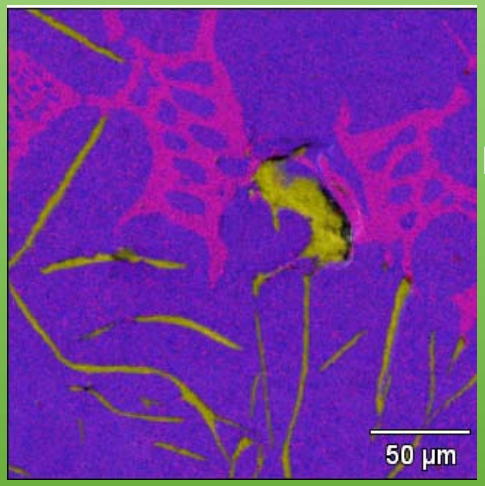
HOW TO APPROACH **SCANNING ELECTRON MICROSCOPY** AND **ENERGY DISPERSIVE SPECTROSCOPY** ANALYSIS

SCSAM Short Course

Amir Avishai

RESEARCH QUESTIONS

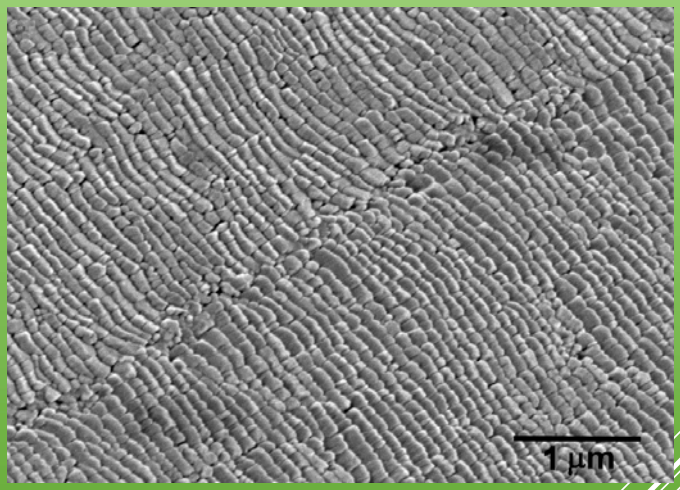


Cast Iron
EDS+SE

Fe
Cr
C

Objective
Ability to ask the right questions!

Sea Shell



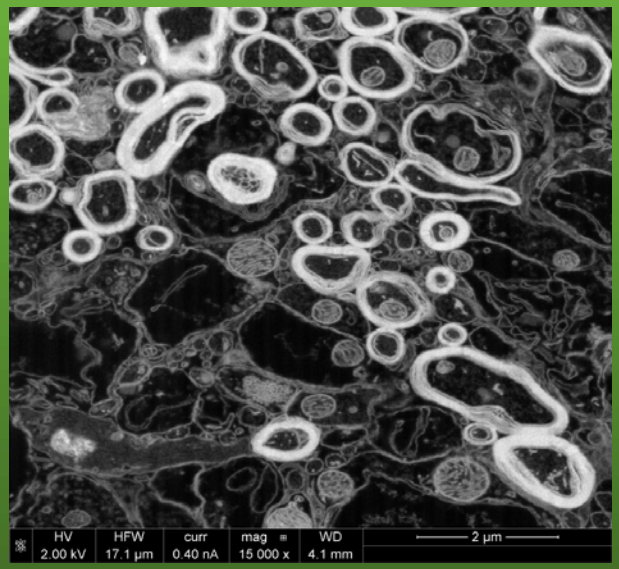
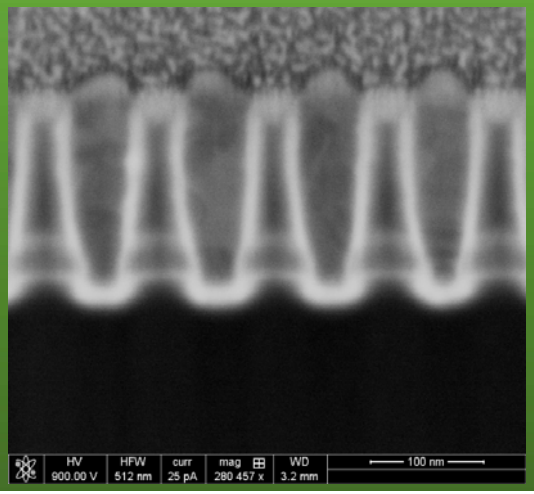
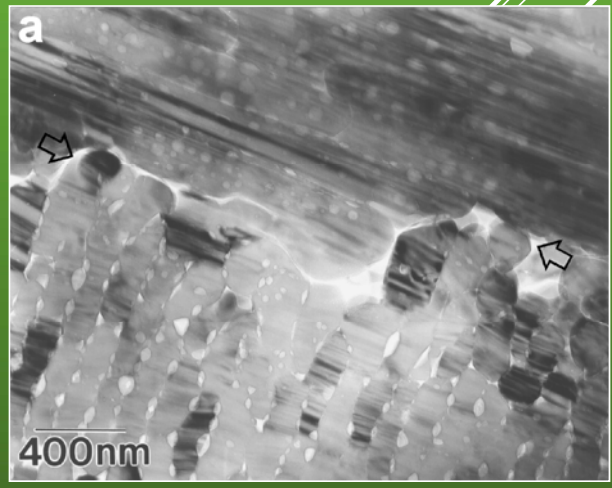


Image showing detail of axons and myelin sheaths, Mitochondria.

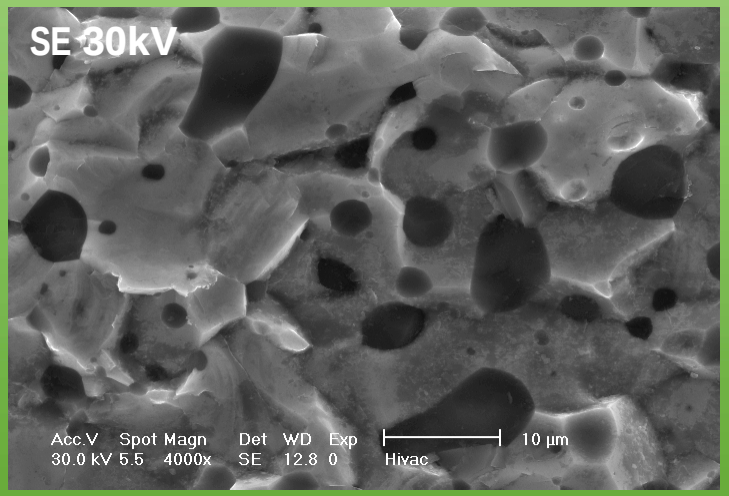
50nm Cu Vias

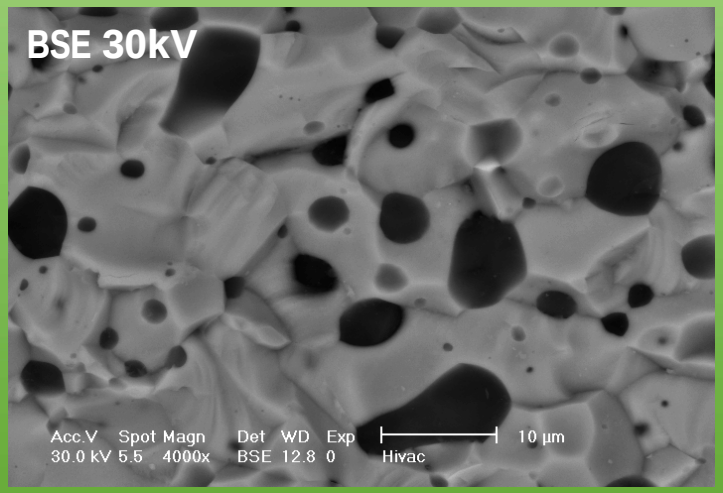


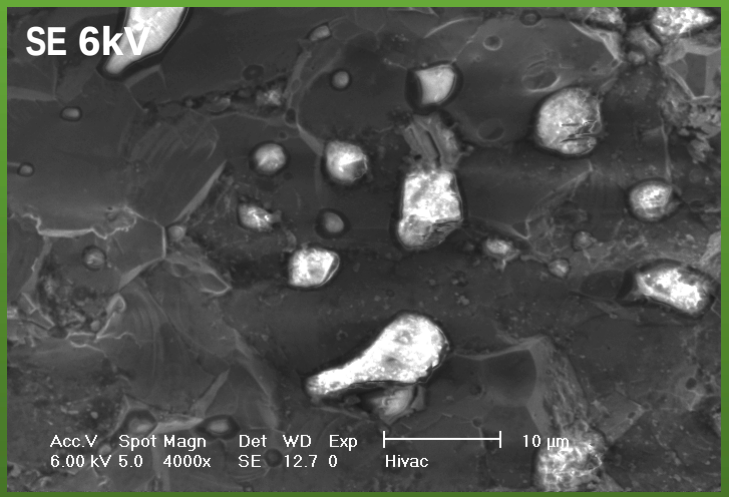
First Order Lamellar Interface

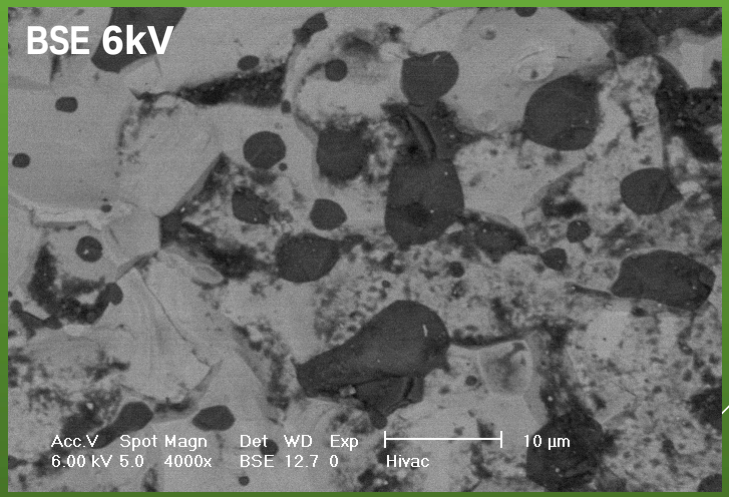


CHARACTERIZATION IS PART OF THE EXPERIMENT!









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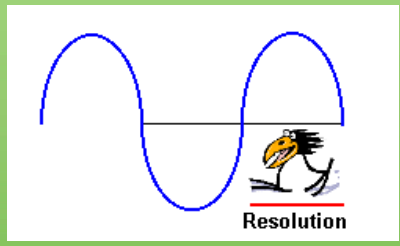
“POKE AND LISTEN”

Dr. Wayne Jennings

Source/Beam/Probe → Interaction/Signal → Detector → Data Interpretation / *Contrast mechanisms*

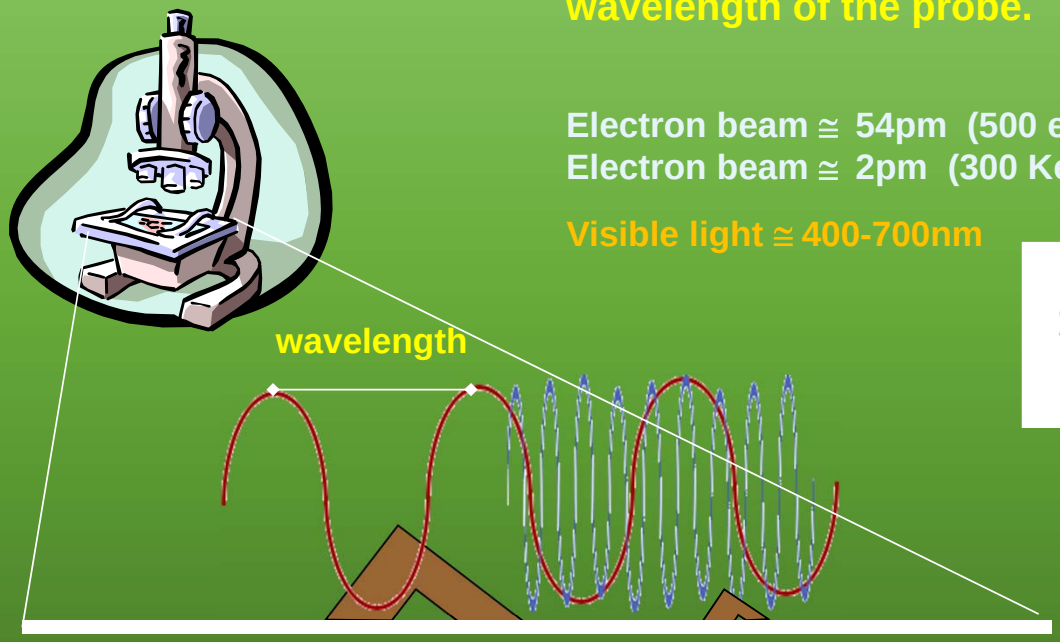
LIGHT VS SEM / TEM



Based on Abbe's theory you cannot resolve structure below about ½ the wavelength of the probe.

Electron beam ≅ 54pm (500 eV)
Electron beam ≅ 2pm (300 KeV) 78% of speed of light

Visible light ≅ 400-700nm



$$\lambda = \frac{h}{\left[2m_0 eV\left(1+\frac{eV}{2m_0c^2}\right)\right]^{1/2}}$$

$$\delta = \frac{0.61\lambda}{\mu \sin\beta}$$

Resolution

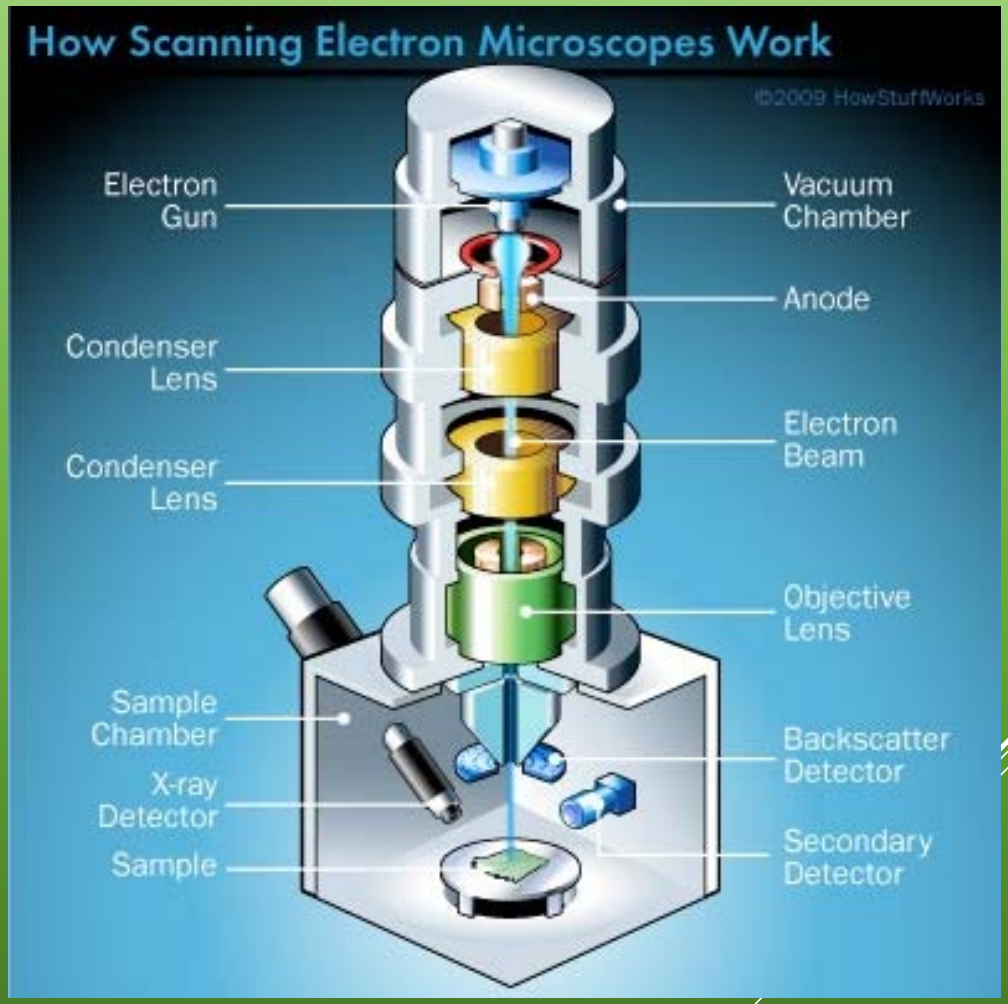
Notes: 1nm=1000pm, typical atomic spacing ≈ 0.1nm

OUTLINE

- **Beam optics and image formation.**
- Signals Generated in an SEM and their detection.
- Beam energy & current.
- EDS - compositional analysis.
- What else can we do with an SEM?
- How do we approach a new sample?

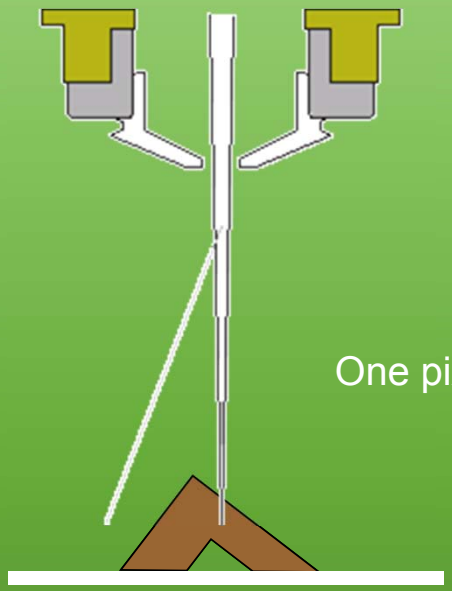
BASIC OPERATION MODE OF SEM

Nova 200 Nano-Lab



Schematic diagram illustrating the essential components of an SEM. Note that an array of useful signals can be collected and analyzed by use of different detectors.

IMAGE FORMATION IN SEM

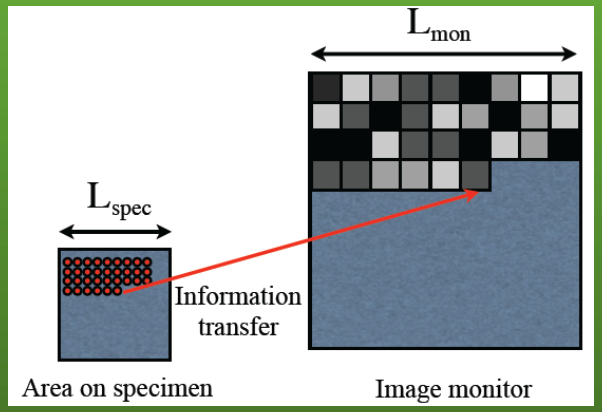


One pixel at a time!

Very small beam convergence angle

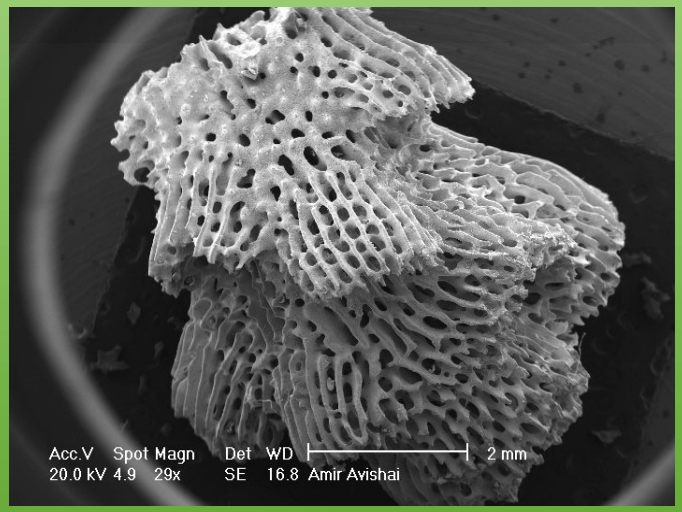
⇓

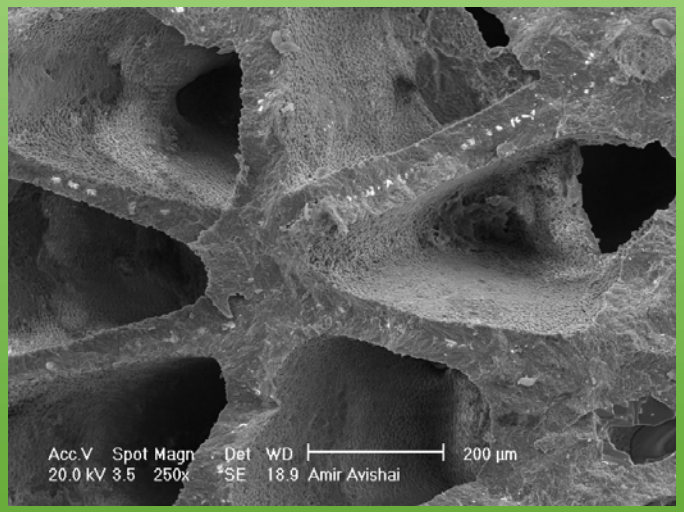
Large depth of field

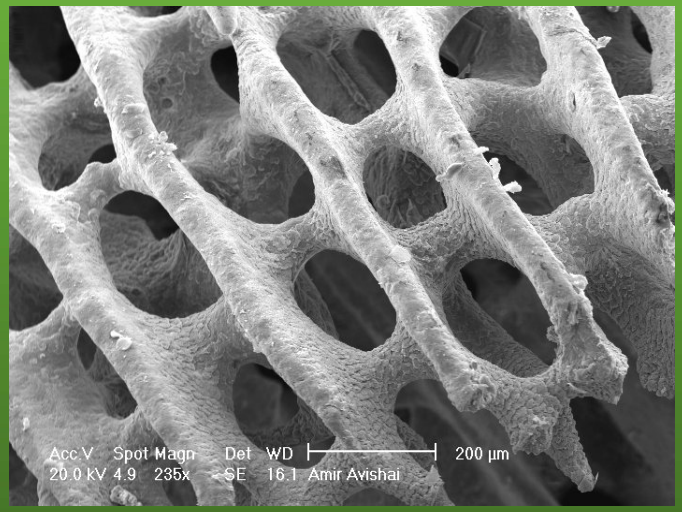


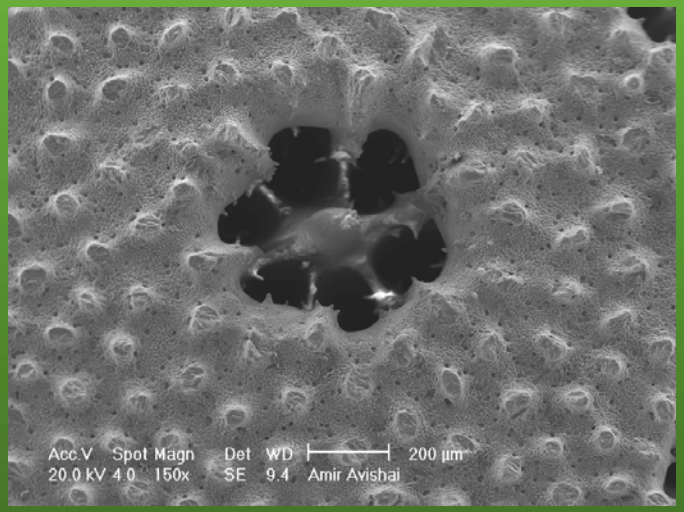
Ratio of the area viewed to the area being scanned is magnification

CORALS – VERY LARGE DEPTH OF FIELD









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β light microscope ≈ π/2 rad (1.57 rad)

β Electron microscope ≈ 10^{-3} rad

$$\delta = \frac{0.61\lambda}{\mu \sin \beta}$$

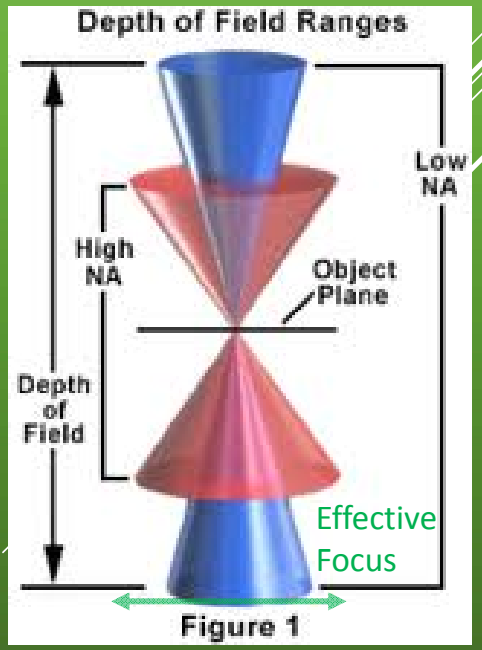

Figure 1

OUTLINE

- Beam optics and image formation.
- **Signals Generated in an SEM and their detection.**
- Beam energy & current.
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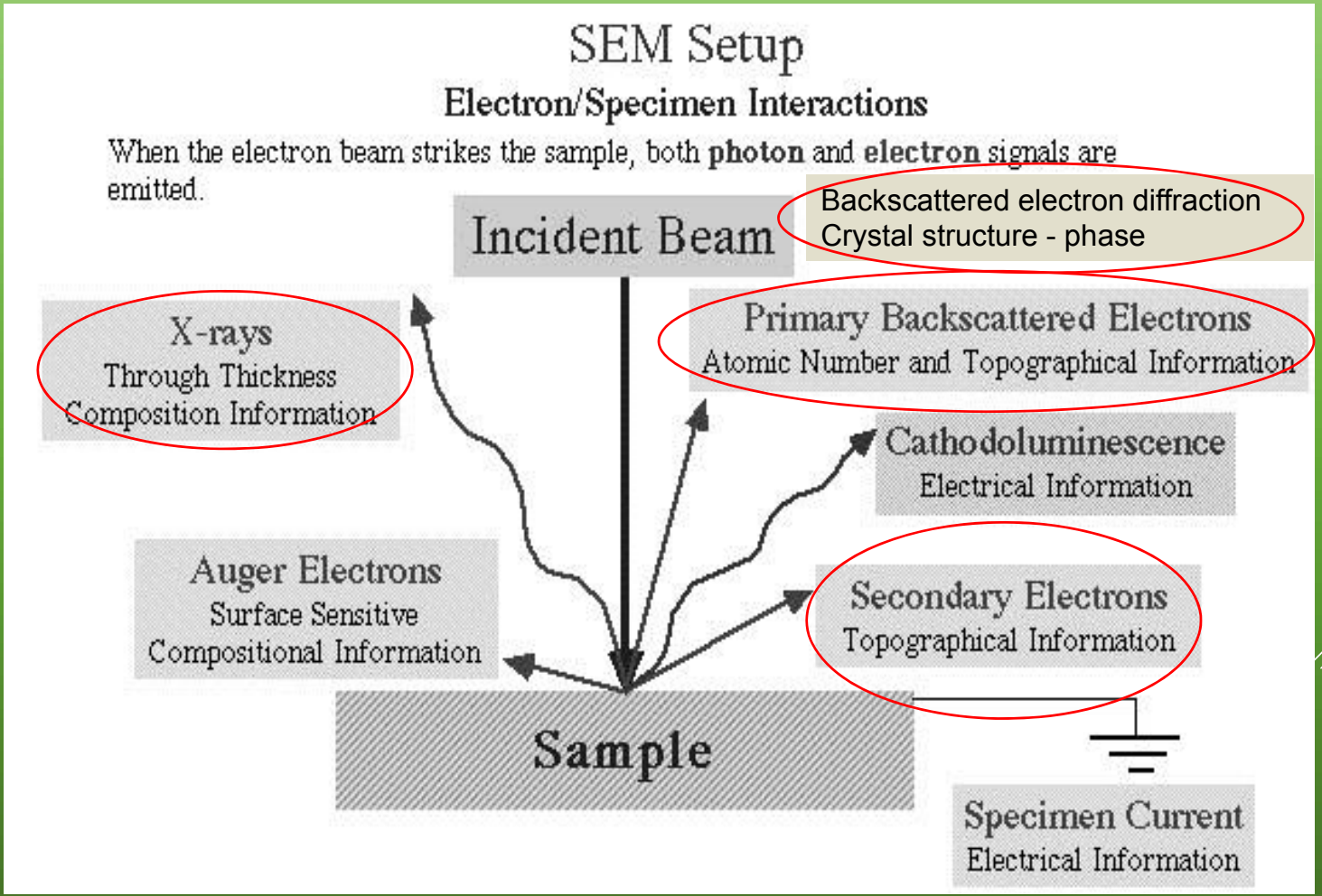
- How do we approach a new sample?

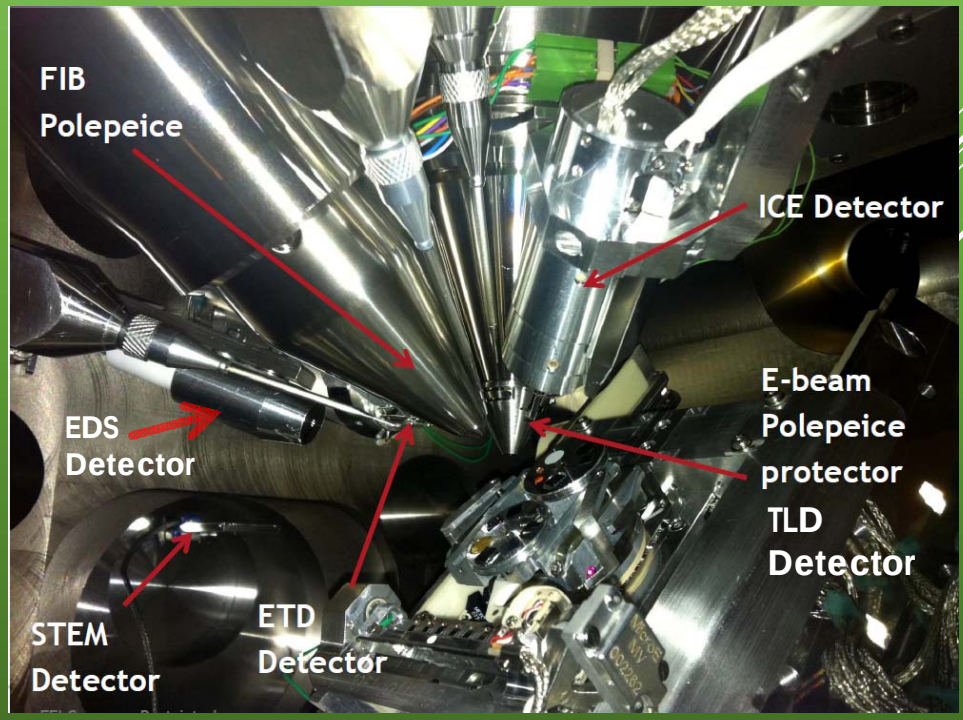
WHAT TYPE SIGNALS ARE CREATED IN A SEM?



DETECTORS AVAILABLE

- Everhart Thornley (**ETD**) Detector (SE, BSE)
- InLens(**TLD**) Detector – SE, BSE Detection
- **ICE** Detector (SE, BSE, ions)
- Retractable **STEM** Detector (BF, DF, HAADF)
- Retractable Solid state **BSE** Detector
- **GSED** SE Detection
- **EDS** Photon Detection and Energy Analysis
- **EBSD** Backscattered Electron Diffraction

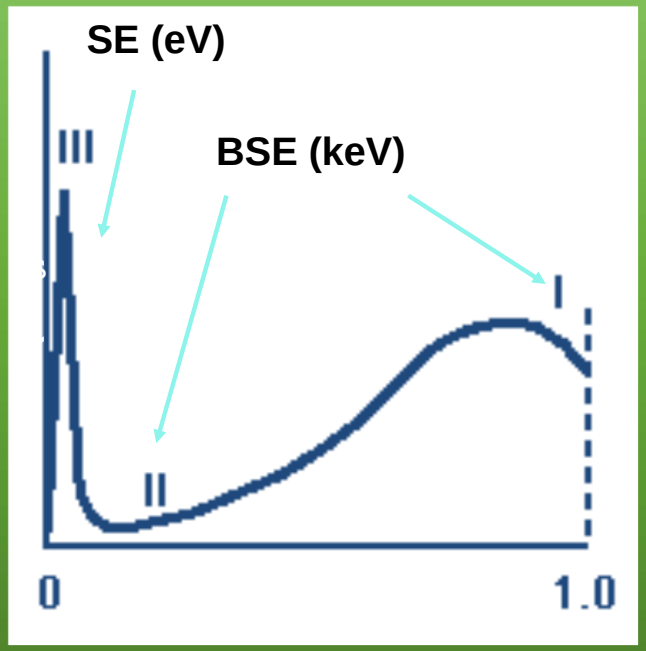
Beam Deceleration

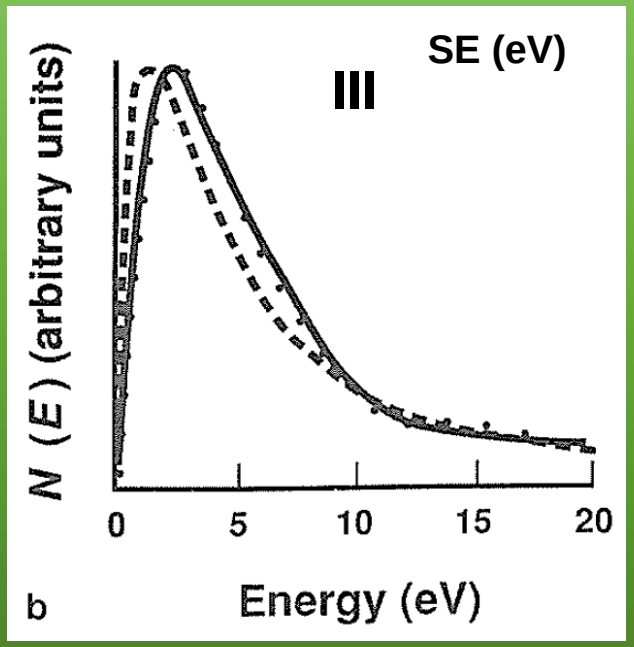


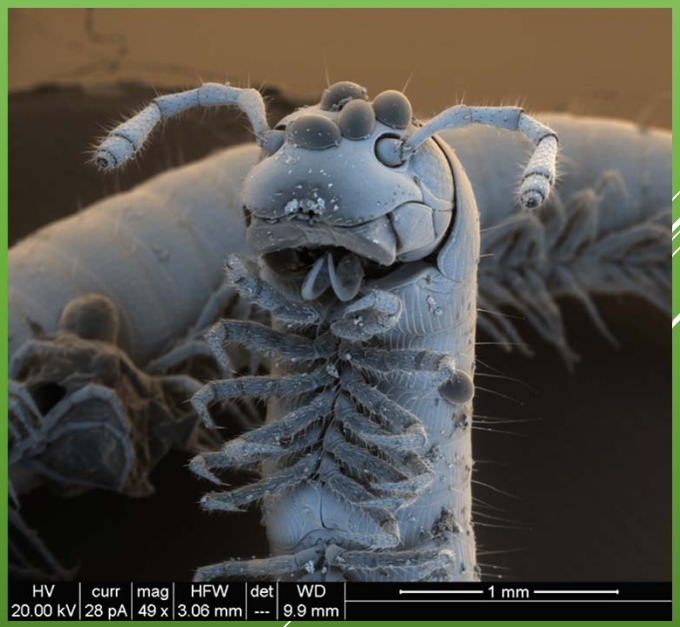
CHARACTERISTICS OF SECONDARY AND BSE ELECTRONS

Energy distribution of all electrons emitted from specimen under keV electron bombardment:

SE: Topographic
BSE: Compositional





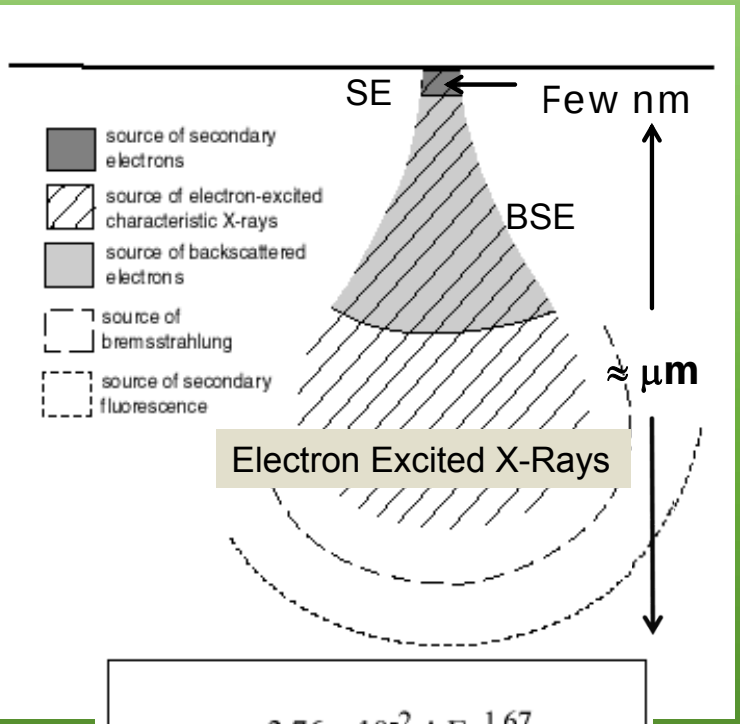


Millipede

SEs are VERY low energy electrons!

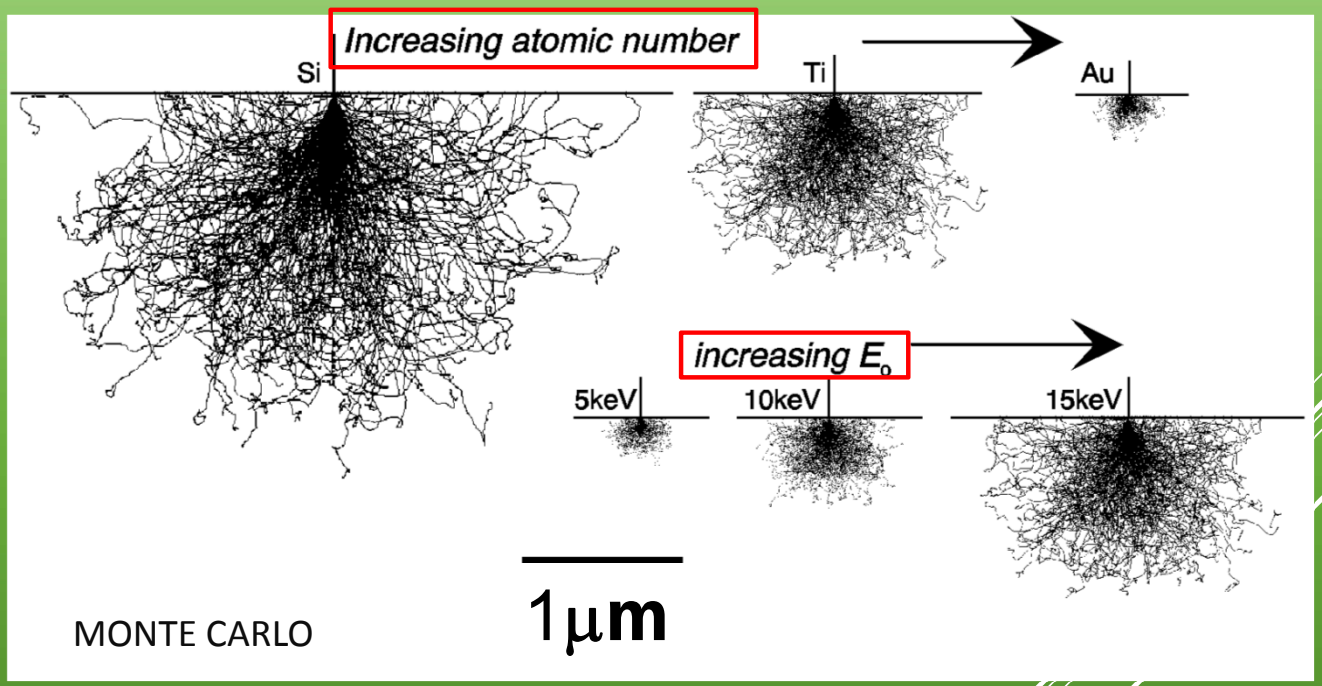
By definition, these secondary electrons are <50 eV, with most 3-5 eV.

ELECTRON BEAM PENETRATION



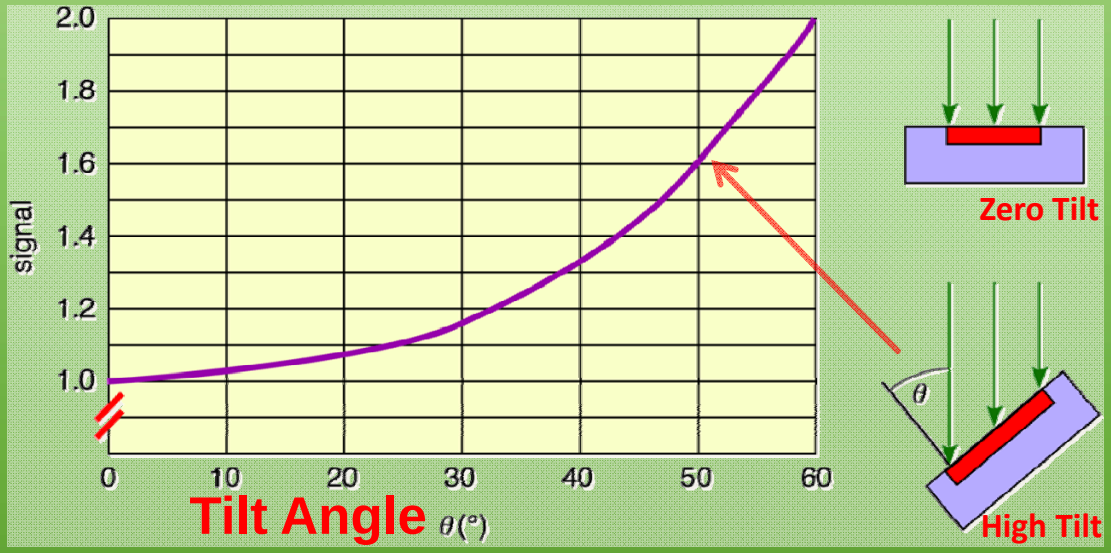
$$r\ (\mu m) = \frac{2.76 \times 10^{-2}\, A\, E_o^{1.67}}{\rho\, Z^{0.89}}$$

where ρ = density of the material (g/cm^3),
Z = atomic number,
A = atomic mass,
and E_o = accelerating voltage.

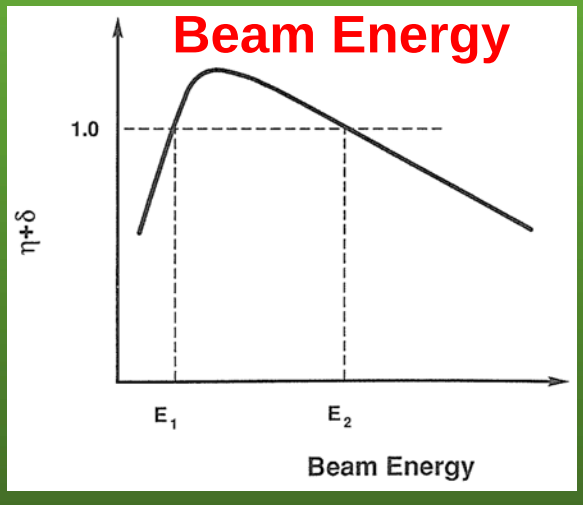


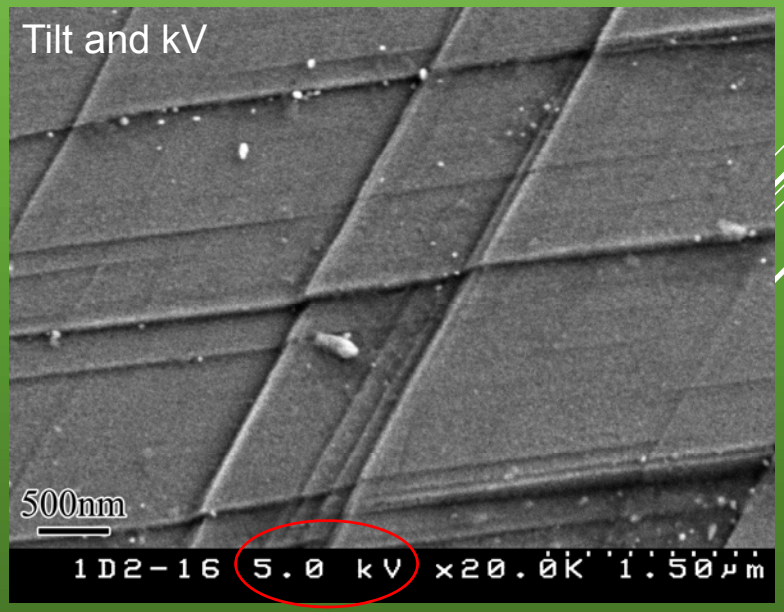
- Beam penetration decreases with Z
- Beam penetration increases with energy
- Electron range ~ inelastic processes
- Electron scattering (aspect) ~ elastic processes

SURFACE IMAGING – TOPOGRAPHY, CRYSTAL SYMMETRY



SE properties





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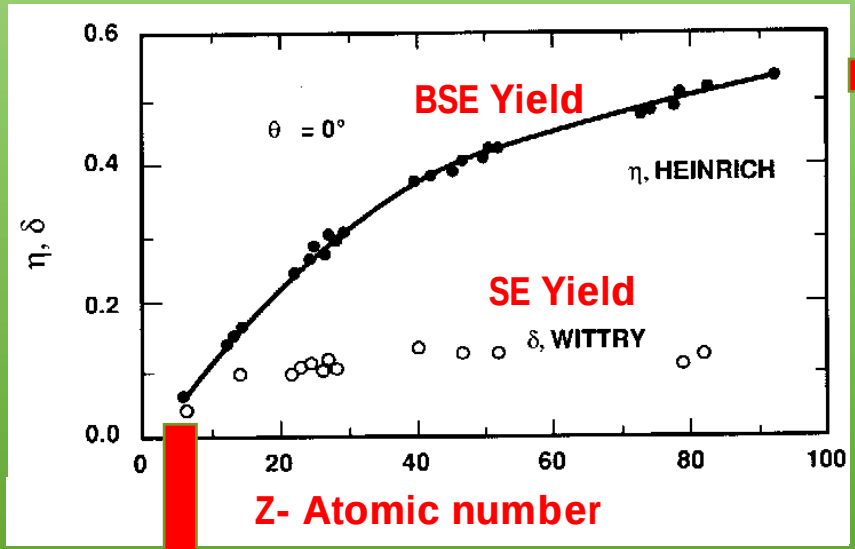
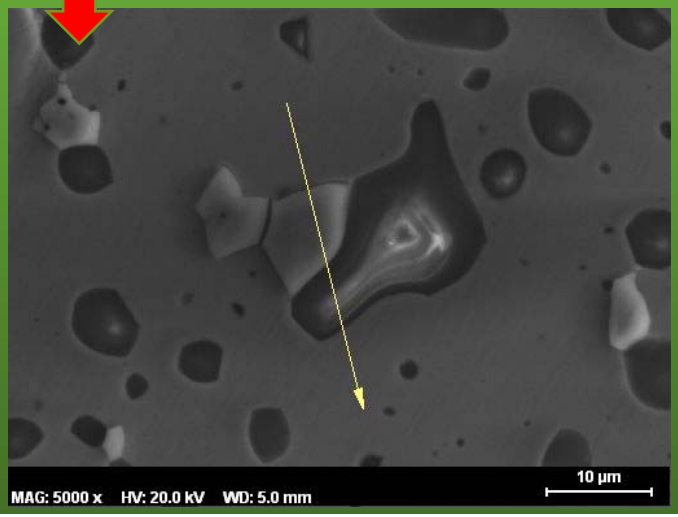
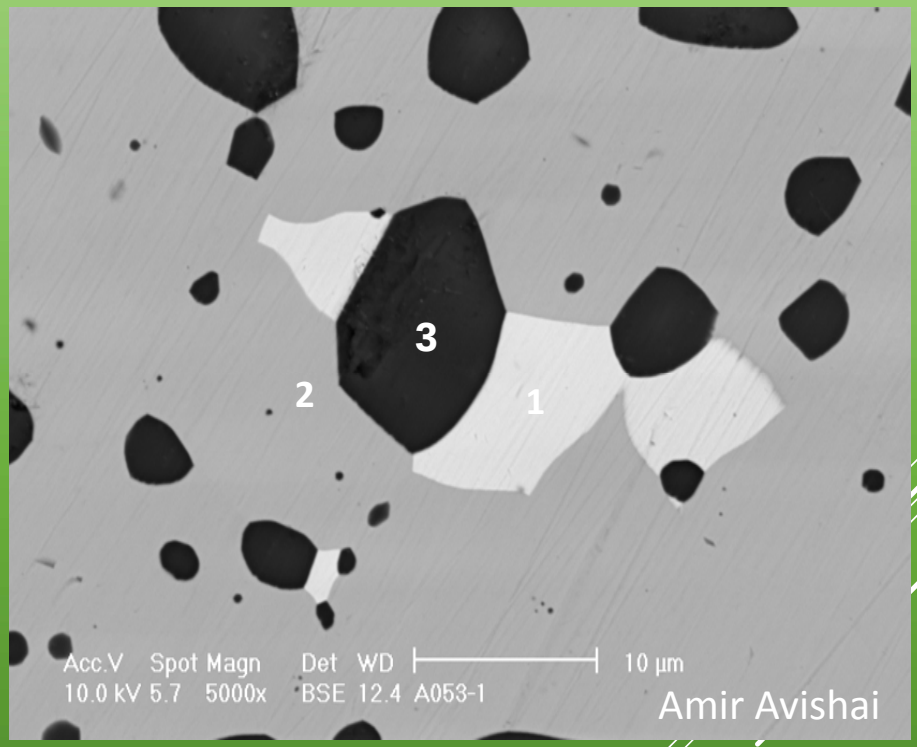
BACKSCATTER ELECTRON PRODUCTION



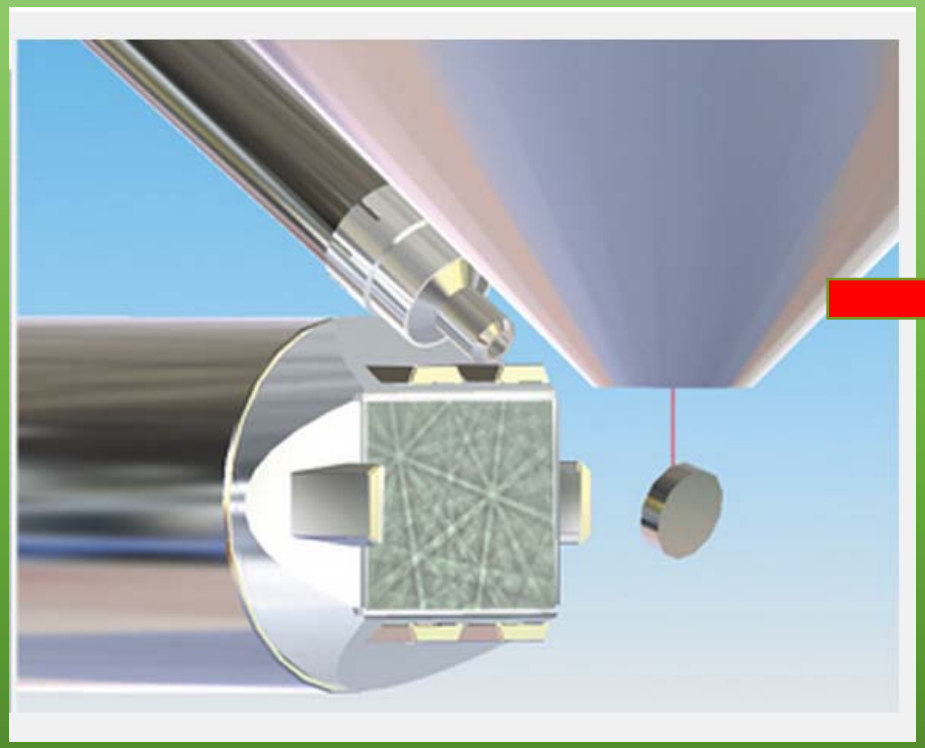


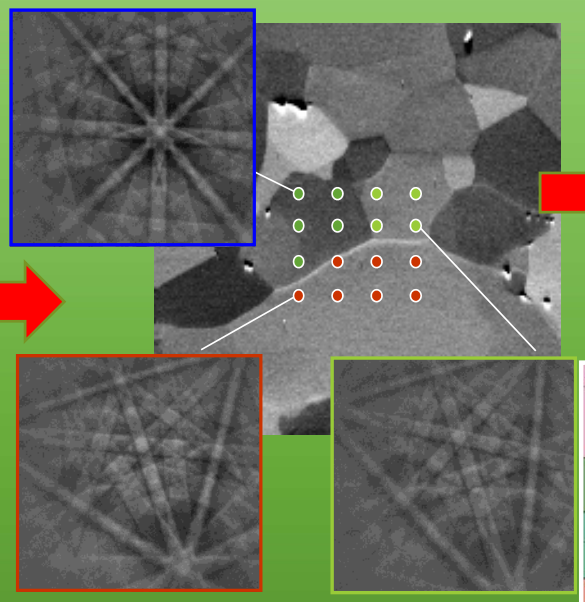
Mo, Si, O

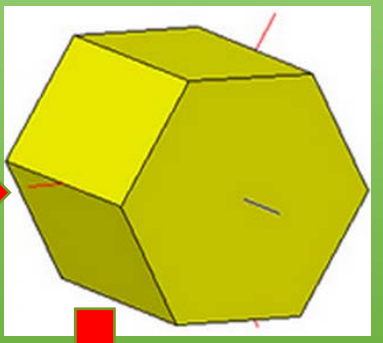


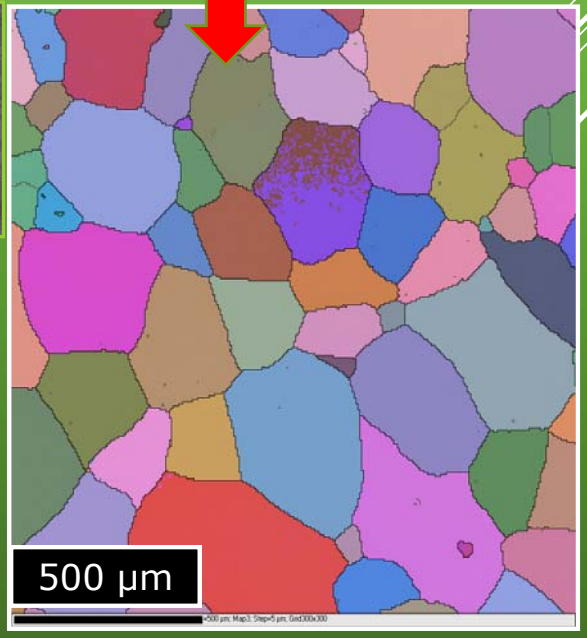
	Si [at%]	Mo [at%]	O [at%]	Other
1	25	**46.5**	28.5	
2	**54**	**31**	15	
3	28.5	0.5	**66**	5 (Al,Mg Ca)

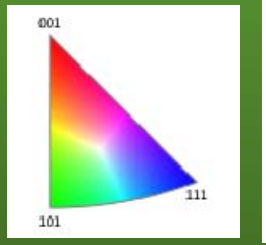
Electron Back-Scattered Diffraction Patterns (EBSD)
Orientation Imaging Mapping (OIM)







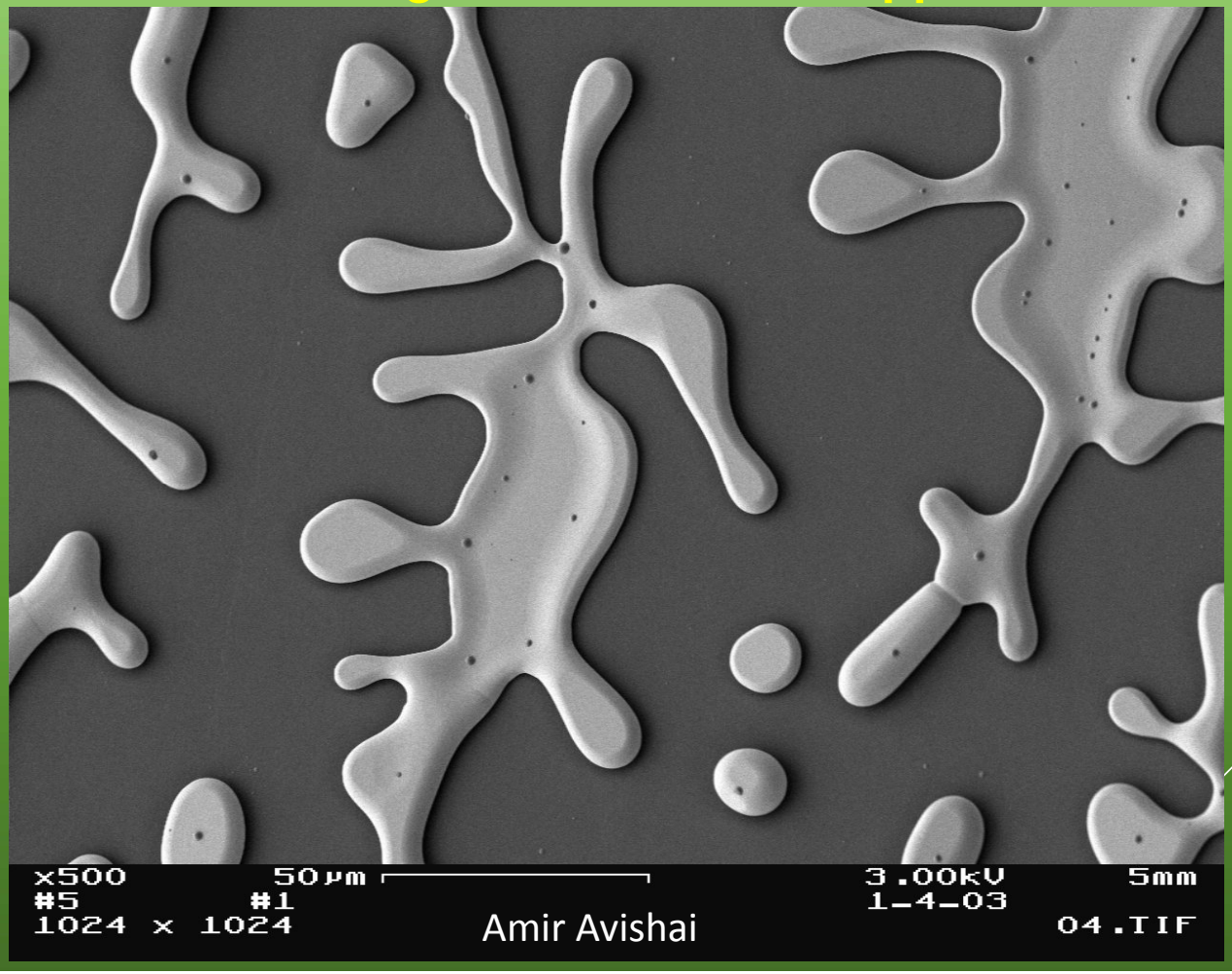




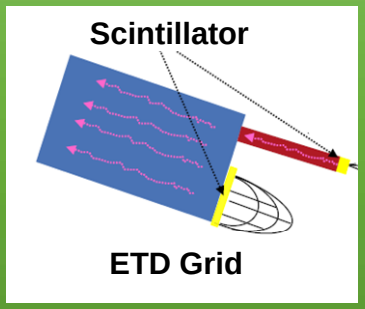
DETECTOR POSITION & CONTRAST

SE Image

Deweting of Ni Film over Sapphire



Where is the detector?



OUTLINE

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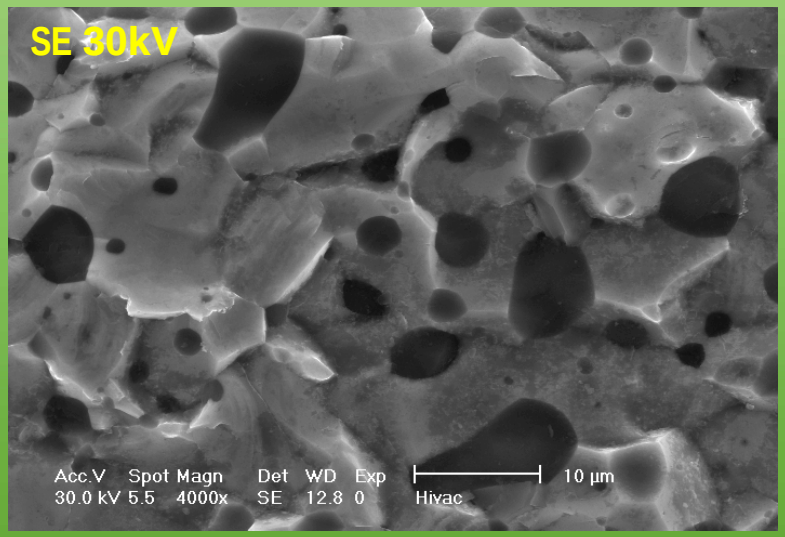
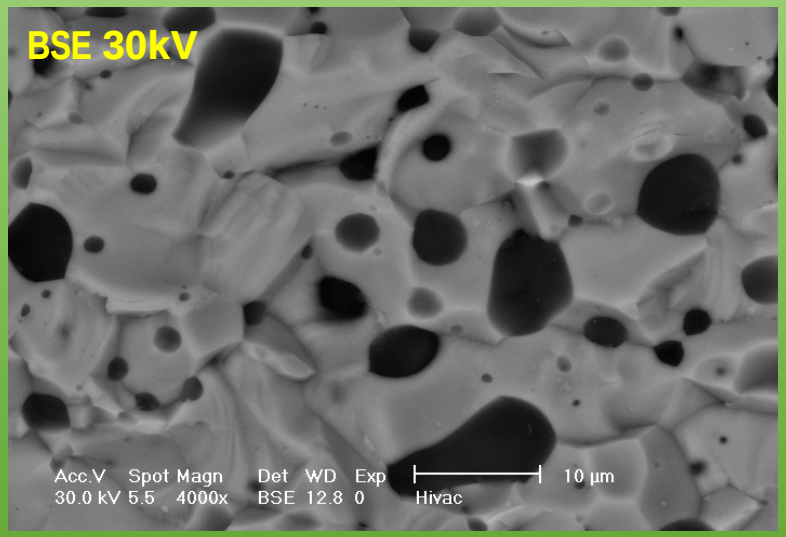
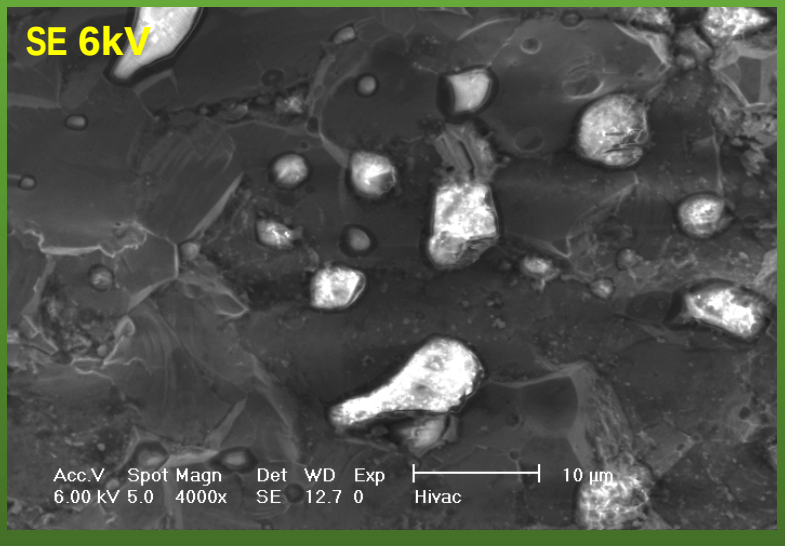
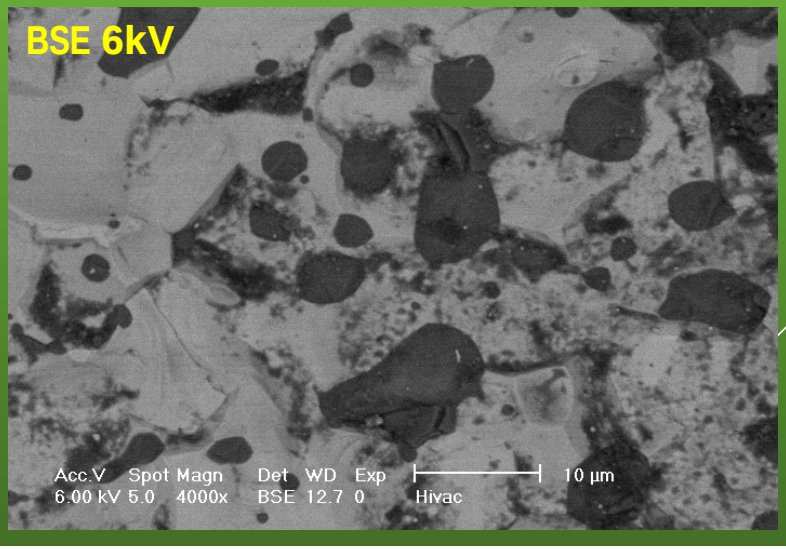
BSE VS SE AND VOLTAGE









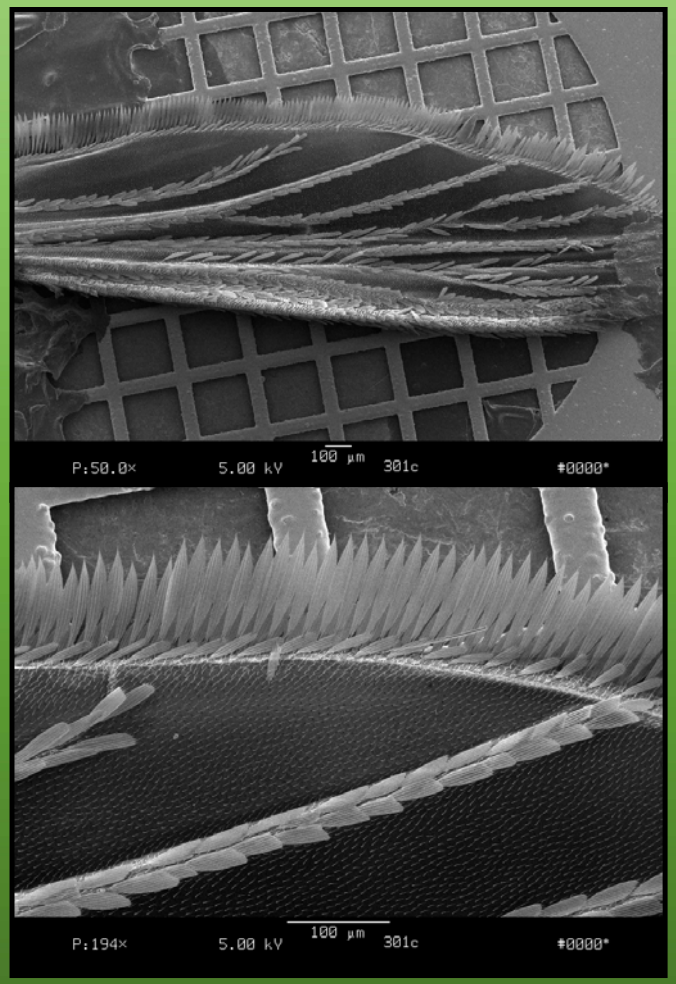
Effects seen here are a result of variation in two parameters only!

Amir Avishai

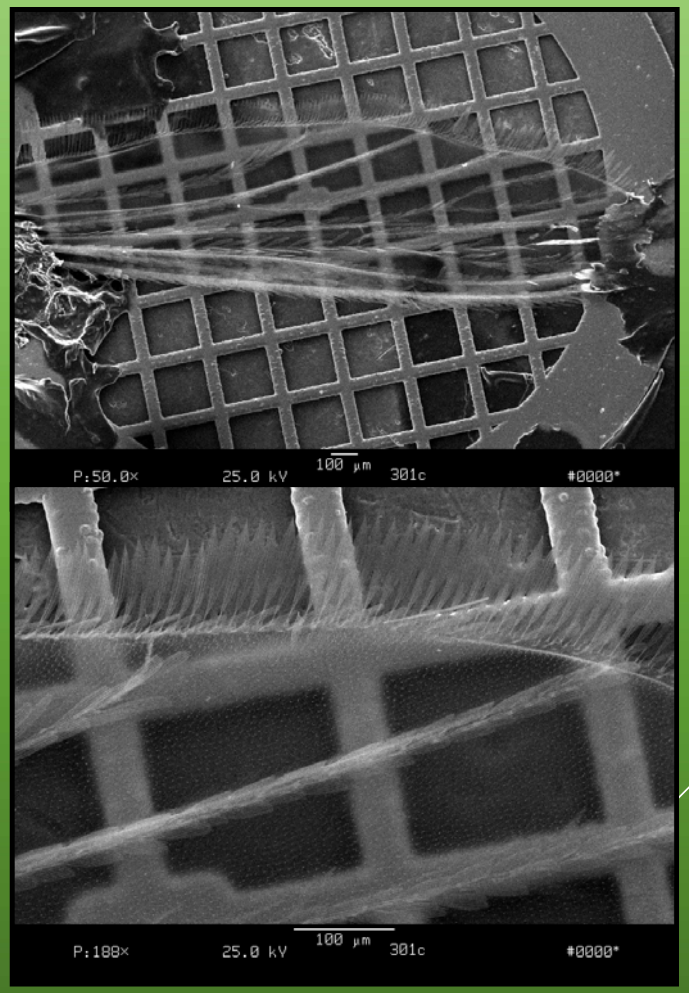
BEAM ENERGY AND PENETRATION

x50

x200

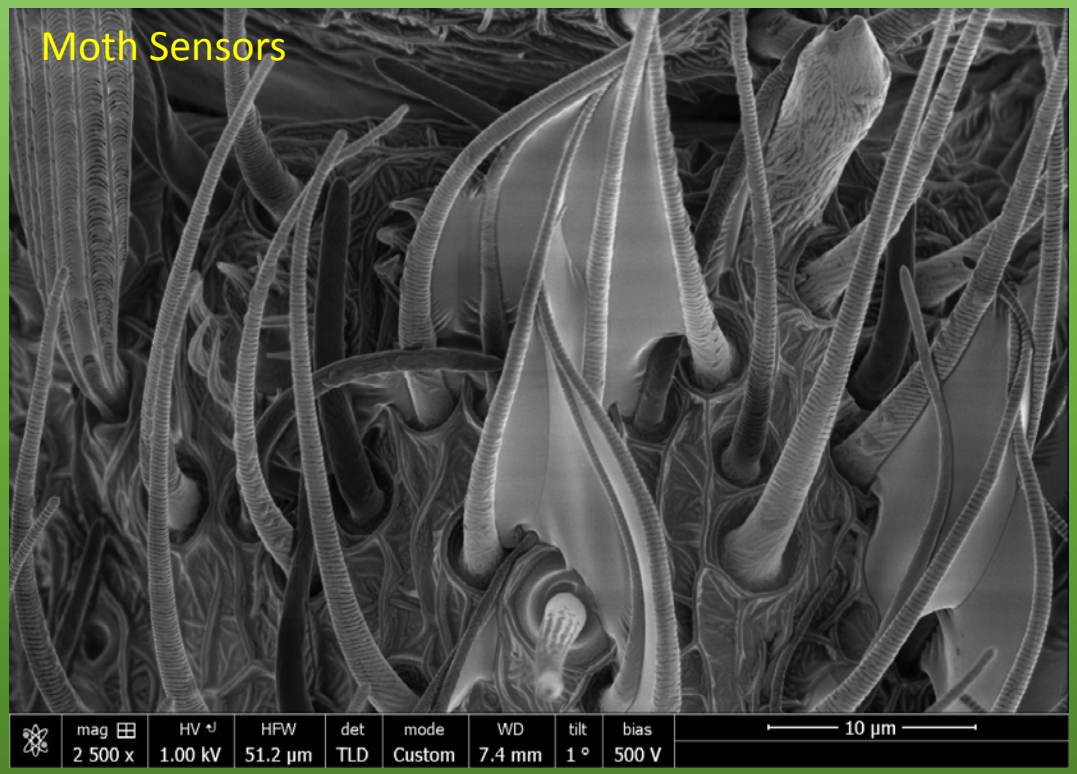


5 kV



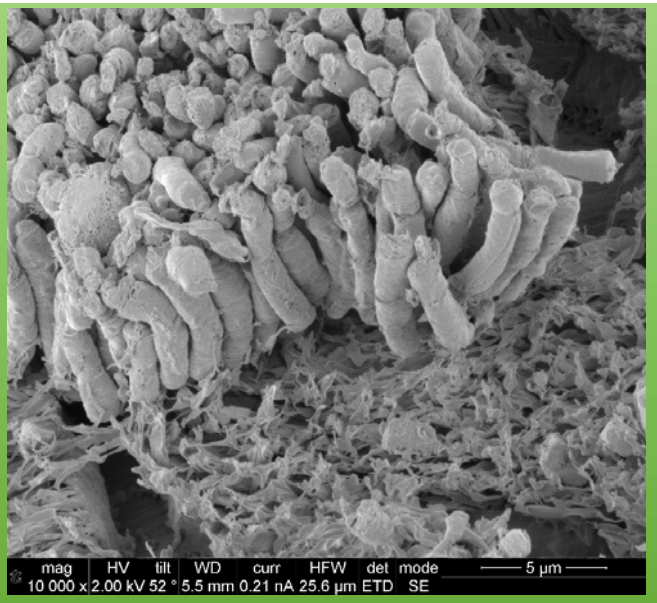
25 kV

BIOLOGICAL TISSUE IMAGING



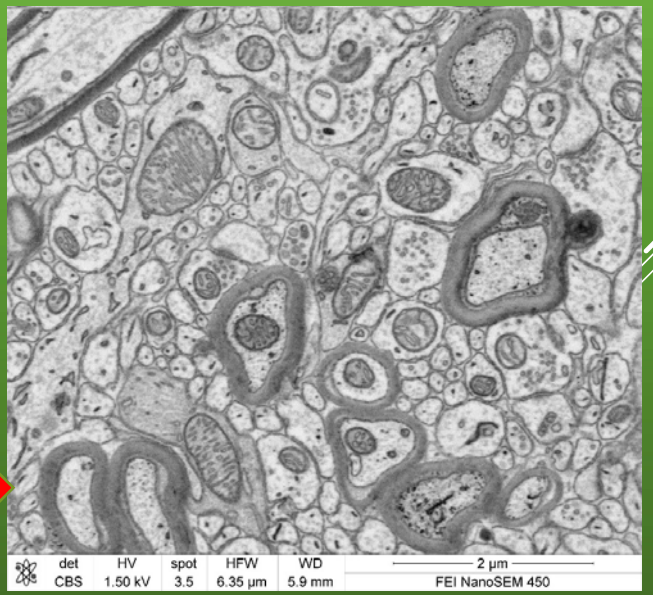
Mark Willis, CWRU, Biology

To obtain BSE contrast samples are stained with heavy metals – Osmium, Uranium, lead and Fe.



Critical point dried Rods in a Wild Mouse Eye

Debarshi Mustafi
CWRU, SOM



Brain Tissue

Grahame Kidd
CCF

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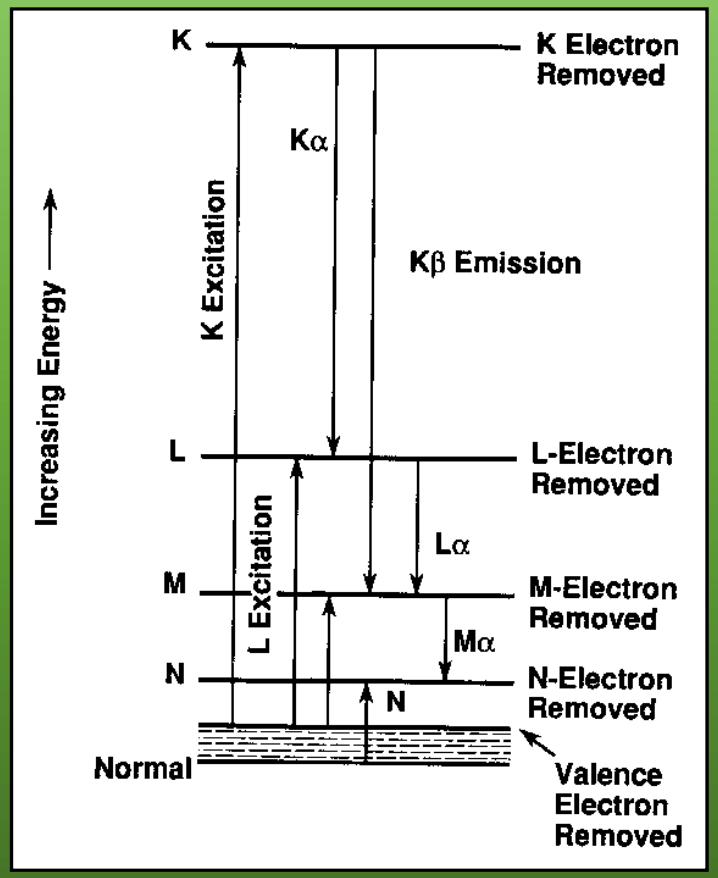
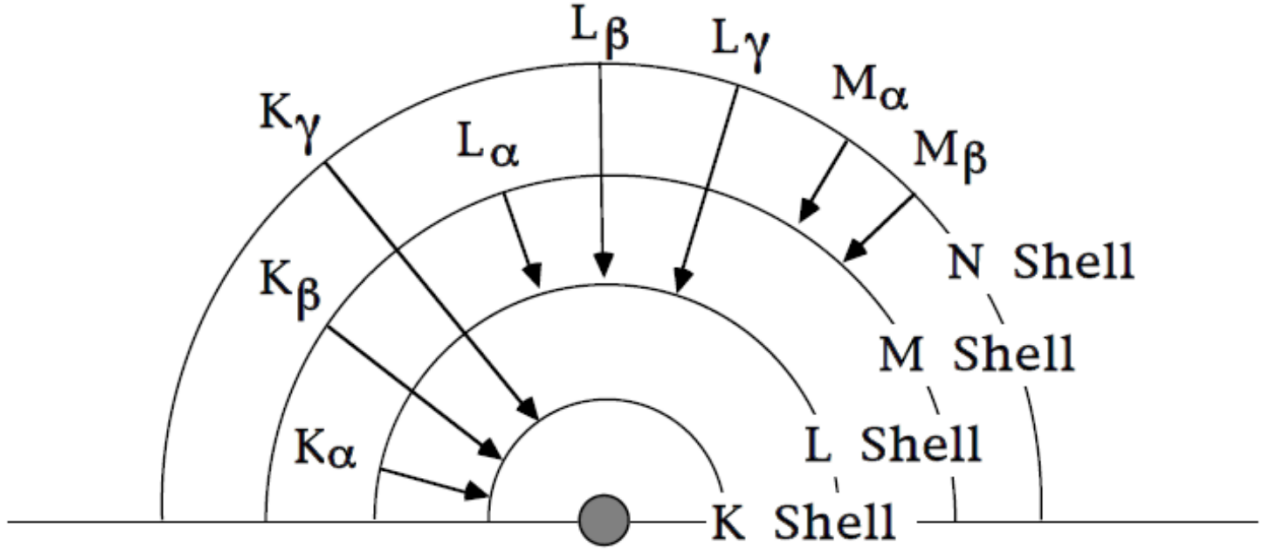
COMPOSITIONAL INFORMATION – ENERGY DISPERSIVE SPECTROSCOPY (EDS)



X-ray Lines - K, L, M

Nomenclature for Principal X-Ray Emission Lines

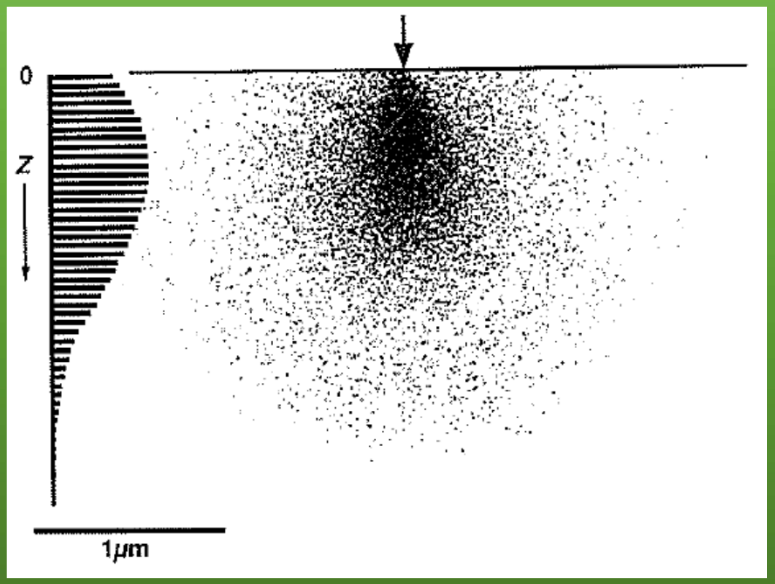


X-RAY GENERATION VOLUME

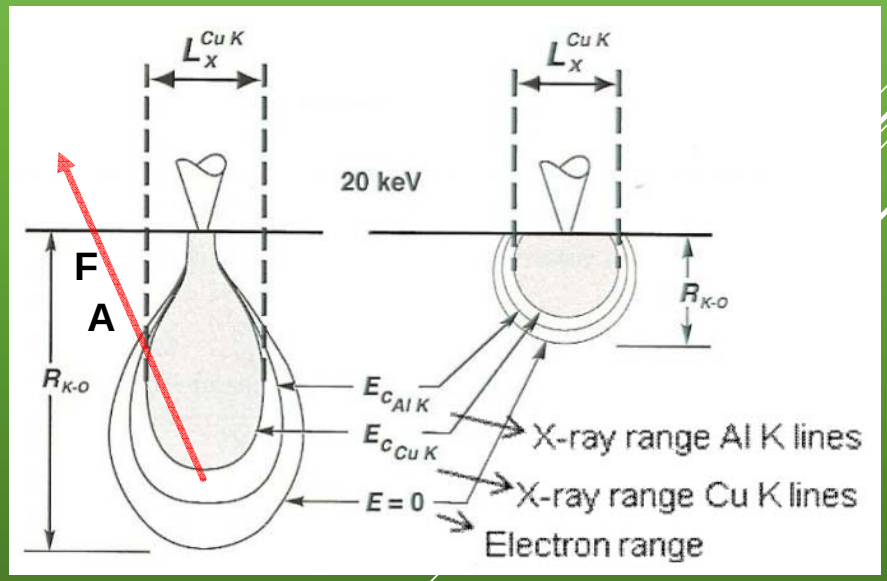
- Atomic number correction (Z)
- Absorption correction (A)
- Characteristic fluorescence correction (F)

$$R_x = \frac{0.064}{\rho}\left(E_0^{1.68} - E_C^{1.68}\right)$$

R_x – [μm]
E_0 - [KeV]
E_C - [KeV]
ρ – g/cm^3



depth-distribution function, φ(ρz),



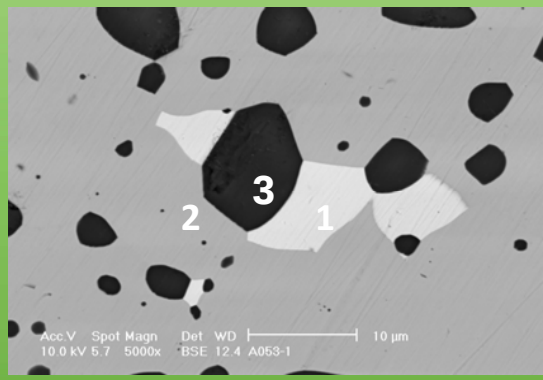
3g/cm3
Cu in Al

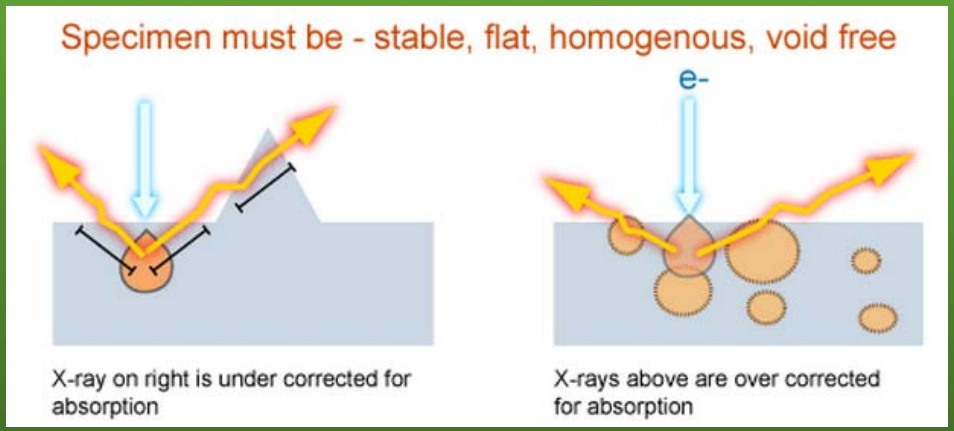
20kV

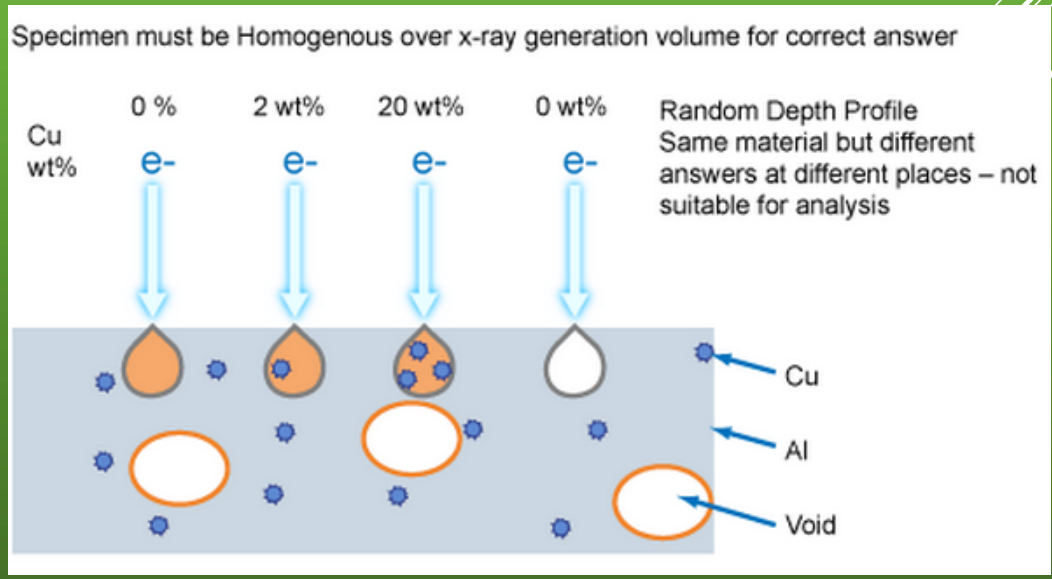
8g/cm3
Al in Cu

REQUIRED CONDITIONS FOR EDS ANALYSIS

- Polished sample (flat).
- Measure on a uniform region.
- No etching, use BSE to identify phases.
- Use a beam energy 2-3 times the highest peak analyzed.
- For charging samples avoid metallic coatings if possible, use carbon.
- Repeat measurement in a few locations.

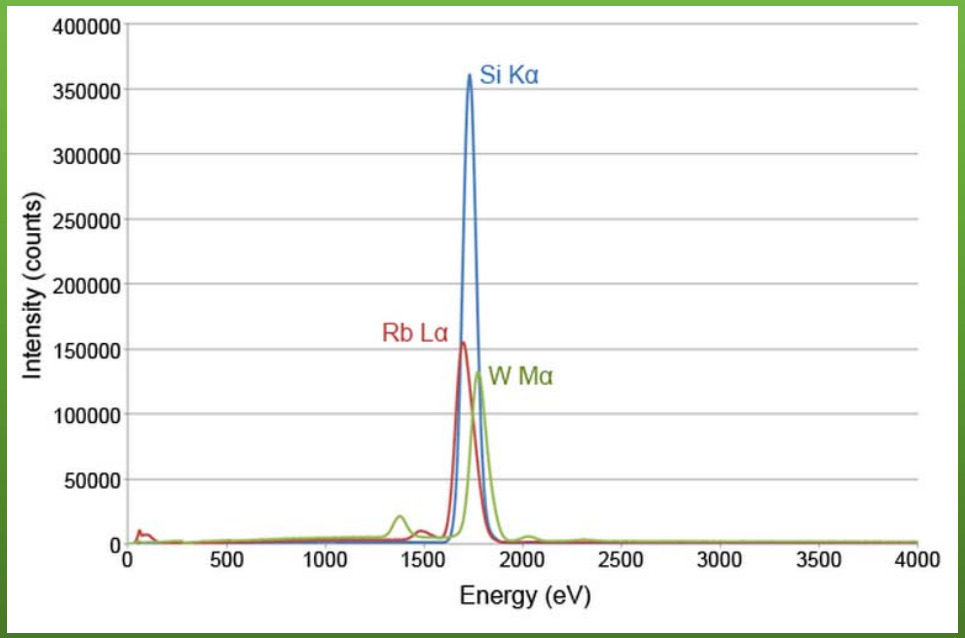


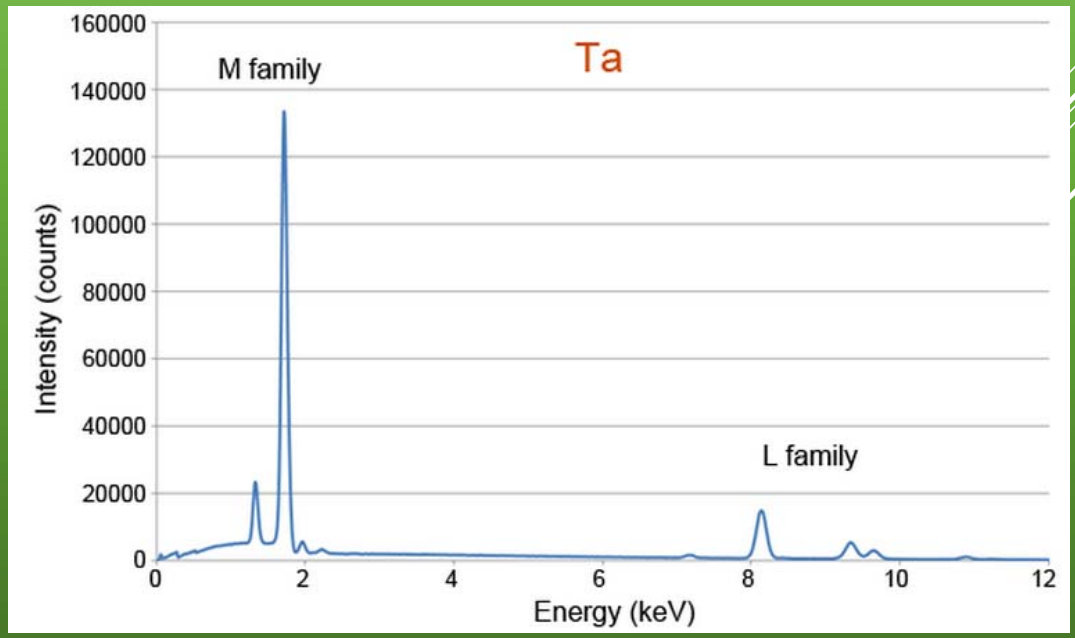




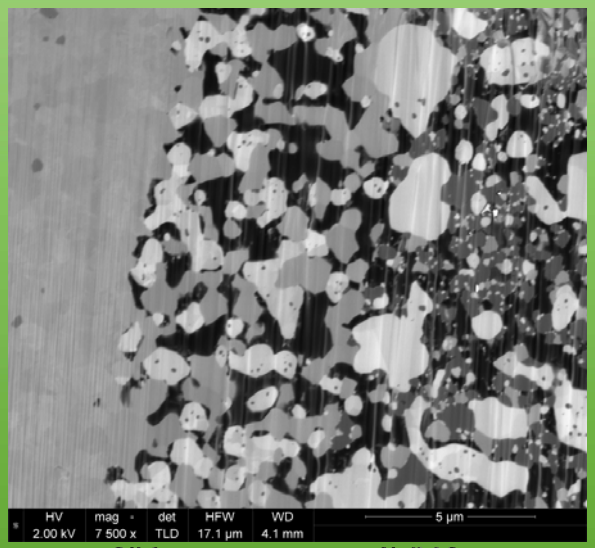
IDENTIFYING ELEMENTS & OVERLAPPING PEAKS

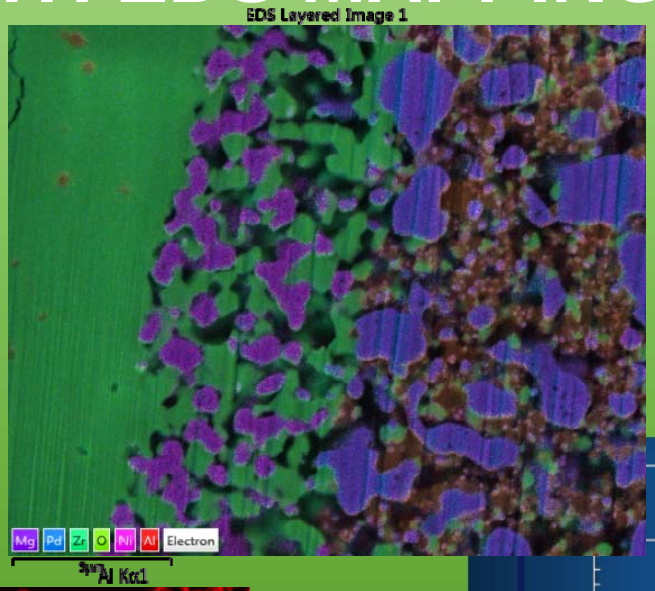
- Never trust auto ID, confirm every peak.
- In case of severe overlaps use higher energies to confirm elements.
- Use longer processing time to better resolve peaks or long collection times (better statistics).





WORKING WITH EDS MAPPING





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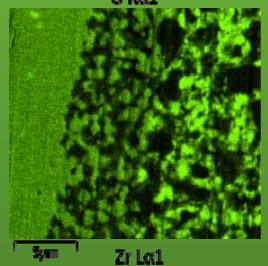
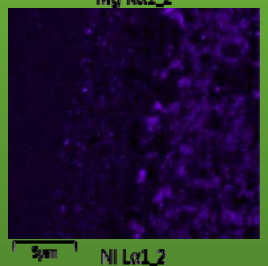
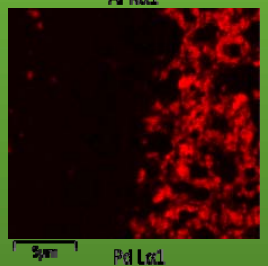
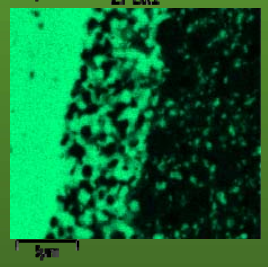
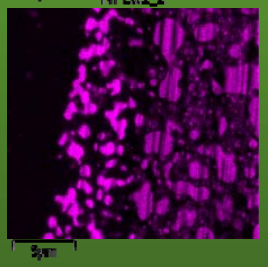
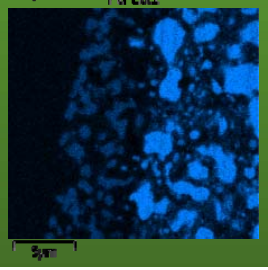


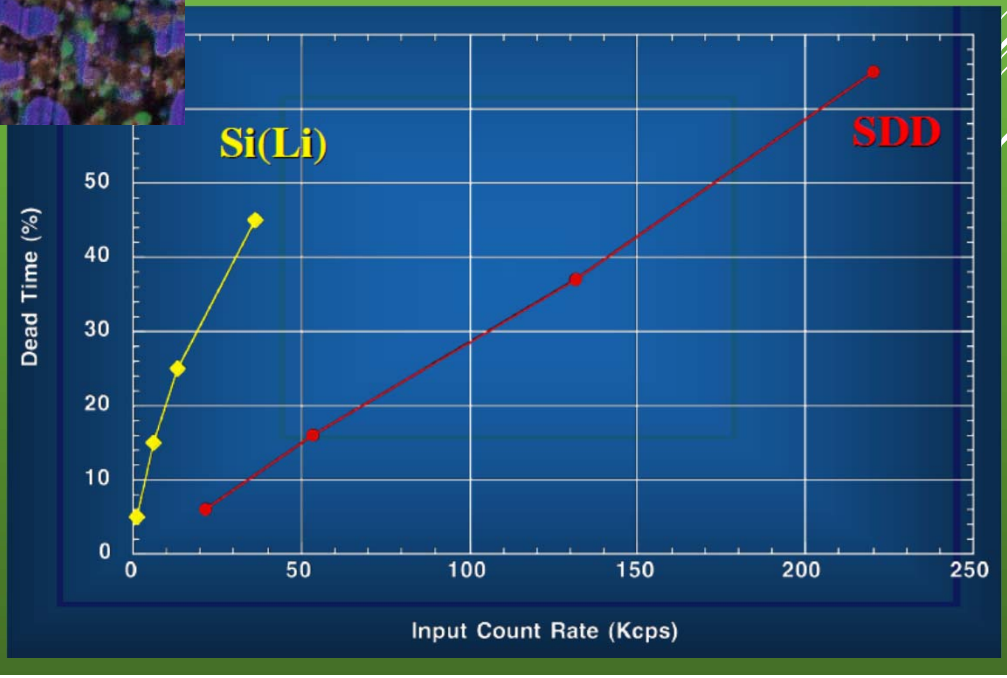




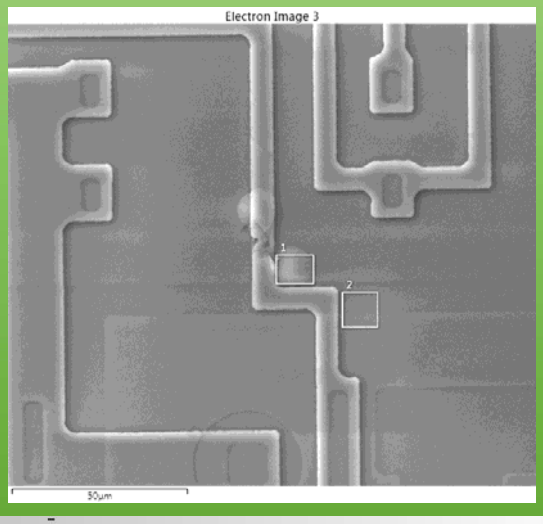


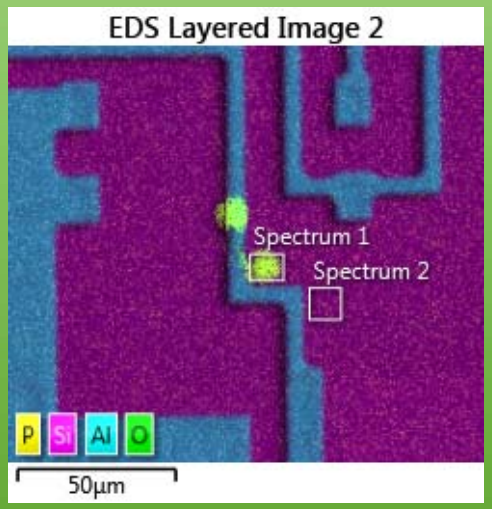


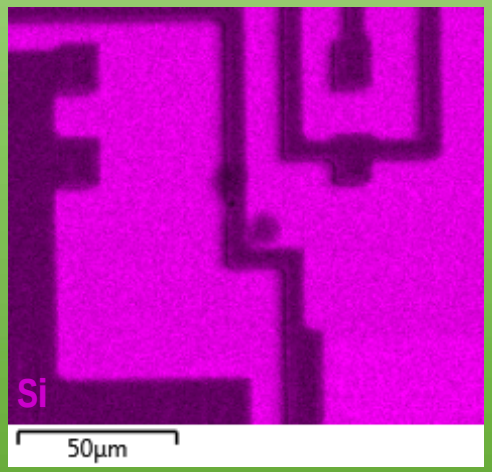


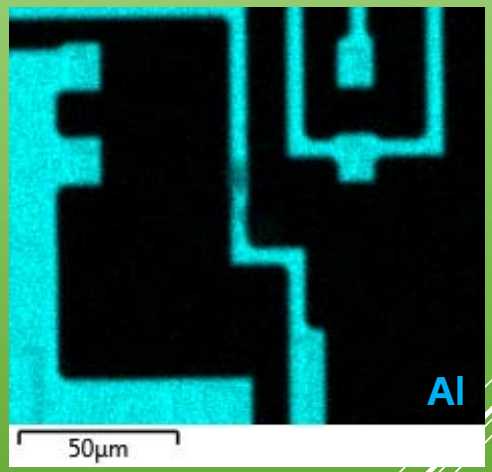


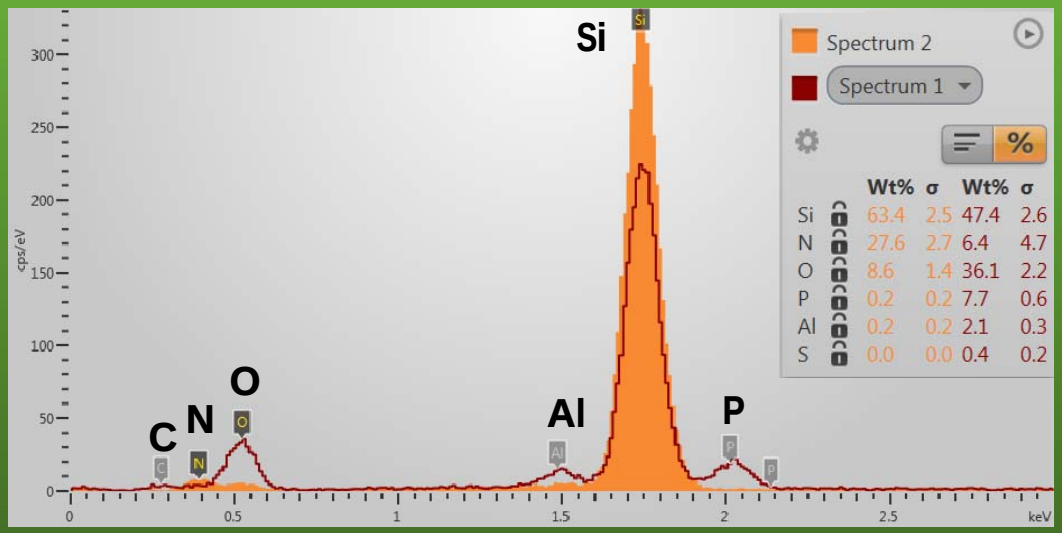
Failure Analysis - Device

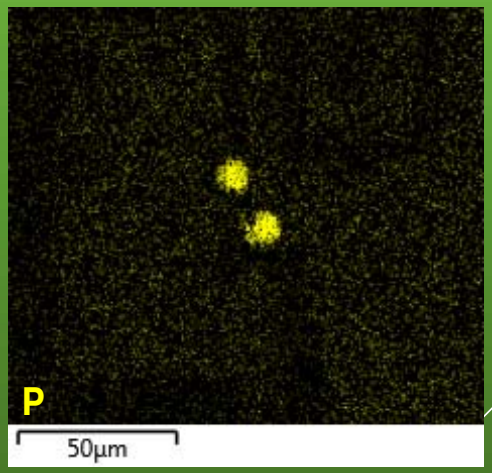


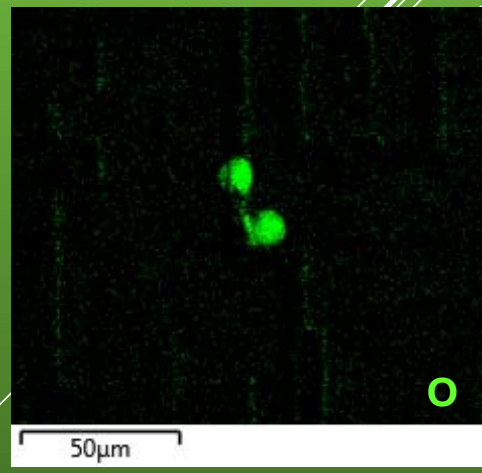






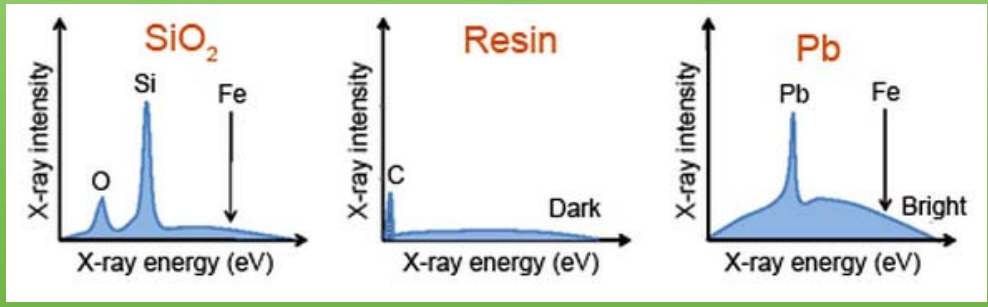


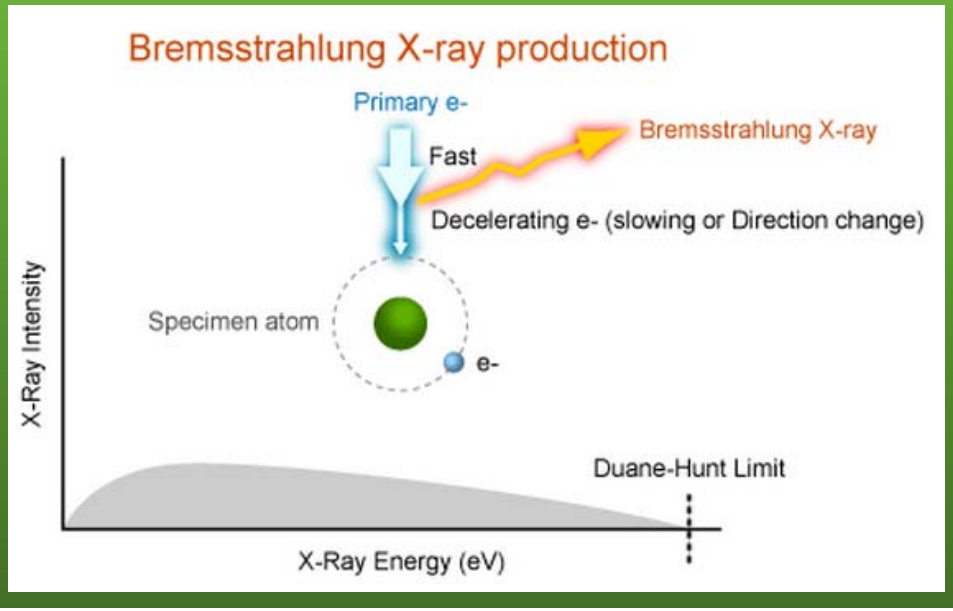


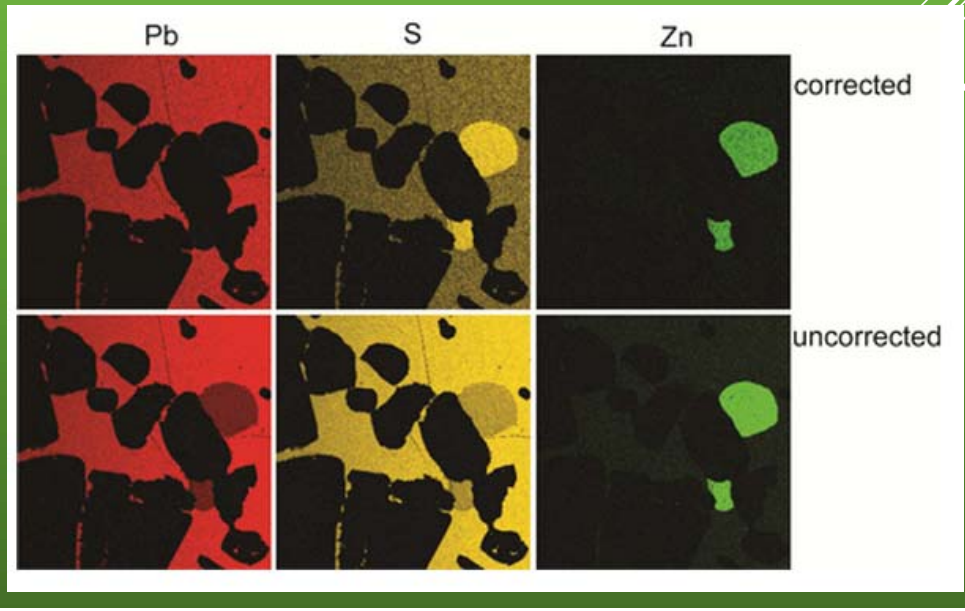


COMMON ARTIFACTS & ERRORS DURING ANALYSIS

- Sum/pileup Peaks
- Si X-Ray Escape peaks
- Errors due to charging (Duane-Hunt limit).
- Background removal in elemental maps.
- Working distance
- Magnification.







OUTLINE

- Beam optics and image formation.
- Signals Generated in an SEM and their detection.
- Beam energy & current.
- EDS - compositional analysis.
- **What else can we do with an SEM ?**
- How do we approach a new sample?

VPSEM CAPABILITIES

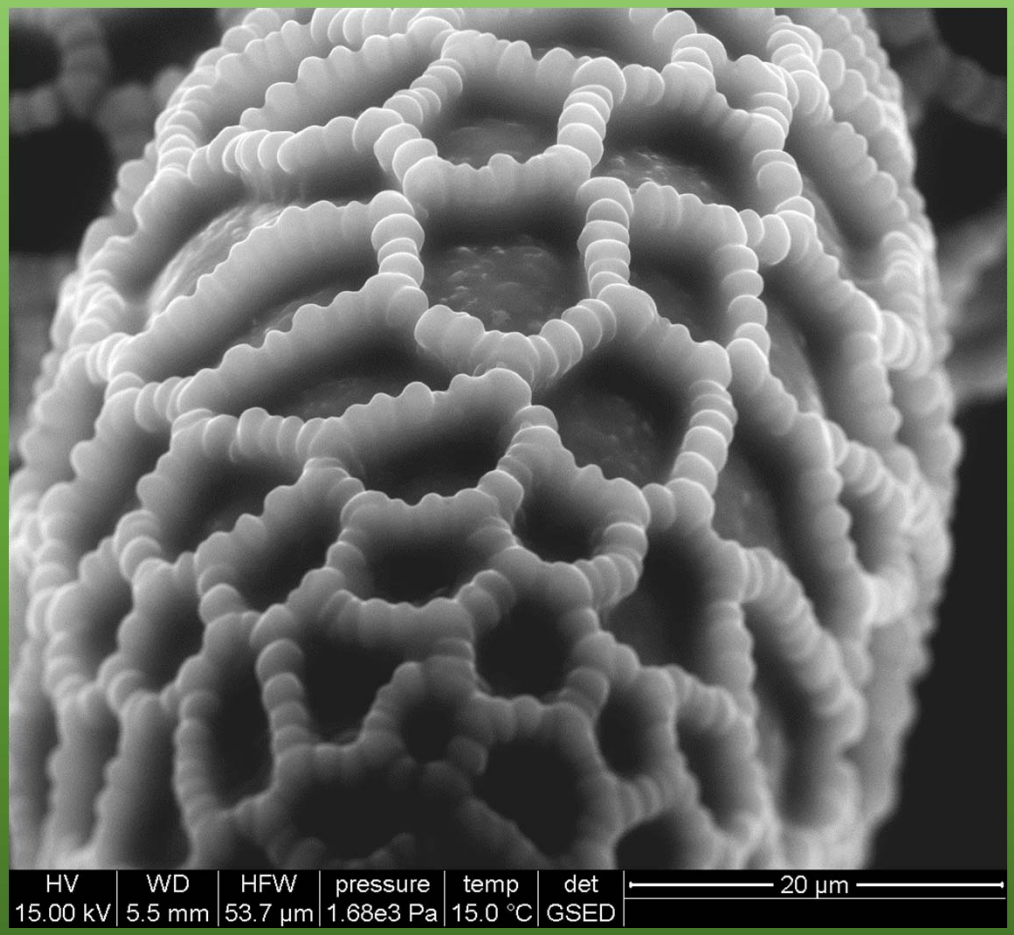
- Conventional High Vacuum
  - Coated/conductive specimens
  - Critical point dried specimens
- Low Vacuum or Wet Mode
  - Charge reduction for non-conductors
  - Surface imaging in a gas (hydration/dehydration, oxidation studies)
  - Vacuum sensitive materials (biological samples)
  - Wet or “dirty” specimens (ESEM)

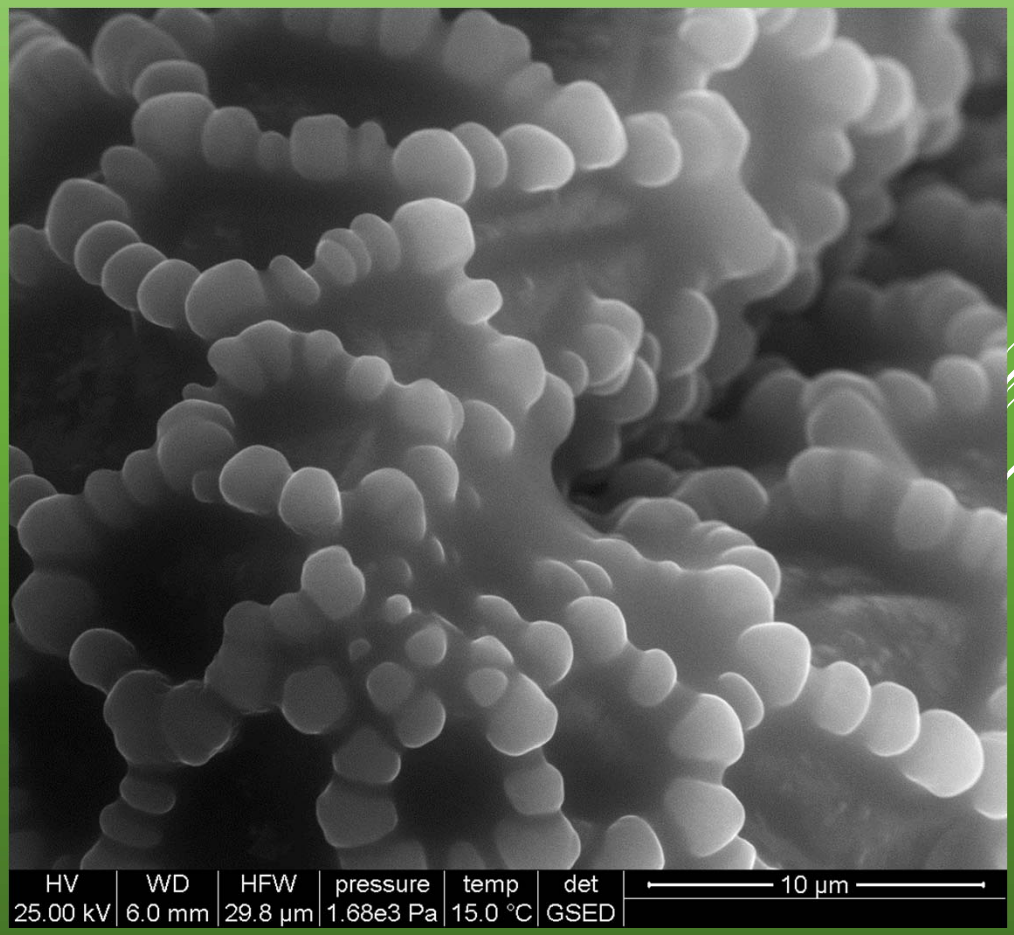
I. **Working Distance**

II. **Gas Pressure**

III. **Accelerating Voltage**

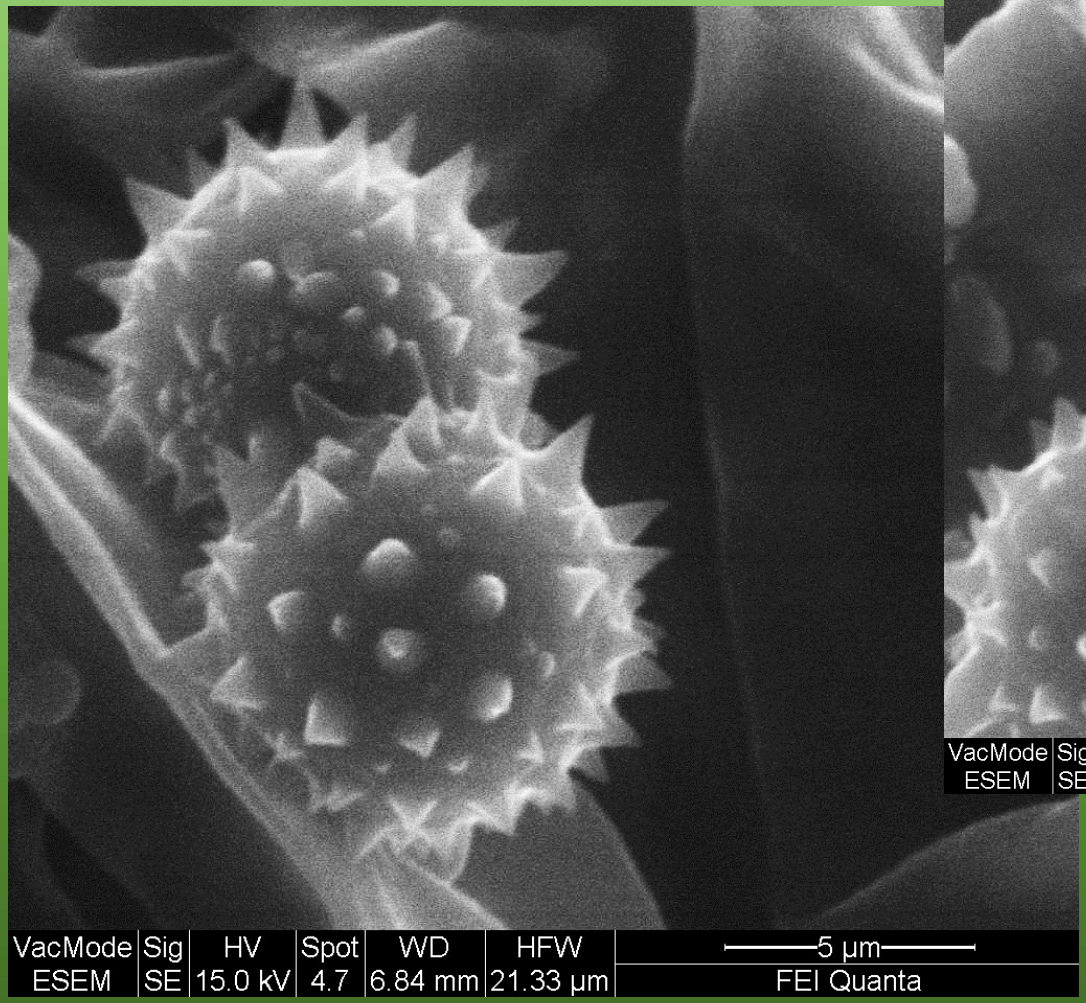
HIGH TEMPERATURE HYDRATION- LILY POLLEN

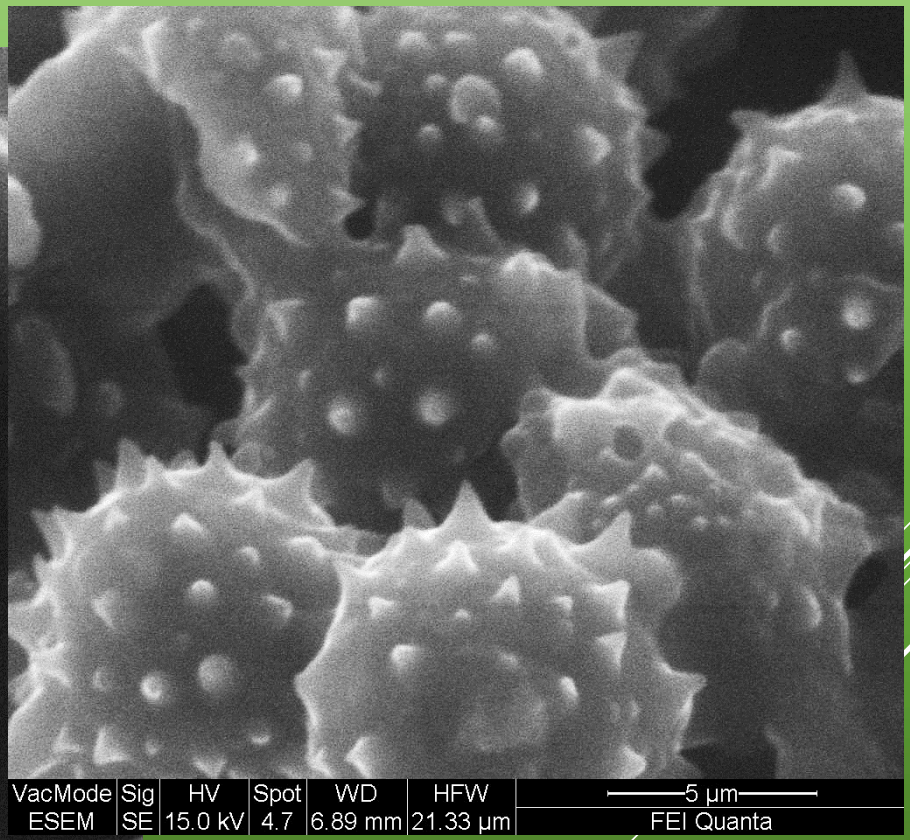




Images compliments of FEI

FRESH LACCARIA (TREE FUNGUS) IN AN ESEM

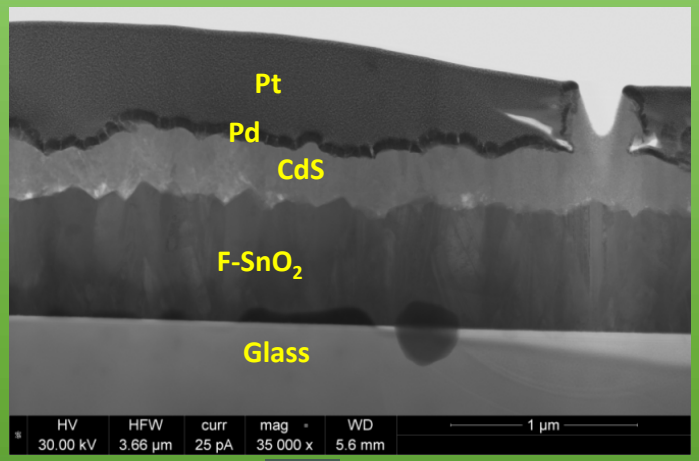


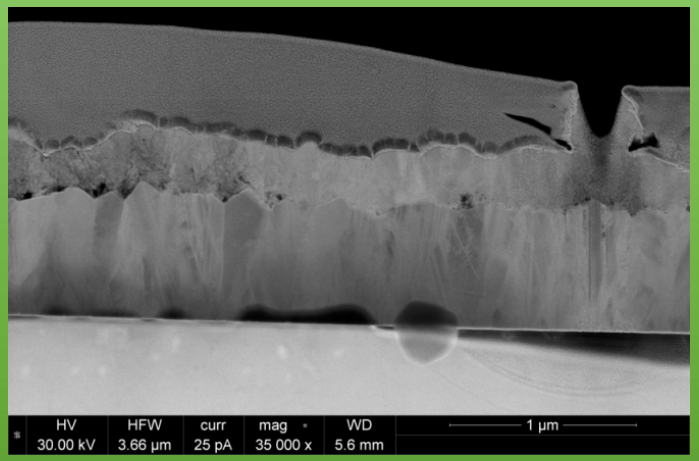


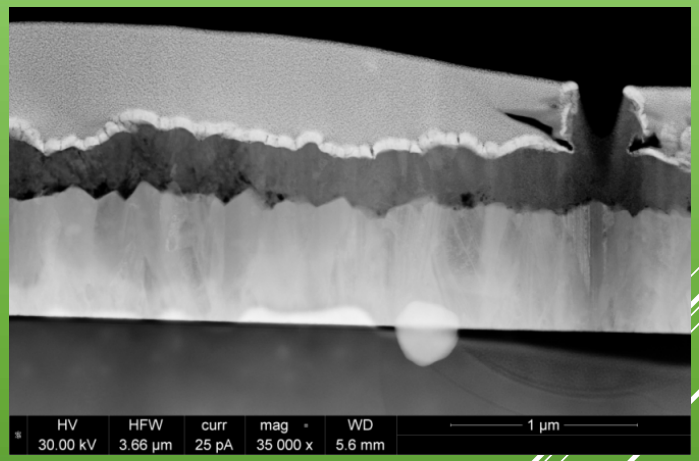
Images compliments of FEI

STEM IN SEM: MULTIPLE SIGNALS COLLECTED SIMULTANEOUSLY

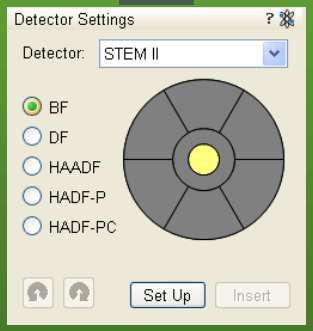
Mark DeGuire CWRU



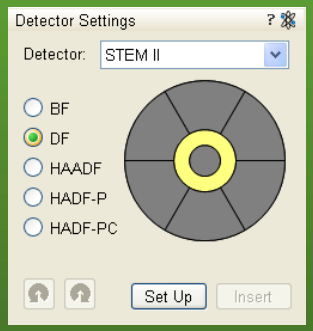




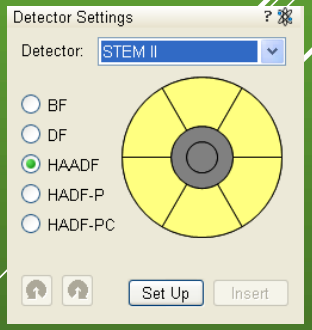
BF



DF



HAADF



The user has not direct control over the “camera length”

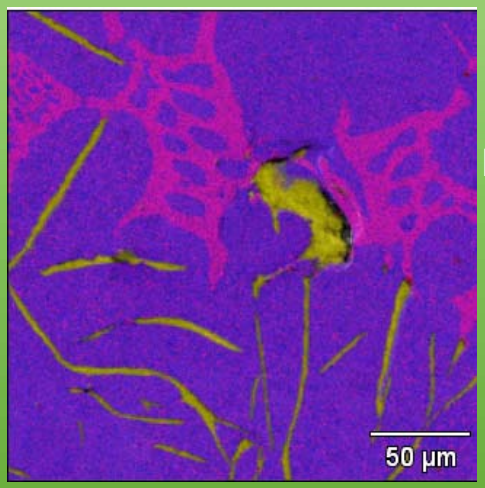
OUTLINE

- Beam optics and image formation.
- Signals Generated in an SEM and their detection.
- Beam energy & current.
- EDS - compositional analysis.
- What else can we do with an SEM?
- **How do we approach a new sample?**

WHAT IS OUR PARAMETER SPACE?

- Beam Energy
- Beam Current
- Working Distance (WD)
- Sample/Stage Tilt and rotation
- Type of signals
- Type of Detector
- Detector setup
- No immersion, Immersion mode
- Scan strategies (slow scan, integrate, average, line average/interlace).
- Stage Bias
- Scan Rotation
- Sample mounting

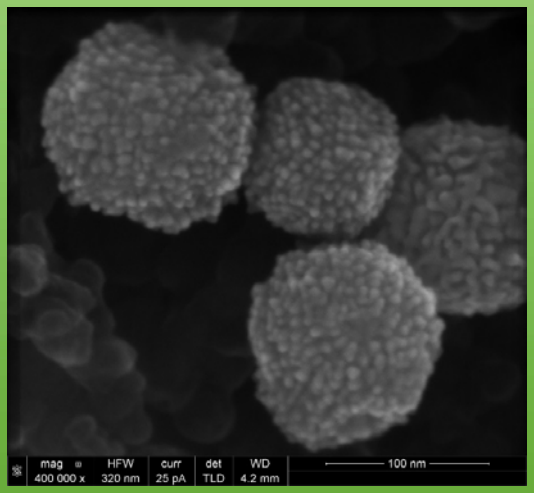
RESEARCH QUESTIONS



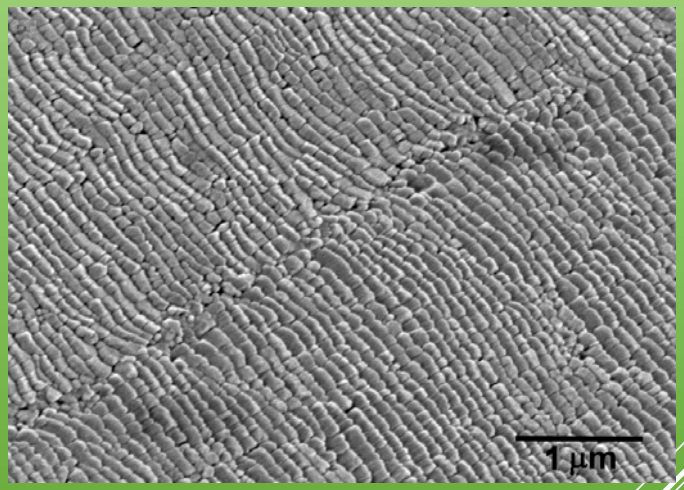
Cast Iron
EDS+SE

Fe
Cr
C

Pt Nano Particles



Sea Shell



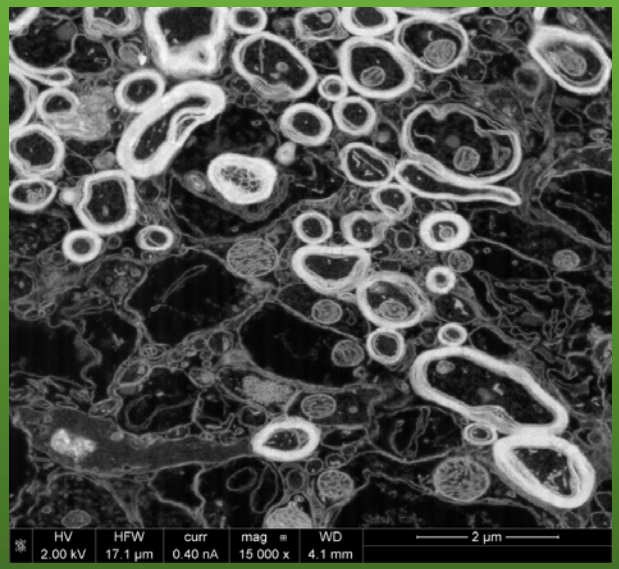
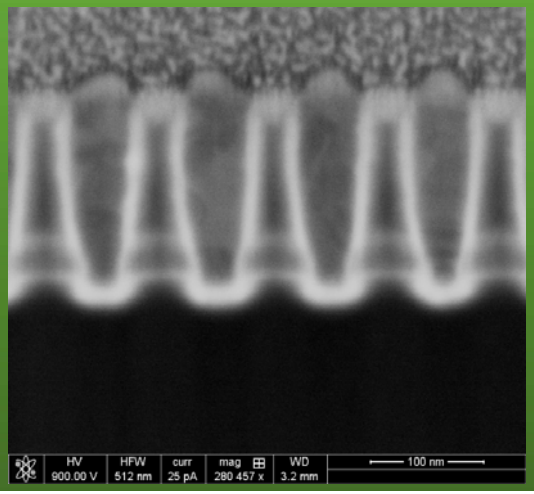
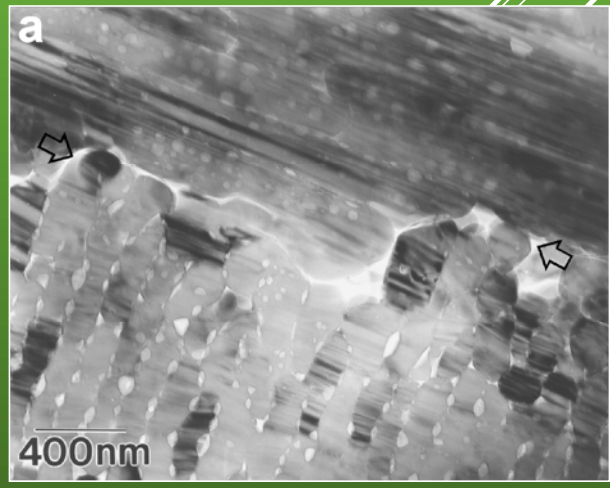


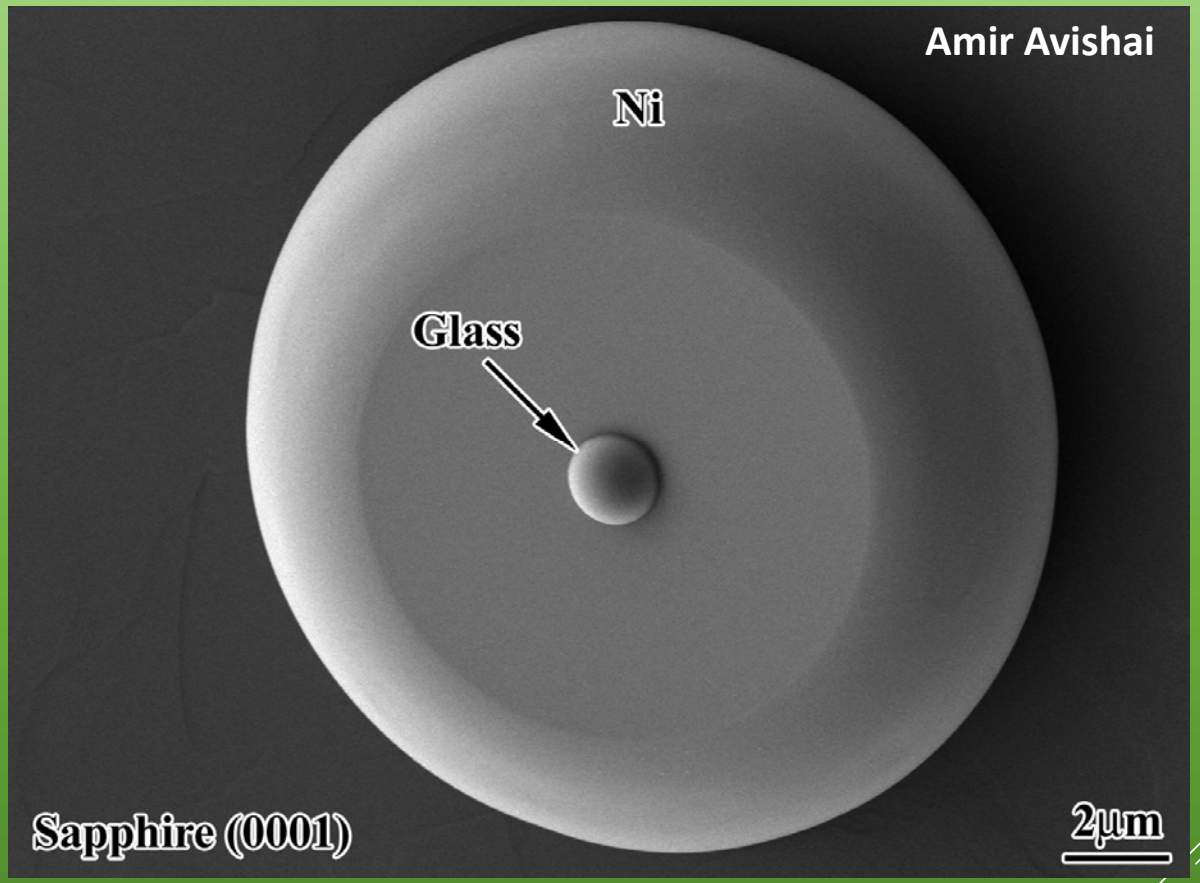
Image showing detail of axons and myelin sheaths, Mitochondria.

50nm Cu Vias



First Order Lamellar Interface





QUESTIONS