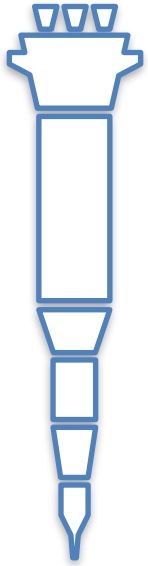
SCANNING ELECTRON MICROSCOPE



Dr. Amar Durgannavar
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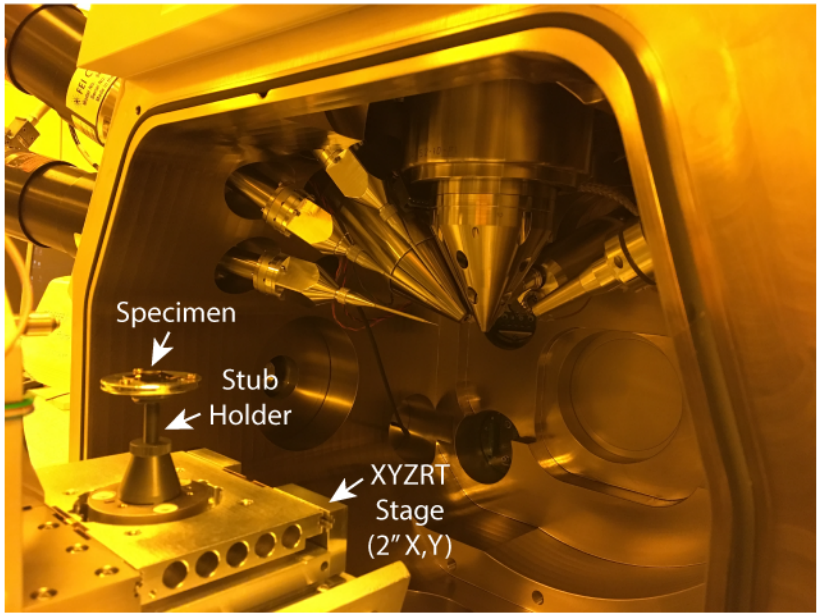
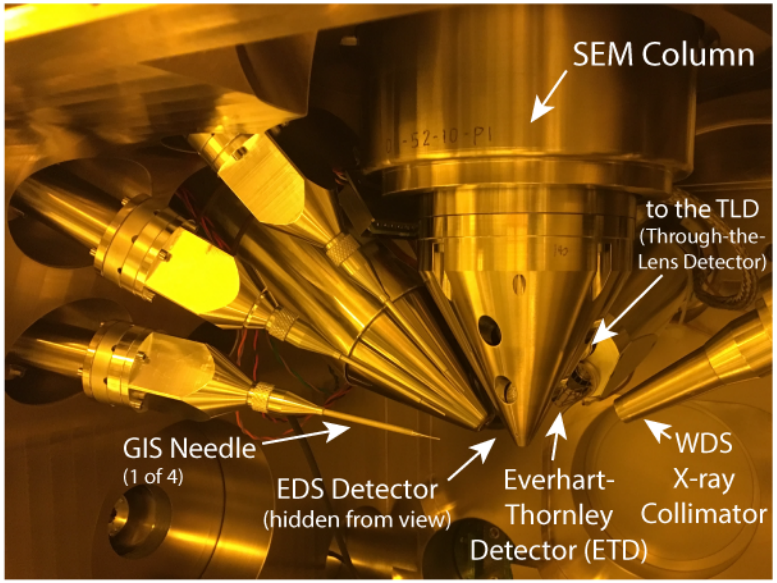
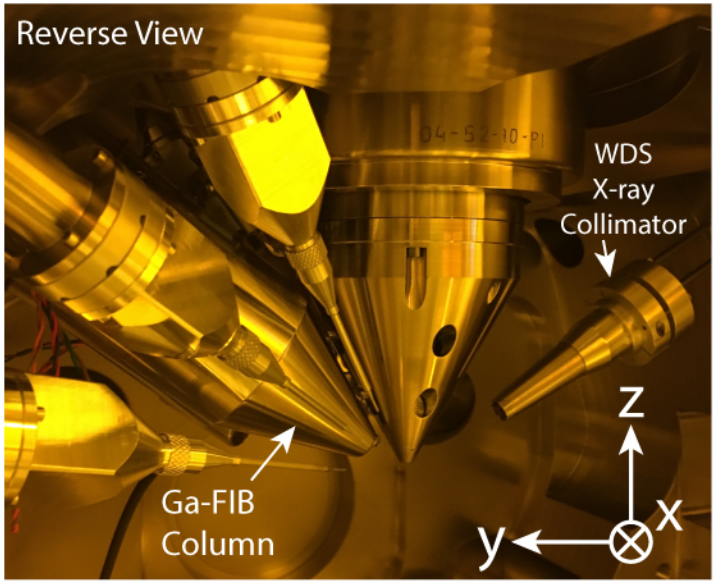
Department of Chemistry
Karnatak University
Dharwad

SEM/Ga-FIB Dual Beam System



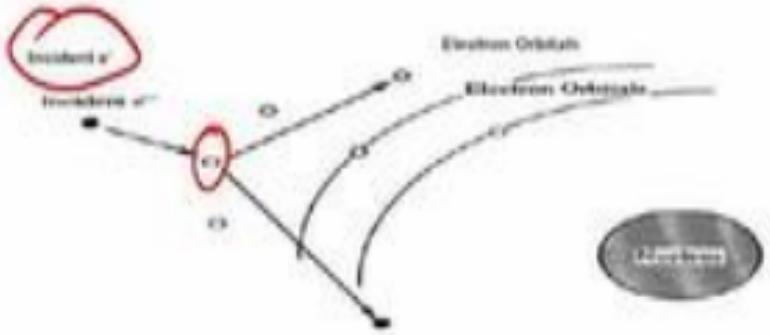
FEI Nova NanoLab 200
Chamber View





Secondary electrons

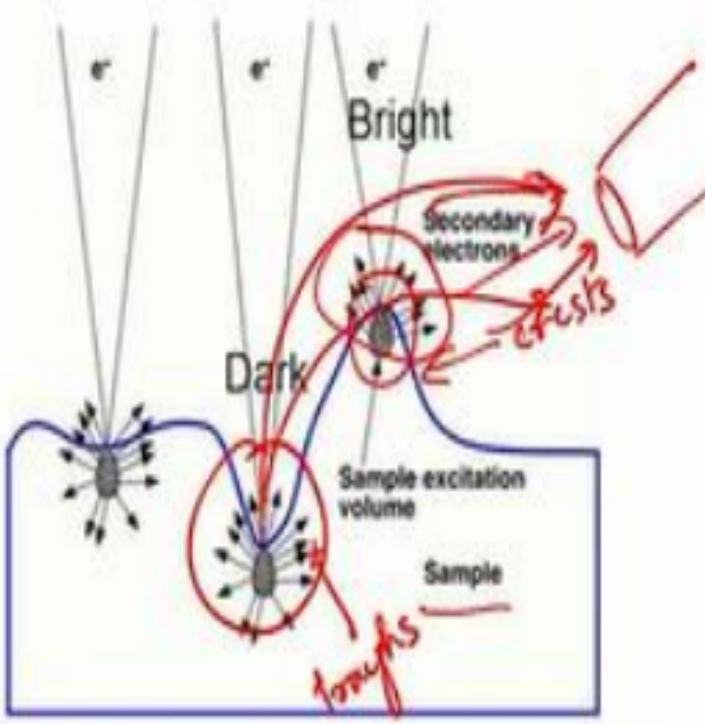
- These electrons arise due to inelastic collisions between primary electrons (the beam) and loosely bound outer shell electrons
- The energy transferred is sufficient to overcome the work function which binds them to the solid and they are ejected.
- The ejected electrons typically have E ≈ 5 - 10 eV. 50 eV is an arbitrary cut-off below which they are said to be secondary electrons.



Since secondary electrons are low energy electrons, they are easily collected by placing a positive voltage (100 - 300V) on the front of the detector (Faraday Cage). A large number of the secondaries (50 - 100%) are collected, enabling a "3D" type of image of the sample with a large depth of field

The type of detector used is called a scintillator / photomultiplier tube.

Secondary electrons



https://slideplayer.com/slide/7279884/

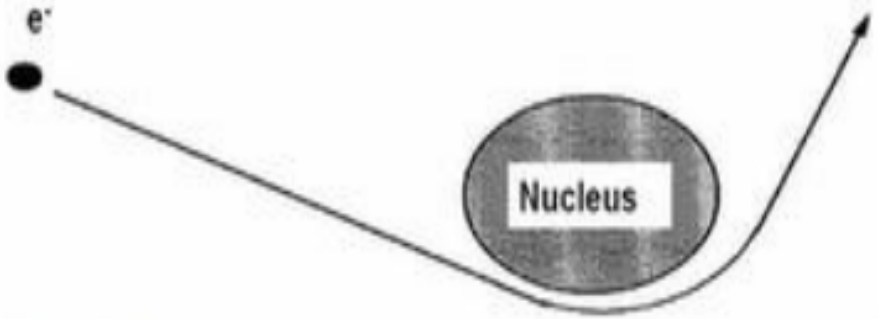
Different numbers of secondary electrons produced at different areas of the sample will provide image contrast.

If at a certain spot on the sample more secondary electrons are produced, a bright spot will appear on the image.

Often many secondary electrons are produced along raised areas of the sample, therefore many electrons will be detected, producing a bright spot on the image. This effect is called the edge effect

Some secondary electrons are produced in a valley of the sample and are difficult to deflect by the Faraday Cage. In this case only few electrons will be detected producing a dark spot on the image.

Backscattered electrons



- Arise due to elastic collisions between the incoming electron and the nucleus of the target atom (i.e. Rutherford scattering). Higher Z, more BSE emitted.
- Elastic scattering results in little (< 1 eV) or no change in energy of the scattered electrons, although there is a change in momentum (p). Since p = mv and the mass of the electron doesn't change, the direction of the velocity vector must change. The angle of scattering can range from 0 to 180° .
- Only 1 – 10% of the BSEs are collected

SEM Voltage

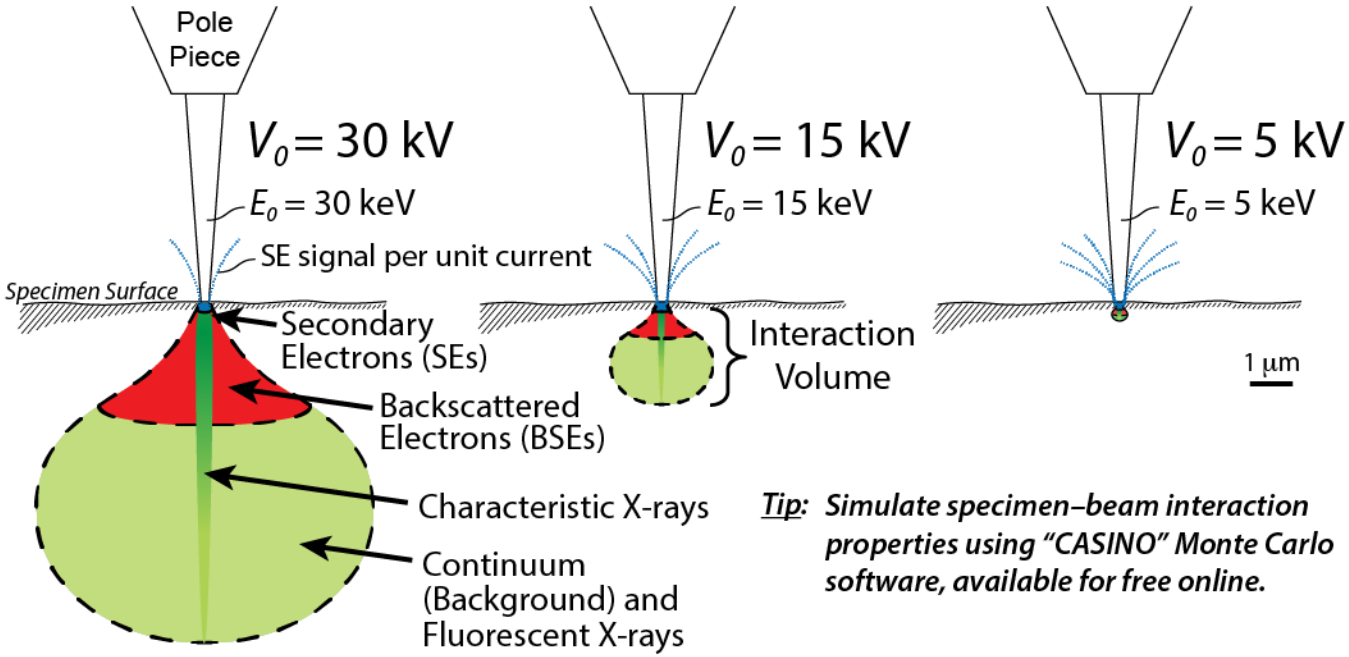
$$\lambda = \frac{1.24}{E_0^{1/2}}$$

$$d_d = \frac{0.61\lambda}{\alpha}$$

V_0 = accelerating voltage
λ = average wavelength of each electron
E_0 = average energy of each electron
d_d = spot size (1/2 Airy disk)
α = aperture angle

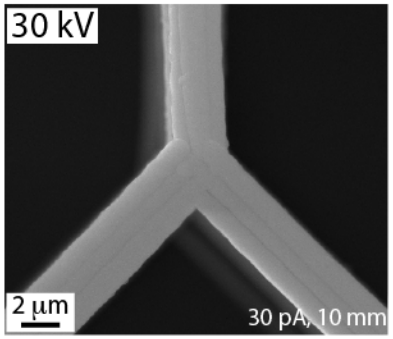
Interaction Volume ∝ Accelerating Voltage ∝ E_0 ∝ $1/\lambda^*$

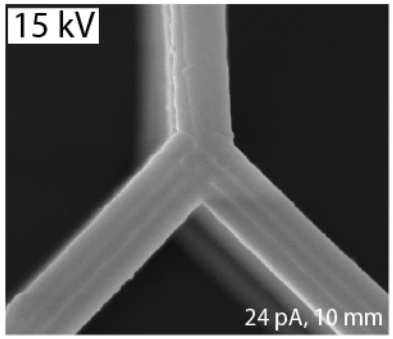
**More crucially than λ effect, chromatic aberrations (E_0 spread from source), spherical aberrations (from lens imperfections), and diffraction aberrations (around aperture) all worsen at low V_0, causing poorer resolution*

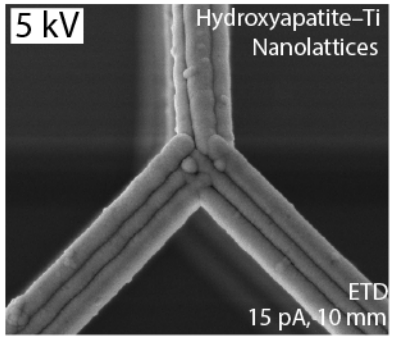


Tip: ***Simulate specimen–beam interaction properties using "CASINO" Monte Carlo software, available for free online.***

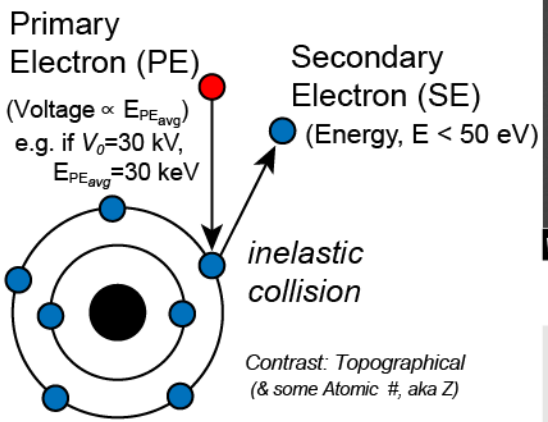
Non-Conductive Specimens: Higher V_0 yields deeper penetration of electrons, which produces charging artifacts that diminish resolution. Use lower V_0 to image non-conductive specimens. SE signal generally increases (per unit current) at lower V_0.



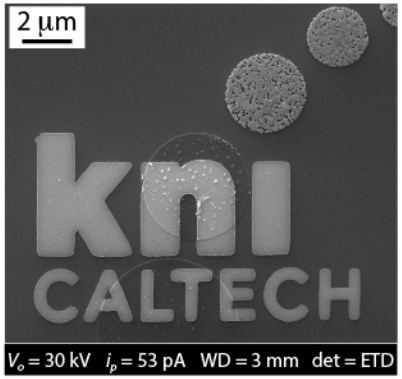


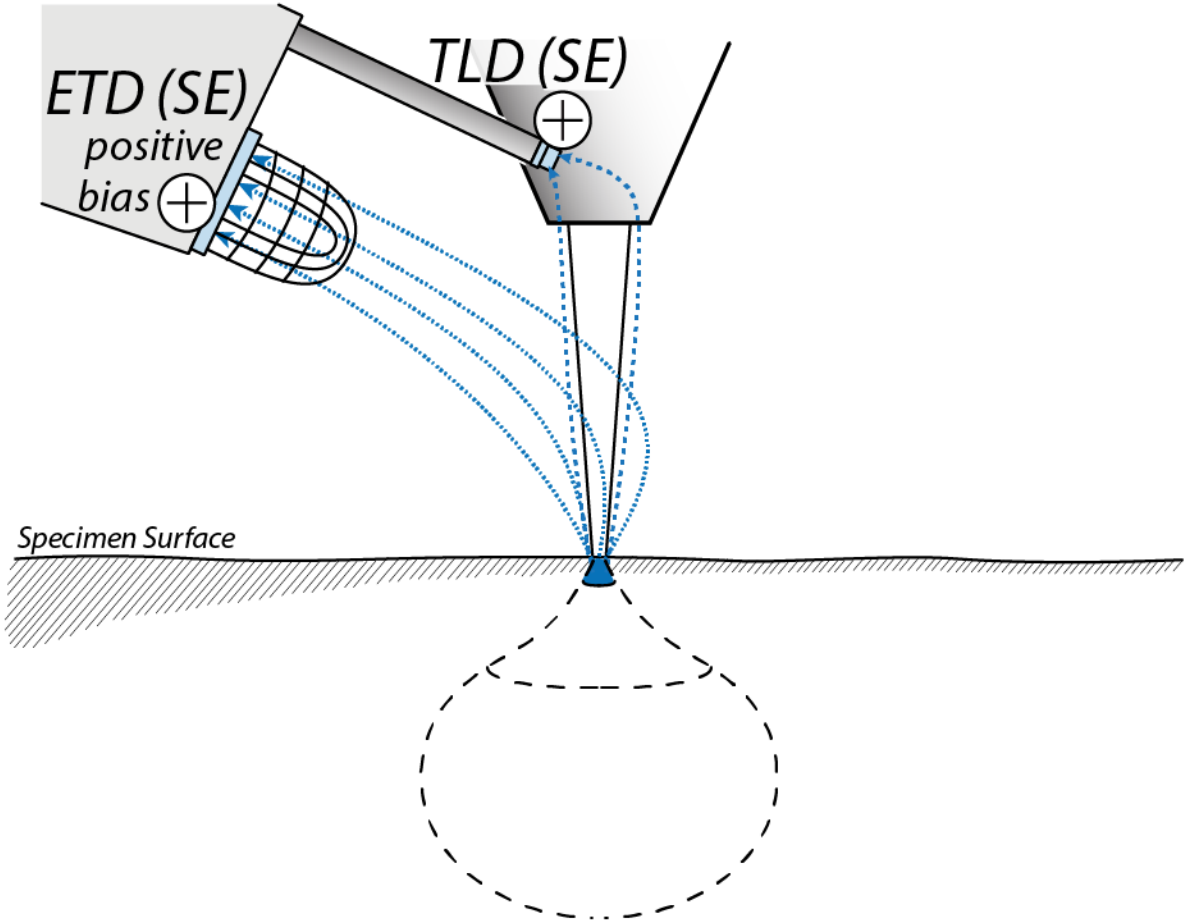


SEM Signals

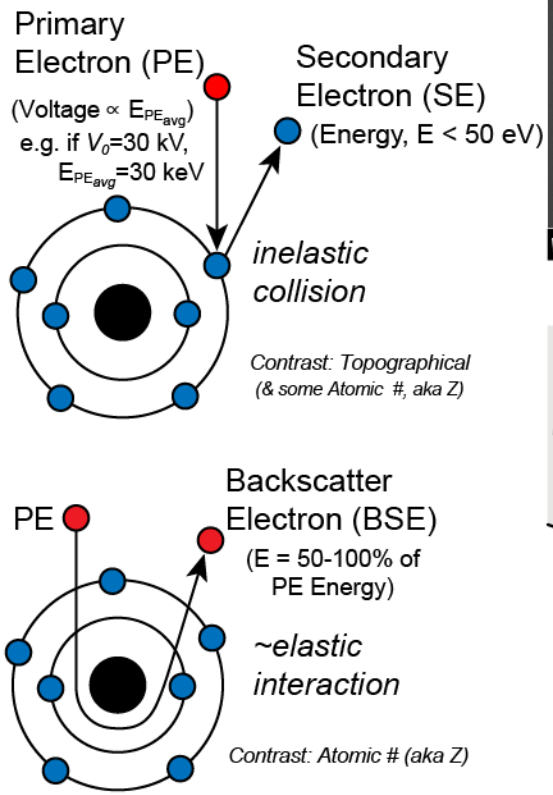


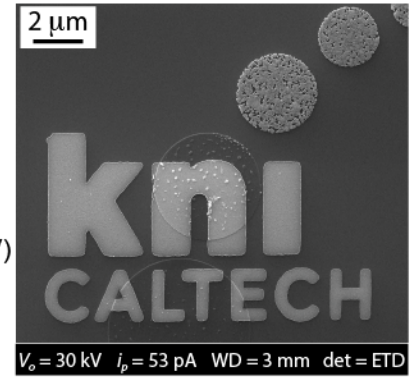
Contrast: Topographical
(& some Atomic #, aka Z)

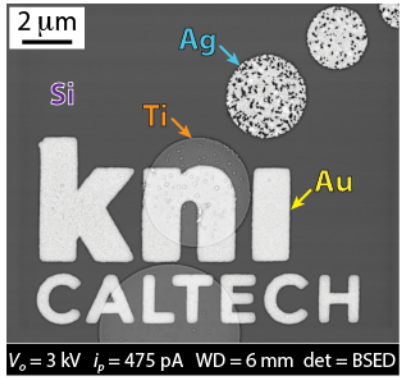


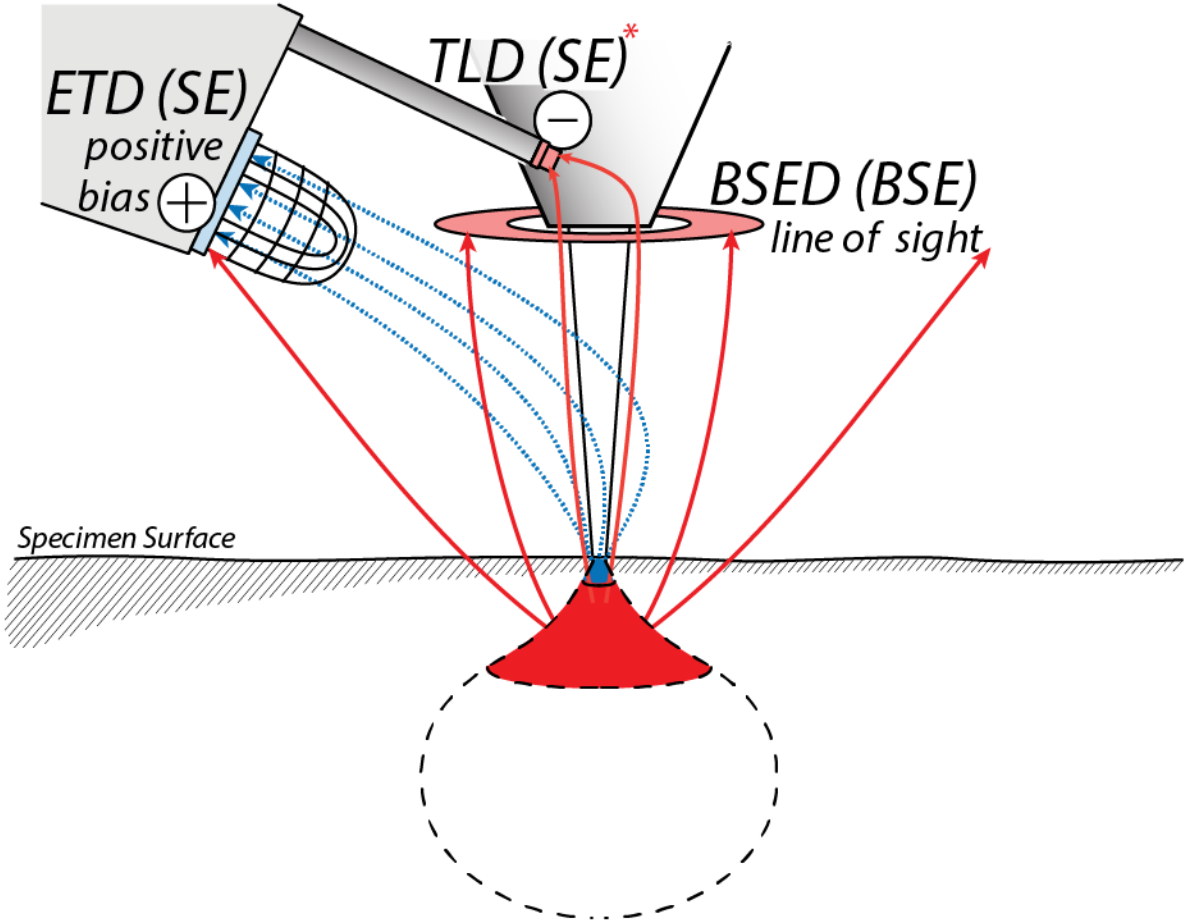


SEM Signals

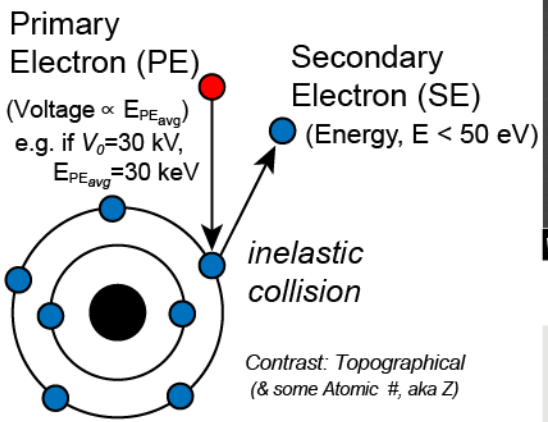

Primary
Electron (PE)
(Voltage ∝ $E_{PE_{avg}}$)
e.g. if V_0=30 kV,
$E_{PE_{avg}}$=30 keV
Secondary
Electron (SE)
(Energy, E < 50 eV)
inelastic
collision
Contrast: Topographical
(& some Atomic #, aka Z)
PE
Backscatter
Electron (BSE)
(E = 50-100% of
PE Energy)
~elastic
interaction
Contrast: Atomic # (aka Z)

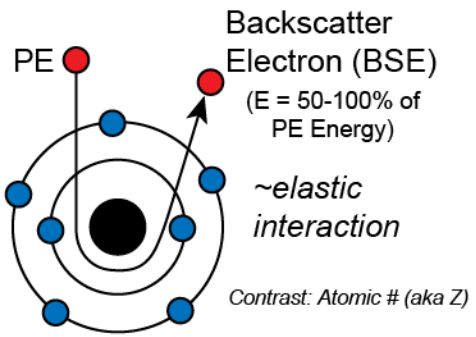

2 µm
kni
CALTECH
V_o = 30 kV i_p = 53 pA WD = 3 mm det = ETD

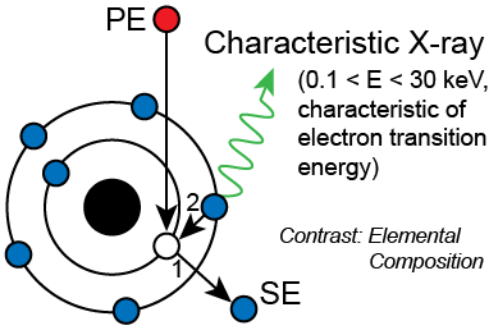

2 µm
Ag
Si
Ti
Au
kni
CALTECH
V_o = 3 kV i_p = 475 pA WD = 6 mm det = BSED

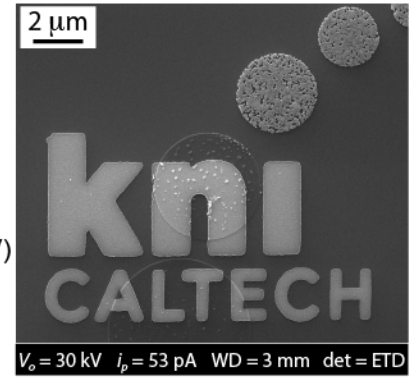

ETD (SE)
positive
bias
TLD (SE)*
BSED (BSE)
line of sight
Specimen Surface

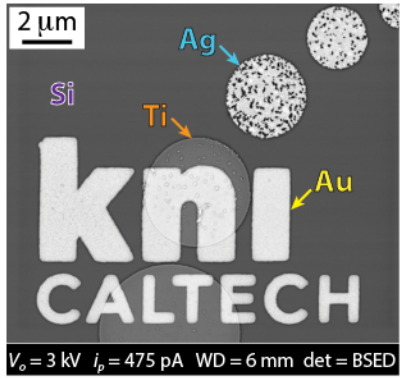
SEM Signals

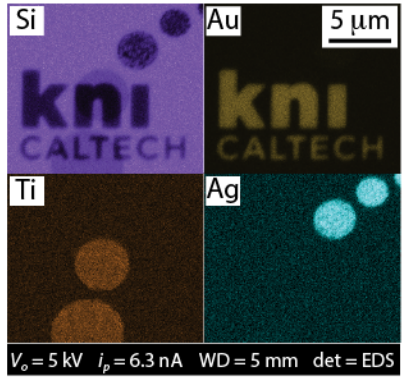

Primary
Electron (PE)
(Voltage ∝ $E_{PE_{avg}}$)
e.g. if V_o=30 kV,
$E_{PE_{avg}}$=30 keV
Secondary
Electron (SE)
(Energy, E < 50 eV)
inelastic
collision
Contrast: Topographical
(& some Atomic #, aka Z)

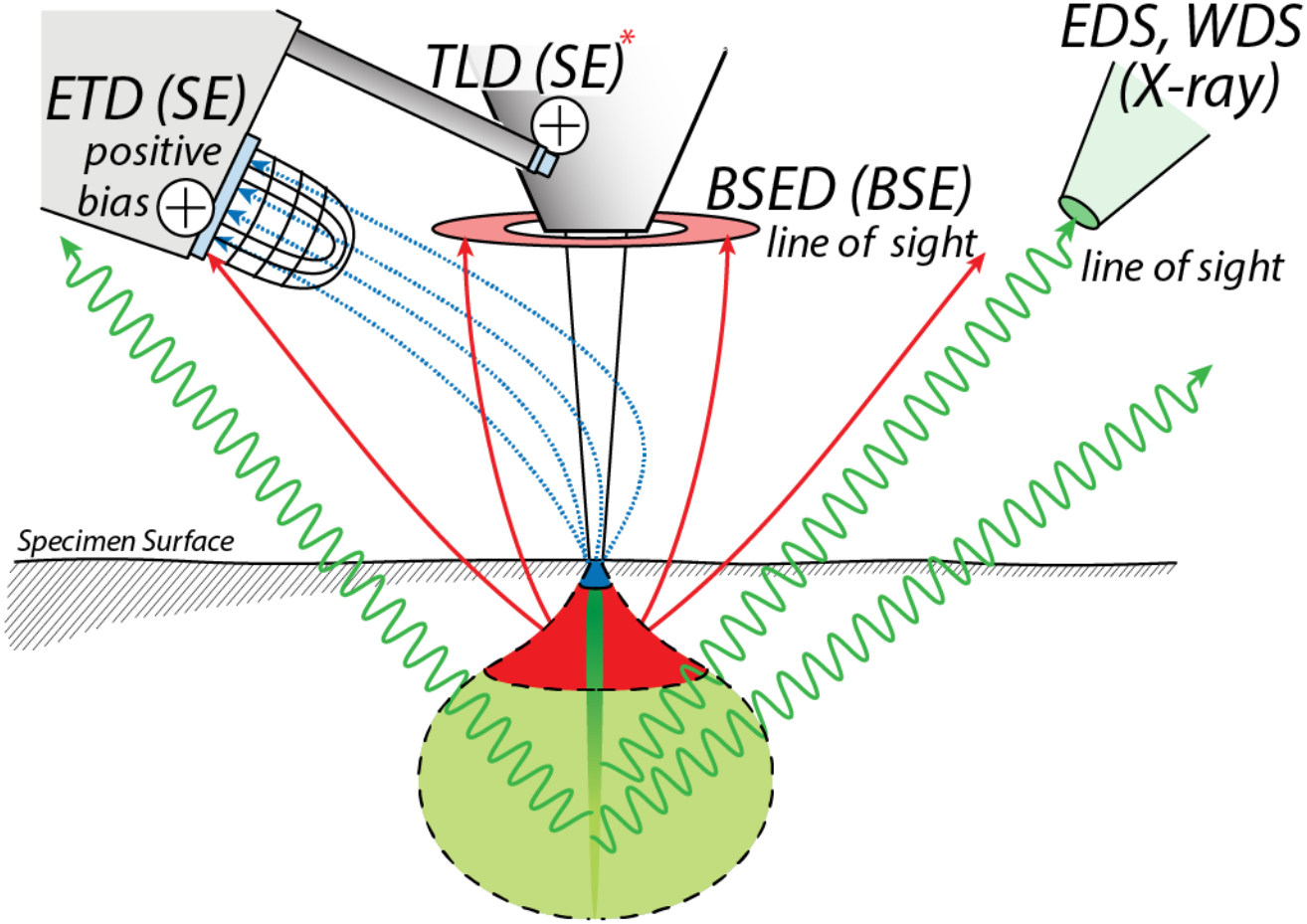

PE
Backscatter
Electron (BSE)
(E = 50-100% of
PE Energy)
~elastic
interaction
Contrast: Atomic # (aka Z)


PE
Characteristic X-ray
(0.1 < E < 30 keV,
characteristic of
electron transition
energy)
2
1
SE
Contrast: Elemental
Composition

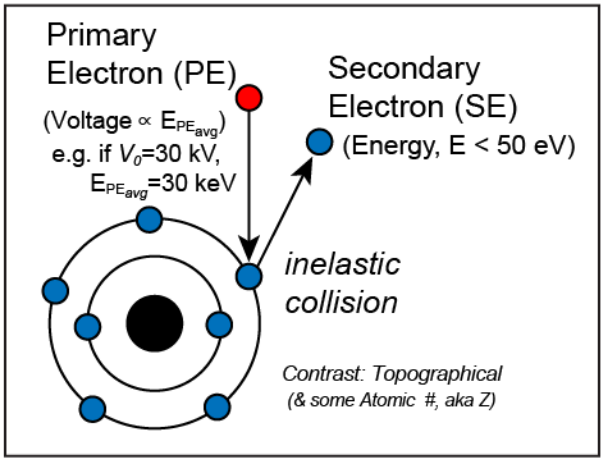

2 µm
kni
CALTECH
V_o = 30 kV i_p = 53 pA WD = 3 mm det = ETD

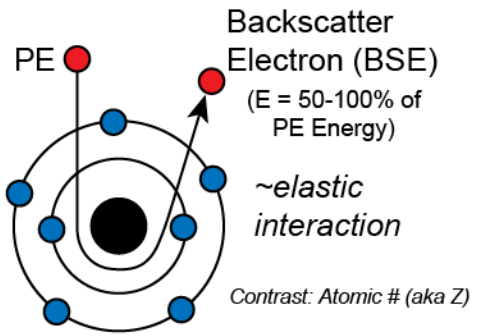

2 µm
Ag
Si
Ti
Au
kni
CALTECH
V_o = 3 kV i_p = 475 pA WD = 6 mm det = BSED

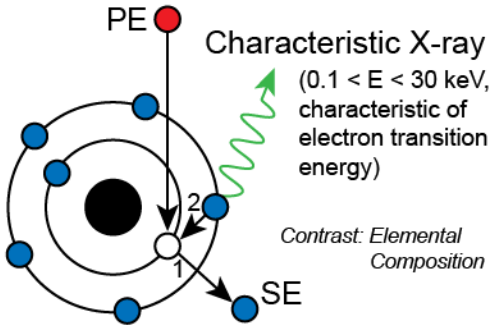

Si
Au
5 µm
kni
CALTECH
Ti
Ag
V_o = 5 kV i_p = 6.3 nA WD = 5 mm det = EDS


ETD (SE)
positive
bias
TLD (SE)*
BSED (BSE)
line of sight
EDS, WDS
(X-ray)
line of sight
Specimen Surface

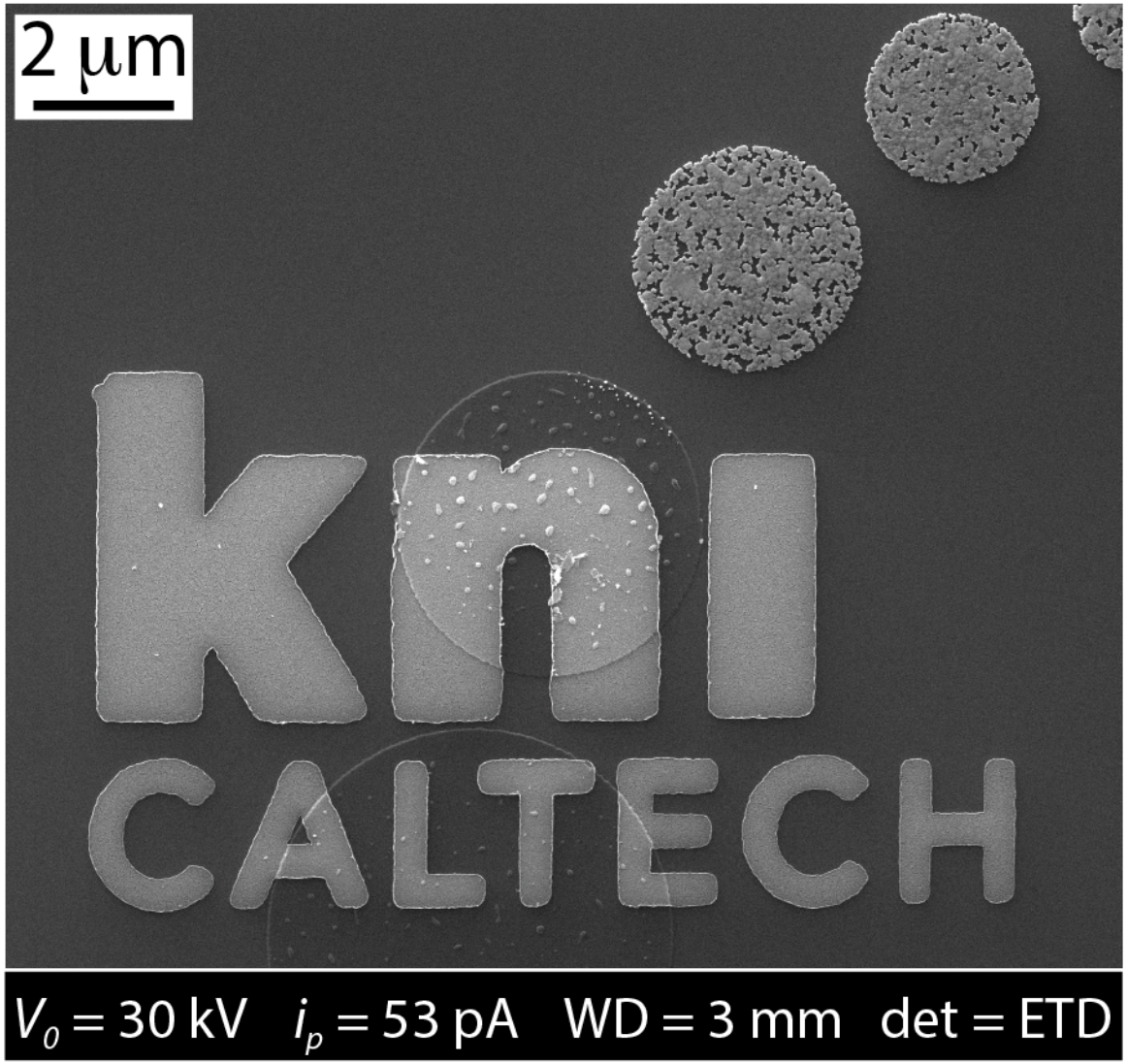
SEM Signals



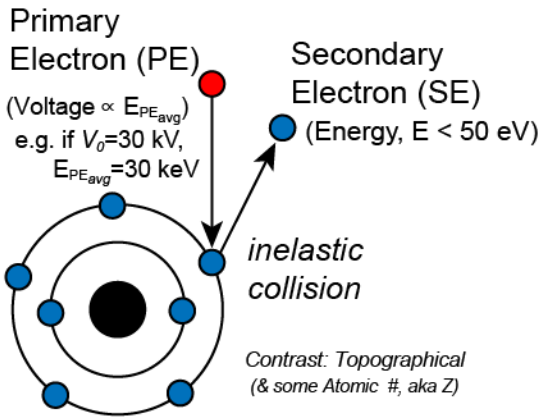


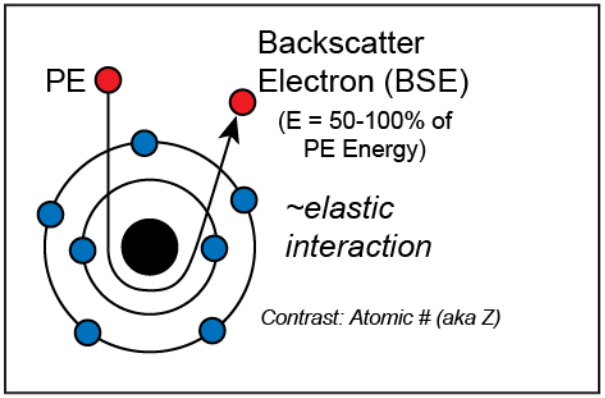


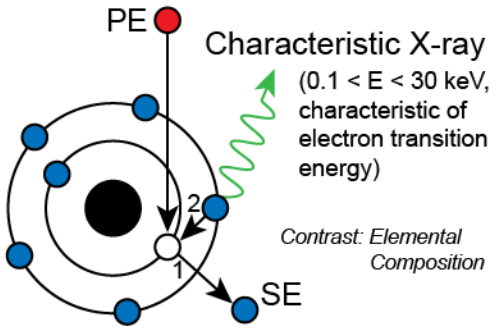
SE = Topographical Contrast (& some Z contrast)



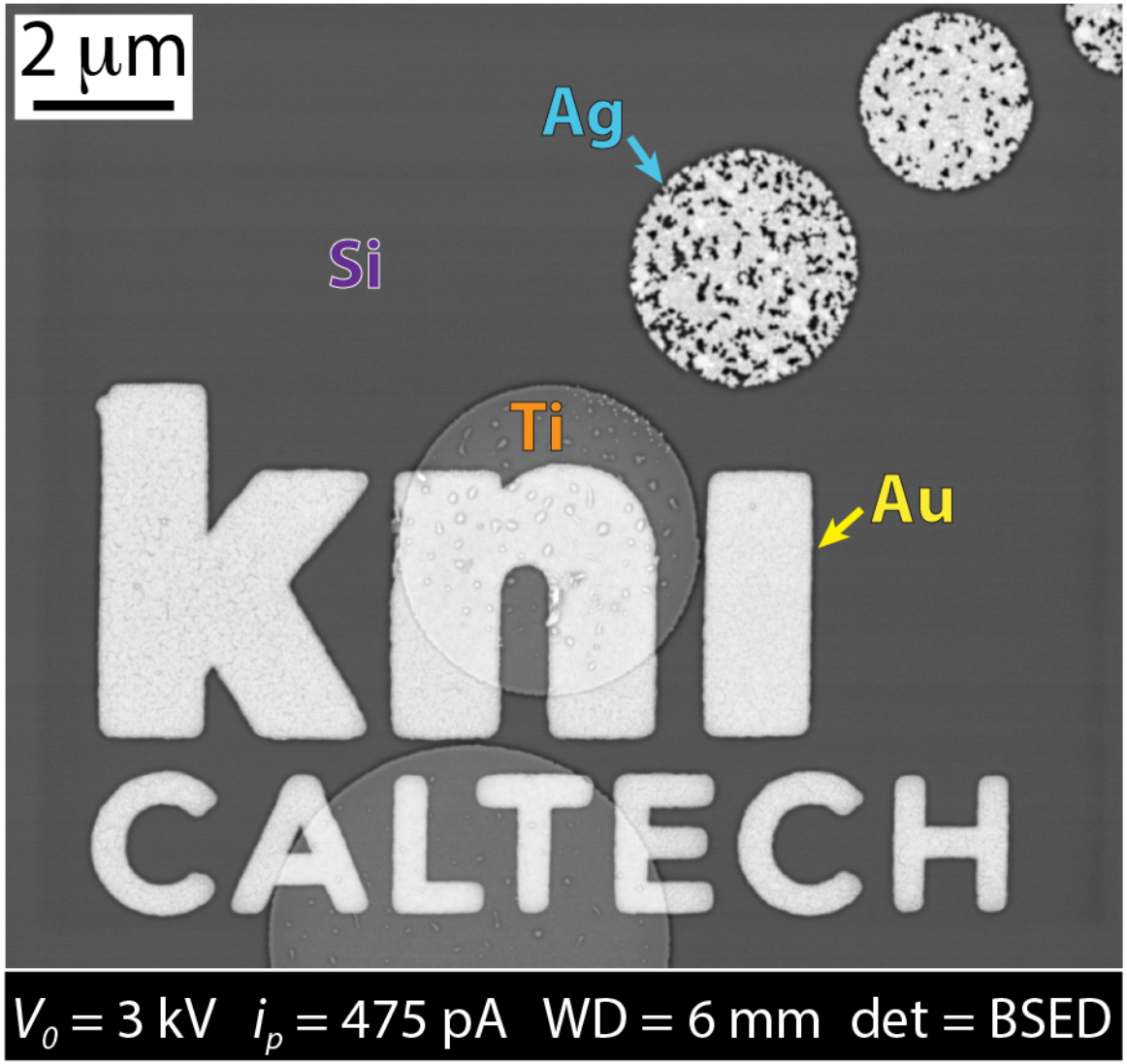
SEM Signals



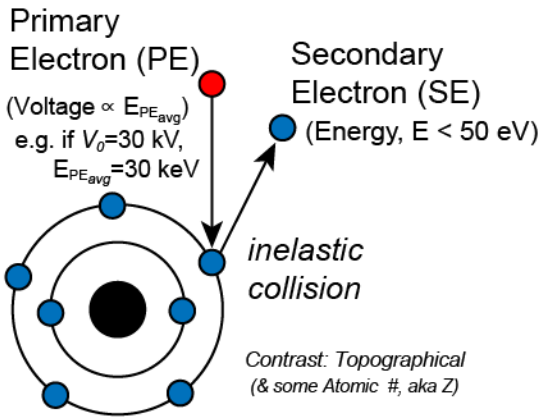


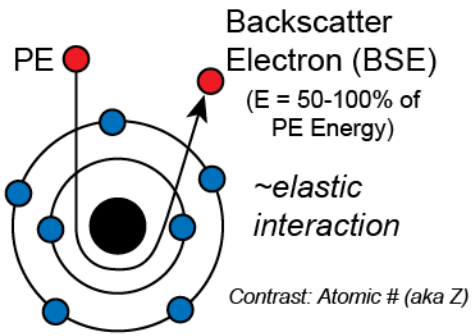


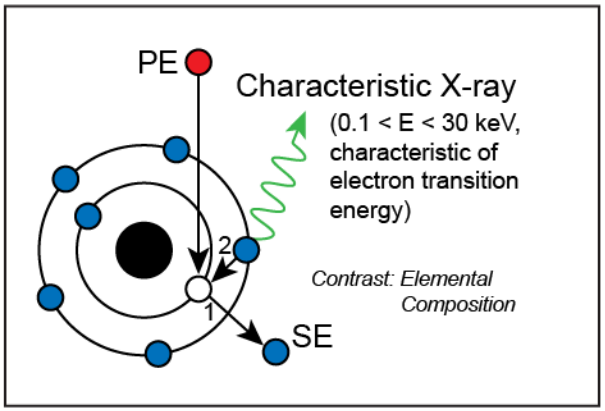
BSE = Atomic Number Contrast (aka Z Contrast)



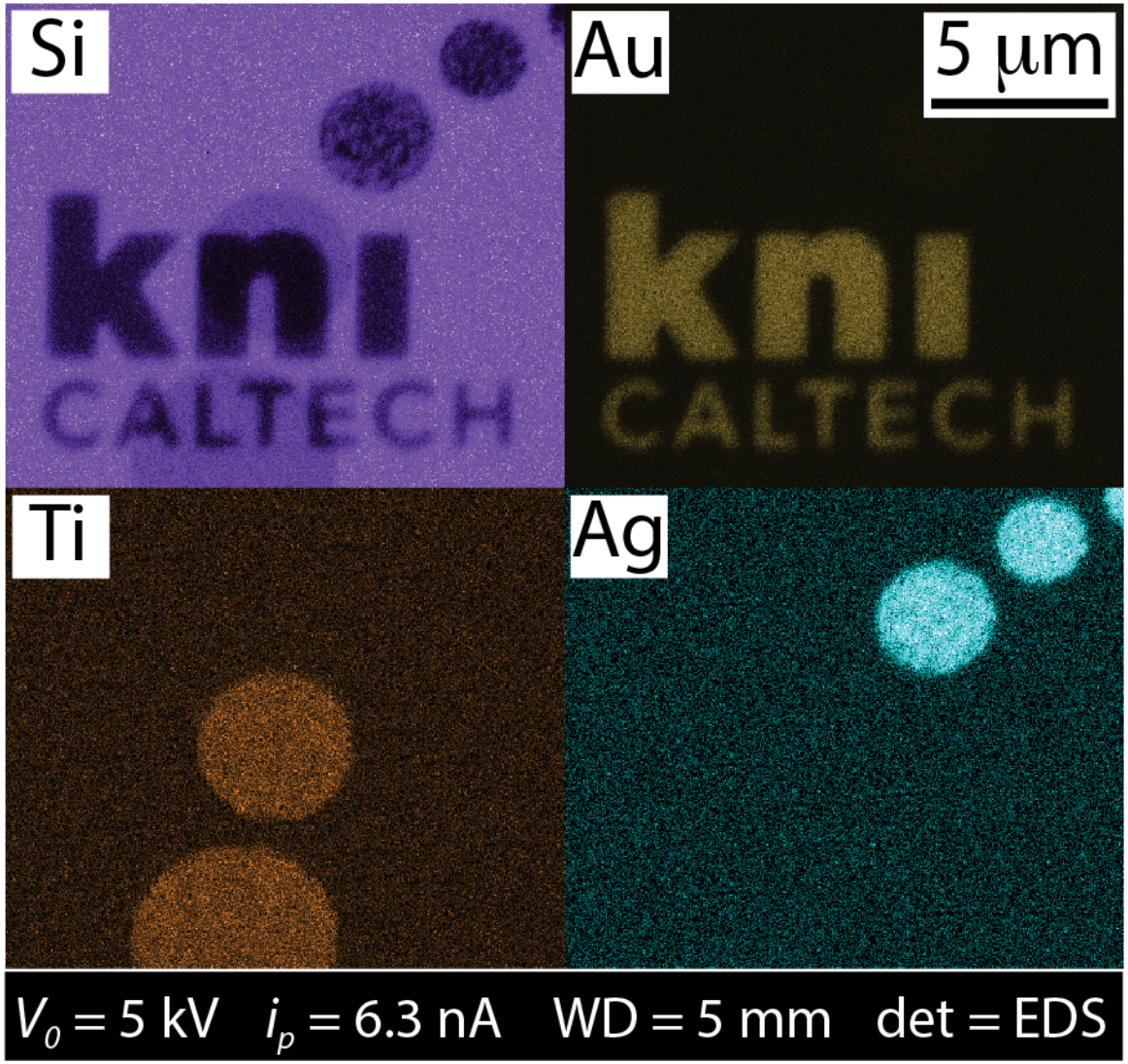
SEM Signals





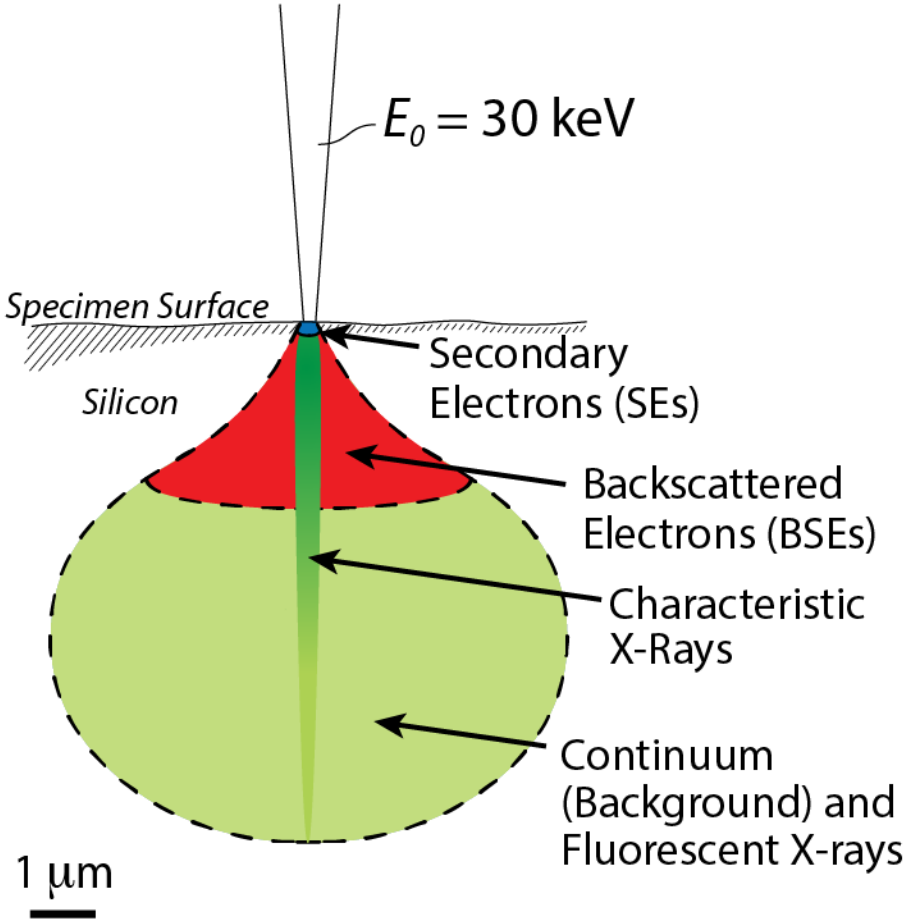


X-ray = Elemental Composition Contrast

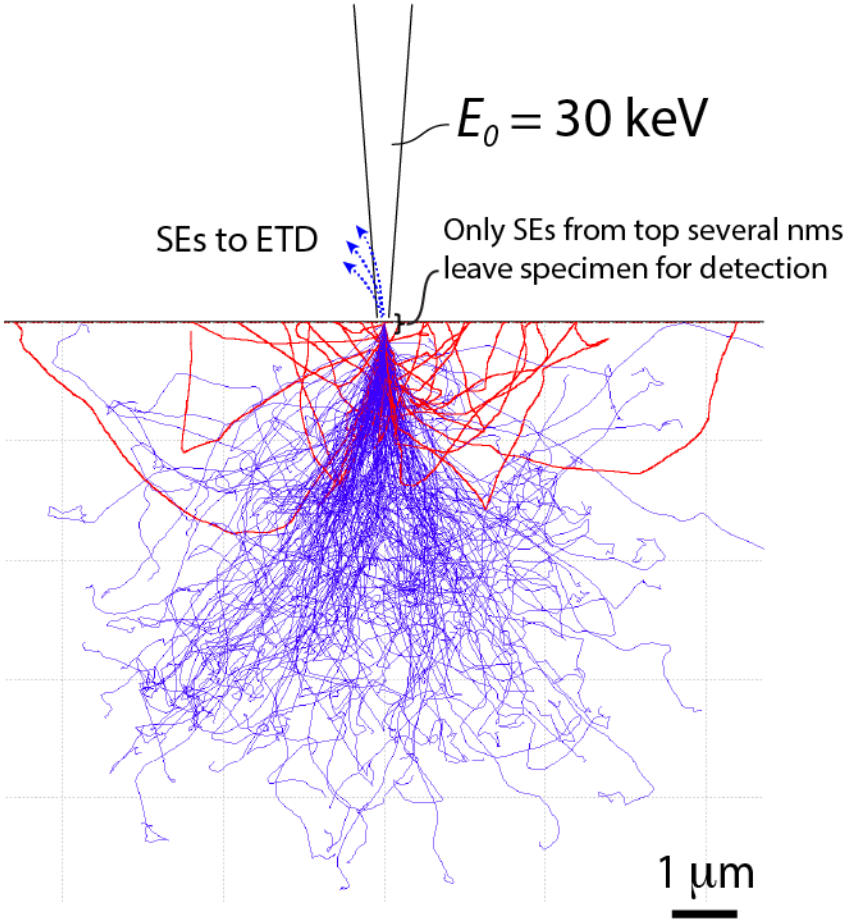


E-Beam Simulations

Simulate electron beam interactions using free Monte Carlo software, e.g. CASINO v. 2.51



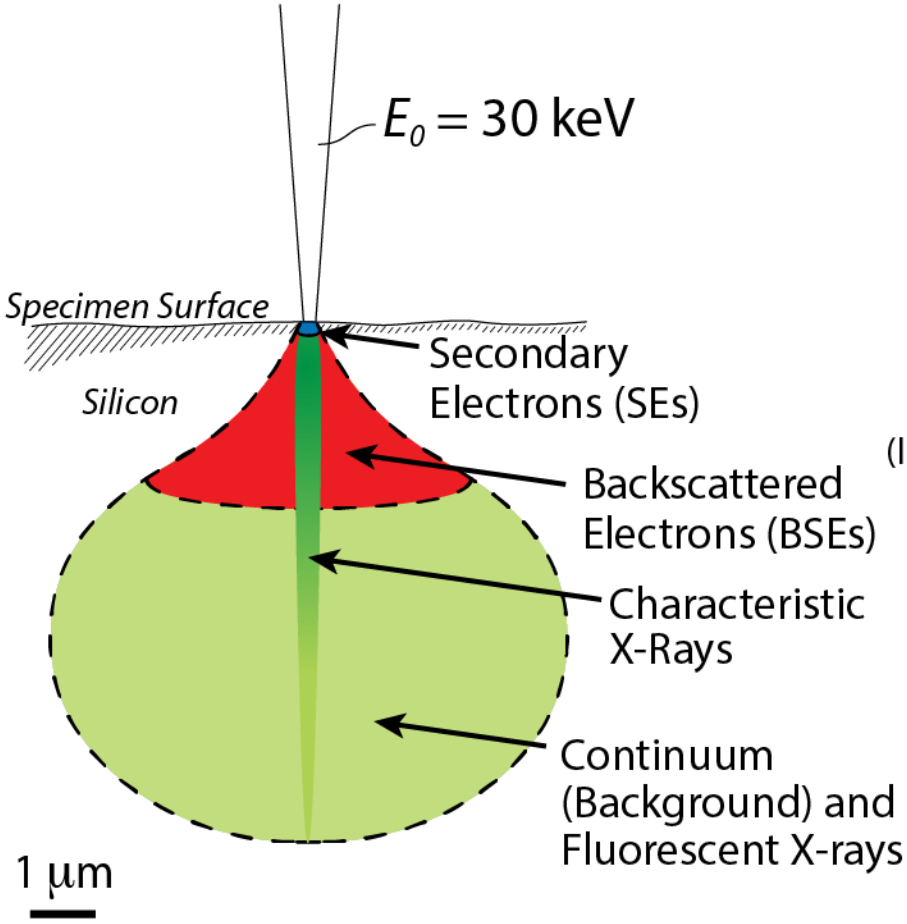
Schematic



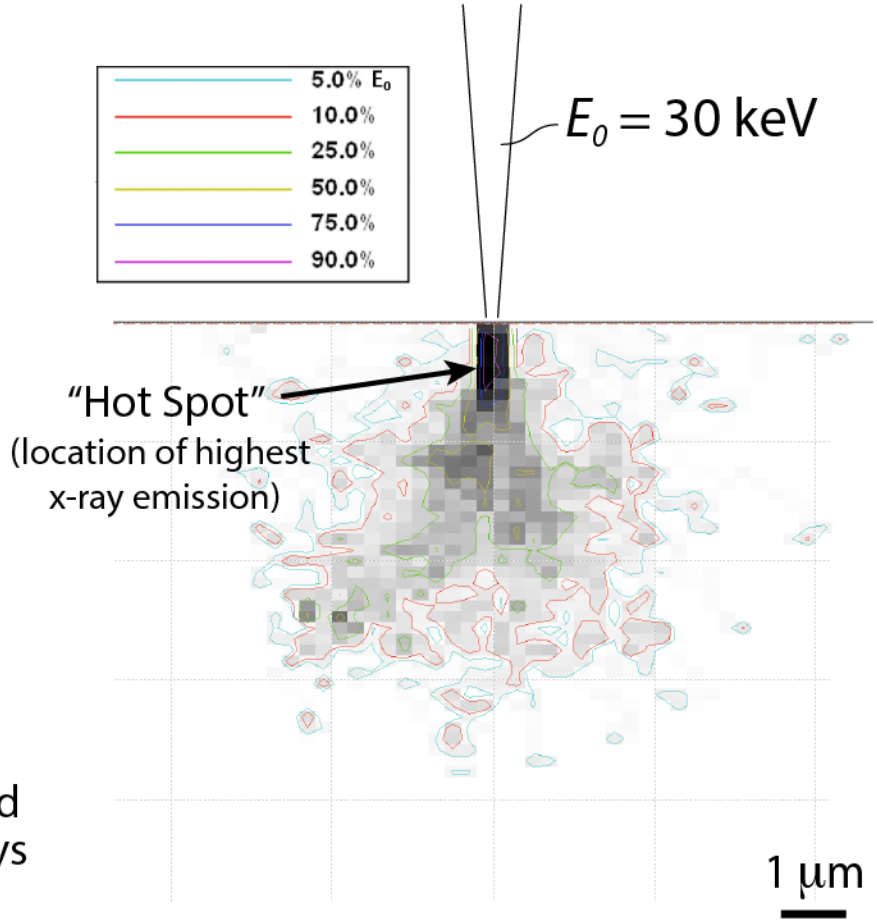
***Reabsorbed* vs. *Backscattered* Electrons**

E-Beam Simulations

Simulate electron beam interactions using free Monte Carlo software, e.g. CASINO v. 2.51



Schematic



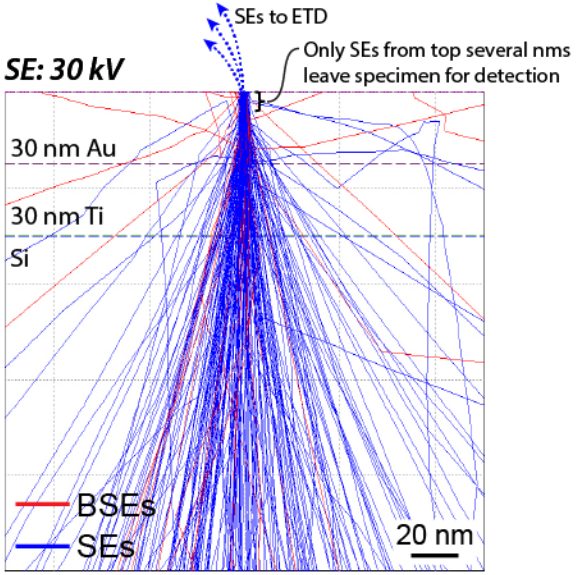
'Energy by Position' Map

E-Beam Simulations

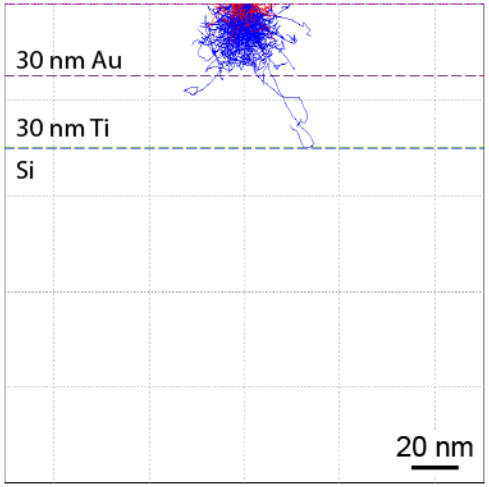
Selecting Voltage for Three Different Signal Captures

Simulate electron beam interactions using free Monte Carlo software, e.g. CASINO v. 2.51

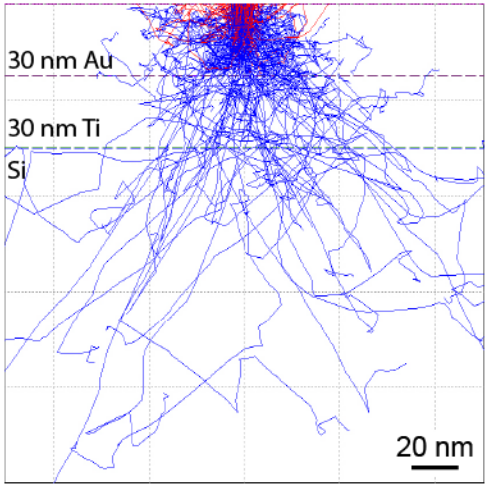
SE: 30 kV

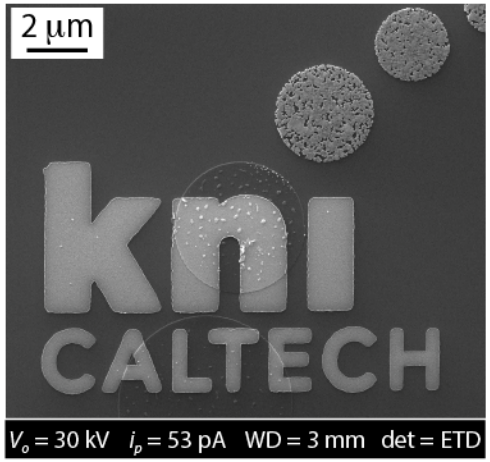


BSE: 3 kV

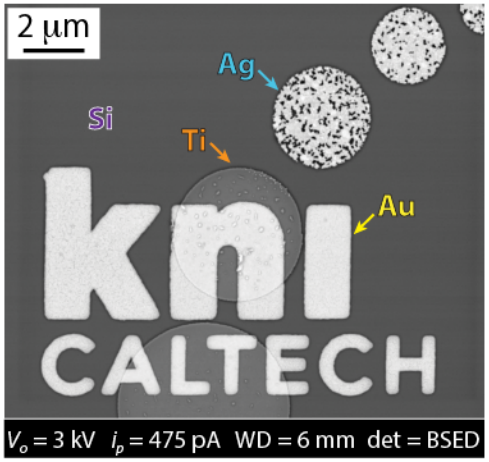


X-Ray: 5 kV

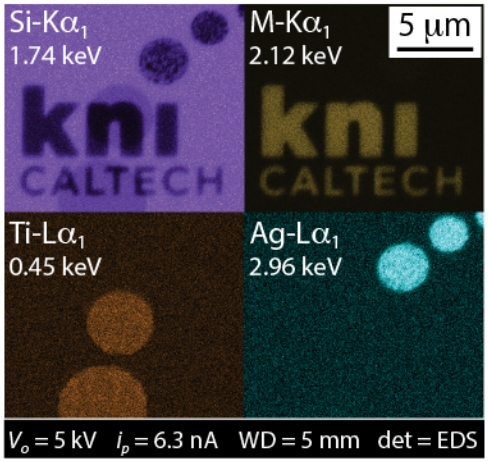




Looking for topographical contrast at high resolution on a conductive specimen: 30 kV

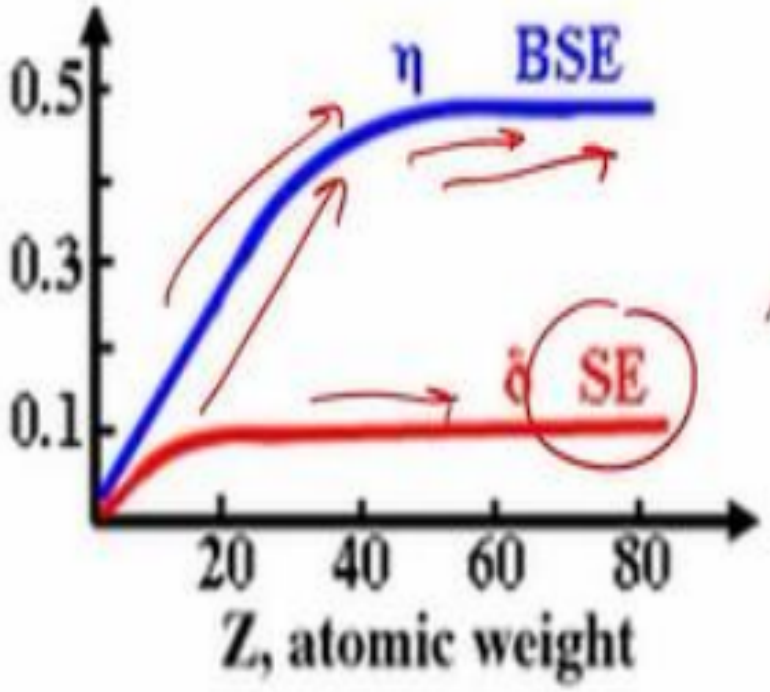


Need to keep BSE signal inside each, respective 30 nm deposition layer, can increase current to maximize signal: 3 kV



Want ~2x the voltage of the highest targeted energy peak (Ag-Lα$_1$ = 2.96 keV), w/out probing too deeply into substrate: 5 kV

Atomic weight effects on yield



What info can be obtained by SEM

Topography

- The surface features of an object or "how it looks", its texture; direct relation between these features and materials properties

Morphology

- The shape and size of the particles making up the object; direct relation between these structures and materials properties

Composition

- The elements and compounds that the object is composed of and the relative amounts of them; direct relationship between composition and materials properties

  BSE intensity = f [Z]

Crystallographic Information

- How the atoms are arranged in the object; direct relation between these arrangements and material properties

Important aspects

- Magnification - An image is obtained by taking the signal from the sample and transferring it to a CRT screen. By decreasing the size of the scanned area (from which we get the signal), magnification is produced.

- Resolution – ability to resolve two closely spaced points. While you may have to be at a high magnification to see small features, resolution is NOT the same as magnification.

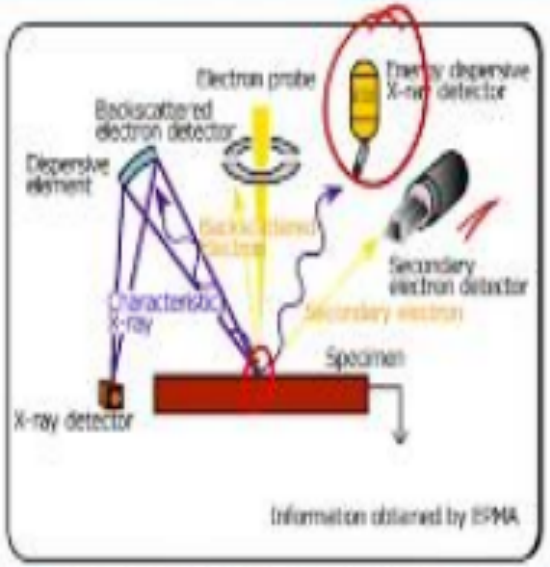
  One way to improve resolution is by reducing the size of the electron beam that strikes the sample

- Depth of field – height over which a sample can be clearly focused

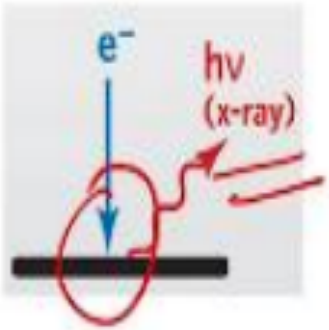
- Spot size - A smaller spot size or smaller diameter of the beam of electrons will resolve more detailed structures of the sample as compared to a beam with a big spot size

- Working distance – distance between bottom of SEM column and top of sample; shorter WD, smaller beam dia, better resolution, but lower DOF

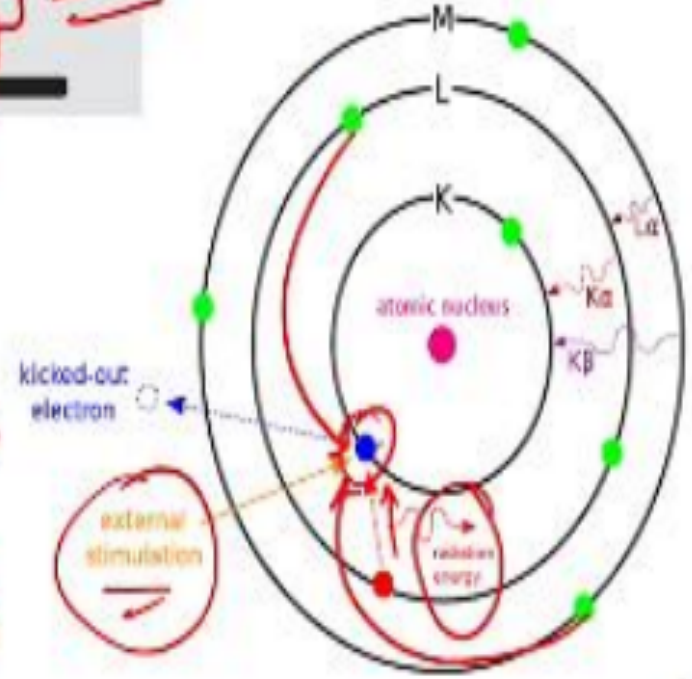
Energy-dispersive x-ray spectroscopy



https://www.jeol.co.jp/en/science/epma.html



EDX
EDS
EDAX



Higher working voltage is necessary good EDX signal

Detectors and stage

Santos, 2016

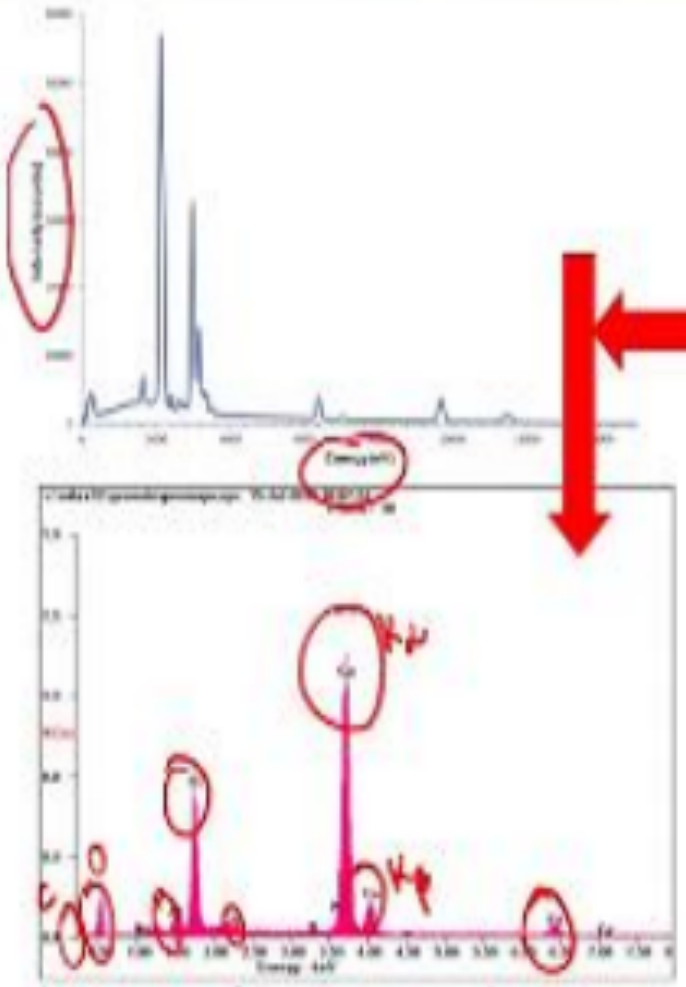
Secondary detector for secondary electrons – provide morphological information

Backscatter detector for backscattered electrons – provide compositional contrast

EDS detector for X rays – provide information about phases

Motorized movement of sample stage possible in all directions

Energy levels depend on atomic number



Energy levels for all transitions in each elements is quantised

Applications of Scanning Electron Microscopy

Topography: The surface features of an object or "how it looks", its texture; direct relation between these features and materials properties (hardness, reflectivity... etc.)

Morphology: The shape and size of the particles making up the object; direct relation between these structures and materials properties (ductility, strength, reactivity...etc.)

Composition: The elements and compounds that the object is composed of and the relative amounts of them; direct relationship between composition and materials properties (melting point, reactivity, hardness...etc.)

Crystallographic Information: How the atoms are arranged in the object; direct relation between these arrangements and materials properties (conductivity, electrical properties, strength.etc.)

Advantages of SEM

- It gives detailed 3D and topographical imaging and the versatile information garnered from different detectors.
- This instrument works very fast.
- Modern SEMs allow for the generation of data in digital form.
- Most SEM samples require minimal preparation actions.

Disadvantages of SEM

- SEMs are expensive and large.
- Special training is required to operate an SEM.
- The preparation of samples can result in artifacts.
- SEMs are limited to solid samples.
- SEMs carry a small risk of radiation exposure associated with the electrons that scatter from beneath the sample surface.

References

Goldstein, J.I., Yakowitz, H.. Newbury, D.E Lifshin, E.. Colby, J.W Colby J.W. and. J.R. Coleman. 1975. Pratical Scanning Electron Microscopy: Electron and Ion Microprobe Analysis.