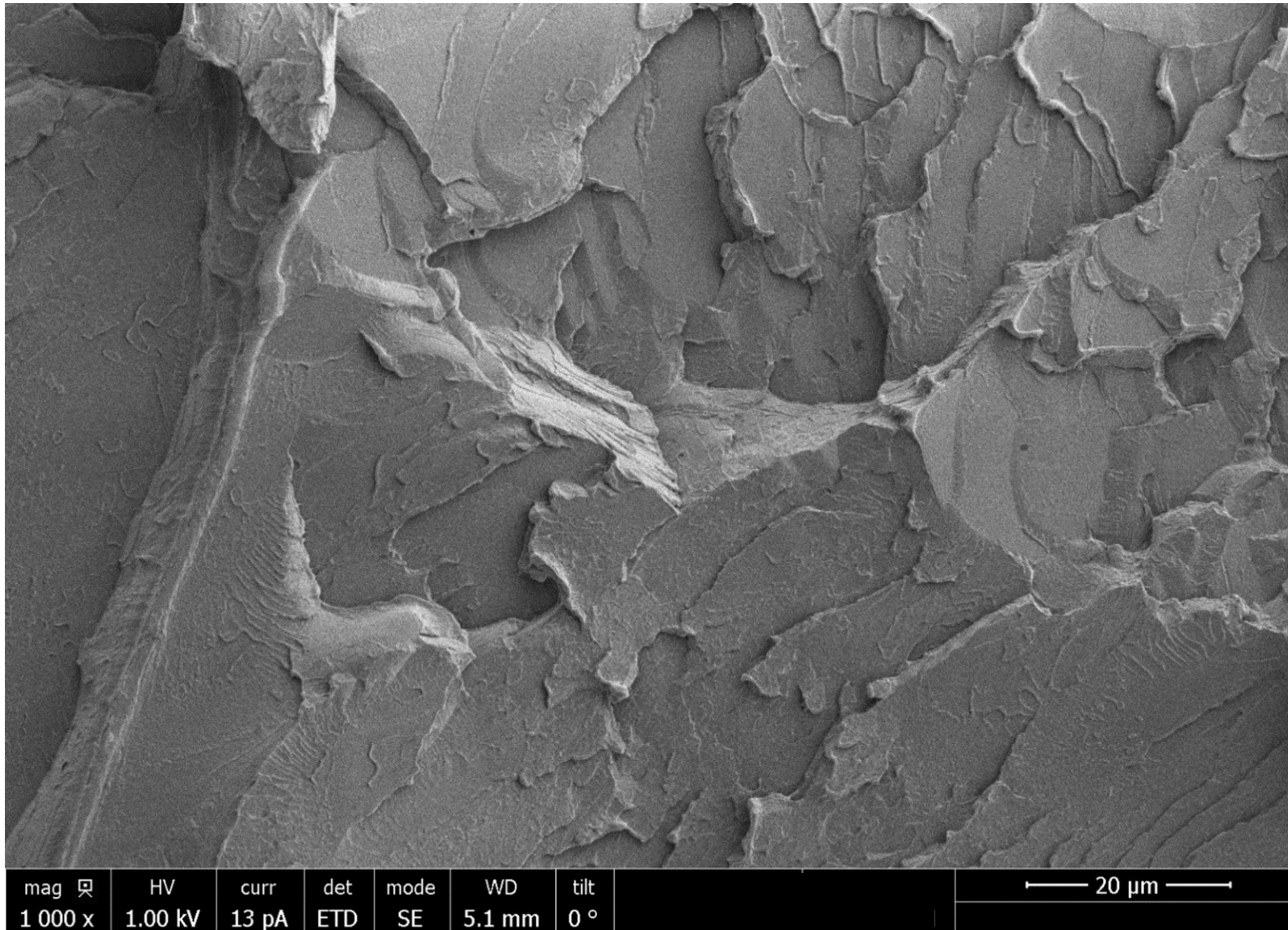


Goldstein, et. al.
from Kanter (1961)

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



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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

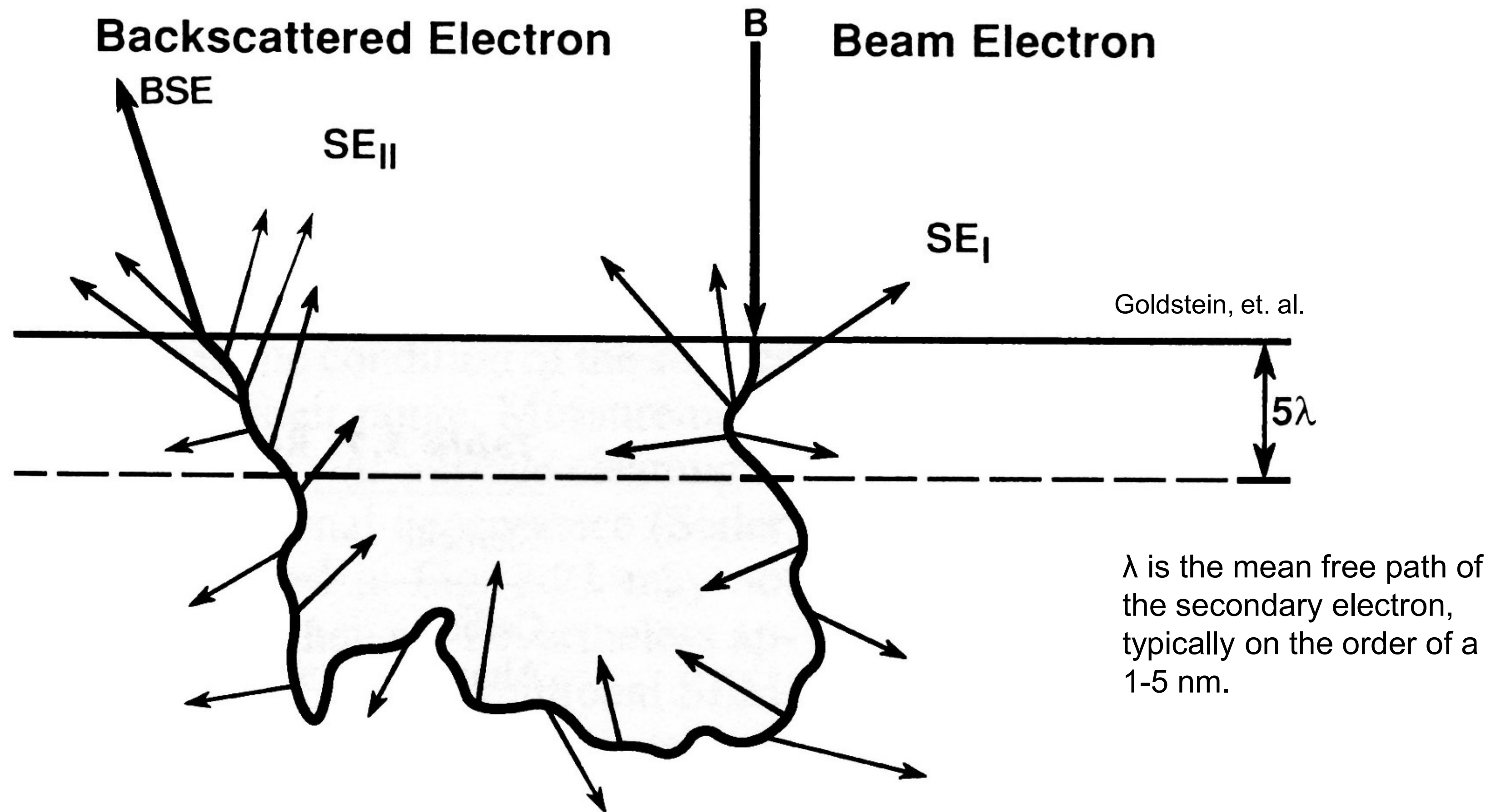
- Secondary electrons have a very shallow sampling depth
  - Maximum escape depth is on the order of 20nm
- The shallow sampling depth is a function of
  - SE's low kinetic energy
  - SEs lose energy quickly in the sample due to inelastic scattering
    - Inelastic scattering has a high cross section 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 the 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!

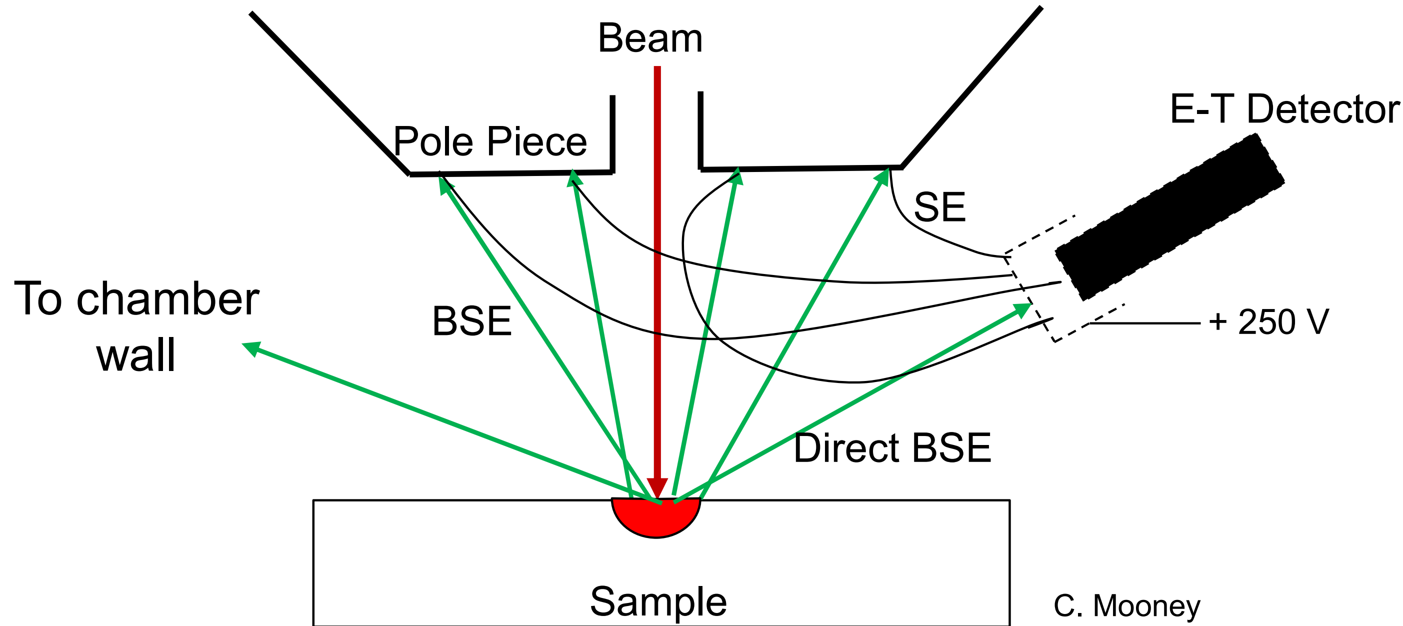
Origins of type I and II SEs



λ is the mean free path of the secondary electron, typically on the order of a 1-5 nm.

Schematic showing origin of type I and II secondary electrons

Source of type III SE

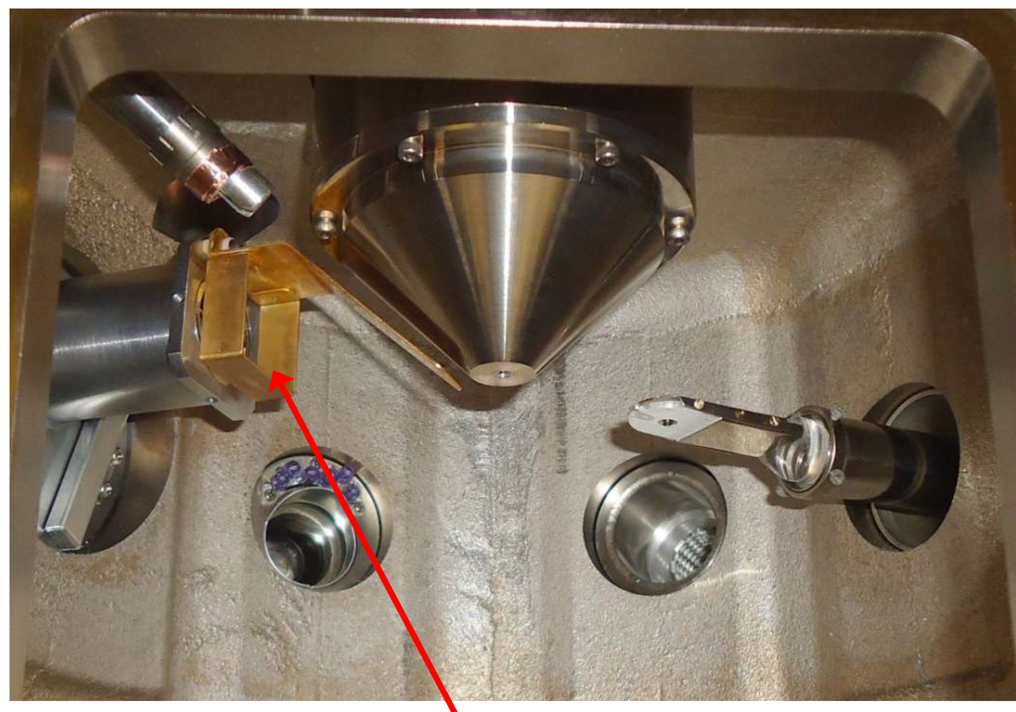


- Type III SEs come from BSEs that are emitted from the sample and strike parts inside the instrument producing SEs that are then detected
- Type III SEs can carry information from the sample since the BSEs that produced them carried information from the sample
- In most SEMs, the pole piece is polished 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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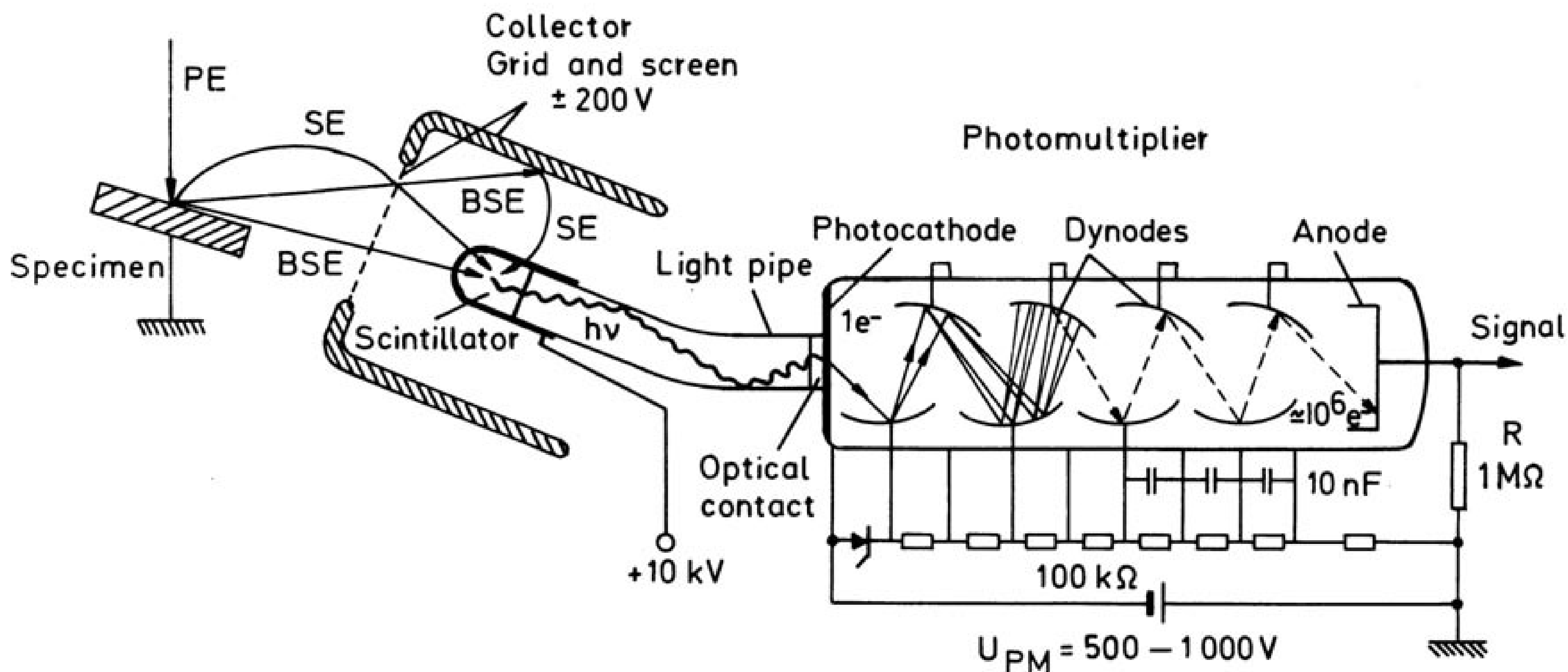
ET Secondary Electron Detector

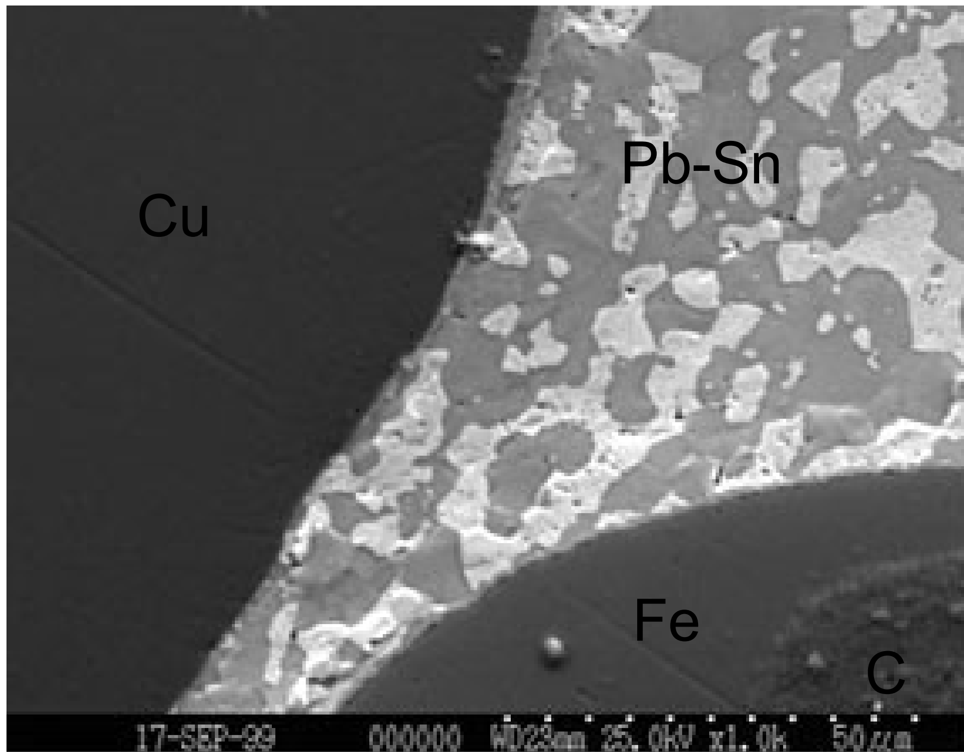
Everhart-Thornley Detector



Goldstein, et. al.

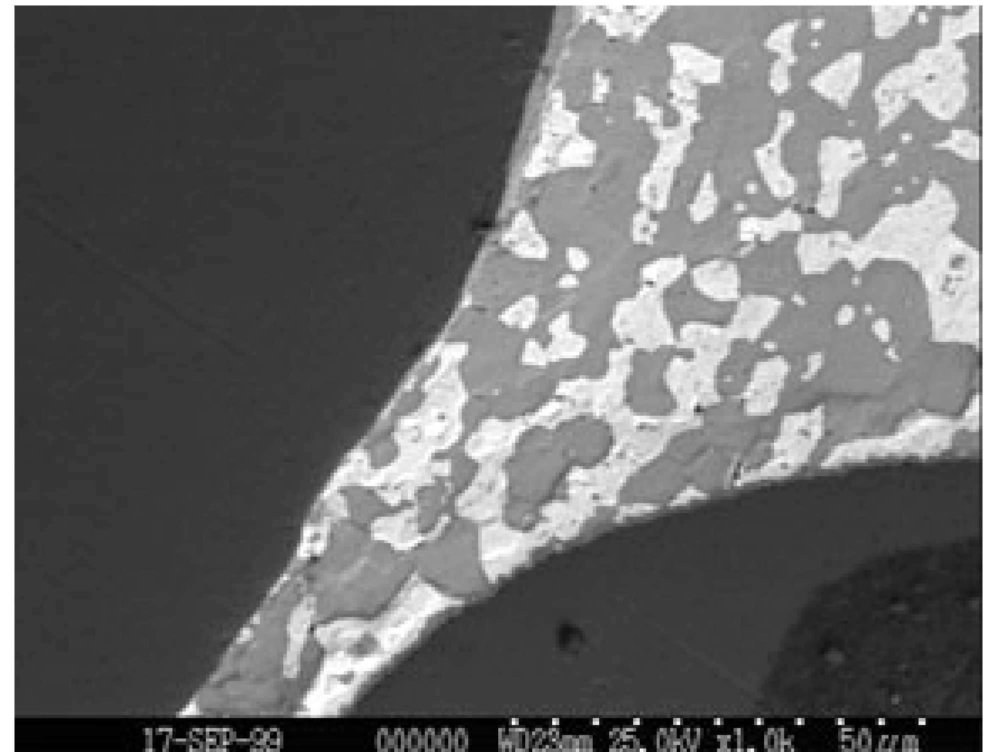
- Collector screen is typically biased to +200V
  - Attracts low energy electrons
- Scintillator is typically biased to +10kV
  - Accelerates the low energy electrons to enough energy to fire a scintillator photon
- Photon enters photomultiplier tube where it is amplified by a factor of ~10^6

SE and BSE images



SE image of PbSn solder

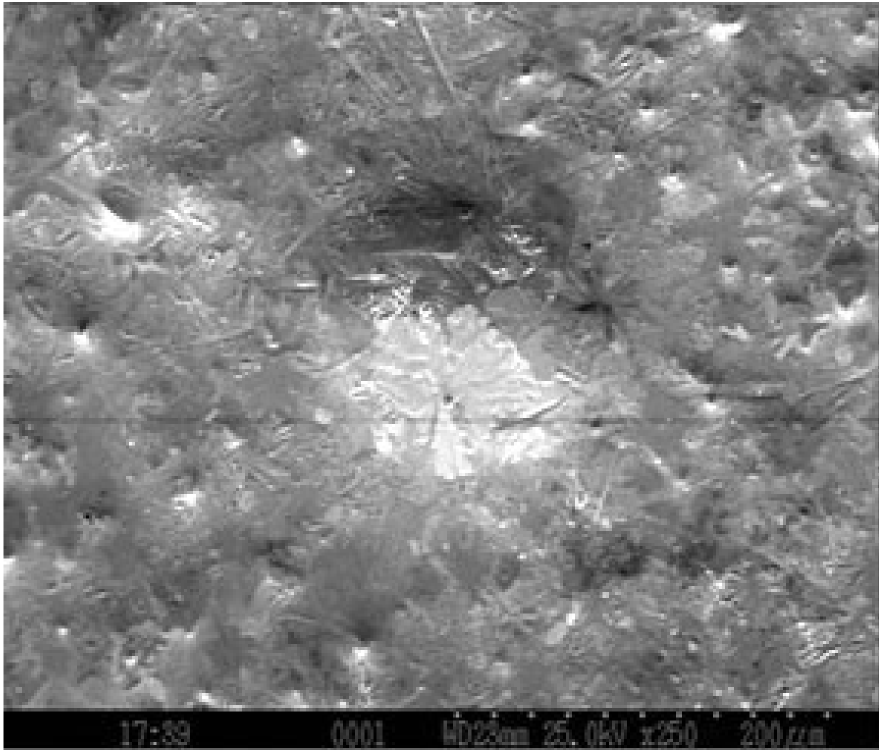
D.Batchelor



BSE image of PbSn solder

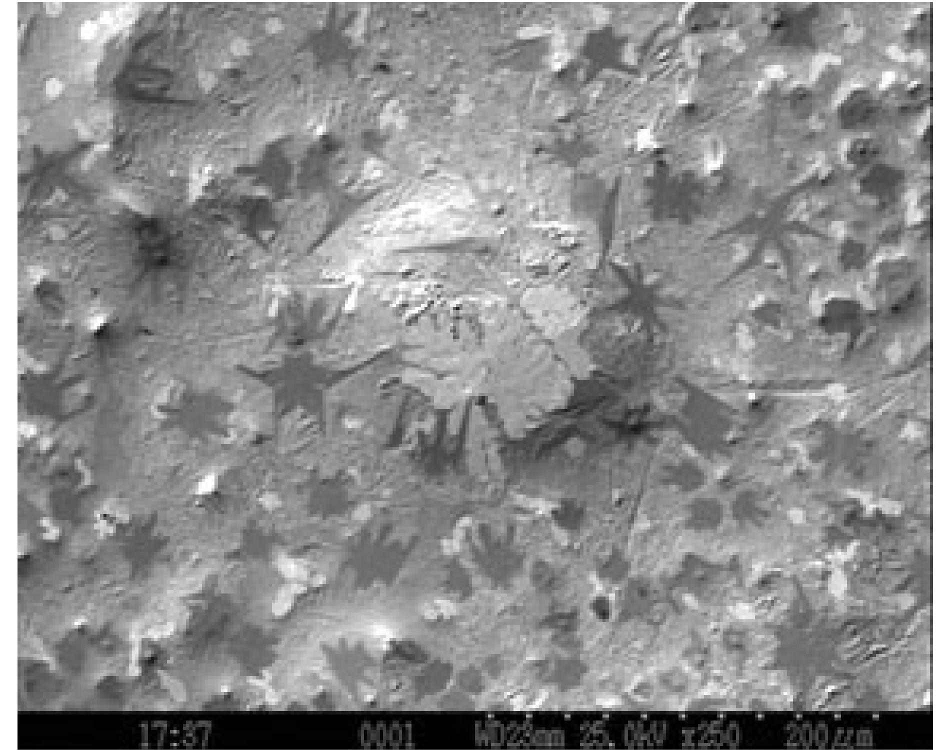
Notice that in the SE image that there is clear topographic contrast while the BSE image appears more flat. The SE image will have some compositional contrast because δ has some variation. The BSE image will have a topographic component due to sample-detector geometry.

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 topograhic 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

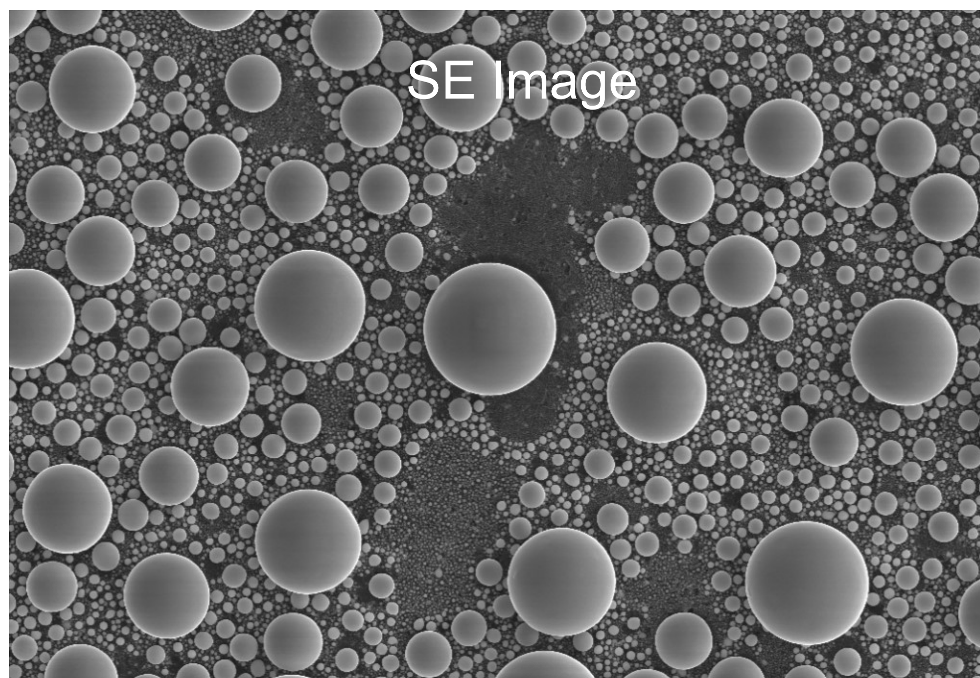
- Considerable topography can be observed in BSE images
  - Similar to a photograph taken with long shadows
  - Bright features point toward the BSE detector
  - Some solid state detectors are divided into sections which can be added or subtracted from one another to enhance topographical contrast

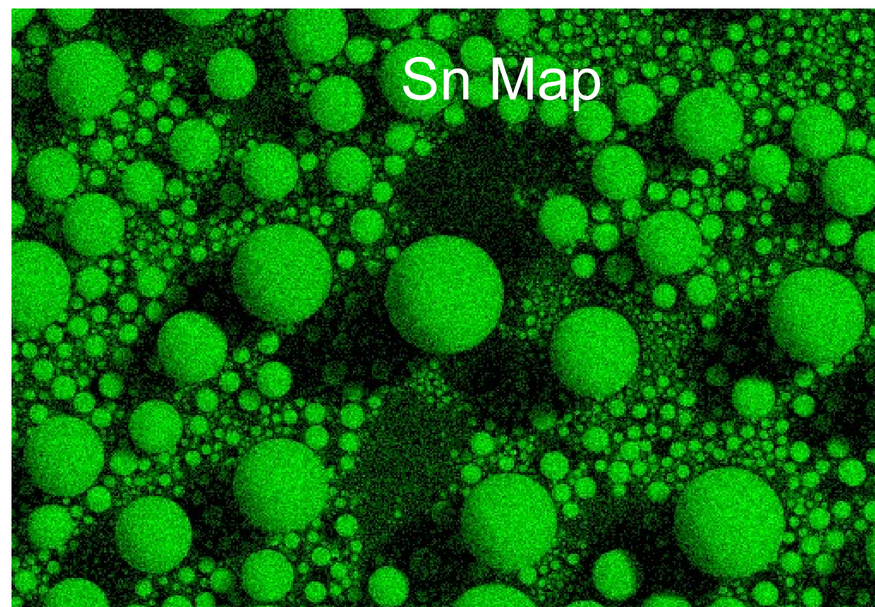
X-rays

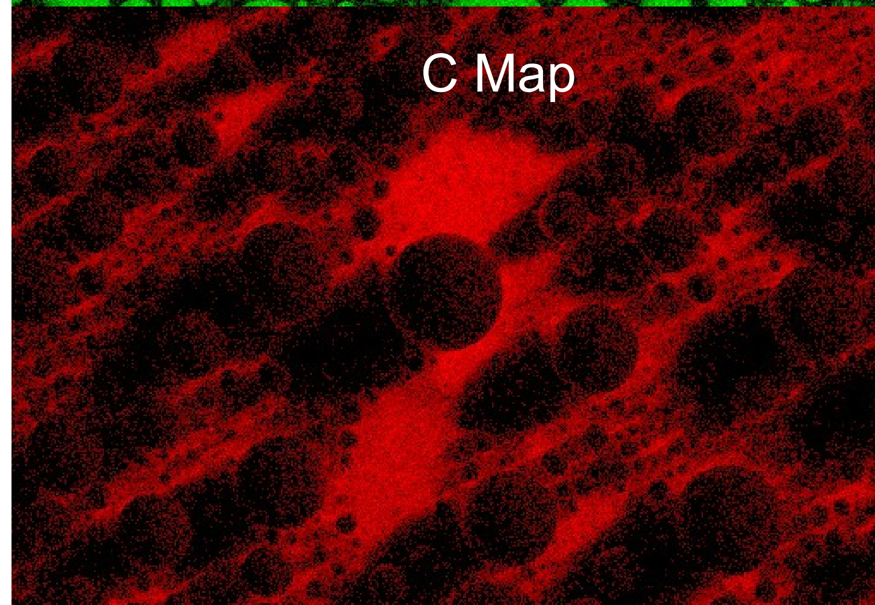
- X-rays are generated in the sample by high energy electrons interacting with the sample
- There are two types of X-rays produced
  - Bremstrahlung and Characteristic
- Characteristic X-rays provide information about the elements in the sample
- X-rays tell us what elements are in the sample and compliment BSE composition images
- X-ray photons don't behave the same as electrons, so most the discussion of X-ray signals will be at the end

X-ray Map

Sn and C X-ray maps of a Sn on C resolution standard.







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Applied Sample Bias

- Some SEMs (e.g., Verios 460L) allow a negative voltage to be applied to the sample

- This decelerates electrons before they strike the sample
  - Can help with charging
  - Allows for high energy in the column and low energy at the sample

- 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!

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, the following controls are available to allow the operator to control the beam

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

- The accelerating voltage is the bias applied between the electron source and the anode plate that accelerates the electrons down the column

- 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

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

Beam Energy Effect: High Resolution SEM

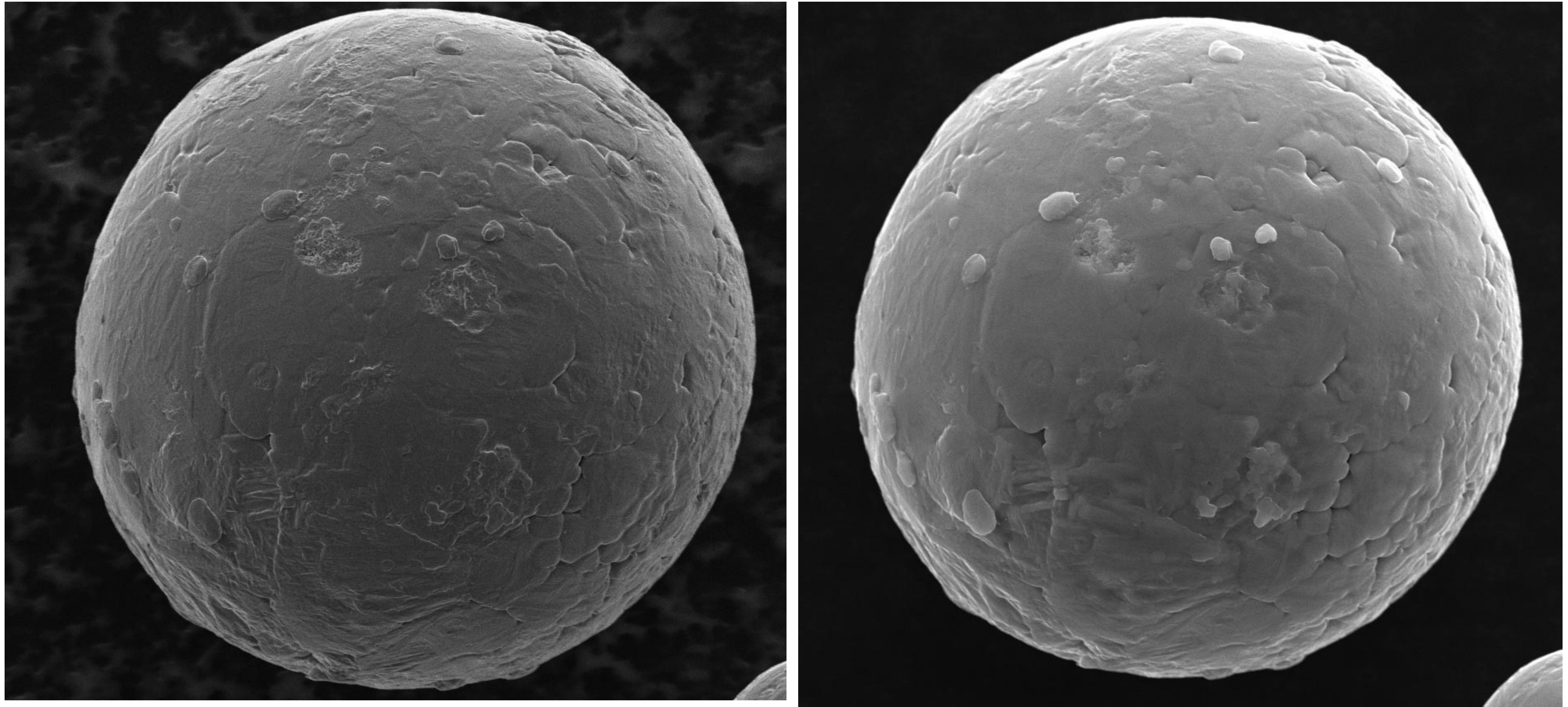
Theory:

- 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, high 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: Conventional SEM



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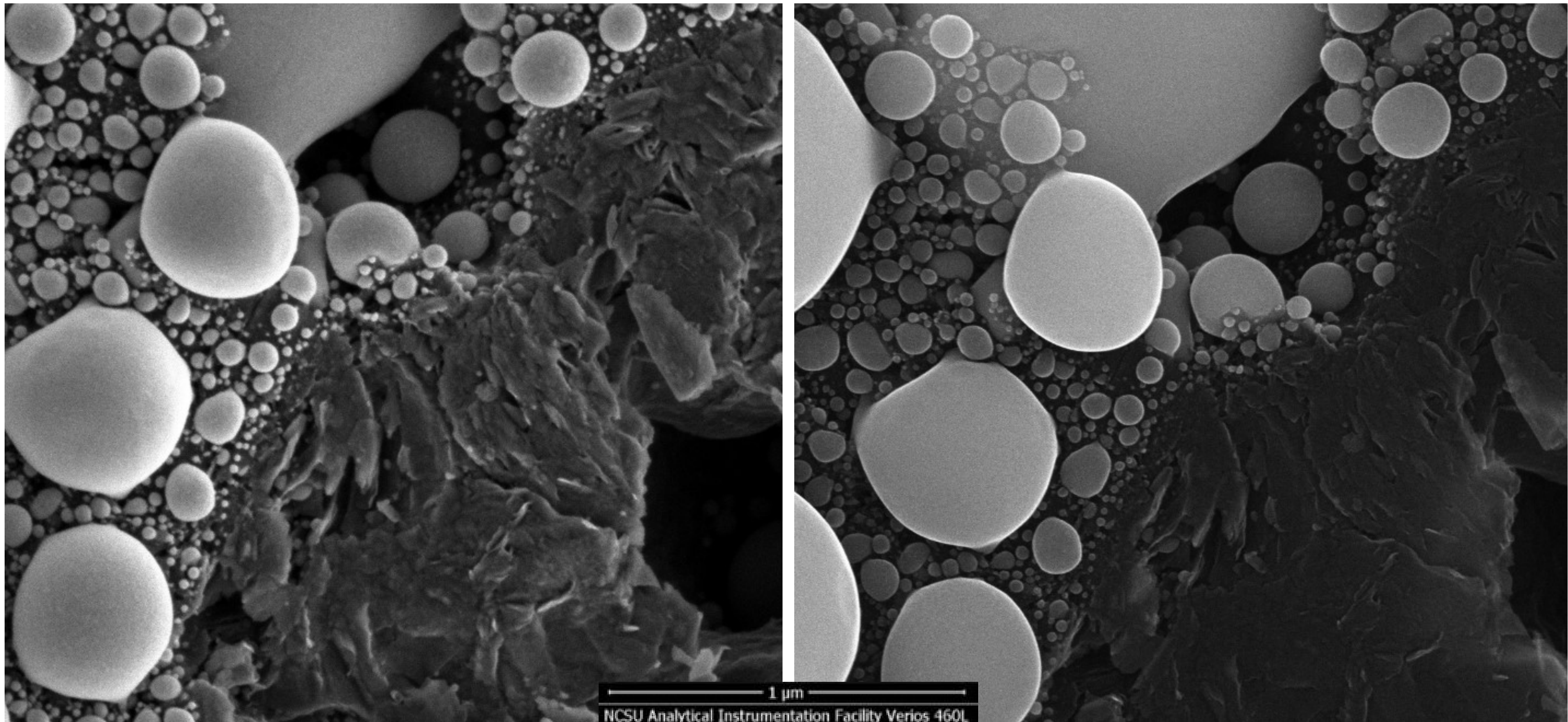
- Ti-6Al-4V alloy sphere used in industrial 3D printing of metal parts
- 5kV image (Champion Data) on left, 30kV image (nice) on right
- Note the increase in surface detail in the 5kV image
- Note edge effects in the 30kV image

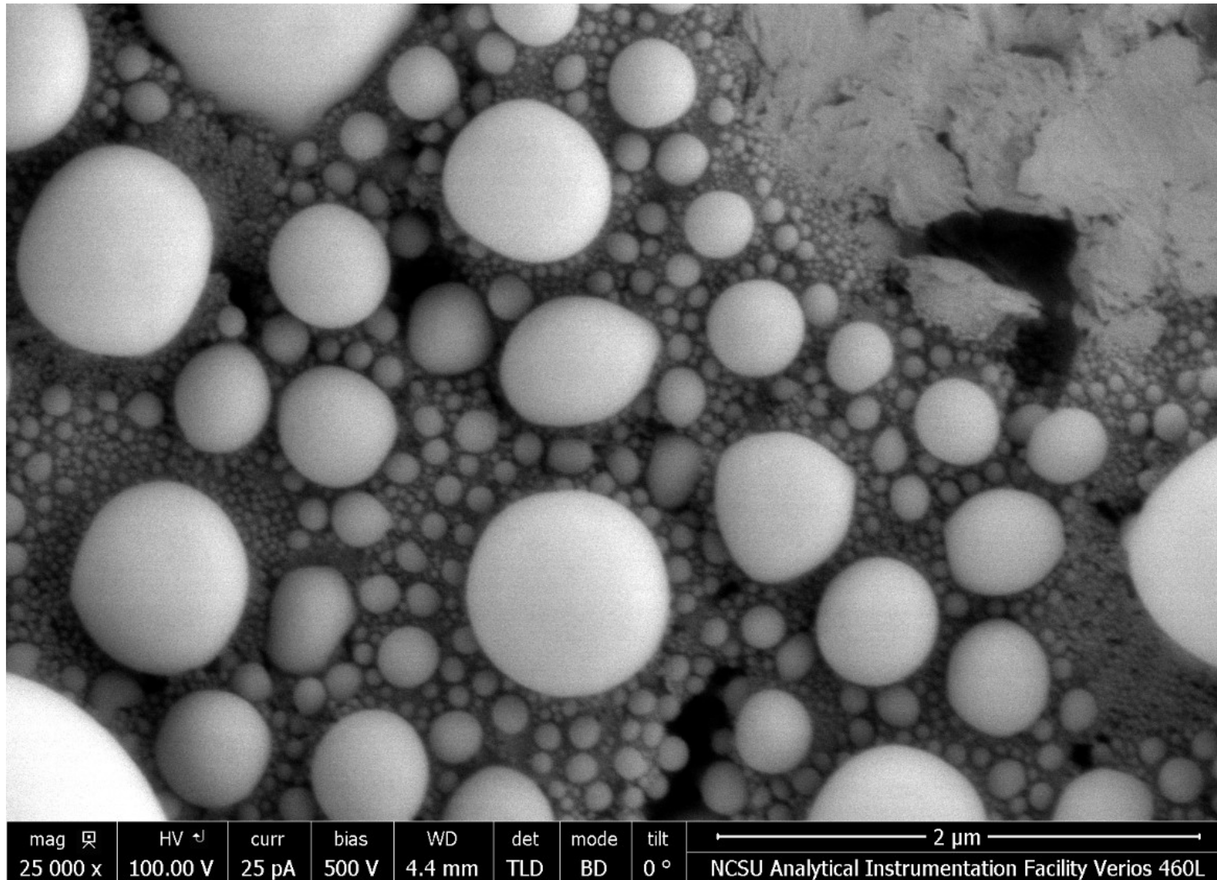
Beam Energy Effect: UHR-SEM



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



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100 eV!

Champion Data

Sample:
Sn spheres
on C

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

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-ish eV (usually with an applied stage bias) is for insulators without a coating

- Experiment! Try different things to see what works the best!

Condenser Lens

- The condenser lens primarily controls the current in the beam (at the sample)
- 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

Practical SEM Rules: Beam Current

- The control for the condenser lens is usually labeled Beam Current or simply Current
  - Often the beam current control is a dimensionless number that is related to the excitation of the condenser lens
  - Some instruments display a current value in amps

- To make the resolution better, choose a smaller beam current
  - **SEM rule: smaller current => smaller spot size**

- SED usually works well with low currents
- BSED often requires more current than the SED
- X-ray analysis (EDS) generally works better with a relatively high current (generates more X-rays)
  - 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 Rules: Beam Current

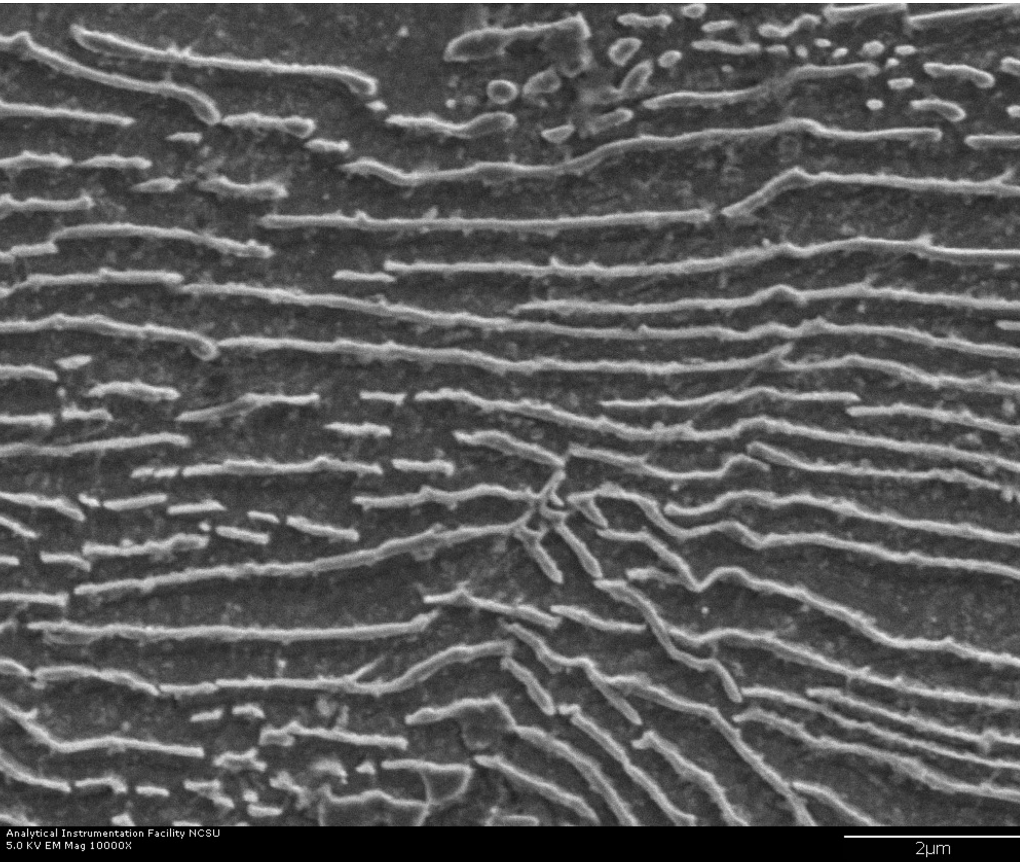
UHR-SEM beam current trends:

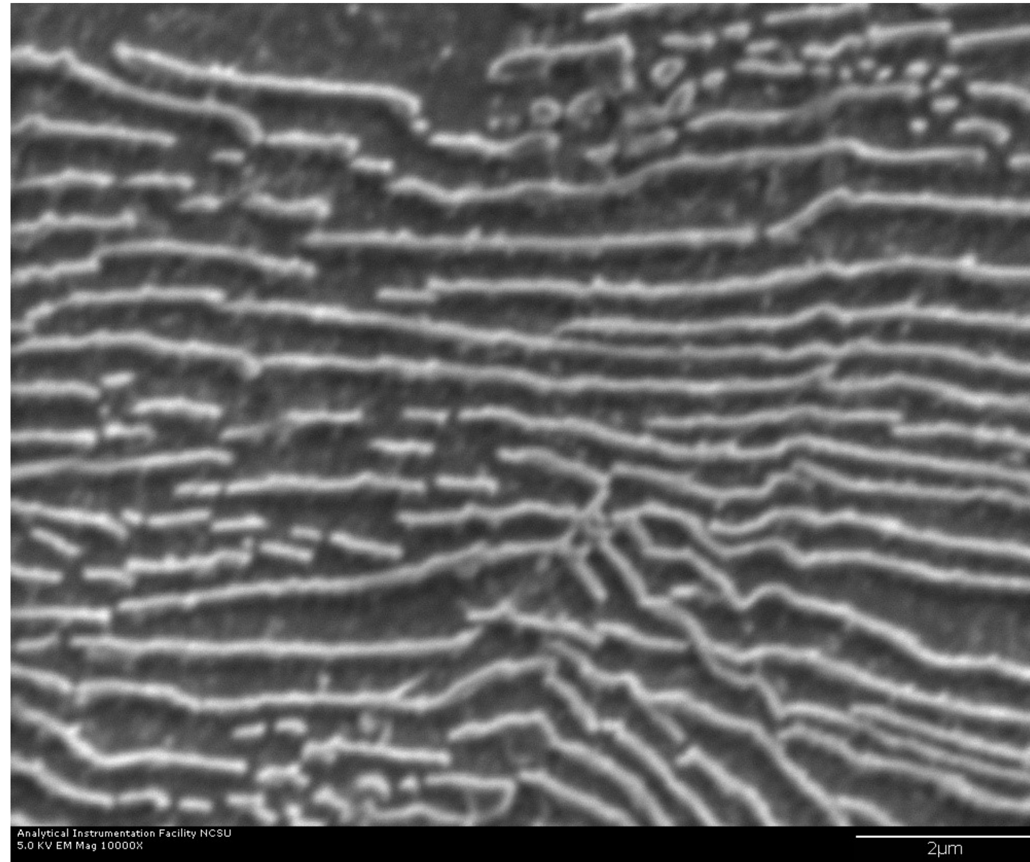
- Follows the same rules as all SEMs
- Smaller beam currents = better resolution

- Difference is that there will be a threshold below which the apparent resolution does not increase

- Verios 460L – hard to tell a difference in resolution between 200pA and 6.3 pA
  - Two orders of magnitude of current change with no discernable difference in resolution!
  - Not just the Verios, all immersion lens UHR-SEMs seem to have this property

Condenser lens effect on resolution

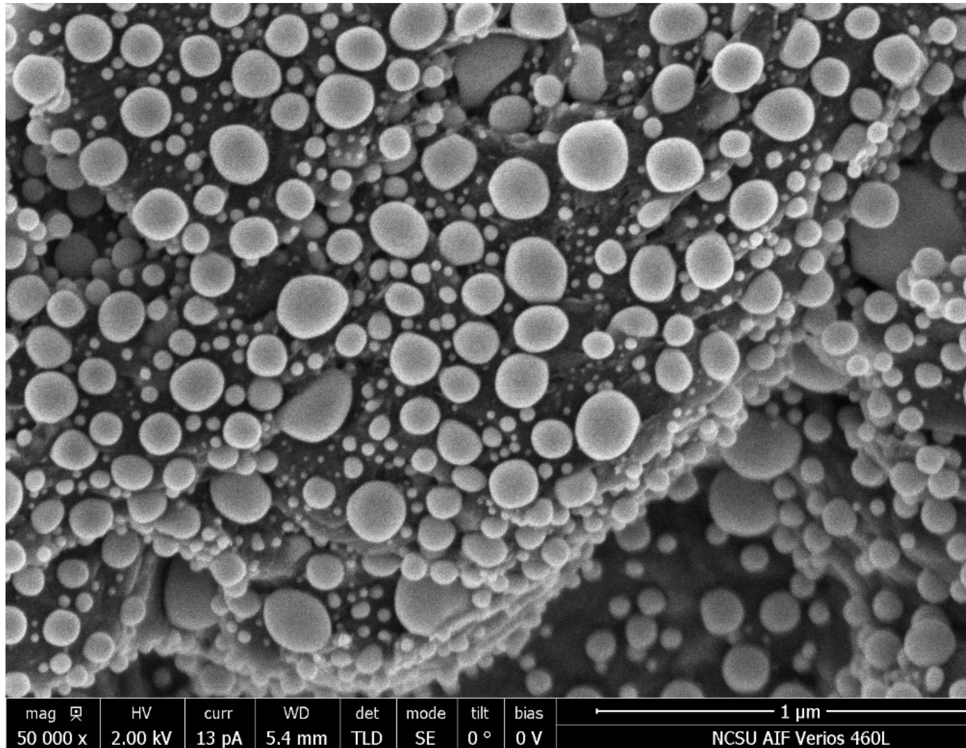


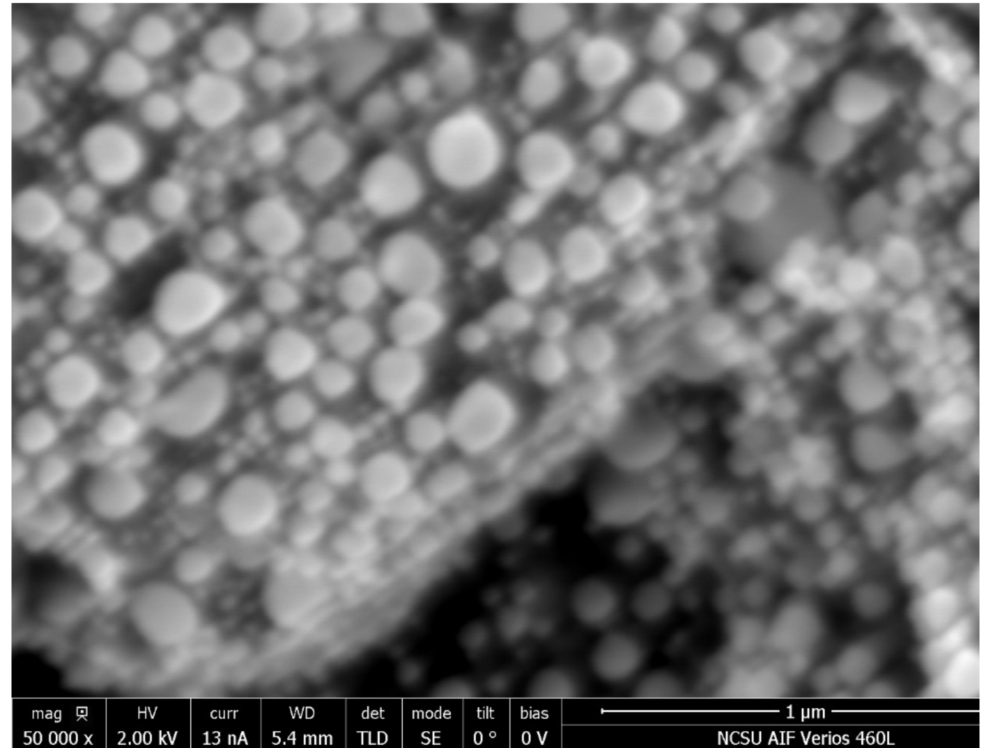


C.Mooney

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

Condenser lens effect on resolution





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- Sn on C sample, UHR-SEM
- All imaging conditions kept constant except for current setting
- Image on left: 13 pA current = low beam current, small spot size
  - Champion data
- Image on right: 13 nA = high beam current, large spot size

Working Distance

- The working distance is the physical distance between the objective lens and the sample
- The pole piece (or pole tip) houses the objective lens

Focal Length of the Objective Lens

- **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 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

Practical SEM: Working Distance

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

- Short WD for high resolution
  - Conventional SEM: ~ 5 mm
  - 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: 10 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 = 10 mm, Verios = 6 mm
- If you don't know:
  - Conventional SEM: Choose 10mm, which is also typically the correct WD for EDS!
  - UHR-SEM: 4 – 6 mm WD will typically give good results

Objective Aperture

- The objective aperture limits the number of electrons that can get to the sample -- merely a hole in a plate that is inserted into the path of the beam

Practical SEM: Objective Aperture

- Small apertures are used in high resolution applications to limit aberrations in the beam that reduce resolution or to increase the depth of field
- Large apertures are used when high current is required (EDS)
- 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!

SEM Rules:

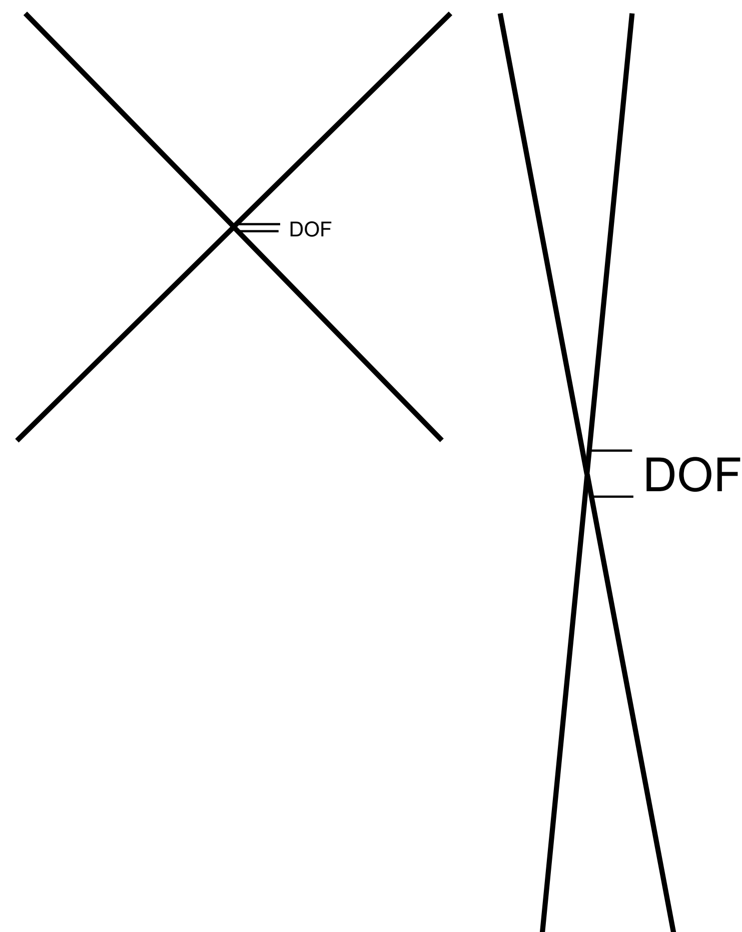
- Use a small OA for high resolution or high depth of field imaging
- Use a large or no OA for EDS
- Use no OA when changing accelerating voltage or condenser lens

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

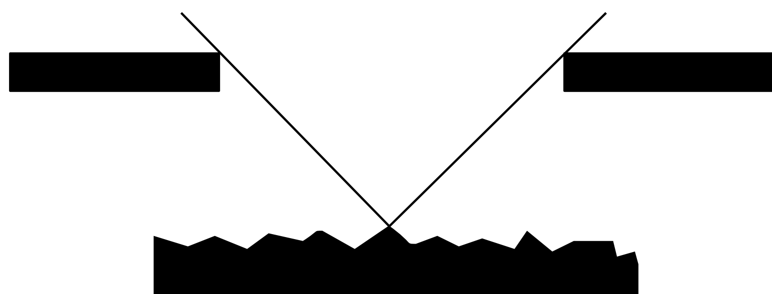
- Small Aperture and long working distance (right) give the highest possible depth of field

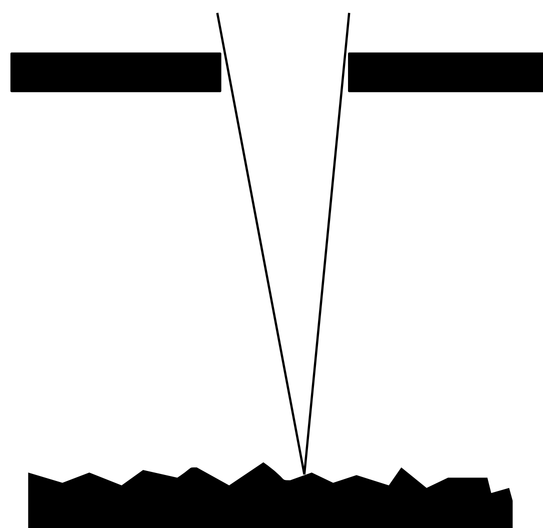


Simple geometric schematic of increasing depth of field.
Low depth of field condition on left.
High depth of field condition on right

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)

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!

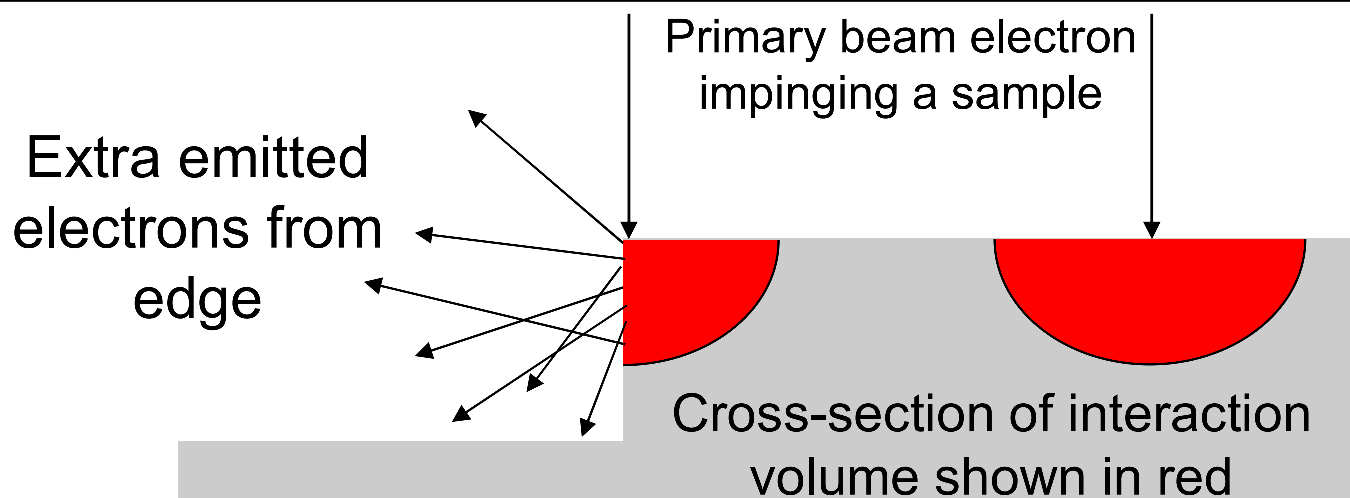
- The UHR-SEM is not designed for high depth of field

- For high resolution, choose a small current!

Practical SEM Issues

- Edge Effects
- Magnification and Resolution
- Dwell time
- Charging
- Beam Damage
- Aesthetics
  - Detector choice and position
  - Composition

Edge Effect Schematic



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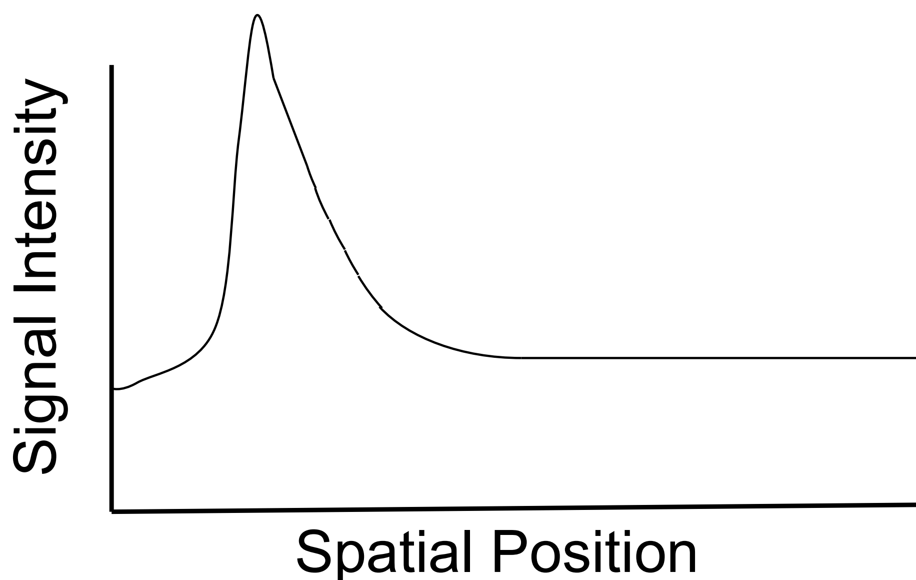
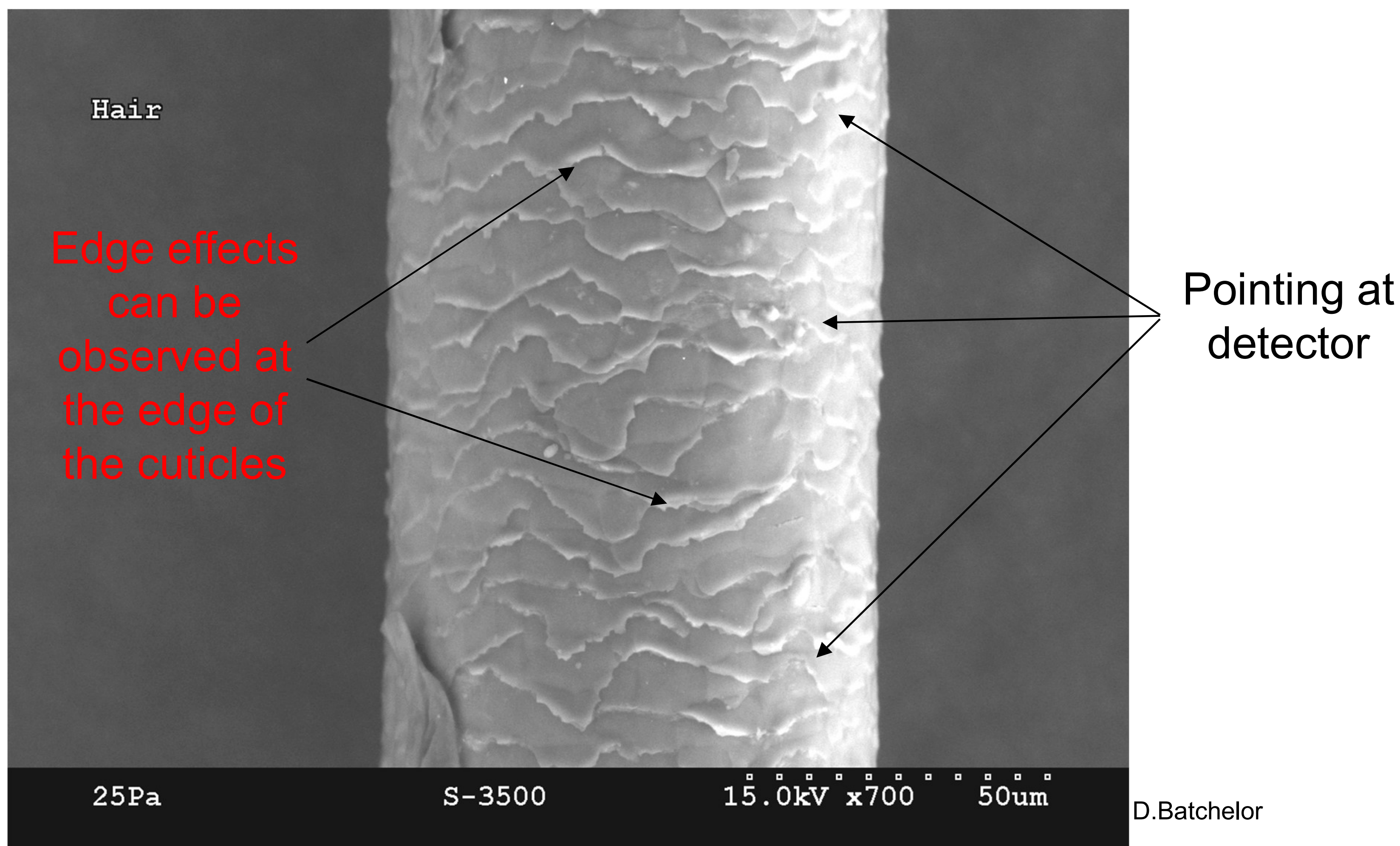


Signal increases at the edge due to increased electron emission

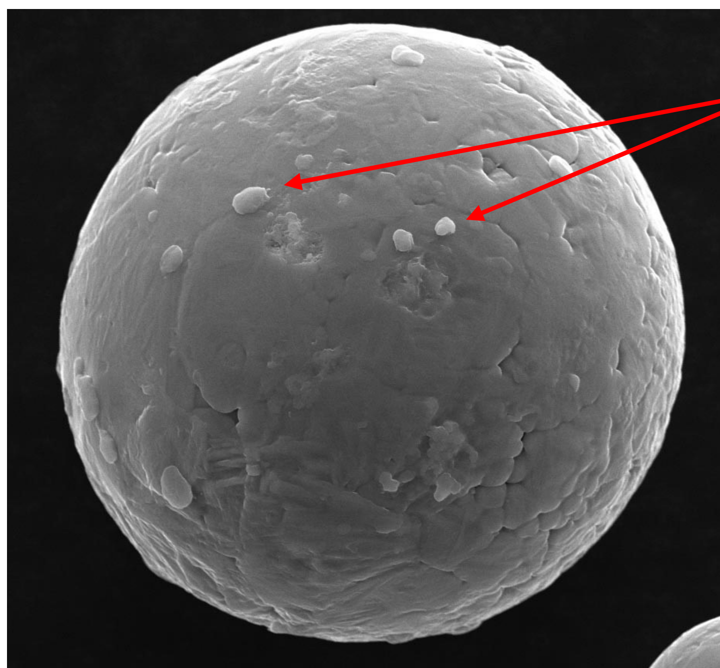
Edge Effects



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

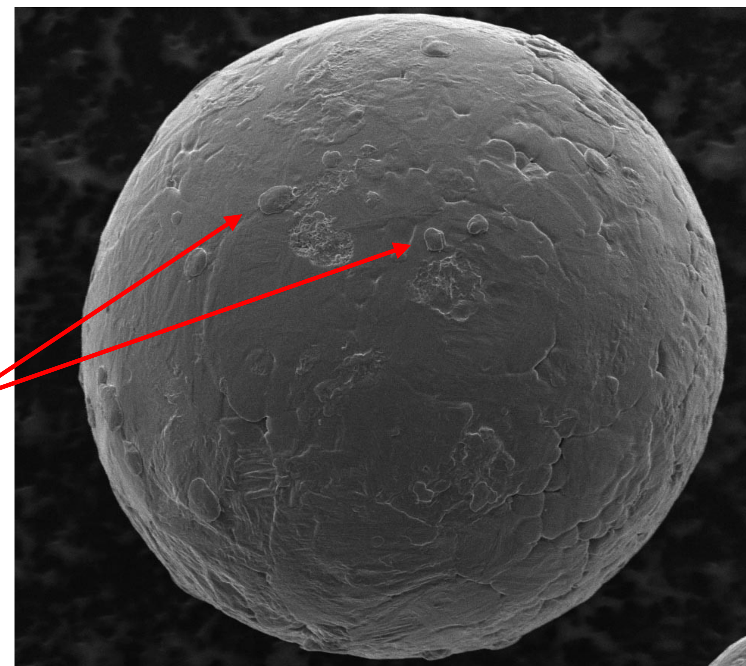
Edge Effect Issues

- 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



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

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

- 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

Note on Magnification in the SEM

- Modern scholarly journals do not like to show the magnification, because it is probably wrong when displayed in the article

- That said, the microscope only knows magnification
  - As far as I know this is true for all SEMs

- So, when specifying what you want from an SEM or trying to repeat work or just being consistent, pay attention to the magnification!
  - All manufacturers have different micron markers and there may be more than one micron marker with the same value but a different length

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!

Resolution Considerations

- In general, it takes on the order of 10 steps across a feature to claim that the feature has been resolved
- This means that the actual resolution must be an order of magnitude better than the smallest feature that is observed
  - The real world resolution is equal to the smallest feature
  - A single pixel does not resolution make!
- Features smaller than 10 – 20 X the resolution limit will appear to be fuzzy
  - That is, if the resolution under current operating conditions is 0.5 nm, then any feature smaller than 10 nm will appear fuzzy and slightly out of focus
  - True for any type of far-field microscope

SEM Resolution Limiting Factors

- Spot size limited resolution: (easy and obvious to define)
  - Where the size of the beam is greater than the size of the feature or the pixel

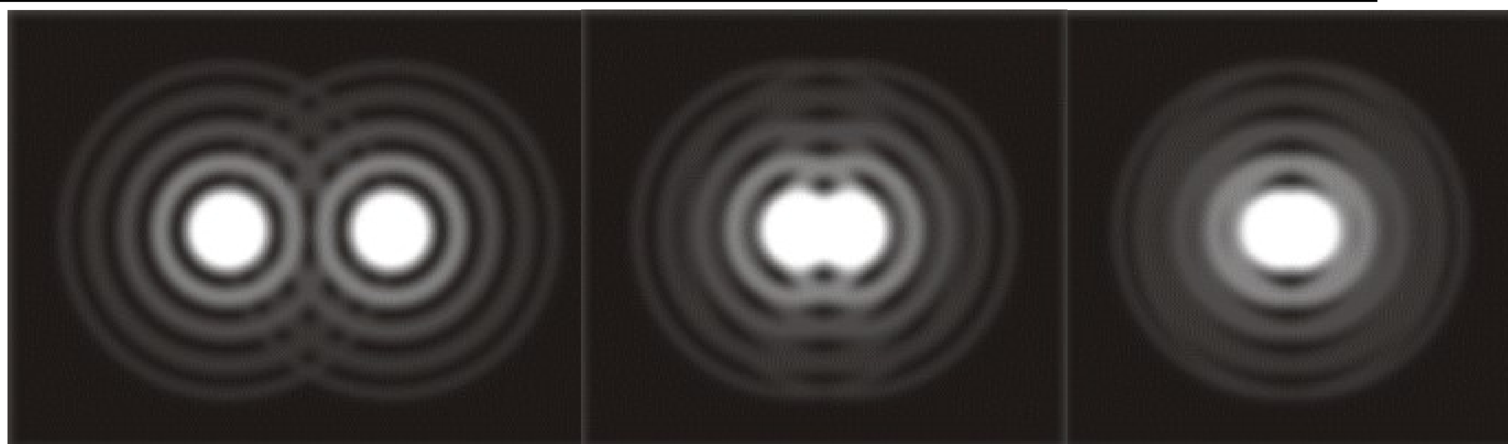
- Pixel limited resolution: (easy and obvious to define)
  - Where the size of the beam is smaller than the size of the pixel but the pixel is larger than the feature of interest
  - Also note that it takes more than one pixel to resolve a feature!

- Interaction volume limited resolution: (not so easy or obvious)
  - 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
  - Leads to the concept of a fundamental SEM resolution limit (0.5 nm)

Spot Size Limited Resolution

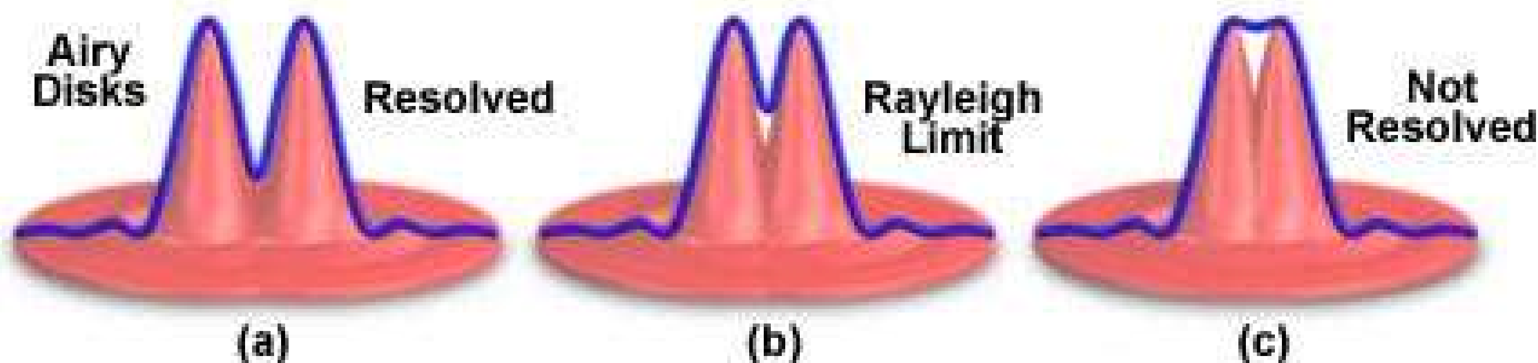
- The best physical definition for spot size limited resolution comes from photon optics and is generally referred to as the Rayleigh criterion
  - This ignores the interaction volume because the interaction volume for photons is very shallow relative to achievable spatial resolution
  - There is an 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
- Rayleigh criterion is named after John William Strutt, 3rd Baron Rayleigh aka Lord Rayleigh (1904 Nobel in Physics for the discovery of Ar)

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

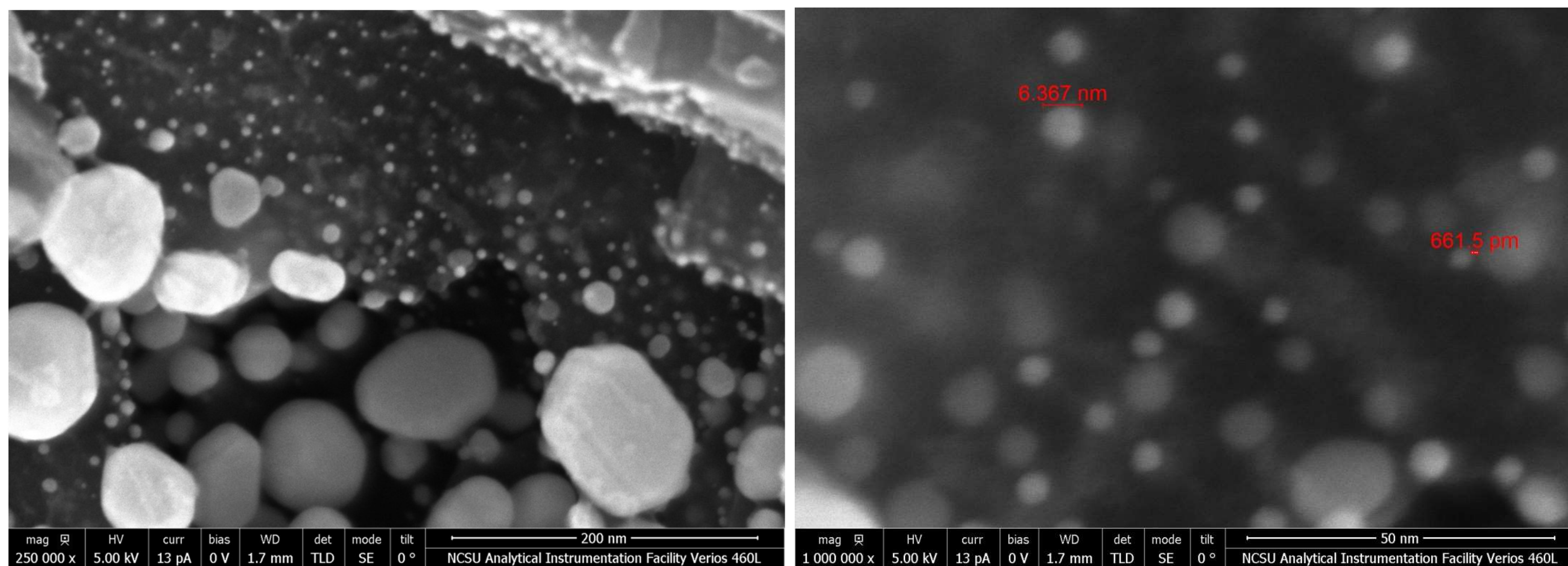
- The electron beam has a Gaussian distribution and appears as an Airy disk without the diffraction rings
  - The electron spot is not diffraction limited!
  - We don't have to worry about the diffraction rings!

- 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



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- 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 Anstroms (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

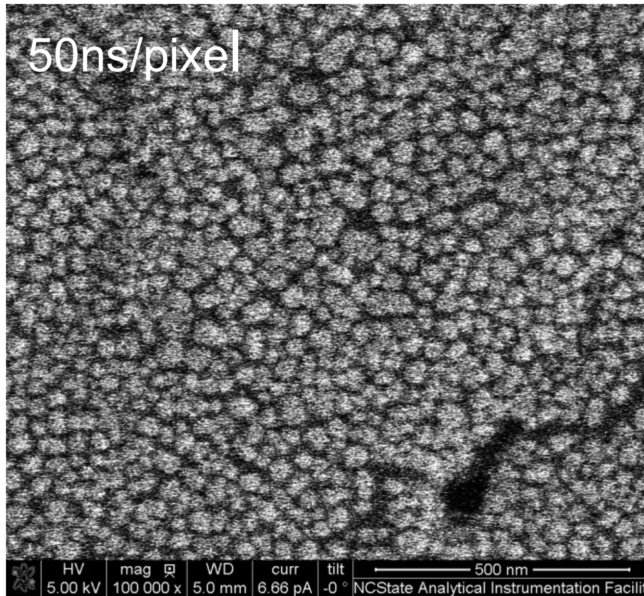
- Resolution will not get any better than 0.5nm
- It will get easier to achieve 0.5nm resolution
- 0.5nm resolution will happen at lower voltages as the optics improve
- Cs correctors will not become common on SEMs
  - Cs correctors allow for smaller, more precisely defined beam sizes and are now used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

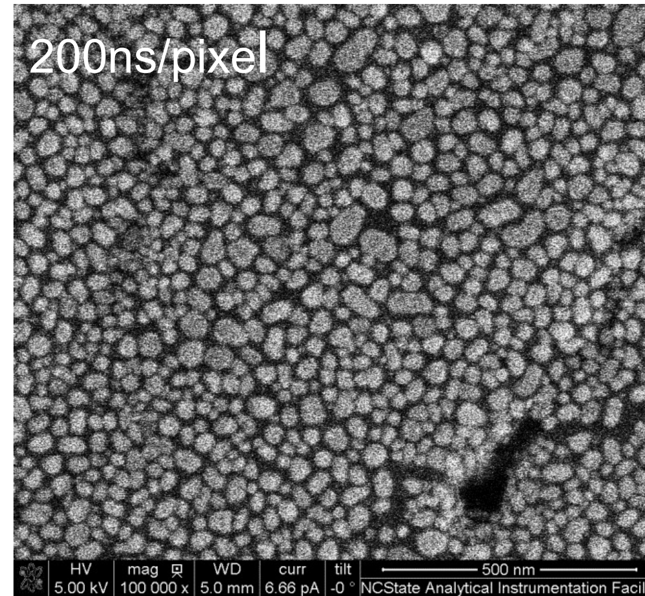
Practical SEM: Dwell Time

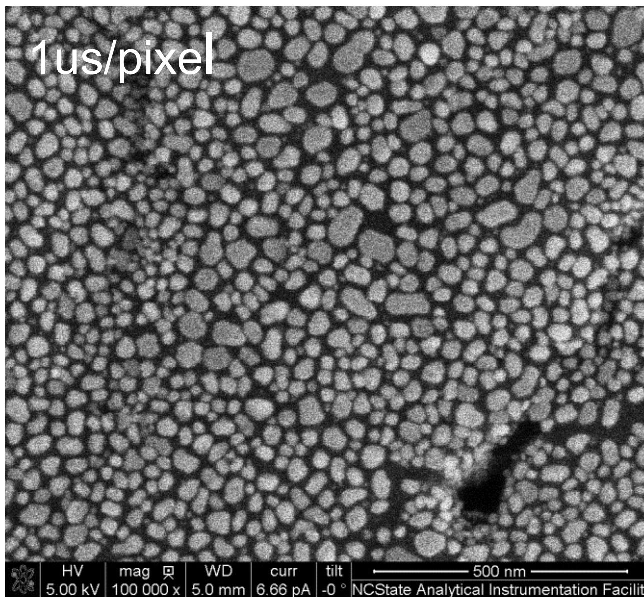
Assuming a conductive or coated sample:

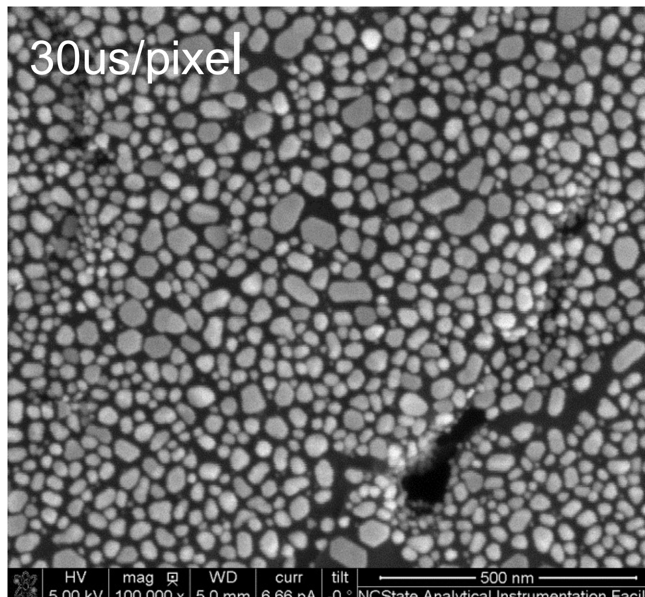
- ETD images are publication quality with ~30us per pixel of dwell time
- BSE images can take longer
- Actual dwell time will depend on beam conditions (current and voltage) and detector efficiency
- How to decide for sure – look at the image!
  - If the image is noisy, increase the dwell time
  - If 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**

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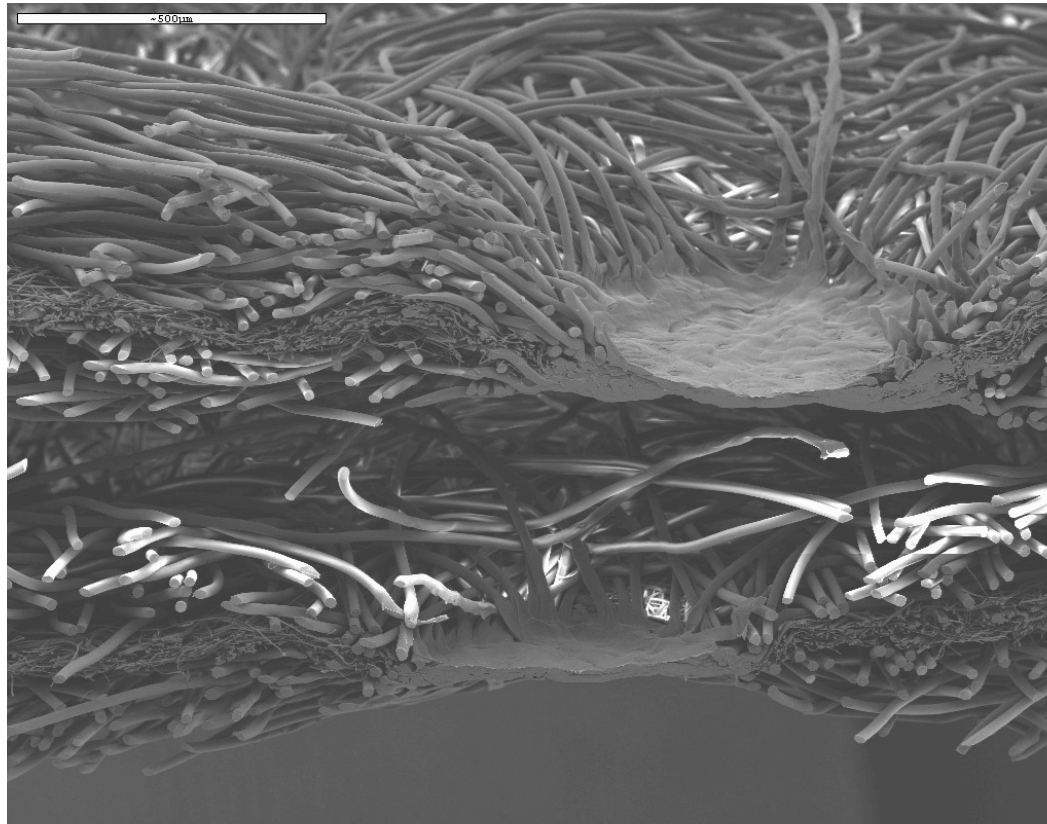
Charging

- Imaging using electrons
  - Not a problem for conductive samples
  - Many samples are not conductive!
- Charging occurs when the number of electrons going in a sample is different from the number of electrons exiting the sample.
- Charge balance is generally achieved by flowing electrons in or out of the specimen directly via a ground path.
  - Note that insulators typically do not flow charge very well!

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.

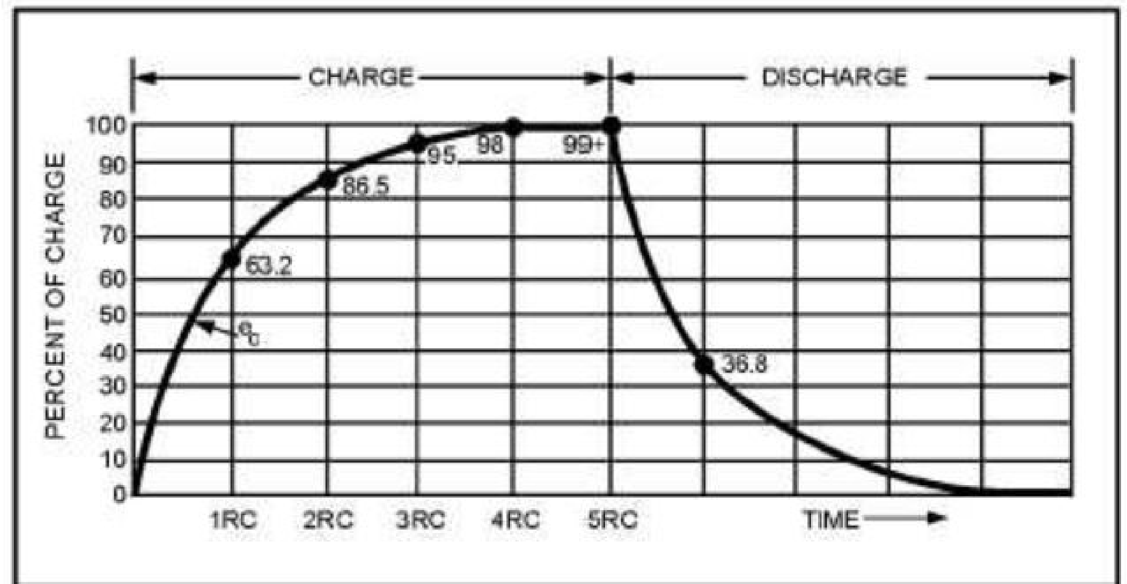


Image source unknown

Charging Example

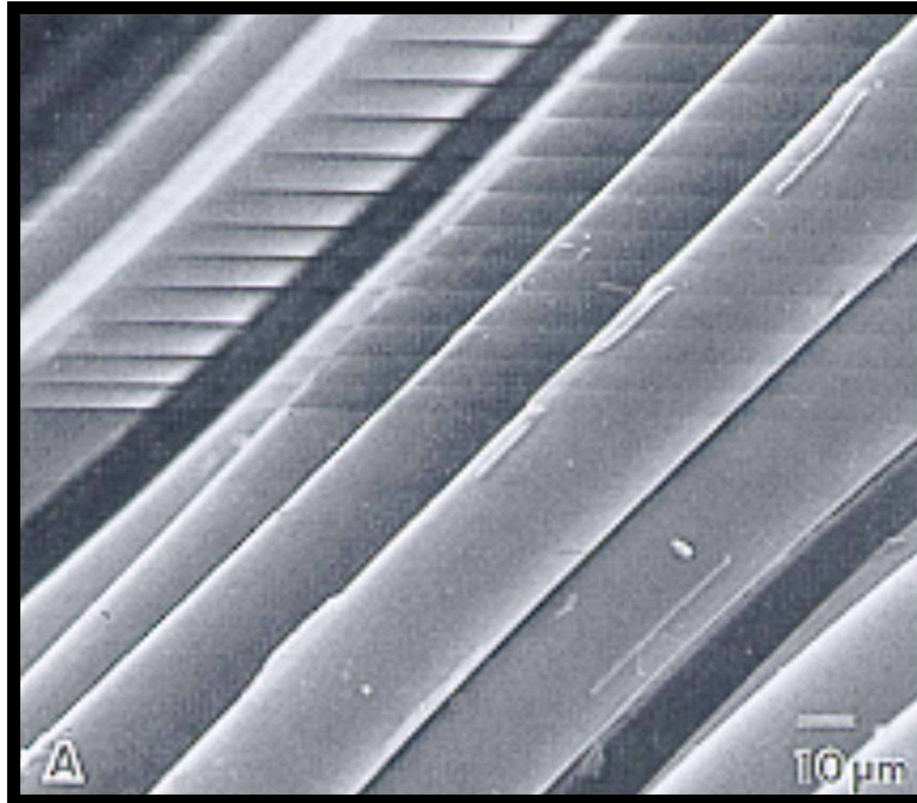


Image source unknown

- 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

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
- Image insulators at the charge balance point
- Operate in variable pressure mode

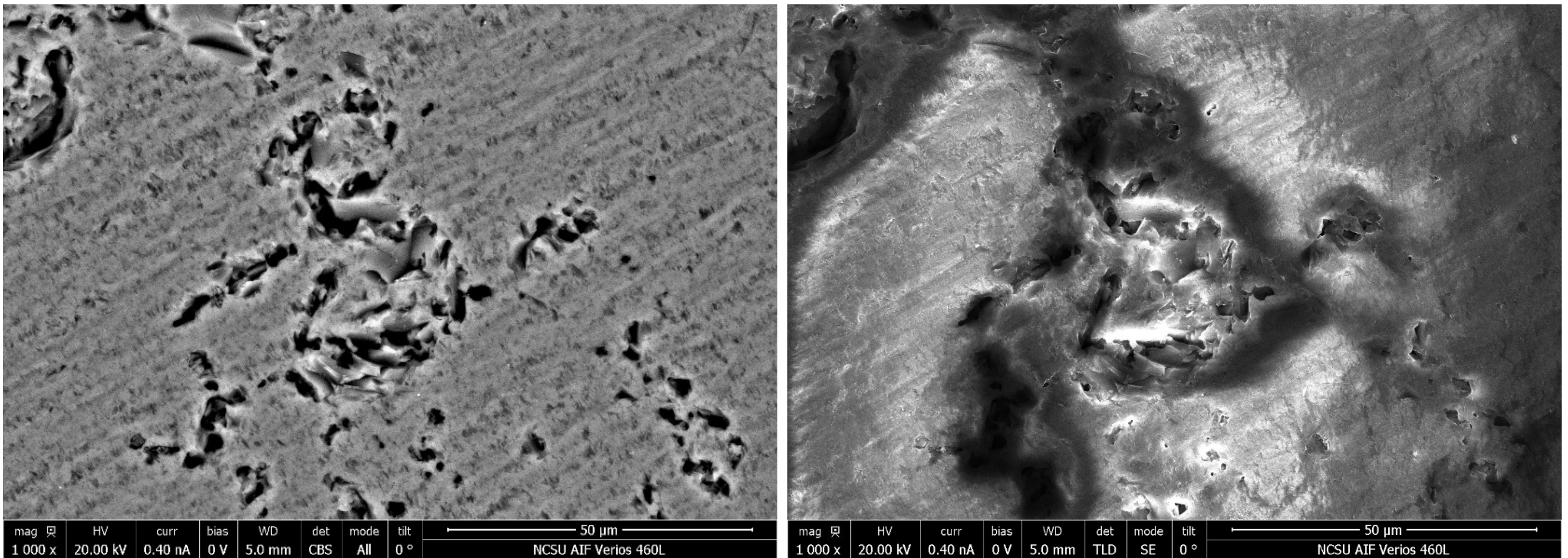
Practical SEM: Ignore the charging

- Some insulators (mostly ceramics and other polycrystalline materials) can be imaged without a conductive coating at high voltage using the BSED

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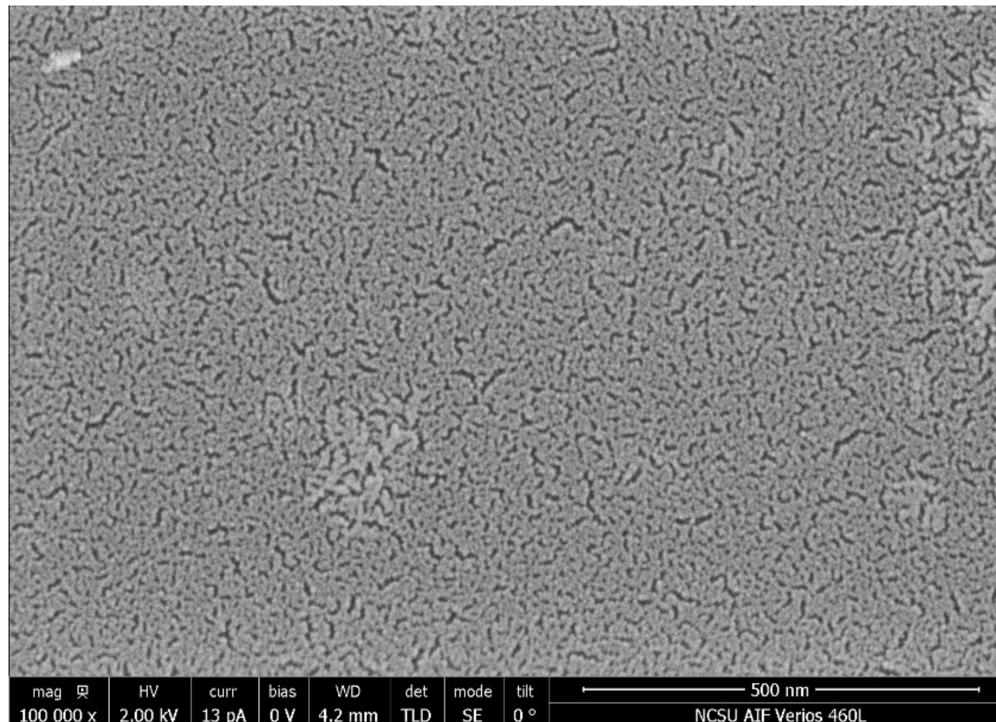
- Ceramic sample imaged at 20 kV with 400 pA of current
  - High voltage, moderate current conditions
- BSE image on the left, SE image on the right
- BSE image is nice
- SE image shows charging

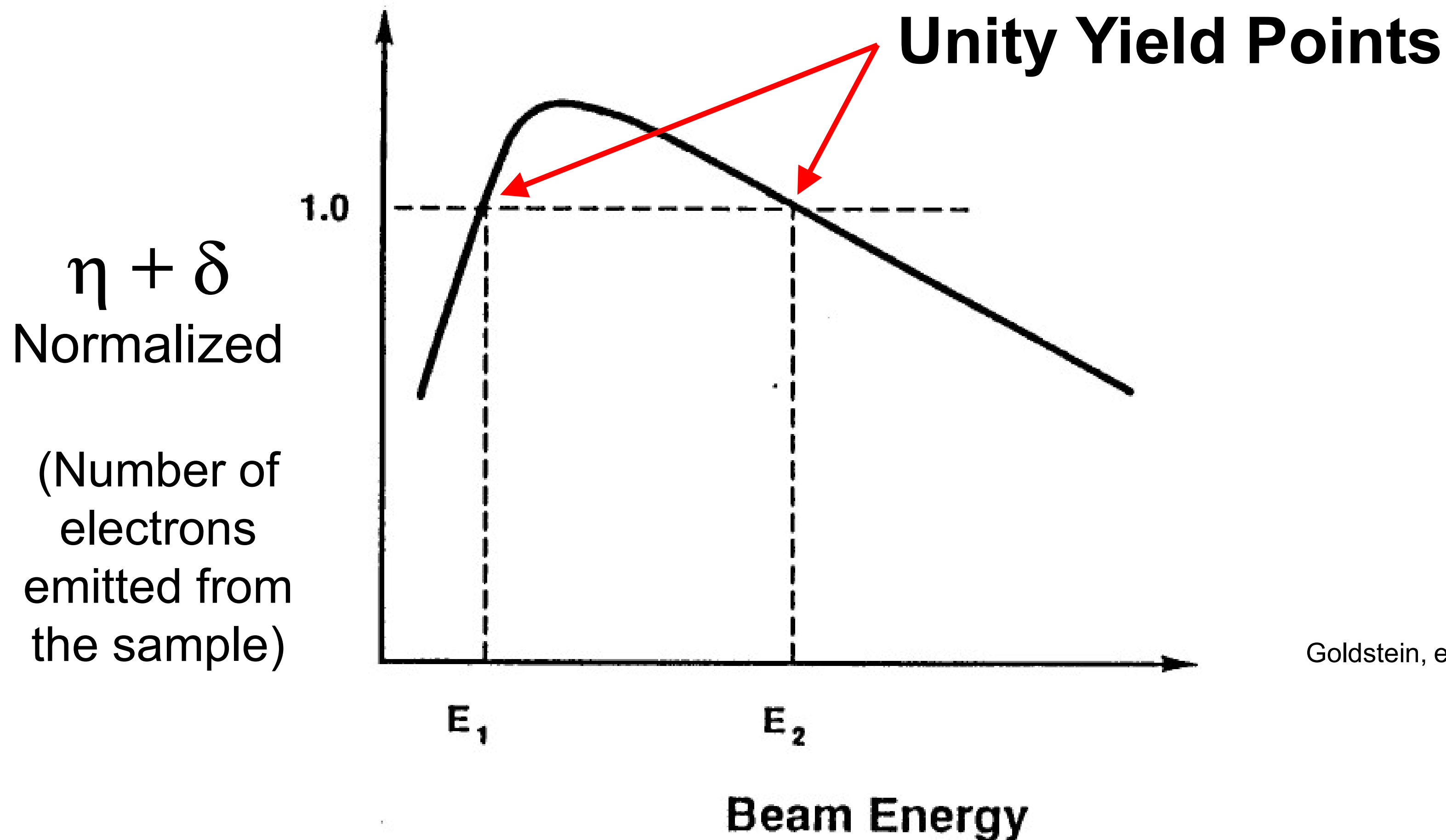
Coating Samples with a Conductor

- This is generally not desirable
- For low resolution/magnification applications (<20kX), it usually doesn't hurt imaging
  - Not so good for EDS
- Typically use Au/Pd (60/40)
  - Smooth, non-toxic, doesn't oxidize
- High resolution
  - Not so good



Most of the structure in the image is from the Au/Pd coating!

Charge Balance Points



- At the unity yield point, the number of electrons going in and out are equal – E2 is best for imaging
- Typical E2 energies ~ 500 eV – 1.5 keV

E1 vs. E2 Charge Balance Points

- 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

Practical SEM: Low voltage

Uncoated insulators in the Verios SEM:

- Start with a 500 V beam and a 500V stage bias
- Low current – 13 pA
  - less than 6.3 pA does not seem to make enough signal
- Play with the beam energy and stage bias to control charging

Pro tips:

- Scan fast! (Charging is time dependent!)
- Set up a photo integration using the slowest scan rate that does not show charging
- Note:
  - BSED often does not work well with low energies
  - EDS requires energy

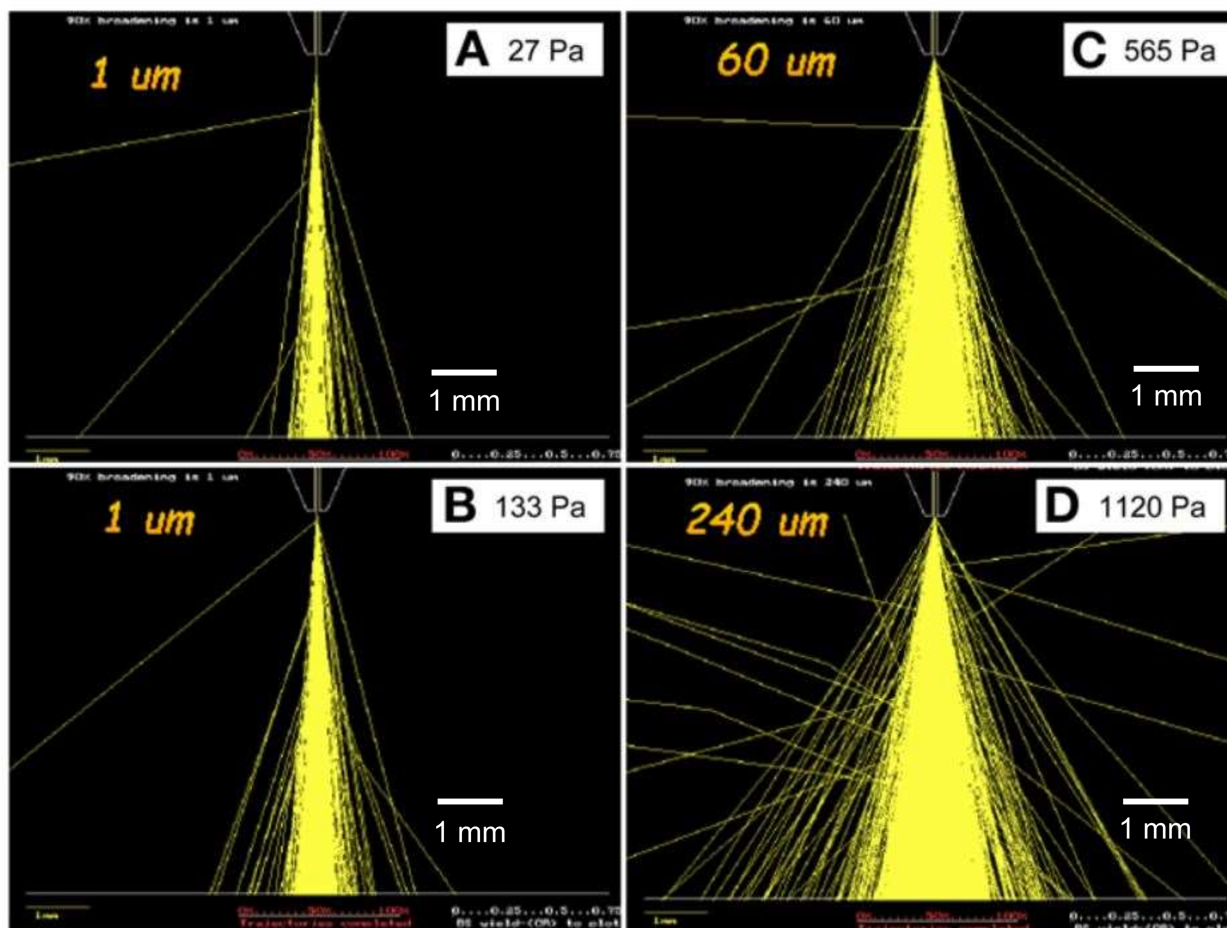
Imaging Insulators – Variable Pressure

- By bleeding a little gas into the sample chamber, charging can be reduced
  - 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.

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

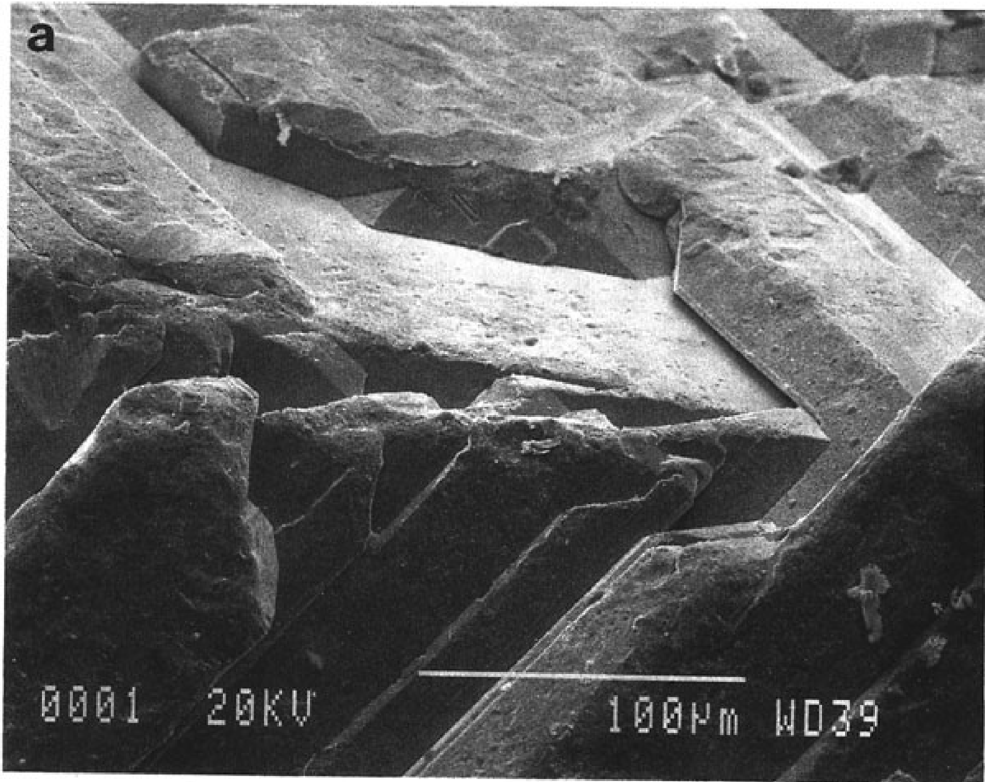
Image Appearance: General

In general in the SEM:

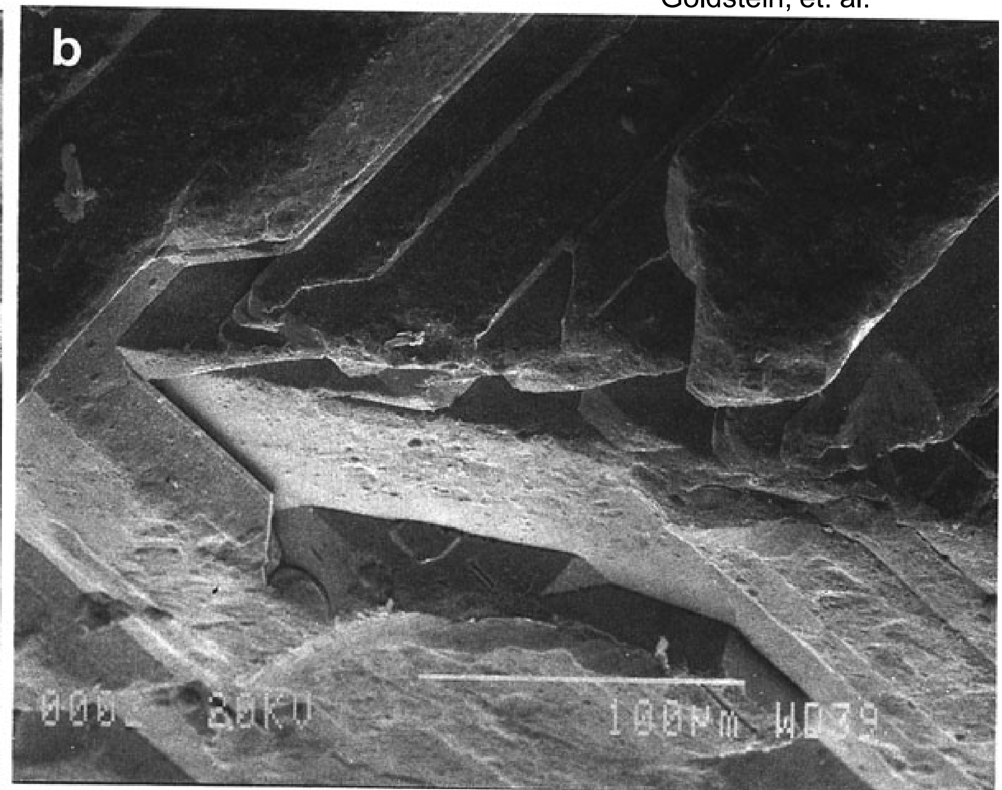
- The illumination source appears to be the detector position
- The view angle appears to be from the electron source, i.e., down the column
- Consider the geometry of the system to make images the most aesthetically pleasing
  - System geometry = beam-sample-detector geometry

Detector Position

Goldstein, et. al.



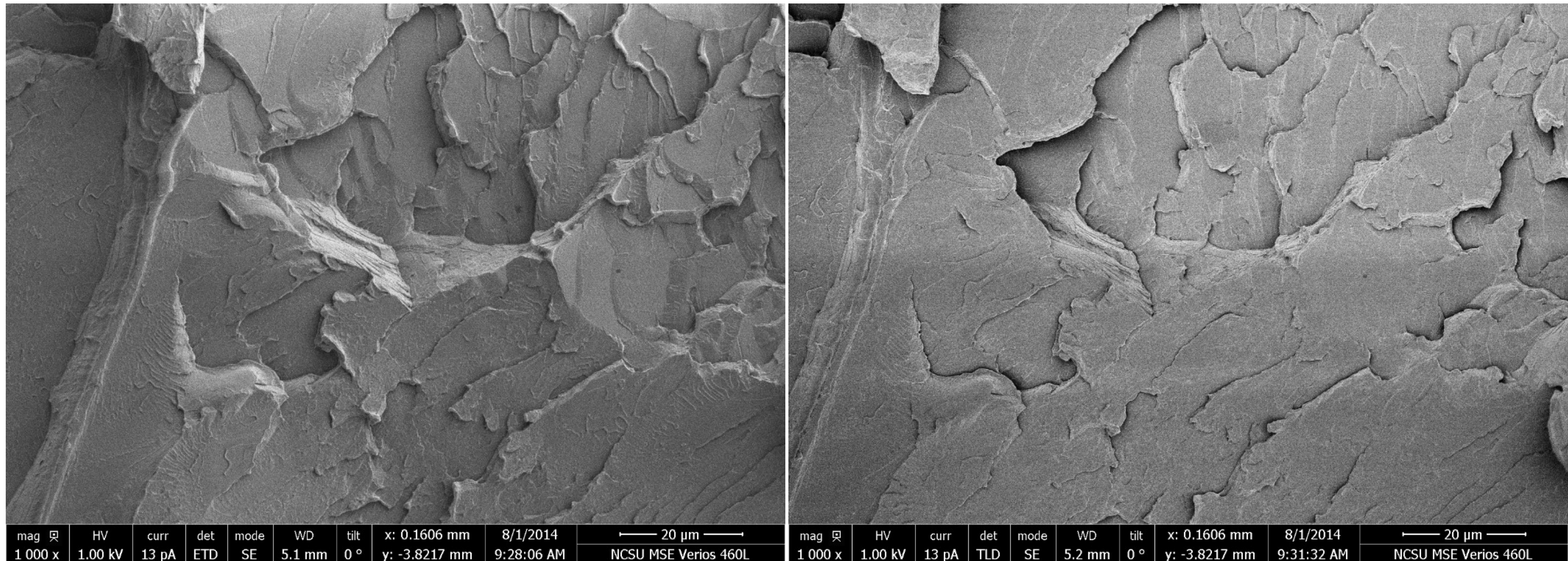
Champion Data: Shadows look natural



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

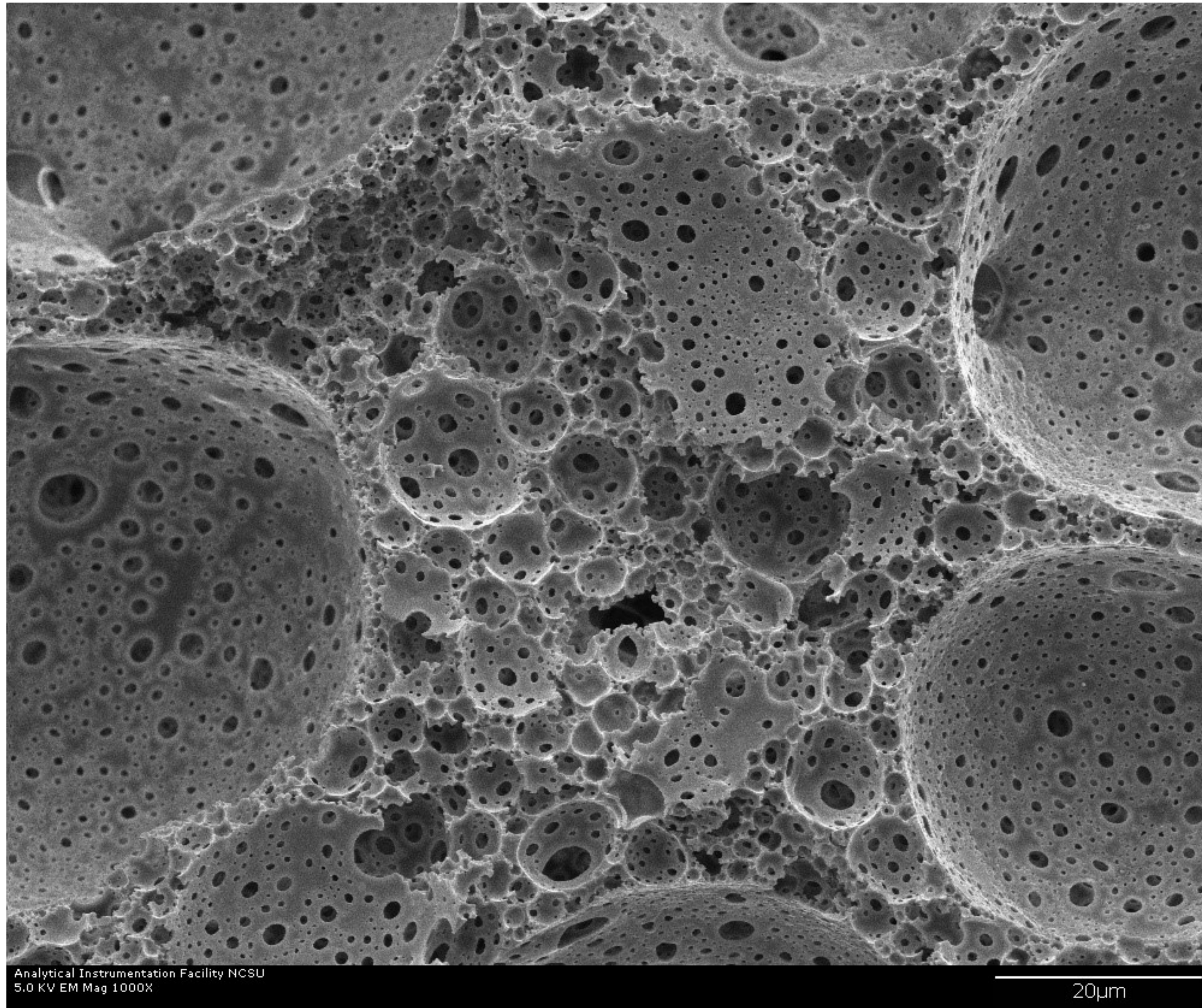
- It is smart to use the same magnification sets when collecting data
  - This way, data from different samples can be directly compared!

- 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

SEM Imaging Done Right…

Aluminum
Foam



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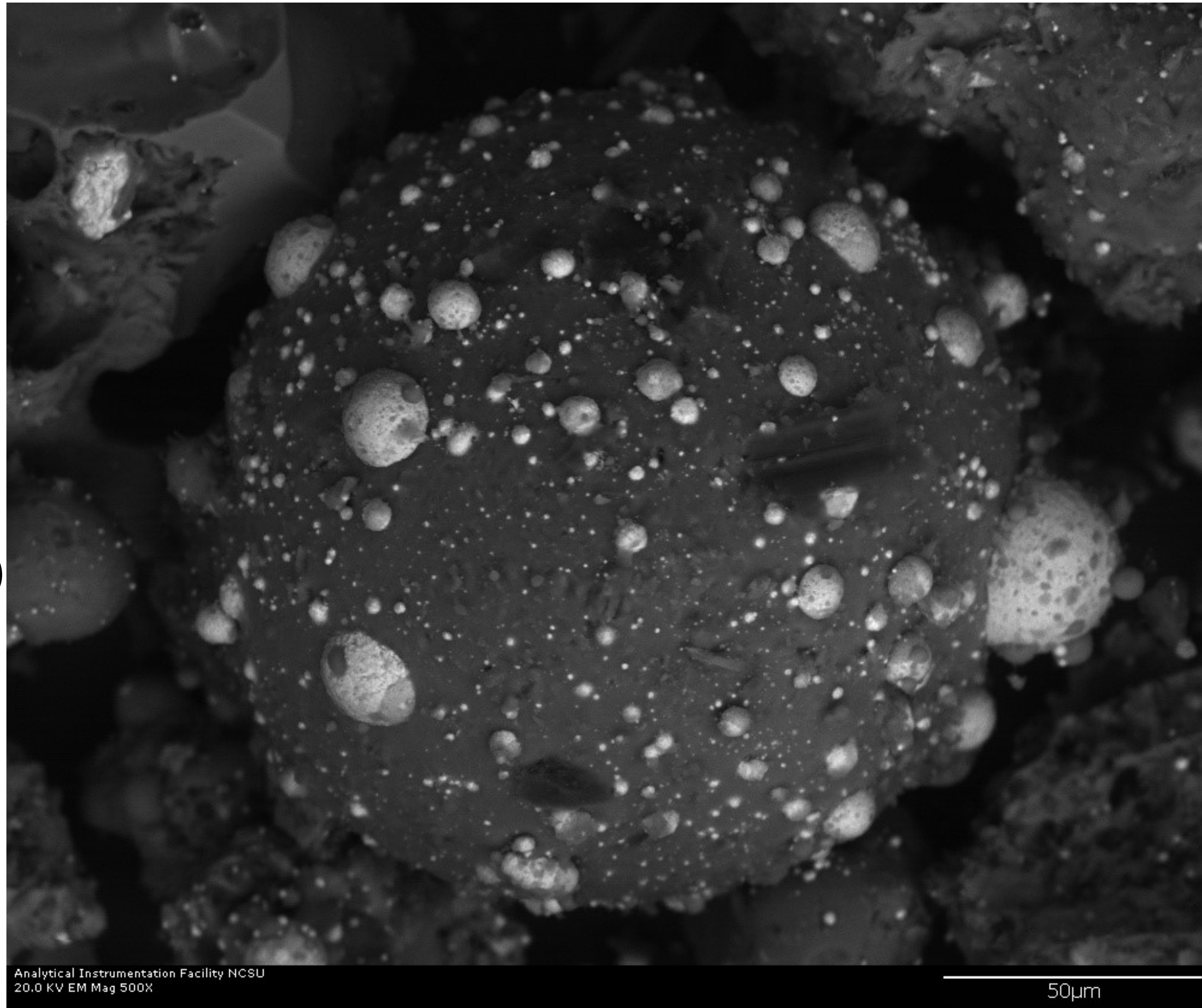
SEM Imaging Done Right…

Carbonaceous sphere with metallic spheroidal inclusions (BSE detector)



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X-ray microanlaysis

- 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 microanlaysis

- X-ray microanlaysis 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 in the SEM

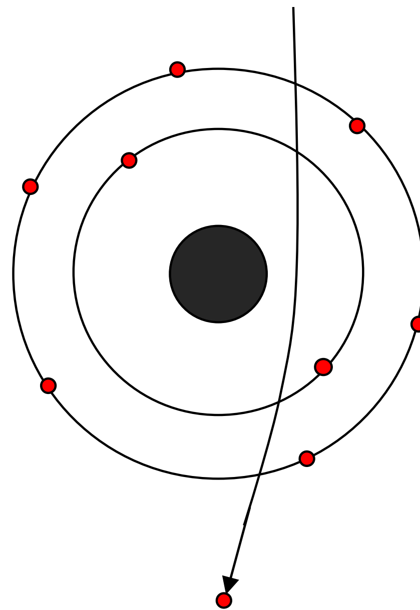
X-rays are produced in the SEM by high energy electrons interacting with the sample.

Two types of X-rays produced:

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

Bremsstralung

- Bremsstralung 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



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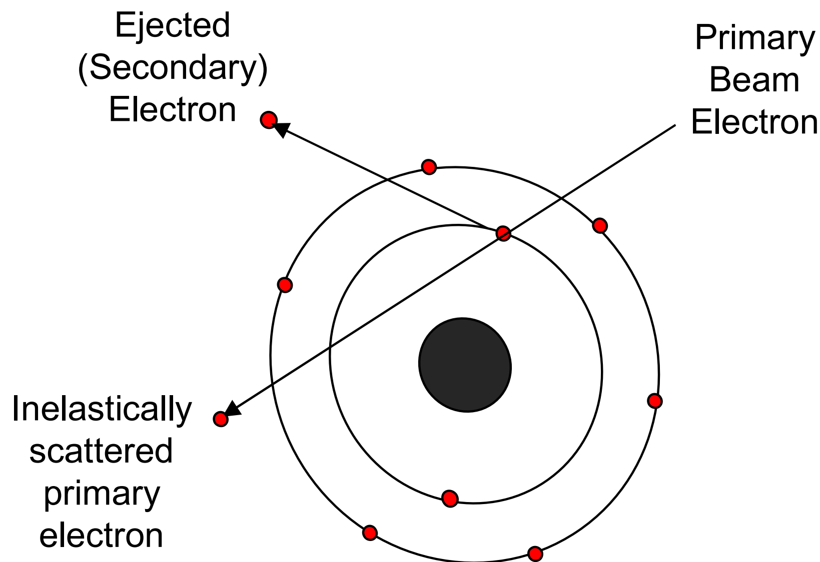
Bremsstralung

- Bremsstralung are not characteristic of any of the elemental species in the sample
- Bremsstralung 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
- Bremsstralung is not to be confused with noise!
  - Noise in an X-ray spectrum are extraneous counts that do not come from detected X-rays
  - Bremsstralung 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



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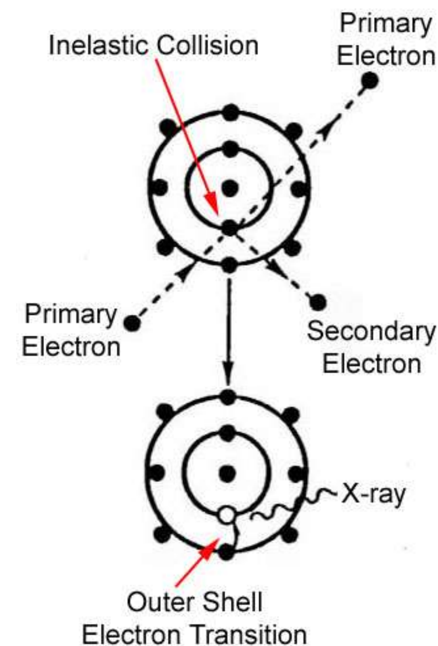


Adapted from Goldstein, et. al.

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 from of a characteristic (X-ray) photon or an ejected characteristic (Auger) electron

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 Kα (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

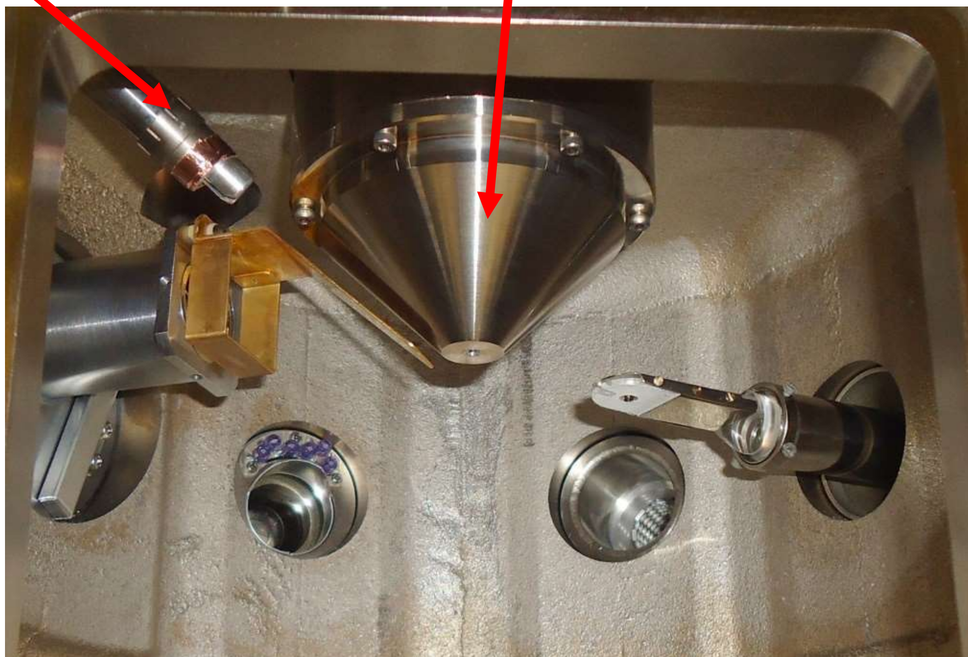
If the spectrum is mostly artifact, why do so many SEMs (>50%) have EDS detectors?

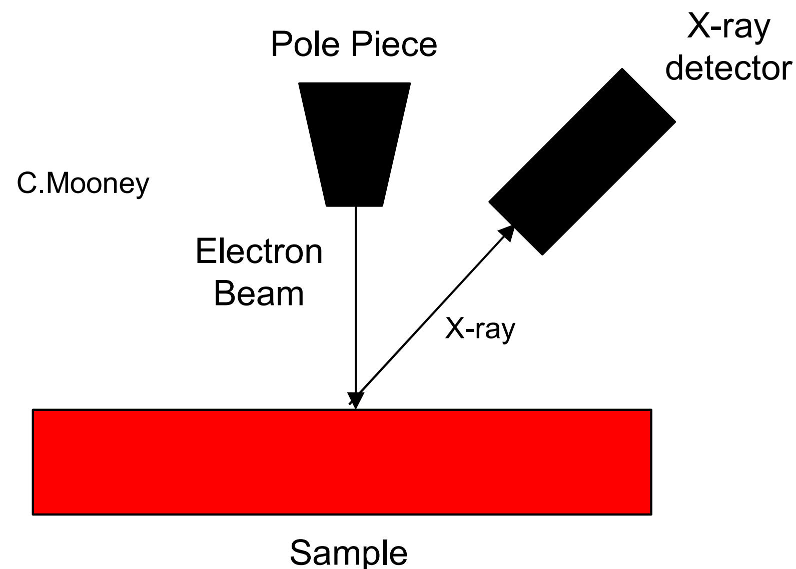
- There is a single detector that detects the X-rays
  - WDS = multiple crystals, usually multiple detectors
  - EDS has much lower overall cost
- All the X-rays are collected at once (faster! – time is money!)
- Determining the unknown contaminant is relatively quick and easy

EDS System

X-ray Detector (SiLi)

Pole Piece (Objective Lens Housing)





- 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

Princeton Gamma Tech, now Bruker

X-ray Detection with an EDS detector

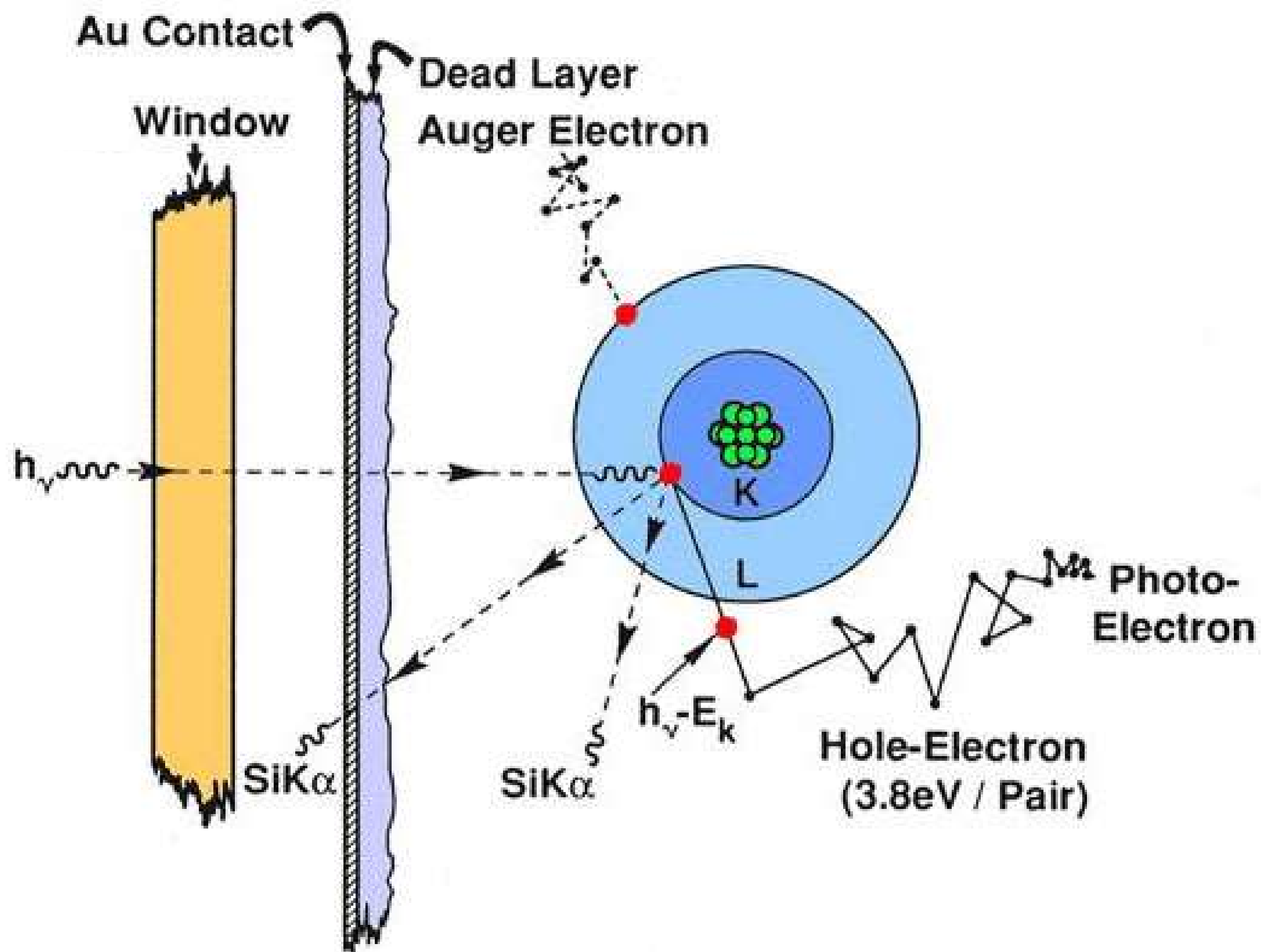
1. X-rays from the sample pass through a protective window and strike the detector crystal
   - Why a window? The detector is cooled: Window protects the detector from contamination collecting on the cold surface
   - Old school detectors had Be windows – absorbed energies below 1 keV, so no elements below Na (Kα = 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 Kα 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

X-ray detection process

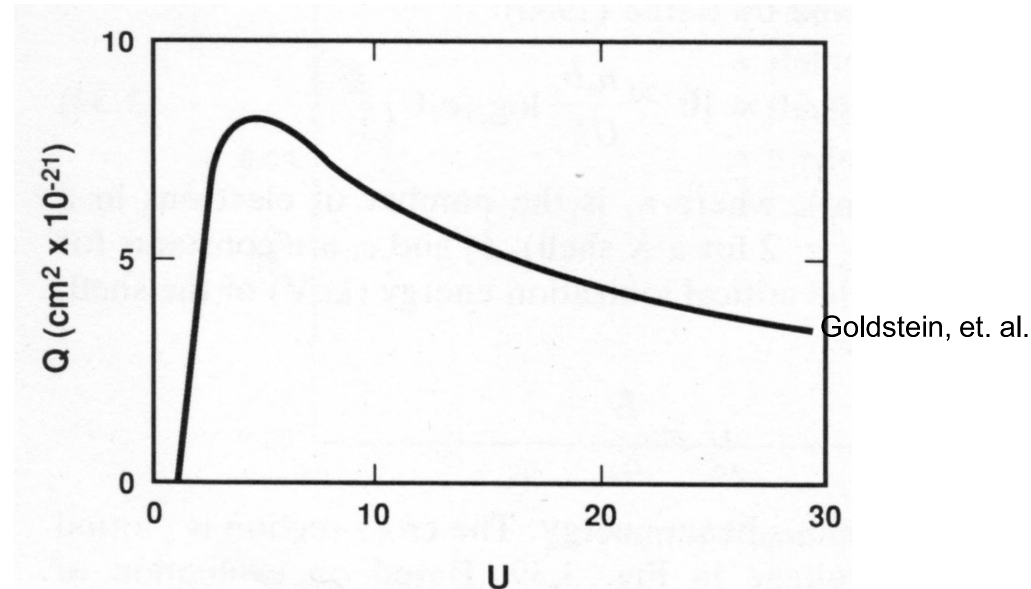


Goldstein, et. al.

Schematic of the X-ray detection process

SiLi detector shown

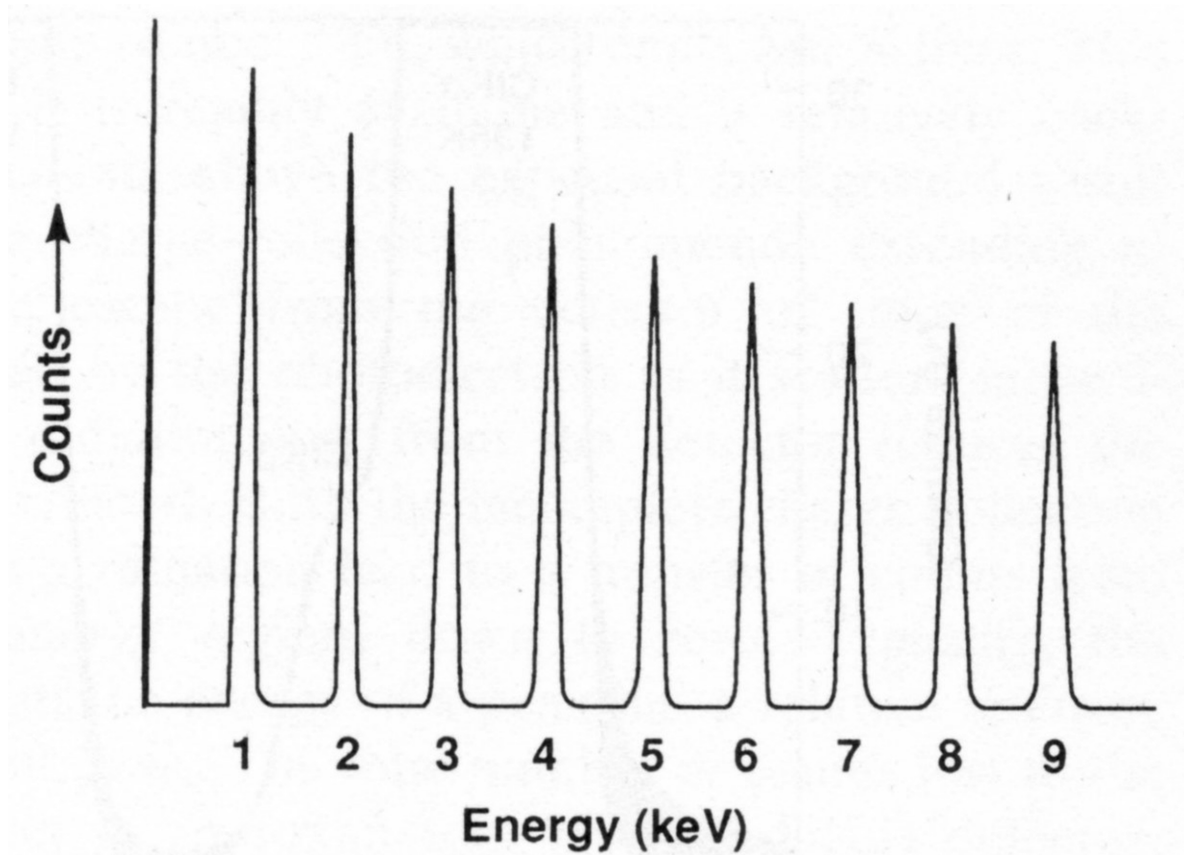
Practical SEM: Voltage for EDS



Plot of cross-section for inner shell ionization as a function of overvoltage U, where U = Beam Energy / Ionization Energy.

- This plot suggests that the optimum energy for producing the most X-rays is about 3 – 5 times the energy of the X-ray
- To minimize the interaction volume, choose U = 2
- For unknowns, start at 20 keV

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

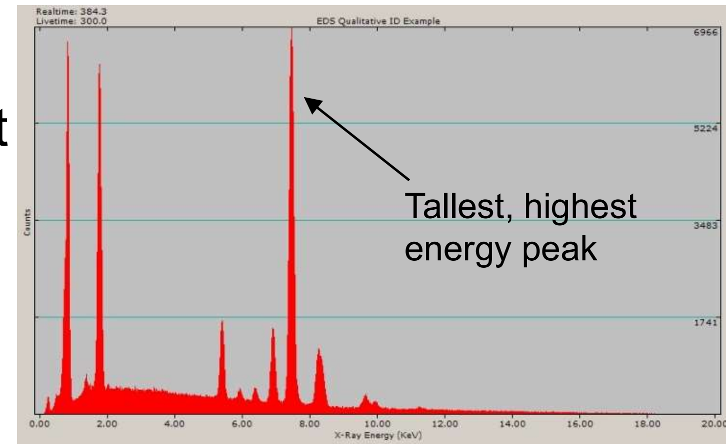
A Method for Qualitative Analysis

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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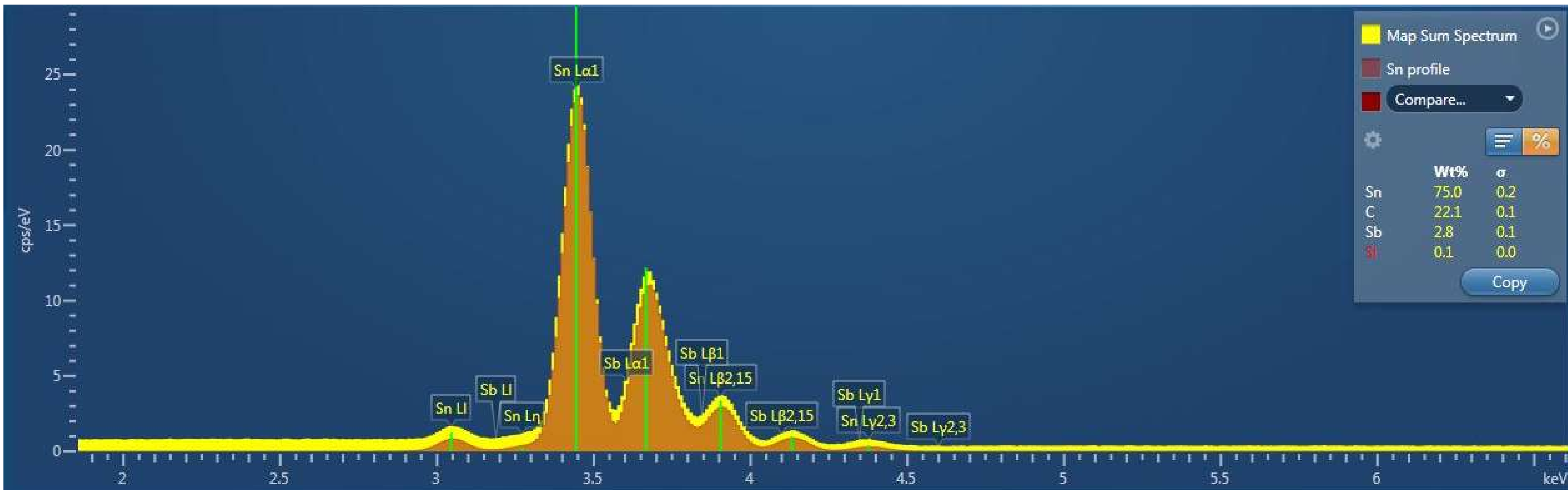
2. Move to the next tallest, high energy, repeat steps 1a – 1c

3. Repeat until ***ALL*** peaks are accounted for
   – If there is a peak, something caused it
   – Account for any sum or escape peaks

EDS Peak Matching



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

- 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

Old School EDS Analysis

- Back in the day, EDS analysis was done with a slide rule or table
- Modern systems have auto ID functions
- ID the spectrum yourself until you feel comfortable
- Never trust the software!
- Trust your brain!

K SERIES	M SERIES						
Kα*	Kβ	Mz	Mα	Mβ	Mγ	M_2N_4	N**

* The combined $K\alpha^1$, $K\alpha^2$ is used for Z less than 40. The $K\alpha^1$ only is used for the higher Z values.

** The data set for the N-Series peaks is not completely known.

EDAX

advanced microanalysis solutions

AMETEK

MATERIALS ANALYSIS DIVISION

3 Li	0.052	35 Br	11.904	67 Ho	47.526
4 Be	0.109	36 Kr	12.628	68 Er	49.091
5 B	0.183	37 Rb	13.371	69 Tm	50.722
6 C	0.277	38 Sr	14.138	70 Yb	52.353
7 N	0.392	39 Y	14.928	71 Lu	54.069
8 O	0.525	40 Zr	15.773	72 Hf	55.801
9 F	0.677	41 Nb	16.614	73 Ta	57.540
10 Ne	0.848	42 Mo	17.478	74 W	59.305
11 Na	1.041	43 Tc	18.364	75 Re	61.122
12 Mg	1.253	44 Ru	19.278	76 Os	62.989
13 Al	1.486	45 Rh	20.214	77 Ir	64.906
14 Si	1.739	46 Pd	21.176	78 Pt	66.834
15 P	2.012	47 Ag	22.162	79 Au	68.804
16 S	2.306	48 Cd	23.171	80 Hg	70.794
17 Cl	2.621	49 In	24.205	81 Tl	72.856
18 Ar	2.956	50 Sn	25.268	82 Pb	74.976
19 K	3.311	51 Sb	26.355	83 Bi	77.078
20 Ca	3.689	52 Te	27.467	84 Po	79.258
21 Sc	4.088	53 I	28.610	85 At	81.499
22 Ti	4.507	54 Xe	29.802	86 Rn	83.757
23 V	4.948	55 Cs	30.966	87 Fr	86.083
24 Cr	5.410	56 Ba	32.191	88 Ra	88.480
25 Mn	5.893	57 La	33.435	89 Ac	90.880
26 Fe	6.398	58 Ce	34.711	90 Th	93.366
27 Co	6.923	59 Pr	36.025	91 Pa	95.870
28 Ni	7.470	60 Nd	37.352	92 U	98.417
29 Cu	8.040	61 Pm	38.647	93 Np	100.780
30 Zn	8.628	62 Sm	40.127	94 Pu	103.300
31 Ga	9.240	63 Eu	41.527	95 Am	105.949
32 Ge	9.873	64 Gd	42.985	96 Cm	108.737
33 As	10.528	65 Tb	44.468	97 Bk	111.676
34 Se	11.204	66 Dy	45.989	98 Cf	114.778

K-Series Relative Peak Intensities

	$18 < Z < 59$
Kα	100
Kβ	10-20

At Z less than 18, the Kβ decreases in height, and becomes closer to, and is eventually not resolved from the Kα. At Z greater than 59, the Kβ increases in size and sub-peaks can be seen.

M-Series Relative Peak Intensities

	$57 < Z < 70$	$70 < Z$
Mz	140 - 13	6 - 4½
Mα	100	100
Mβ	0 - 45	45 - 57
Mγ	20 - 3	3½ - 1
M_2N_4	0 - 2	0 - 2

At Z less than 57, the Mα becomes 0% and the Mz is the dominant peak. The Mγ becomes 0 at $Z < 50$.

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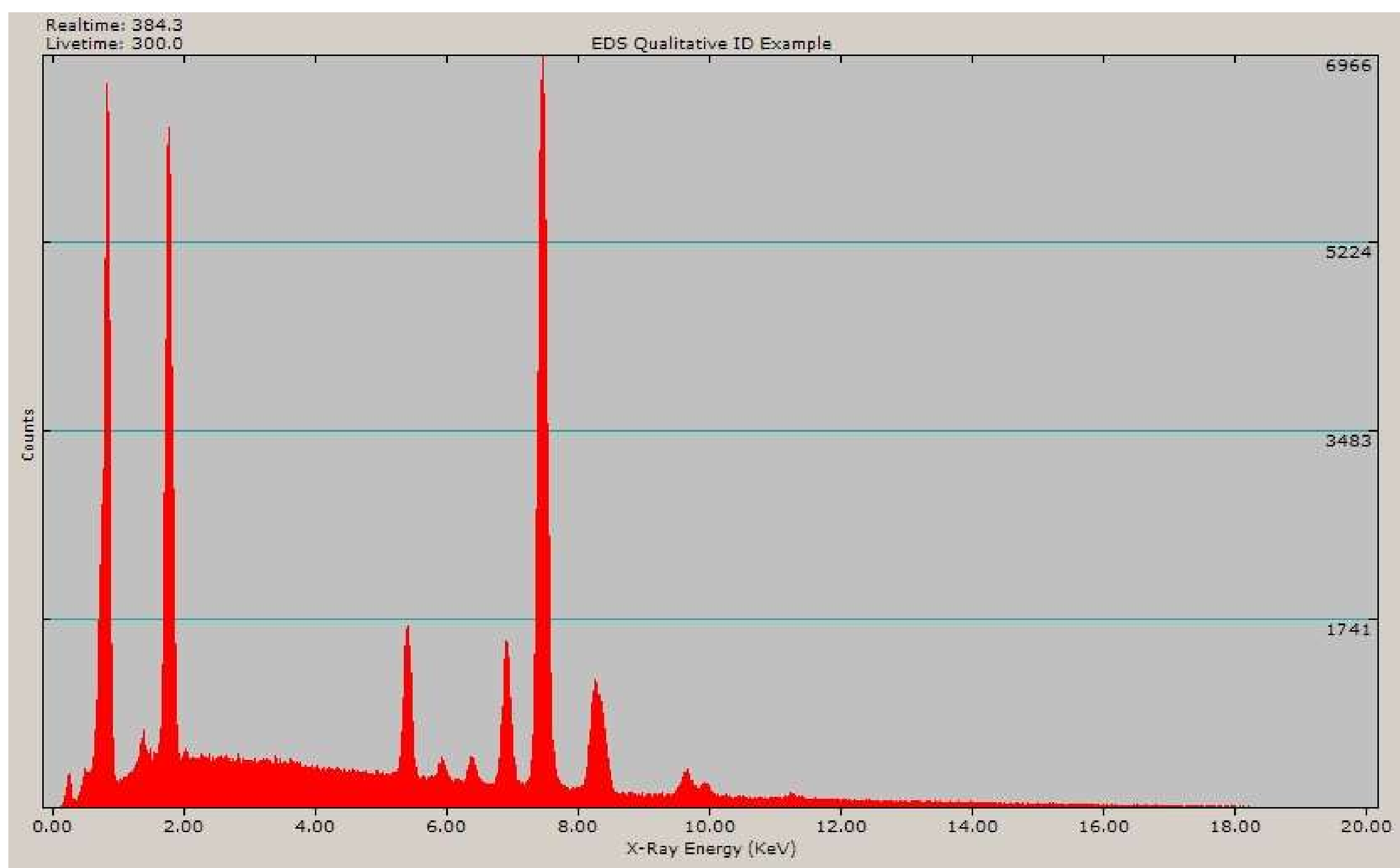
EDS Qualitative Analysis Example



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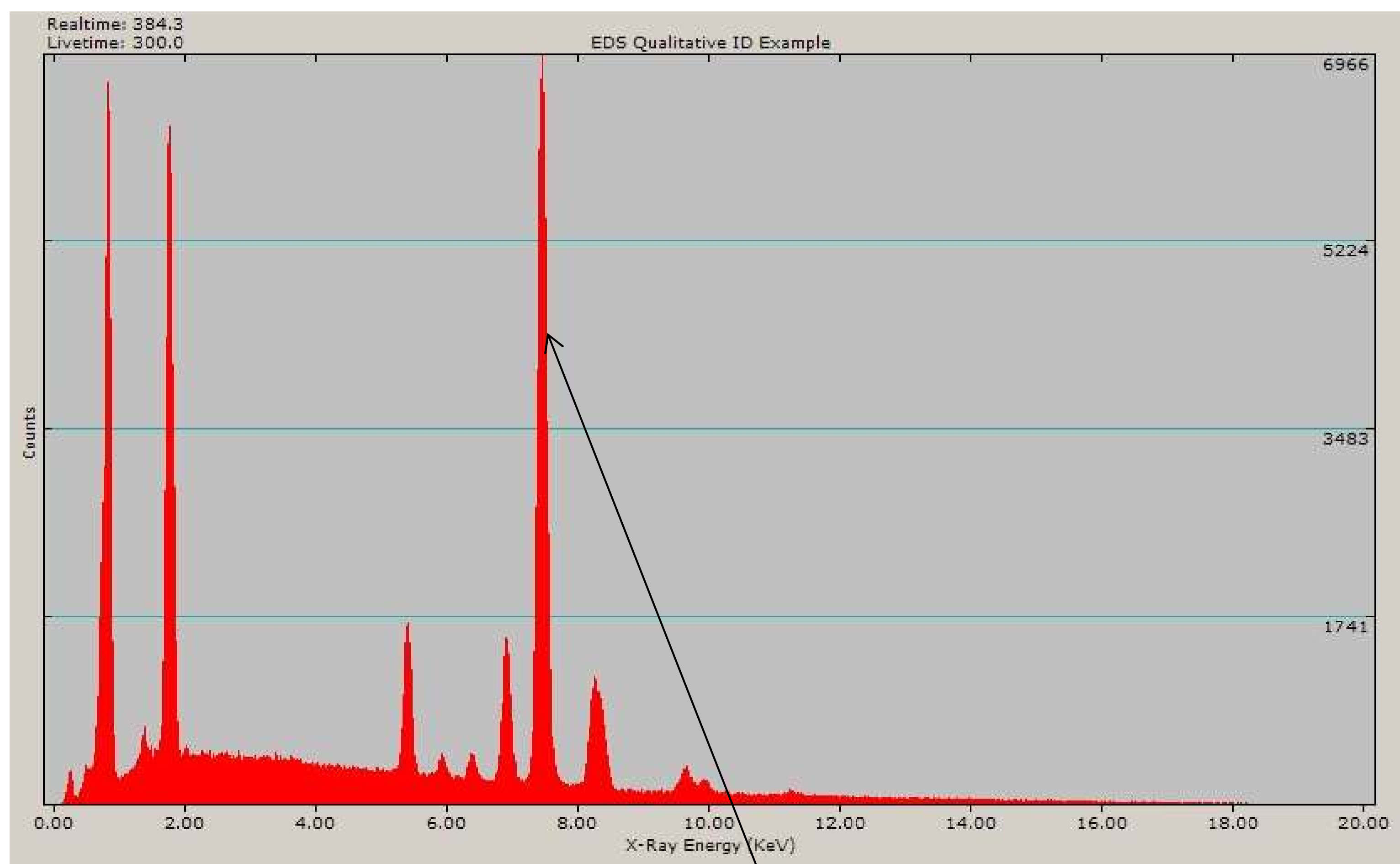
- Example of an unknown metallic sample for analysis
- EDS spectrum collected at 30keV
- Step by step analysis using an X-ray energy slide rule (and X-ray line markers in the software!)

EDS Qualitative Analysis Example



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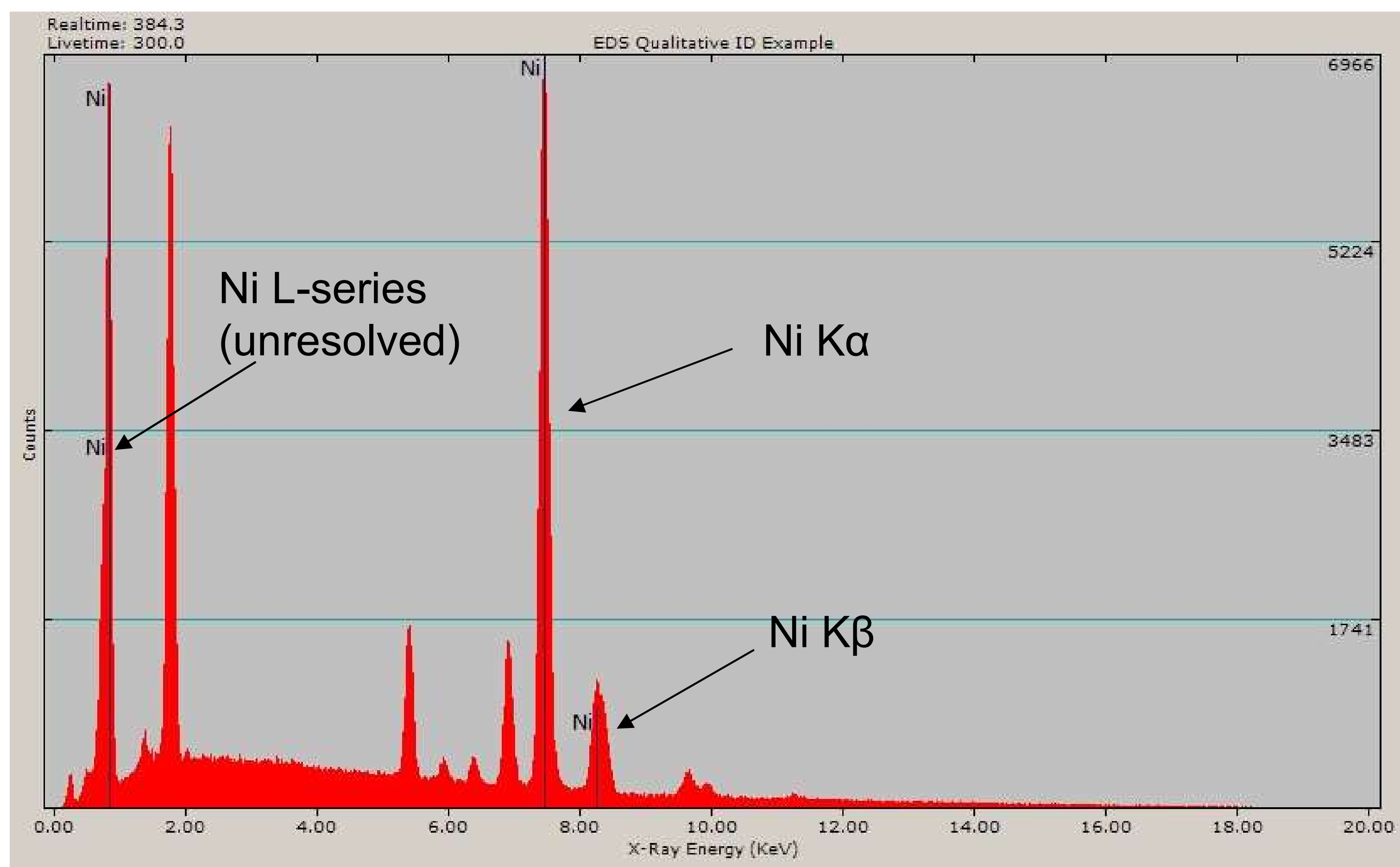
- Step 1: Identify the tallest, highest energy peak
- Energy ~ 7.5 keV = Ni Kα peak

EDS Qualitative Analysis Example



- Step 1 continued: Mark ***ALL*** Ni peaks
  - Kα, Kβ, and the L-series
  - Notice that the Ni Kβ peak looks a little wide and tall and offset

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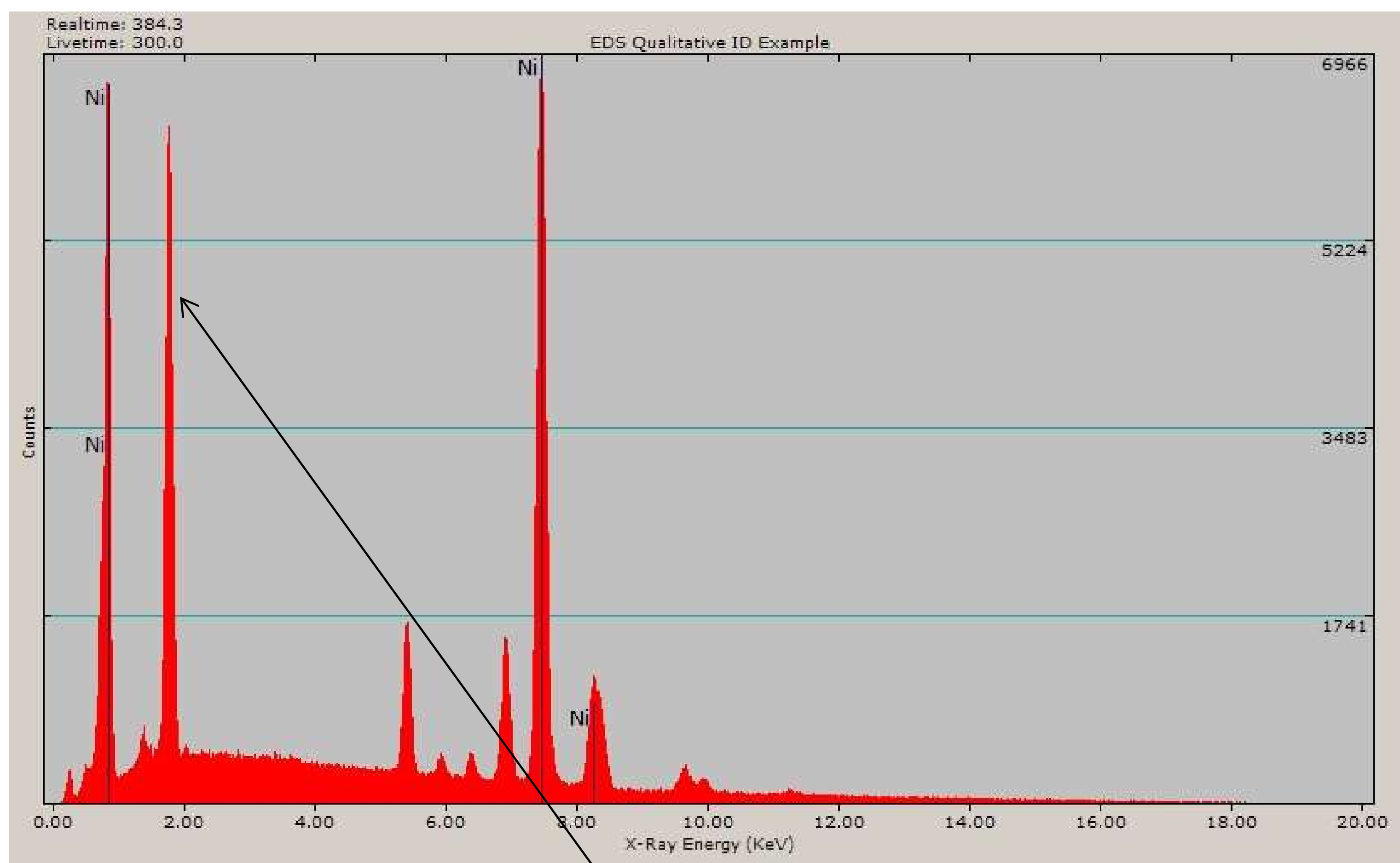
EDS Qualitative Analysis Example



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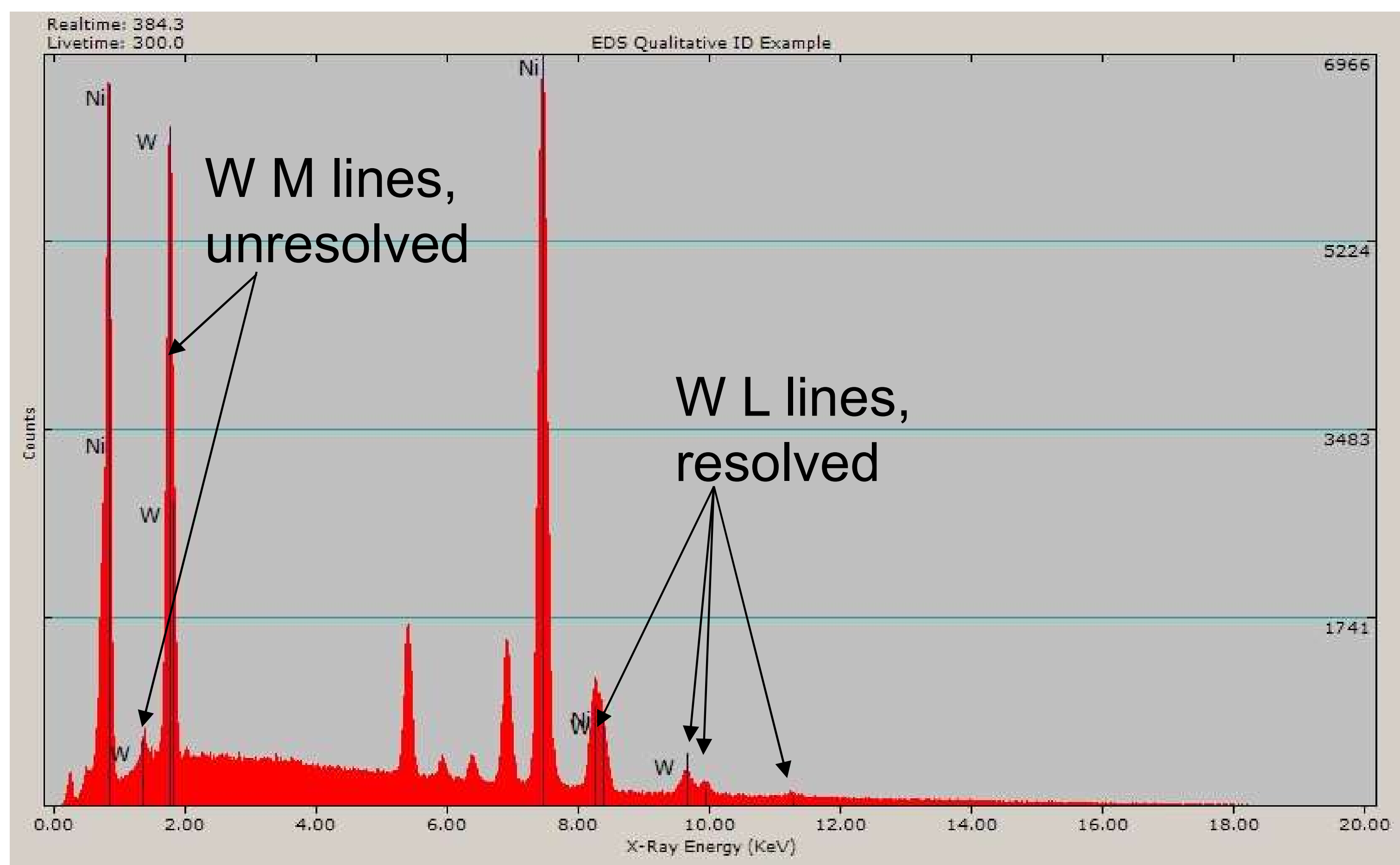
- Step 2: Identify the next tallest, highest energy, unmarked peak
- Energy ~ 1.7 keV = W Mα peak

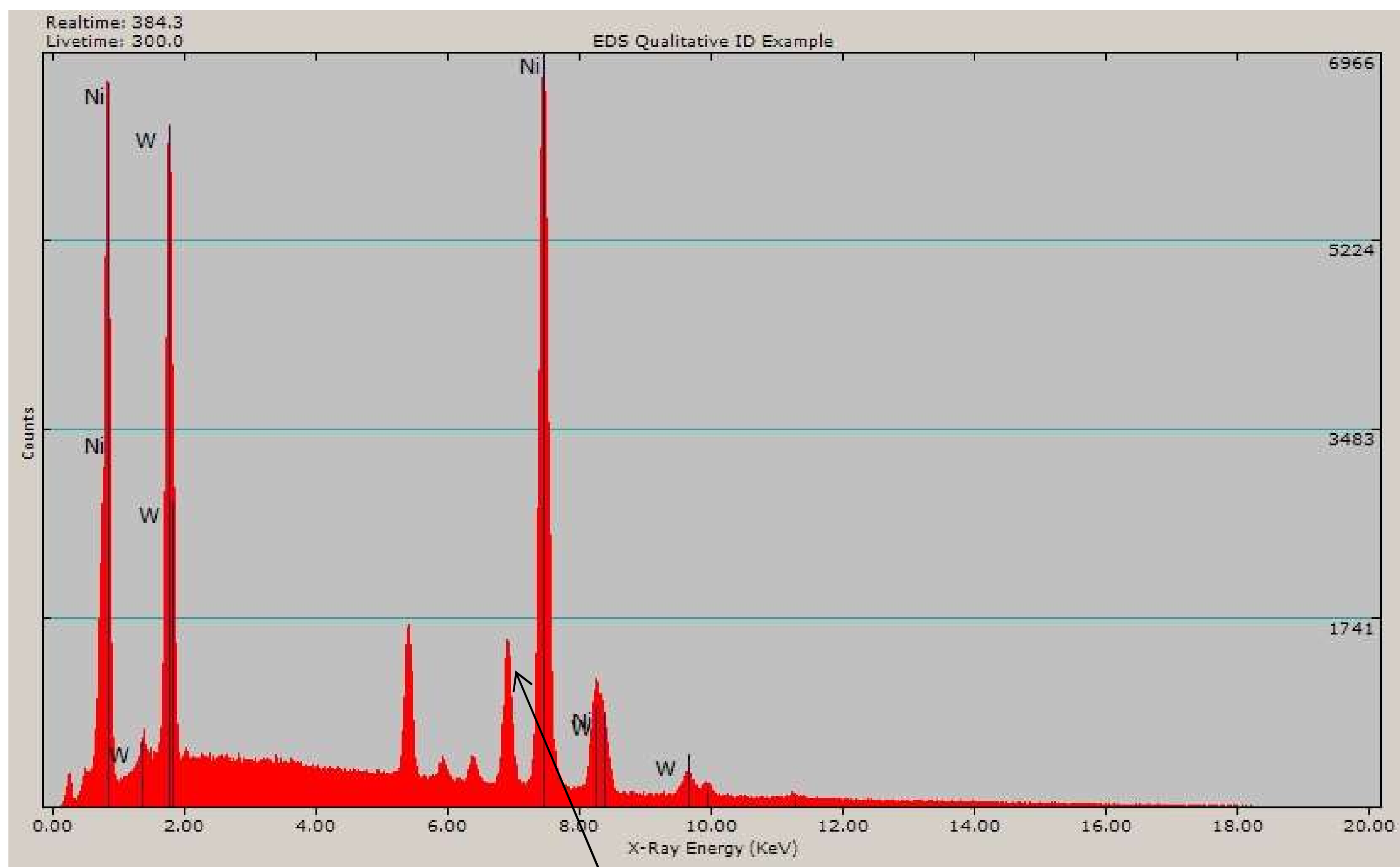
EDS Qualitative Analysis Example



C.Mooney

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

EDS Qualitative Analysis Example



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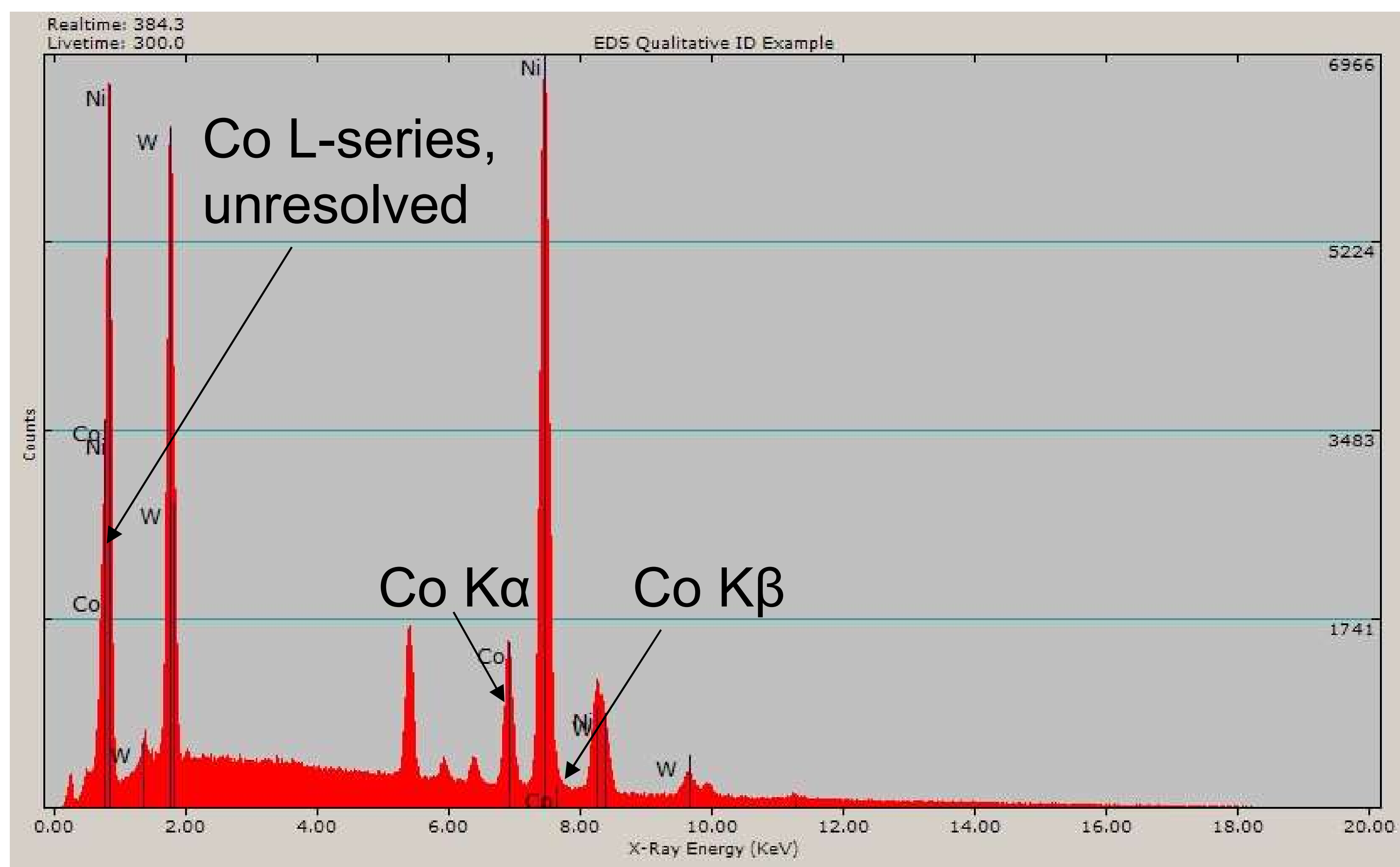
- Step 3: Identify the next tallest, highest energy peak
- Energy ~ 6.9 keV = Co Kα

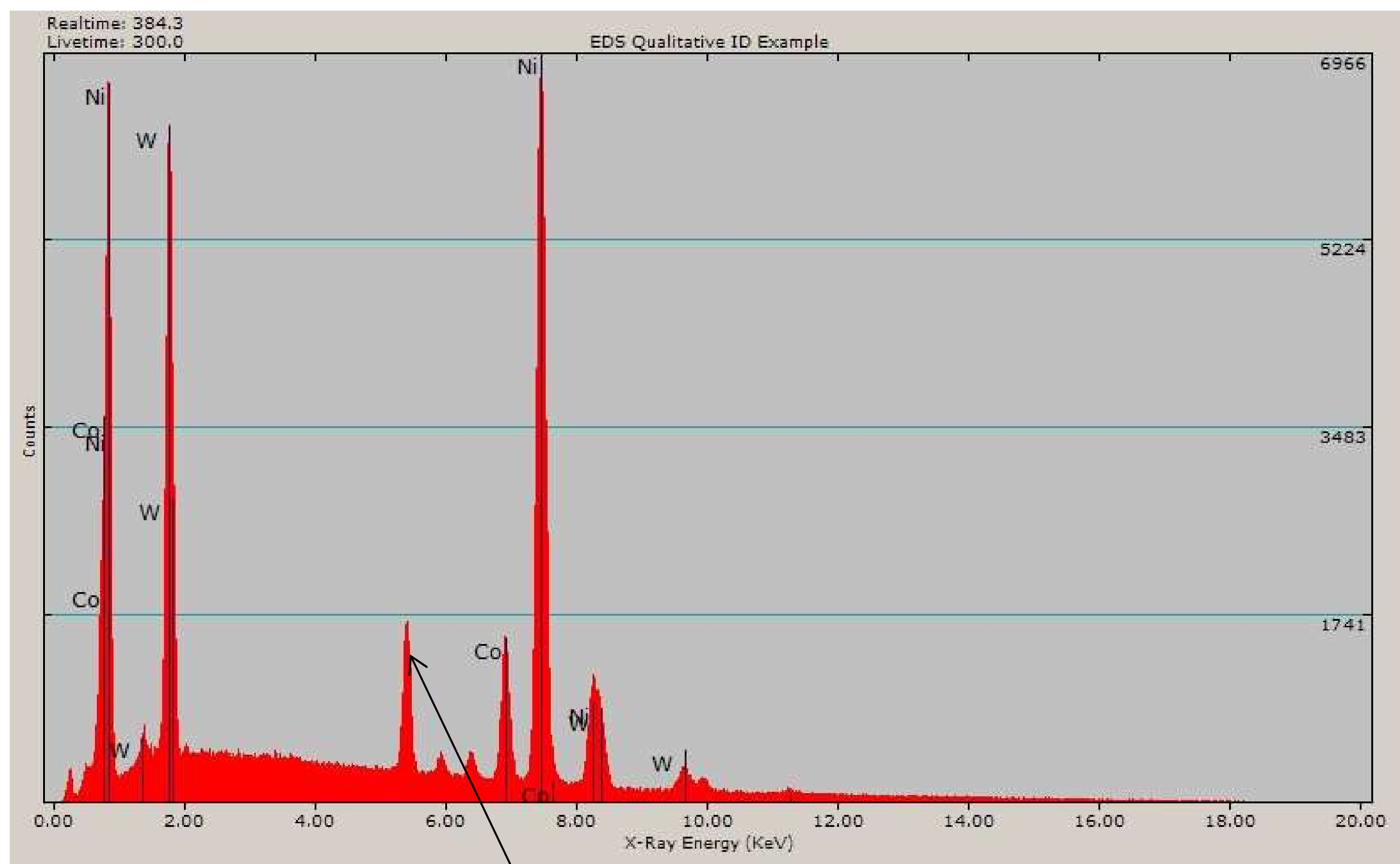
EDS Qualitative Analysis Example



C.Mooney

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

EDS Qualitative Analysis Example



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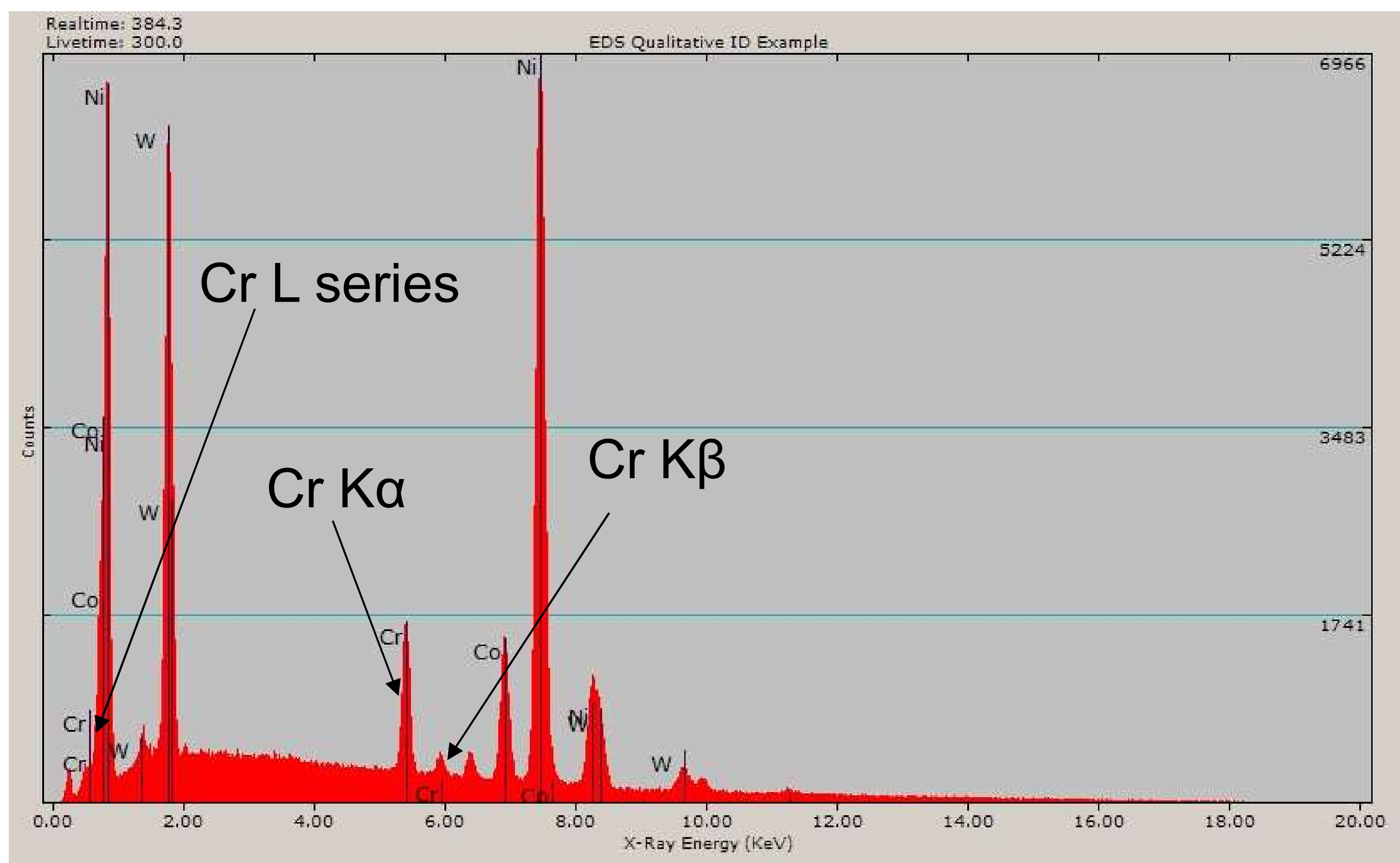
- Step 4: Identify tallest, highest energy, unmarked peak
- Energy ~ 5.4 keV = Cr Kα

EDS Qualitative Analysis Example



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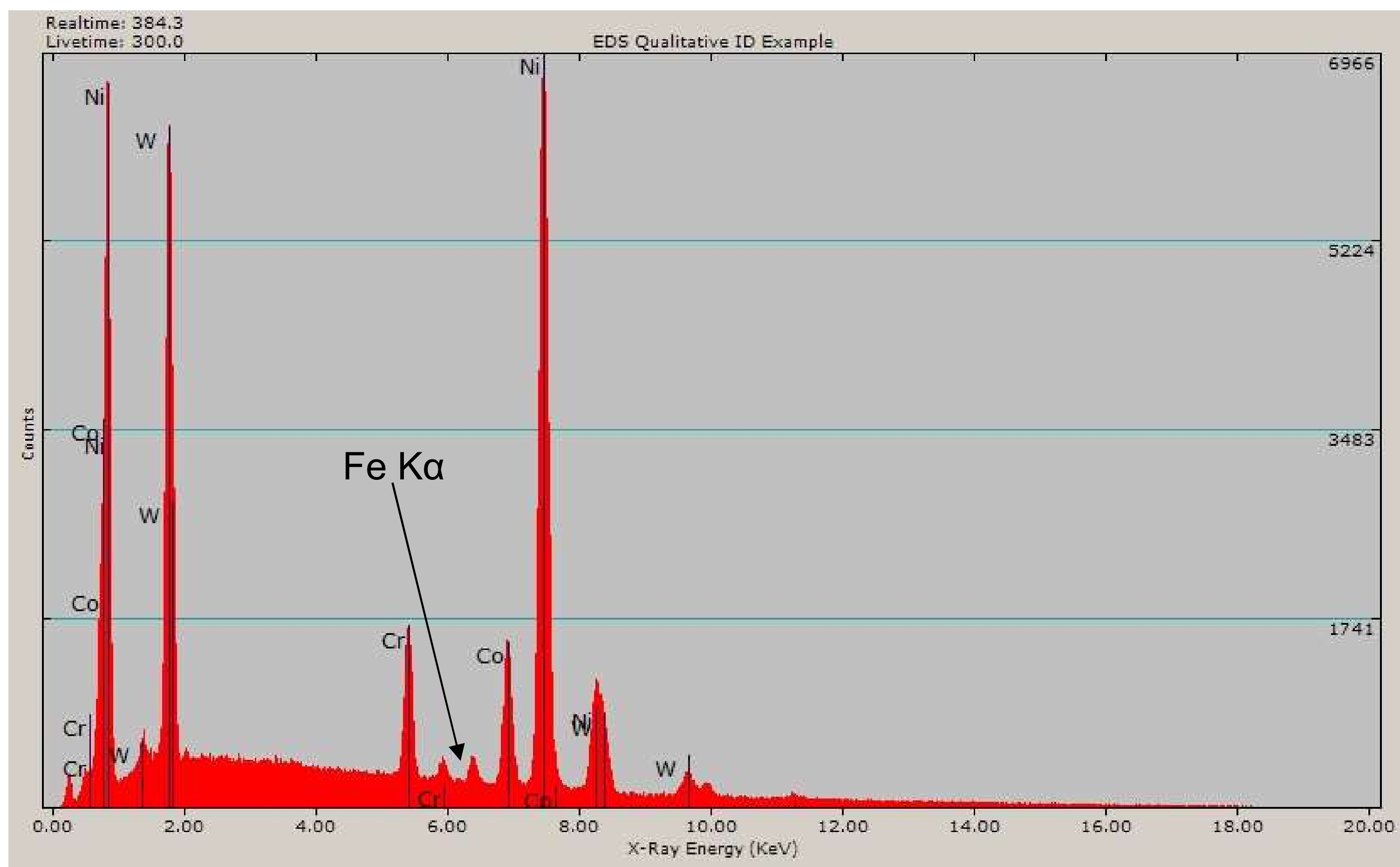
- Step 4 continued: Mark ALL Cr peaks

EDS Qualitative Analysis Example



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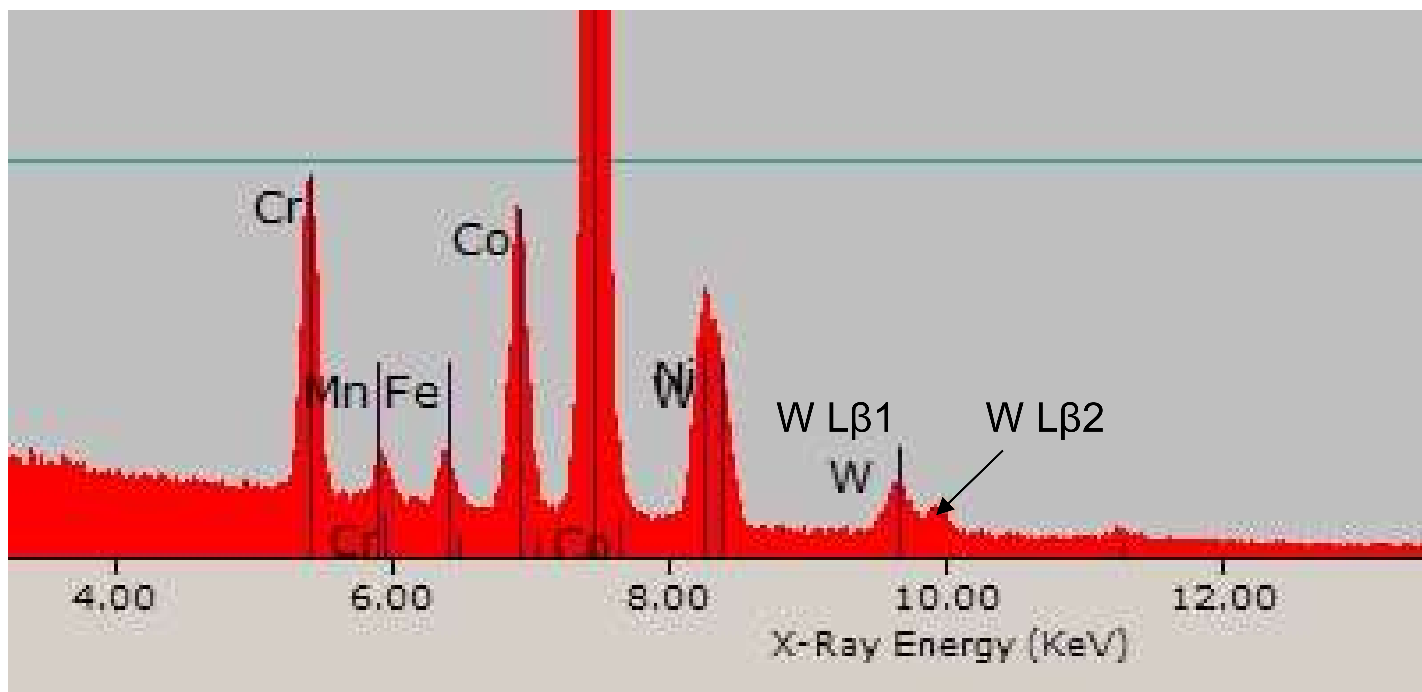
- Step 5: Identify remaining peak
- Energy ~ 6.0 keV = Fe Kα
- Mark all Fe peaks

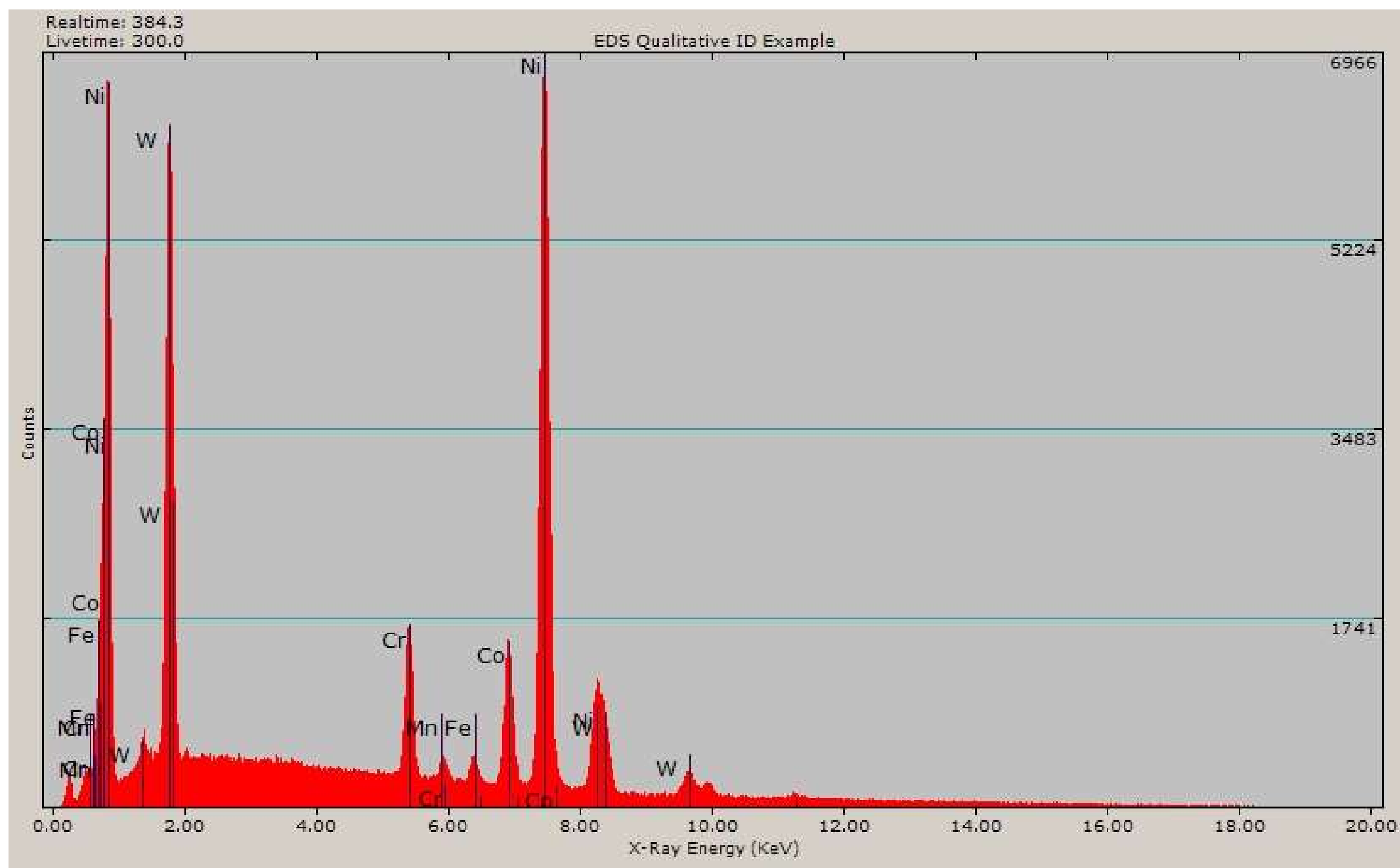
EDS Qualitative Analysis Example



C.Mooney

- Look for discrepancies
- Cr Kβ can't account for the peak height and position
- Mn Kα 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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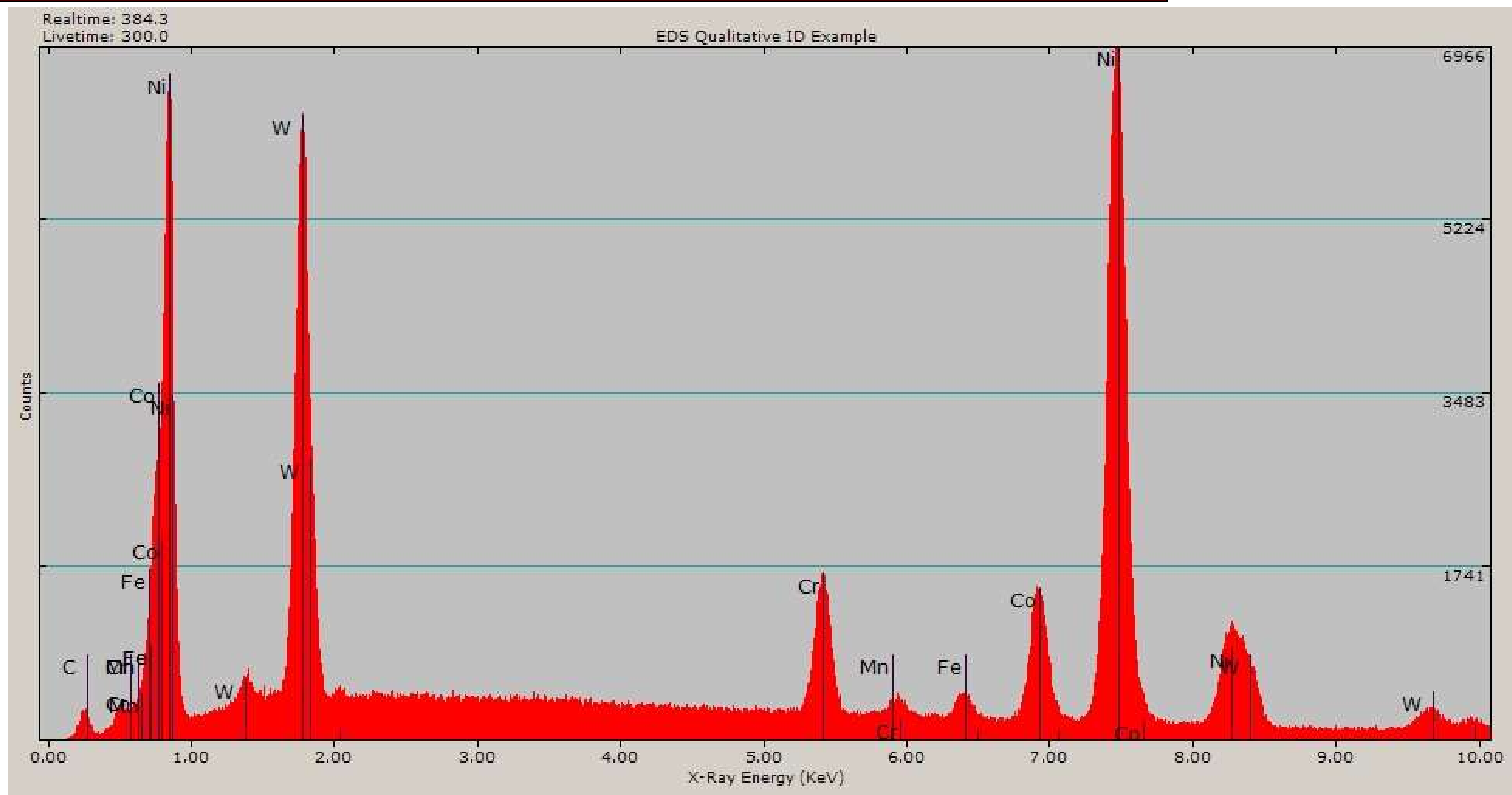
- Finished! All peaks are accounted for!
- The unknown alloy is composed of Ni, W, Co, Cr, Fe, and Mn

EDS Qualitative Analysis Example



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- 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!)

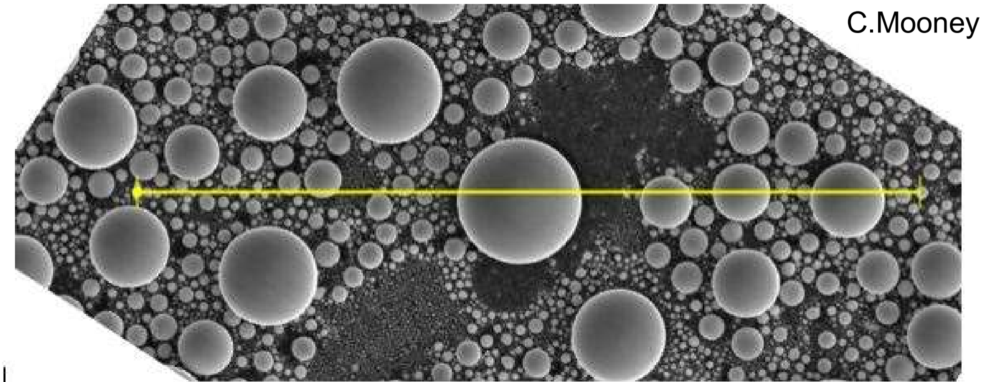
EDS Artifacts

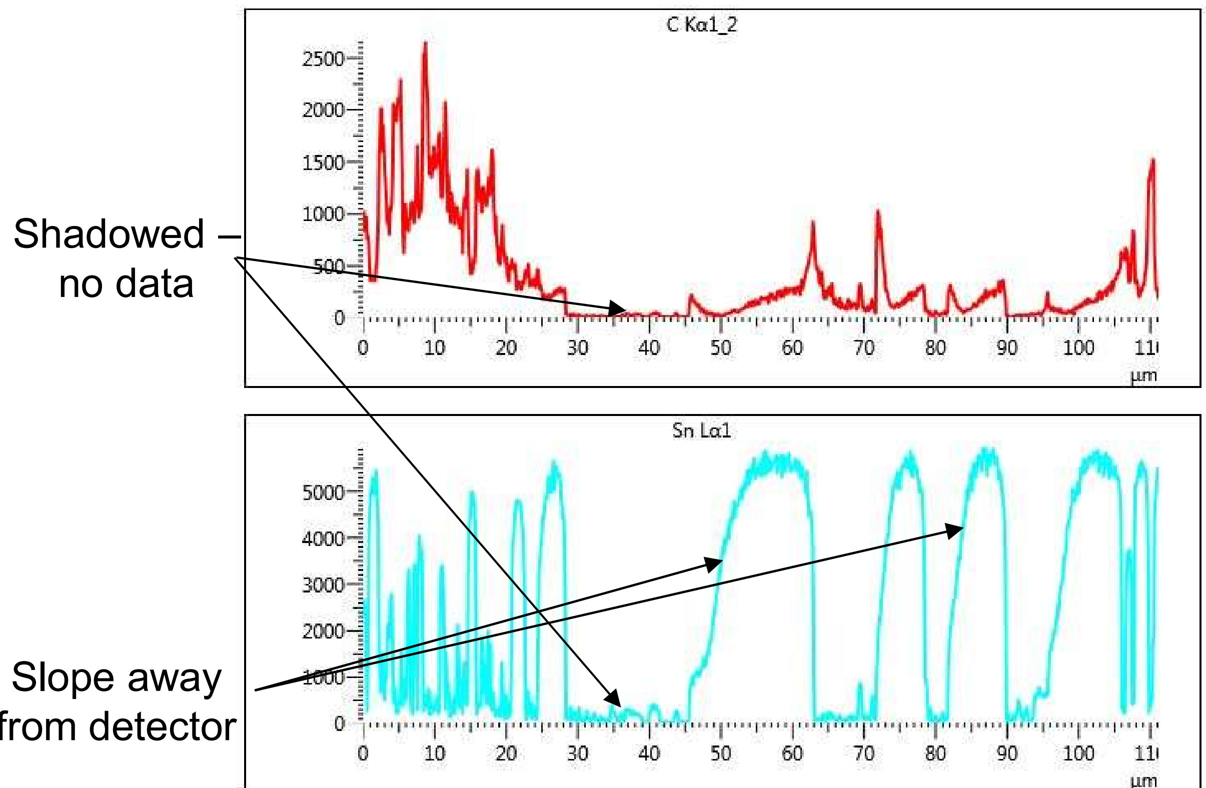
- Sum peaks
  - If more than one X-ray strikes the detector at the same time, we get a peak at the sum of the two X-rays
  - This can be avoided by not overdriving the detector and not generally a problem with modern detectors

- Escape peaks
  - If the incoming X-ray has enough energy to cause the Si detector to fluoresce (> 1.9 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

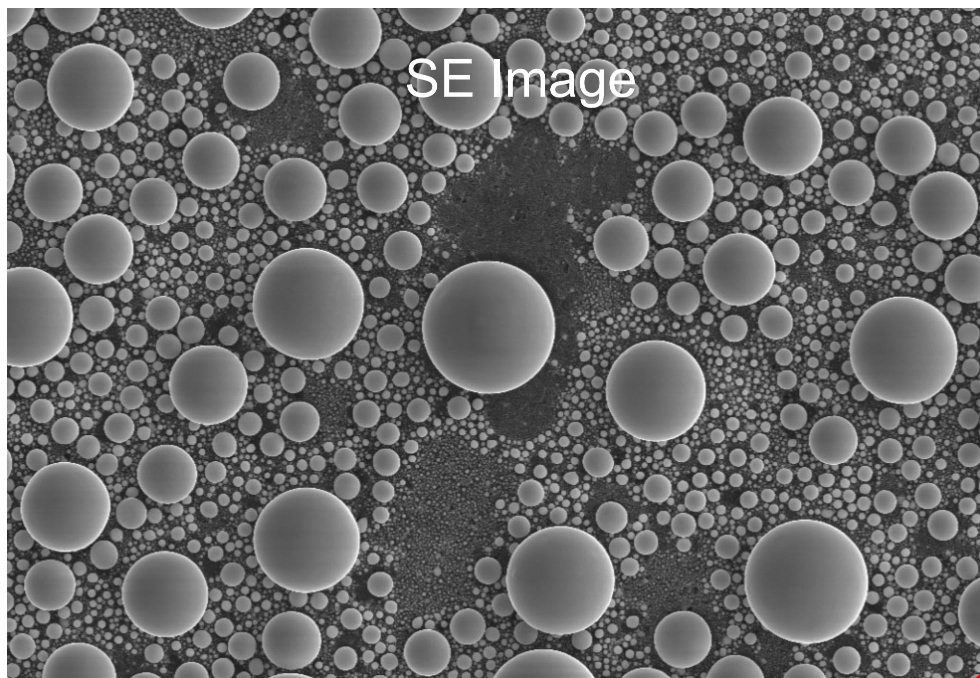
X-ray Line Scan

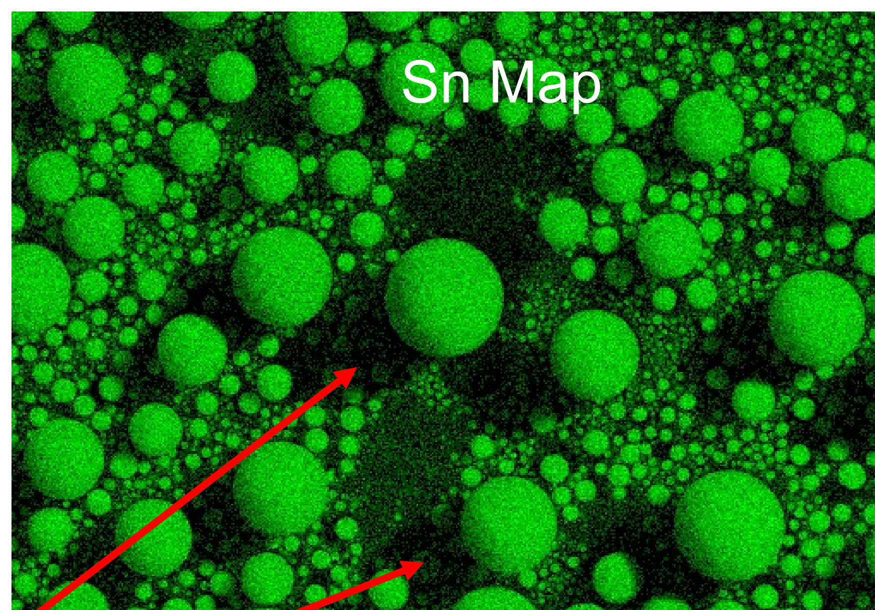
- Instead of an area scan, a line scan can be generated
- This was smart when detectors were slow and mapping took a long time, but mapping with an SDD is faster than line scans used to be with a SiLi detector
- Note that shadowing effects give the X-ray data a slope away from the detector

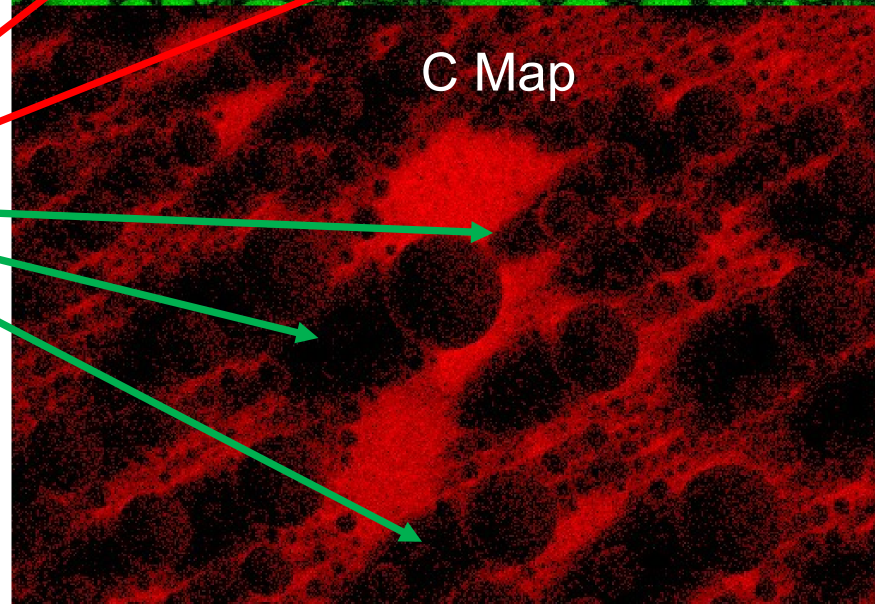




X-ray Map







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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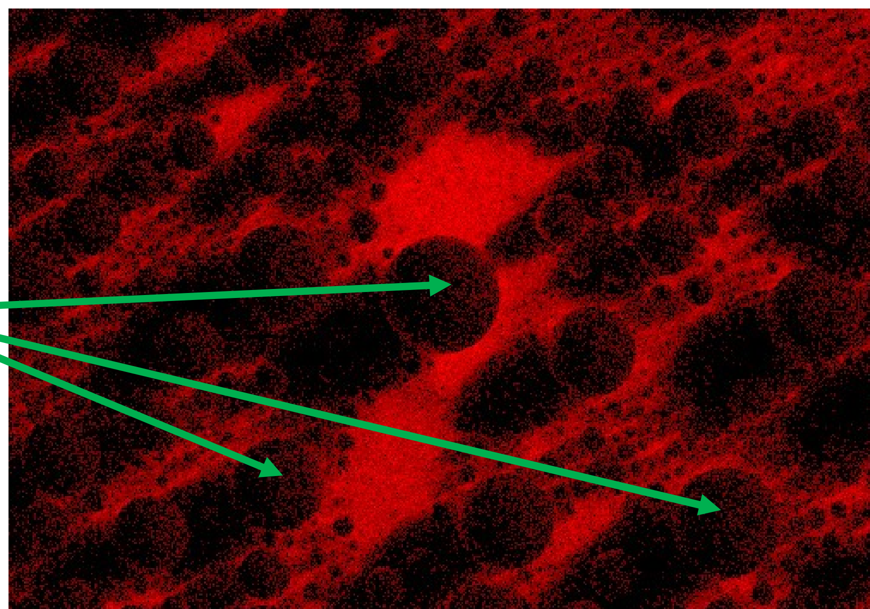
Dangers of X-ray Mapping

- 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…

Only a Taste of SEM…

For More Information see:

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