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Theory and Operation of a Scanning Electron Microscope Equipped with an Energy Dispersive Spectrometer

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Lecture Goals

What I hope to achieve today is the following…

- **Provide some basics concerning SEM operation and capabilities.**
- **Briefly discuss the most common signals you have available to you using an SEM.**
- **Show some examples of how an SEM can be used and discuss things to watch for that may cause your analysis to be flawed.**

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Outline

I. Introduction

- Basics of Operation

II. Specimen Electron Interactions

- Backscattered Electrons
- Secondary Electrons
- X-rays

III. Examples of Signal Detection and Analysis

- Imaging (Backscattered and Secondary)
- Chemical Analysis (X-rays)

IV. Summary

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Optical vs SEM

SEM has numerous advantages over optical microscopy…..

Magnification (1000x vs 100,000x)

Resolution (0.2 µm vs 0.005 µm)

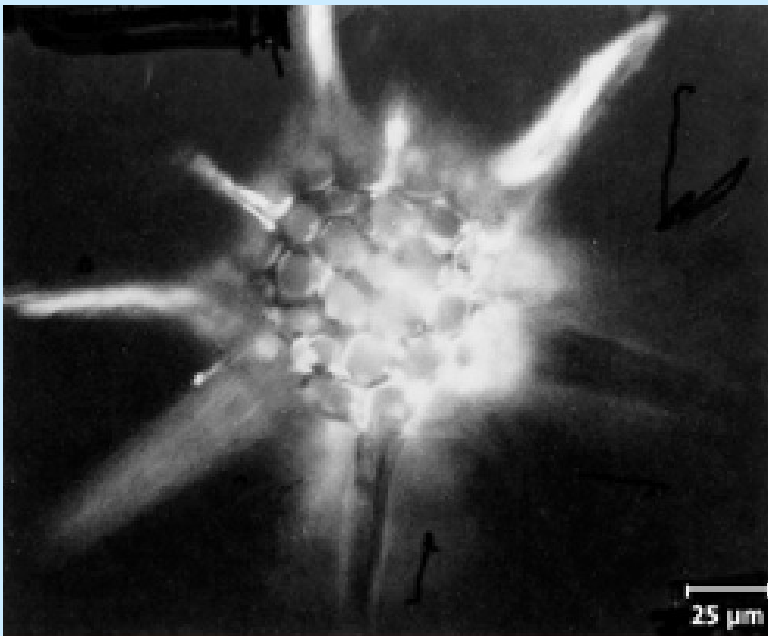
Composition Information (limited vs extensive)

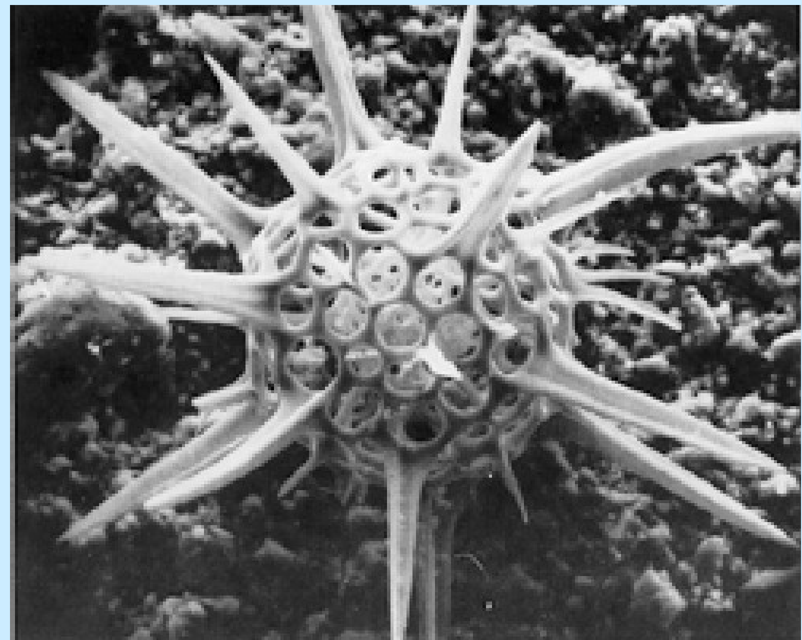
Sample Preparation (flat vs varied)

Depth of Focus (15 µm vs 4 mm)

Example

Pollen imaged using optics (left) and electrons (right).





Even at relatively low magnifications and resolutions the Depth of Field (DF) for optical systems is low.

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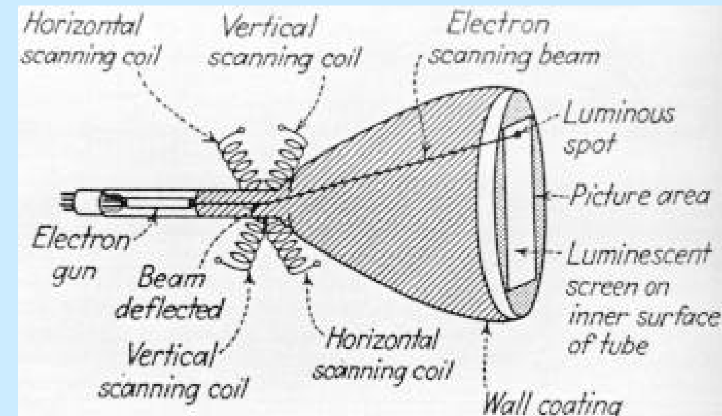
Basics of Operation

In operation, the SEM is really nothing more than a very expensive Black & White Television. Consider…

Both have an electron gun in a vacuum.

Both scan in a serial manner.

The image depends on the quality / strength of the incoming signal.



For a TV, the quality of the image depends on the strength of the signal coming from the station. For SEM, it depends on the signal collected by detectors, which depends on operation.

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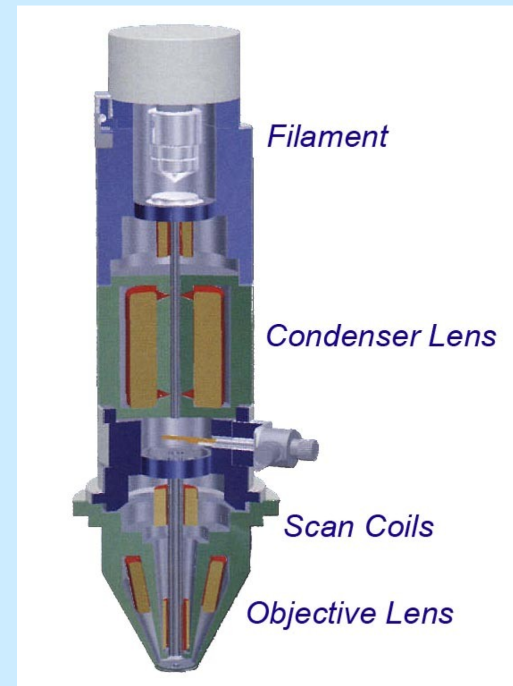
Basics of Operation

The SEM is analogous to a conventional optical microscope in terms of lenses...

In the SEM we ...

- **Heat a filament to get electrons.**
- **Use electromagnetic lenses to produce a beam.**
- **Using scan coils to direct the beam across the sample.**
- **Collect the emitted signals to form an image.**



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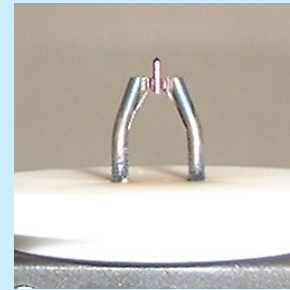
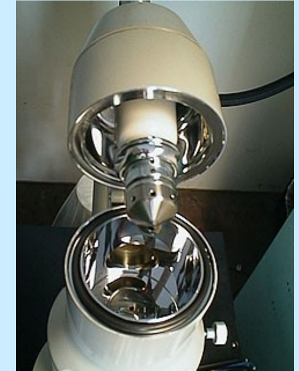
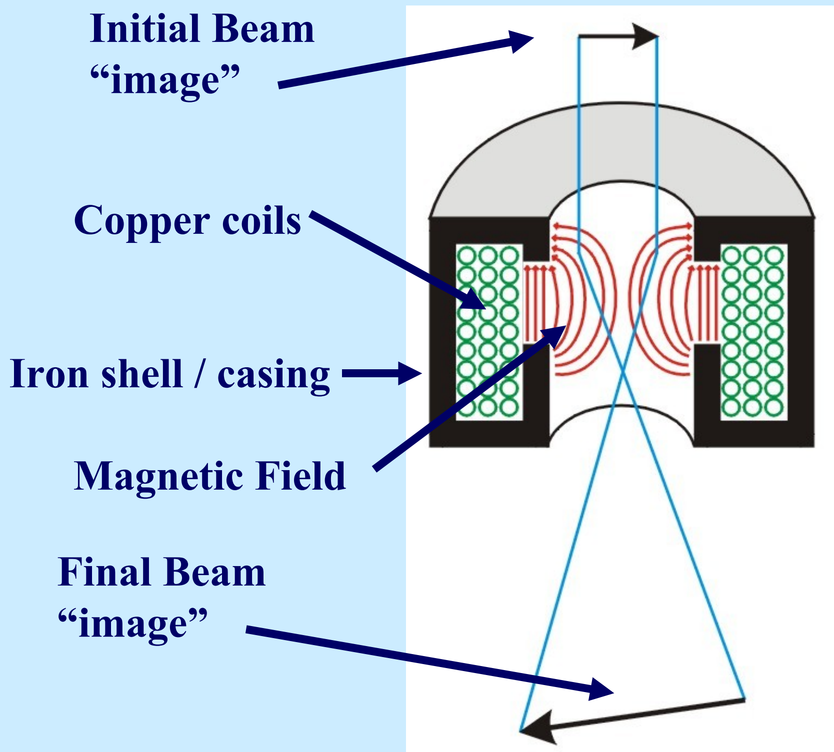
Hardware

The filament is usually tungsten or Lanthanum Hexaboride (LaB_6).

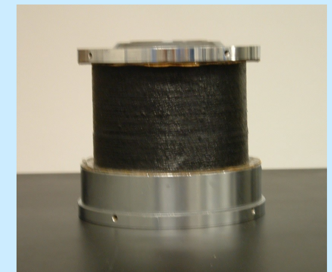




Typical Electron Gun



The lenses are electromagnetic, i.e. coils of Cu wire wrapped around an Fe shell. Changing the current to the Cu changes the lens strength.



View of an actual lens. Cu wires covered with black insulator

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Control Knobs

The Condenser Lens affects the resolution and signal strength. It is usually labeled *Spot Size*, *Probe Current,* or *Resolution*.

The Objective Lens is always set to focus the beam on the surface of the sample and is labeled *Focus*.

Scan Coils move the beam left/right and up/down to create a square raster or a line scan. They control *Magnification*.

Stigmation Coils enable you to make the beam round and get a sharper image and are labeled *Stigmator.*

The Objective Aperture defines the angular spread of the beam and determines how many electrons hit your sample.

The Complete SEM Column

When you put everything together you have an SEM!

Now we need to ask: How do I get an image now that I have e^-s hitting the sample?

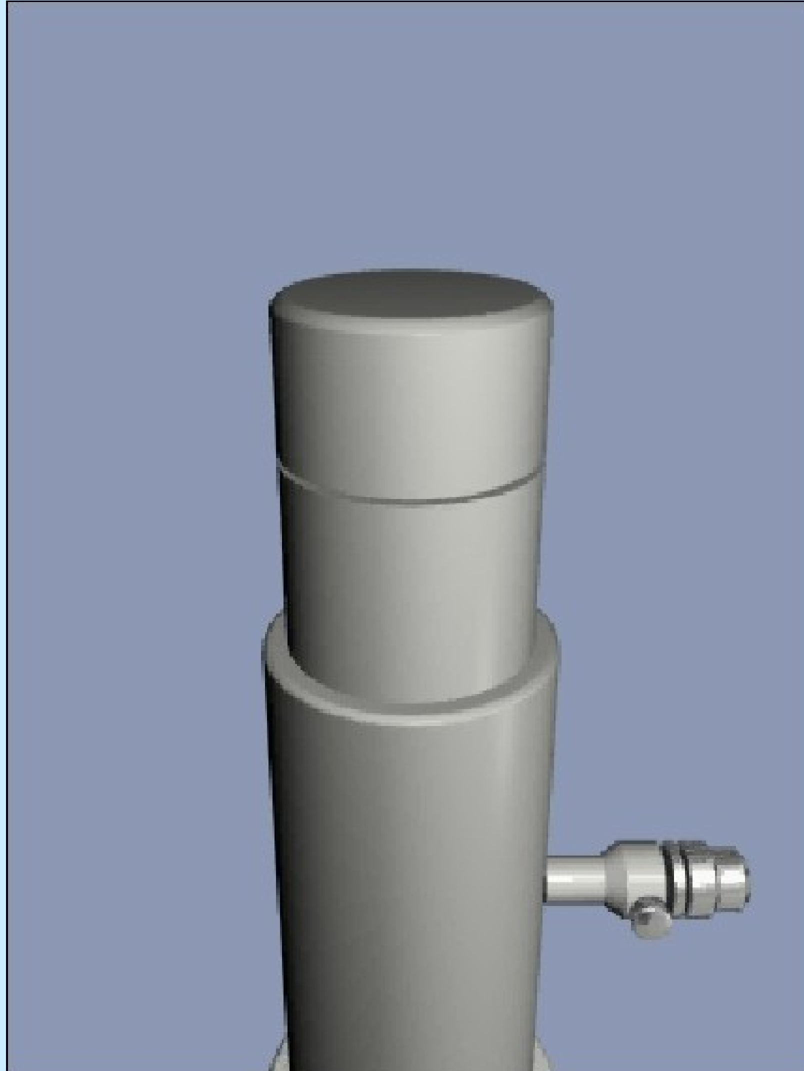
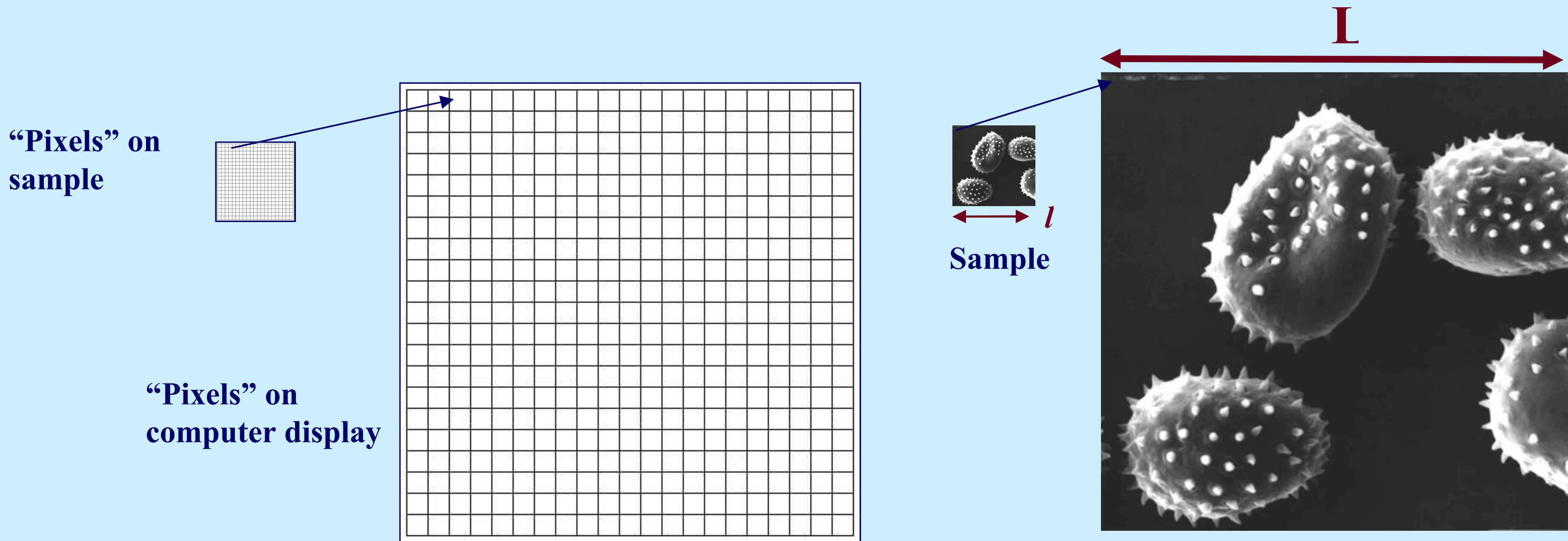


Image Formation

Image formation is achieved by transferring information pixel by pixel, from sample to the display.

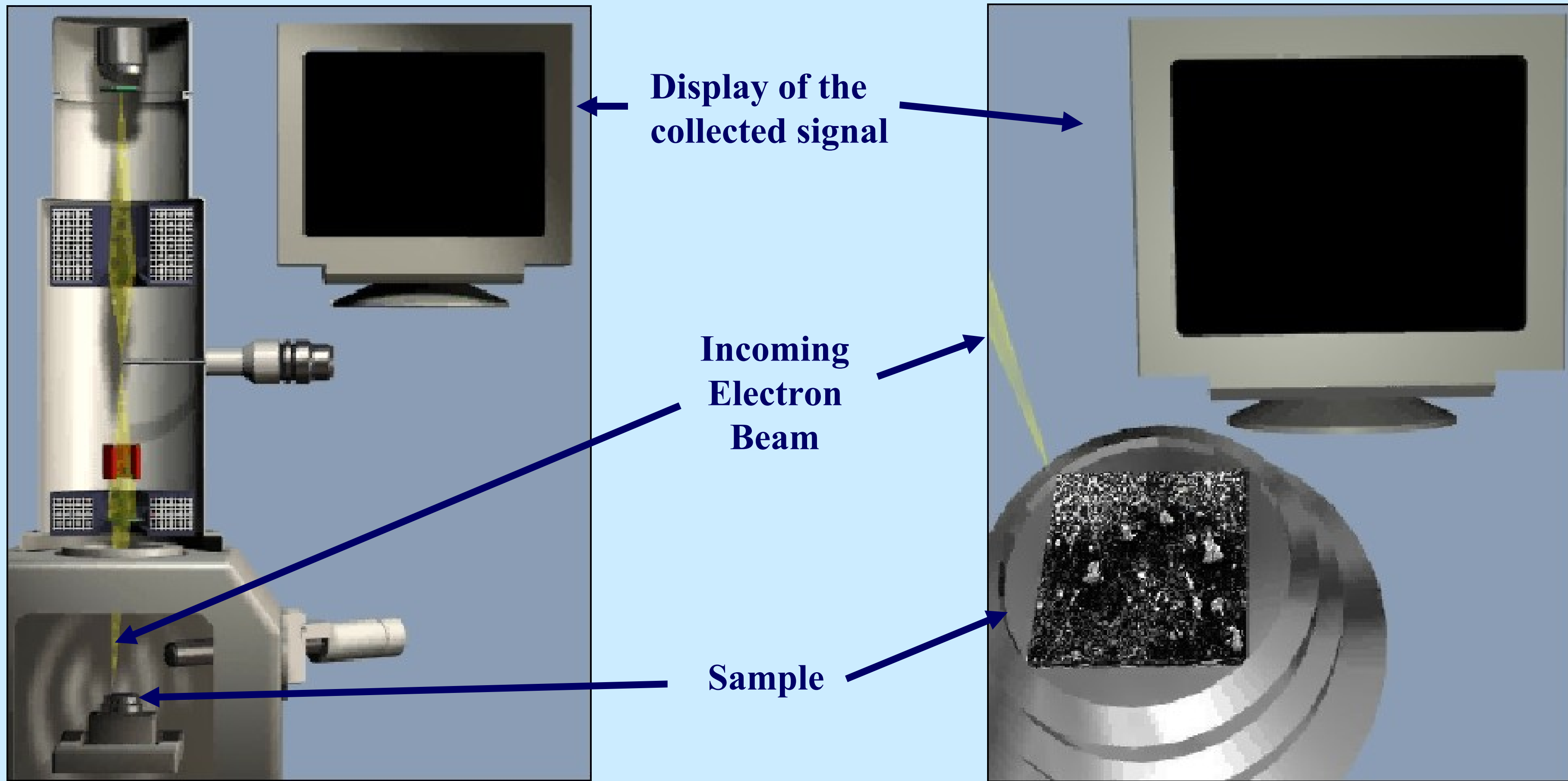


The number of "pixels" is equal for the sample and the display.

Image on computer display screen reflects sample.

$$\text{Magnification} = \frac{\text{Scan Length on Display}}{\text{Scan Length on Sample}} = \frac{L}{l}$$

Image Formation

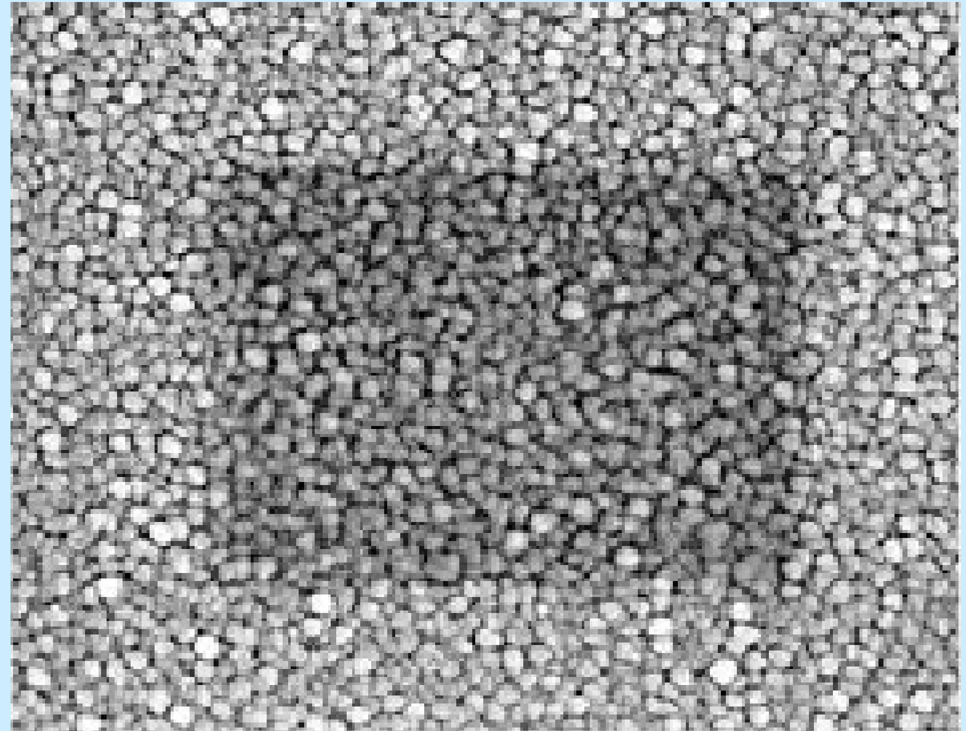


I increase magnification by decreasing the scan size on the sample.

Example

How this all works is usually easily revealed by the contamination that appears on the surface due to heating of the beam.

Contamination area shows size of scan when at a higher magnification.



Definitions

How I set up my lenses and how much of the electron beam I allow to hit my sample by inserting different sized apertures in the column determine my operating conditions, such as….

Resolution: Ability to resolve two closely spaced points.

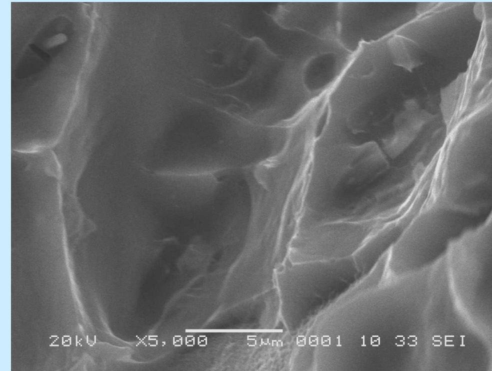
Depth of Field: Ability to clearly image features at different distances from the lens or viewer.

Signal Strength: Number of electrons / x-rays are hitting your sample and / or your detectors.

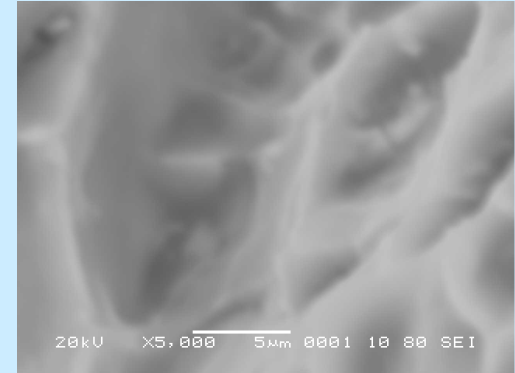
Working Distance: The distance the sample is from your objective lens. Since the objective lens is always focused on the sample, you can't change one without the other.

Operation

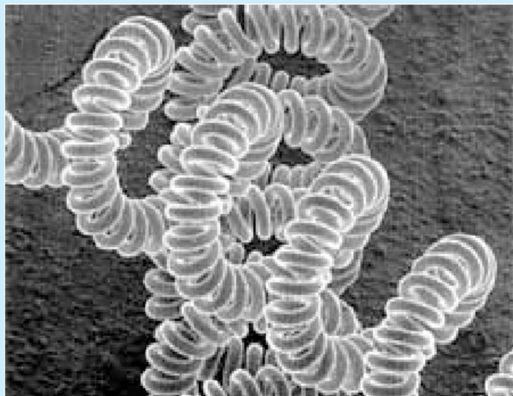
Best resolution requires a small beam (strong lenses, weak signal, sample close to the lens, i.e. short working distance).



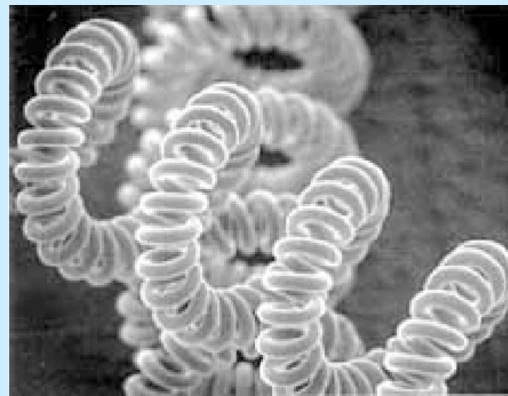
Good resolution, weak signal



High signal, poor resolution



Good depth of field



Poor depth of field

Best depth of field requires a narrow beam (sample far from the lens, small aperture).

High signal requires a large diameter beam (weaker lenses, poorer resolution).

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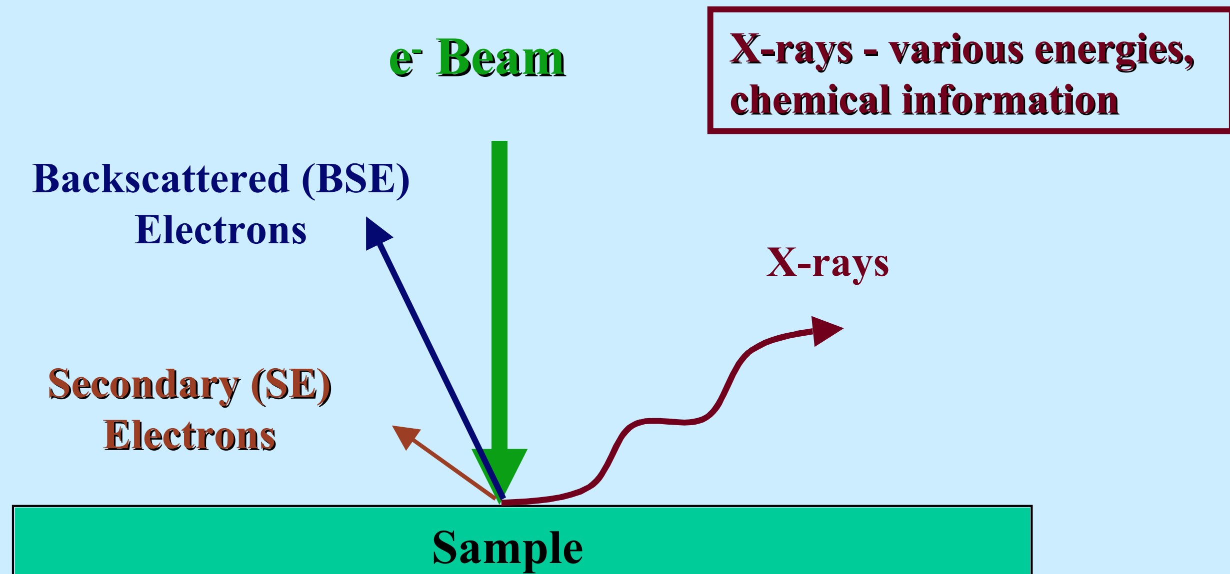
Electron / Specimen Interactions

When an electron strikes a sample a large number of signals is emitted. The most commonly signals are secondary electrons (SE), backscattered electrons (BSE), and x-rays.

BSE electrons - high energy, come off at high angle, atomic number dependence, directional

SE electrons - low energy, "sucked" in to the detector, depend on beam / specimen angle

X-rays - various energies, chemical information



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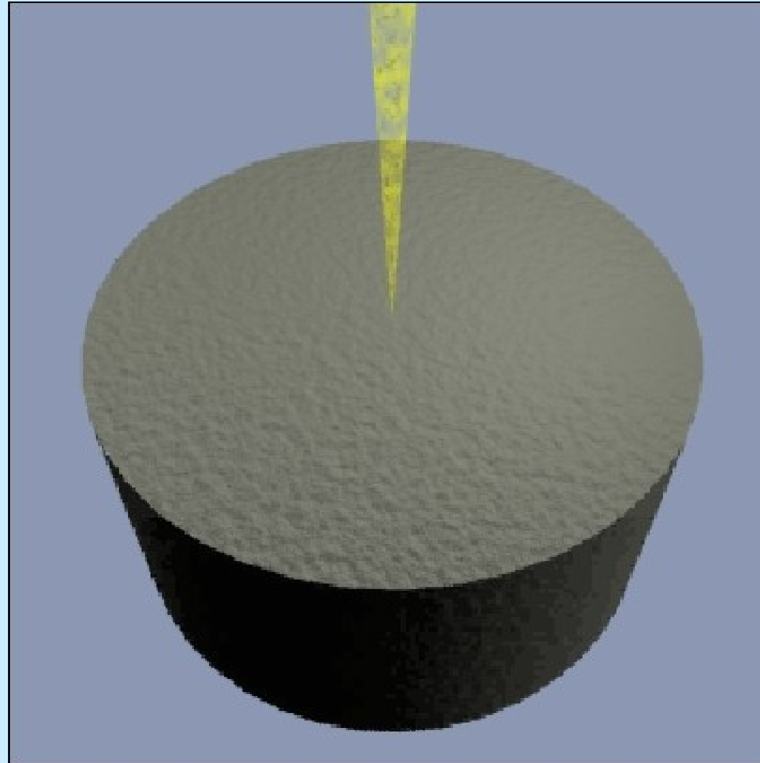
Interaction Volume

When the electron beam strikes a sample, the interactions that result occur over a volume much larger than the size of the beam. This is called the Interaction Volume.

Electron beam strikes sample and starts to interact with the atoms. This interaction can be modeled.



The shape of the interaction volume depends on such things as accelerating voltage, composition of the sample, and angle the beam enters.

Interaction volume varies from a hemisphere to a bottle shape.

Signal Generation

The signals come from various depths within the interaction volume in the sample.

Secondary Electrons - 100Å

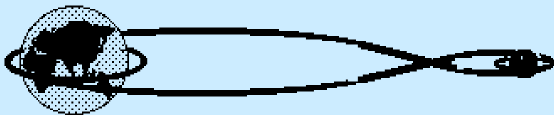
Characteristic X-rays (K & L) -

Backscattered Electrons

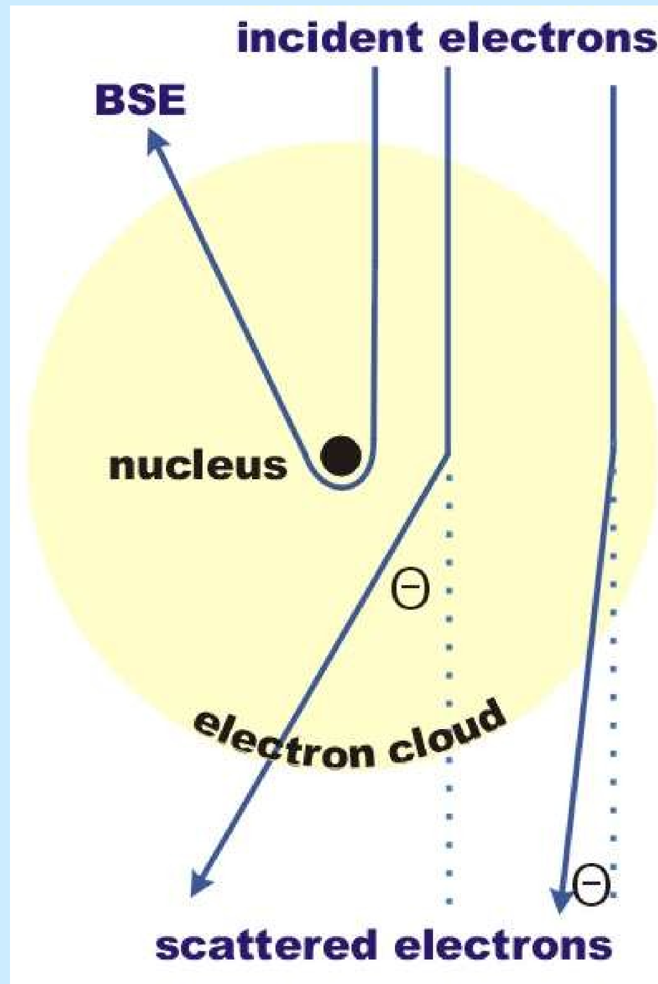
X-rays and BSE can come from several microns below surface depending upon sample and accelerating voltage.

Electron Scattering

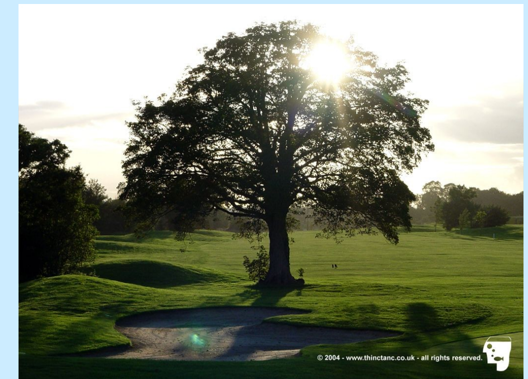
Backscattered electrons come from elastic scattering of the incident electrons around the nucleus.



Best analogy - spacecraft using gravity to "slingshot" around planets.



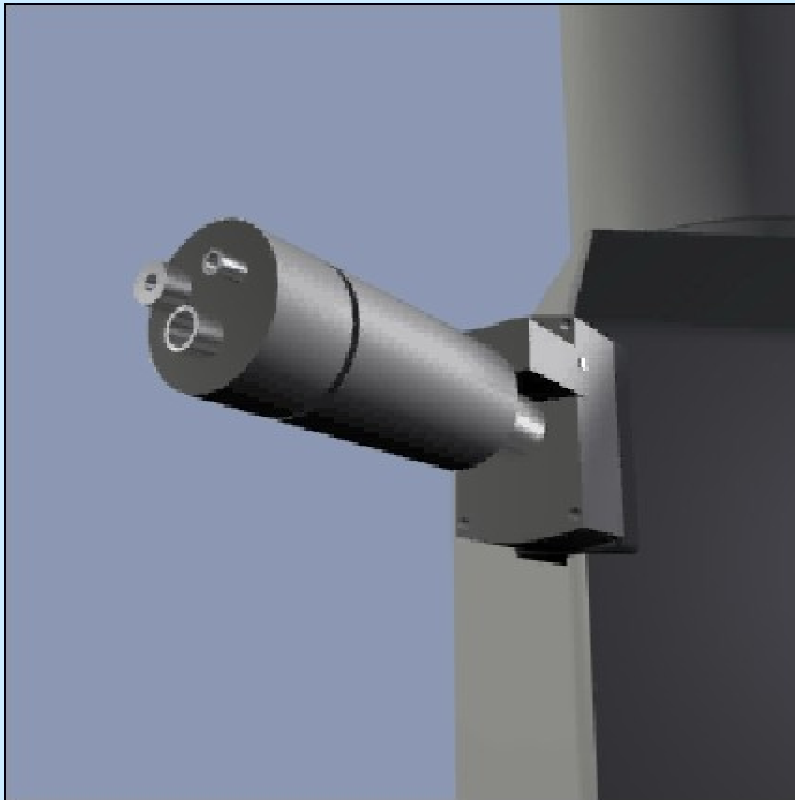
Secondary electrons come from inelastic scattering of the incident electrons with the electrons that surround the atom.



Best analogy - trying to hit a golf ball through a tree.

Detection

SE can be collected by placing a positive voltage on the detector.



SE images appear very 3-dimensional.

A thin detector over the sample detects the BSE electrons that strike

BSE signal is very directional.

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Backscattered Images

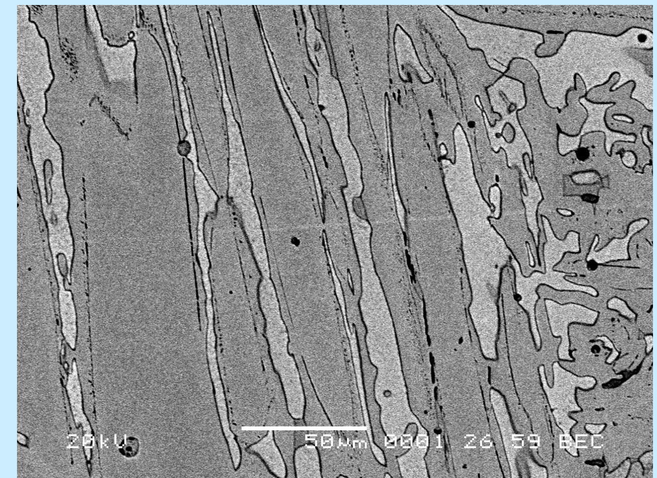
BSE electrons produce an image related to the size of the nucleus. The larger the nucleus, the more BSE I get.

Al-Cu alloy. Copper has a larger nucleus so Cu rich areas appear white, Al rich areas, dark gray.

Important:

2) **The observed contrast is always relative!**

3) **Sample must be polished flat!**



Steel sample. The lighter regions rich in Cr, black areas are carbides. But the sample is entirely steel!

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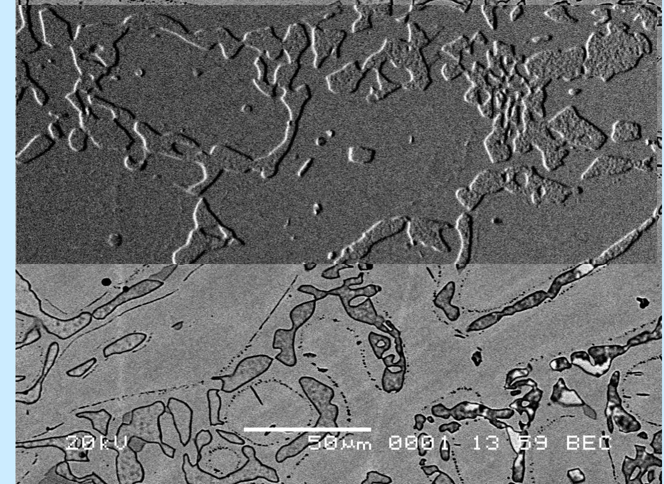
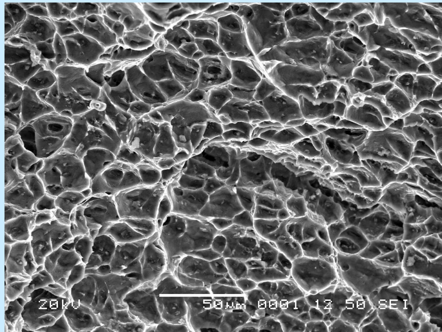
Topo / Compo Imaging

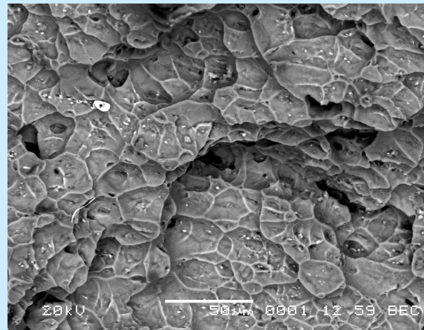
BSE emission is directional. Manipulation of multiple signals can produce a topographical or compositional image.



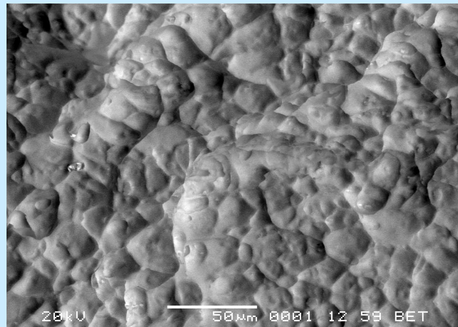
Top = Topographical (signals subtracted)
Bottom = Compositional (signals added)



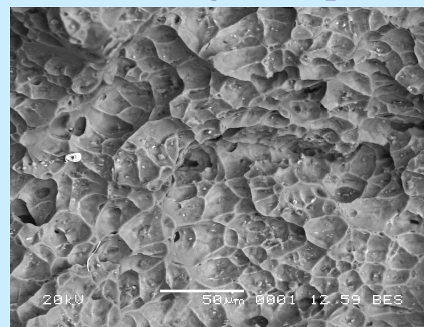
SE image



BSE image, compo



BSE image, topo



BSE image, combination

The directional nature can produce confusing images from rough samples.

All 4 images on the left are of the exact same area!

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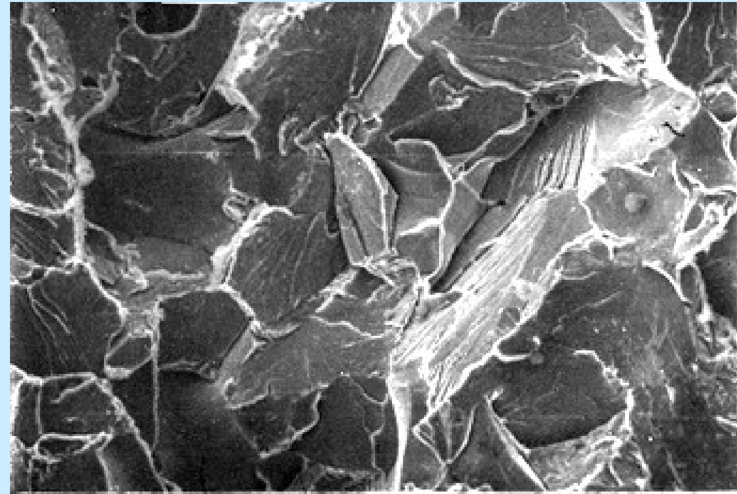
Secondary Electrons

SE produce an image related to the surface roughness of the material. More SE are emitted from steep slopes and sharp points than flat surfaces.



SE image from broken steel. Ridges, steep sides show up brighter.

Collection efficiency of SE is higher from the sample side facing the detector. Thus, shadows from low points and regions away from the detector produce a “3-d” image.

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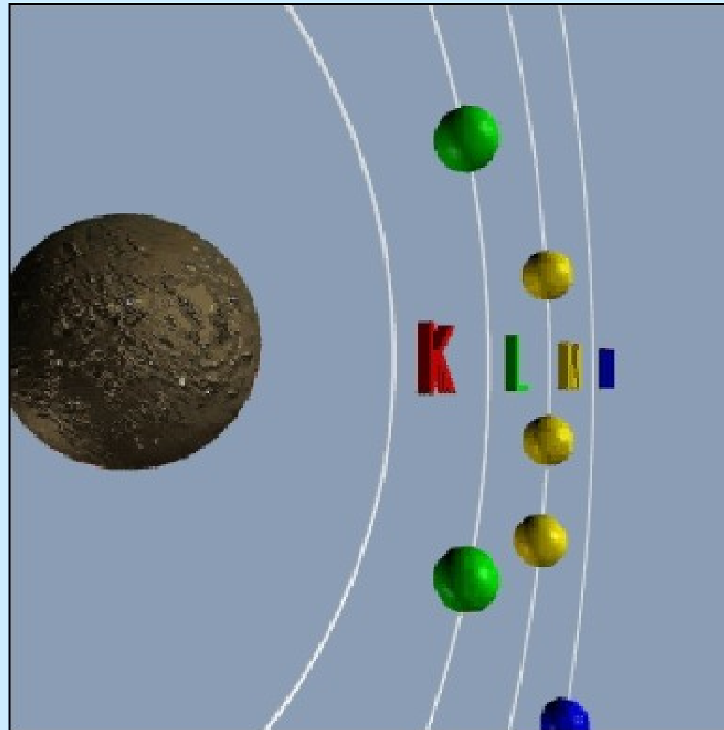
X-rays

X-rays are also generated by inelastic collisions. There are two basic kinds, those that are characteristic of the atom the e- collides with and continuum x-rays which really are just background and can't be used for identification.

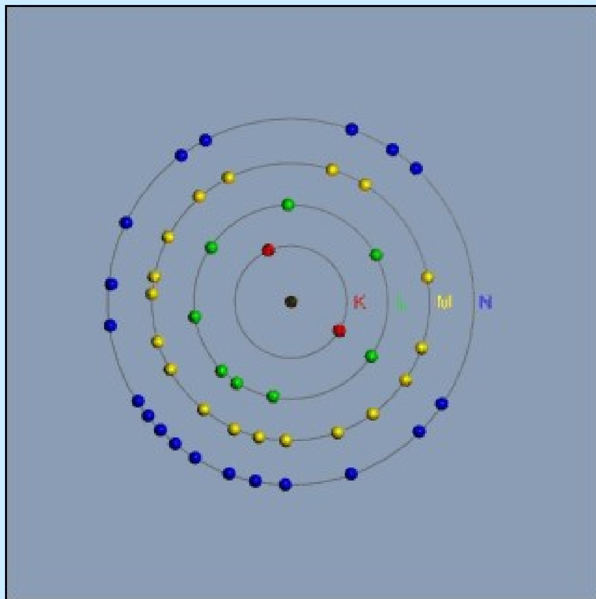
Electron from beam interacts with the atom's electrons and loses energy.



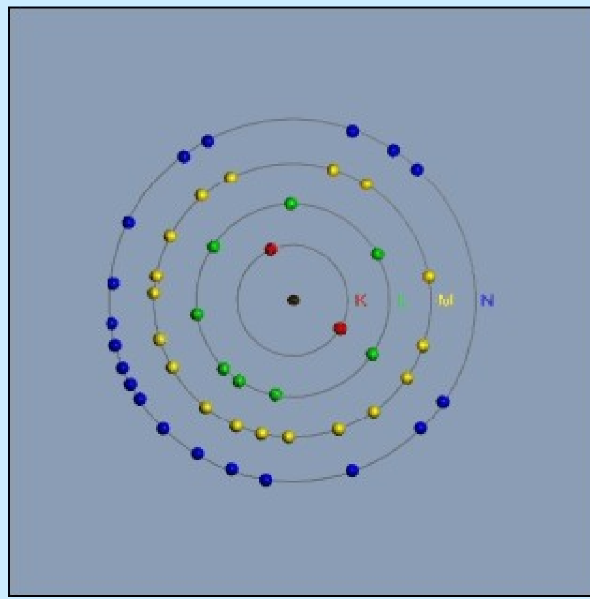
Energy from the interaction is converted into an x-ray.

Characteristic X-rays

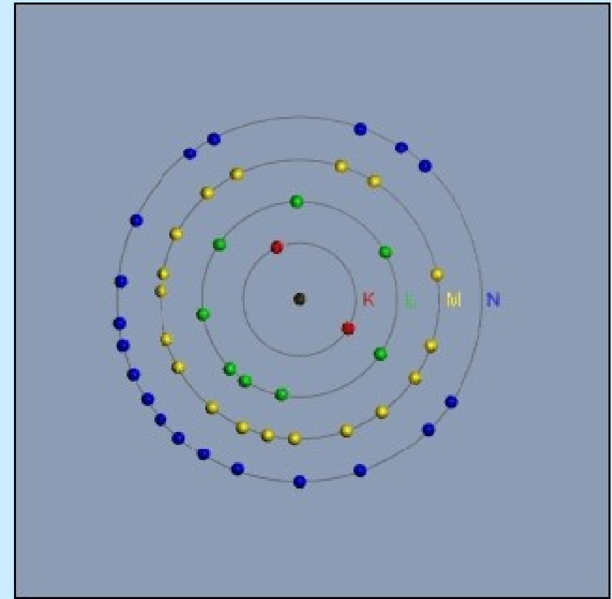
Characteristic X-rays are much more useful! Their energy is related to the atom because they involve collisions from actual electrons in the sample.



Innermost shell gives K x-ray



2nd shell gives L x-ray

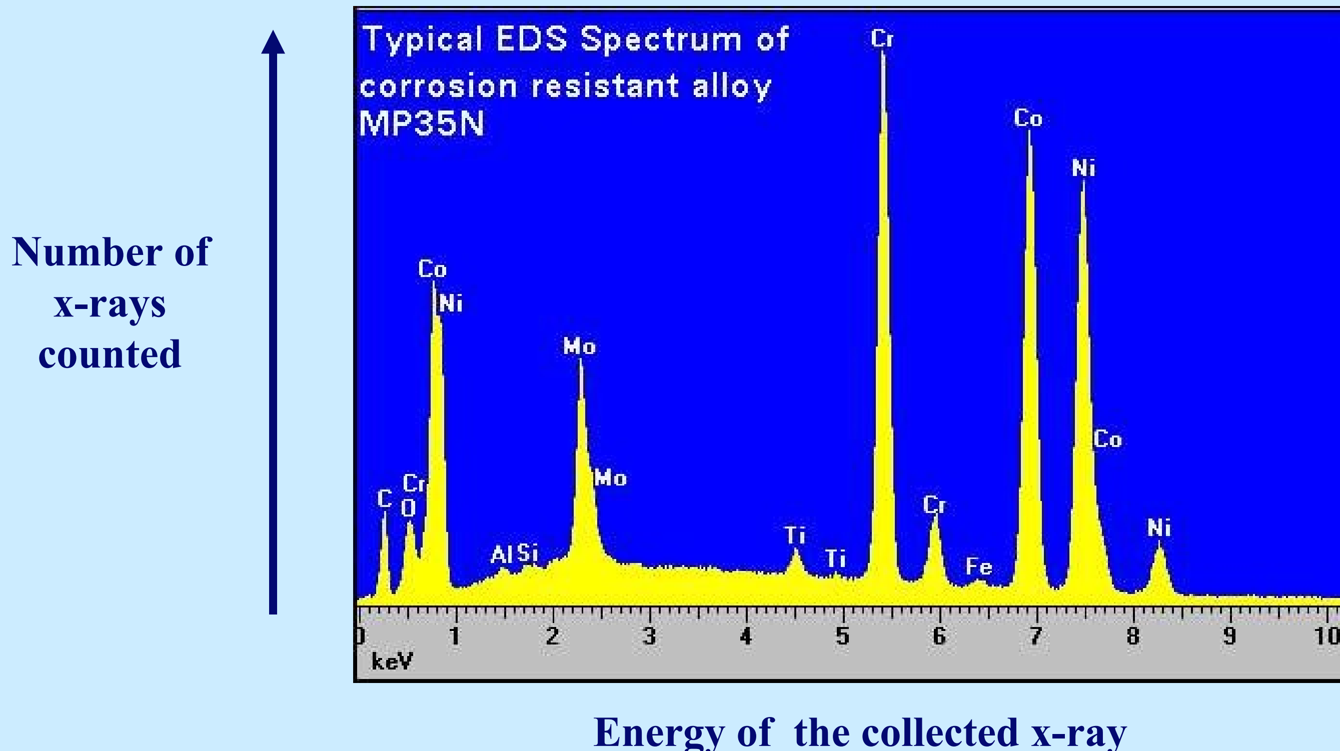


3rd shell gives M x-ray

K and L x-rays are most commonly observed and used.

Characteristic X-ray Spectrum

By collecting all of the x-rays and counting and separating them according to energy we can get a spectra that is characteristic of any particular material or element.



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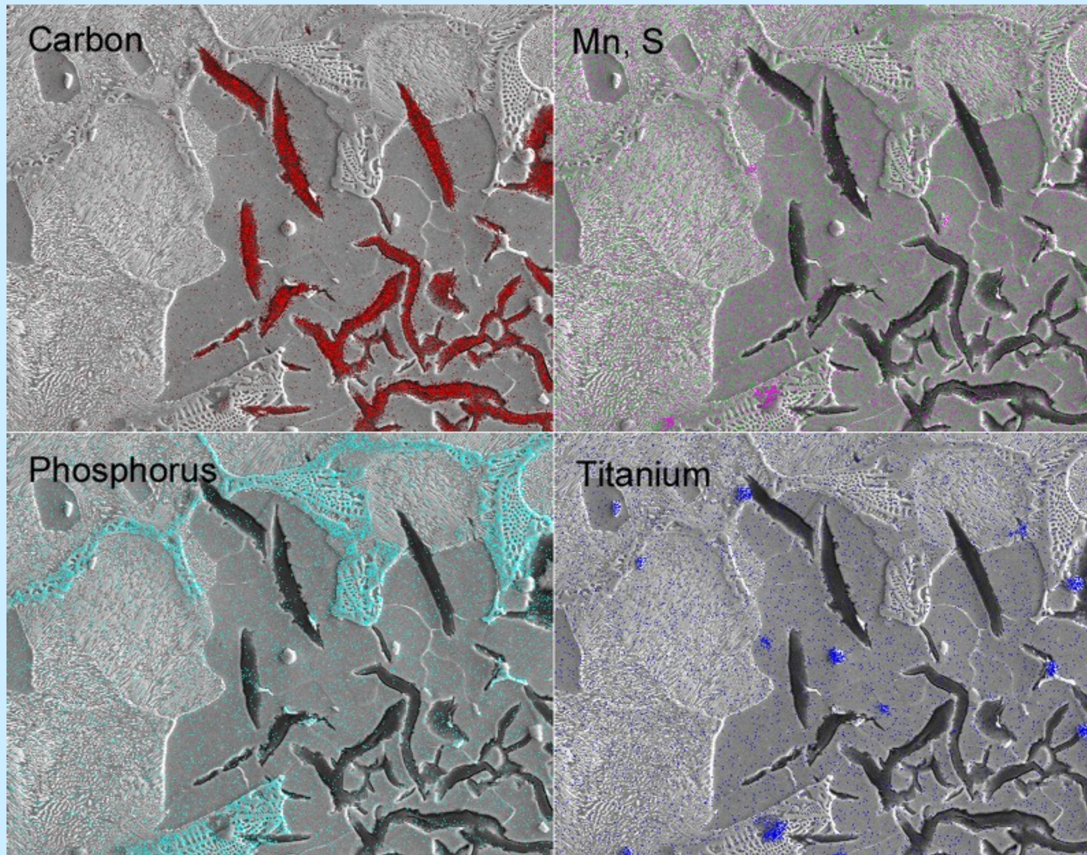
"Dot" Maps

It is possible to have the computer display a colored dot at the location where the scanning beam was when a particular energy x-ray was counted. This is called a "dot map".



Same image of an alloy cast iron. Different elements are selected to show as different colors.

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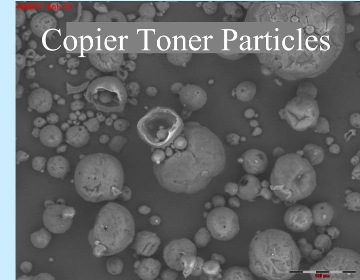
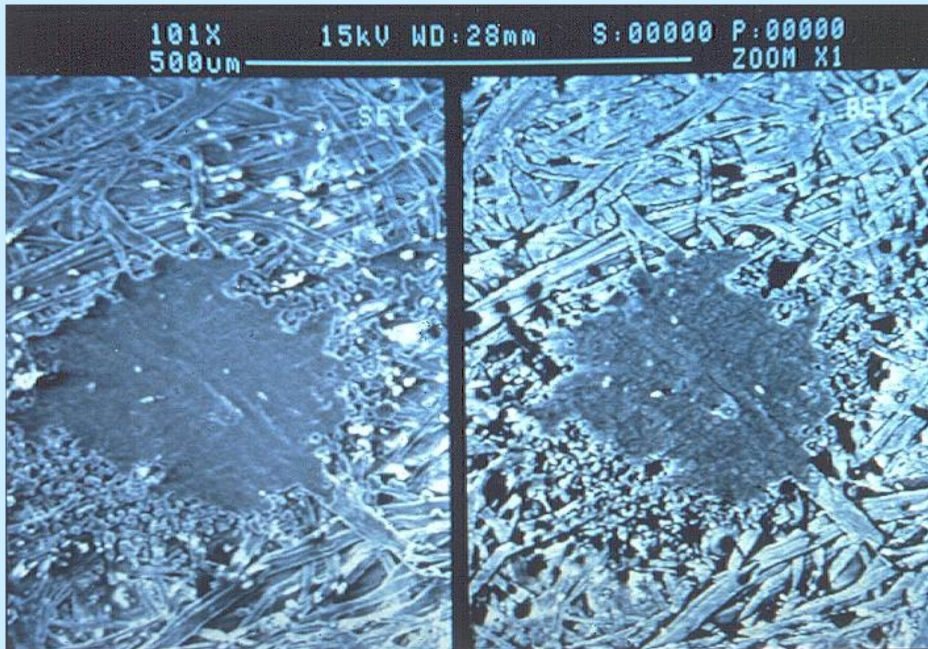
Secondary vs. Backscattered

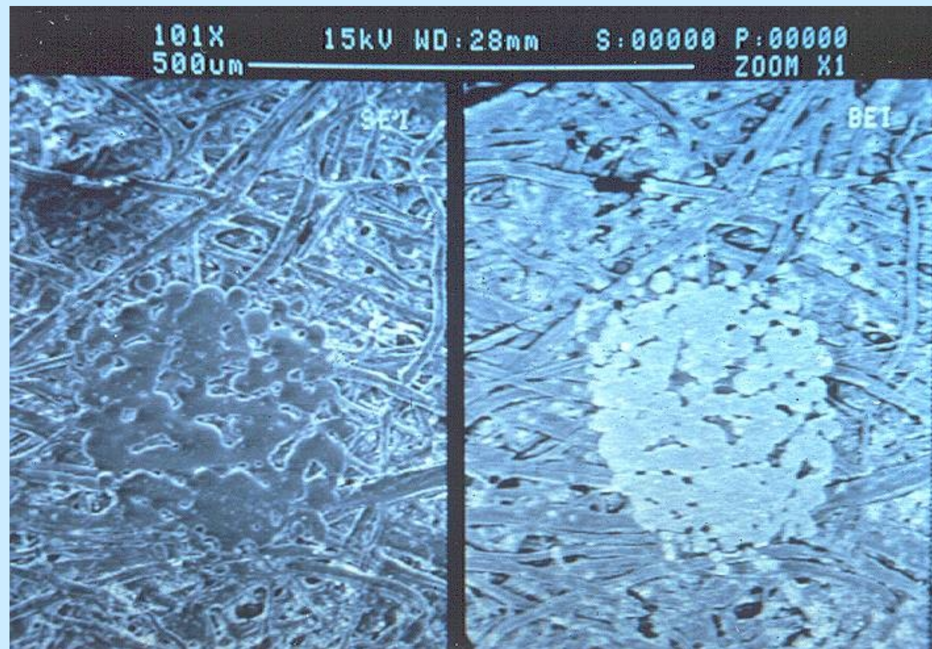
The correct choice of imaging signal can allow you to differentiate between two suspect possibilities…



Imaging of a "period" on paper, produced by two distinct copiers



Copier "A" Toner



Copier "B" Toner

The difference is clearly seen using BSE, indistinguishable with SE.

(Data from F. Platek, FDA)

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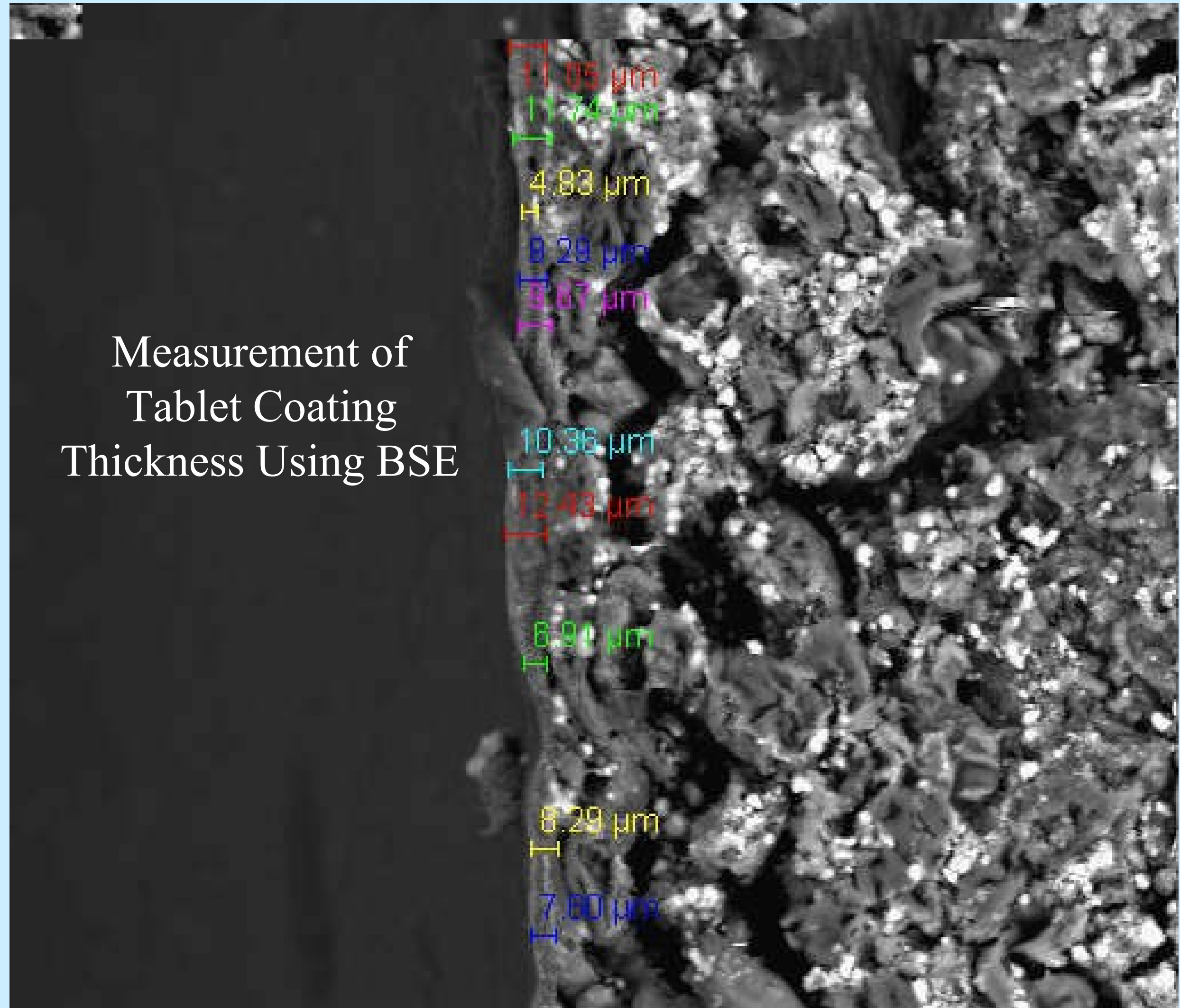
Imaging of Layers

Since this coating is an edge, using SE imaging it would all appear bright and the thin layer would be lost.



(Data from F. Platek, FDA)

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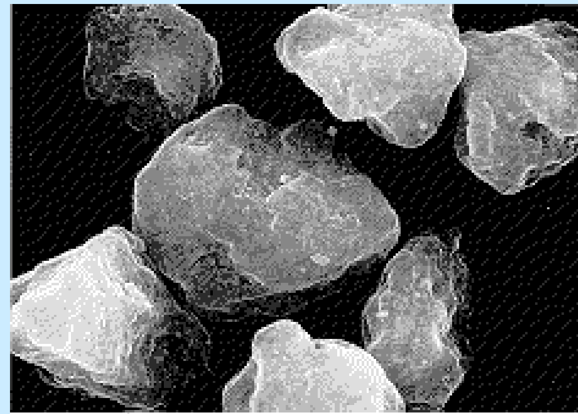
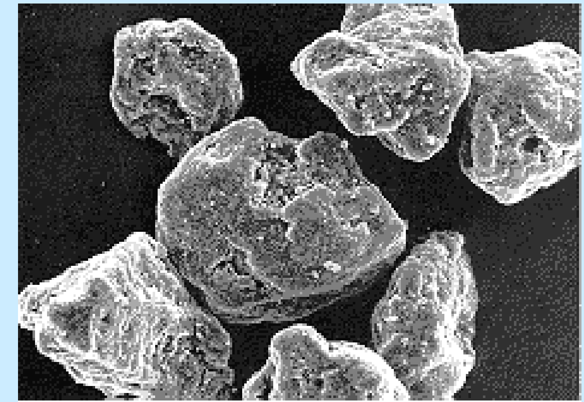
Microscope Operating Parameters

How I operate the microscope will to a large extent determine what I see. One of the major considerations is the choice of accelerating voltage.

A low voltage limits penetration of the beam and produces an image that is more surface sensitive.

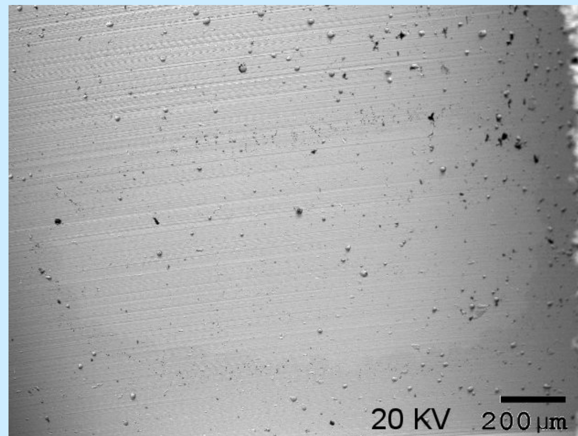


(a) 30 kV x 2,500



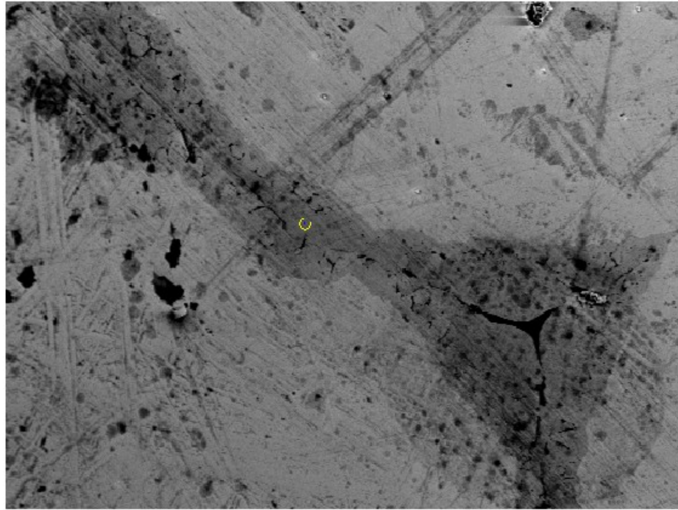
(b) 5 kV x 2,500

A lower voltage can more clearly reveal surface films or contaminants.



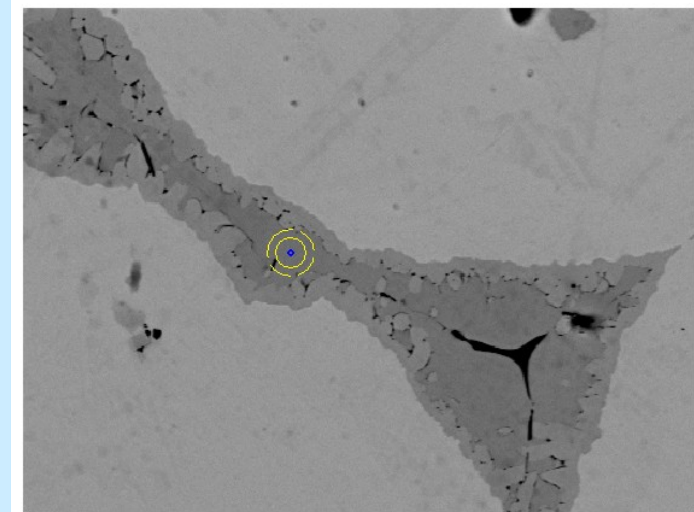
Imaging and EDS Analysis

While imaging may improve, X-ray analysis may suffer…



60µm

5 kV



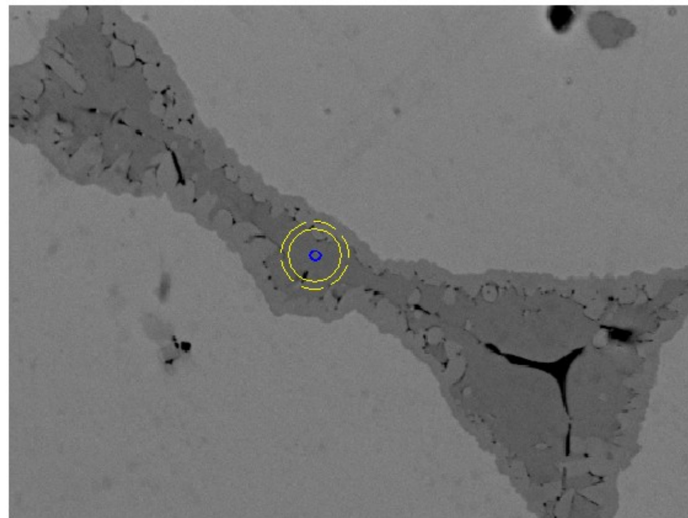
60um

20 kV

Higher voltage gives a clearer image of other phases,

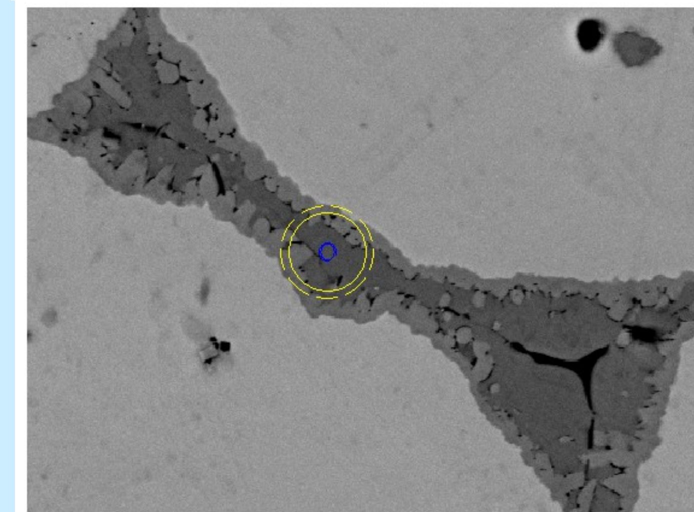
HOWEVER,

It comes with increased spreading of the beam (yellow circles) which can compromise EDS analysis.



60µm

25 kV

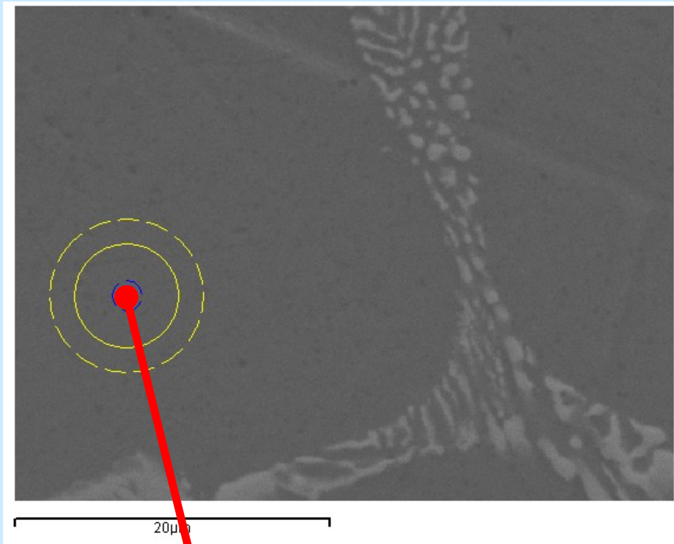


60µm

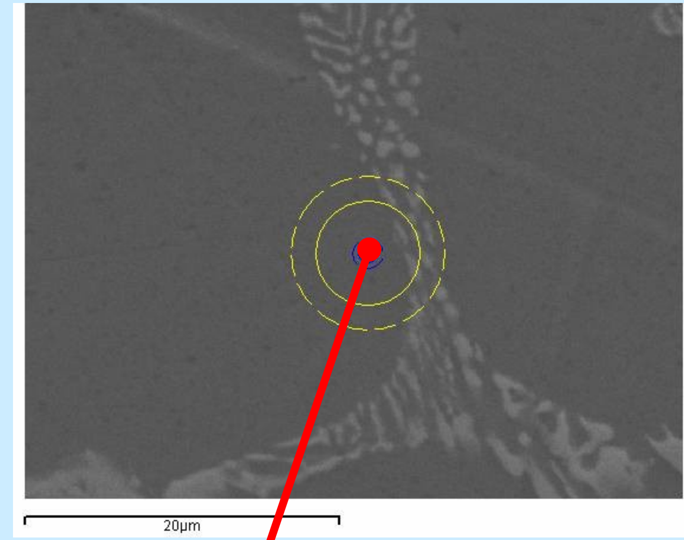
30 kV

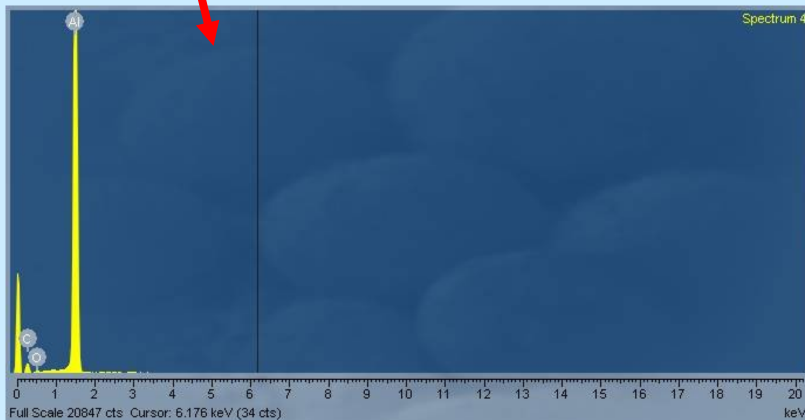
Interaction Volume

Remember, X-rays are generated from an area much larger than your beam. Consider these two Al-Y sample analyses…



Solid and dashed lines show extent of x-ray generation. Even though the beam is outside the Y-rich phase (right), beam spreading below the surface causes Y to be detected.

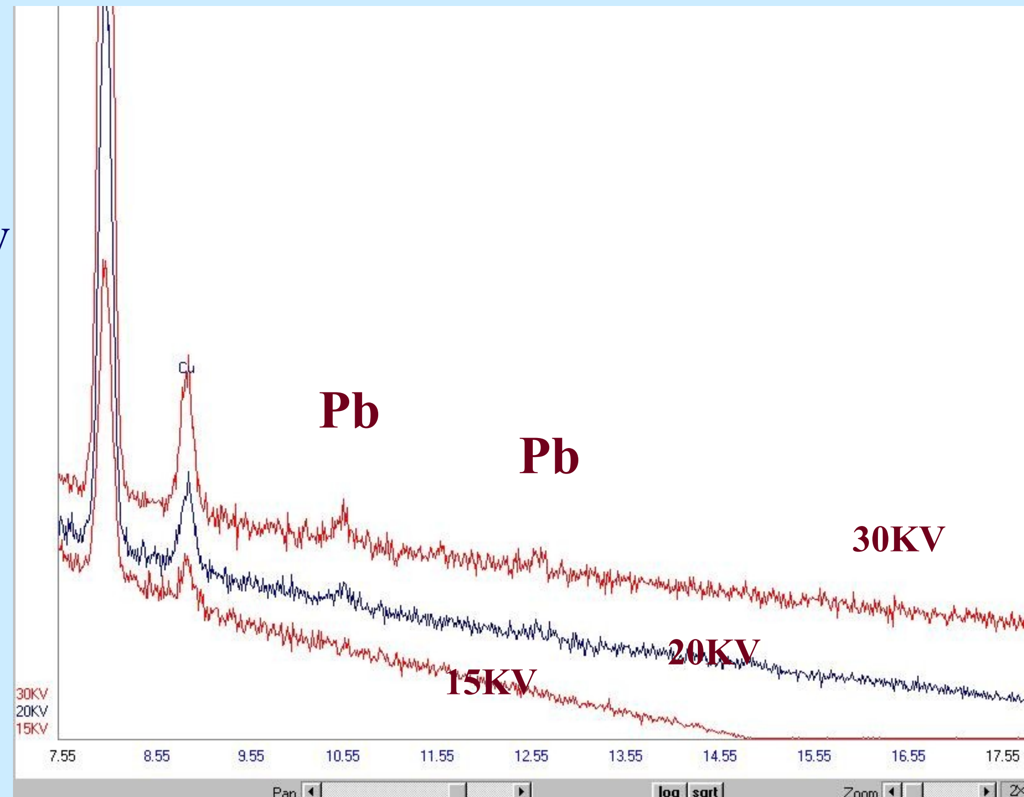




Effect of Overvoltage

In order to excite an x-ray of a particular energy, the beam needs to have at least 1.5x the energy (overvoltage) just to generate the x-ray. For example, if my x-ray has an energy of 10 KV…

If Beam Voltage is too low you may be unable generate higher energy (shorter wavelength) x-rays!



(Data from D. Ward, FBI)

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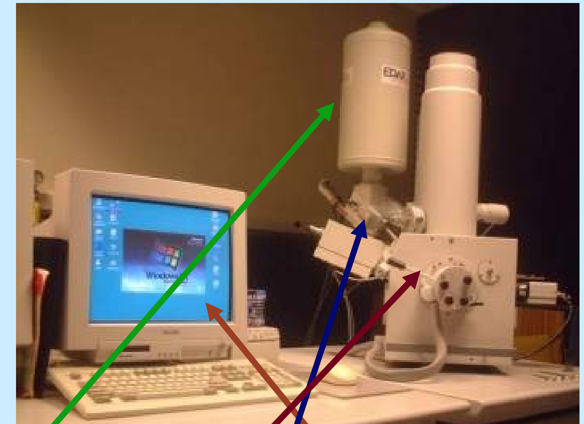
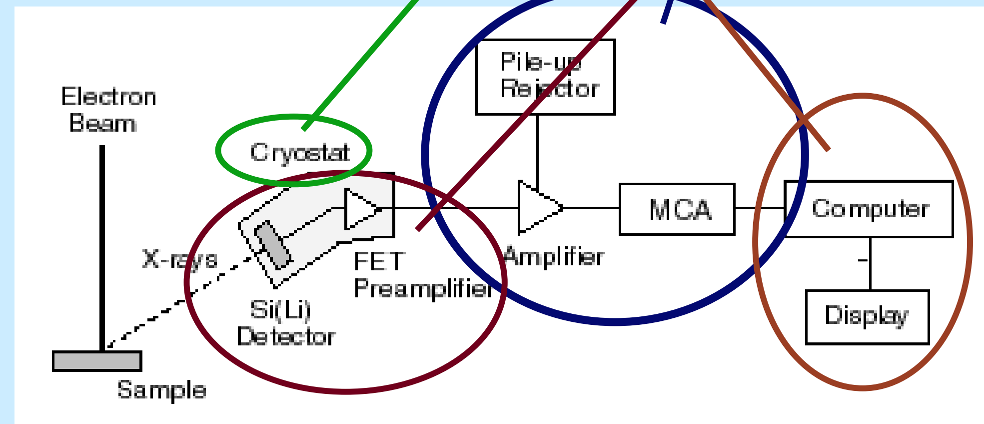
Detector Operating Parameters

The Energy Dispersive Spectrometer is the most often used detector since it is the easiest, least expensive, flexible, and gives good results.

Effective use requires a knowledge of detection parameters (e.g. deadtime, count time, pulse processor time constant), and how the software operates.

Typical EDS with schematic showing the main coponents.





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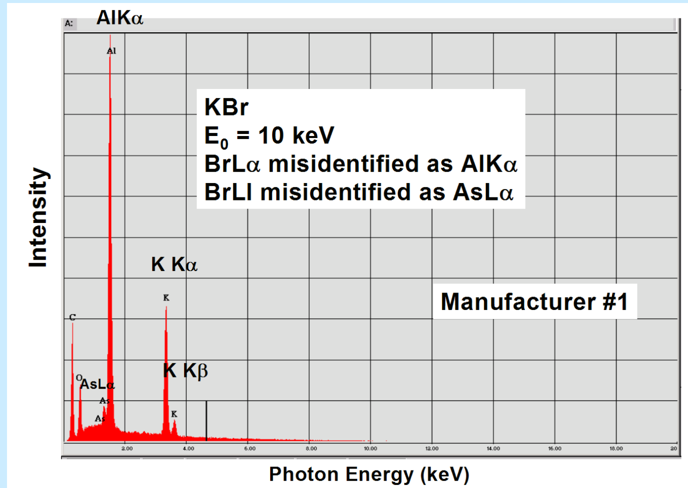
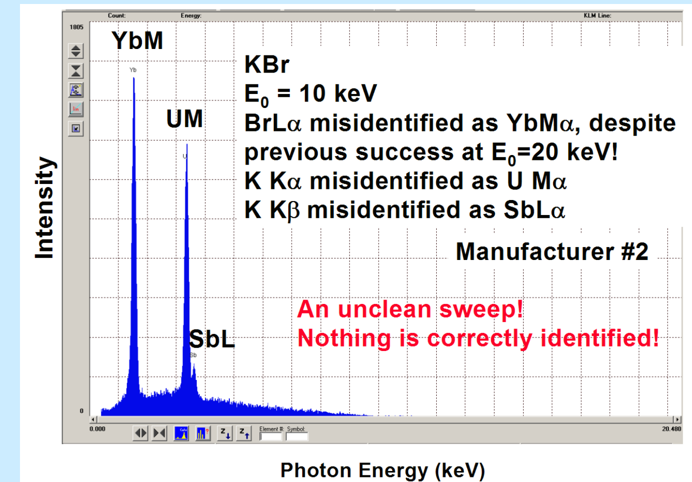
Peak Misidentification

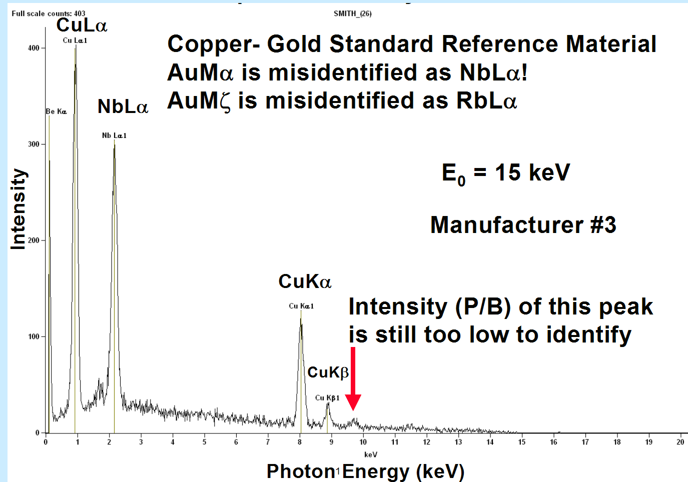
Relying too heavily on the software can lead to mistakes.

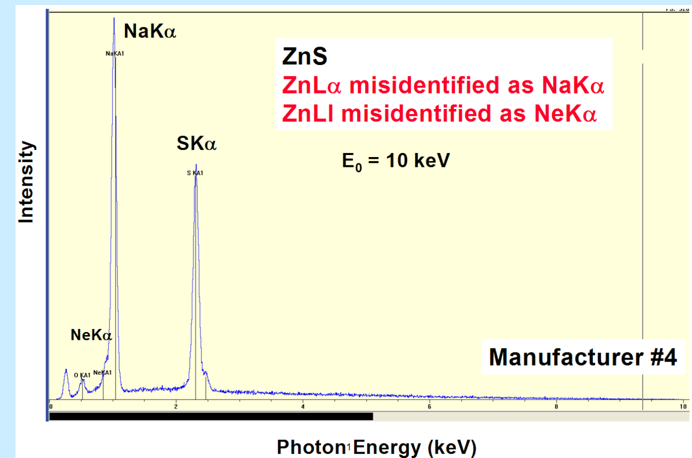




4 companies, all misidentify various samples and elements

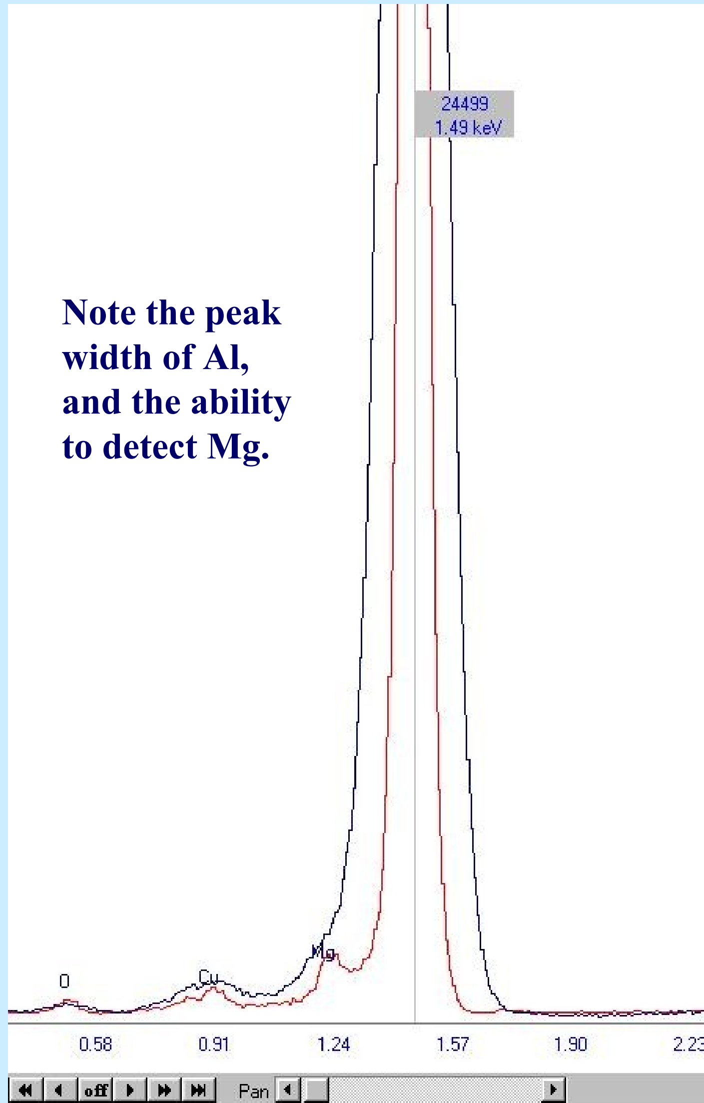
(Data from D. Newbury)





Commercial routines commonly make mistakes!

Example: Processing Time



(Data from D. Ward, FBI)

Effect of Pulse Processor Time Constant:

Red = 102us, Blue = 3.2us

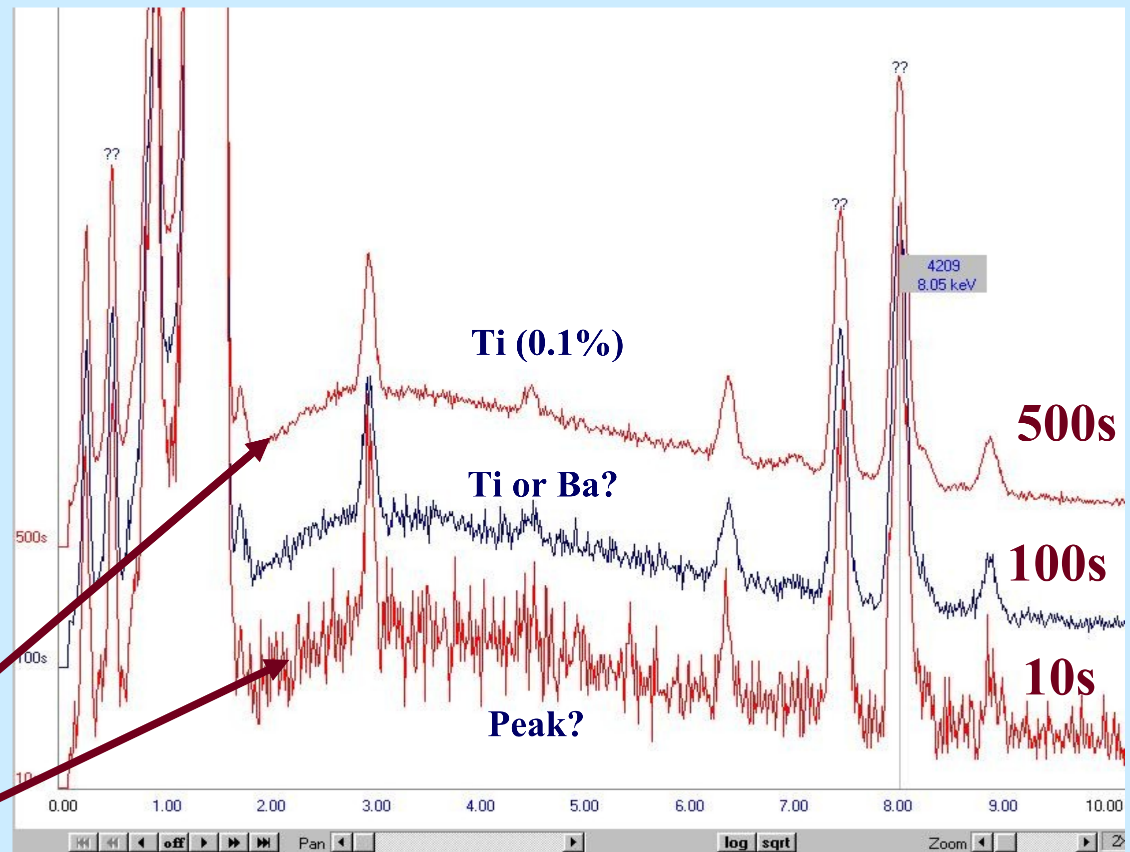
As Time Constant increases, resolution, sensitivity increase.

Note that increasing the time constant is different than simply counting for a longer period of time.

Example: Counting Time

Trace element detection and good statistics requires long acquisition times.

The smaller the amount desired to detect, the longer the processing and counting times must be.



Rule of thumb: Make sure your peaks are gaussian and the background looks like

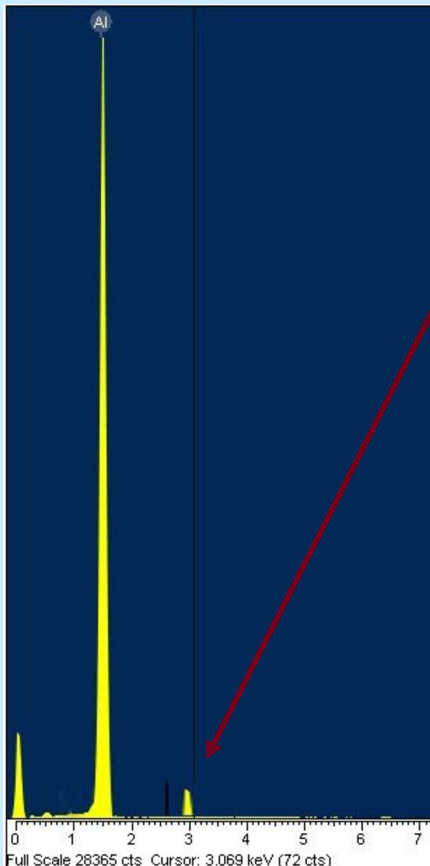
This:

Not this:

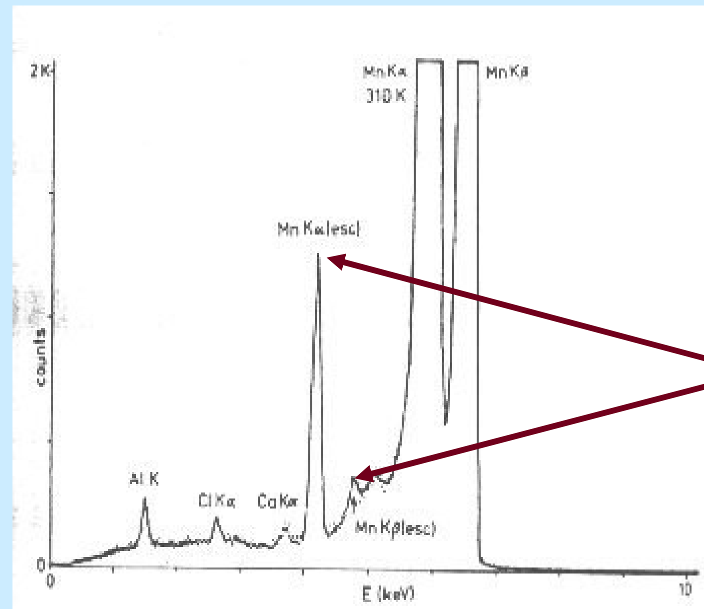
(Data from D. Ward, FBI)

Example: False Peaks

Increasing the beam current will increase the count statistics. However, a number of false peaks can appear that may cause problems…



SUM PEAKS result when 2 x-rays enter at the exact same time, producing false peaks at energy $E = E_1 + E_2$

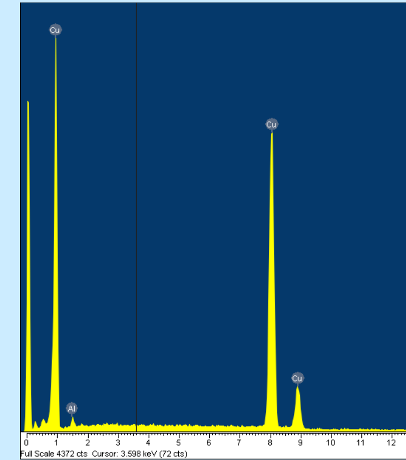


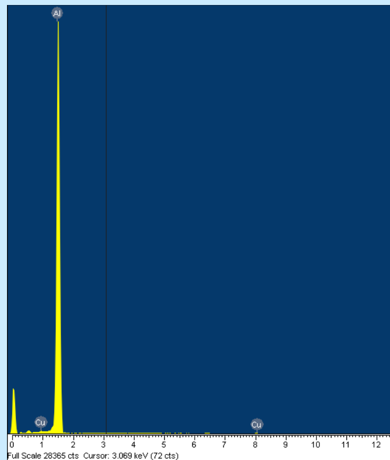
Sometimes Si x-rays are generated in the Si detector, producing false peaks at energy $E = E_1 - E_{Si}$ called Si Escape peaks.

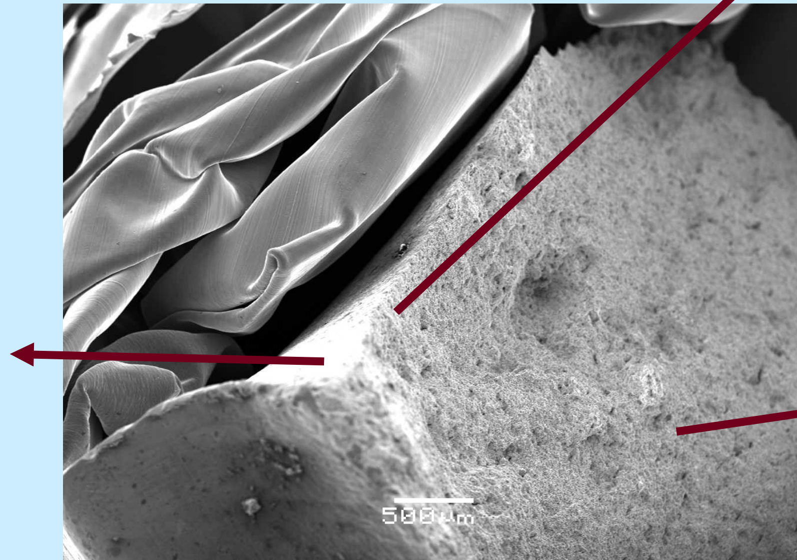
These peaks usually result during high count rates.

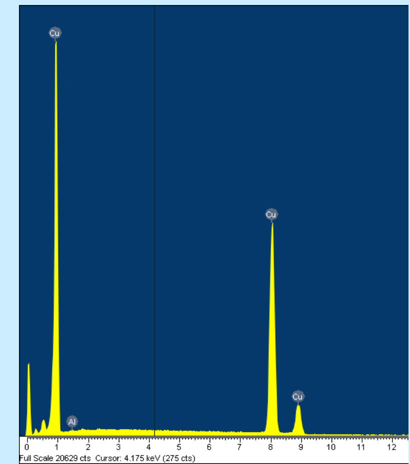
Sample Parameters

If sample is not flat, BSE emission can fluoresce x-rays that are not there, absorption can eliminate x-rays that are there. It is important to know the location of the detector with respect to the sample surface.









Cu sample held in place by Al foil.

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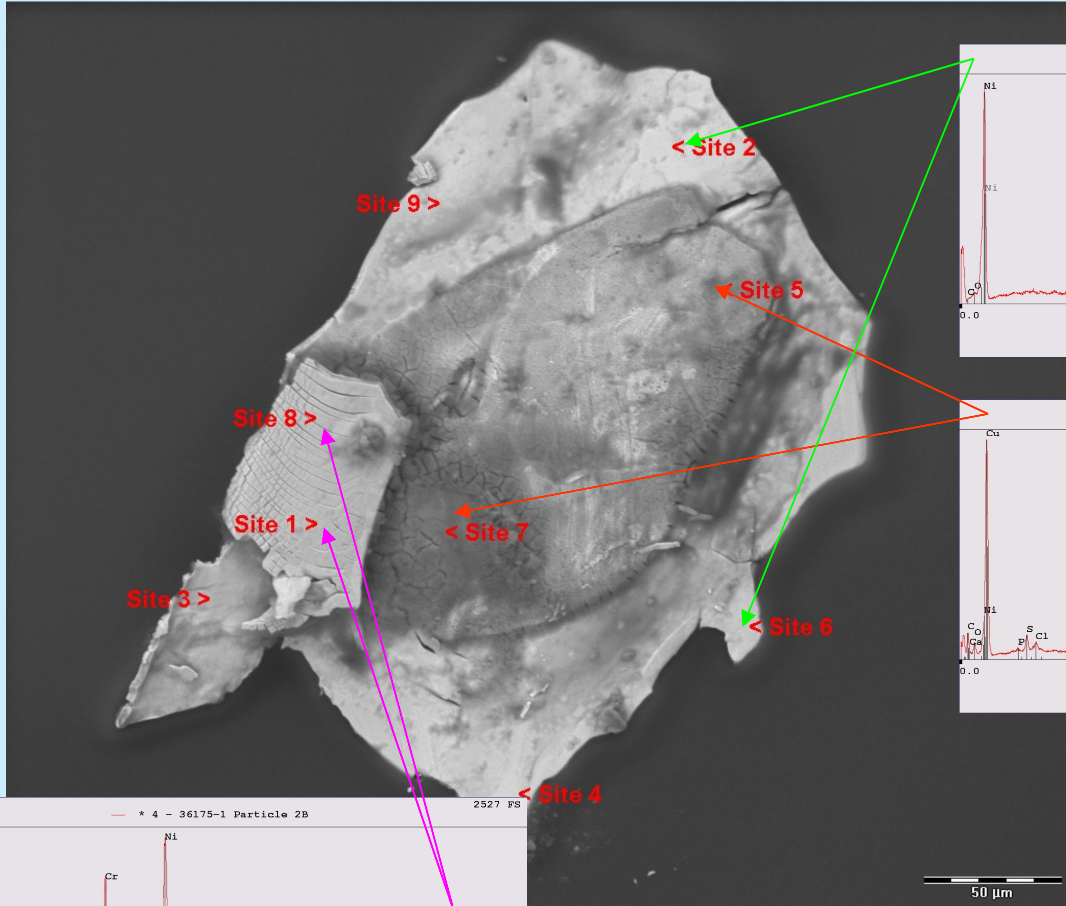
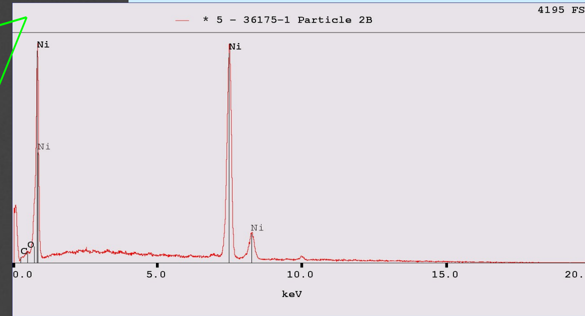
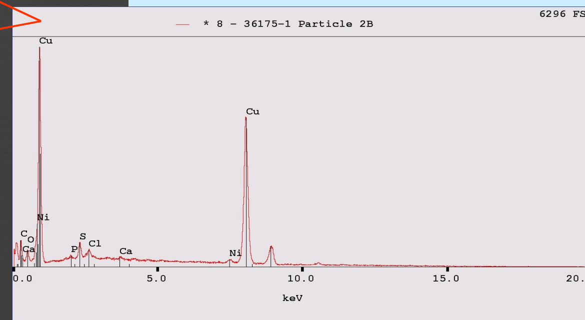
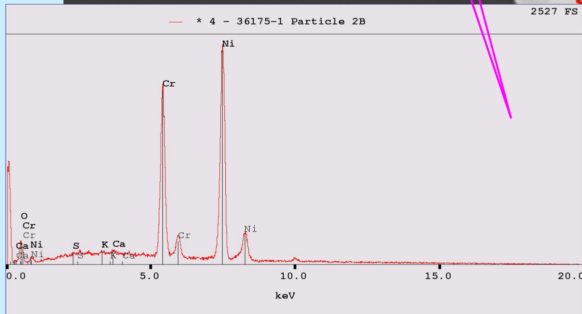
Multiple Site EDS









(Data from F. Platek, FDA)

Taking data from various sites can produce varying results.

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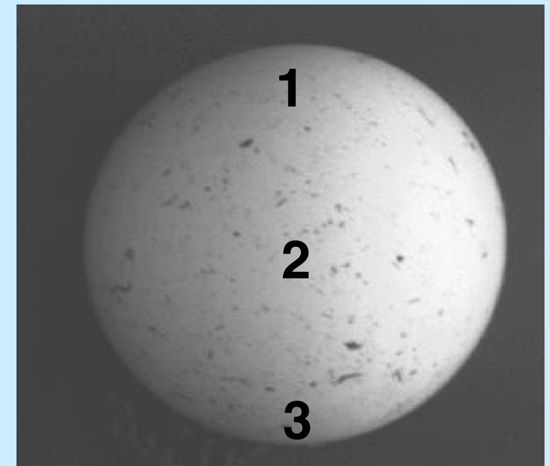
Examples

Similar sample geometry is VERY important when comparing spectra from two different samples.

Si
Al
Cr
Cr, (Mn)
1
2
3
detector side
normal
non detector side
0.37 1.37 2.37 3.37 4.37 5.37 6.37 7.37 8.37 9.37 10.37
Pan log sqrt Zoom 2X

Detector



(Data from D. Ward, FBI)

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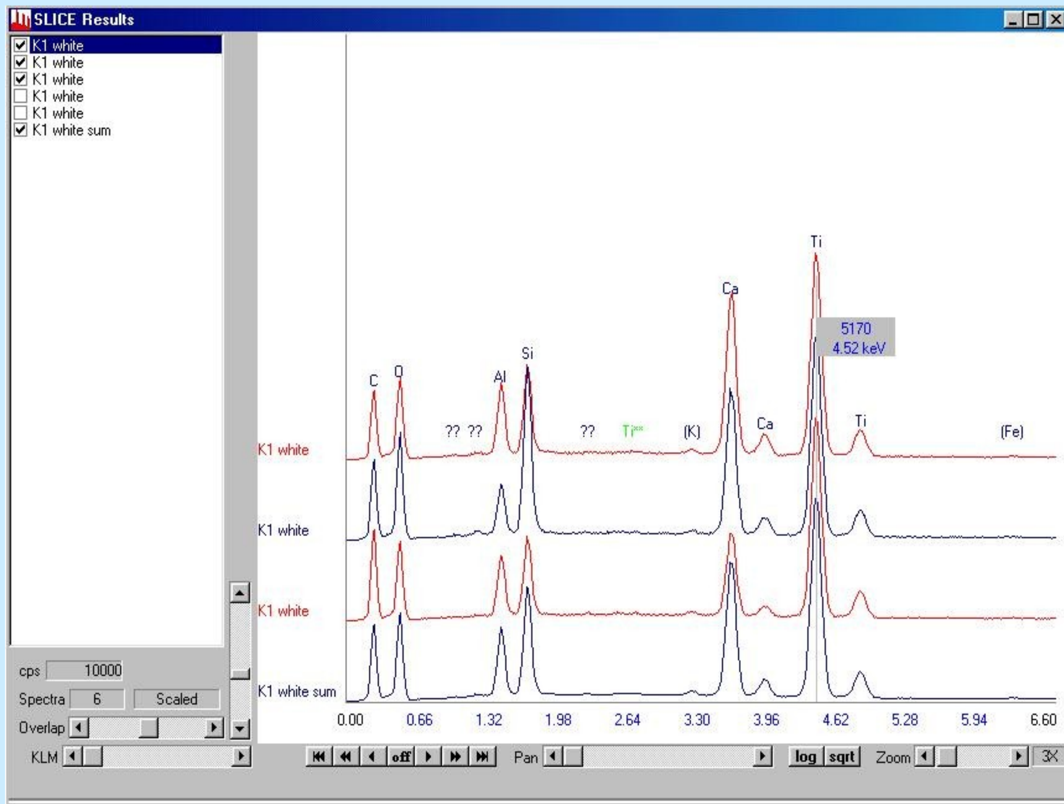
Composition Averaging

When trying to get an average composition from a large number of particles it is important to obtain numerous spectra that encompasses as many particles as possible...



In example, the 3 "small area" spectra show peak ratio differences (spectra are scaled on Ti). Summing the spectra (bottom spectra) approaches true sample composition.

(Data from D. Ward, FBI)

Quantitative Analysis

Quantification involves comparing data from your unknown to data from known standard compositions.

Calibration standard containing known elements and / or compounds.

The Cliff-Lorimer expression relates the known compositions to the unknowns in terms of x-ray intensity.

$$\frac{C_i}{C_{(i)}} = \frac{I_i}{I_{(i)}} \times (ZAF)_i$$

C_i = wt % of i in unknown

$C_{(i)}$ = wt % of I in standard

I_i = intensity of I x-rays in unknown

$I_{(i)}$ = intensity of I x-rays in standard

ZAF = correction factor for atomic number, absorption and fluorescence

Samples must be polished flat to obtain the best quantitative results.

Methodology

When conducting a quantitative analysis, best results will involve…

- **Using known standards.**
- **Using a flat sample to minimize absorption / fluorescence.**
- **Ensuring proper EDS parameters (time constant, calibration, counting time)**
- **Ensuring proper microscope parameters (beam current, overvoltage)**

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Precision and Accuracy

The questions of importance for any quantitative analysis method are always:

1. **How accurate are my measurements?**
2. **How precisely can I measure something?**
3. **What are my detection limits?**

These are always hard questions since the answer can depend on the system, the operator, and the sample. But, in general, if you have good data and know what you are doing….

#1: About 5% relative **#2: About 5%** **#3: About 0.1 wt%**

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Outline

I. Introduction
   - Basics of Operation

II. Specimen Electron Interactions
   - Backscattered Electrons
   - Secondary Electrons
   - X-rays

III. Examples of Signal Detection and Analysis
   - Imaging (Backscattered and Secondary)
   - Chemical Analysis (X-rays)

IV. Summary

Summary

Hopefully now we have covered all of these basic objectives...

- **You know a little bit about the basics of operation of an SEM.**
- **You are aware of the most common signals and information you can get using an SEM.**
- **You have seen how different results can be obtained depending upon which signal is used and how the SEM is operated.**

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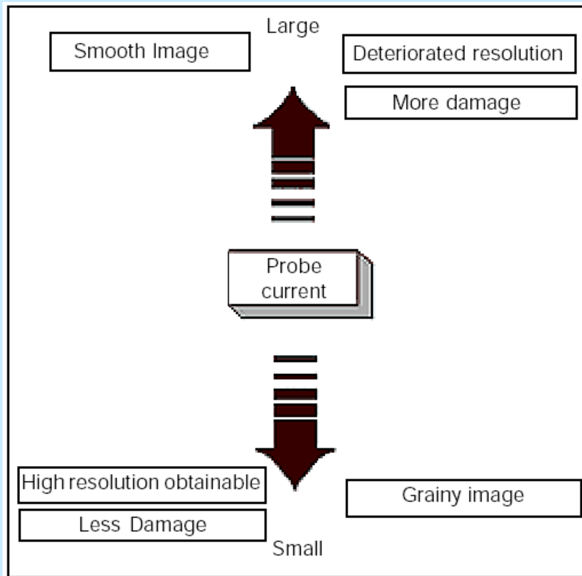
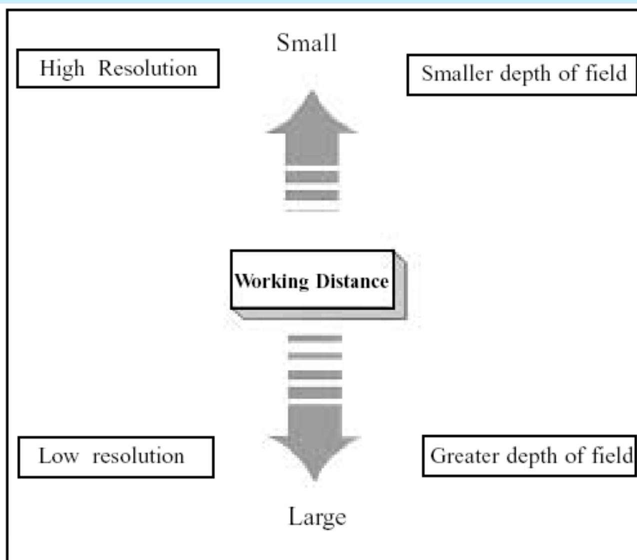
Summary of Lens Operation



Weak Condenser Lens

Strong Condenser Lens

Remember: Condenser lens = probe current, spot size



Strong Objective Lens

Weak Objective Lens

Remember: Objective lens = focus. Can only be changed in conjunction with working distance

(from JEOL operation manuals)

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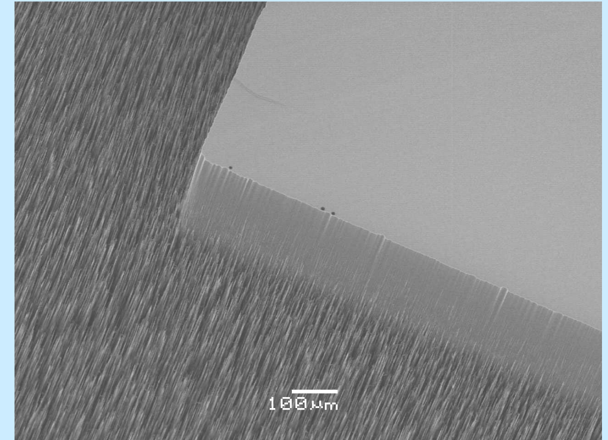
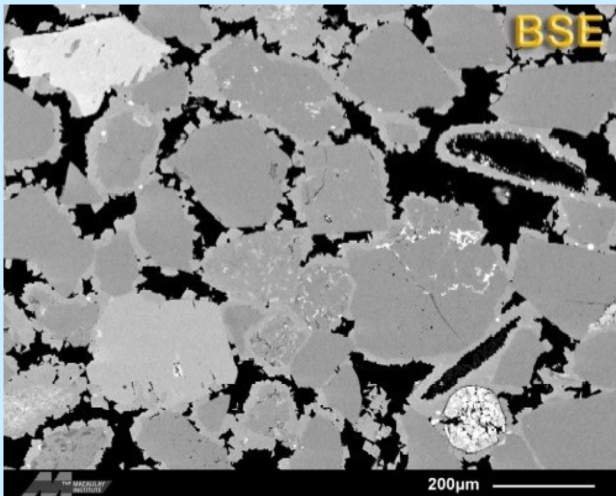
Summary of Signals

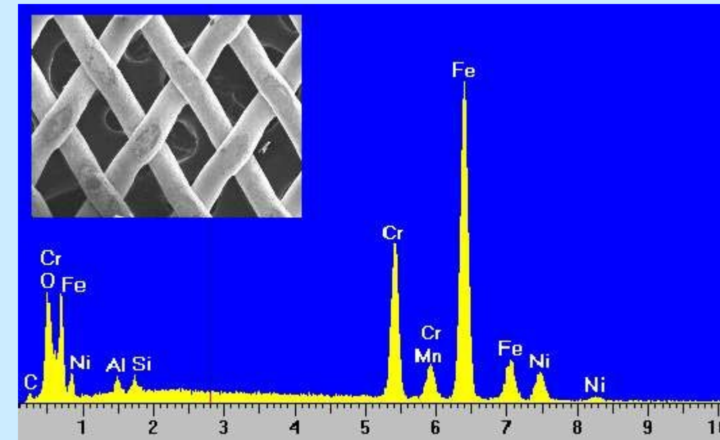


Secondary electrons are good for rough samples.



Backscattered electrons are good for samples with varying composition.

X-rays tell us the elements in the sample and how much there is.



Conclusions

The SEM is a very flexible instrument for numerous applications.

- **One size does NOT fit all. Choose the signal, operating parameters for the desired application.**
- **The information can change depending upon many different things.**
- **Trace analysis requires special care and knowledge of your system and your sample to get the best results.**