Building College-University Partnerships for Nanotechnology Workforce Development

Charged-Particle Interaction Analysis

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Outline

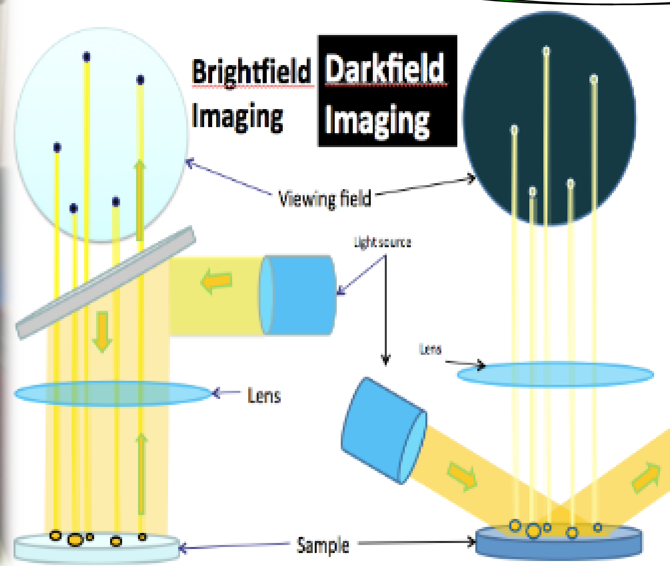
- SEM
  - Limits of Optical Microscopy
  - SEM: The Big Picture
  - Electron Emission
  - Electromagnetic Lenses
  - Apertures
  - Raster Coils
  - Noise Reduction
  - Stage
  - Beam Sample Interaction
  - Beam Detection
  - Charging
  - Image Enhancement
- EDS/EDX
- EPMA
- EBSD

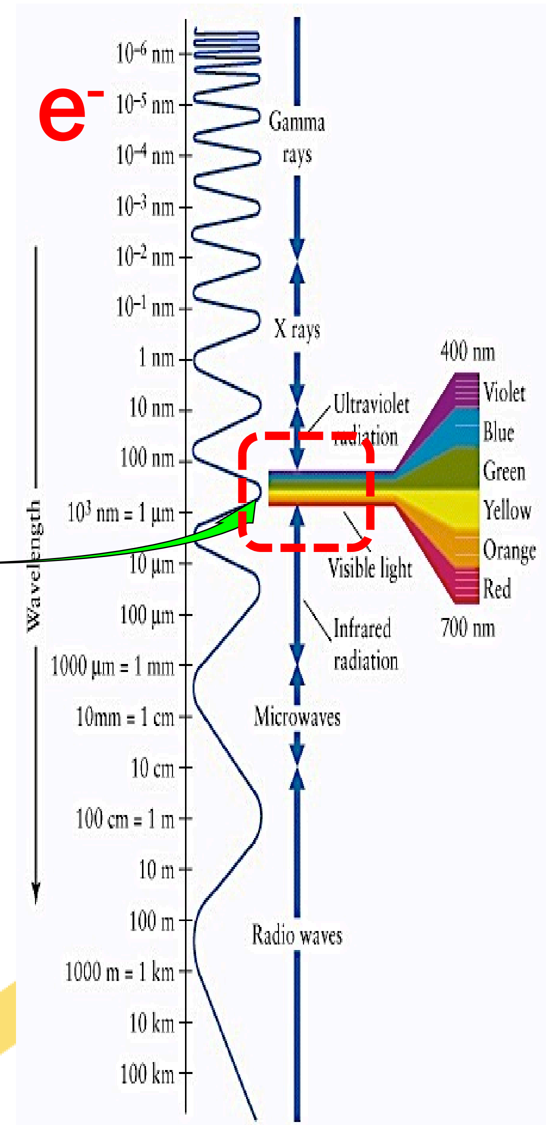
Limits of Optical Microscopy

Top end Mag. ~10,000 X; >200 nm

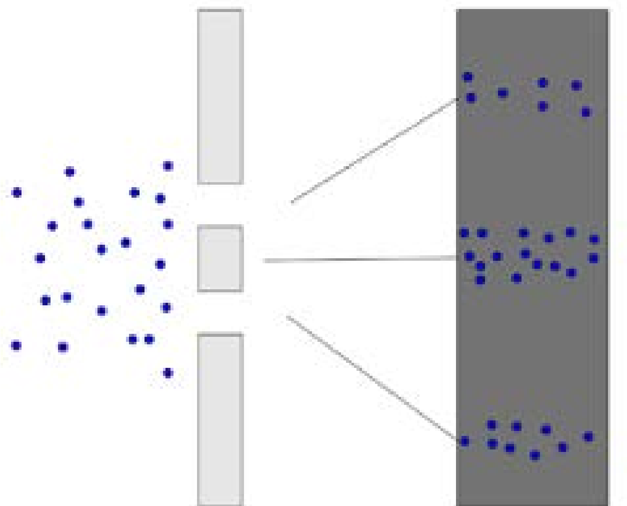
Resolution limit

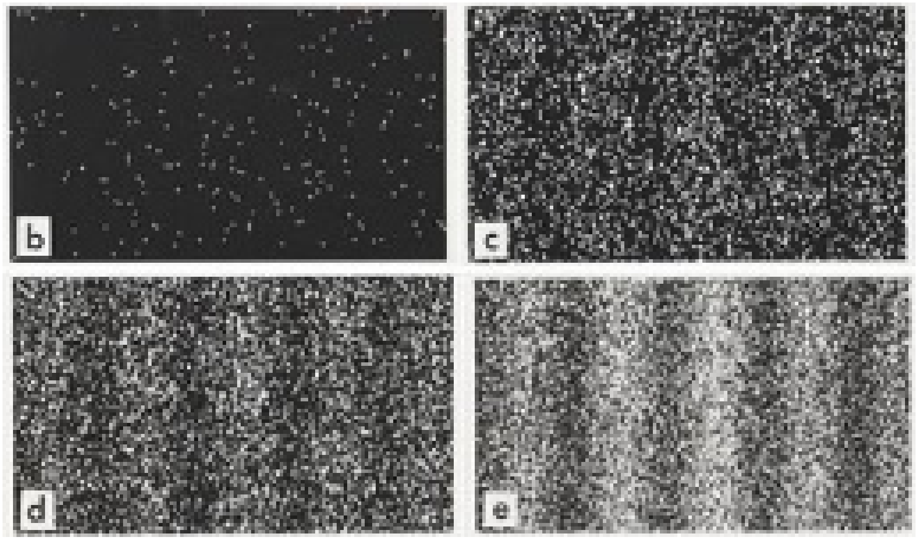
$$d = \lambda/2NA$$



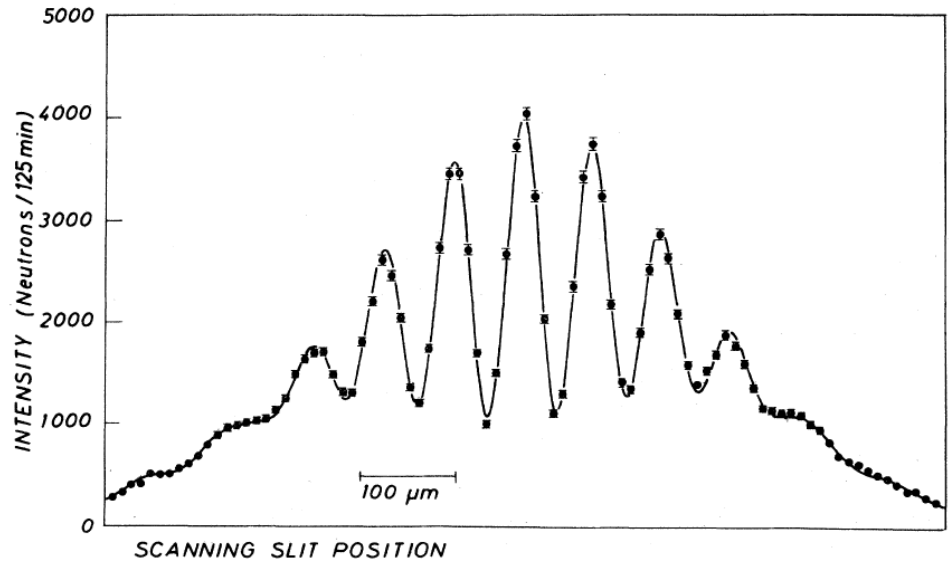


Limits of Optical Microscopy



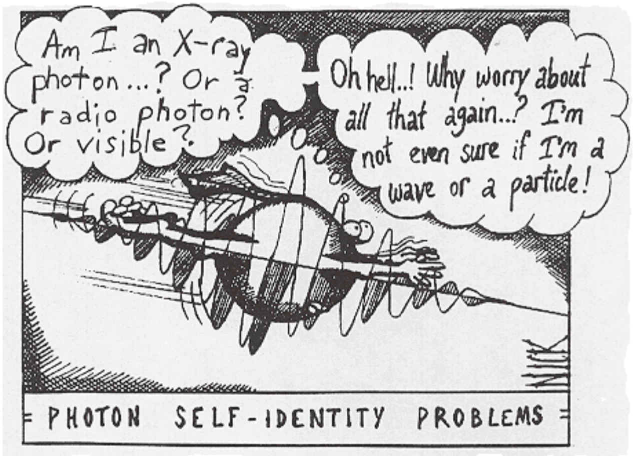


Louis de Broglie



Limits of Optical Microscopy

- Anything and everything has a wavelength
- $\lambda = h / P$
  - λ is the particles wavelength
  - h is Plank's constant
  - P is the relativistic momentum of the particle



- The electrons wavelength is also inversely related to a field acting on it (*U*), like an acceleration voltage
- So…10 kV gets λ 12.3 pm…200 kV gets λ 2.5 pm!
- ~50 million electrons hit the sample every second

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Limits of Optical Microscopy

1858
James Plücker:
cathode rays

1935-1937
Max Knoll/Manfred von Ardenne:
Scanning of electron beam → SEM

1942
Zworykin, Hillier, and Snijder:
Functional SEM →
50 nm resolution

1960
Everhart and Thornley:
Secondary electron detector

1965
Cambridge Scientific Inc.:
Commercial SEM
"Stereoscan"

1986
Ruska, et. al:
Nobel Prize

1940 | **1950** | **1960** | **1970** | **1980** | **1990** | **2000** | **2010**

Nano gets HOT!

1938
Siemens Corp.:
Commercial TEM

1931
Max Knoll, Ernst Ruska, and Bodo von Borries: TEM

1938
Manfred von Ardenne:
Scanning-TEM

1897
J.J. Thomson:
discover electron

1969
Ong Sing Poen (Philips):
EM200 STEM
25 nm resolution

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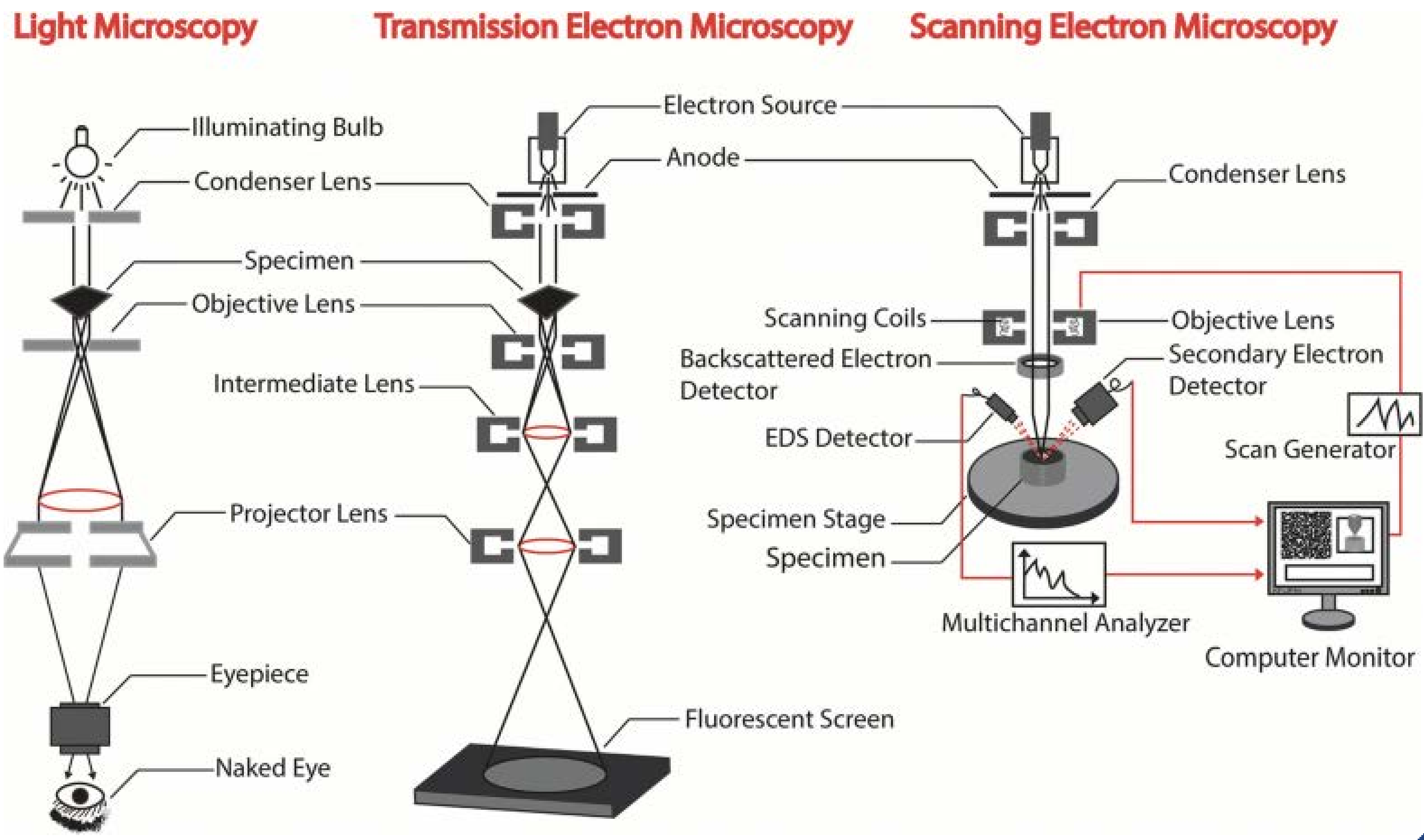
2009
Lawrence Berkeley and Argonne National Labs and FEI and CEOS corps.:
TEAM Aberration corrected TEM →
50 pm resolution!

Limits of Optical Microscopy



SEM: The Big Picture

SEM: The Big Picture

Operation:

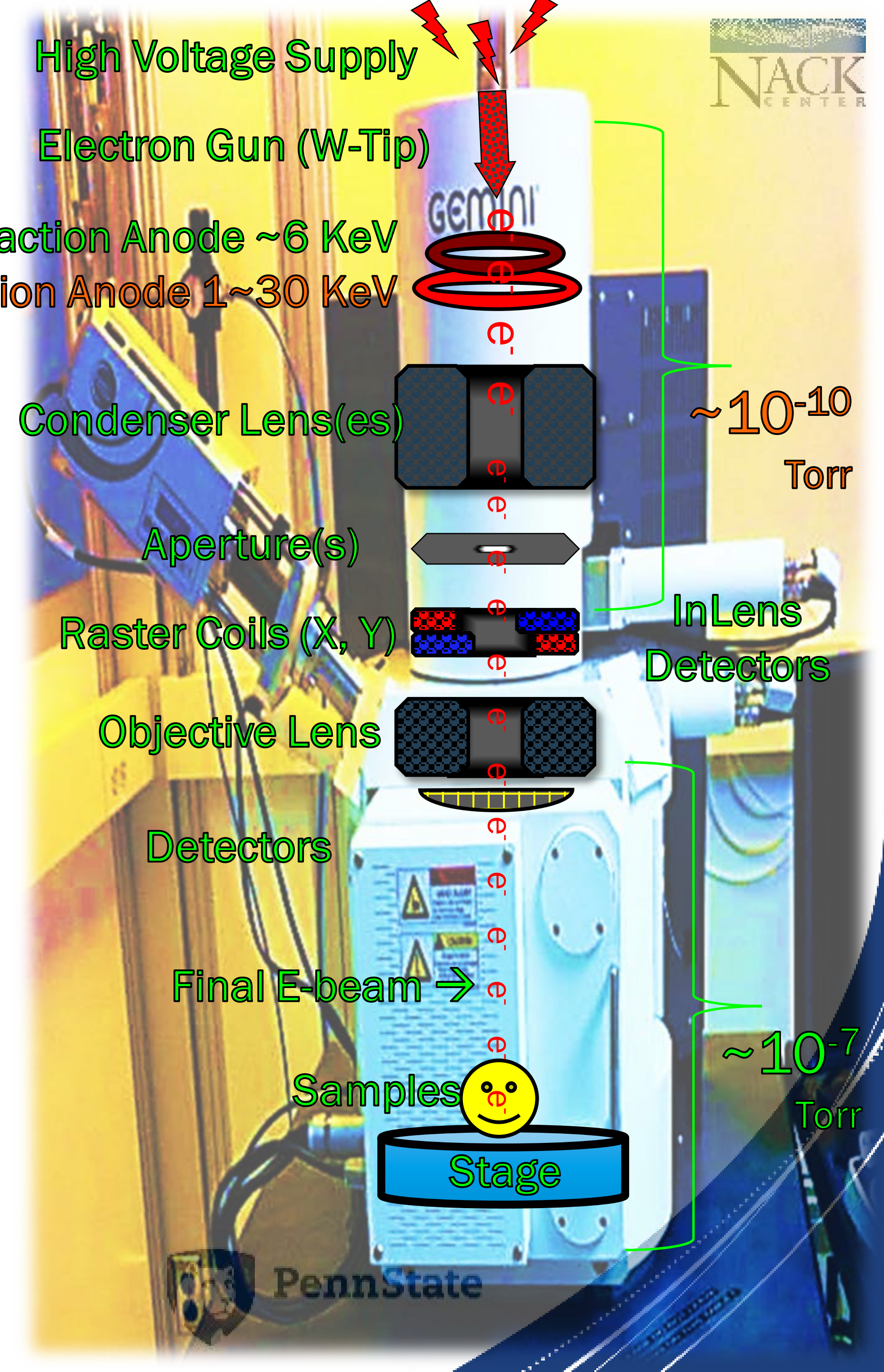
- Electrons are generated by the “tip” and acceleration down the “column”
- Electromagnets demagnify, focus, and raster e-beam across the sample
- Electrons and other radiation are produced when e-beam hits sample

Investigative modes (detectors):

  - Topography (Secondary electrons)
  - Composition (Backscattered electrons)
  - Chemical (Energy Dispersive Spectroscopy—X-rays)

Details:

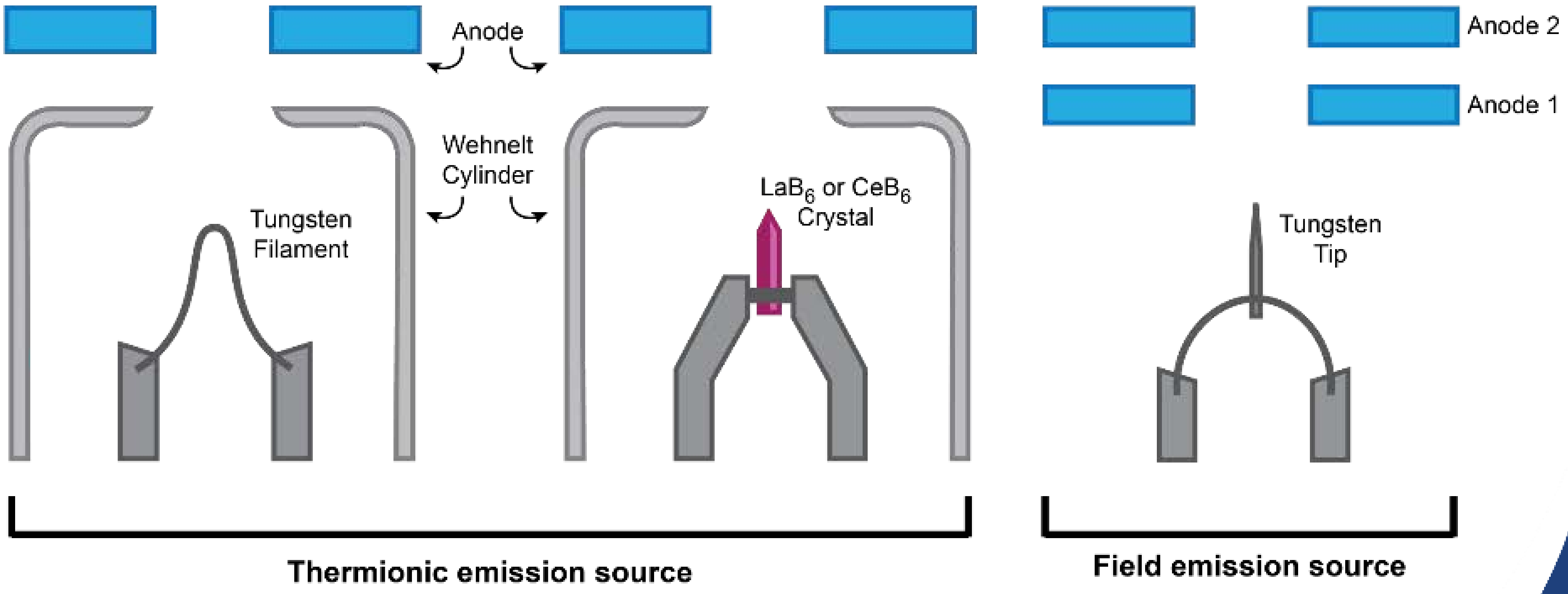
- Samples are placed in vacuum—cells POP!
- Non-conductive samples are coated with gold, or like



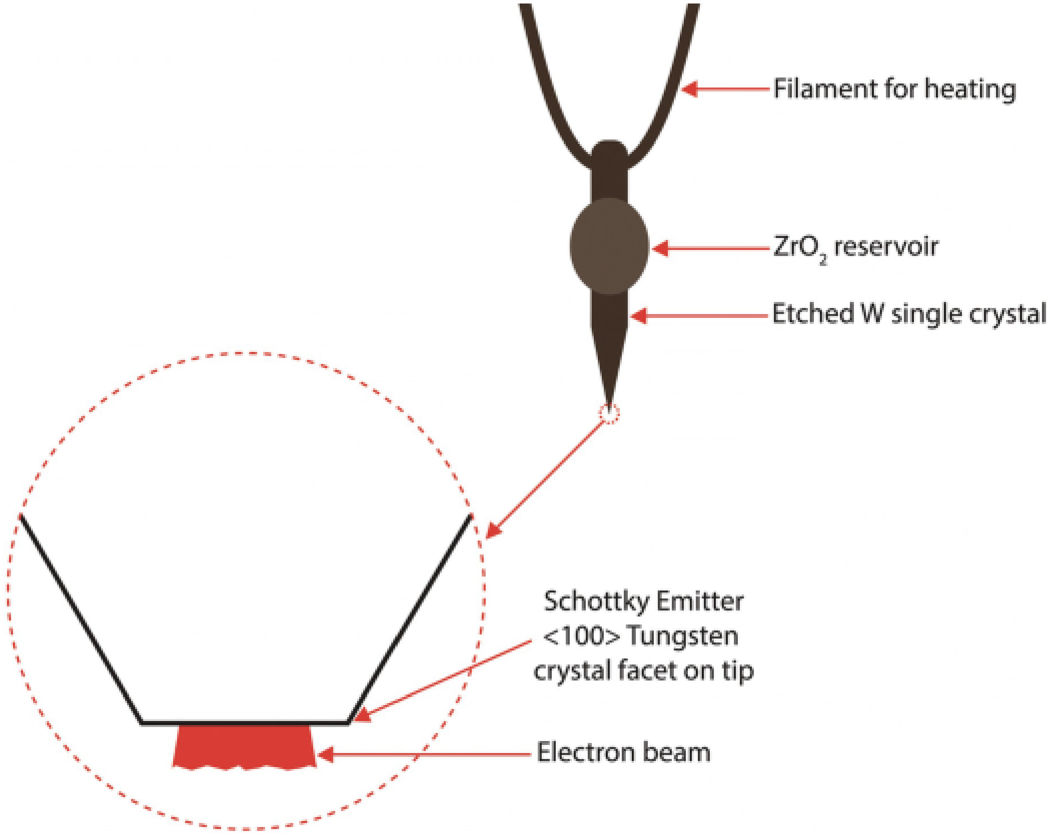
Electron Emission

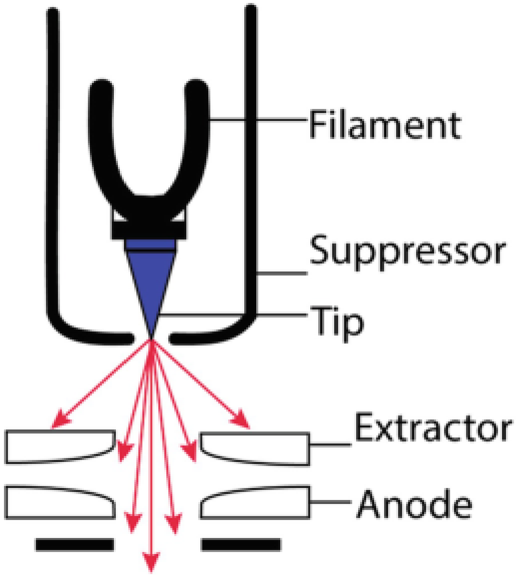
- Goal is to produce the greatest number of electrons with a consistent energy in the smallest diameter beam
- Two emission methods
  - Thermionic: Large current flows through wire inducing resistive heating to the point of incandescence with the emission of electrons
    - Electrons are 'pushed' off tip
  - Field Effect: Large electric potential (5-10kV) concentrates upon small tip geometry enables electrons to surpass tip material work function and emit into free space
    - Electrons are 'pulled' off tip
    - Intrinsically smaller beam → gives better resolution

Electron Emission

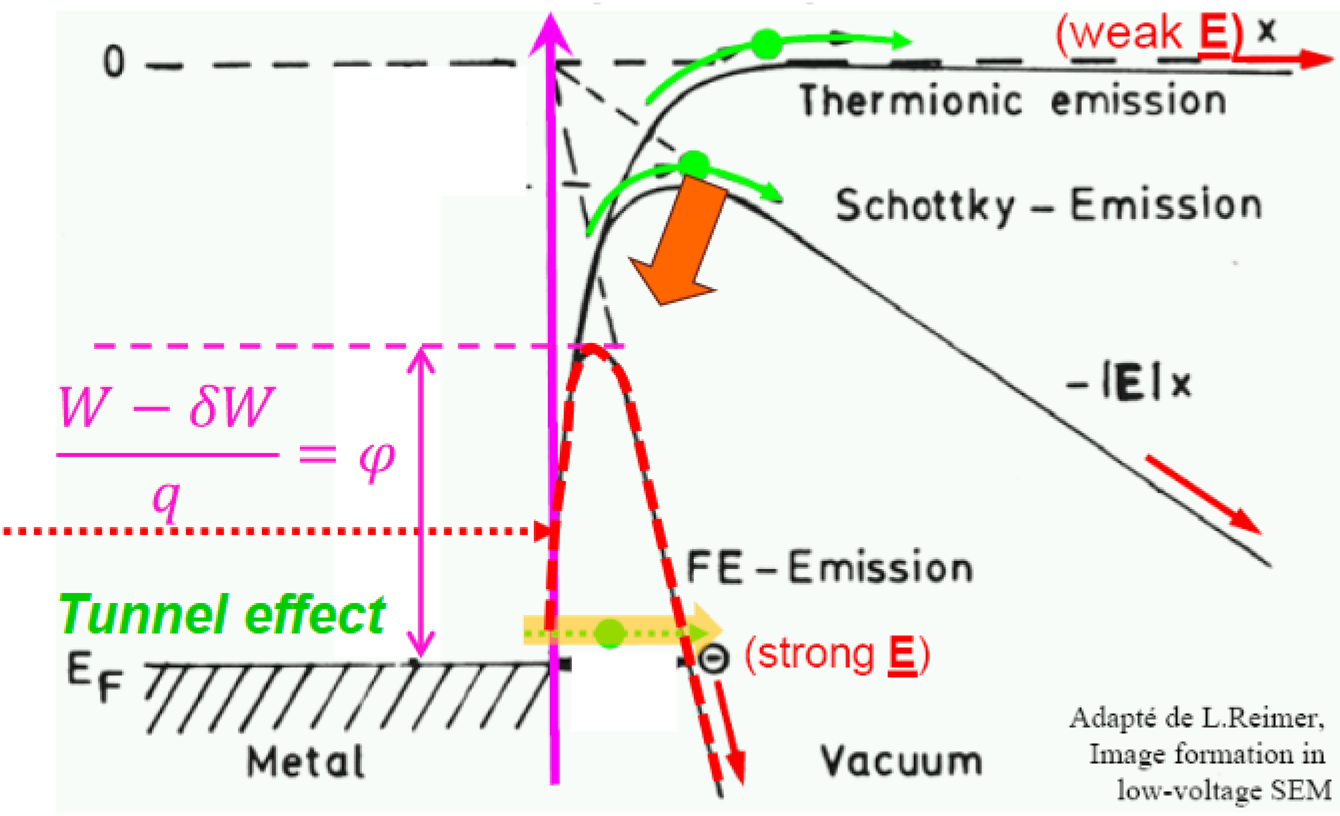


Electron Emission





Electron Emission



Electron Emission

	Thermionic		Field Effect (FEG)	
→	W Hair pin	LaB_6	Cold	Schottky (ZrO)
Life (hr)	50-100	200-1000	>1000	>1000
Flashing	N/A--Replace	N/A--Replace	Daily	Weekly-Monthly
Apparent size. (nm)	25,000-100,000	500-50,000	<100	100-500
Operating Temp.	2,700 ˚C	~2,000 ˚C	25 ˚C	1500 ˚C
Energy Spread (eV)	1-3	1-2	<1	1
Vacuum (Torr)	10^{-5}	10^{-7}	$<10^{-11}$	10^{-11}
Brightness (A/cm^2 Strand)	10^5	10^6	10^8	10^{7-8}
Optimal Mag.	<50 kX	50-100 kX	>>100 kX	>100 kX
Cost	Lowest $100's	Low ~$1,000	High ~$5,000	Highest ~$7,000

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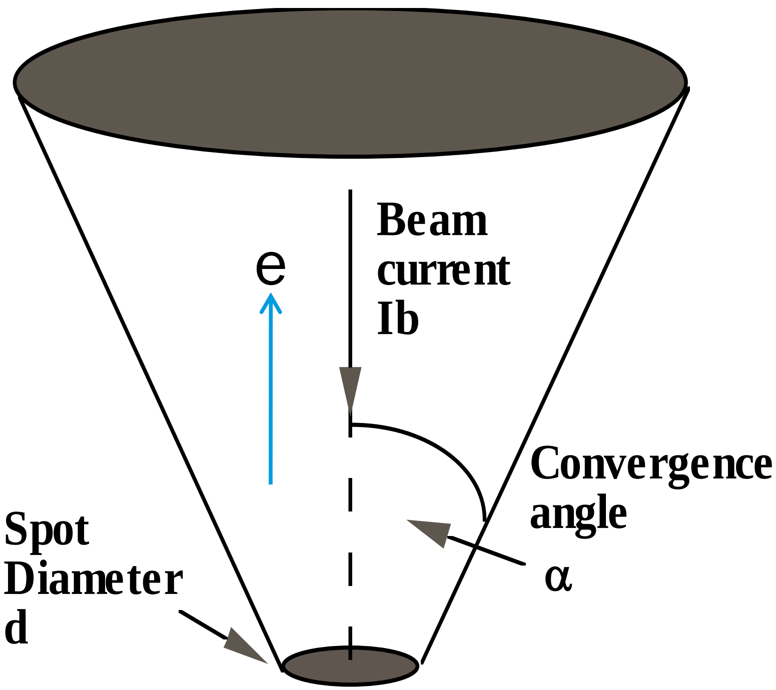
Electron Emission

$$\beta = \frac{4I_b}{\pi^2 \alpha^2 d^2}$$



- Brightness is defined as current per unit area per solid angle, with unit amp/cm^2/steradian.
- Brightness is the **most useful measure** of gun performance.
- Brightness depends on energy, so one must compare different guns at the same beam energy (acceleration voltage).
- High brightness is **not the same as high current.**

E.g. thermionic emission can have very high beam current, but low brightness (due to large d). Most current will then be **blocked by a small aperture** (to limit α) in order to have an acceptable small beam spot onto the specimen for high resolution imaging.

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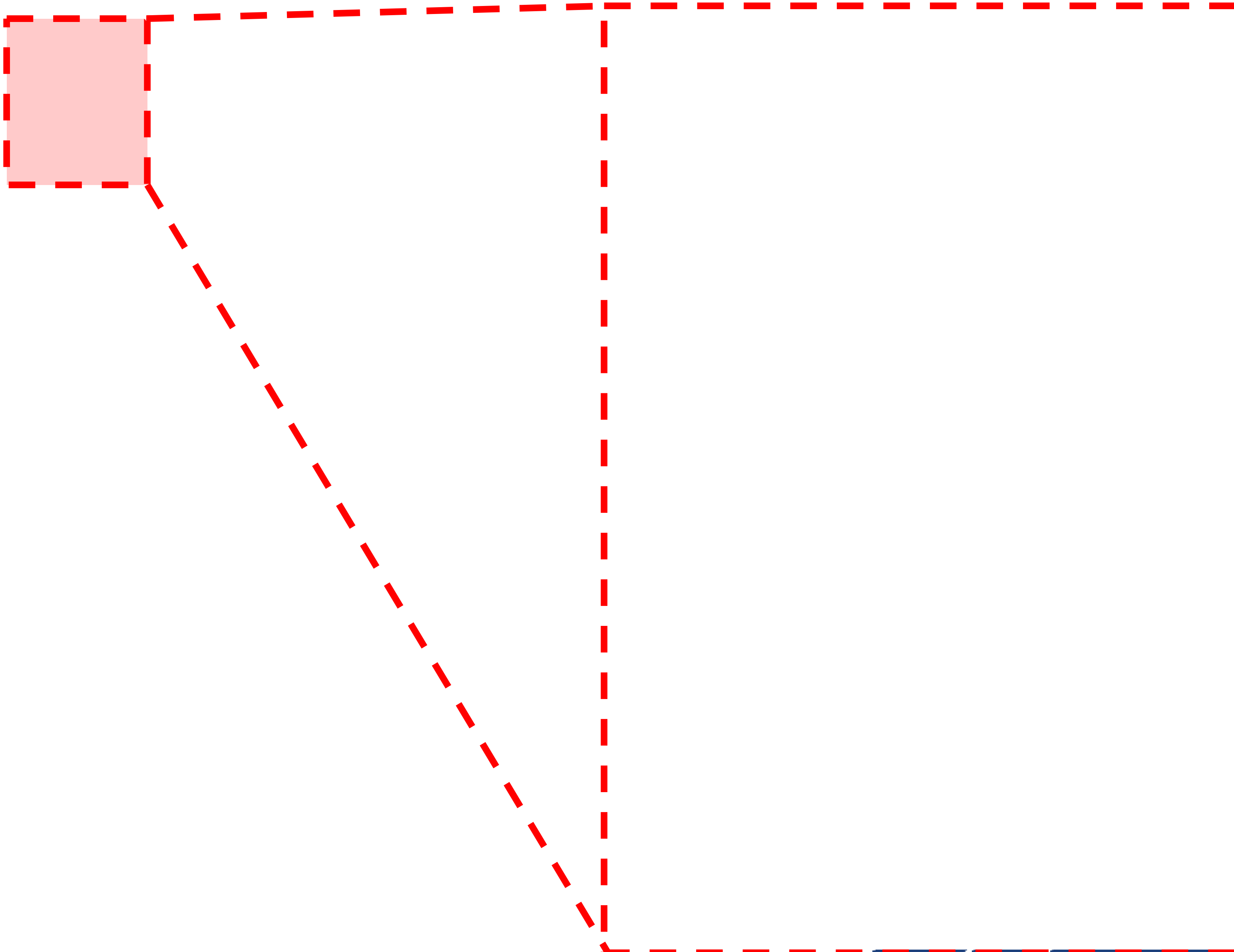
Electron Emission



Electron Emission

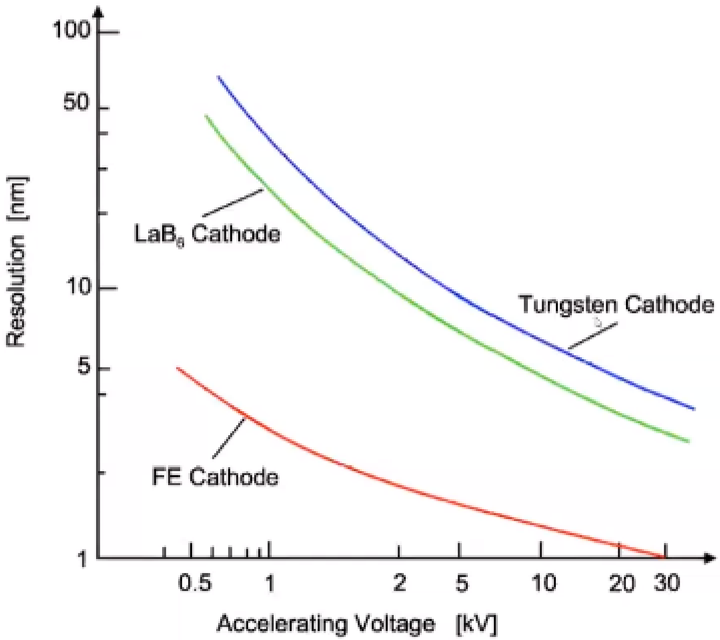
Electron Emission

SEM

- Thermionic emission
- $ ~5,000-10,000's
- Mag. 10 kX to 100 kX
- Spot size 2–10 nm
- Gun vac. ≤10^{-5} Torr

FESEM

- Field emission gun (FEG)
- $ 50,000-100,000's
  - Zeiss 55 ~$500,000
- Mag. 100 kX to 1 MX
- Spot size < 2 nm
- Gun vac. ≤10^{-8} Torr



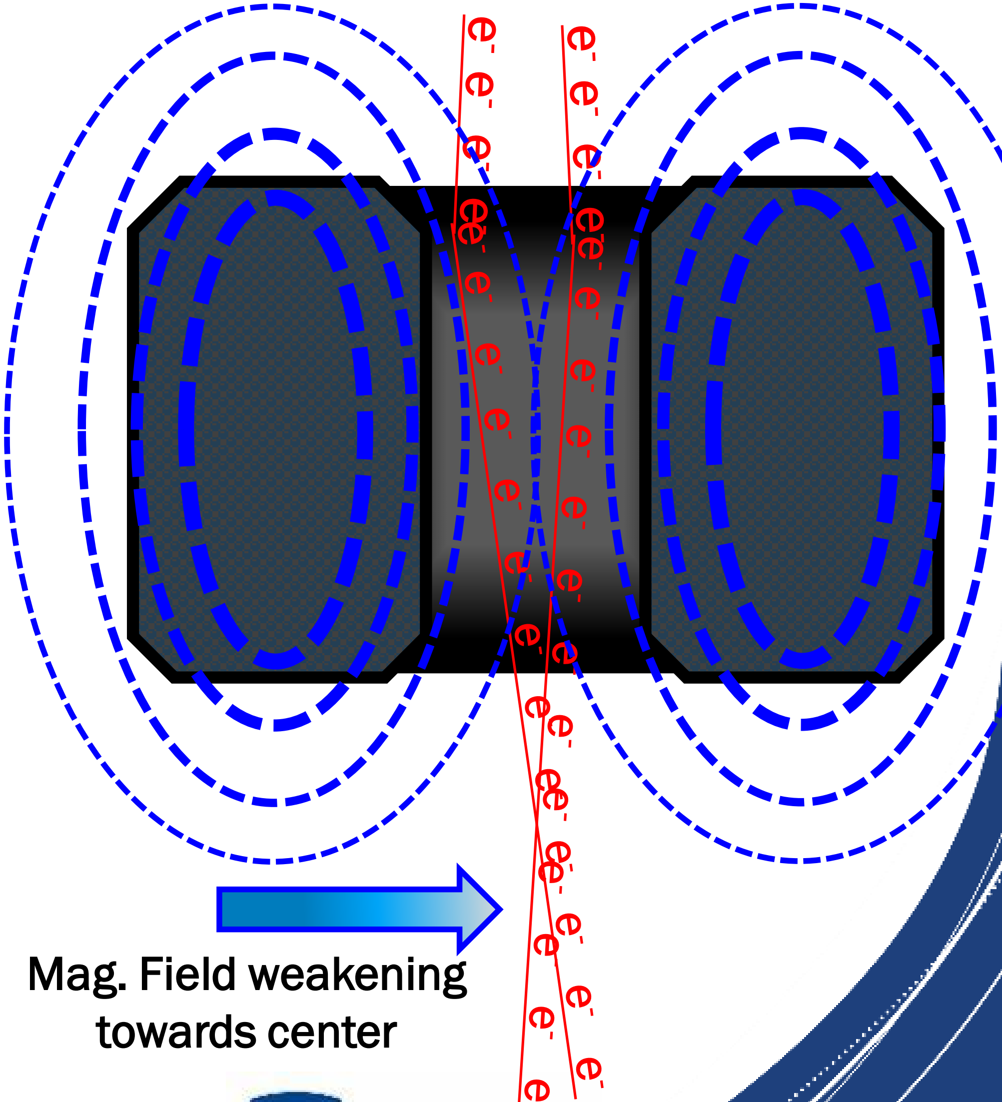
Different electron guns allow for different spatial resolutions.

For high resolution imaging (spatial resolution of some nm) FE cathodes are absolutely recessary !

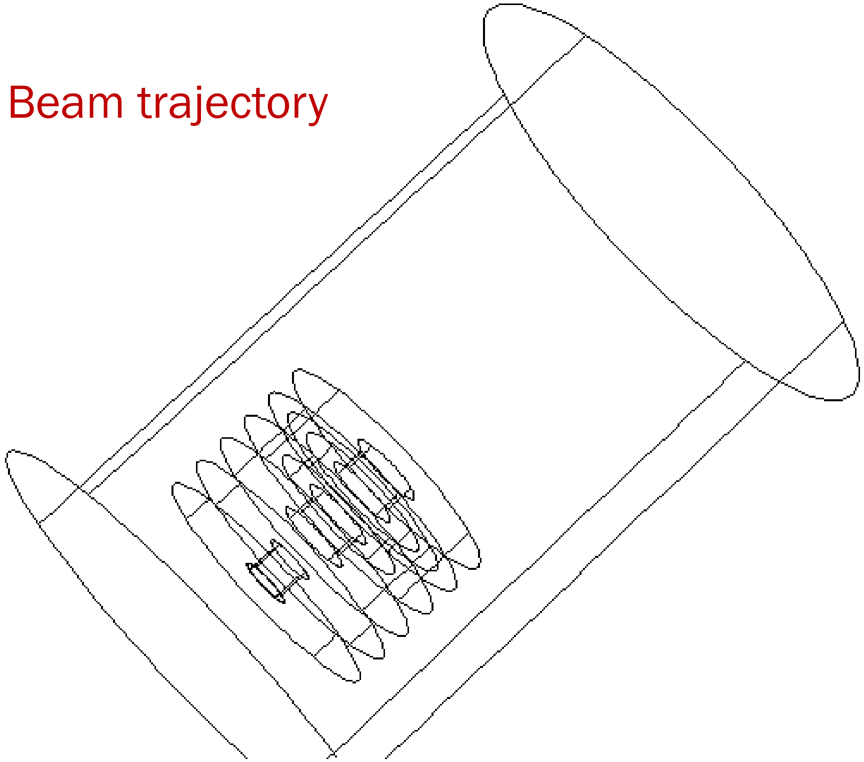
Electromagnetic Lens

Electromagnetic Lens

- current through coils (Cu) generates magnetic field
- Mag. field deflects electrons to focus beam, etc.
  - Intensity of filed determines degree of change
  - Solid state (no moving parts)
- Much "weaker" than optical lenses
- very poor quality (compared to optical)

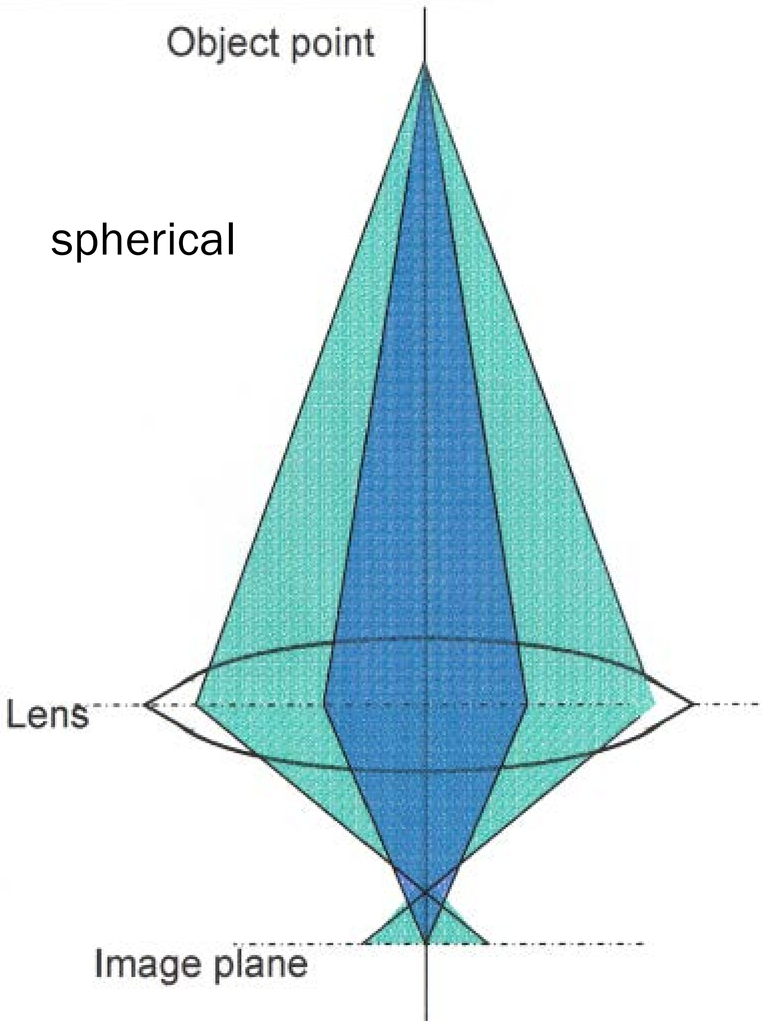


Electromagnetic Lens

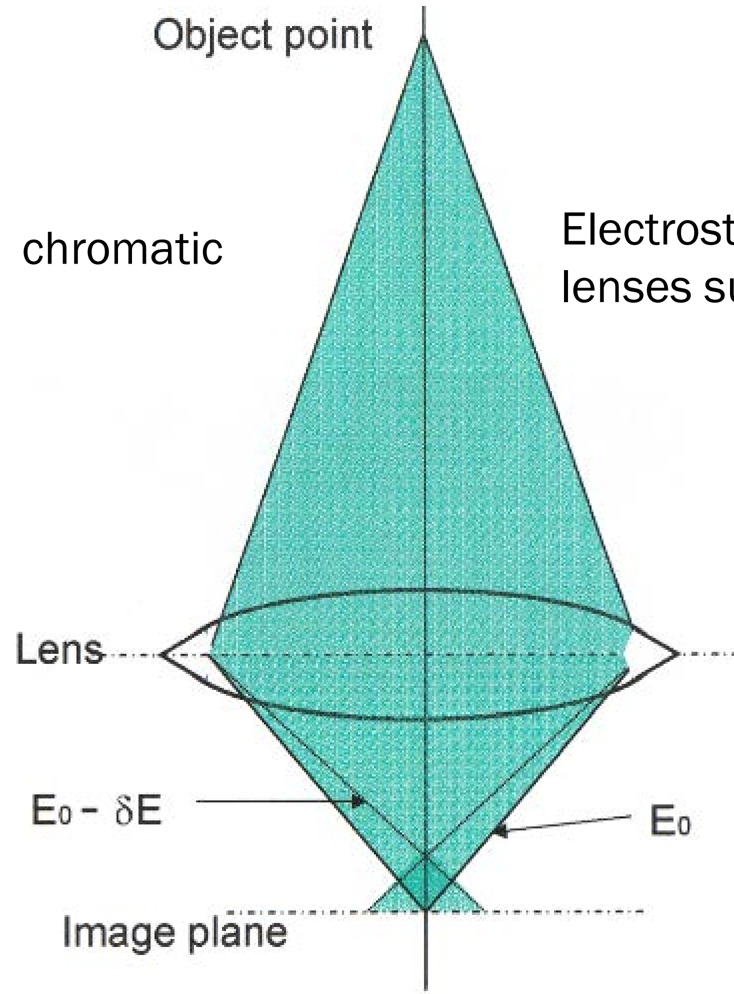


Electromagnetic Lens

spherical



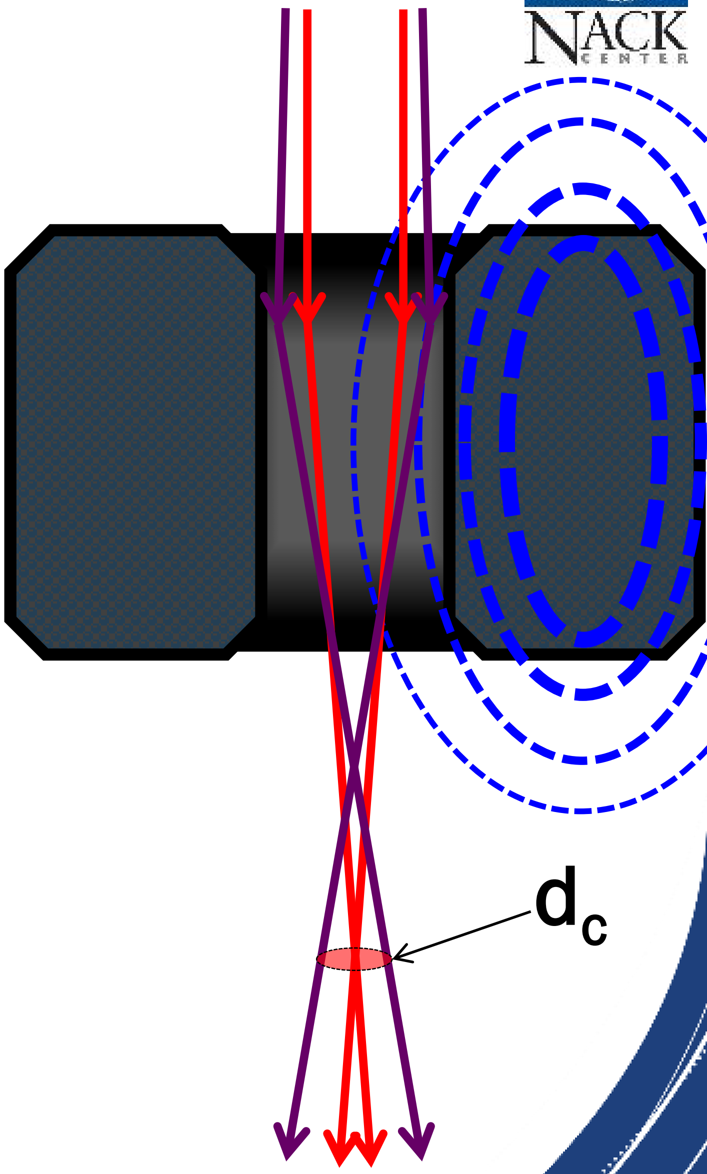
chromatic



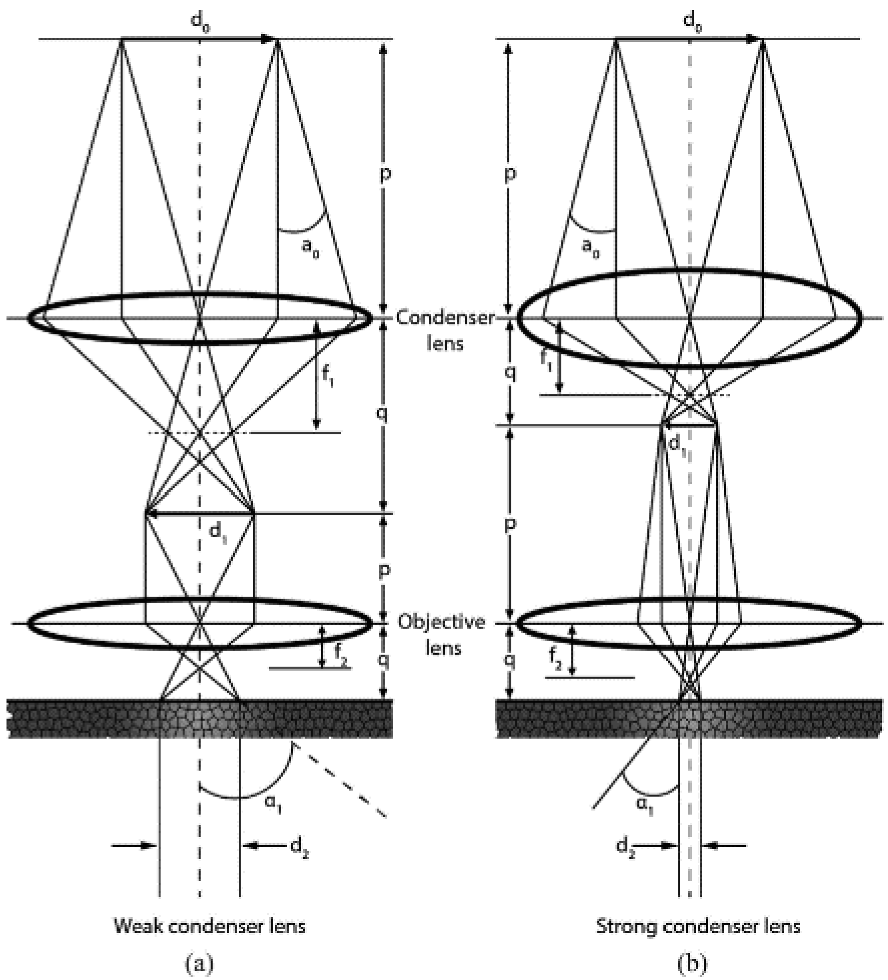
Electrostatic lenses suffer more

Electromagnetic Lens

- Spherical Aberration (A. K. A. Angular)
  - Electrons of different lateral points forced through the lens by different incident angles converge at separate axial focal points
  - Recall: apparent size
- Chromatic Aberration (A. K. A. Temporal)
  - Electrons emitted with different energies are deflected dissimilarly by mag. field
- Aberrations lead to a Disc of Minimal Confusion (d_c)
  - Minimal lateral spread electrons can be focused to by lens
  - Essentially is probe diameter



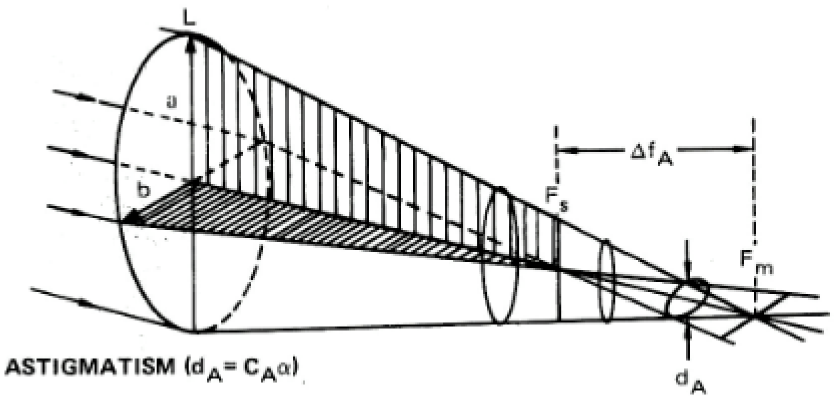
Electromagnetic Lens

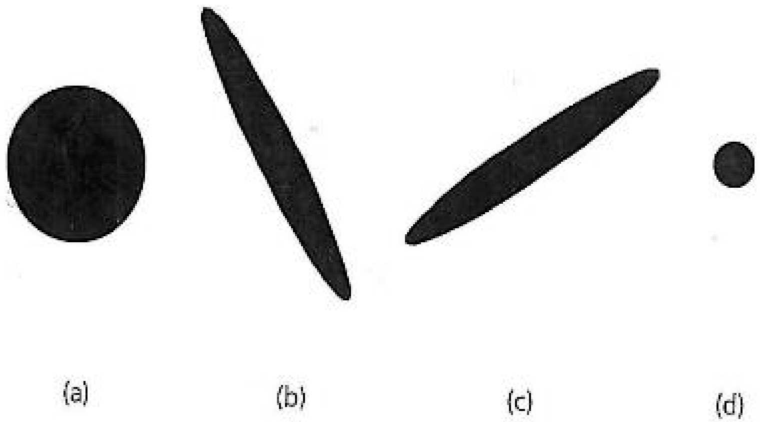


Larger beam probe, smaller magnification
High current passes through=> High signals but low resolution

Smaller beam probe, higher magnification
Low current passes through=> Lower signals but higher resolution

Electromagnetic Lens



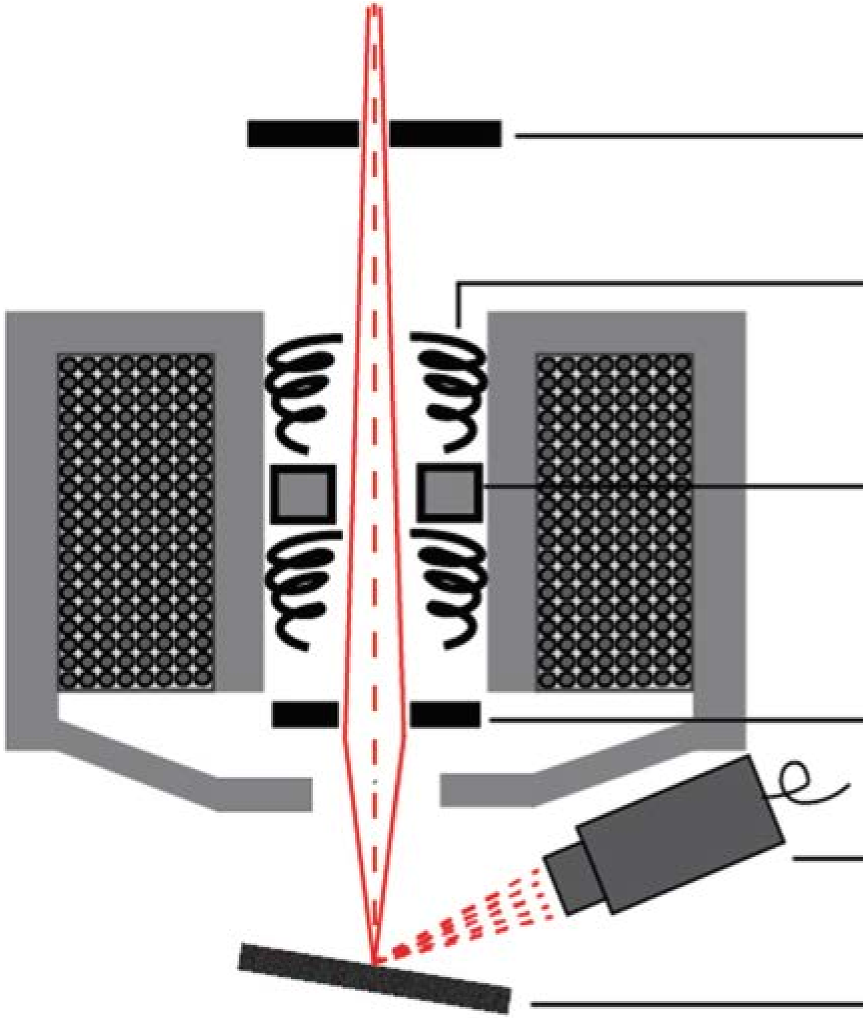


Astigmation:
focal points for x- and y-directions are different

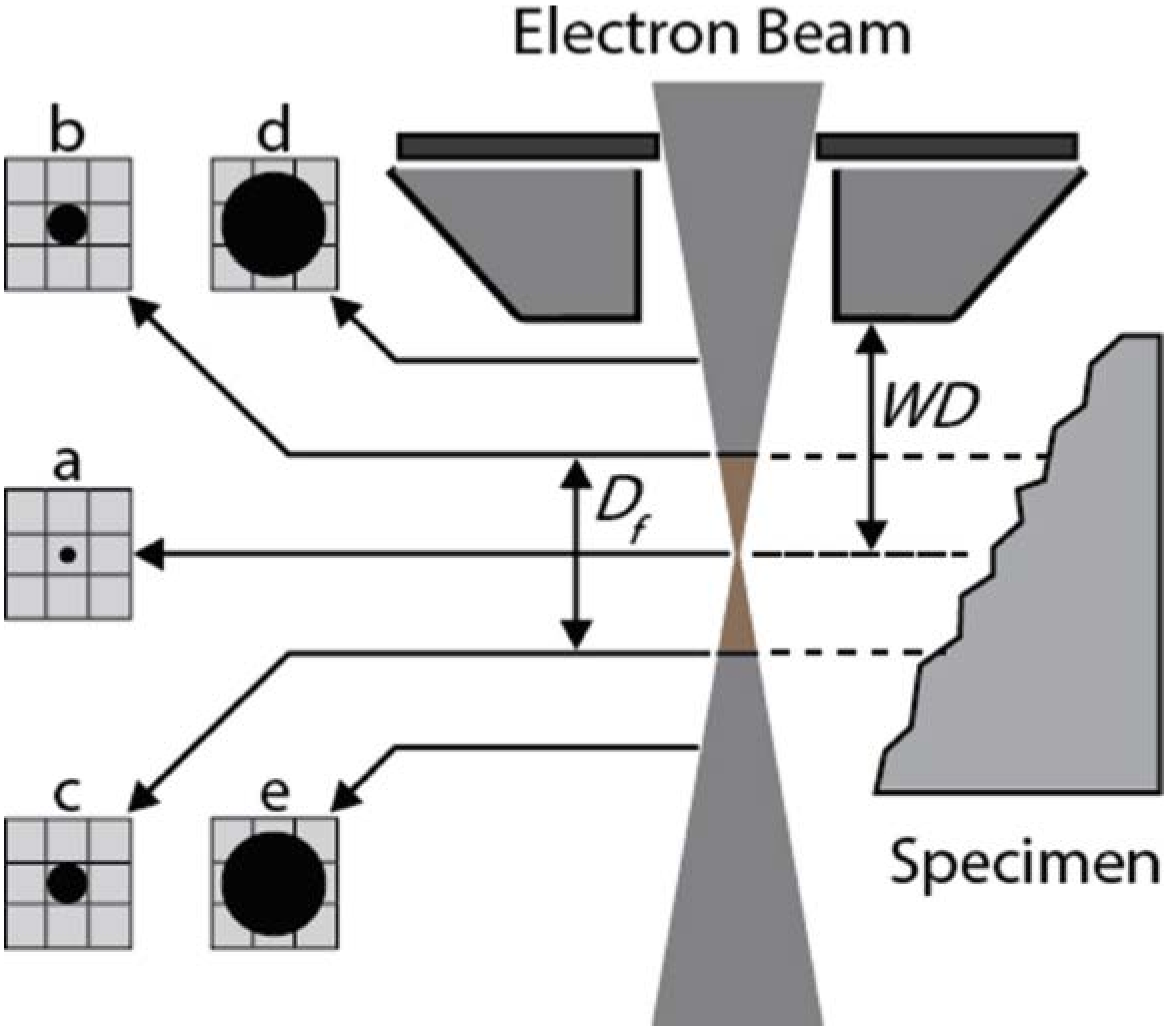
Beam shape at different planes

- Every time one switch on or adjust an electron lens (magnetic, not electrostatic lens), the **magnetization** of the metal in the lens **changes.**
- Because of hysteresis, the lens **never quite goes back** to where it was.
- The lens will then have non-round features due to different magnetization around the pole-piece, which is the focusing part of the electron lens.
- Stigmators eliminate/compensate astigmation by adding a small quadrupole distortion to the lens => Focus in one direction, defocus in the other.

Electromagnetic Lens



Electromagnetic Lens



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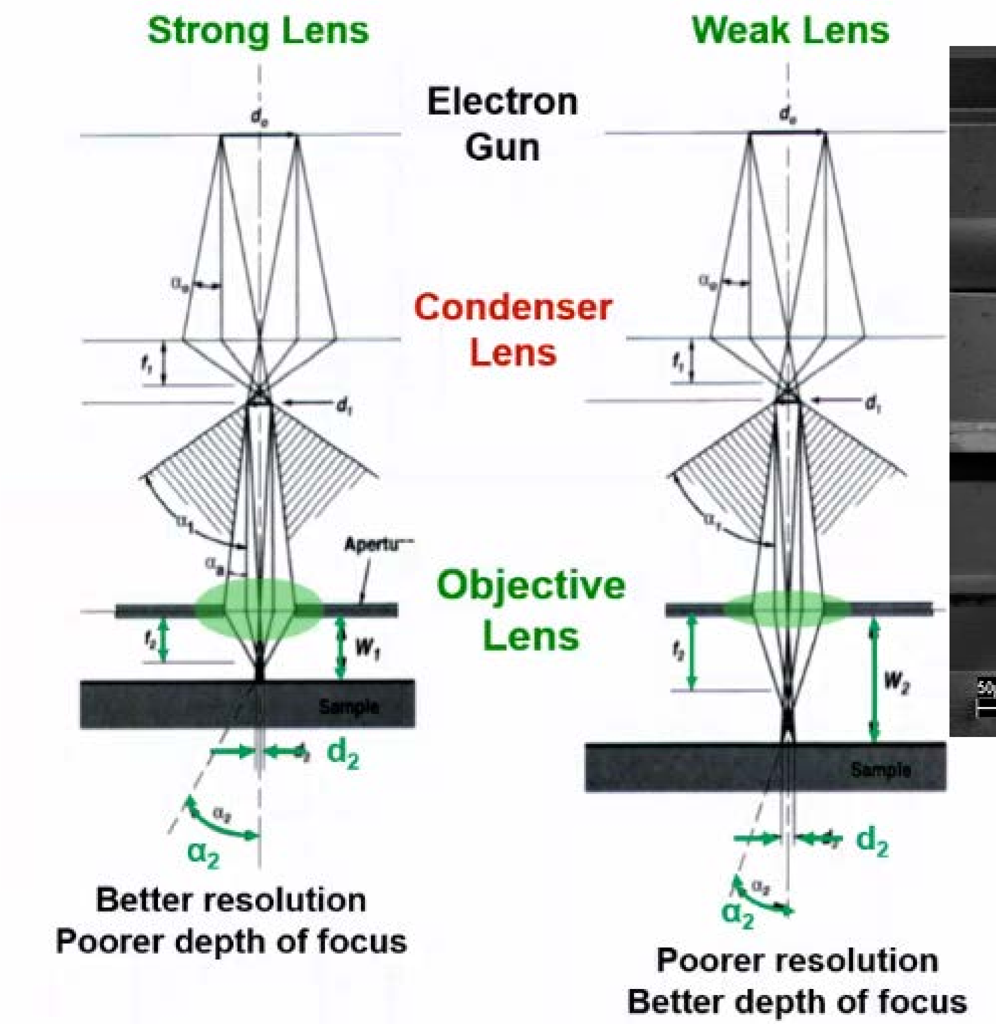
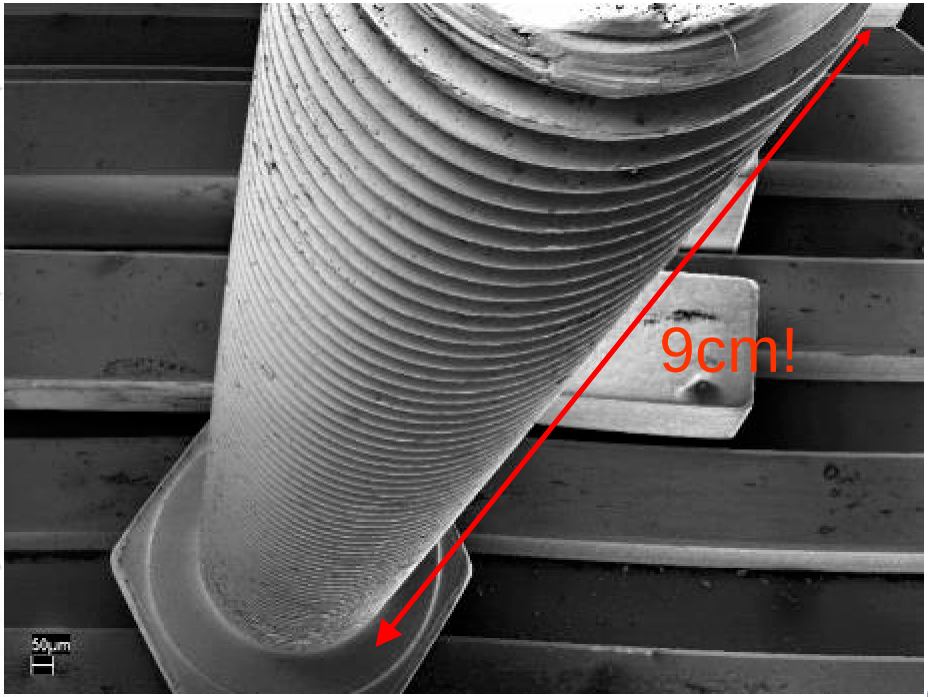
Electromagnetic Lens





M8x90 screw;
SE-detector, HV,
10 keV

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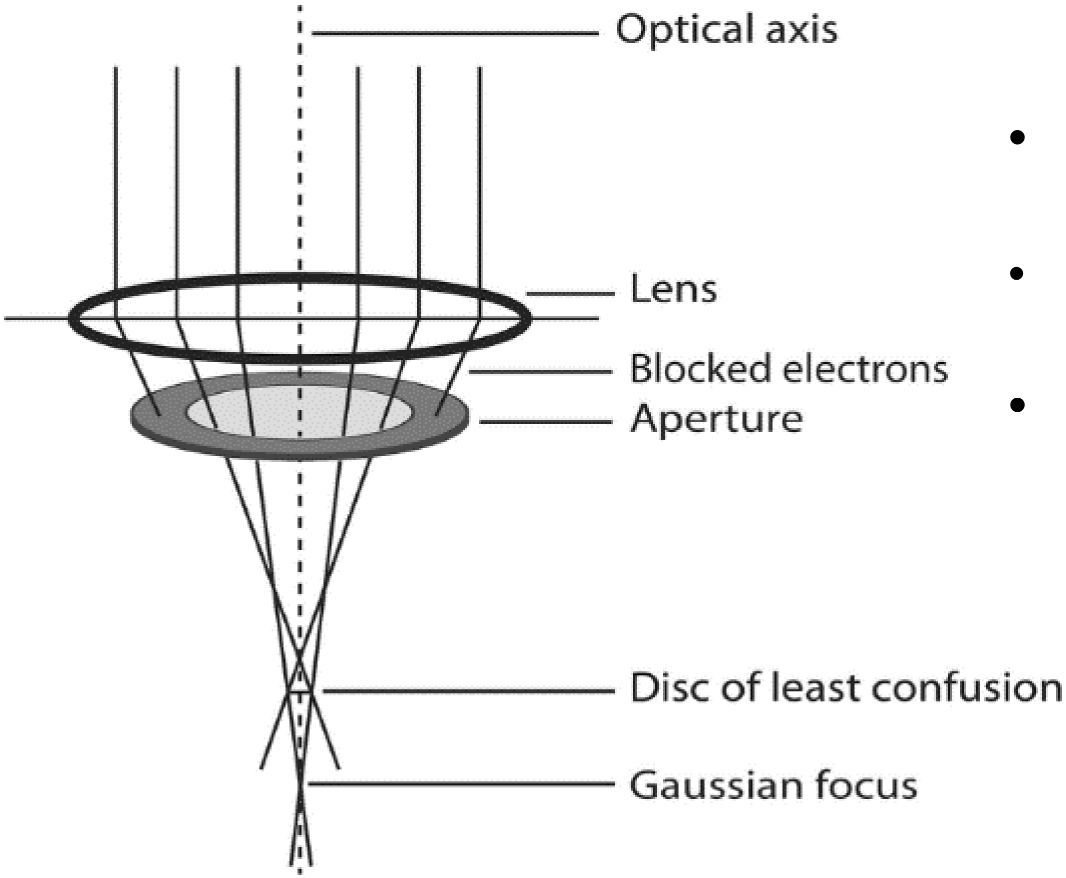
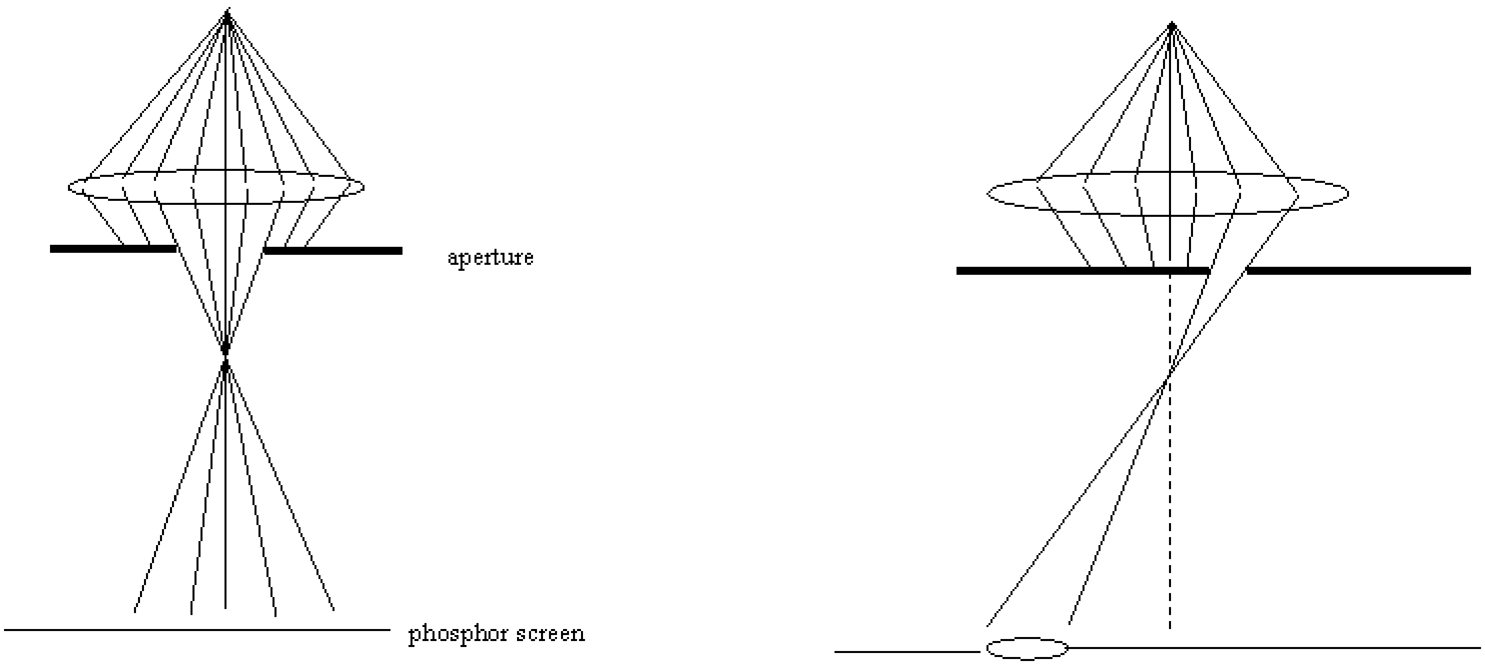
Apertures



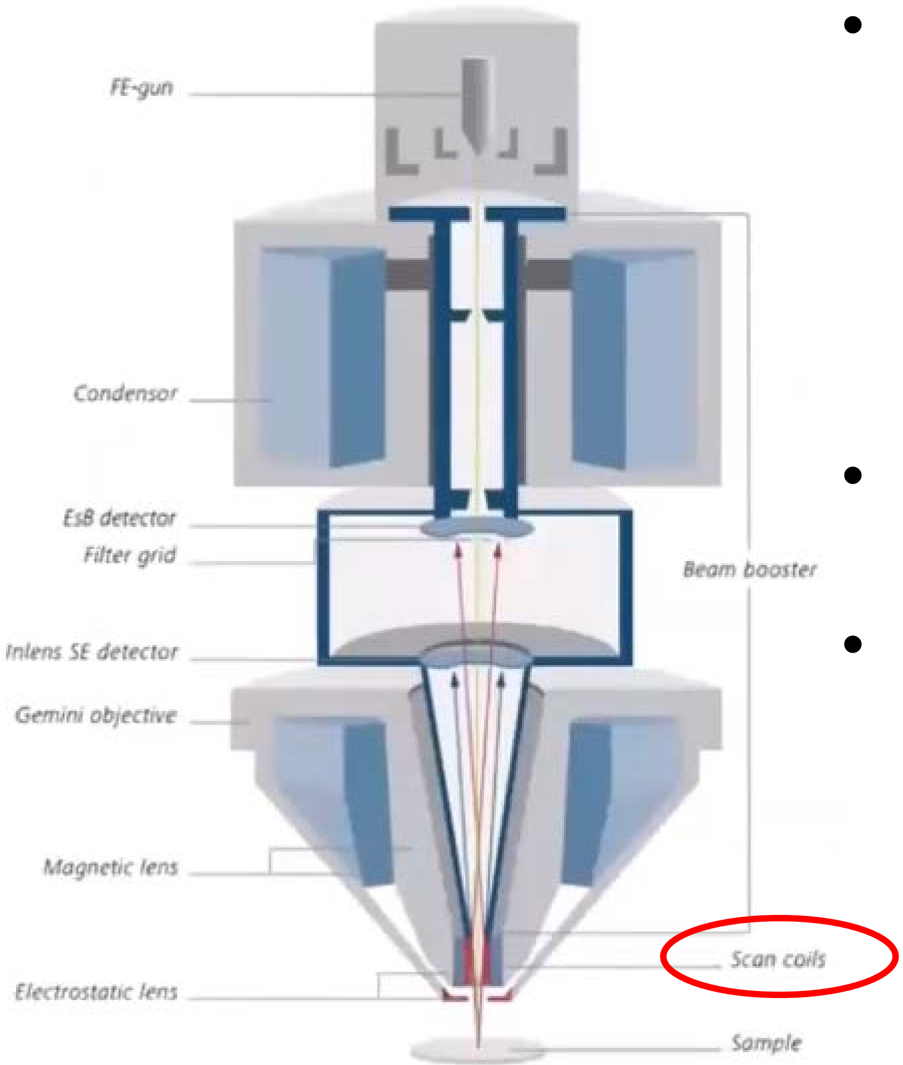
- Series of micron-scale (30 µm) holes in metal disk block stray parts of beam
- Reduces effects of lens aberrations
- Must be aligned to center of e-beam (X & Y coordinates)
- If misaligned will cause raster pattern to shift when objective lens (focus) is adjusted
  - Monitor image show shifting
  - Wobble: automating oscillation of focus will make image ‘bounce’ in direction of misaligned aperture

Apertures



Aperture Misalignment Problem

Raster Coils



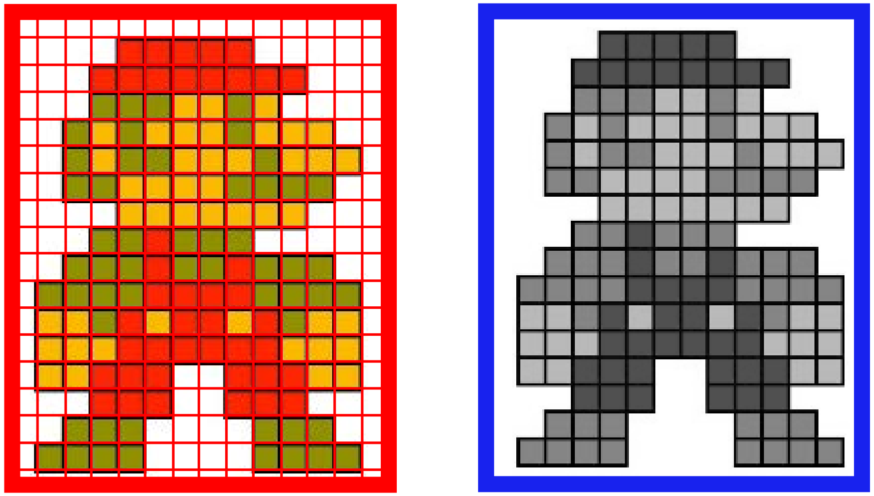
- Rasters e-beam across sample surface
  - Follows discrete X & Y coordinates
  - Coordinates are time "stamped"
- Detectors register resulting radiation
- Software compiles coordinate and intensity, according to time, and the 2D image is formed

Raster Coils

- Raster coils dictate magnification in “scanning” electron microscopes
  - Mag. = Image pixel width / Raster width

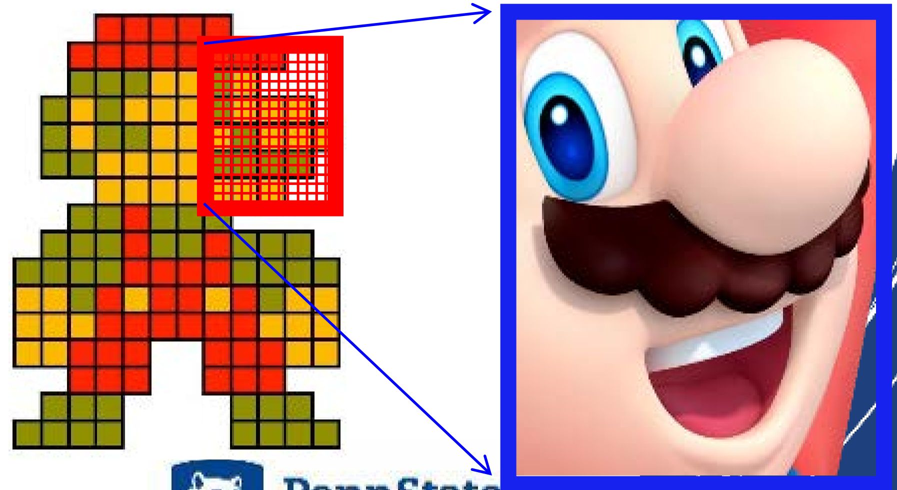
Larger raster areas = Low mag.

Raster on Sample | Pixels on screen image



Smaller raster areas = Higher mag.

Raster on Sample | Pixels on screen image



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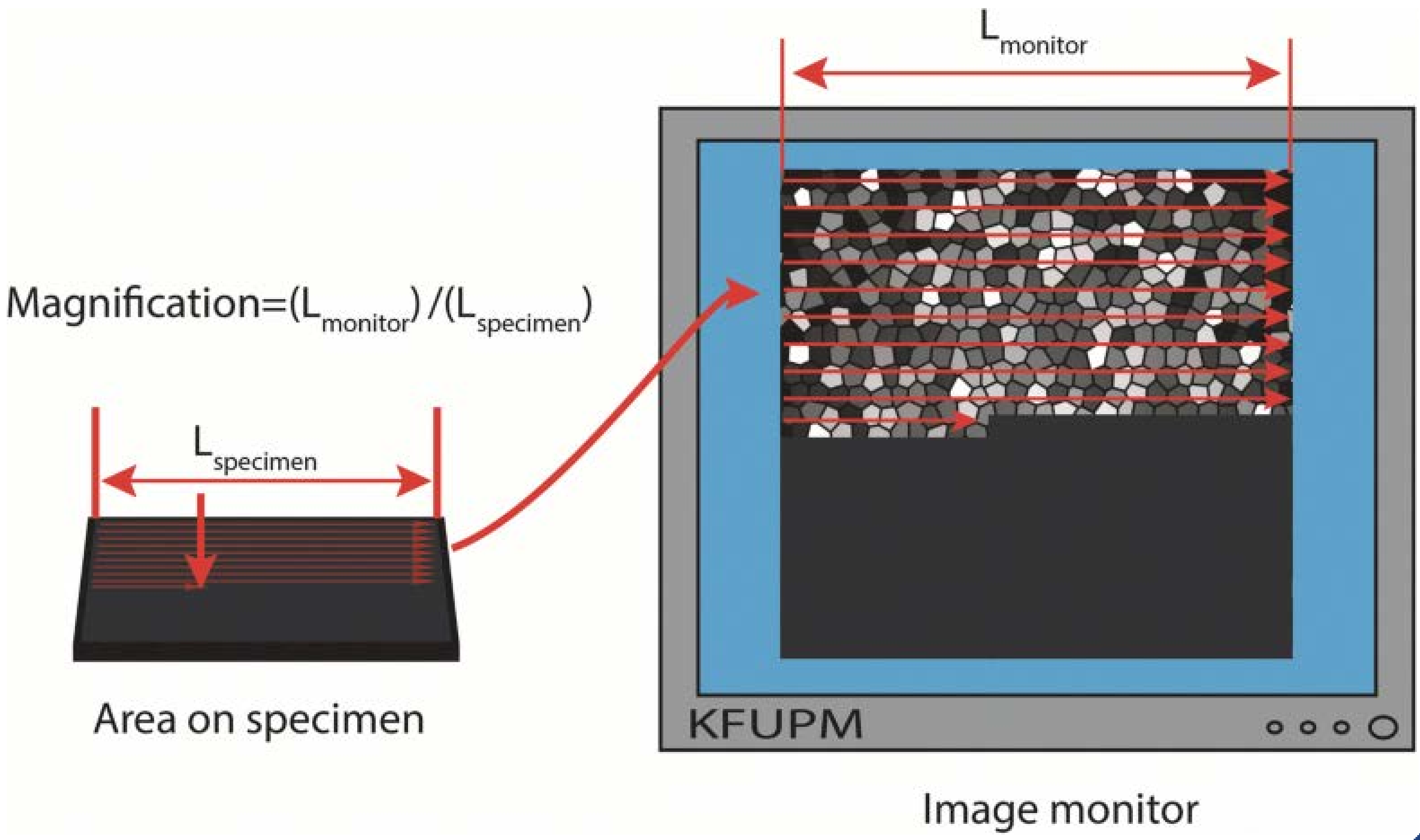
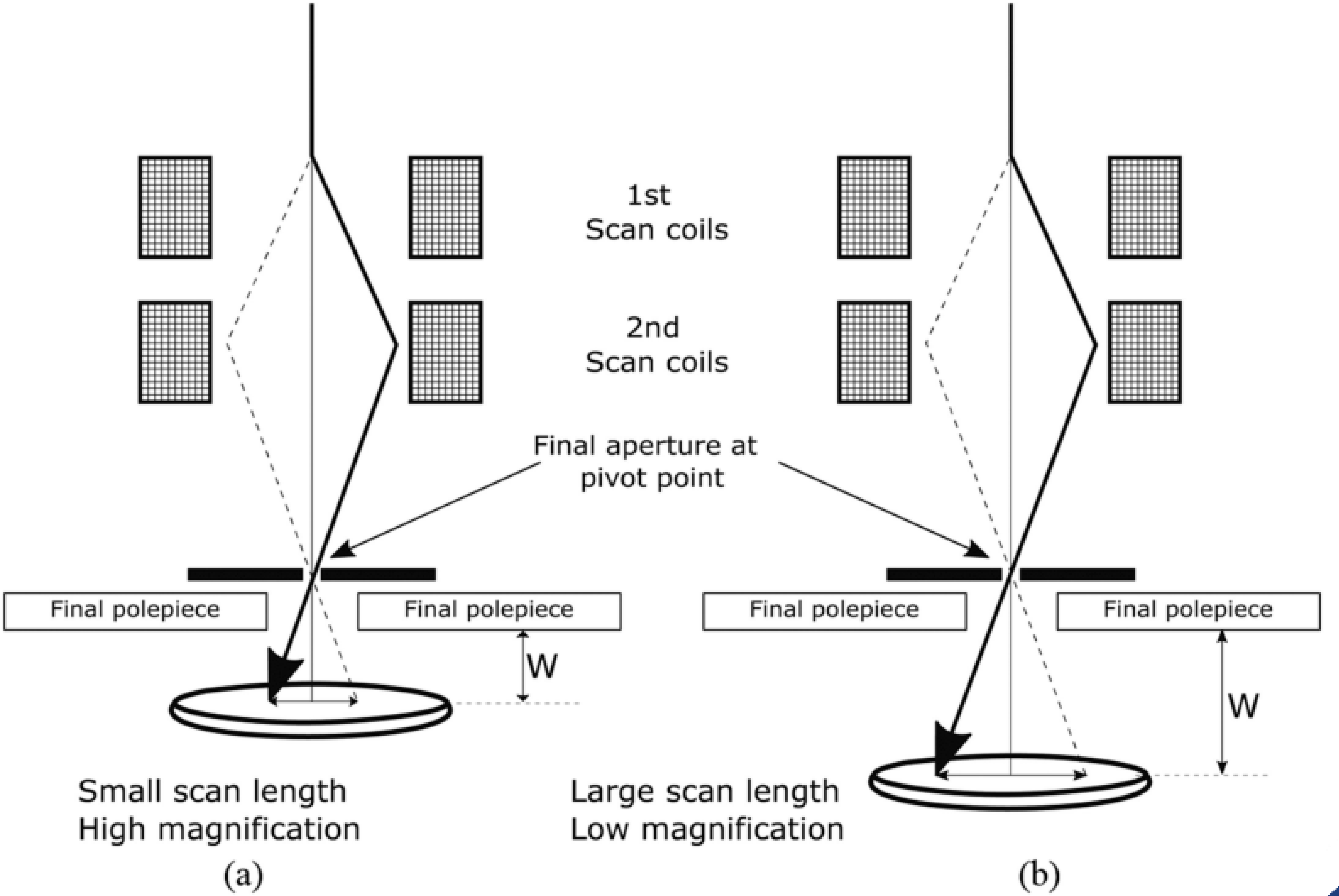
Raster Coils

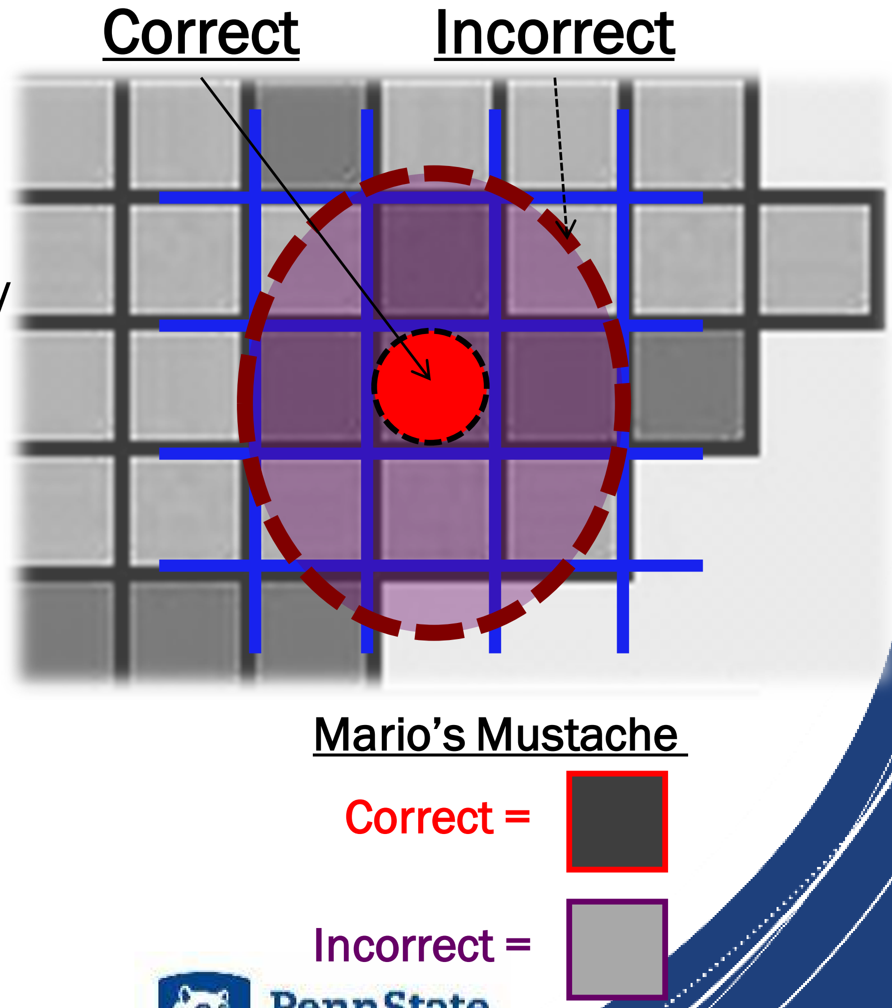


Raster Coils



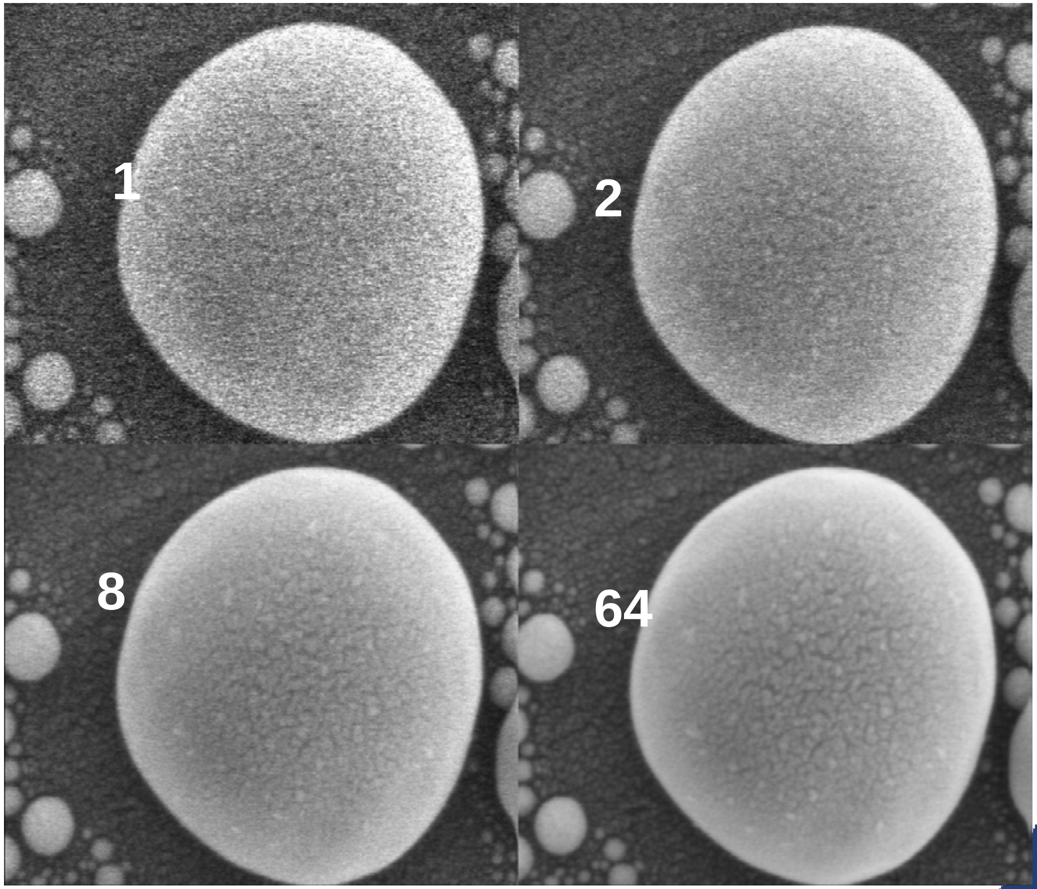
Raster Coils

- Pixel overlap is when the spot size is larger than the raster patterns coordinate
  - Results in erroneous intensity data being read from multiple spots
  - Images appears blurry
  - Corrected by operator adjusting, focus, stigmation, aperture alignment, etc.



Noise Reduction

- The line and frame averaging options for the framestore can greatly reduce noise
- This should not used as an excuse to use beam currents that are too low!
- Whenever possible take a single slow speed scan rather than accumulating multiple high speed scans
- This eliminates blurring due to drift, and distortions in the video amplifier chain and usually produces a higher signal to noise ratio and better contrast
- Higher pixel resolution images require longer acquisition times compared to low pixel resolution images – dwell time should remain the same



Effect of the averaging on noise

Stage

- Electron beam (gun column) are fixed in position—never moves
- Stage with sample move under e-beam
- Allows operator to navigate search samples
- Movement far less precise than e-beam raster

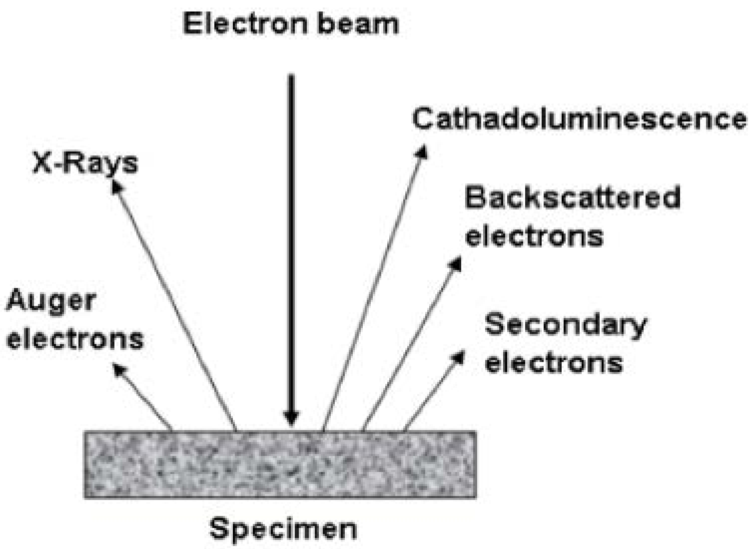
(a) (b)

(a) (b)

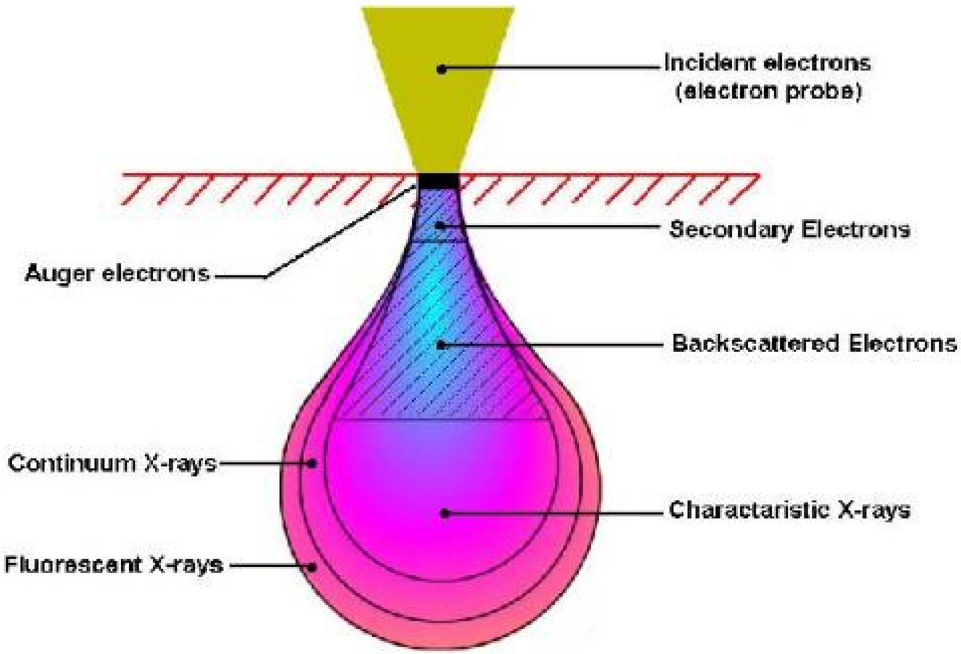
(c)

Beam Sample Interaction

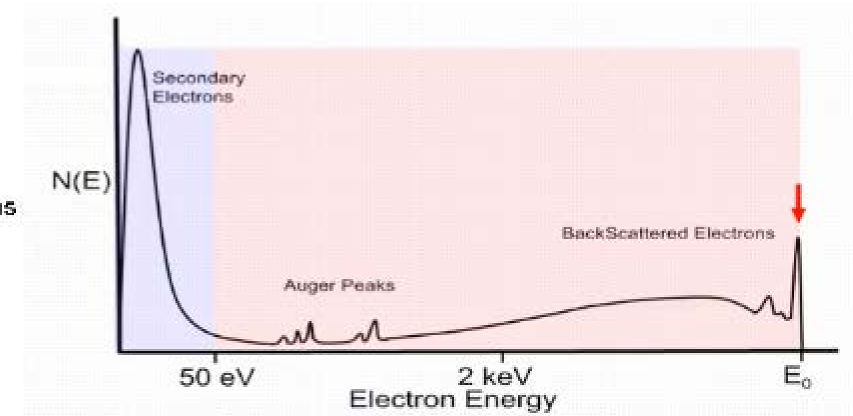
- Contrast from (1) type of byproduct(s), (2) energy level of byproduct, (3) and angle of takeoff/collection
- Incident beam transfers energy to the sample, generating and ejecting different types of beam-specimen interaction species
- Scattering byproduct are collected by detectors to register an intensity signal (brightness of image pixel)
- Types of byproducts
  - Secondary electrons (SE)
  - Backscattered electrons (BSE)
  - X-ray
  - Auger electron
  - Etc.



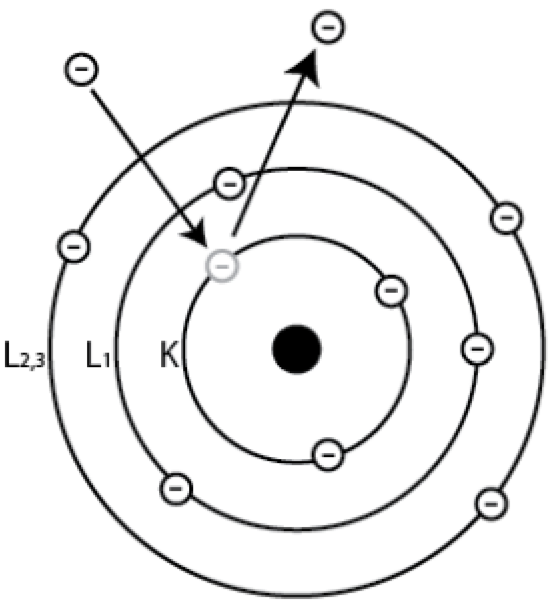
Beam Sample Interaction



Electron Beam Interaction Diagram



Beam Sample Interaction

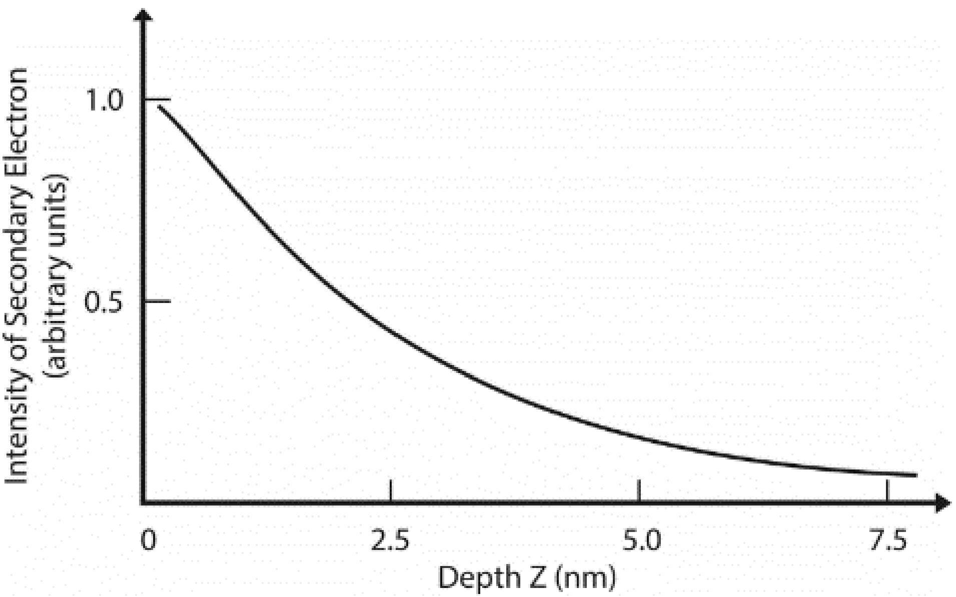


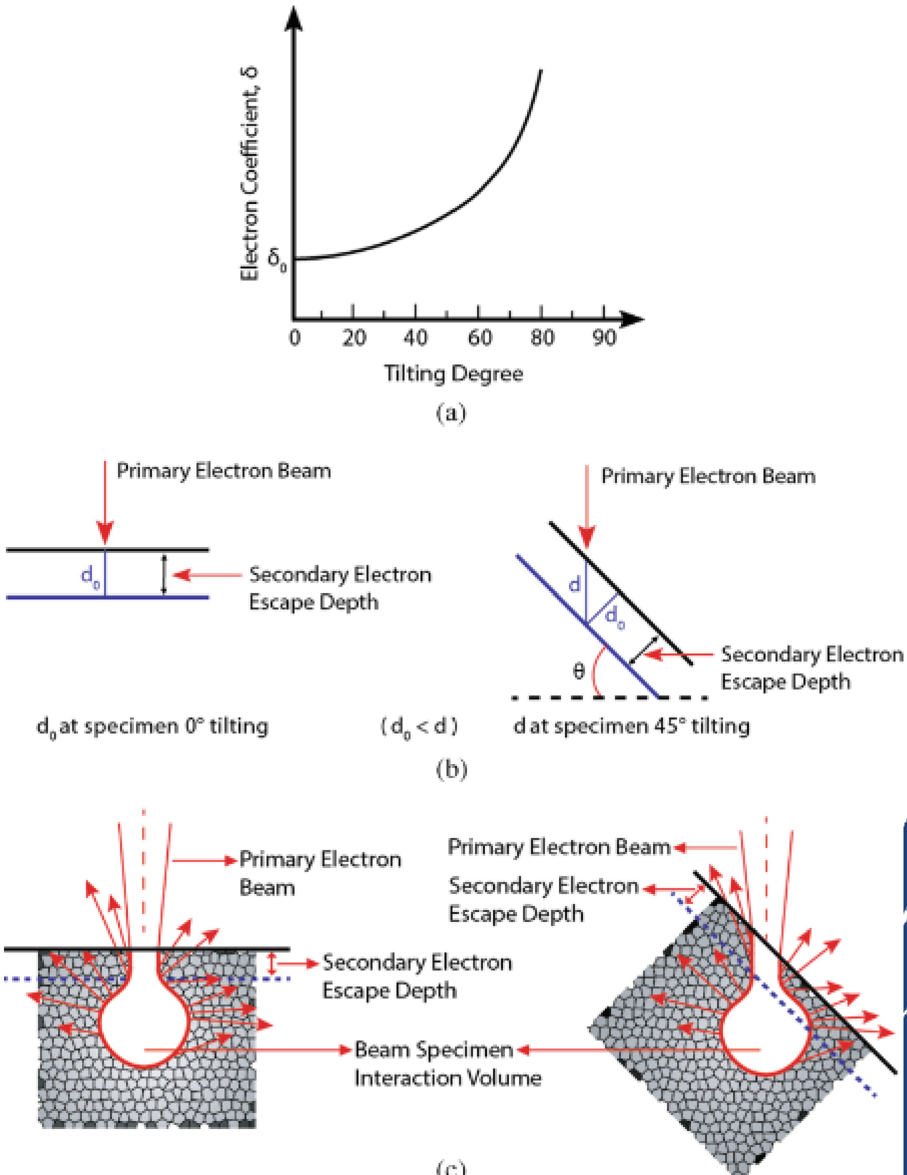
Secondary Electrons

Secondary Electrons

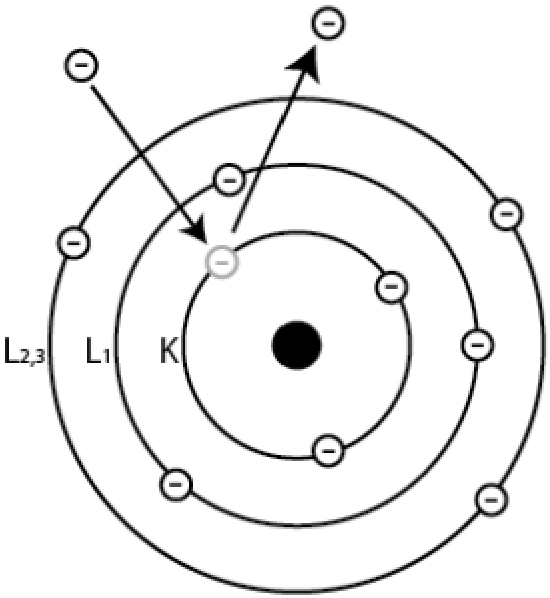
- Incident electron strikes the sample ejecting an electron from the sample
- Several secondary electrons can be produced from one incident electron
- Secondary electrons are low energy
  - <50 eV (most ~10 eV)
  - Shallow escape depth (5 nm)
  - Highly influenced by the topography of the sample
- Detectors may be biased (+ 1 kV) to collect adequate signal

Beam Sample Interaction

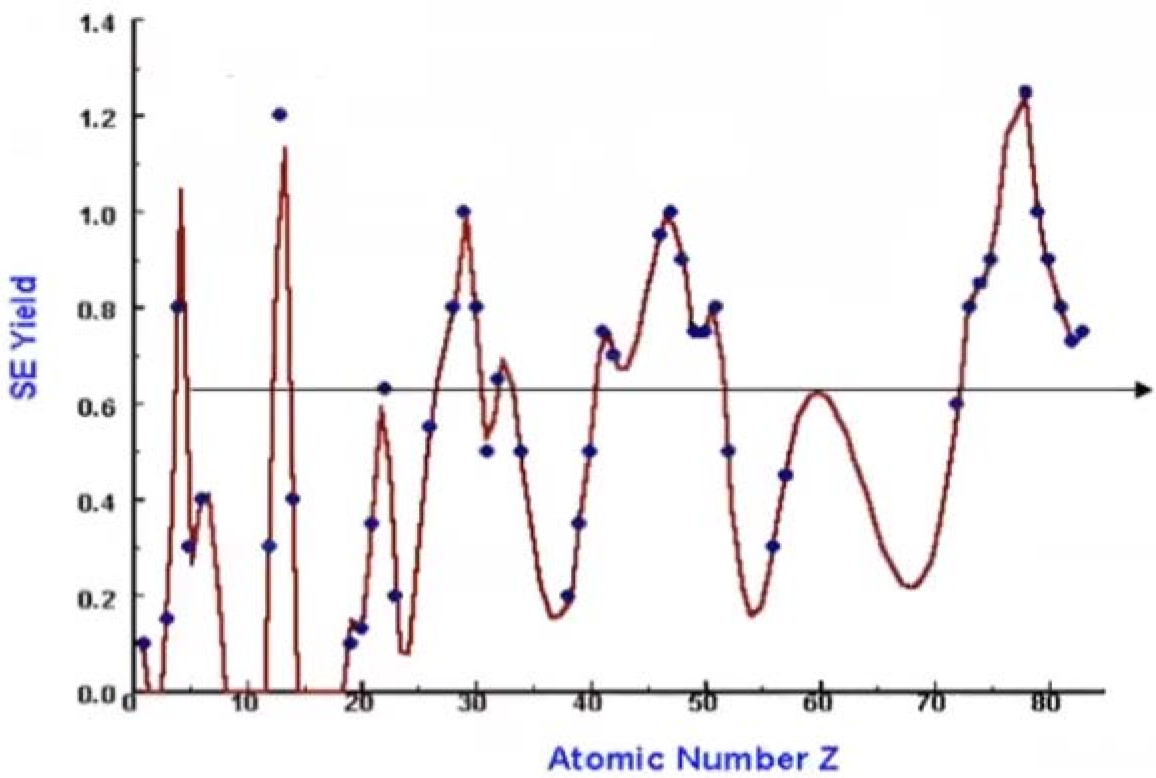




Beam Sample Interaction

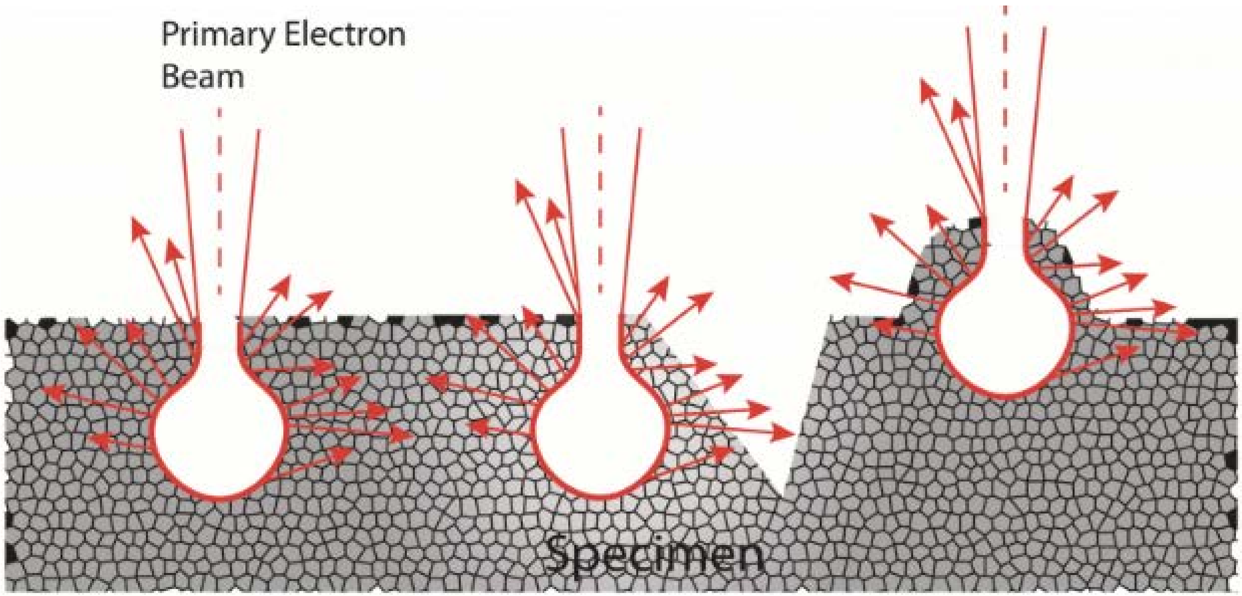


Secondary Electrons

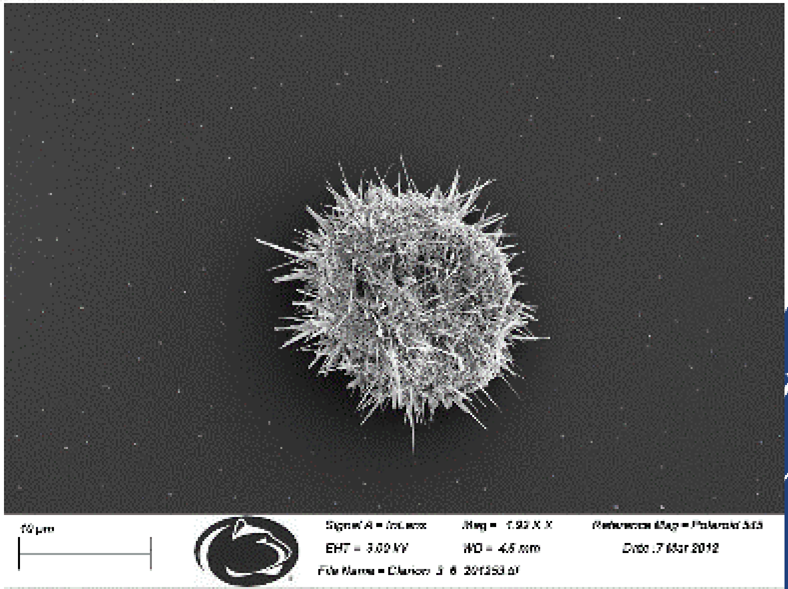


Variation of SE yield at 2keV with atomic number

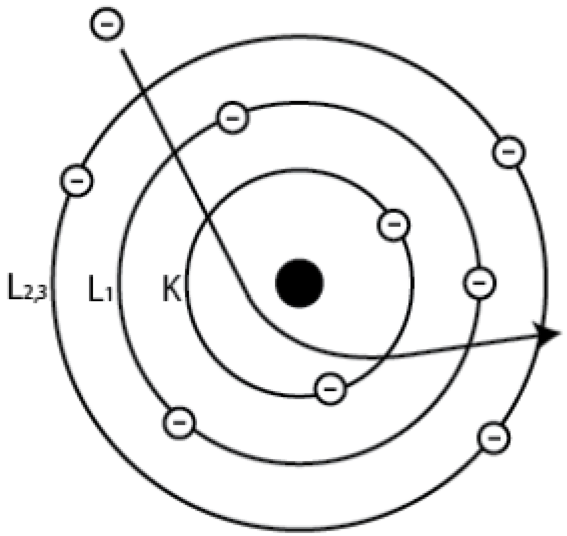
Beam Sample Interaction



Edge Effect



Beam Sample Interaction

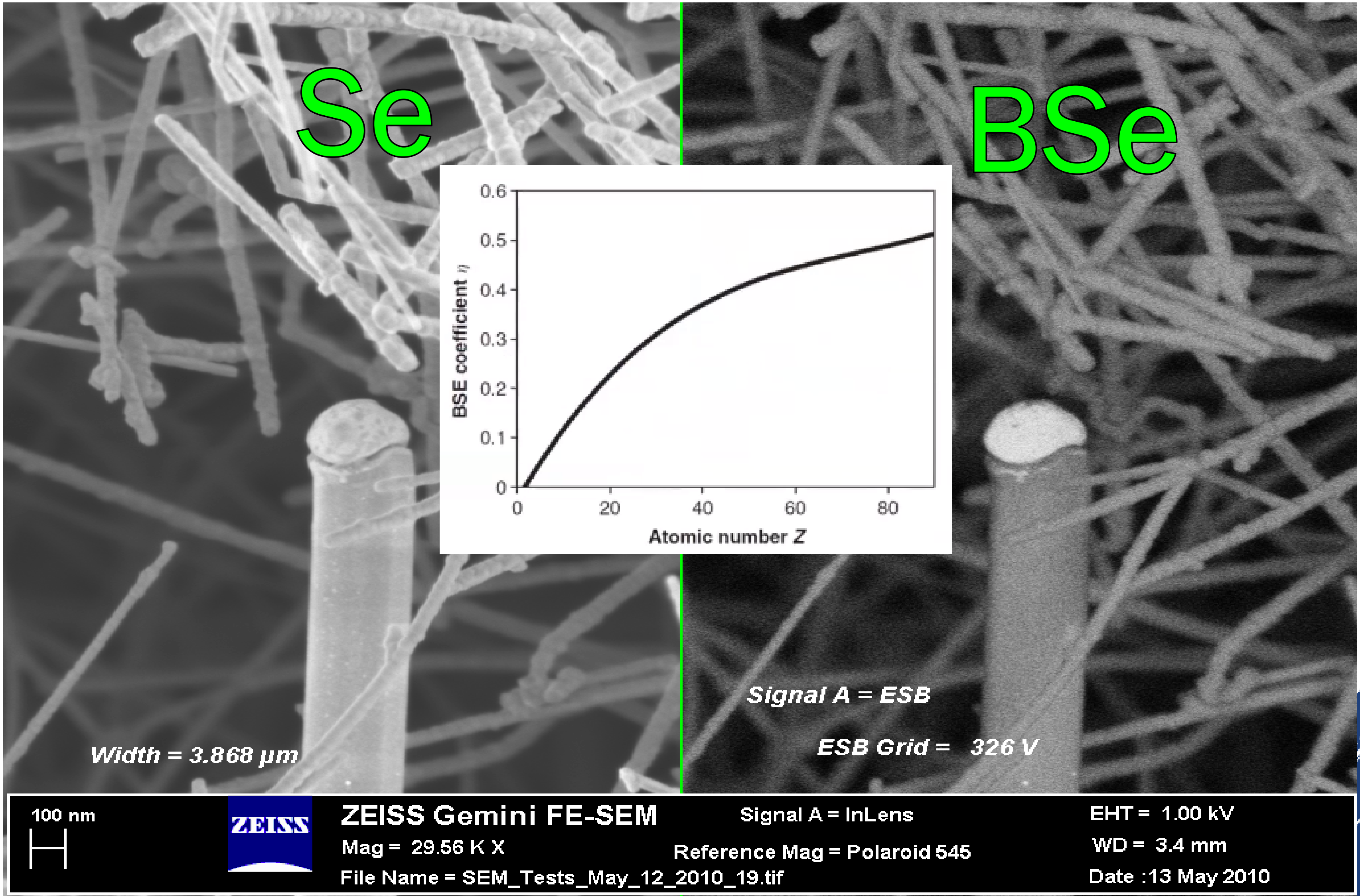


Backscattered Electrons

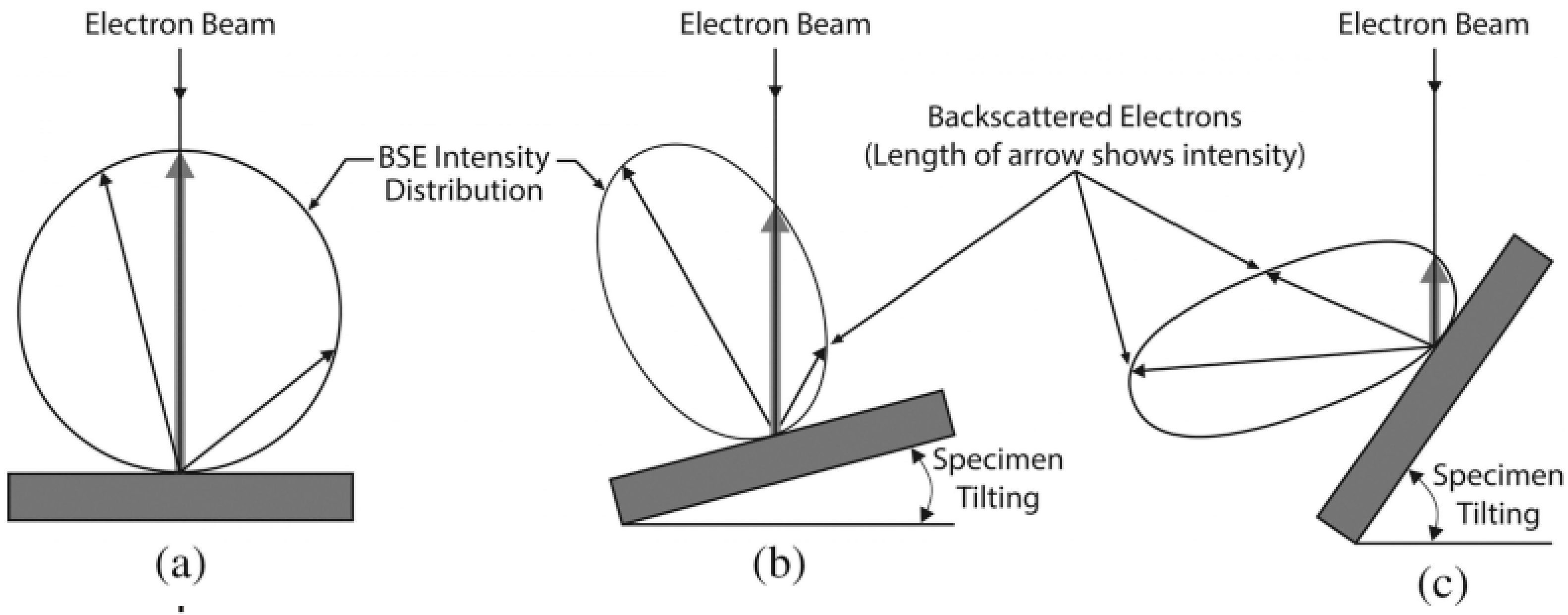
Backscattered Electrons

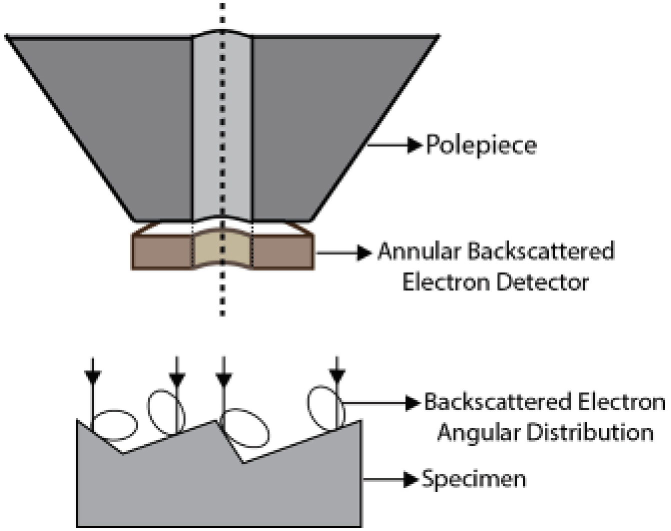
- Formed when an incident beam electron strikes the sample and is redirected back out of the sample
- BSE's retain much of the e-beam's original energy
  - 50 eV to >50% acceleration voltage
  - Large (10's nm) escape depth
- Elements with higher atomic numbers redirect more incident electrons allowing more to be backscattered
- This gives contrast to different materials in the sample
  - Higher Z or denser materials (more atoms) appear brighter on image

Beam Sample Interaction

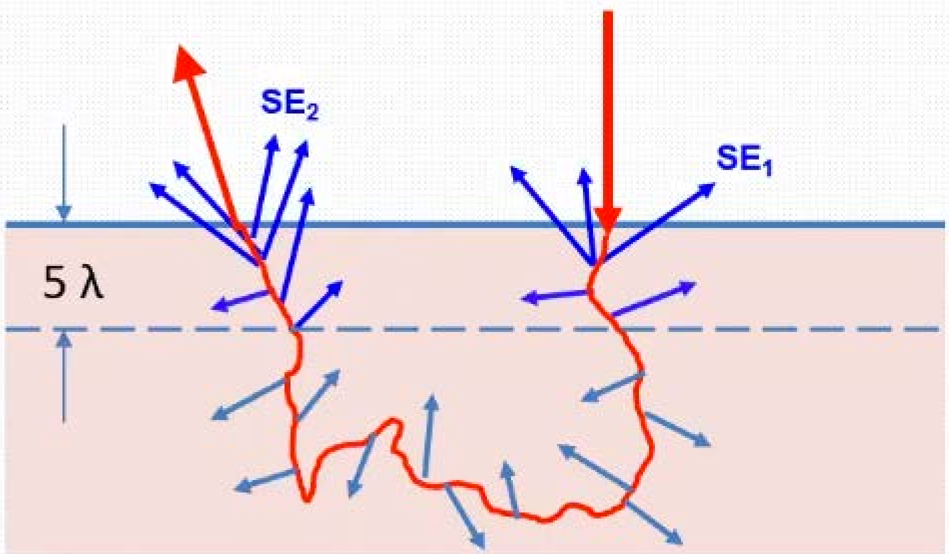


Beam Sample Interaction

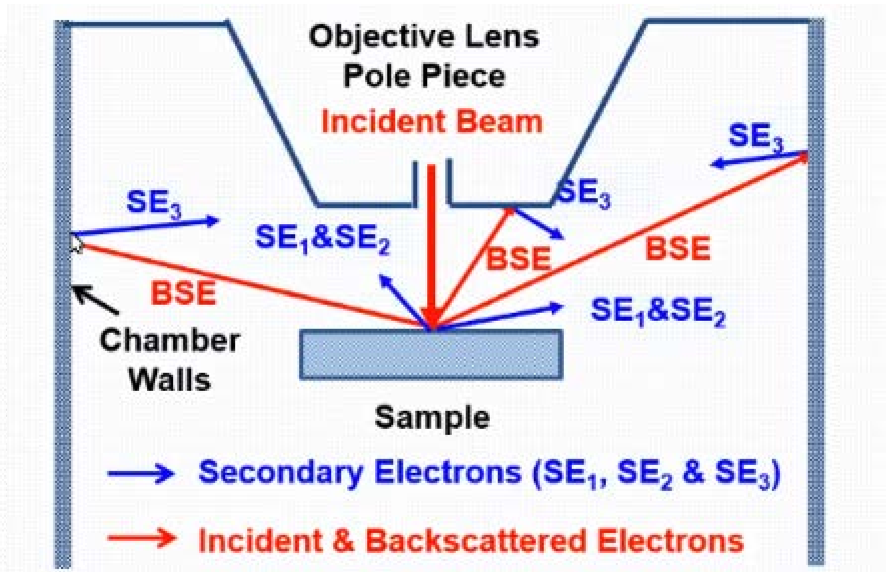


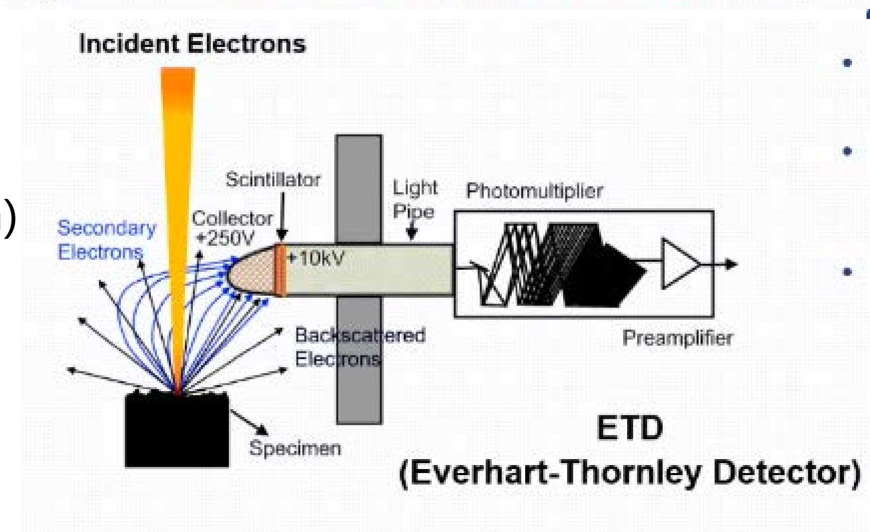


Beam Detection



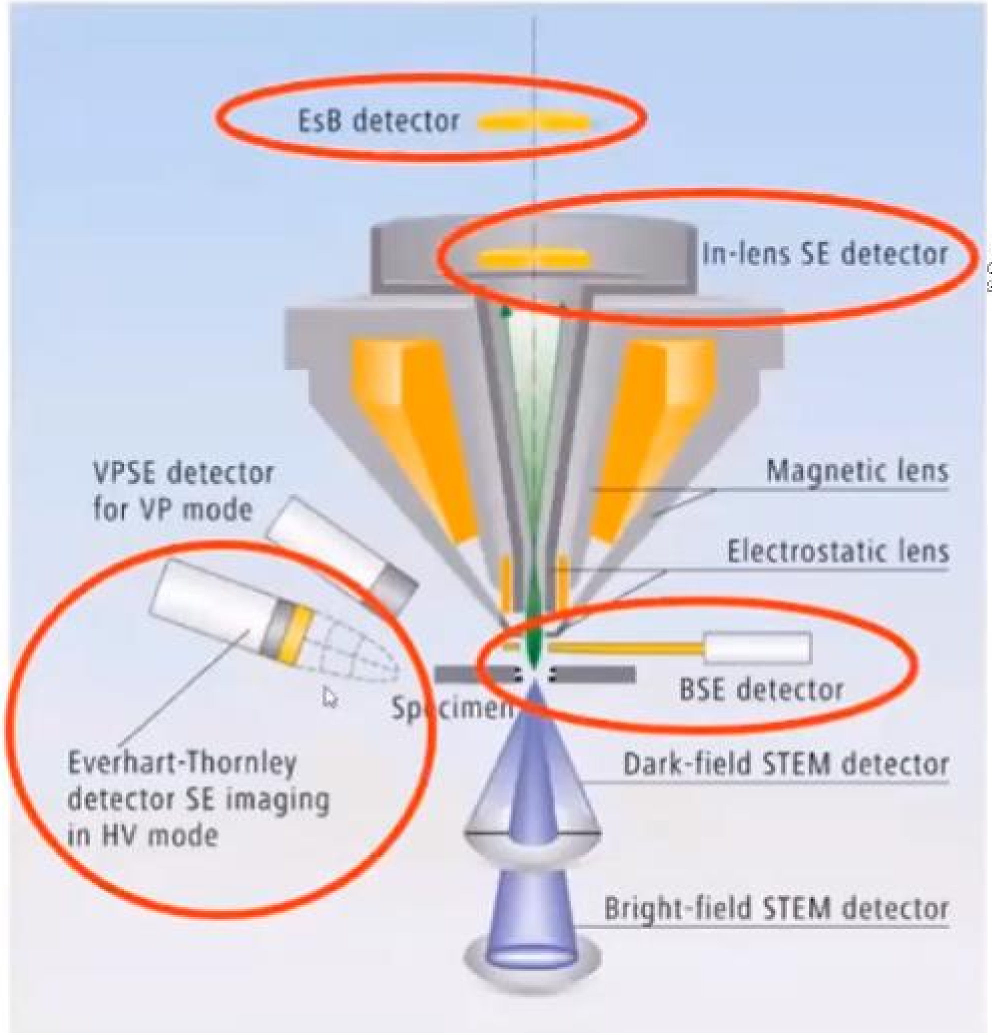
λ: Mean free path of 2nd electrons

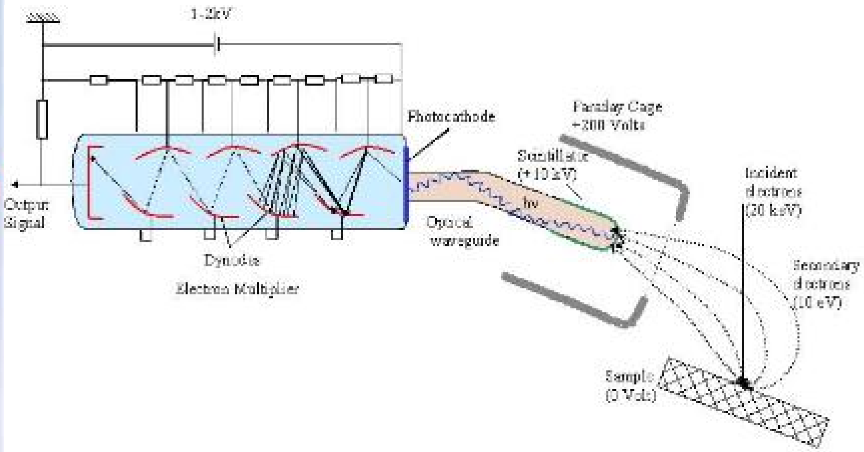




SE1: high resolution SE (generated with incident beam)
SE2: low resolution SE (generated due to BSEs)
SE3: indirect generation from the chamber

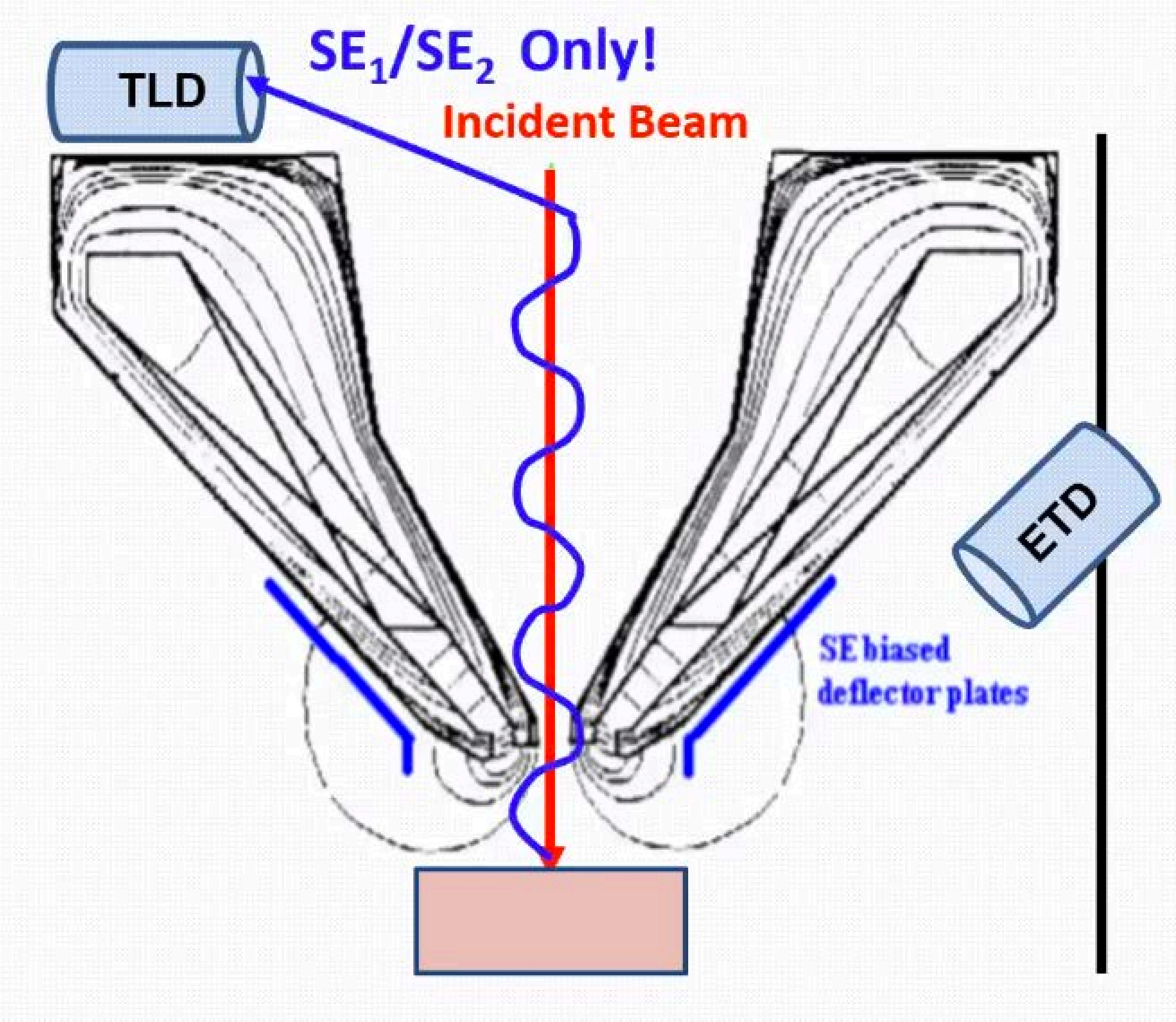
Beam Detection



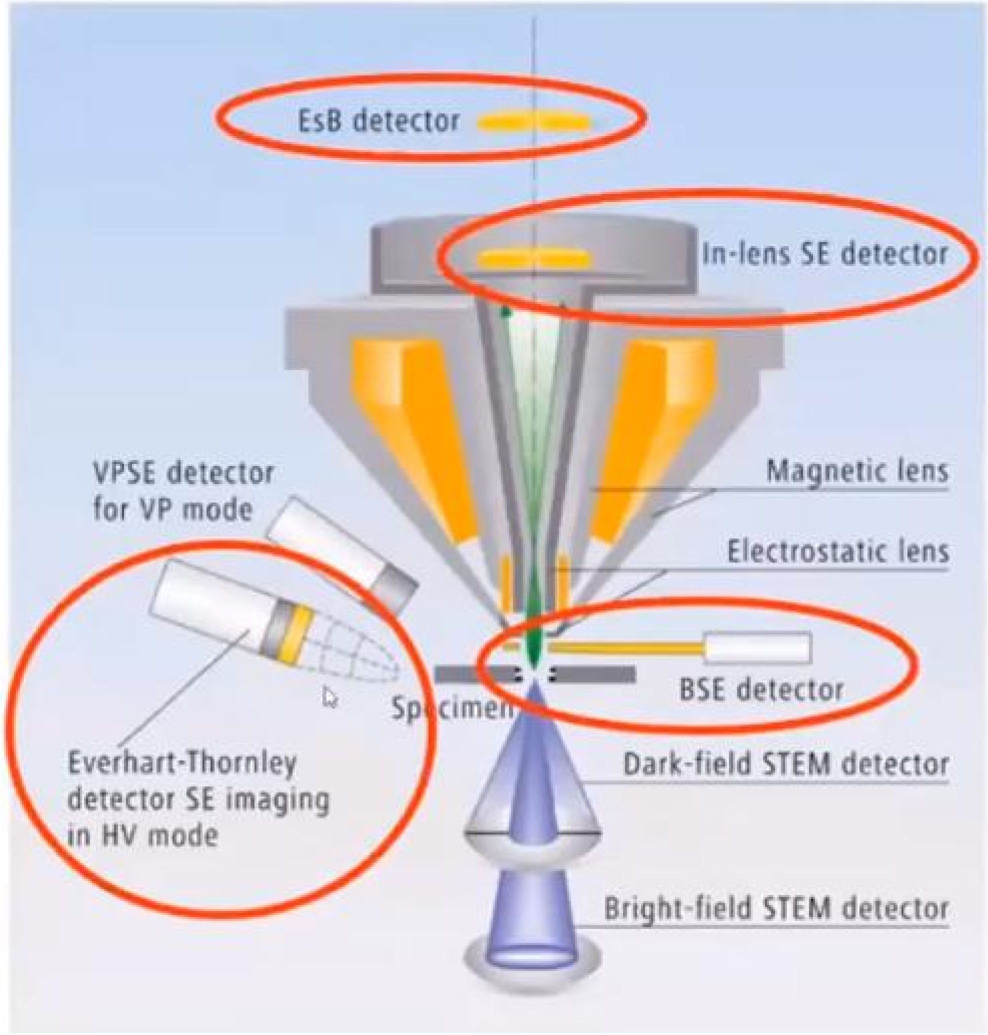


Requires High Vacuum

Beam Detection



Beam Detection



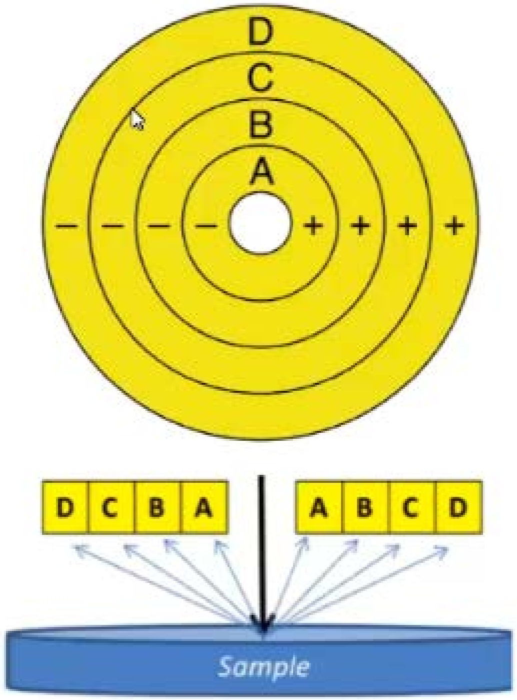
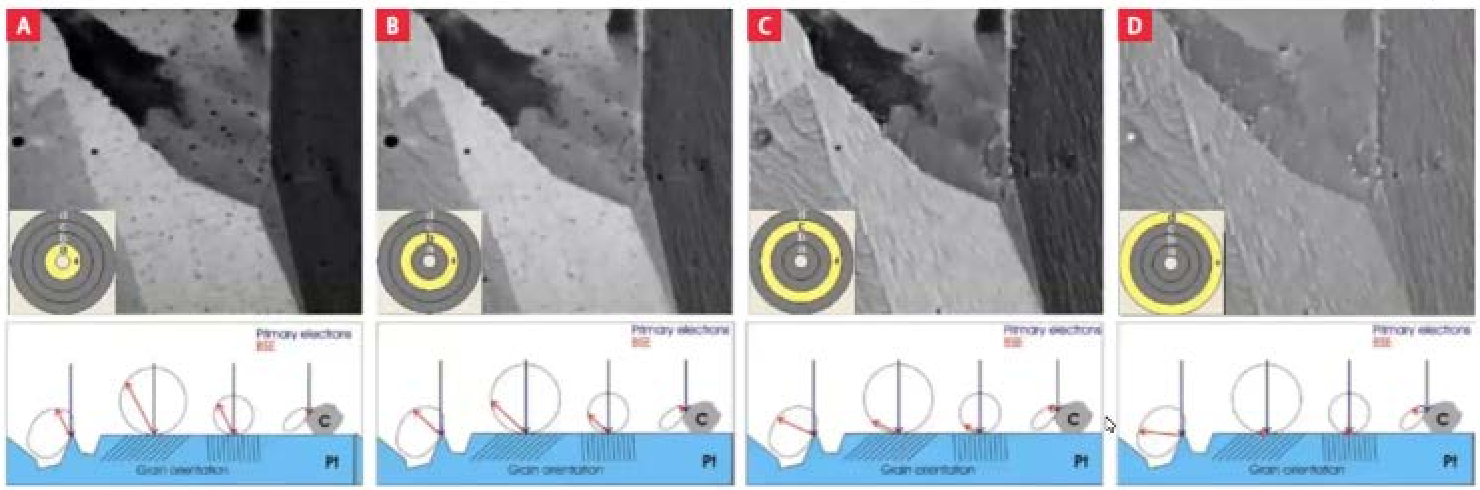
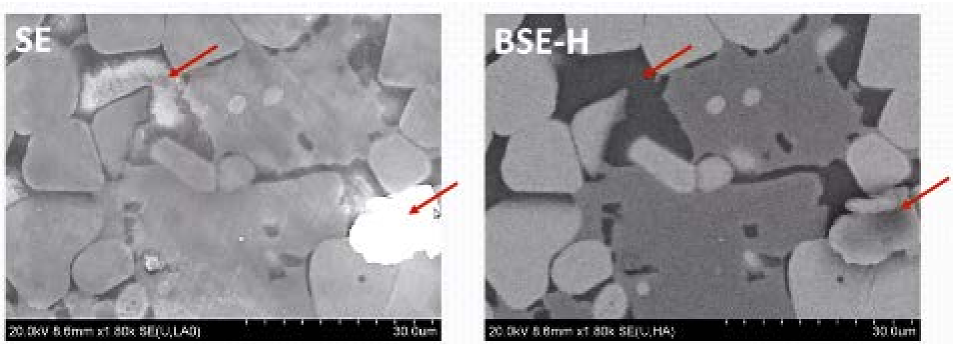


Figure 8: Schematic of angular separation of BSE detection and detector layout

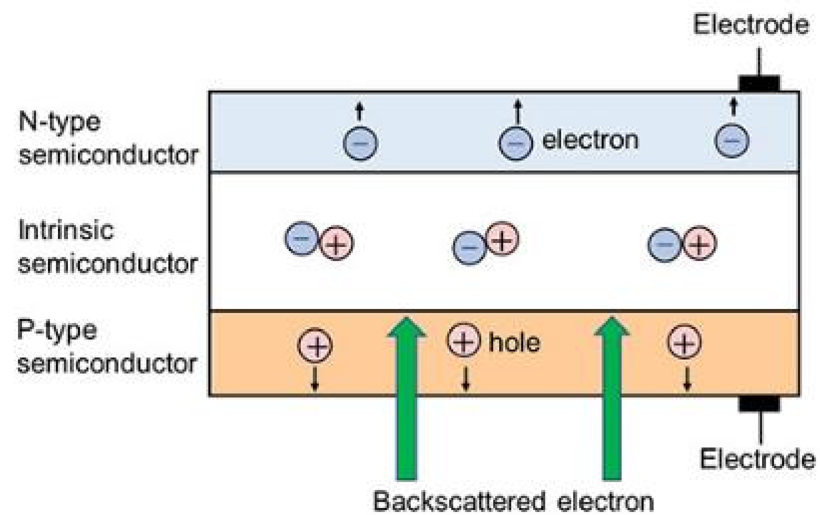
Beam Detection

Inner Rings: High Angle BSE, mostly atomic contrast information
Outer Rings: Low Angle BSE, topographical contrast

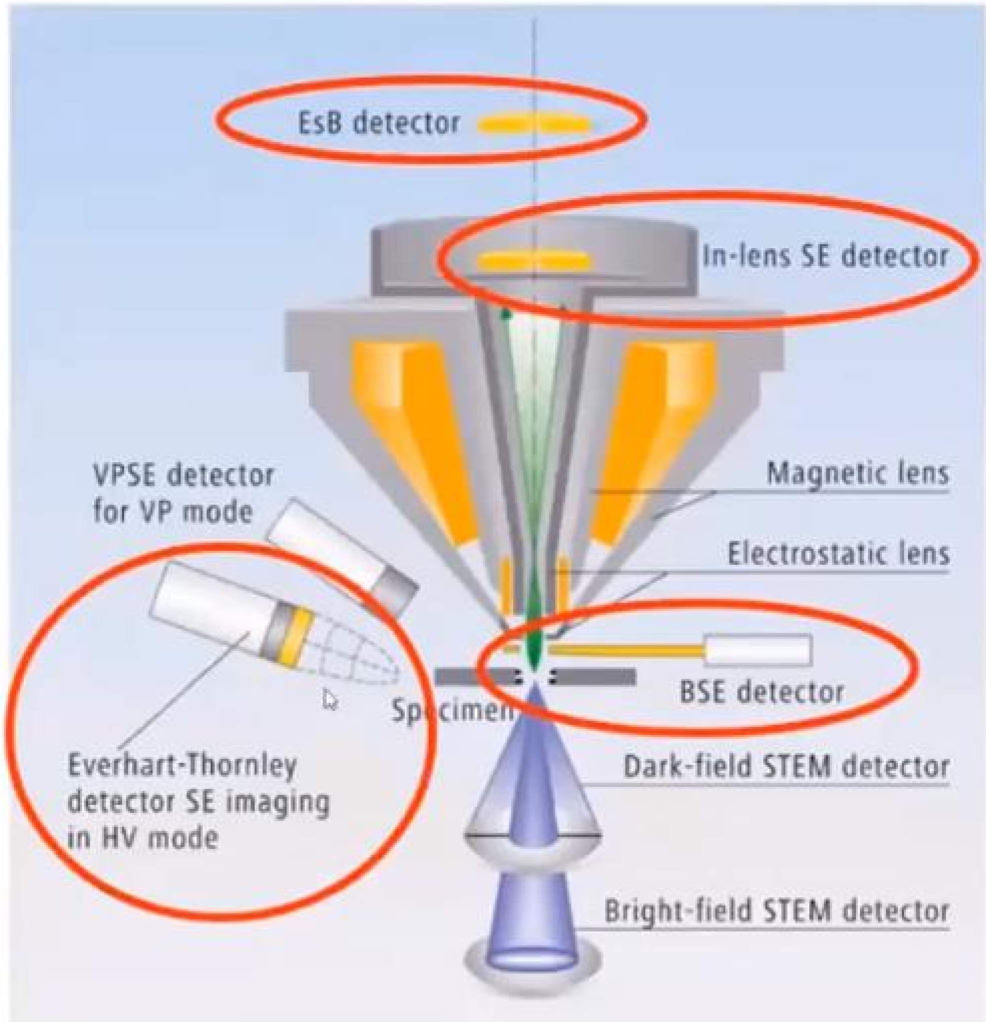


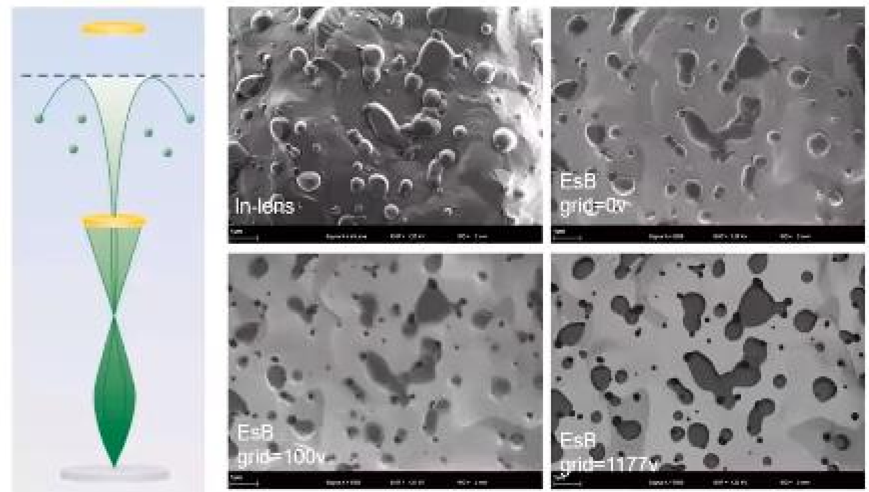


BSEs are less affected by Charging Effect
→ Charge artefact free image



Beam Detection





Energy Selective BS

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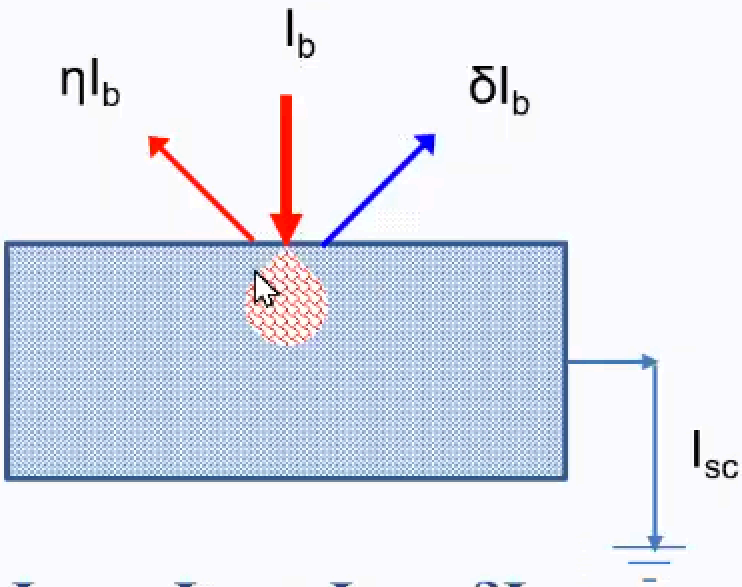
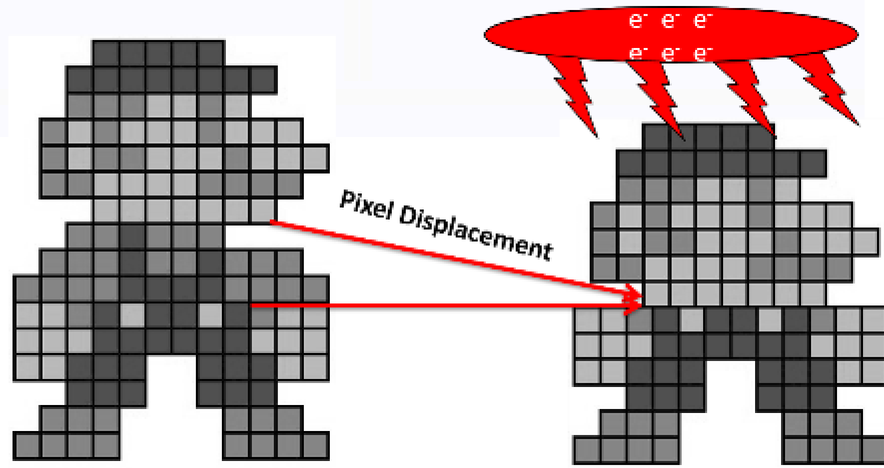
Charging



$$I_{sc} = I_b - \eta I_b - \delta I_b$$

I_b – incident beam electrons
δI_b – secondary electrons
ηI_b – backscattered electrons
I_{sc} – specimen current

- No charging
- Charging



Charging

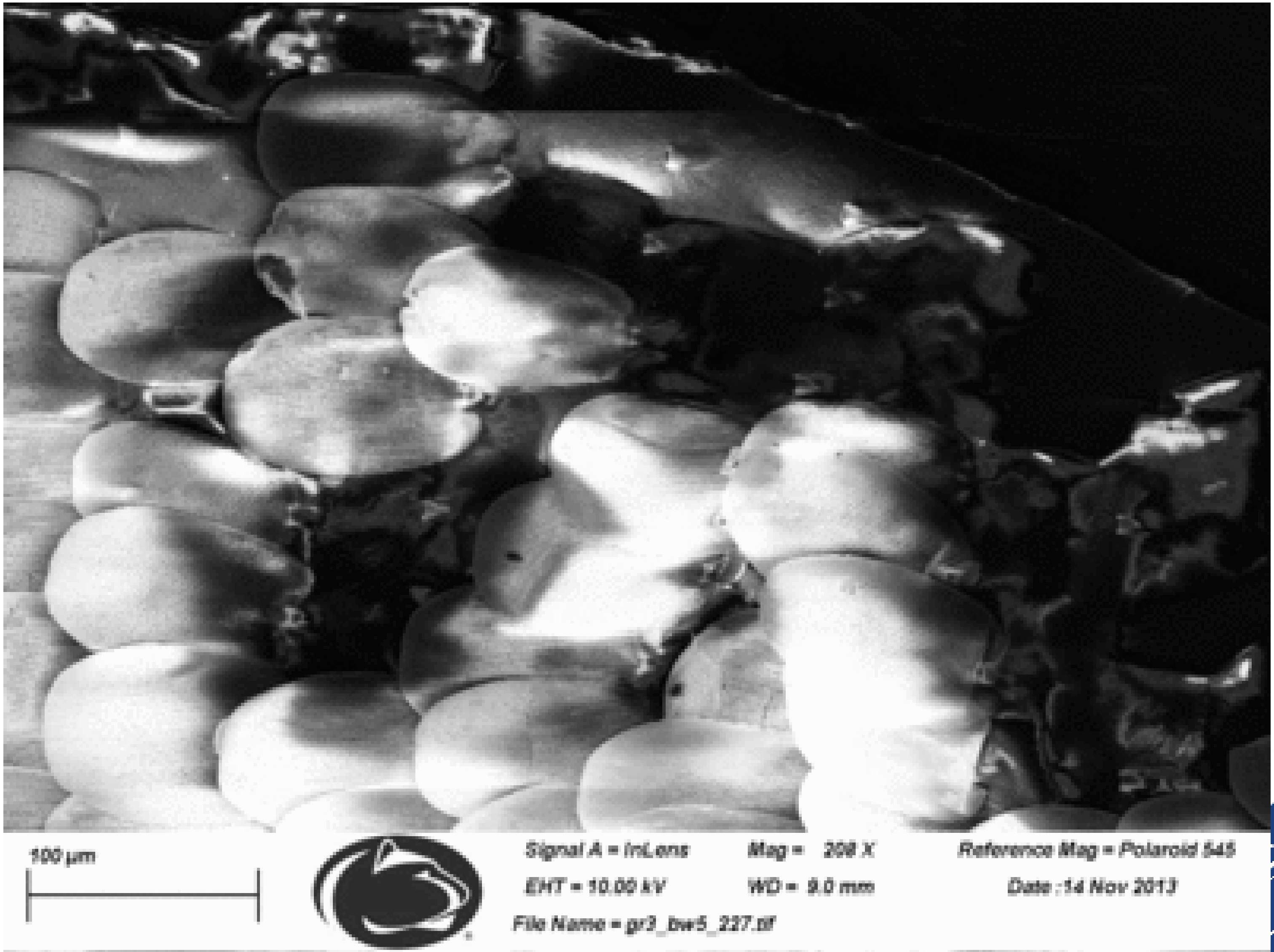
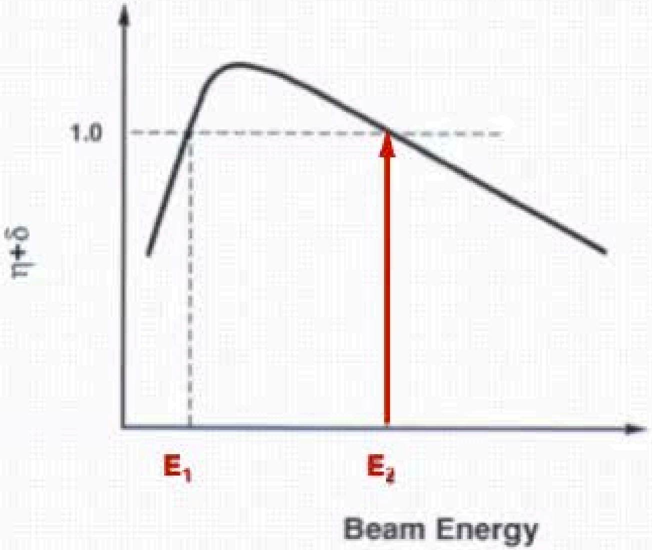

100 µm
Signal A = InLens
Mag = 208 X
Reference Mag = Polaroid 545
EHT = 10.00 kV
WD = 9.0 mm
Date :14 Nov 2013
File Name = gr3_bw5_227.tif

Image Enhancement

Operate where emitted electron current balances beam current

$$I_{sc} = I_b - \eta I_b - \delta I_b = I_b\,(1-(\eta+\delta)) = 0$$



Total emitted electron coefficient η+δ as a function of beam energy
When η+δ =1, charge reaches balance

Upper cross-over energy, E_2, for several materials

Material	E_2(keV)
Kapton	0.4
Electron resist	0.55–0.70
Nylon	1.18
5% PB7/nylon	1.40
Acetal	1.65
Polyvinyl chloride	1.65
Teflon	1.82
Glass passivation	2.0
GaAs	2.6
Quartz	3.0
Alumina	4.2

From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press

PennState

Image Enhancement

Carl Zeiss

- Enables imaging at near atmospheric conditions (5 to 10 Torr)
  - Water behaves like water ("wet limit" 0 ˚C at ~4.5 Torr)
  - Biological, etc., samples are studied in virtually natural environment
- Gun aperture (valve) maintains high/ultra high vacuum at tip
- Variable Pressure (Gaseous) SE Detector
  - MFP too low in chamber for typical imaging
  - Input gas depends on material typically water vapor and nitrogen mixture
  - Gas molecules undergo cathodoluminescence when hit by SE emitted from the sample
  - Detector registers photon intensity from cascading gas molecule ionization

Image Enhancement

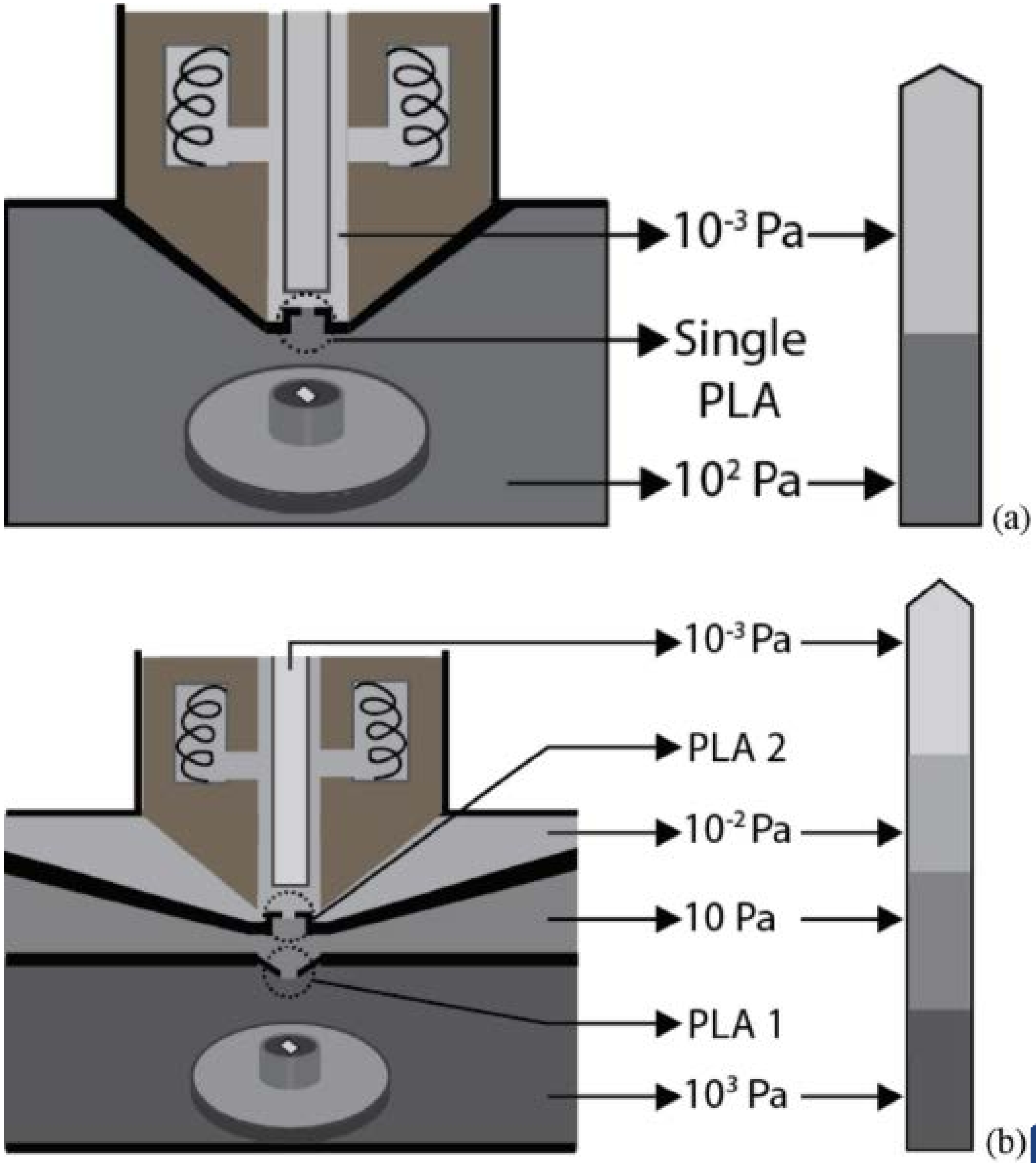
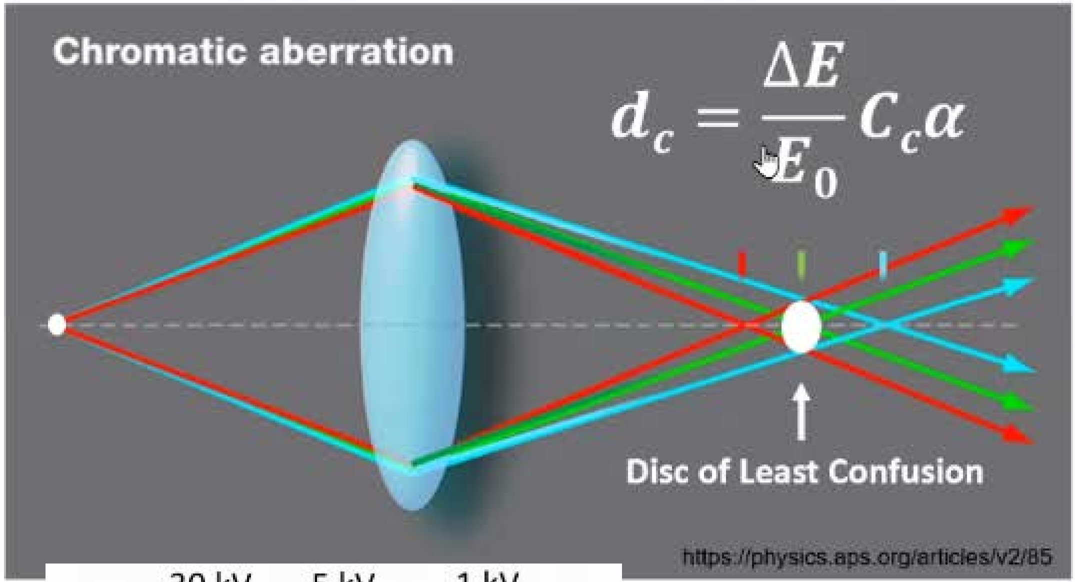
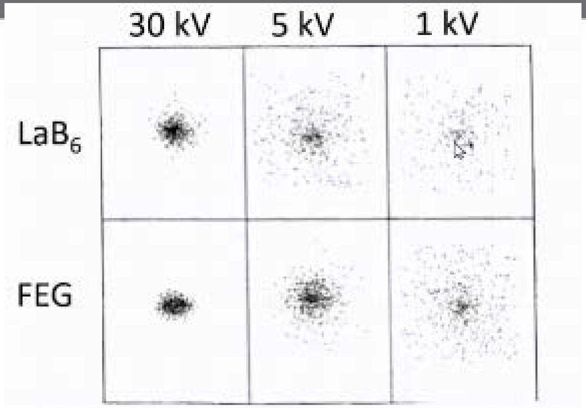


Image Enhancement



- d_c increases with source energy spread ΔE
  – electron source
- d_c increases when electron energy E_0 drops
  – accelerating voltage



Simulation of the first 250 electrons in the probe at the plane of the disc of least confusion

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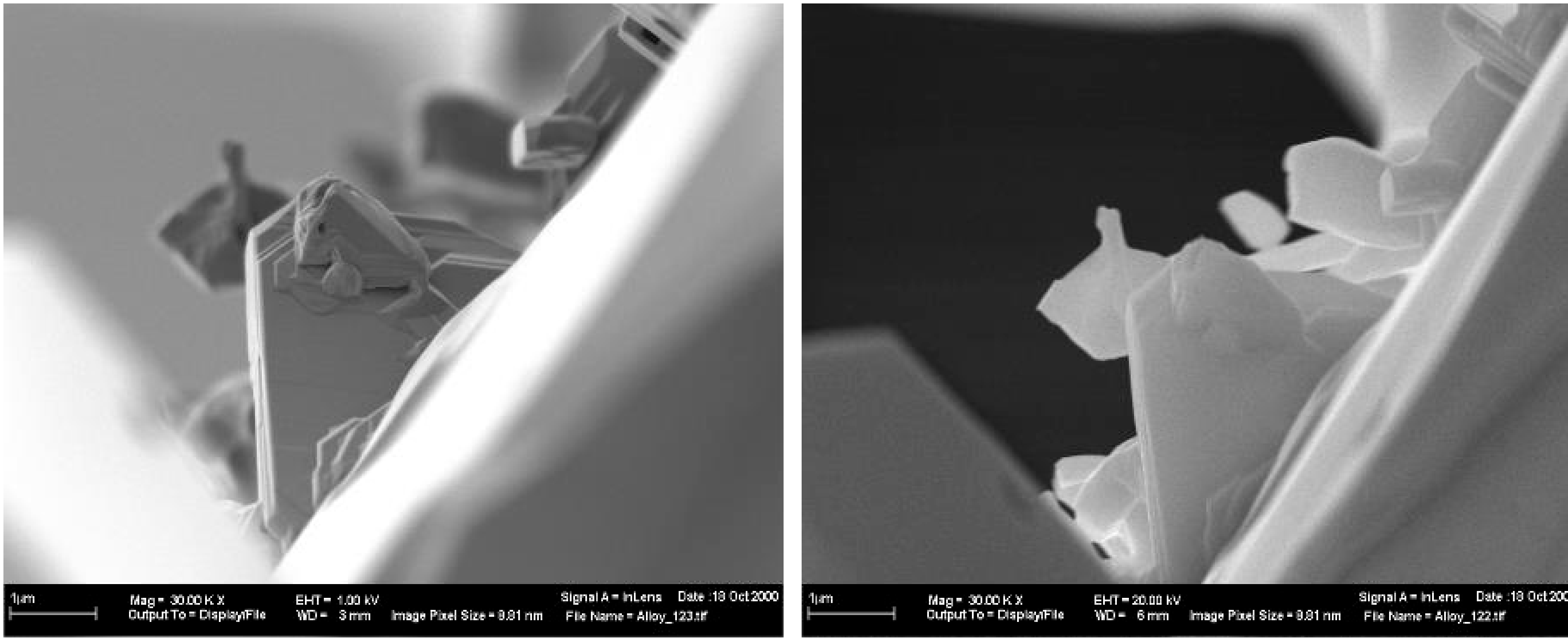
Image Enhancement



Platinum Rhodium Alloy Crystals at 1kV (left) and at 20kV (right)

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Image Enhancement

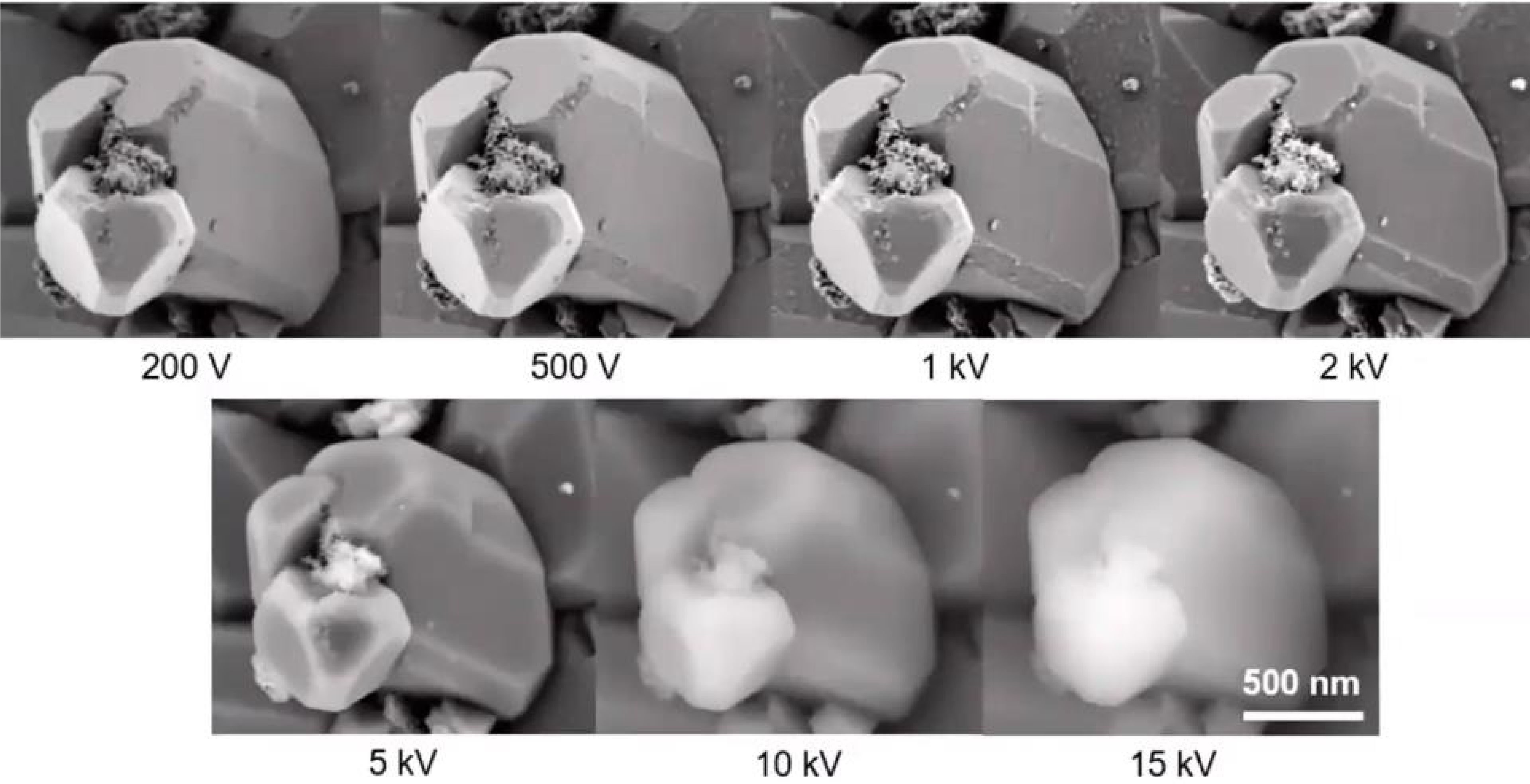
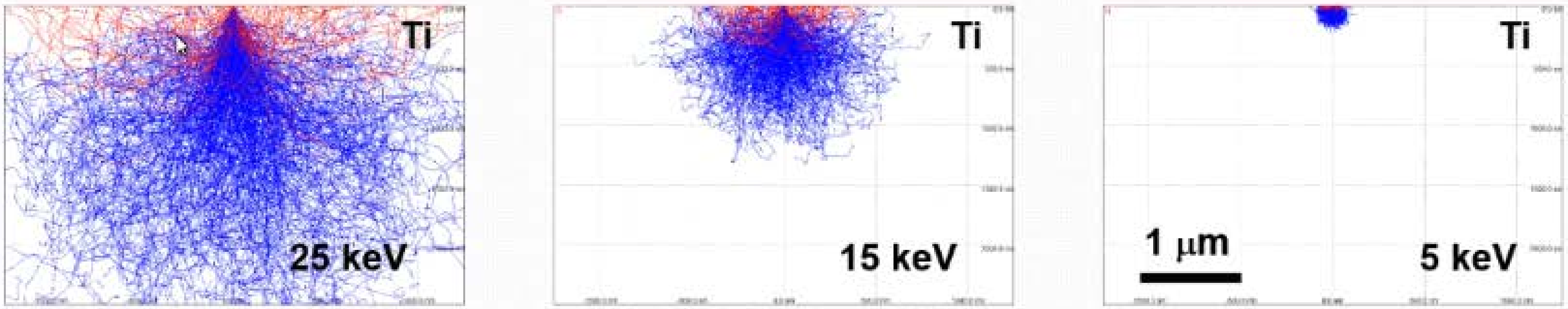


Image Enhancement



Monte Carlo Calculations, CASINO

Beam Energy Decreases

Higher Accelerating Voltage

- Higher beam energy
  → Larger interaction volume
- **SE_2 & BSE** exit from a much larger region
  → Surface spatial resolution is reduced

Lower Accelerating Voltage

- Lower beam energy
  → Smaller interaction volume
- **SE_2 & BSE** exit from a much smaller region
  → Surface spatial resolution is enhanced

Image Enhancement

How would a low keV come handy?

- **Increase spatial resolution for both SE and BSE**
  - but only if chromatic aberration effect is not an issue
- **Minimize charging effect**

- **Chromatic aberration**
- **Low BSE yield**
  - Yield coefficient of heavy elements drops when the beam energy falls below a few keV
- **Detector detection sensitivity**
  - Solid state detectors lose sensitivity as the incoming electron energy falls below a few keV
- **Detector detection efficiency**
  - Low angle BSEs tend to go uncollected

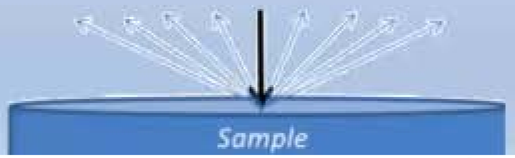
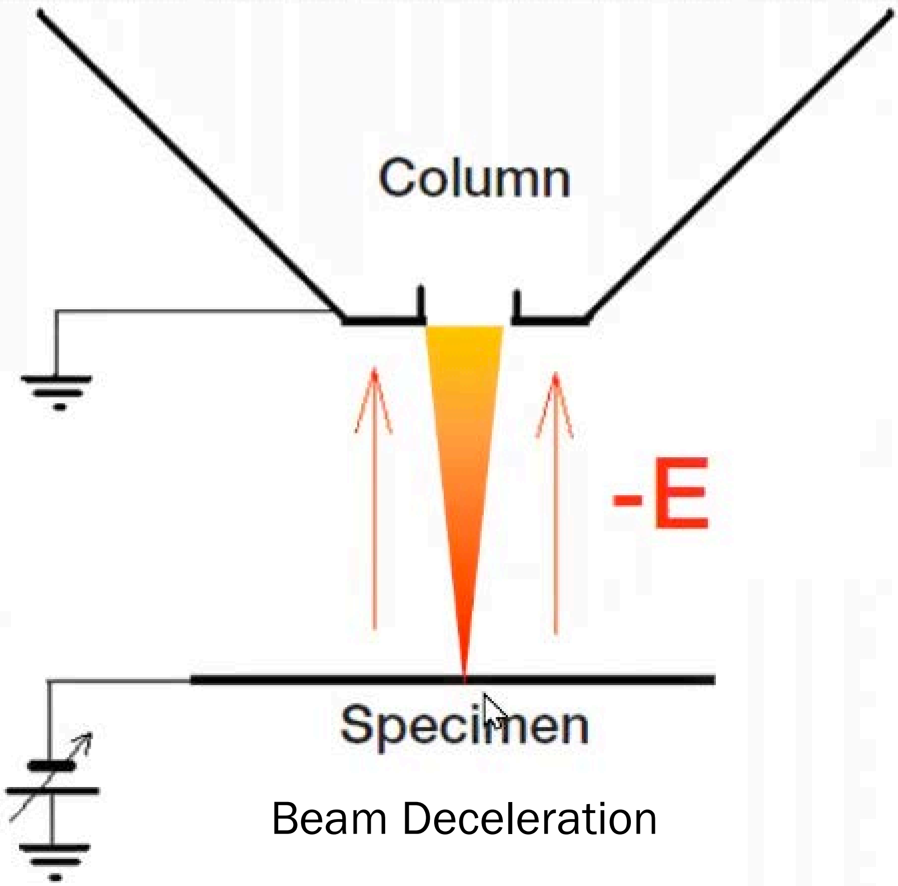
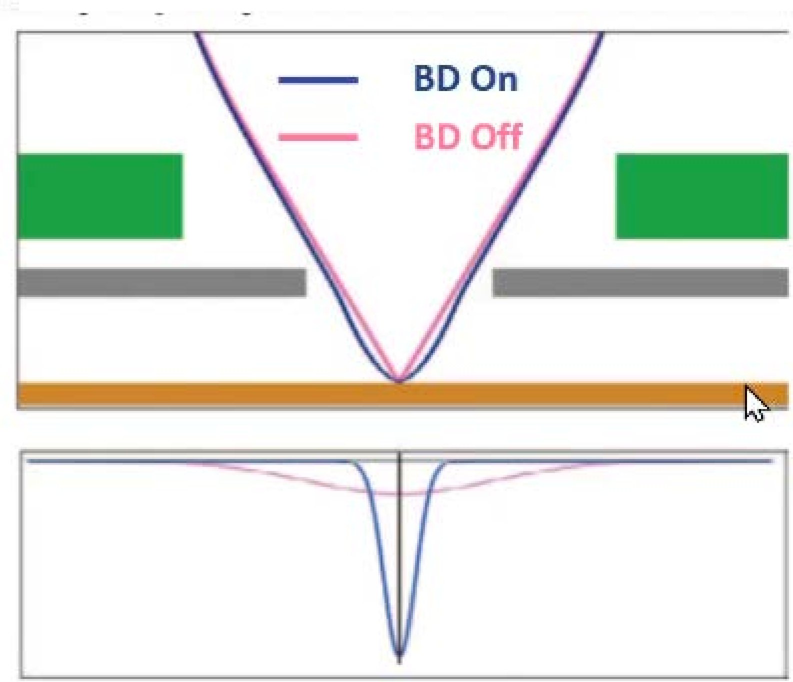


Image Enhancement





D. Phifer*, L. Tuma, T. Vystavel, P. Wandrol, and R.J. Youn
Microscopy Today, 40, 2009

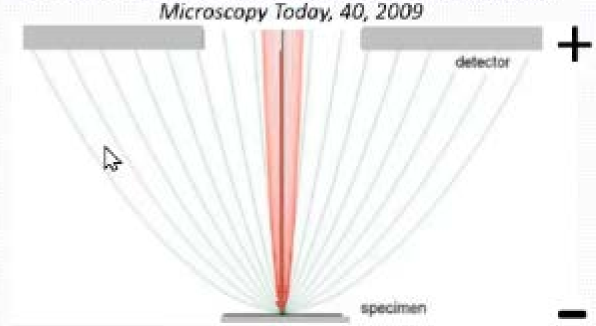
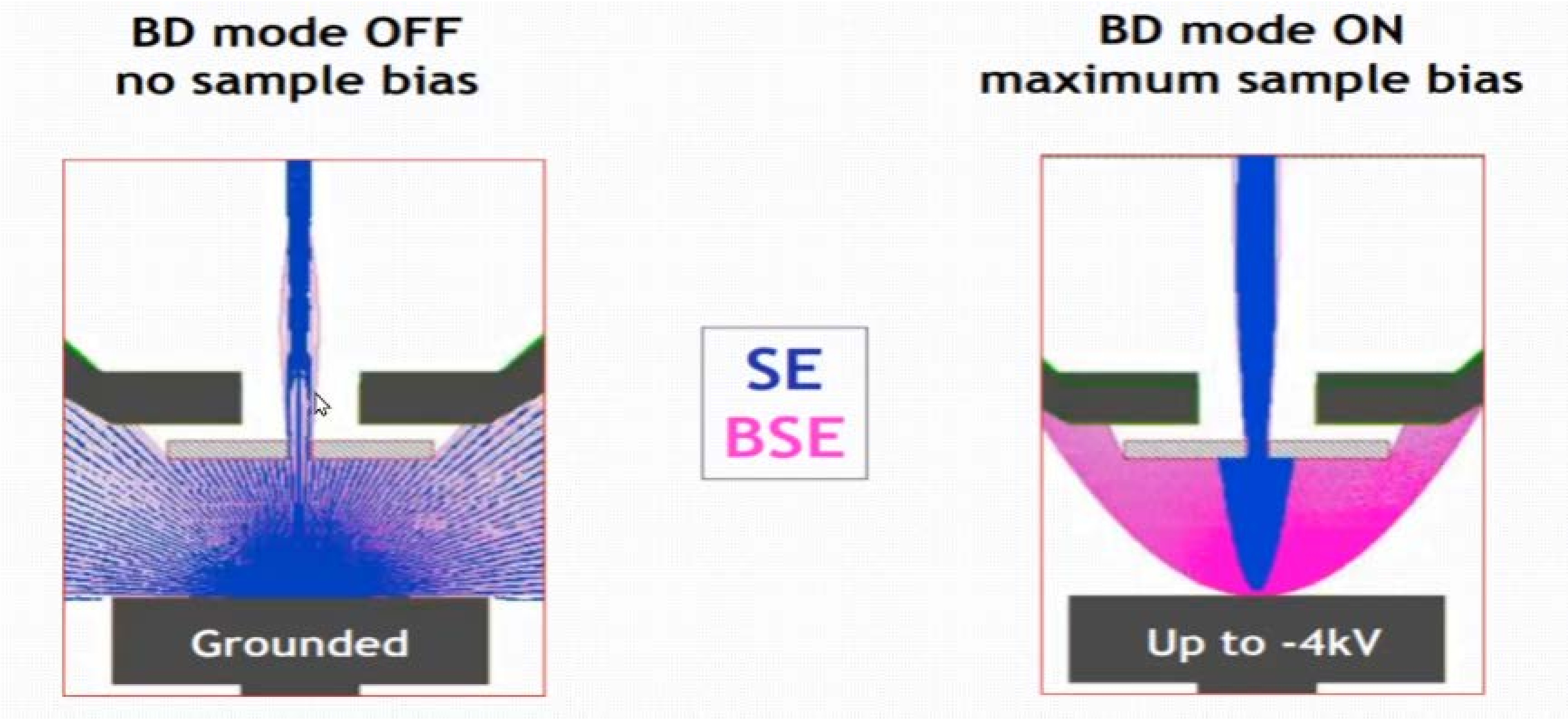
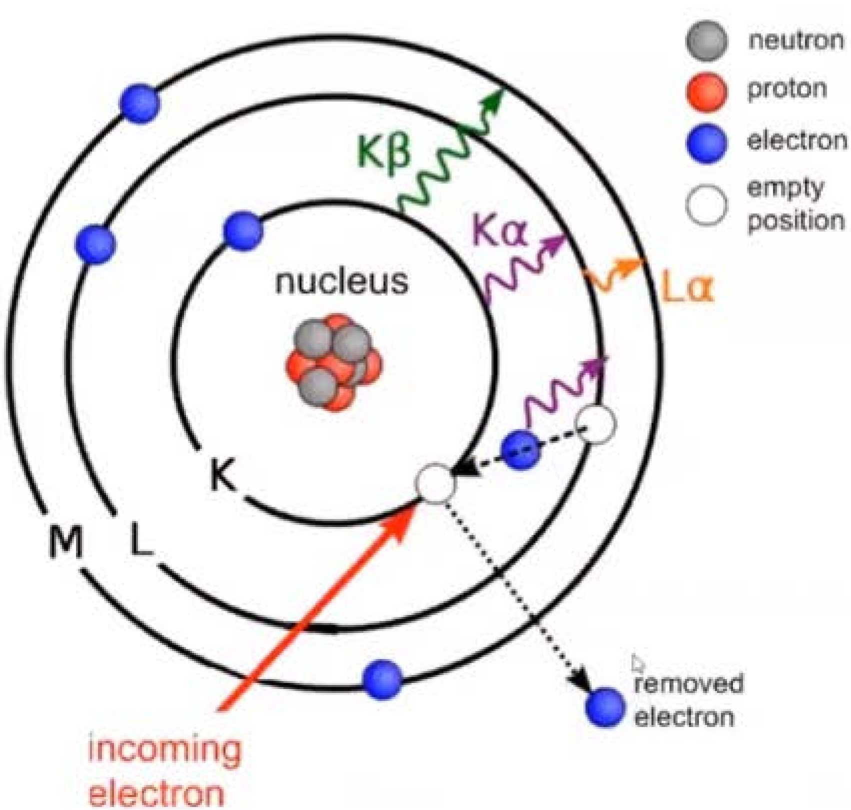


Image Enhancement

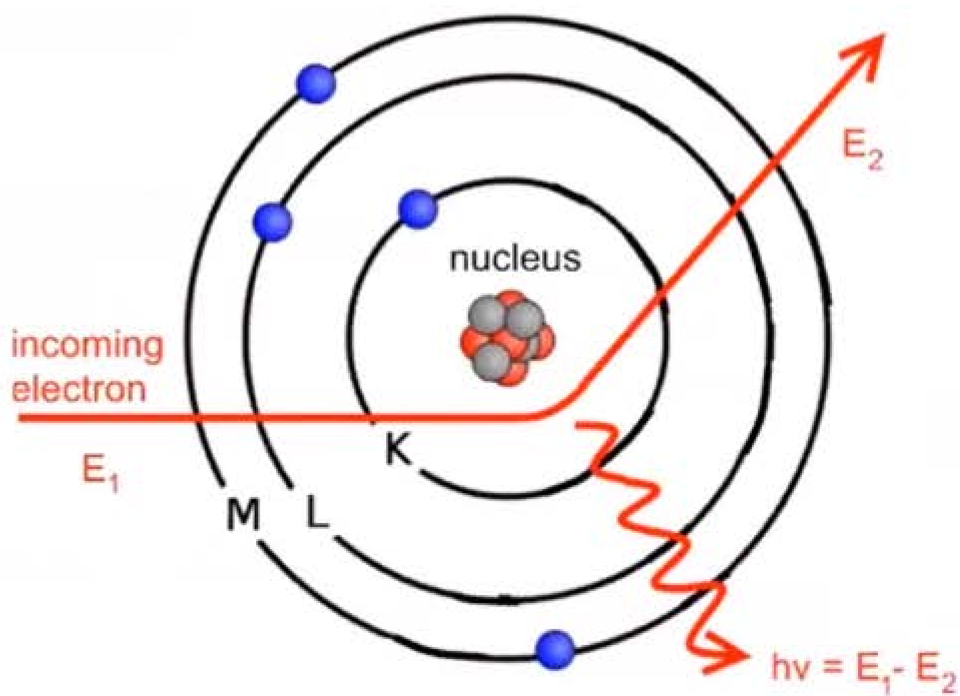


Energy Dispersive X-Ray Spectroscopy (EDS/EDX)

Characteristic X-Ray Emission Lines



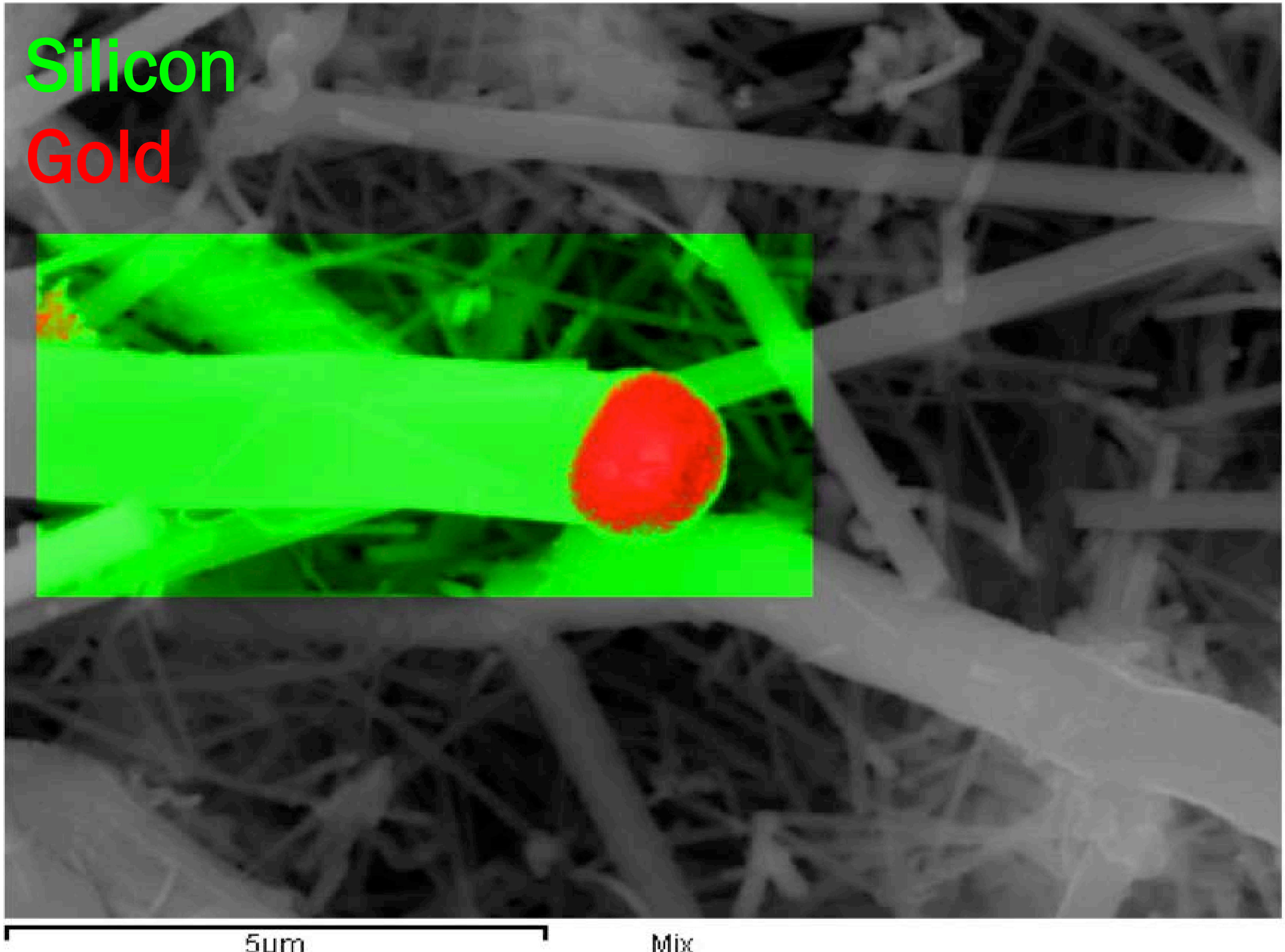
Bremsstrahlung



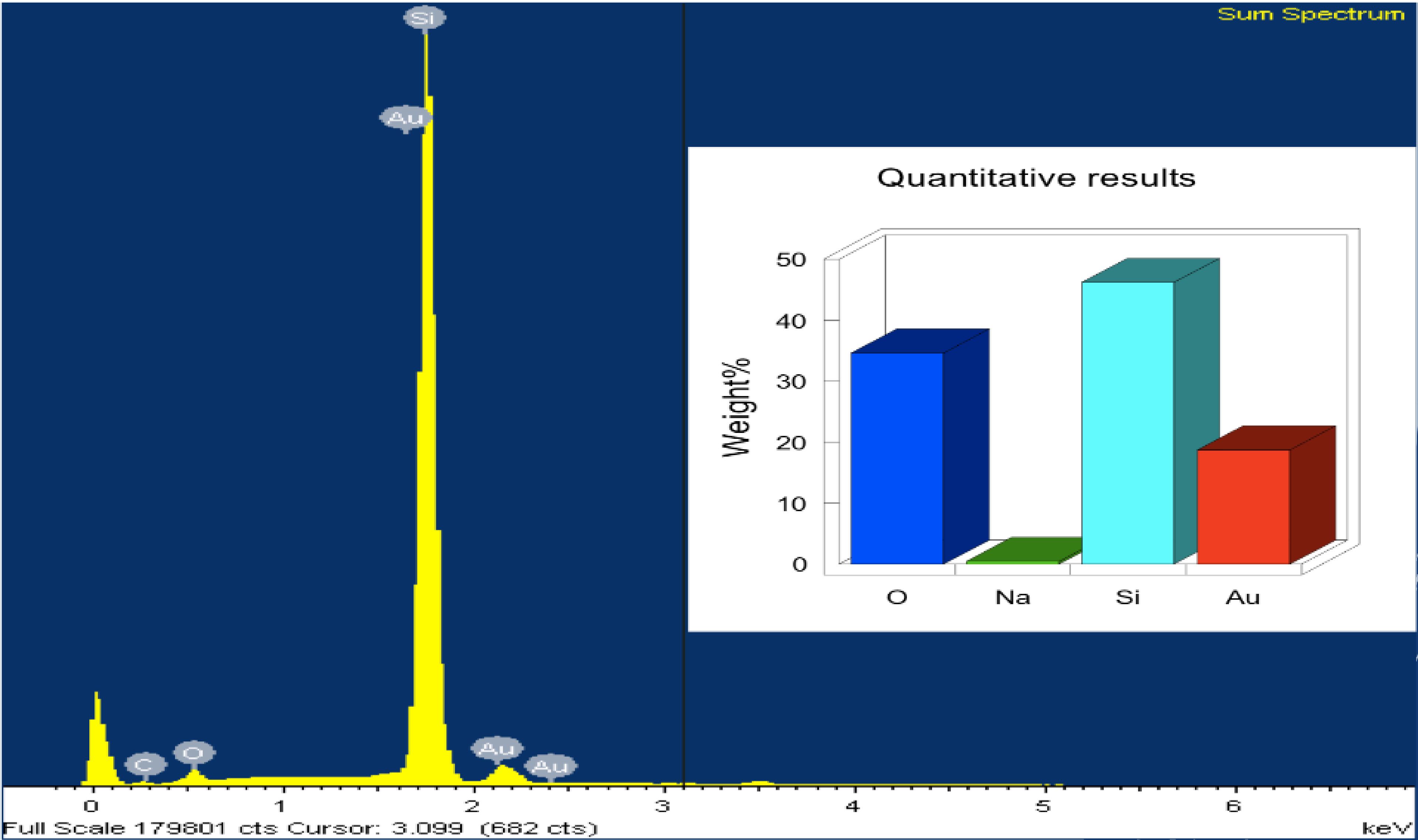
Energy Dispersive X-Ray Spectroscopy (EDS/EDX)

- Energy Dispersive X-ray Spectroscopy (EDS)
  - Chemical (elemental) analysis technique compatible with SEM/TEM tools
- Sometimes referred to as EDXA (energy dispersive x-ray analysis)
- SEM gun electrons collide with the electrons within the sample, causing some of them to be knocked out of their orbits. These electron shell positions are filled by higher energy electrons which emit x-rays in the process.

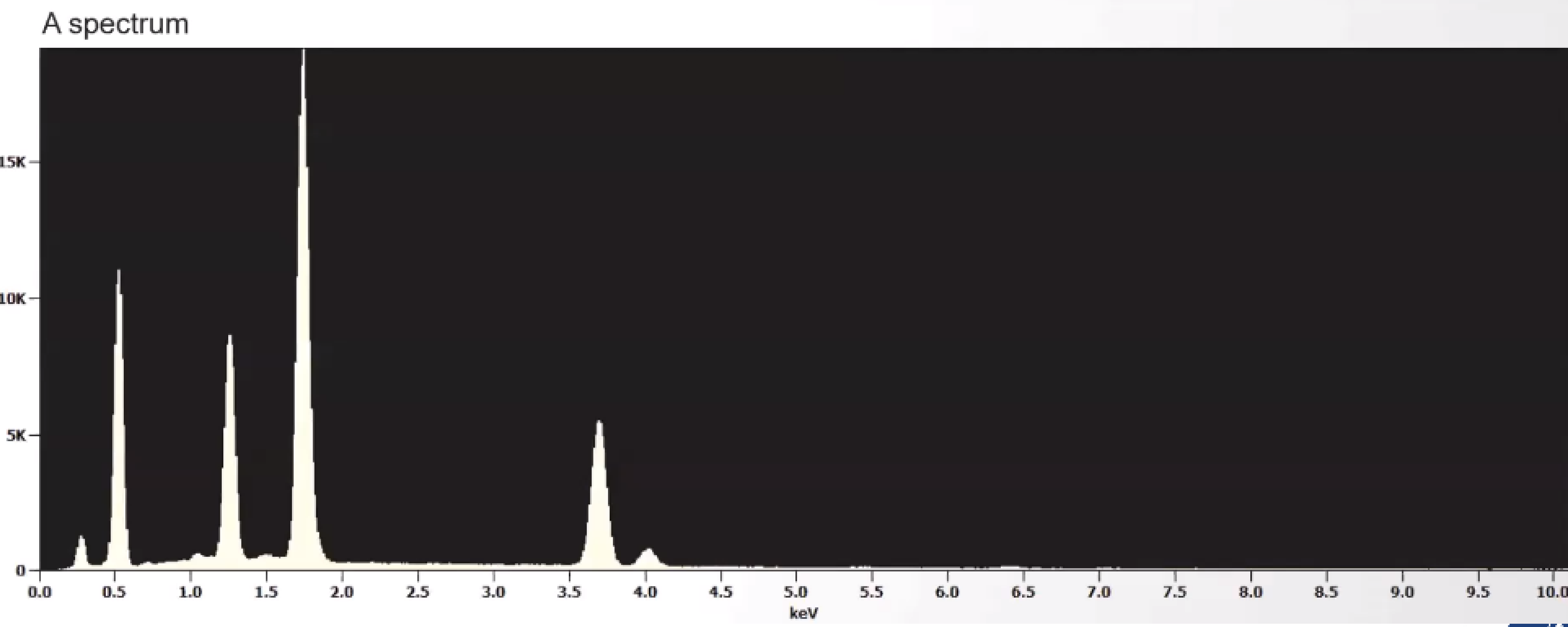
Energy Dispersive X-Ray Spectroscopy (EDS/EDX)



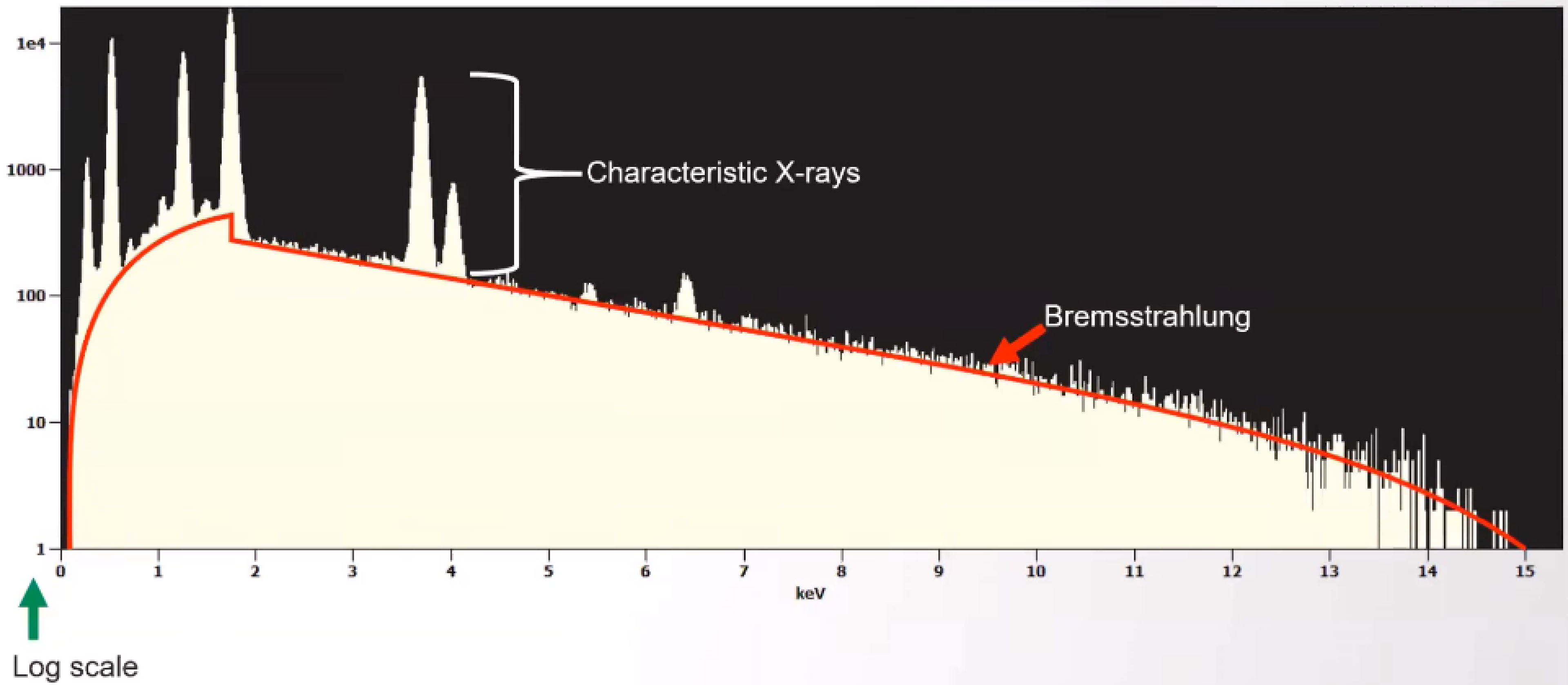
Energy Dispersive X-Ray Spectroscopy (EDS/EDX)



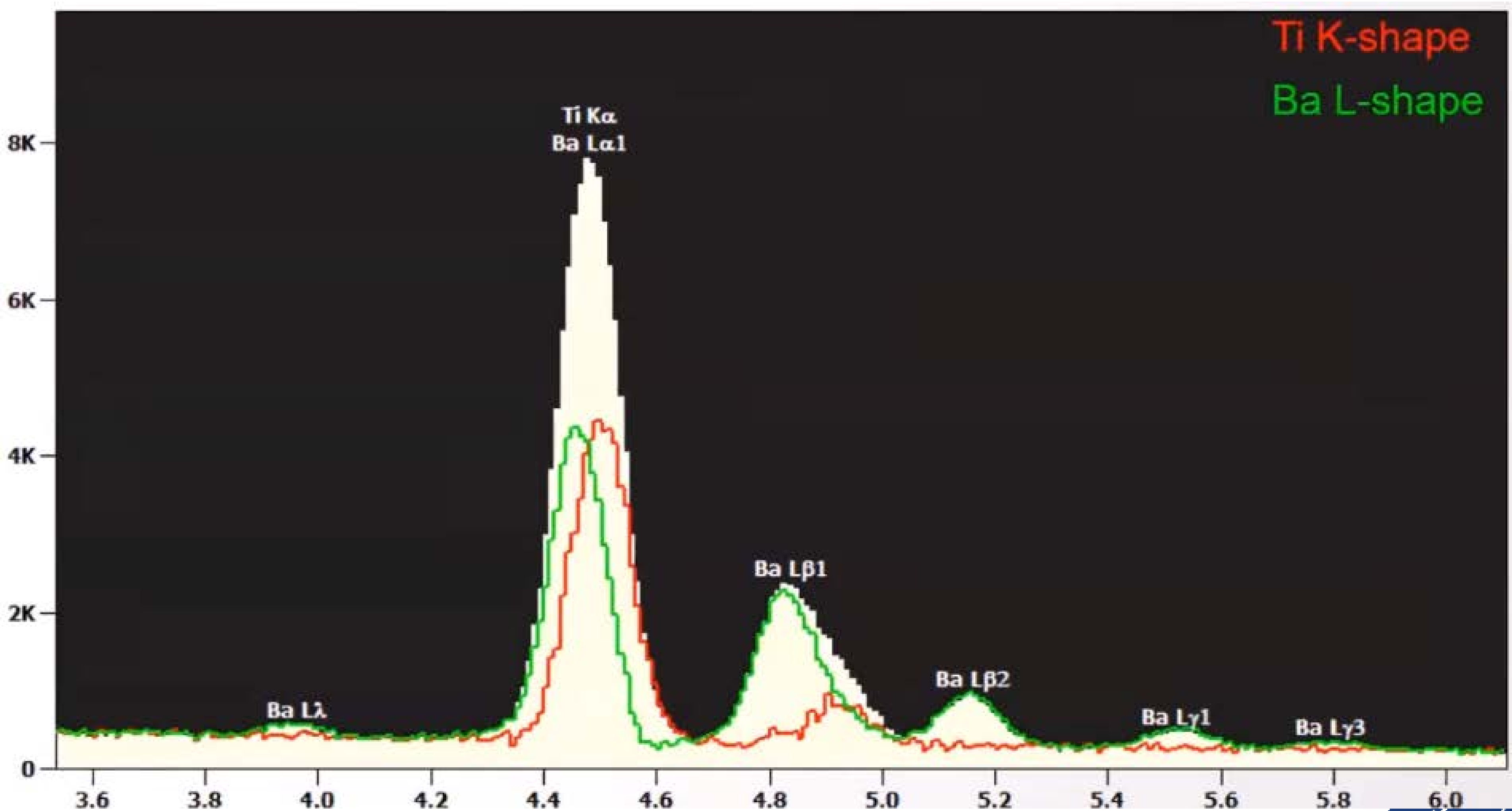
Energy Dispersive X-Ray Spectroscopy (EDS/EDX)



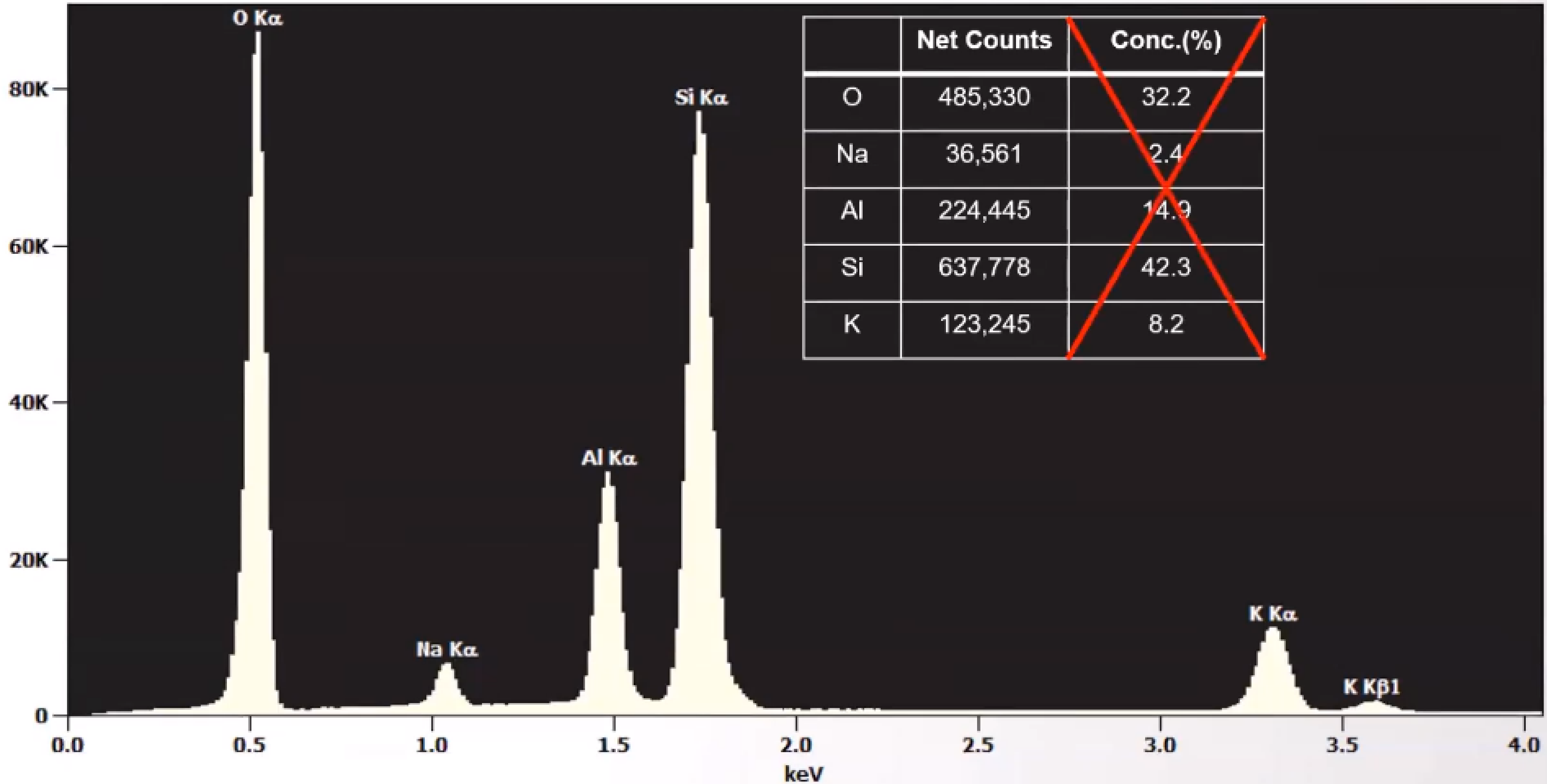
Energy Dispersive X-Ray Spectroscopy (EDS/EDX)



Energy Dispersive X-Ray Spectroscopy (EDS/EDX)

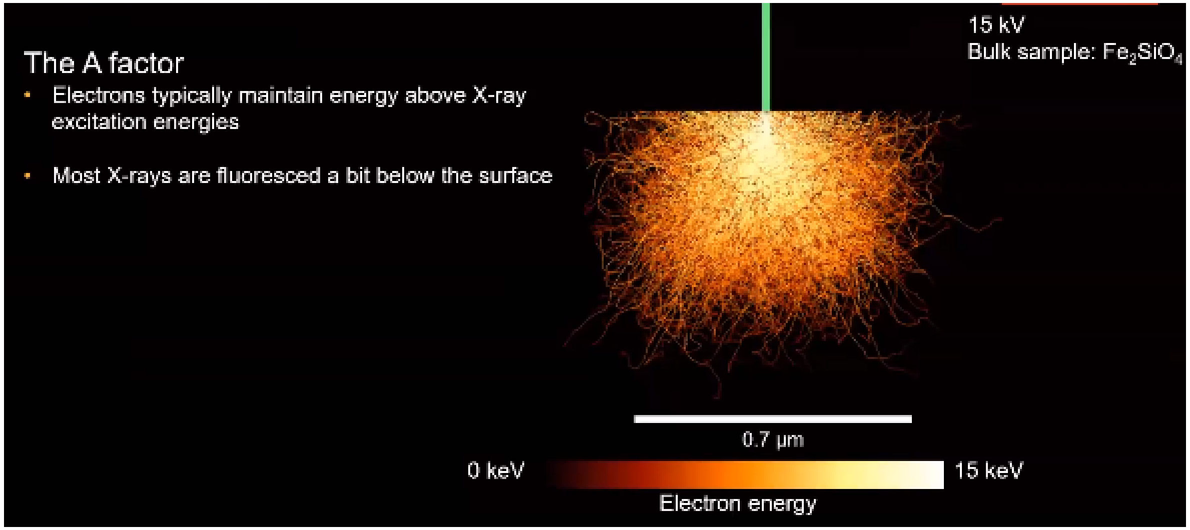


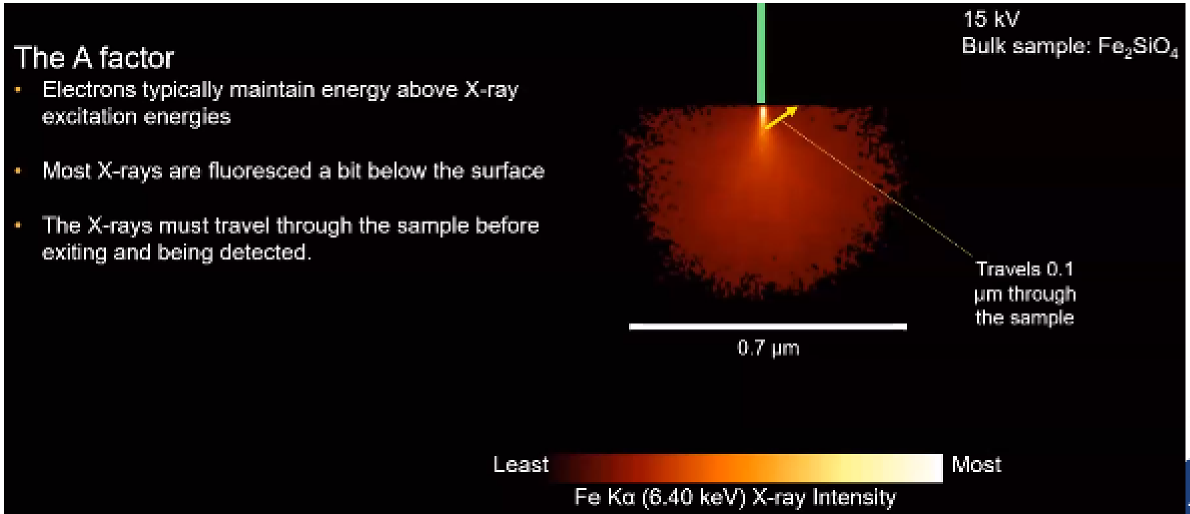
Energy Dispersive X-Ray Spectroscopy (EDS/EDX)



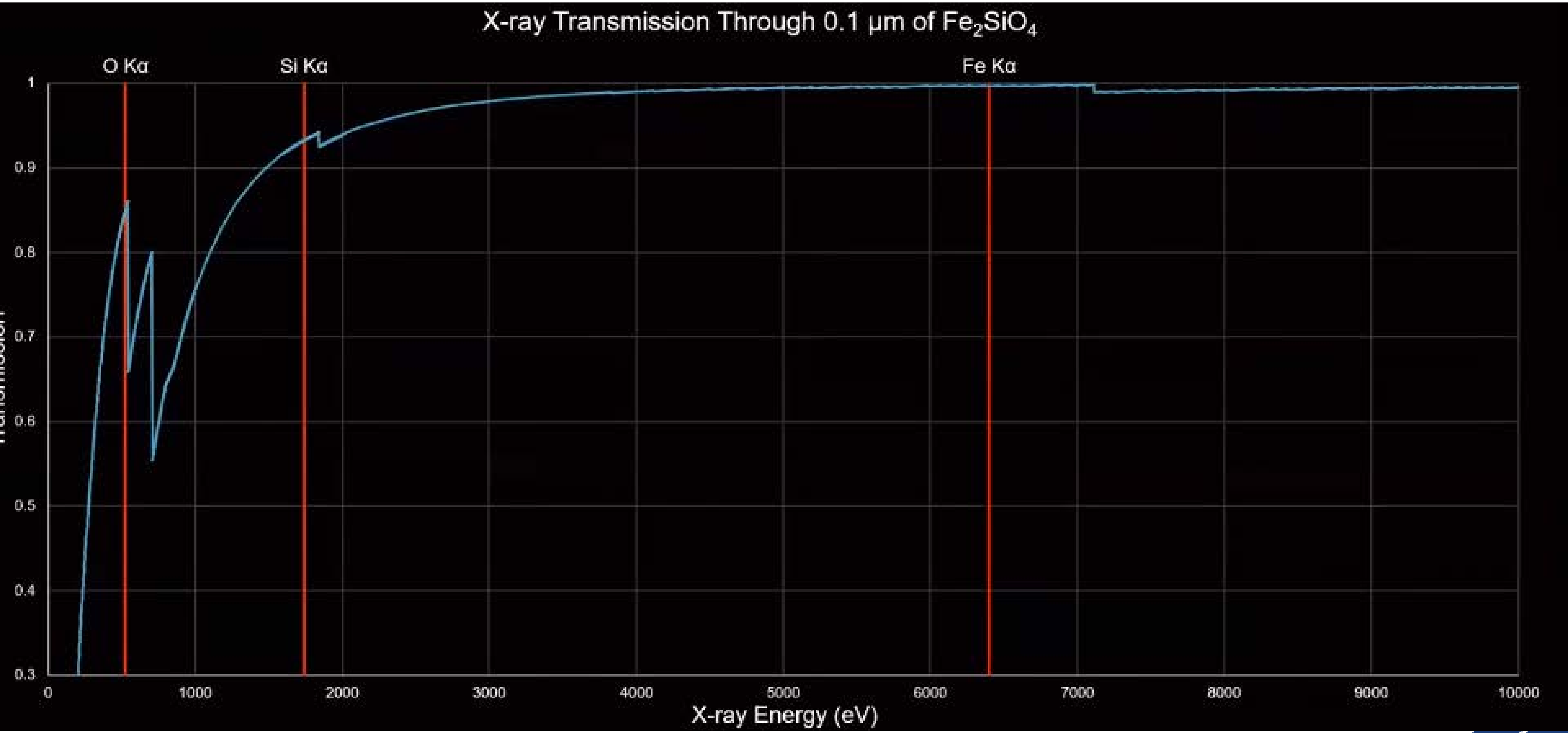
	Net Counts	Conc.(%)
O	485,330	32.2
Na	36,561	2.4
Al	224,445	14.9
Si	637,778	42.3
K	123,245	8.2

Energy Dispersive X-Ray Spectroscopy (EDS/EDX)

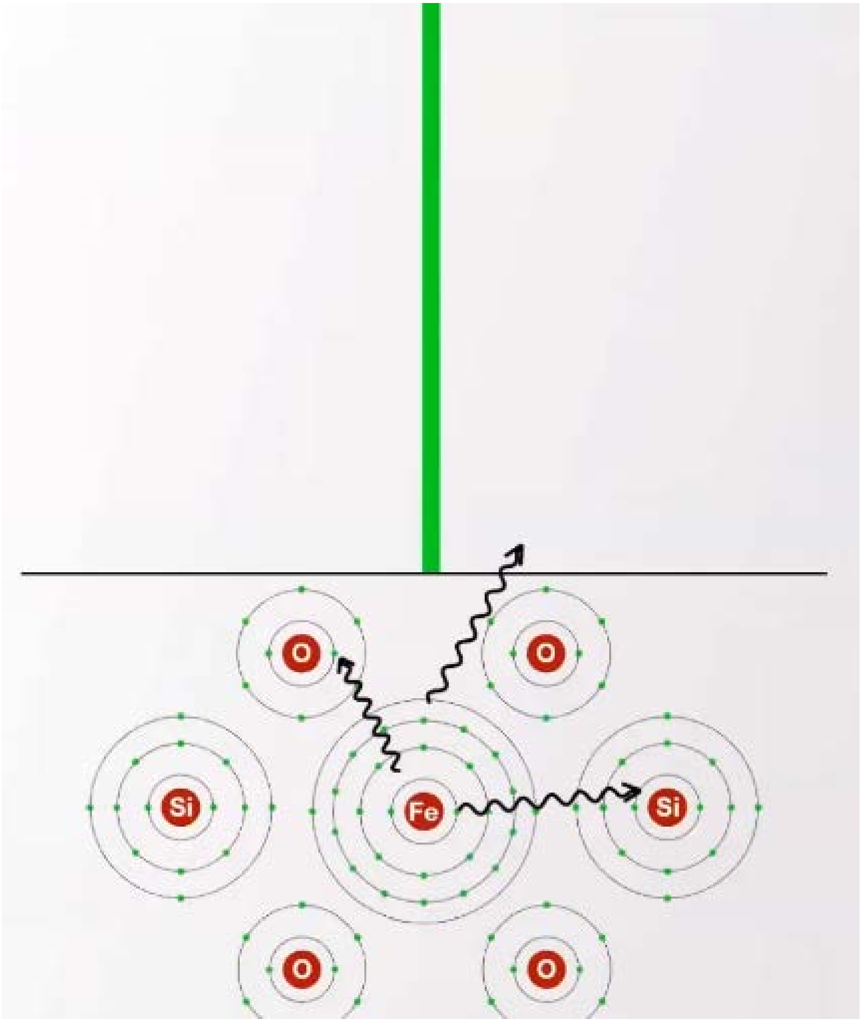


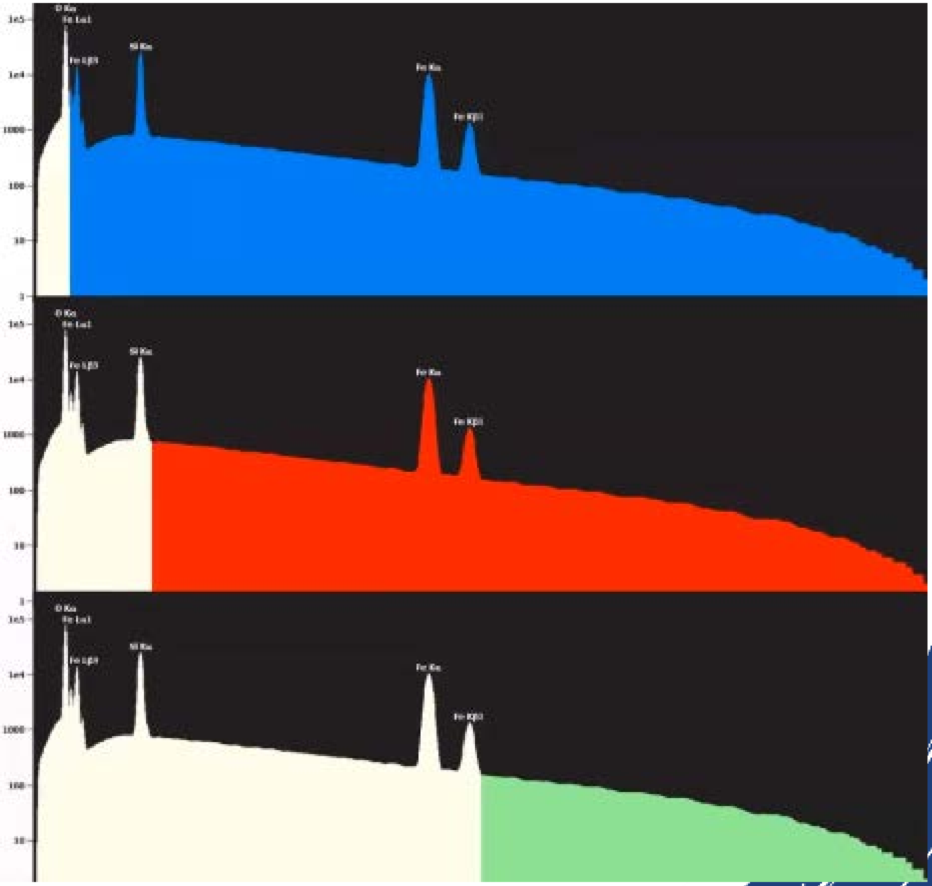


Energy Dispersive X-Ray Spectroscopy (EDS/EDX)



Energy Dispersive X-Ray Spectroscopy (EDS/EDX)





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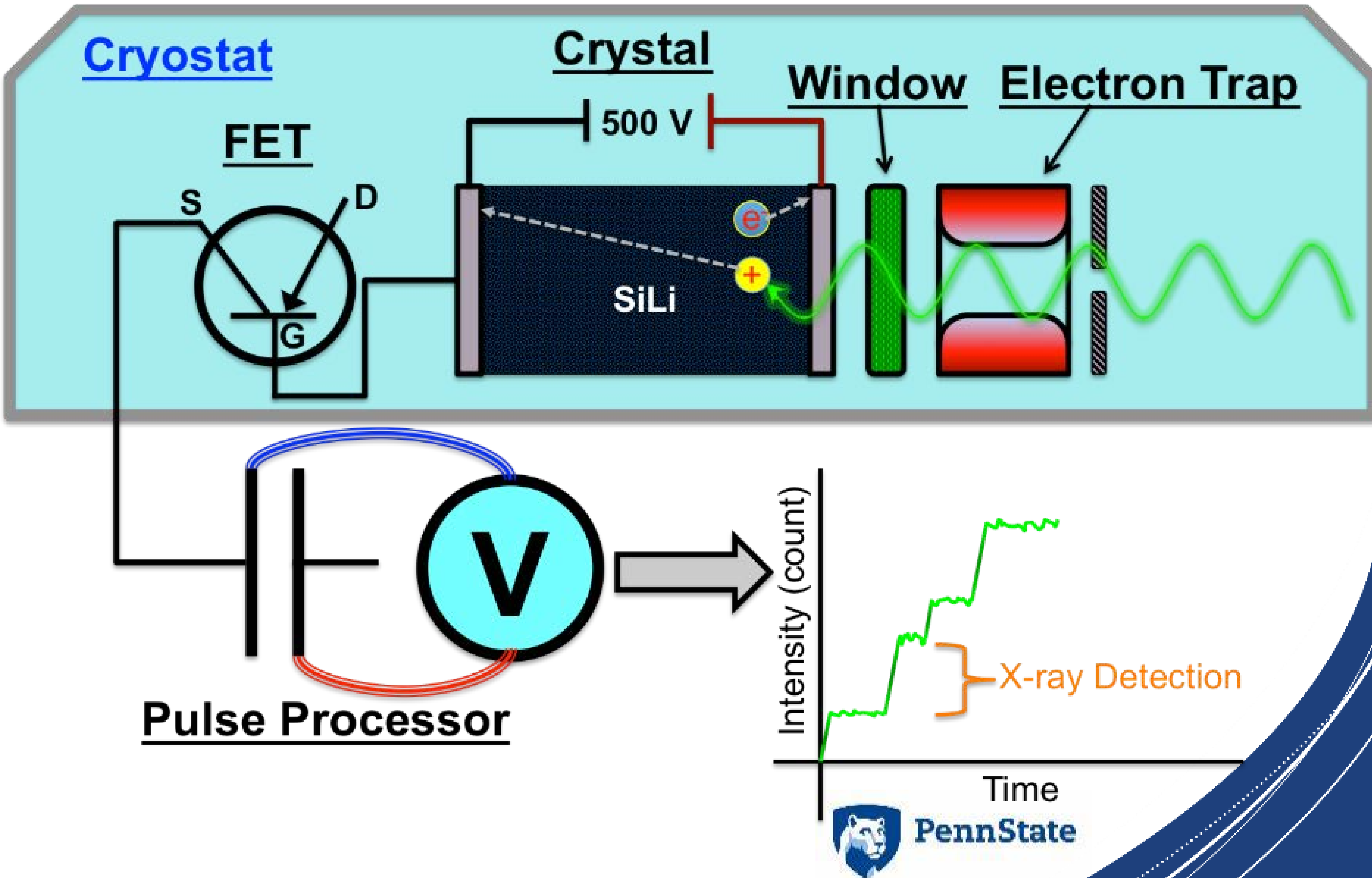
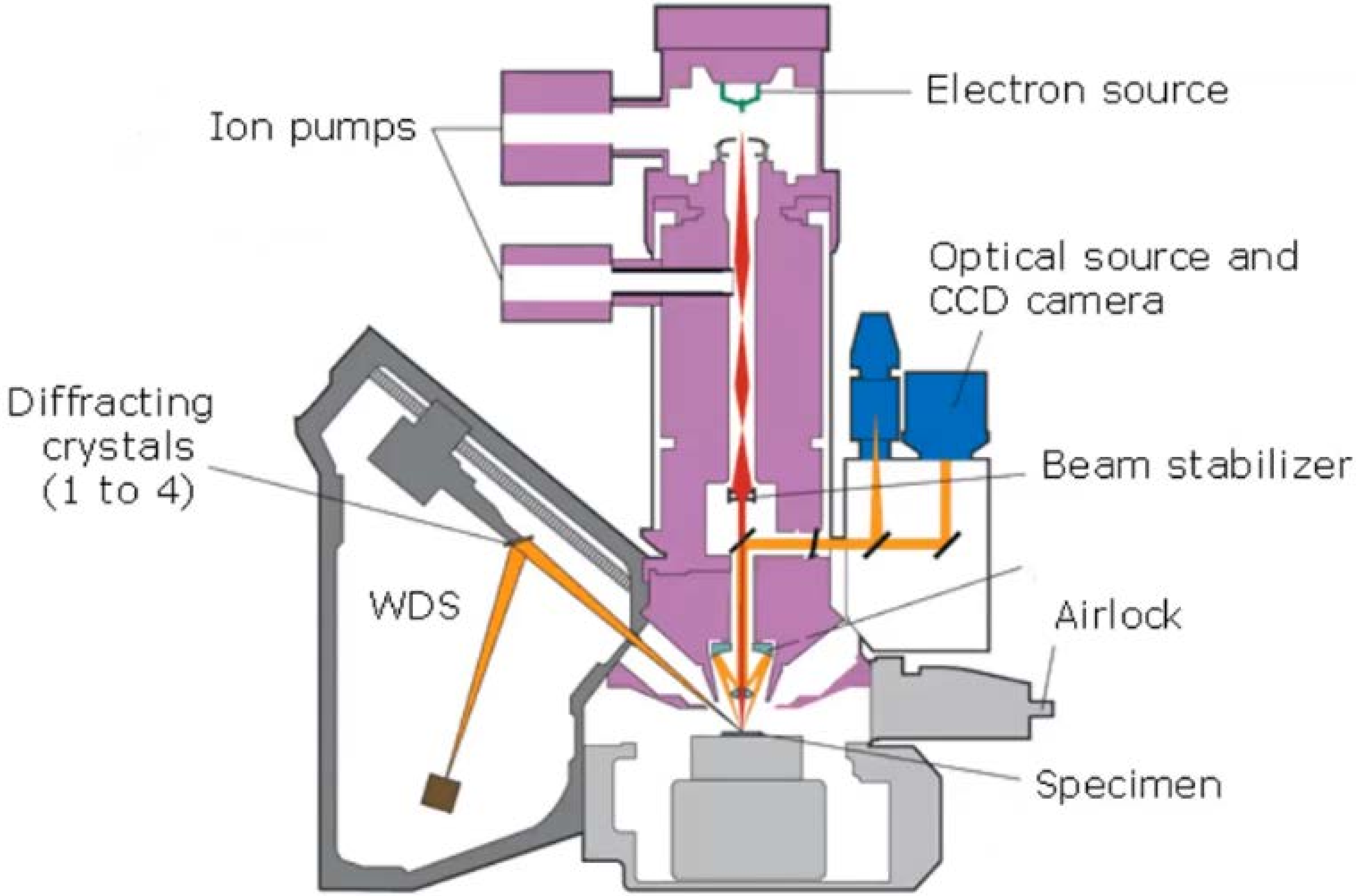
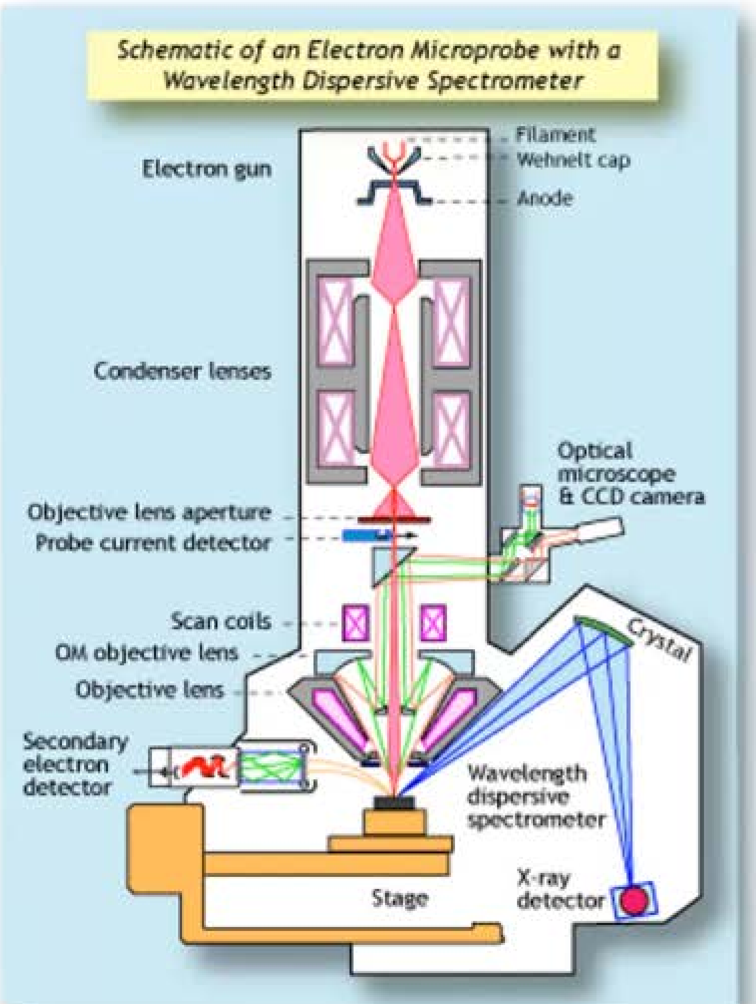
Energy Dispersive X-Ray Spectroscopy (EDS/EDX)

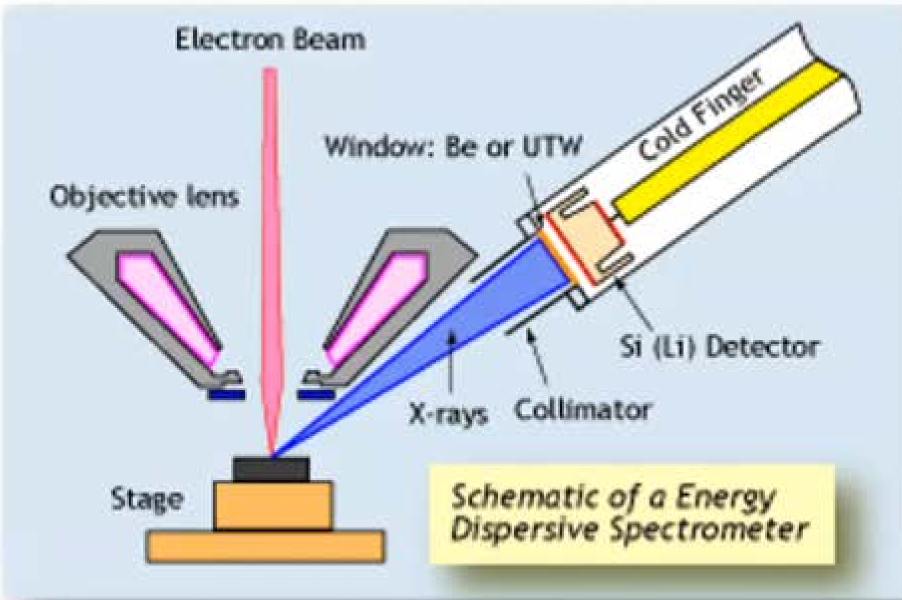


Electron Probe Microanalysis (EPMA)



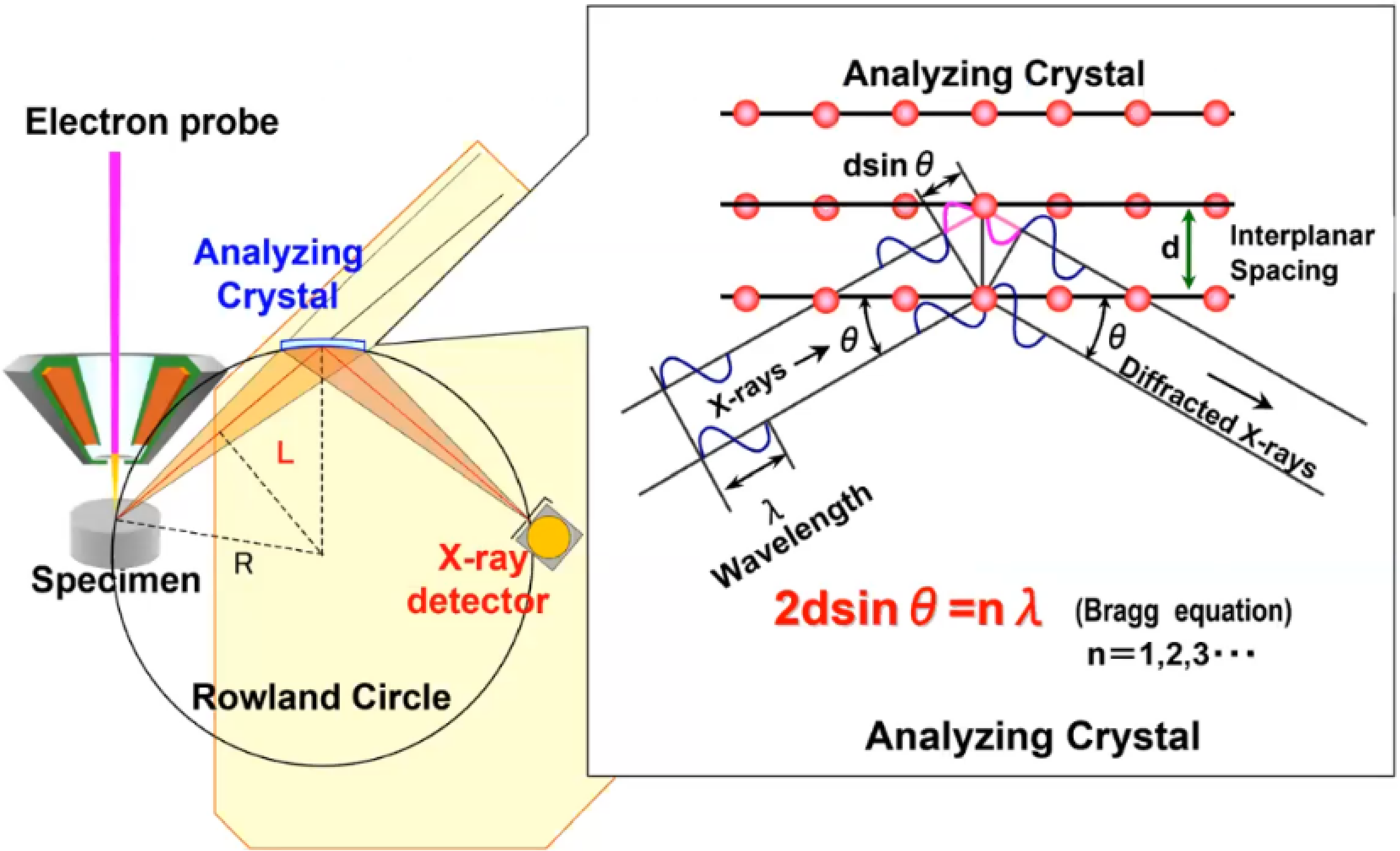
Electron Probe Microanalysis (EPMA)





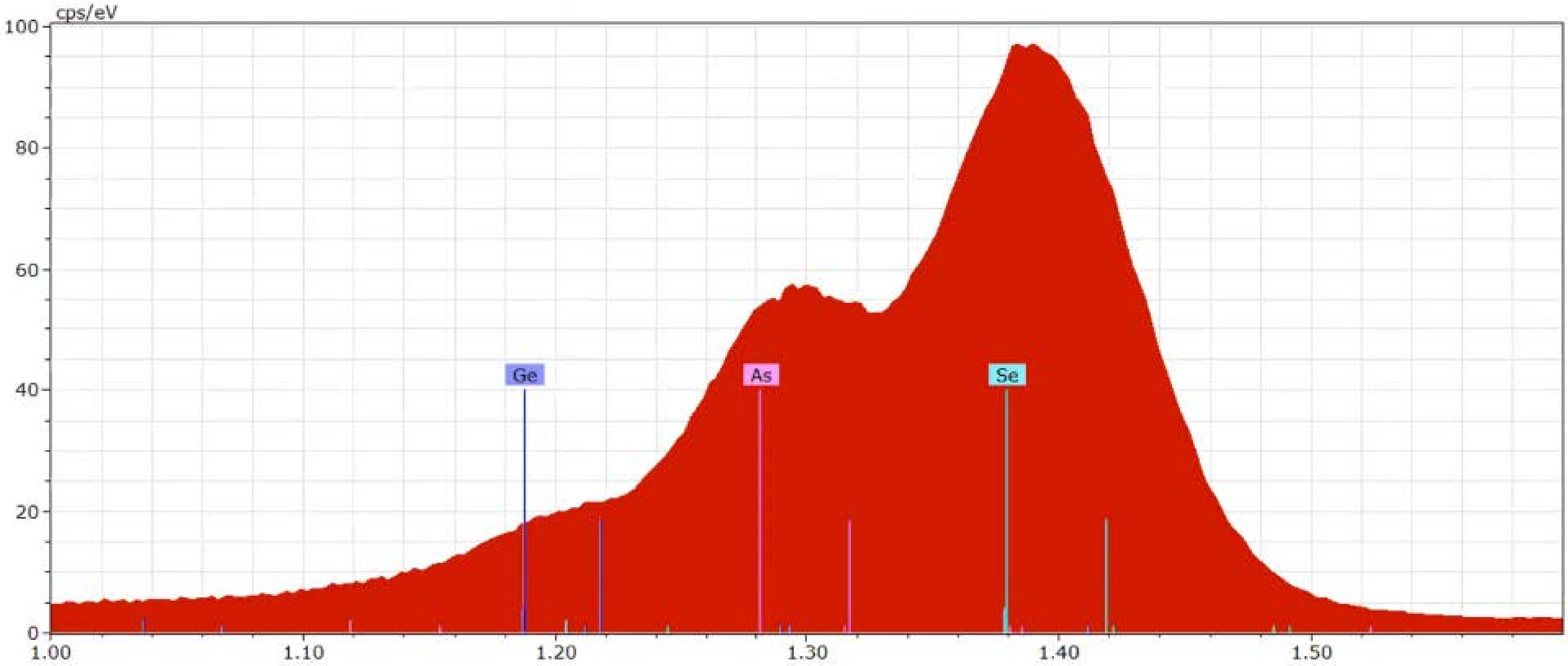
Analysis of characteristic X-rays – 2 Types of data collection

Electron Probe Microanalysis (EPMA)

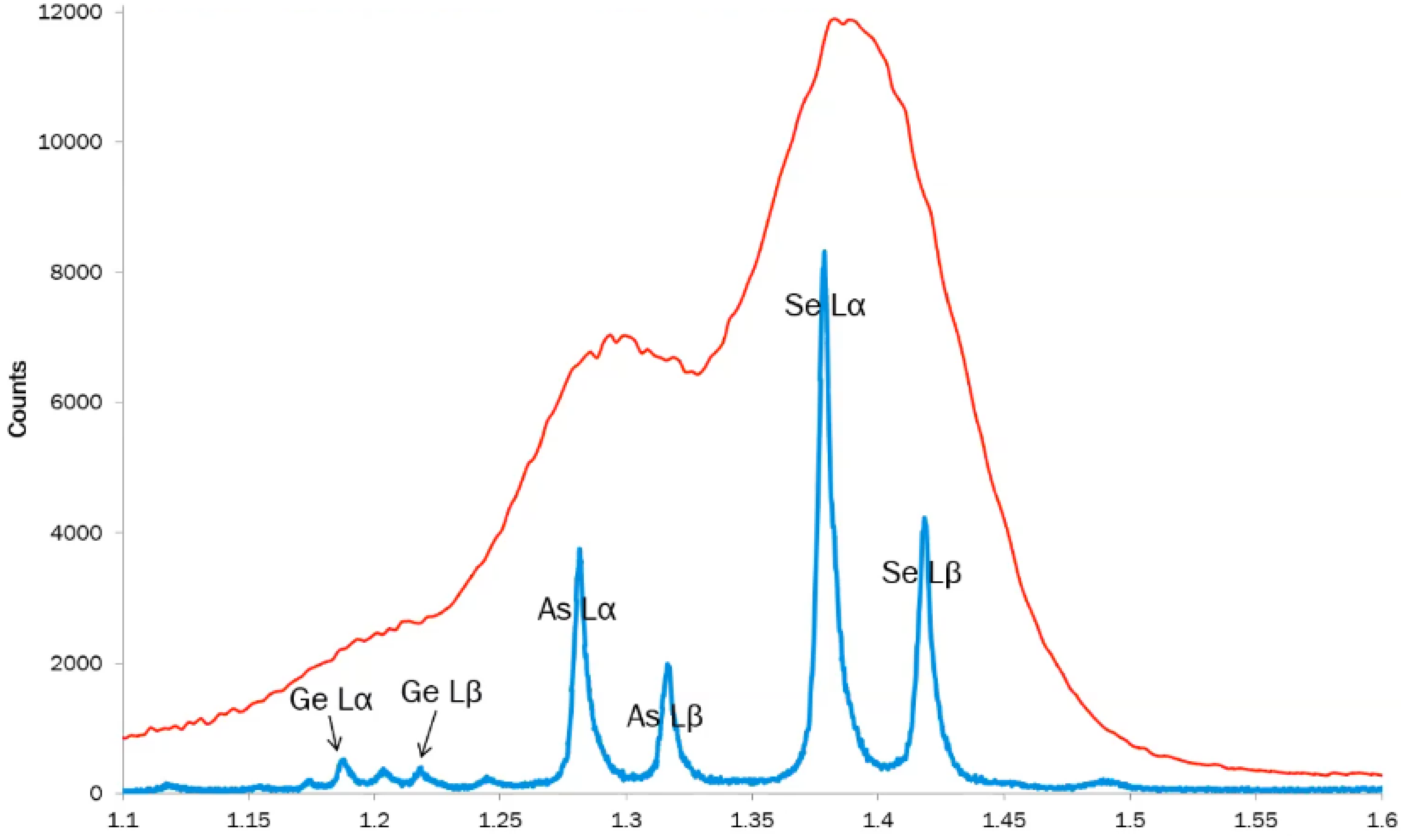


X-ray detection system (WDS)

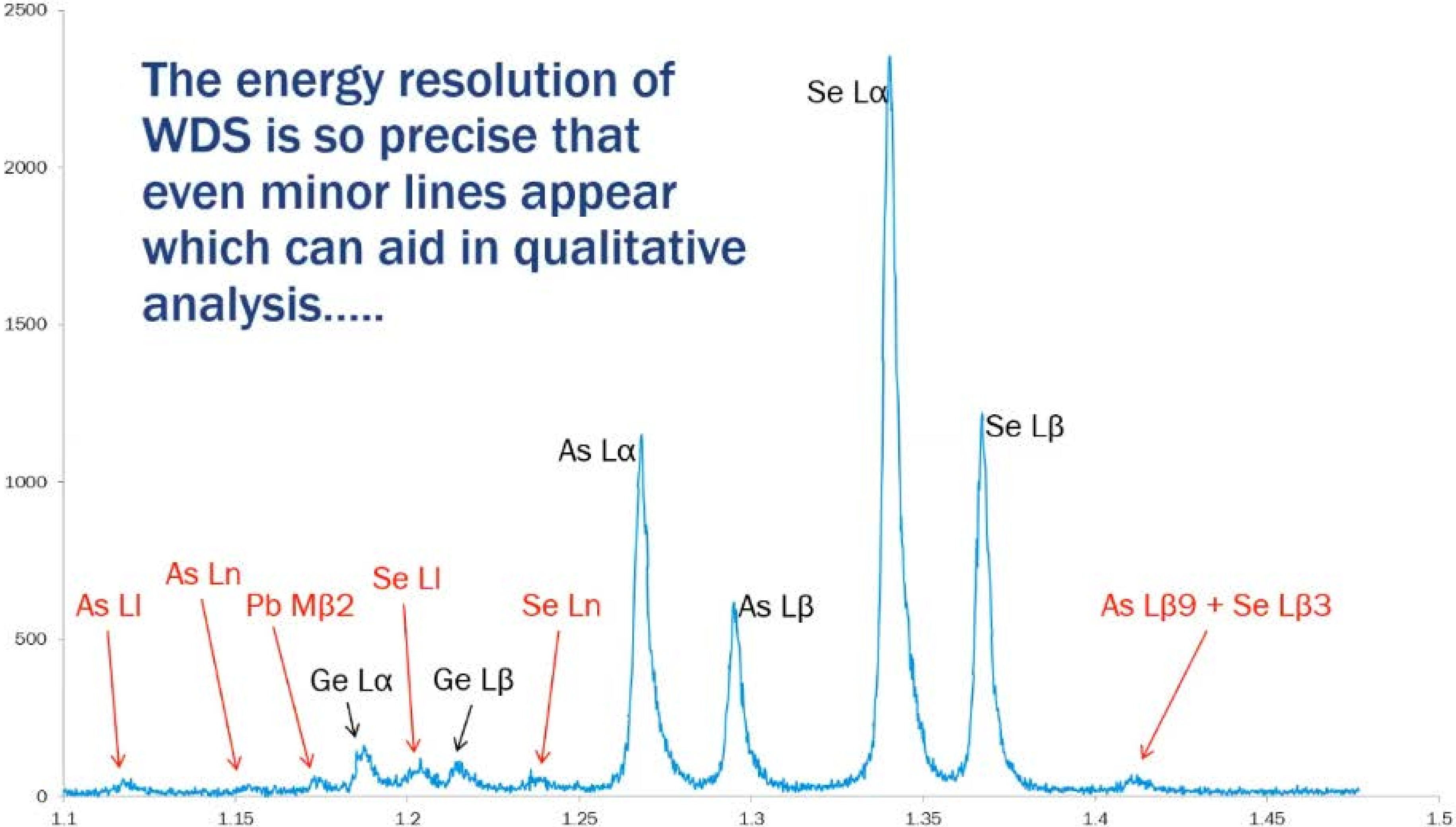
Electron Probe Microanalysis (EPMA)



Electron Probe Microanalysis (EPMA)



Electron Probe Microanalysis (EPMA)



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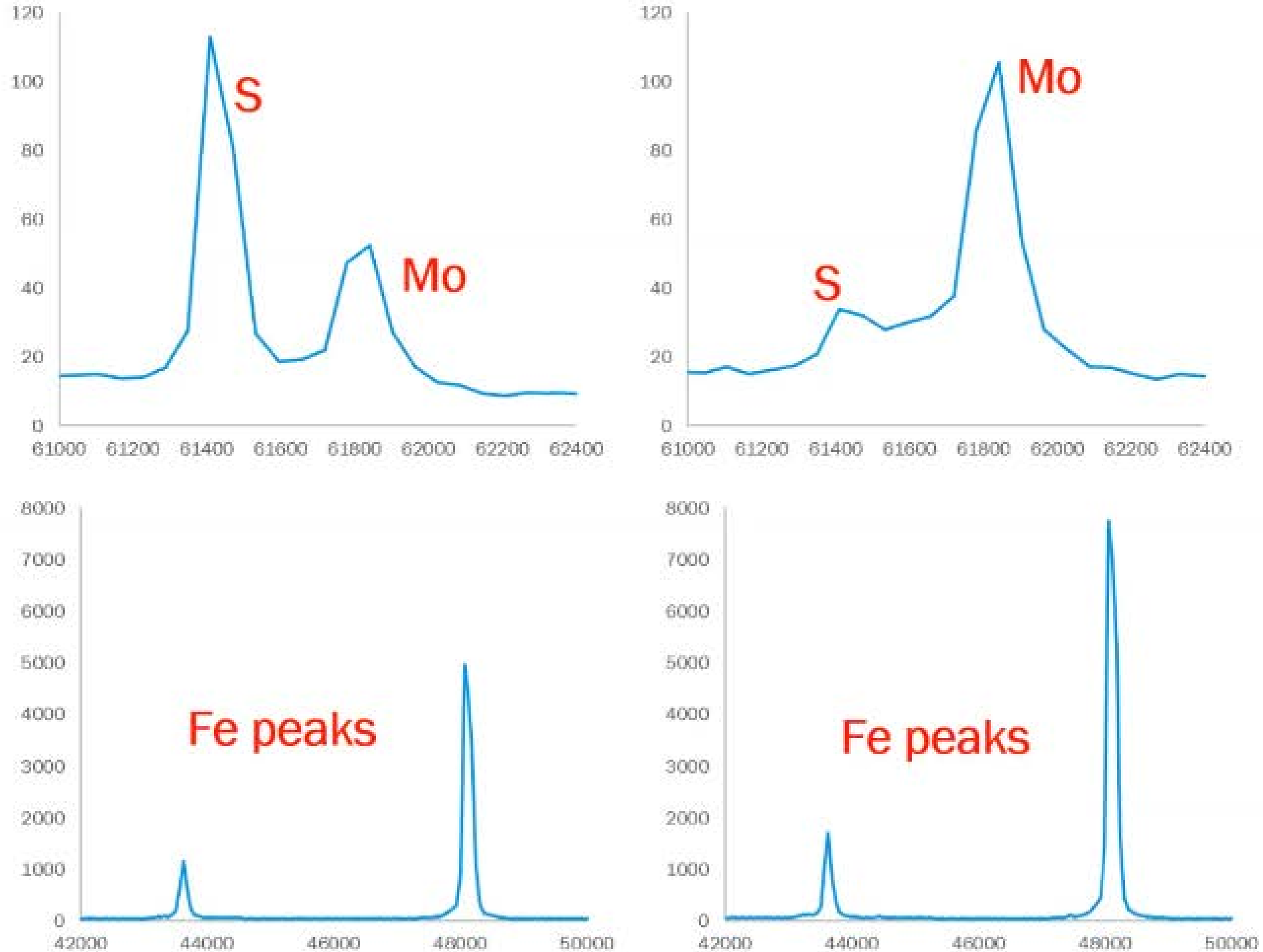
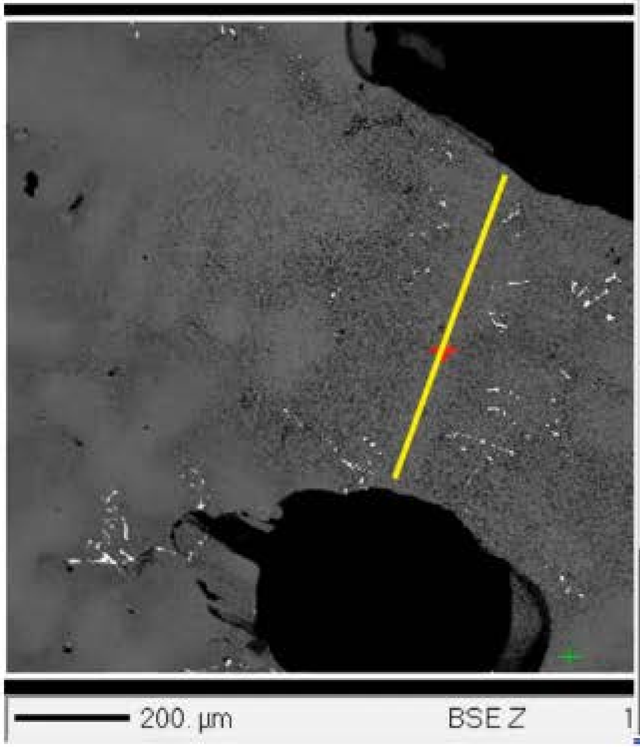
Electron Probe Microanalysis (EPMA)

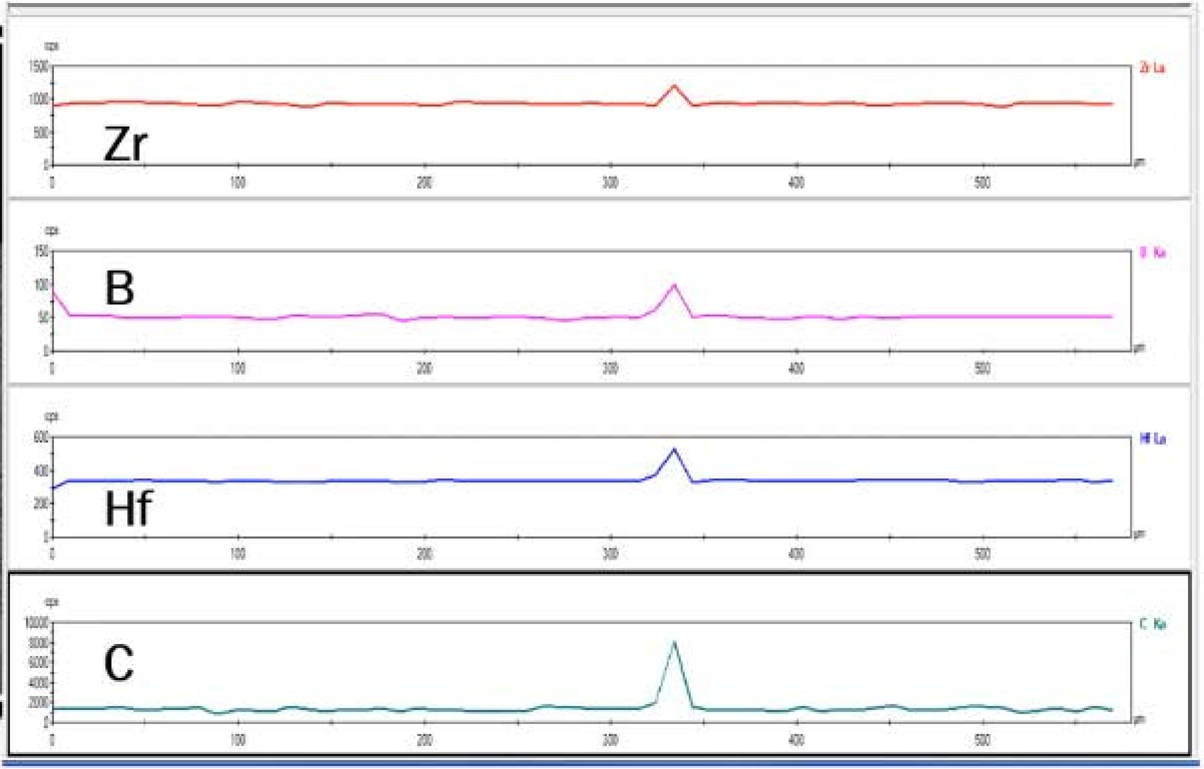
WDS – Compositional difference of elements that overlap in EDS spectra



Electron Probe Microanalysis (EPMA)

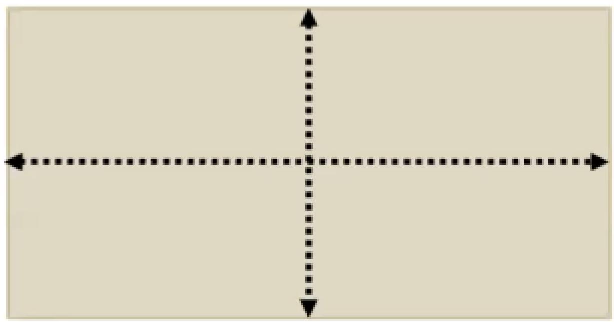
Scan of B, Zr, Hf and C across a grain boundary





Electron Probe Microanalysis (EPMA)

Homogeneity in advanced materials

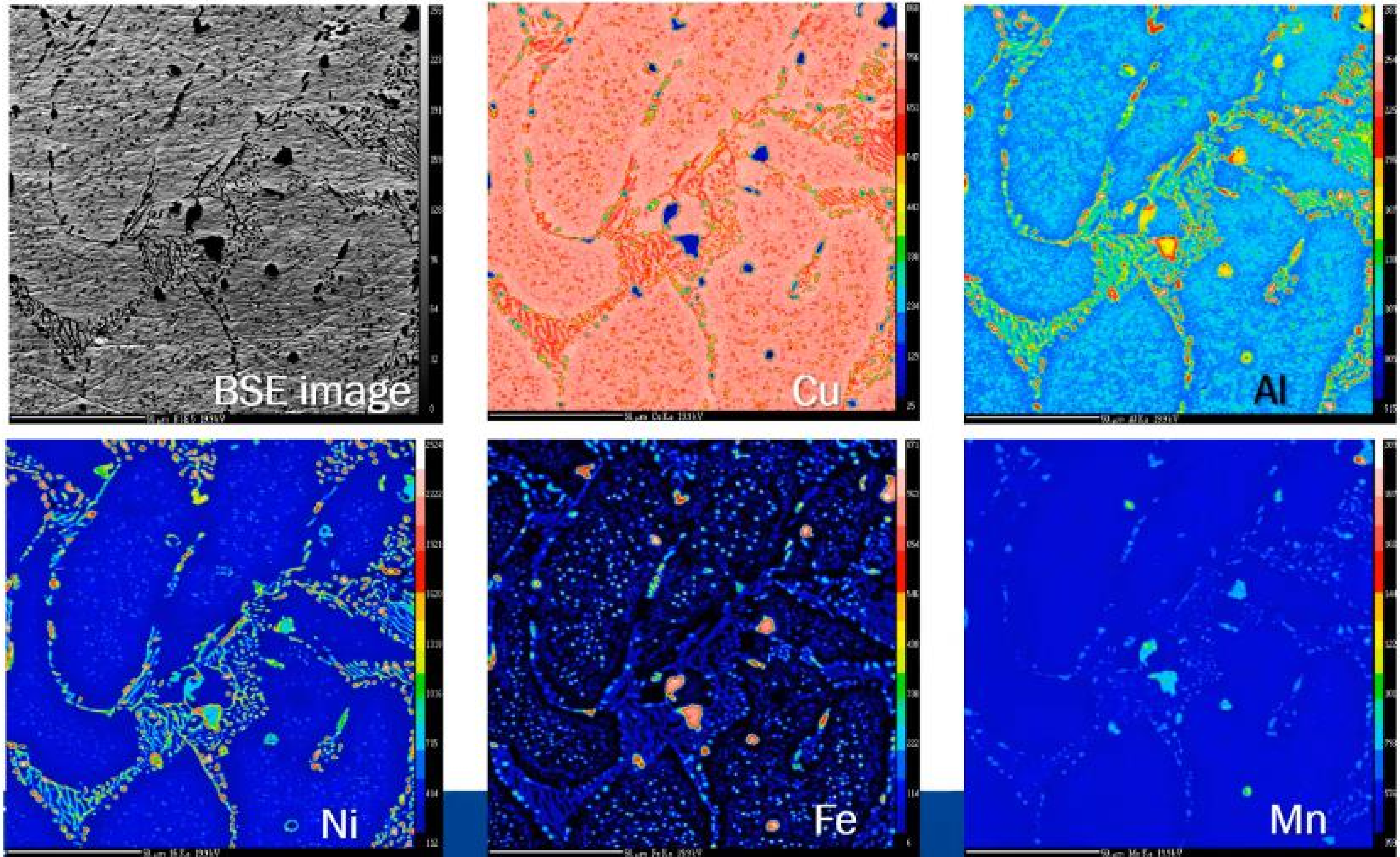


	Horizontal Direction		Vertical Direction	
Element	Wt% avg	2 σ	Wt% avg	2 σ
O	16.736	0.575	16.633	0.482
Mg	1.056	0.023	1.052	0.017
Ti	4.508	0.080	4.516	0.077
Nb	13.046	0.209	13.043	0.157
In	4.432	0.086	4.443	0.077
Pb	61.063	0.642	61.219	0.722

	Horizontal	2 σ	Vertical	2 σ
In/Pb	0.1310	0.0029	0.1310	0.0031
Mg/Nb	0.3094	0.0104	0.3083	0.0070

Electron Probe Microanalysis (EPMA)

Phase ID in bronze samples

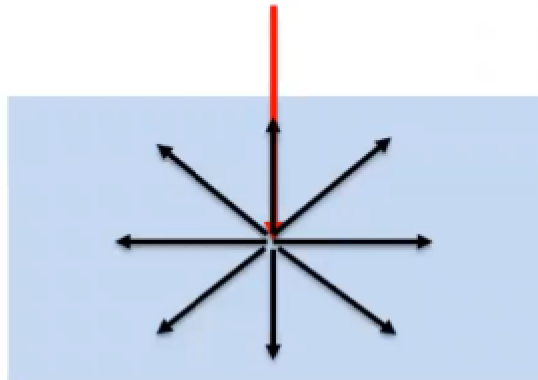


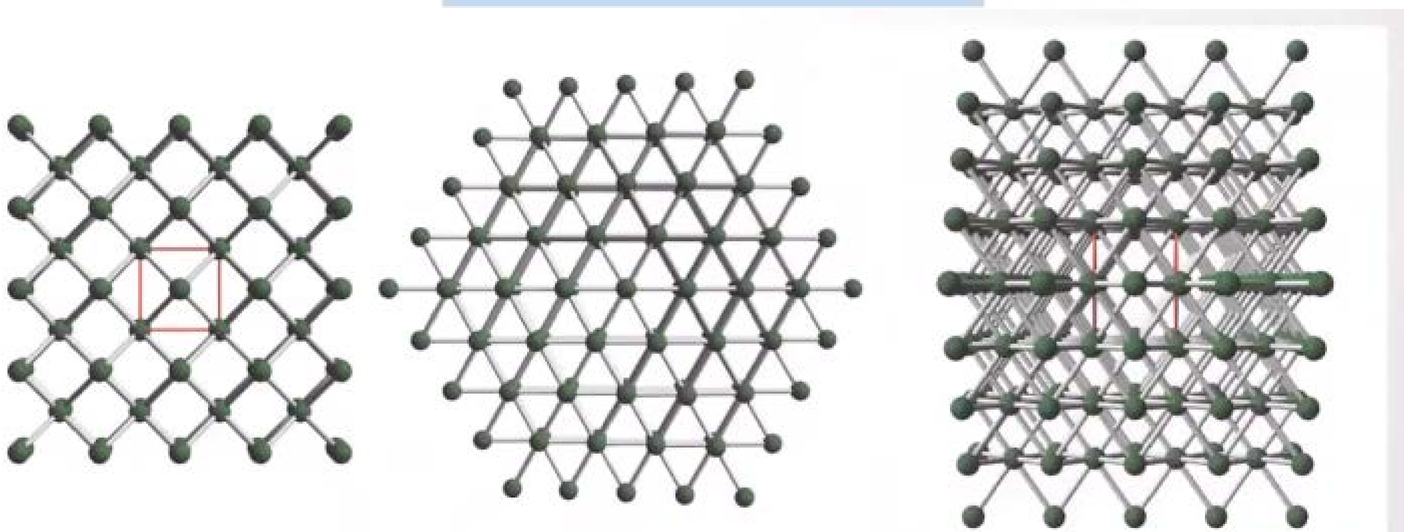
Electron Probe Microanalysis (EPMA)

- Both qualitative and quantitative information can be obtained
- Sampling volume ~1 cubic micron
- Useful for:
  - Non-destructive elemental analysis
  - Determining exact stoichiometry
  - Detection and identification of contaminants down to the 10s or 100s of ppm
  - Mapping out phases precisely – even with only slight variation
  - Quality control to ensure uniform manufactured composition of materials over time
  - Separation of elements that overlap badly in EDS analysis
  - Higher precision, accuracy and repeatability over EDS
- Major limitations: no "wet" samples, micron volume not nano

Electron Backscatter Diffraction

The effect of scattering is to create a 'point' source of electrons, with all possible trajectories, within the material.

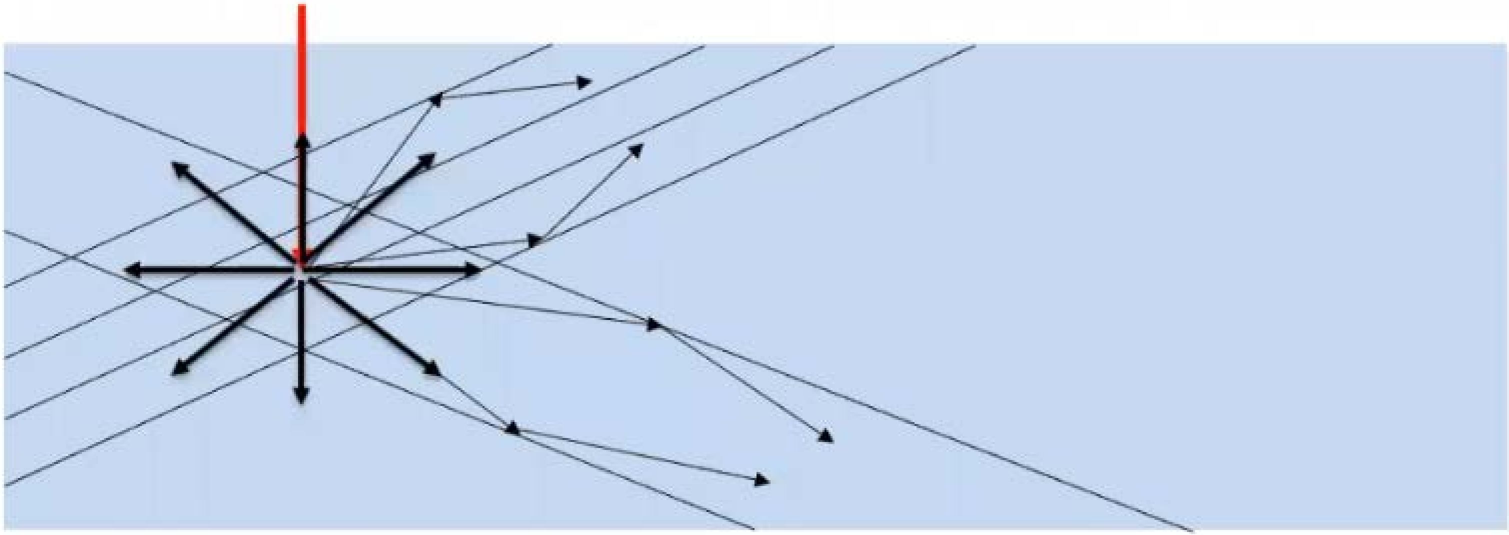




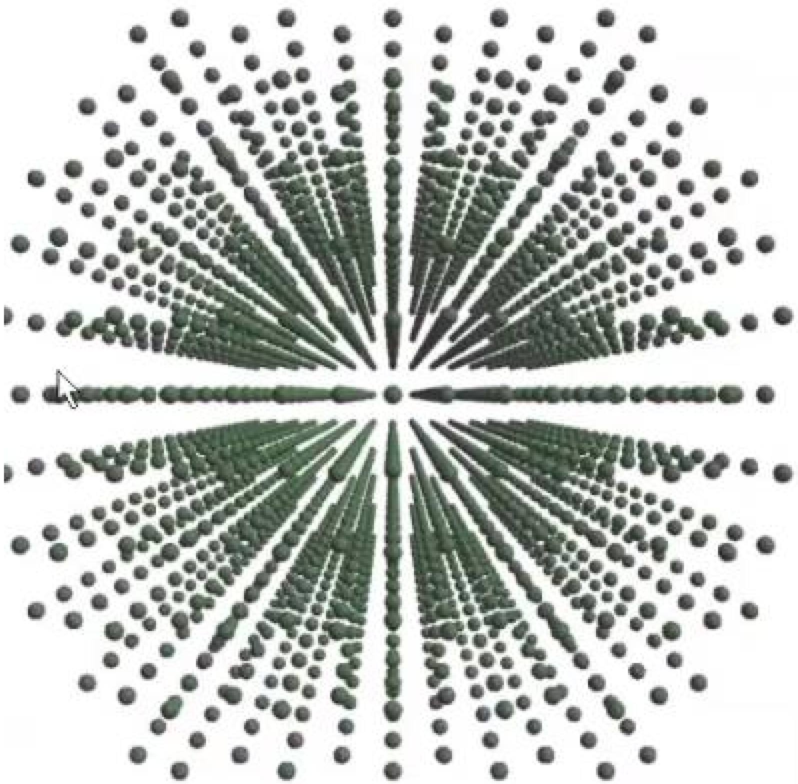
Views of a small lump of crystal down [001], [111] & [110] crystal directions – looking straight on a cube face, down a cube diagonal and at a cube edge.

Electron Backscatter Diffraction

If the material is crystalline, electrons will be diffracted by lattice planes where the Bragg condition is satisfied

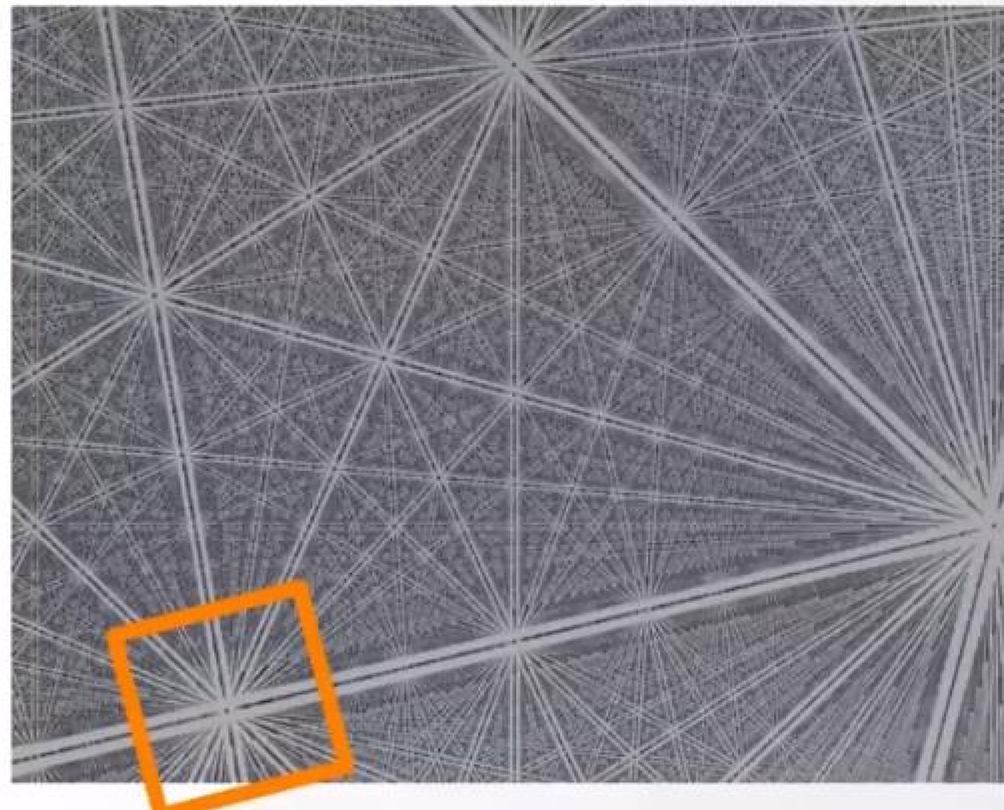


Electron Backscatter Diffraction



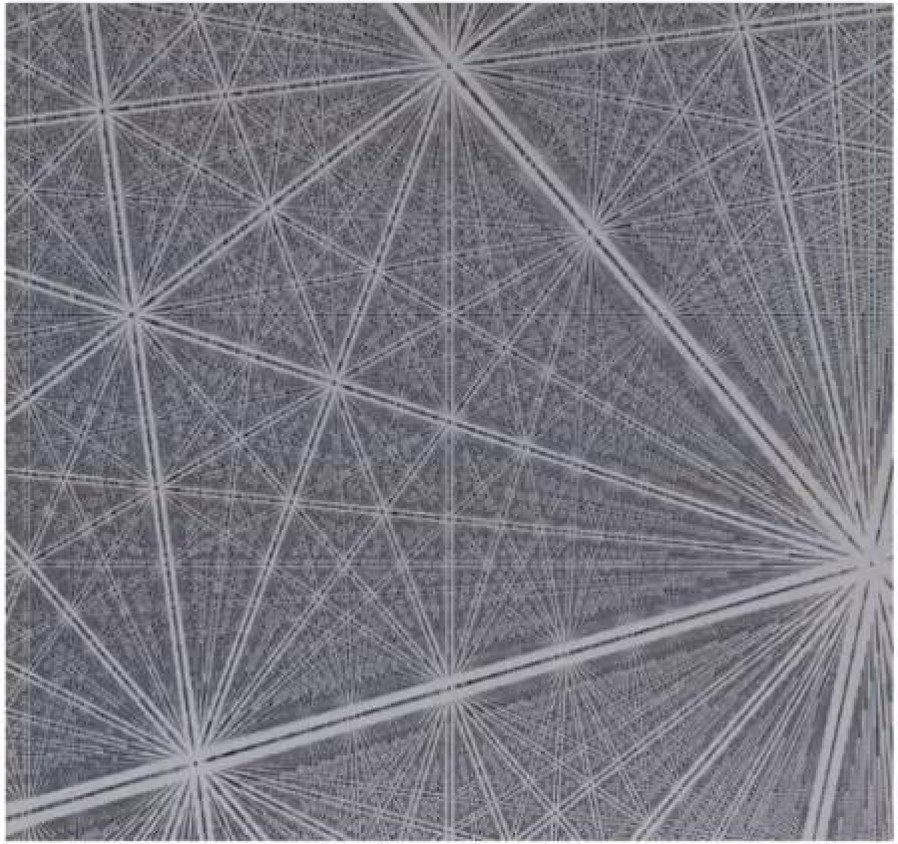
Iron "crystal" ~20Å diameter & 80Å deep, looking down a [110] direction (edge of a cube is horizontal)

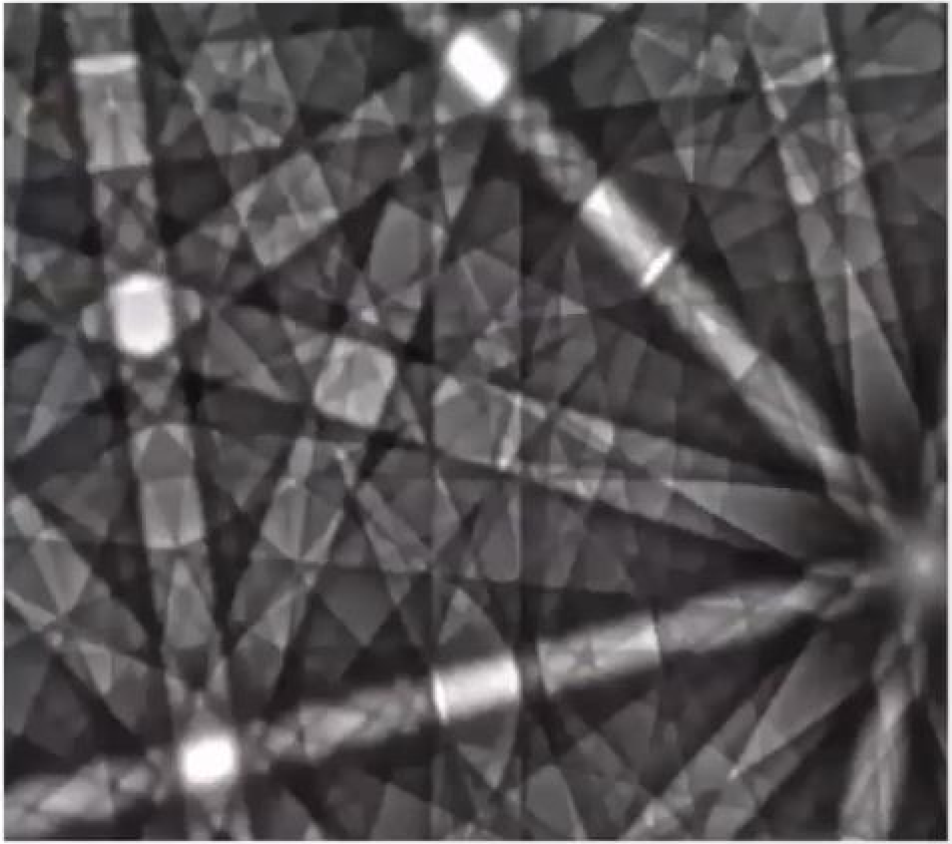
Kikuchi Lines



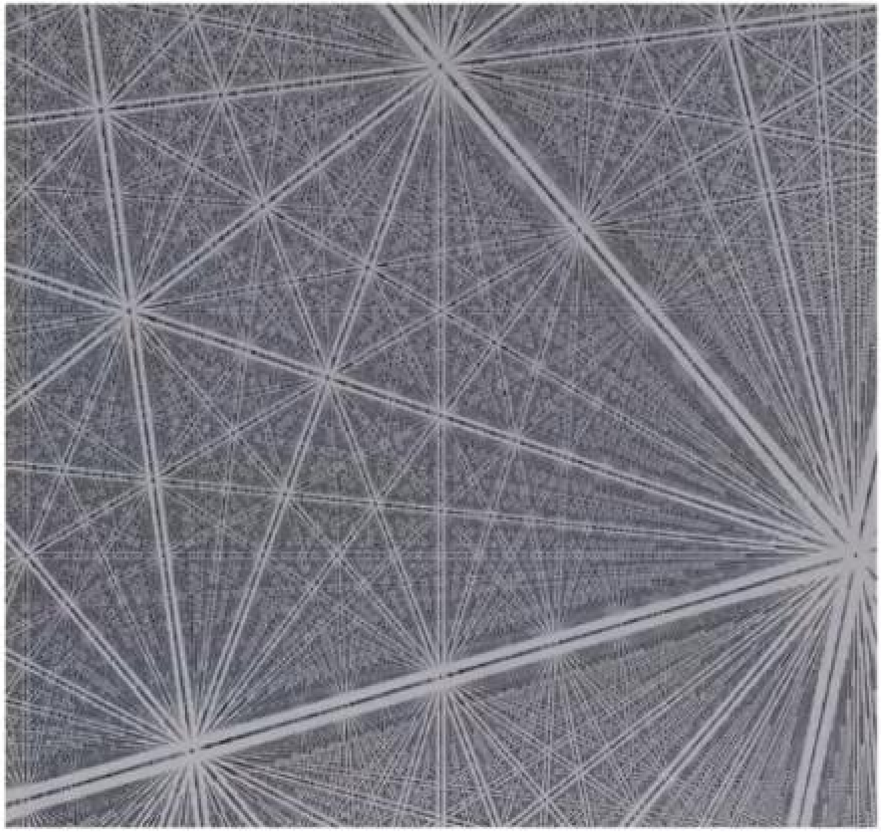
Add lots more atoms and look in a more interesting direction

Electron Backscatter Diffraction

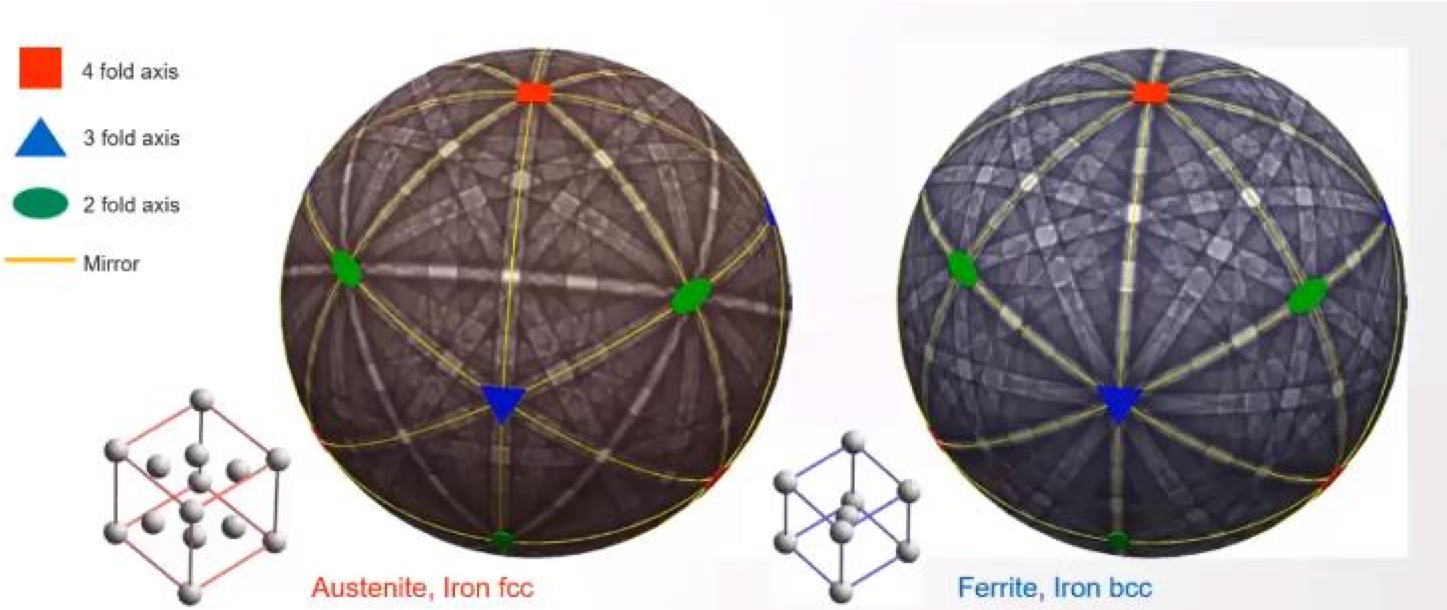


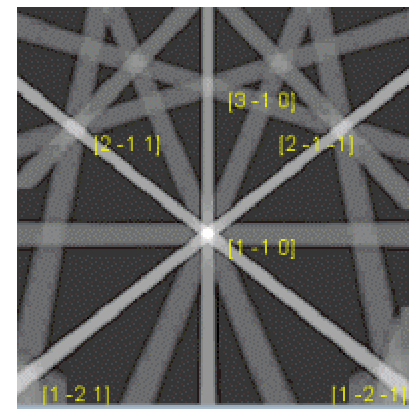


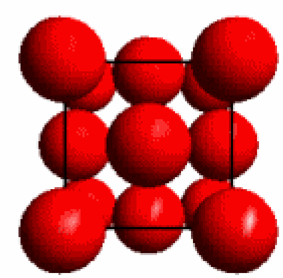
Electron Backscatter Diffraction



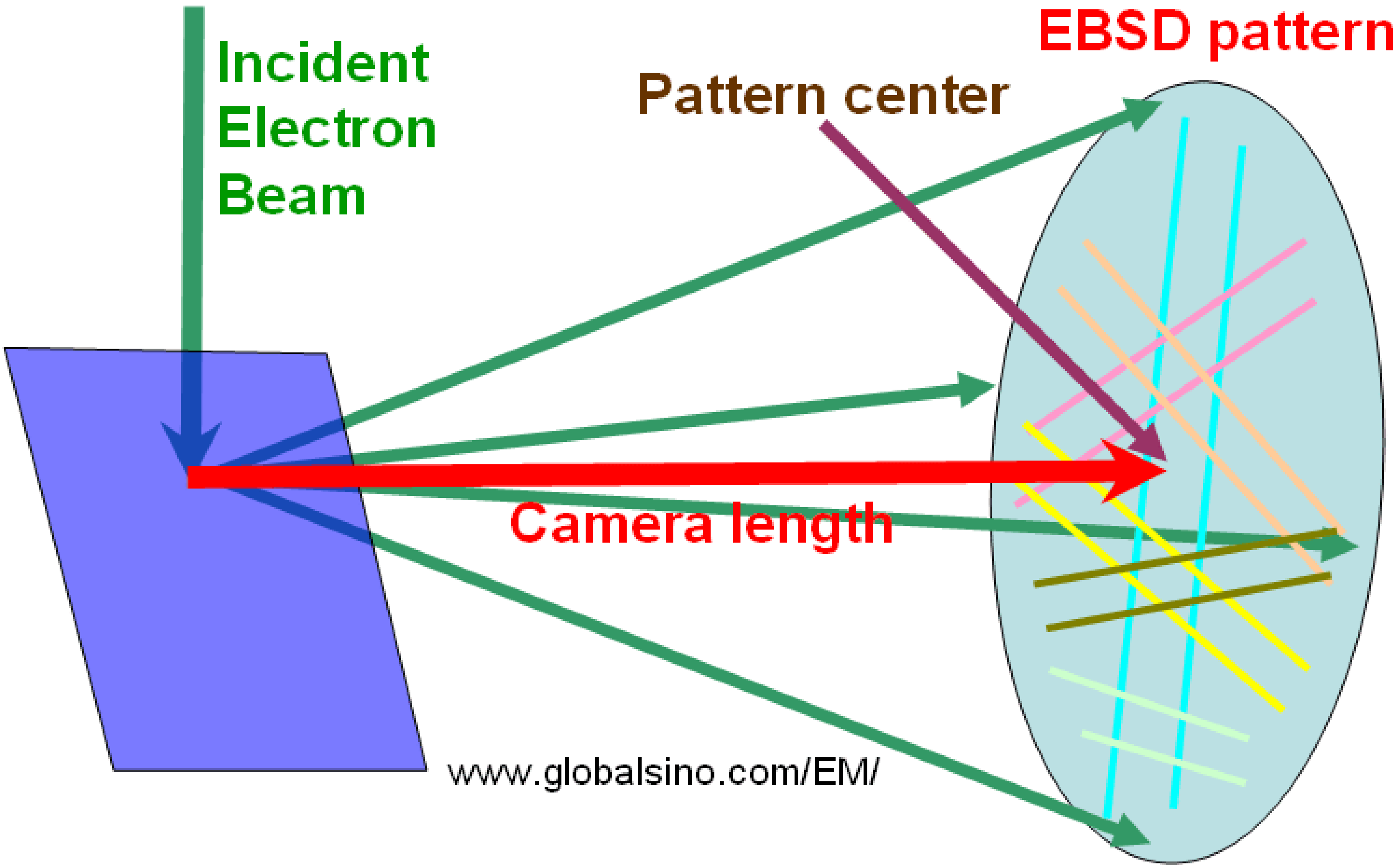
Electron Backscatter Diffraction



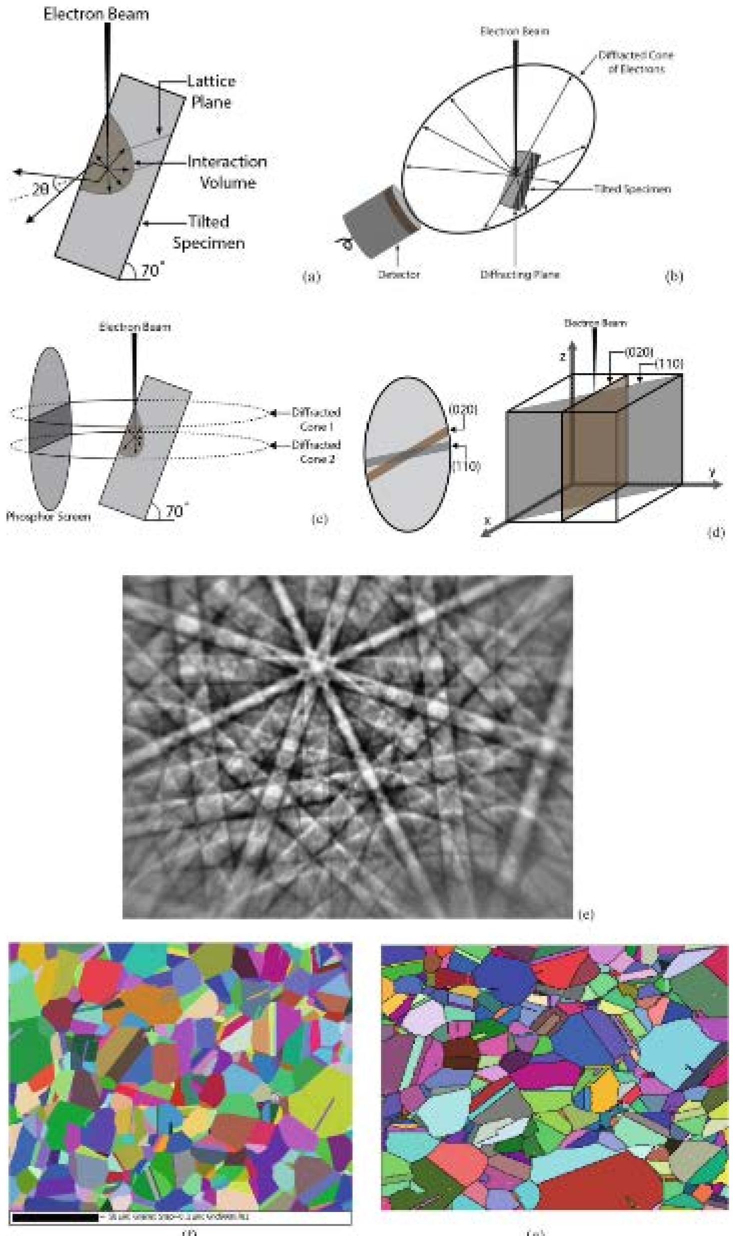


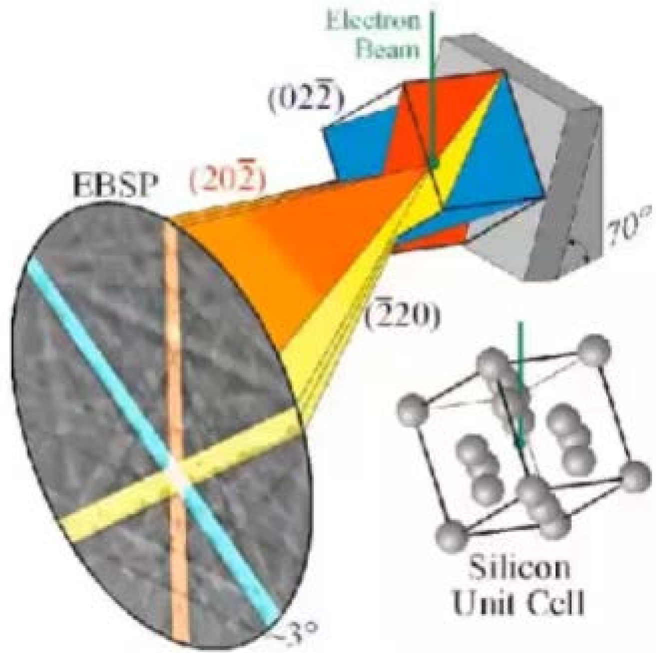


Electron Backscatter Diffraction



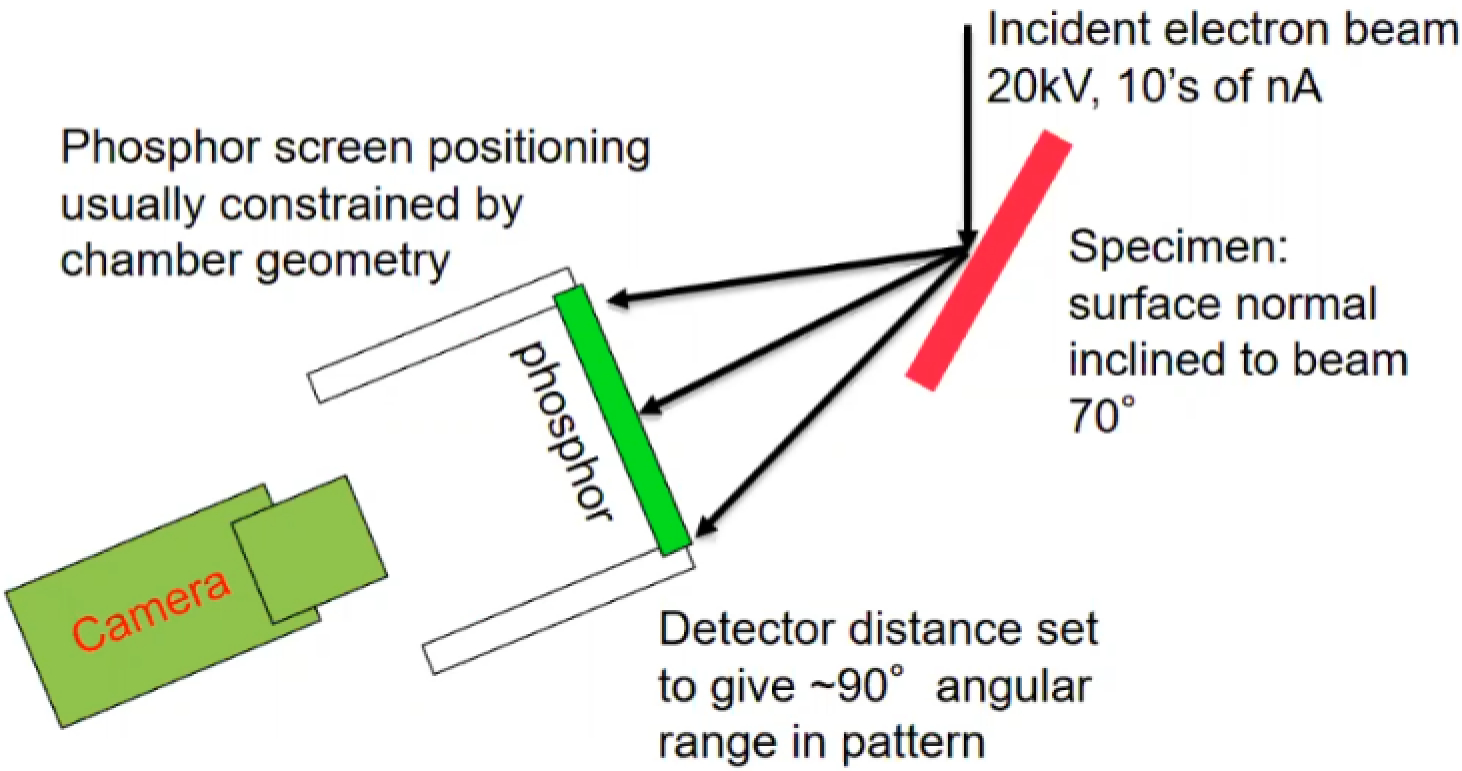
Electron Backscatter Diffraction





Electron Backscatter Diffraction

Typical EBSD set-up



Electron Backscatter Diffraction

EBSD provides a wealth of sample information

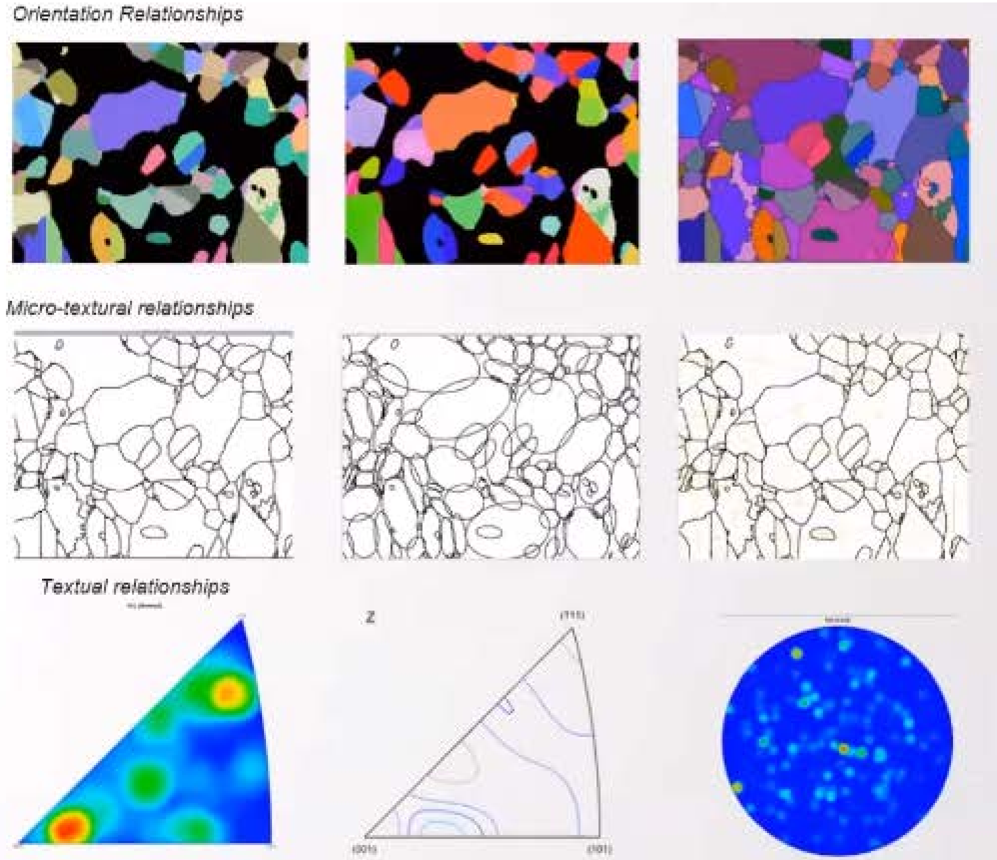
- Grains
  - *Size, distribution, morphology, internal deformation, crystallographic alignment, slip system analysis…*
- Grain boundaries
  - *Boundary axis*
  - *Boundary angle*
  - *Special boundaries, e.g., Twins / CSL*
- Texture – macro & micro

Phases

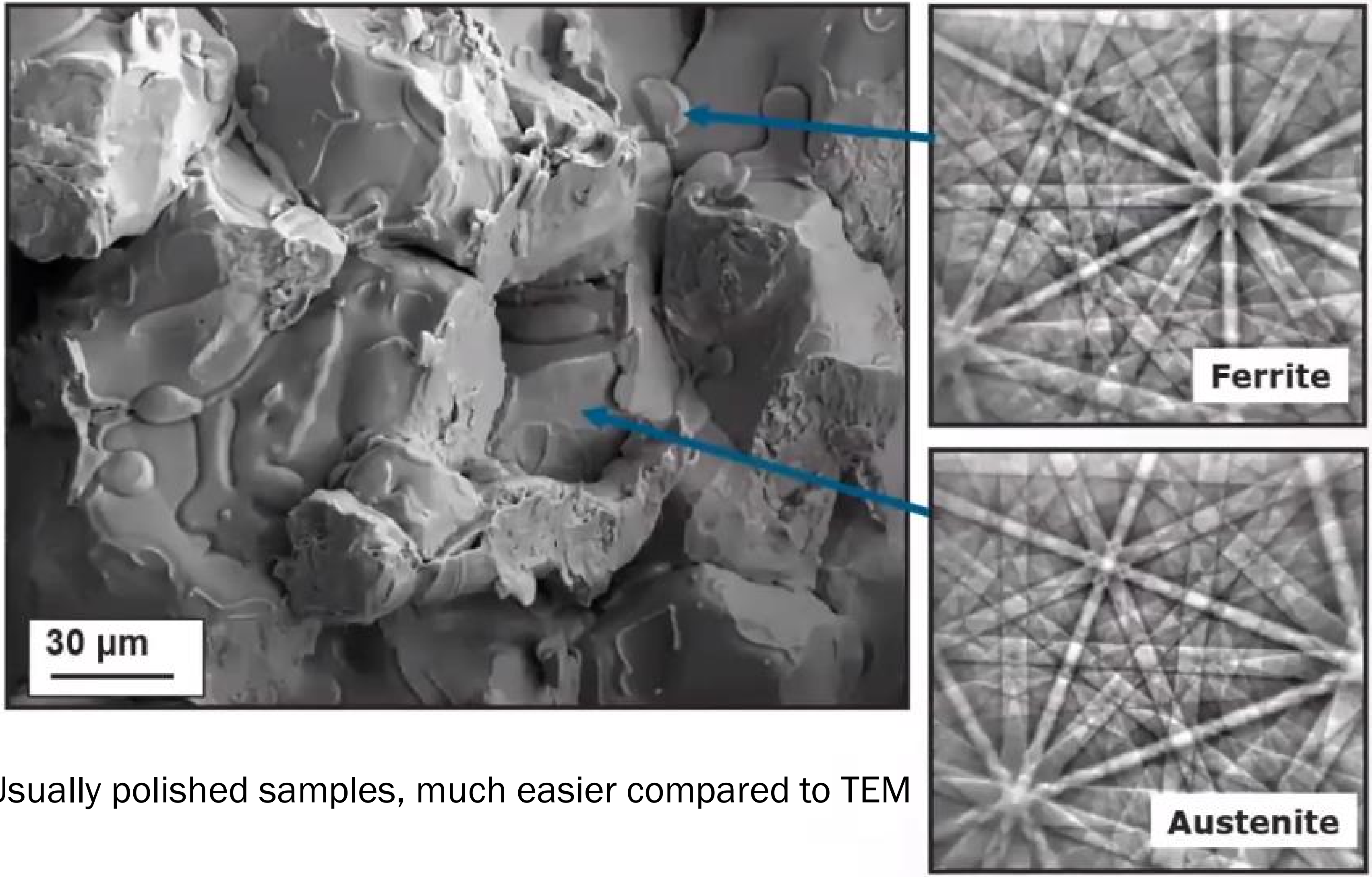
- Identification, discrimination and distribution
- Interphase relationships, transformations

Deformation

- HR EBSD strain analysis - cross correlation

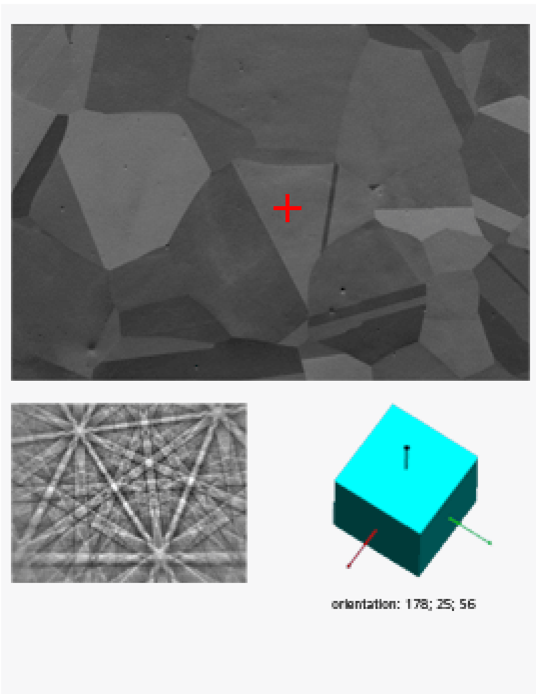


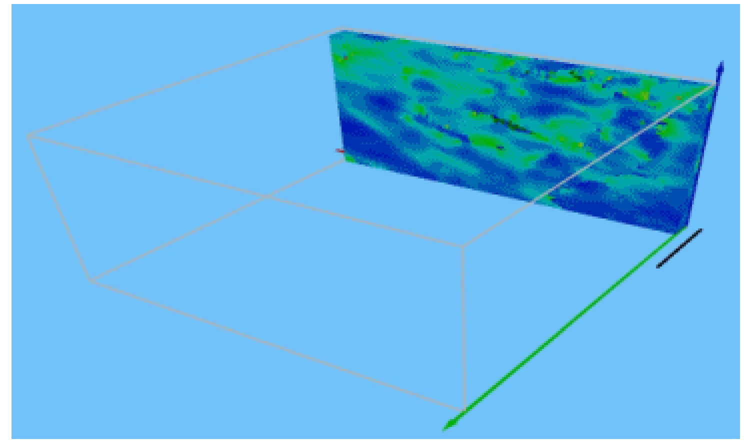
Electron Backscatter Diffraction

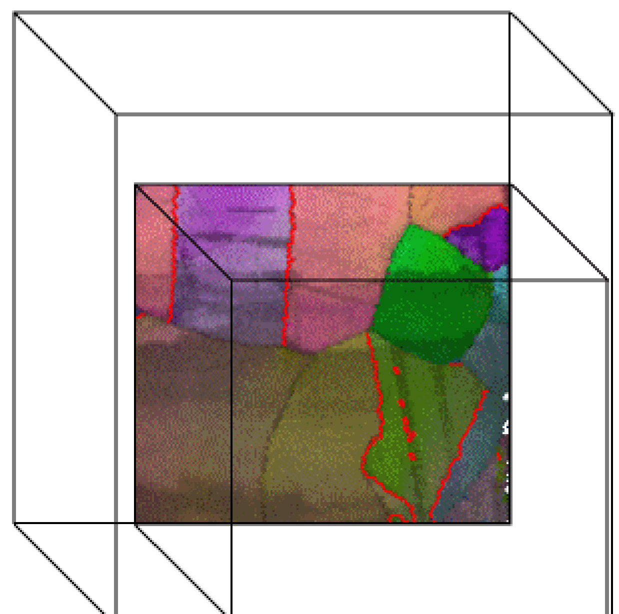


Usually polished samples, much easier compared to TEM

Electron Backscatter Diffraction







Building College-University Partnerships for Nanotechnology Workforce Development

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