Scanning Electron Microscopy (SEM)

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Physical Inspection and AttacKs on ElectronicS (PHIKS)

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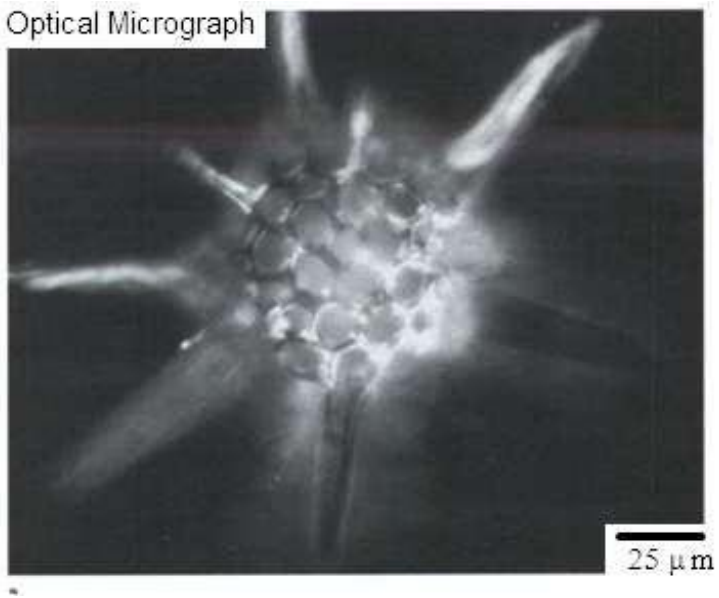
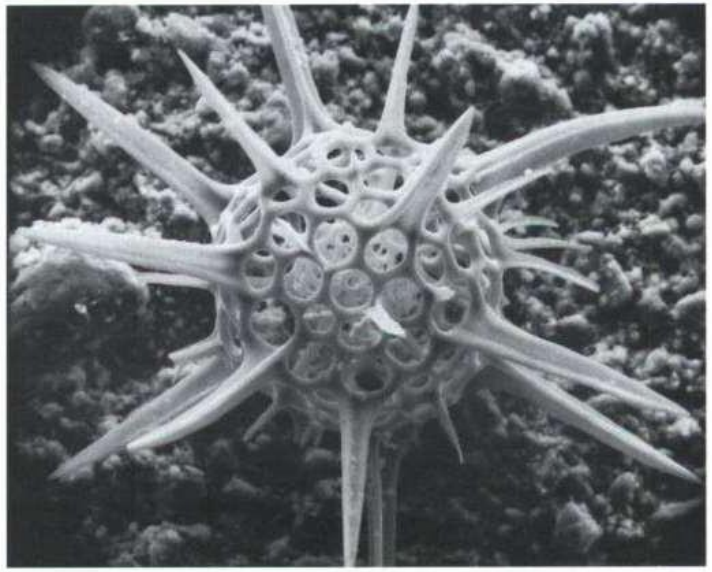
Comparison to Optical Microscopy



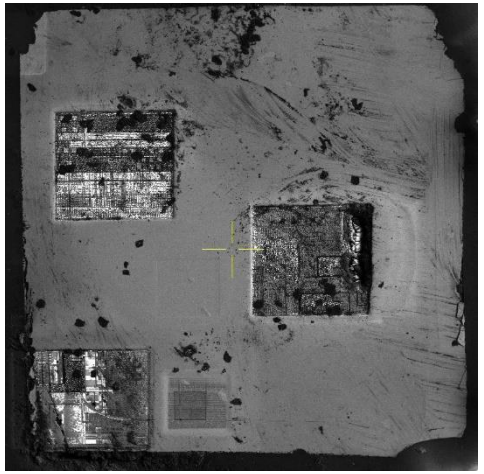
OPTICAL

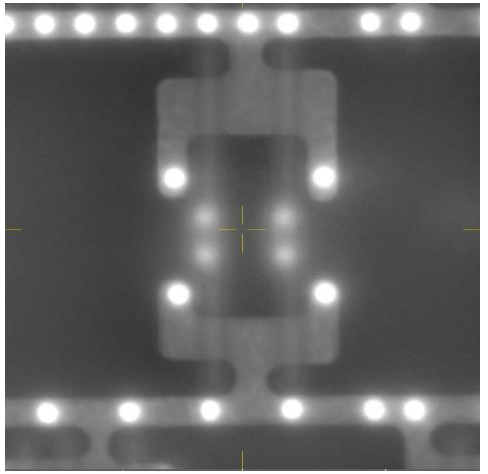


SEM (Secondary Electron)

- The higher resolution and depth of focus available with the SEM are clearly observed.
- It also provides a very wide, easily adjustable range of magnifications as compared to optical microscope.
- The high resolution attainable (very small probe size) is due to very low mass and short wavelength of energetic electrons(0.007nm @30kV).
- The combination of high brightness sources of electrons and electron optics allow the formation and manipulation of very fine focused electron beams to probe the sample surface for imaging and analysis.

ADVANTAGES OF USING SCANNING ELECTRON MICROSCOPE (SEM)

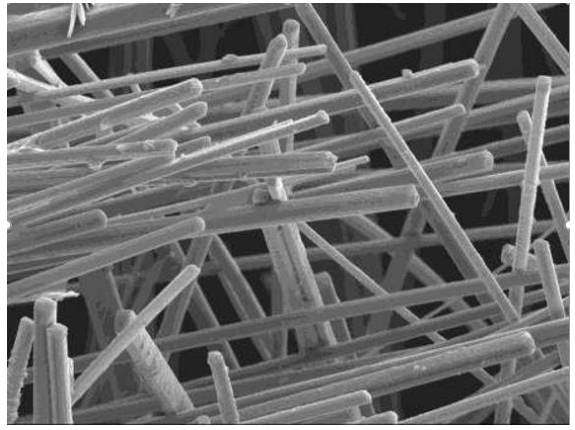


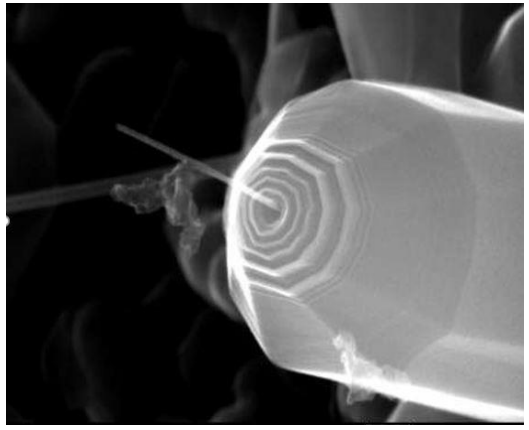


Extremely wide range of Magnifications

Magnification : 142x Magnification :45000x

Exceptionally High Depth of Focus





Magnification :2,200x Magnification : 100,000x

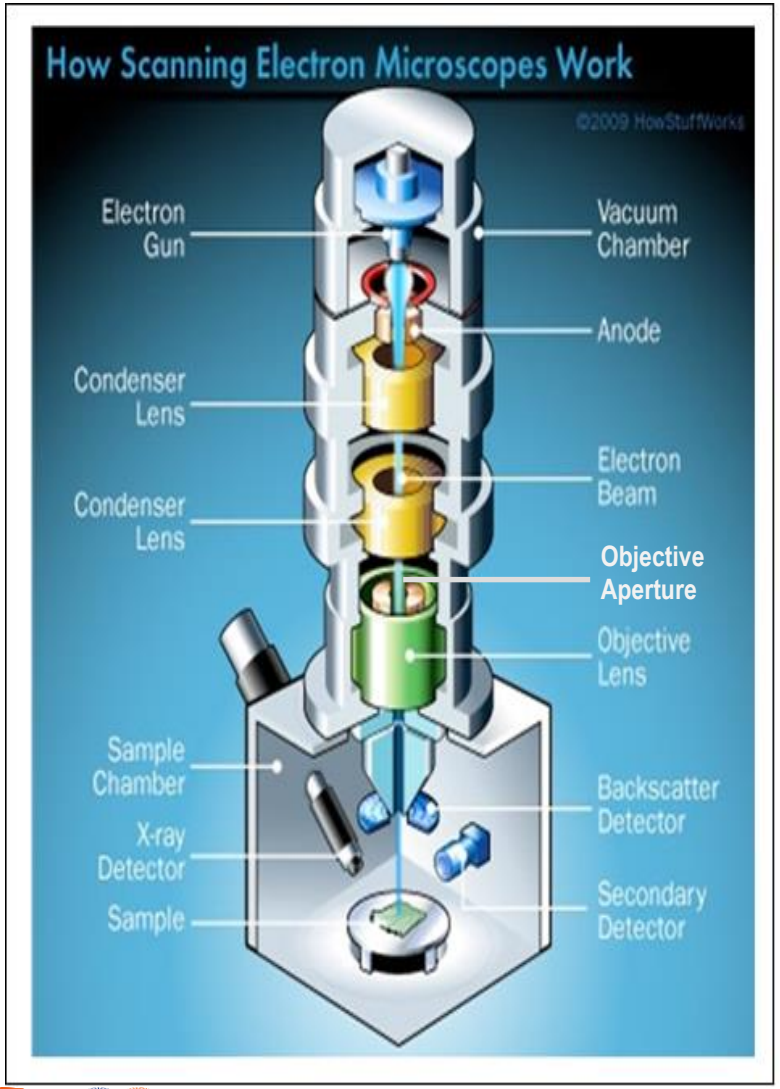
SCANNING ELECTRON MICROSCOPY

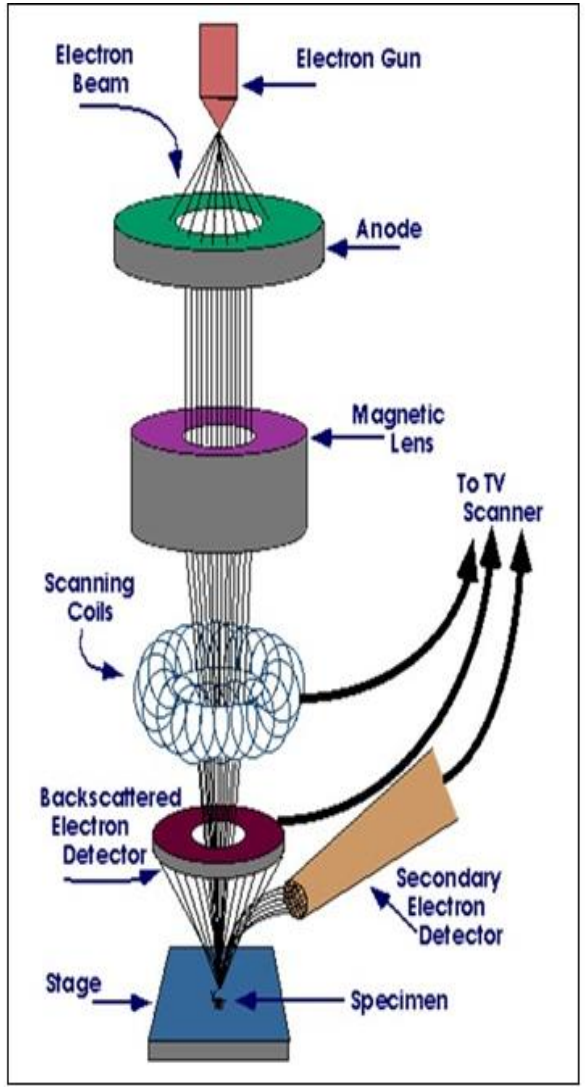
- A Scanning Electron Microscope is an instrument for observing and analyzing the surface microstructure of a bulk sample using a finely focused beam of energetic electrons.
- An electron-optical system is used to form the electron probe which may be scanned across the surface of the sample in a raster pattern.
- Various signals are generated through the interaction of this beam with the sample. These signals may be collected or analyzed with the application of appropriate detectors.
- For imaging, the signal amplitude obtained at each position in the raster pattern may be assembled to form an image.

APPLICATIONS:

- Surface topography / morphology
- Composition analysis
- Crystallography (electron diffraction and channeling techniques)
- Optical/Electronic properties (Cathodoluminescence, EBIC)
- Many other more specialized applications.

GENERALIZED CONSTRUCTION OF SEM





- Vacuum System
- Electron Source and Accelerating Voltage
- Electron Lenses (electromagnetic)
  - Condenser Lens(es)
  - Objective Lens
    - Stigmator Coils
- Beam Deflectors (electromagnetic)
  - Alignment
  - Scanning (raster)
- Objective Aperture
- Multi-Axis Specimen Stage
- Detectors
  - Imaging detectors
  - Analytical detectors
- Operating / Display Systems

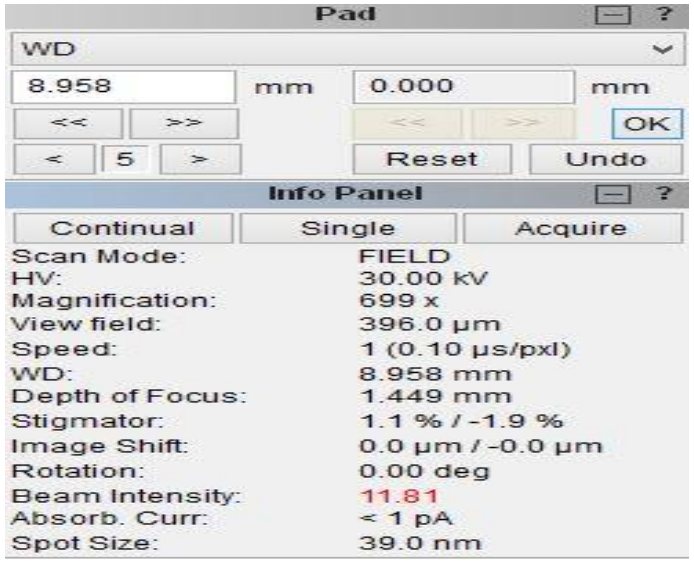
MAJOR PARAMETERS OF ELECTRON BEAM

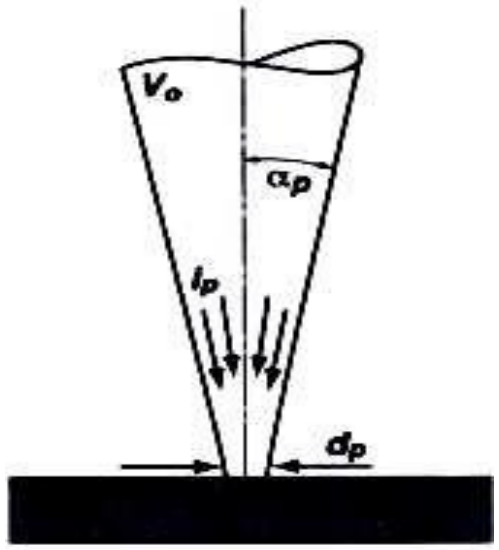
Four electron beam parameters define the probe:

- Probe diameter – d_p
- Probe current – I_p
- Probe convergence angle – α_p
- Accelerating Voltage – V_o

These interdependent parameters must be balanced by the operator to optimize the probe conditions depending on needs:

- Resolution
- Depth of Focus
- Image Quality (S/N ratio)
- Analytical Performance



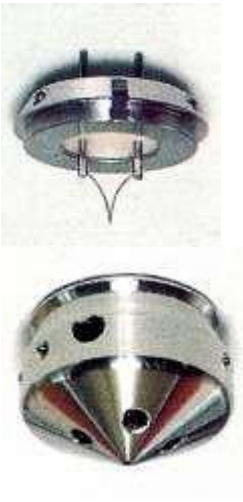


ELECTRON SOURCES – THERMIONIC SOURCES

1. Tungsten Filament
2. LaB_6
3. Cold Field Emission
   (Sharp Single Crystal (310) Tungsten Tip)

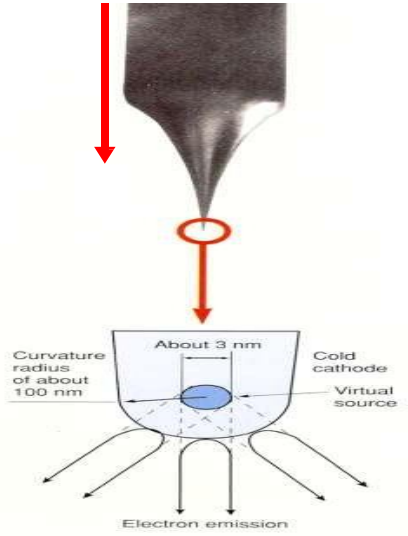
4. Thermal Field (Schottky) Emission
   (Sharp Single Crystal (100) Tungsten Tip with $Zr0_2$ Film)

Note: TESCAN FERA3 AND LYRA3 have High brightness Schottky Emitter



Tungsten Filament

LaB_6



Thermal Field (Schottky) Emission

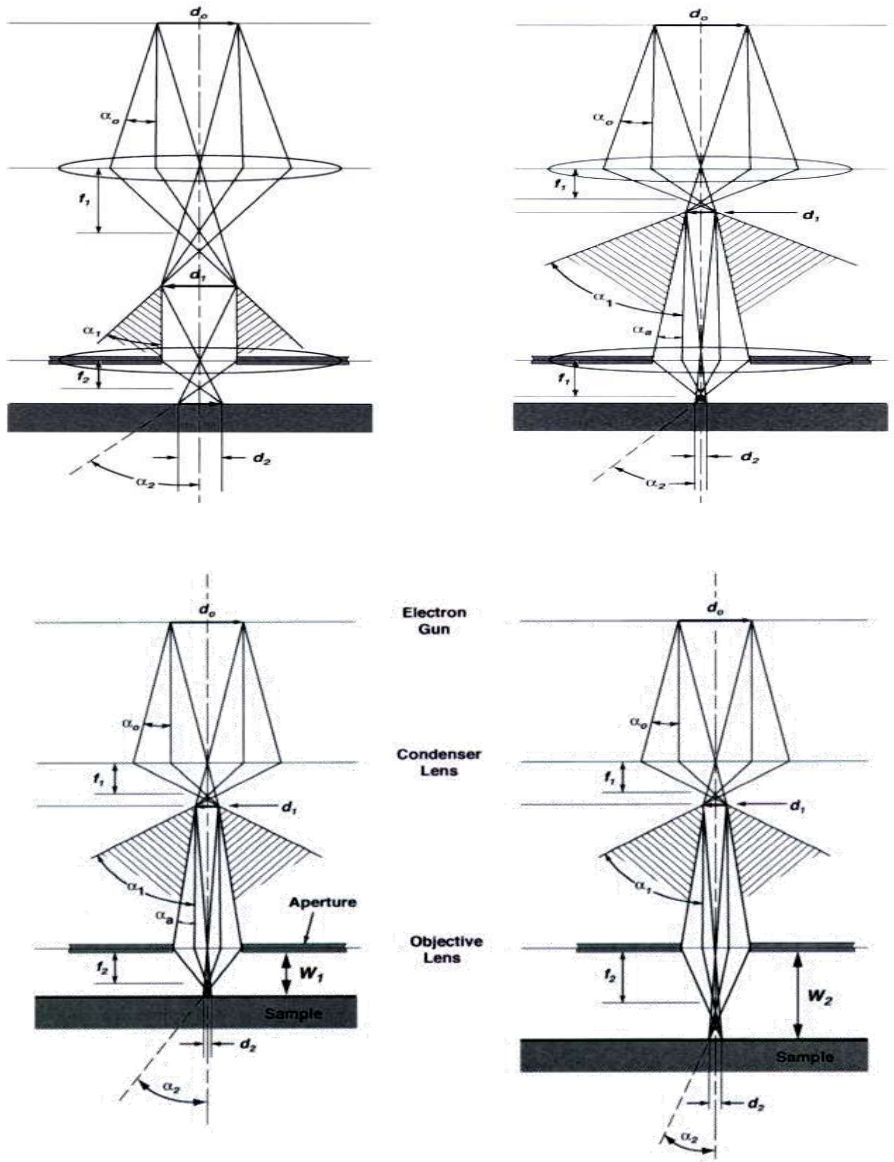
Comparison of Electron Sources

	TUNGSTEN	LAB6	SCHOTTKY	COLD-FIELD
Cost	$	$$	$$$	$$$
Effective Source Size(nm)	15000	5000	15	3
Brightness	10^5-10^6	10^6-10^7	>10^8	10^9
Energy Spread (eV)	1.0	1.0	0.5-1.0	0.3
Emission Current	<150uA	<100uA	<150uA	<20uA
Maximum Probe Current (SEM)	1000+nA	<1000nA	10-500nA	<2.0nA
SEM Resolution	3-4 nm	2-3 nm	<1-2 nm	<1-1.5 nm
Probe Current Stability (%hour)	<1	<2	<1	>10
Operating Temperature (K)	2800	1850	1800	300
Operating Vacuum (Pa)	<10^{-2}	<10^{-5}	<10^{-7}	<10^{-7}
Typical Service Life	100 hours	1000 hours	>>1 year	>>>1 year

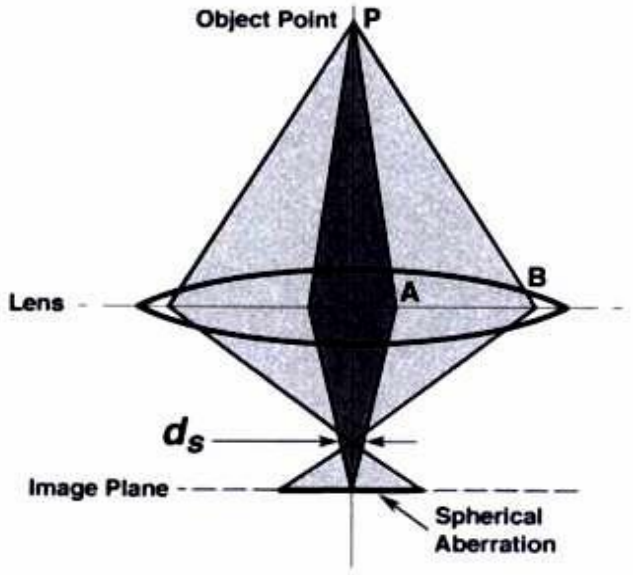
Lenses In Electron-Optical Column

Condenser Lens : De-magnify the beam extracted from the source to enable a small spot to be obtained on the sample . Multiple lenses may be used in the condenser lens system.

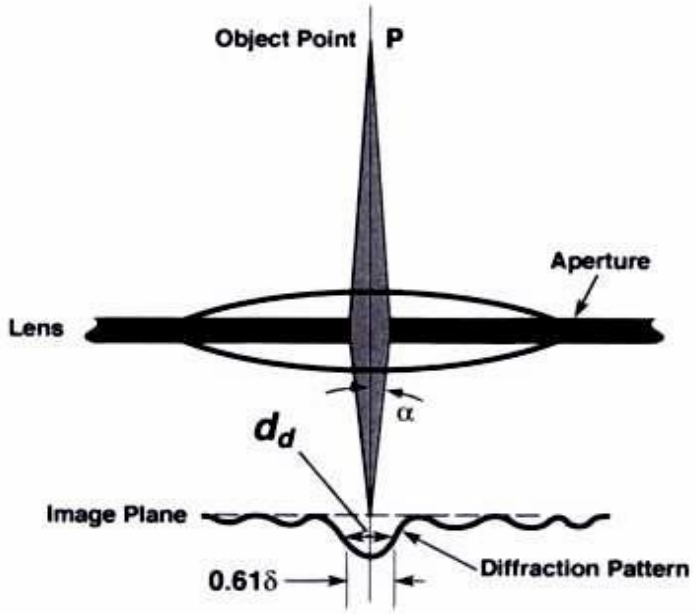
Objective Lens : Focus the electron beam on the specimen with minimal lens aberrations, astigmatism is corrected.



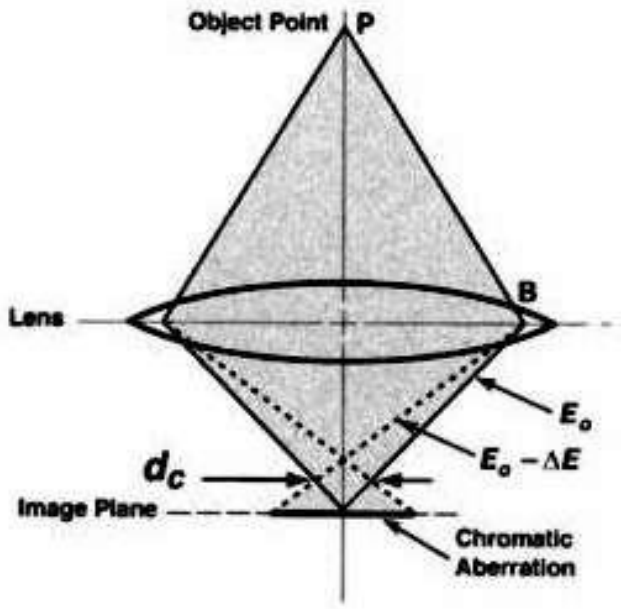
Limiting Parameters



Spherical Aberration



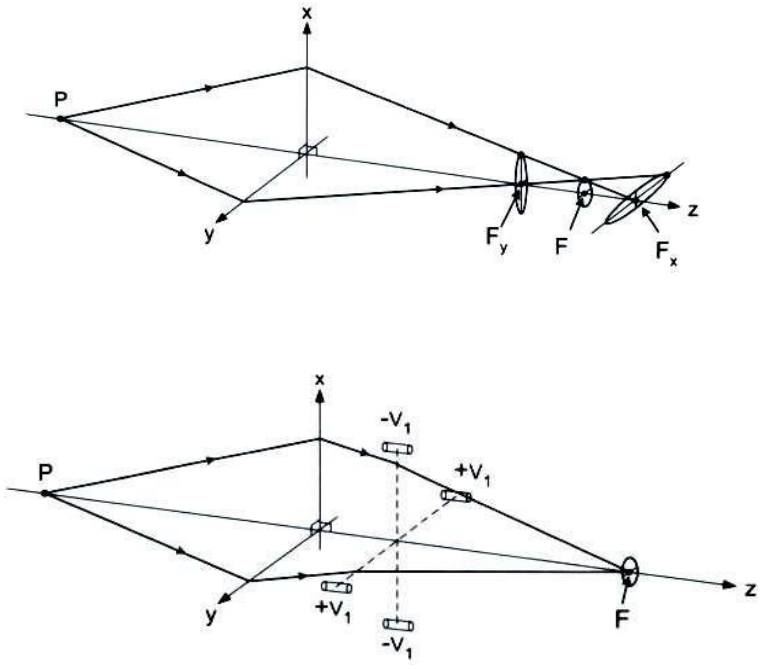
Aperture Diffraction

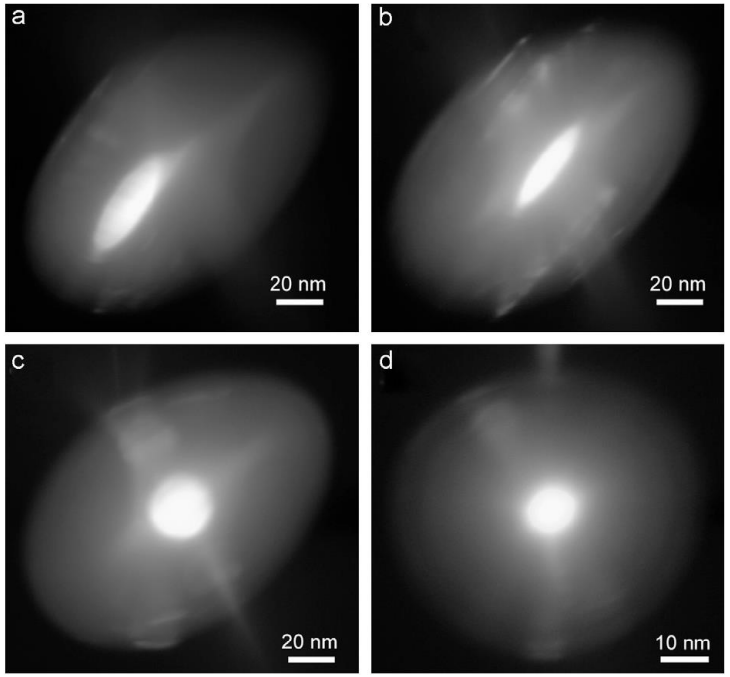


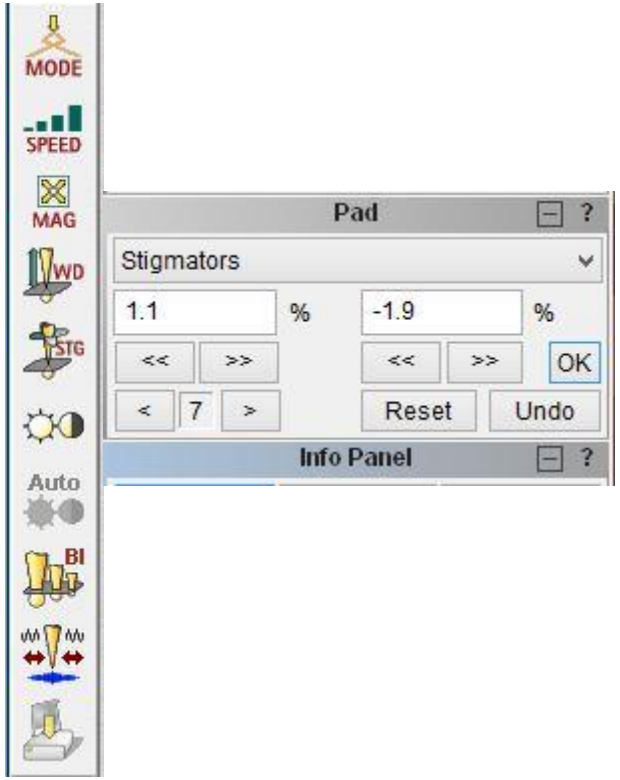
Chromatic Aberration

Astigmatism

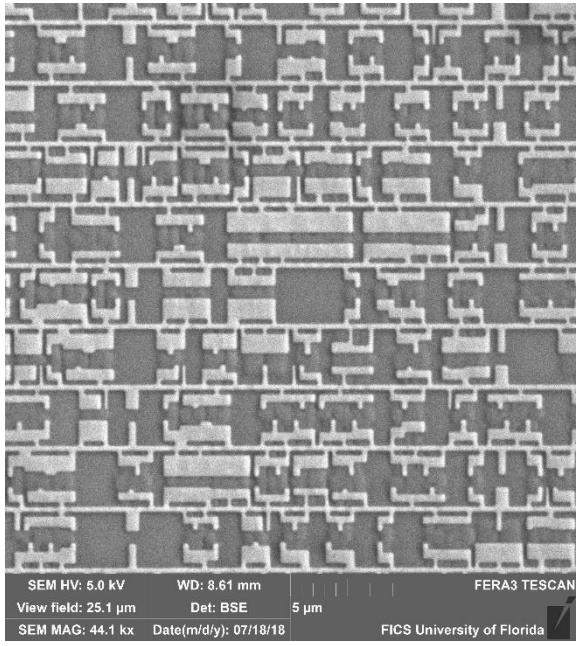
Astigmatism is caused by imperfections in the lens or other interference It can be corrected using additional elements called stigmators contained inside the objective lens.



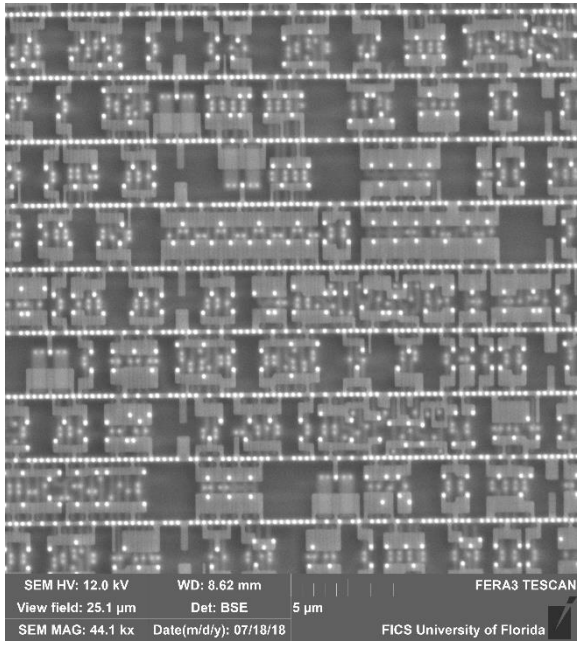




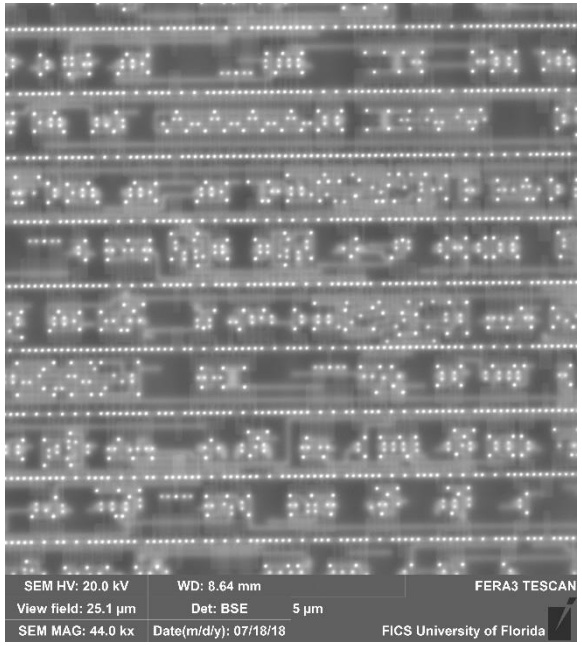
Effect of Beam Energy on SE Imaging



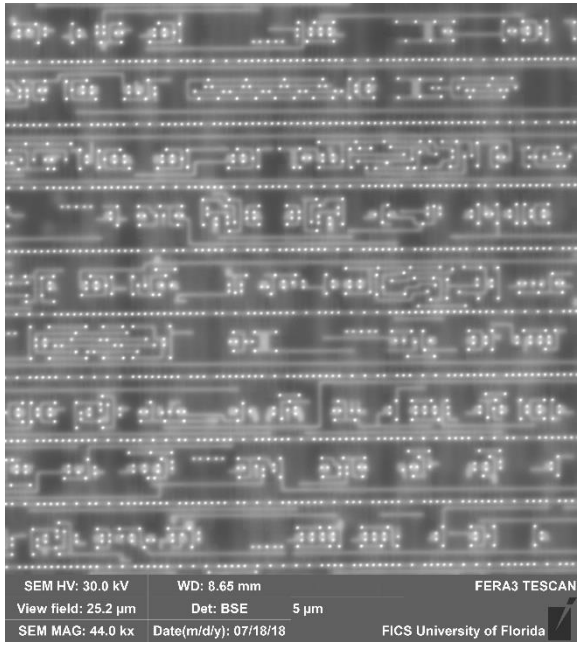
5KV



12 KV

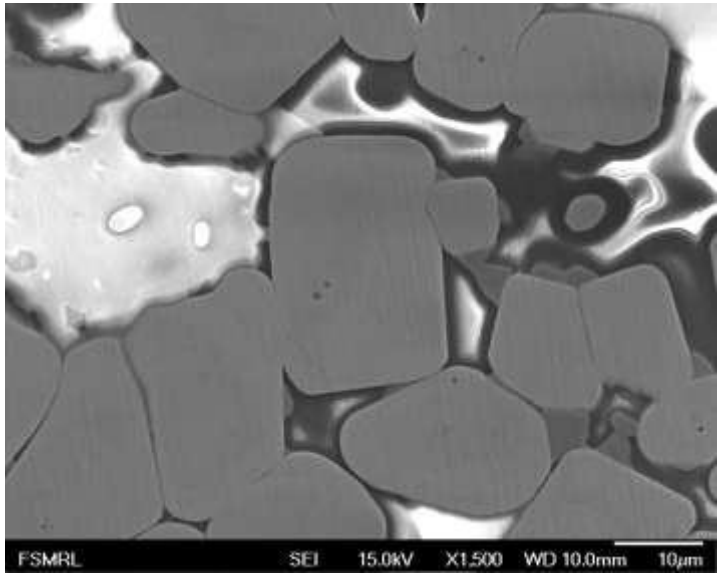


20 KV

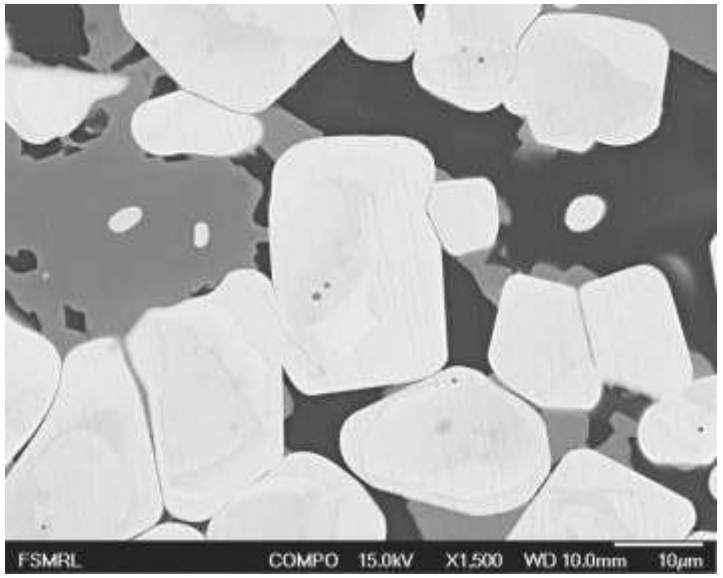


30 KV

BACKSCATTERED COMPOSITIONAL CONTRAST

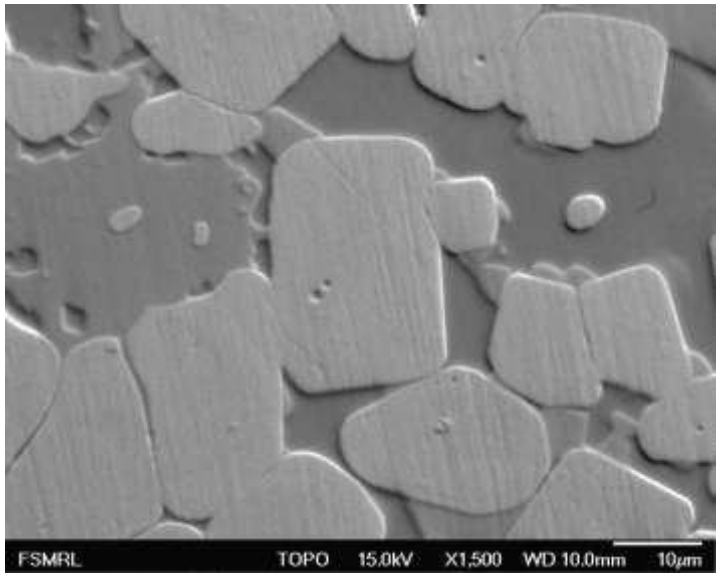


Secondary Electron Image



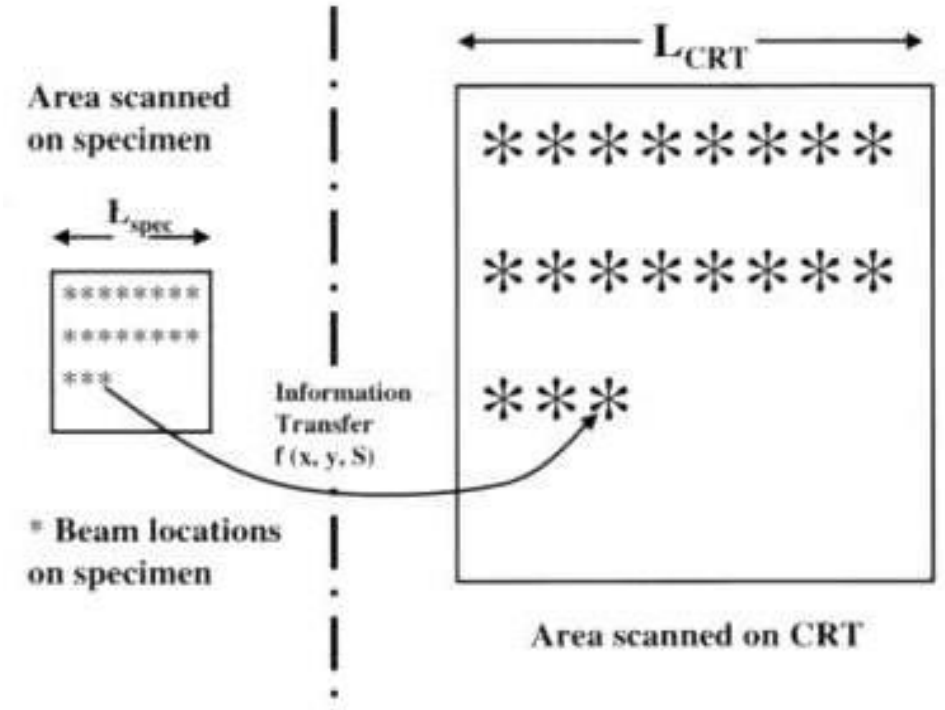
Backscattered Electron Image (Compositional)

Backscattered Electron Image (Topographical)



- Compositional mode imaging most useful on multi-phase samples.
- Sensitivity can be as low as 0.01 average Z differences.
- Flat-polished specimens preferable for best sensitivity in compositional mode.

SEQUENTIAL IMAGE ACQUISITION IN SEM



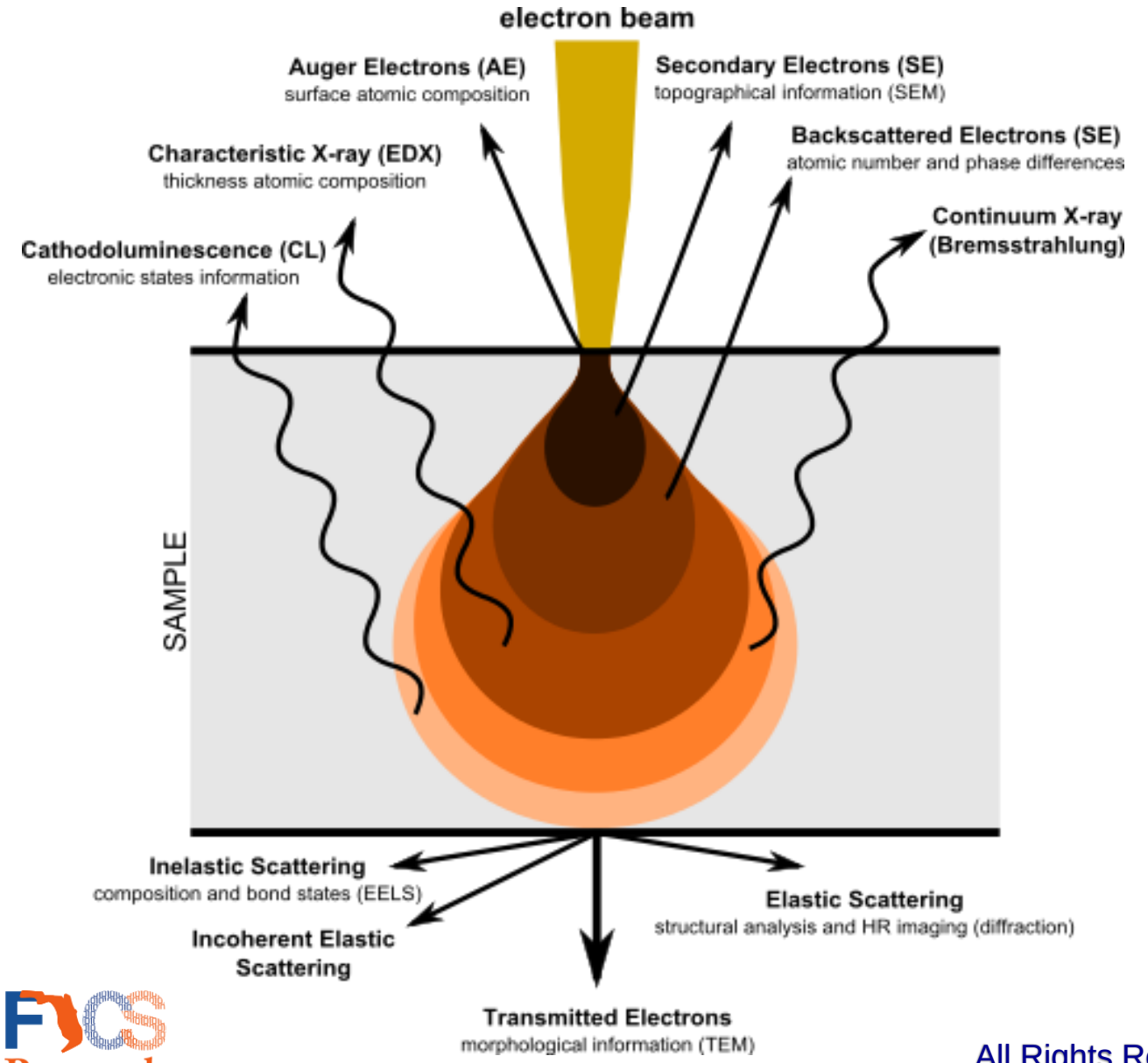
$M = L_{display}/L_{specimen}$

Caution:

Instrument magnification value is based on reference image size which may vary

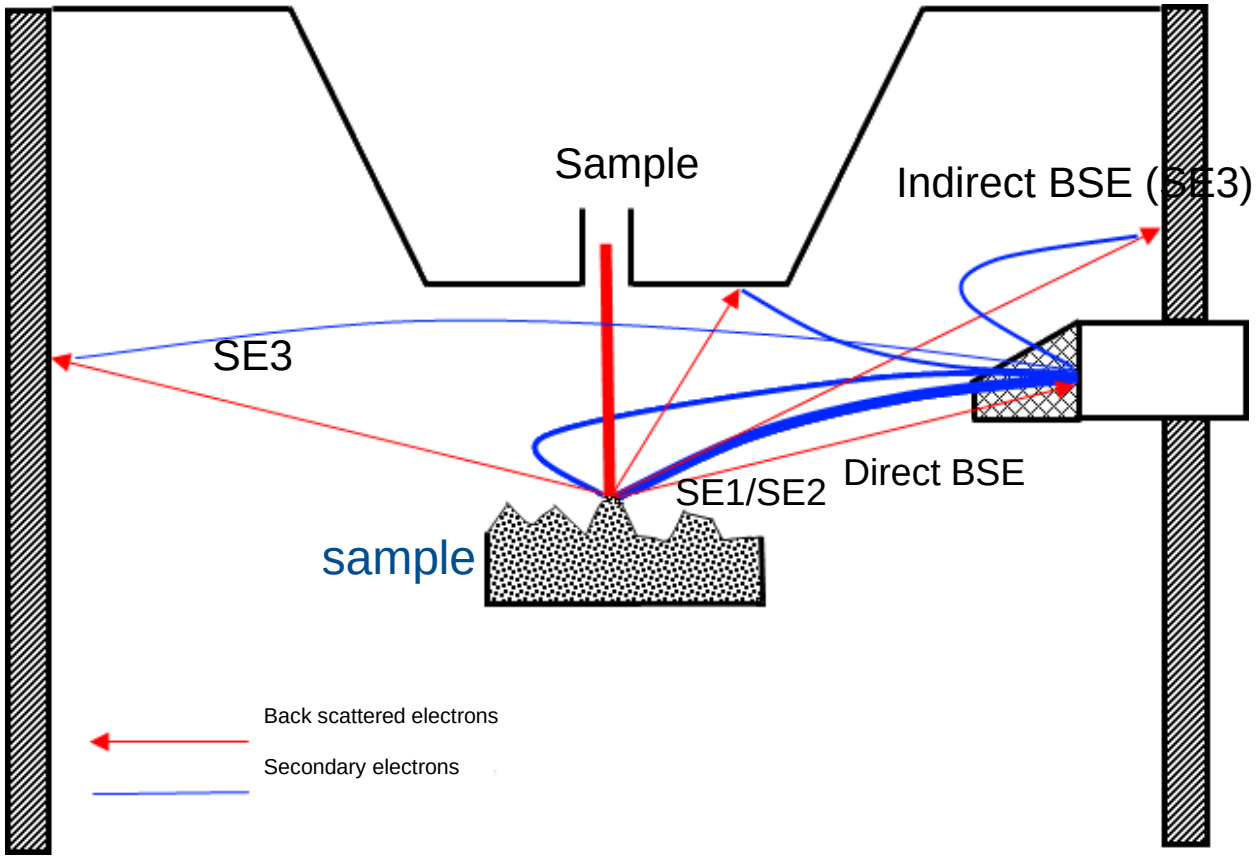
- The scan of the electron beam and the digitization of the image pixel value are synchronized with intensity proportional to the collected signal.
- Typically electrons emitted from the sample are detected to assemble the image.
- Magnification is given by the ratio of the length of the line on display device to length scanned on the real sample.

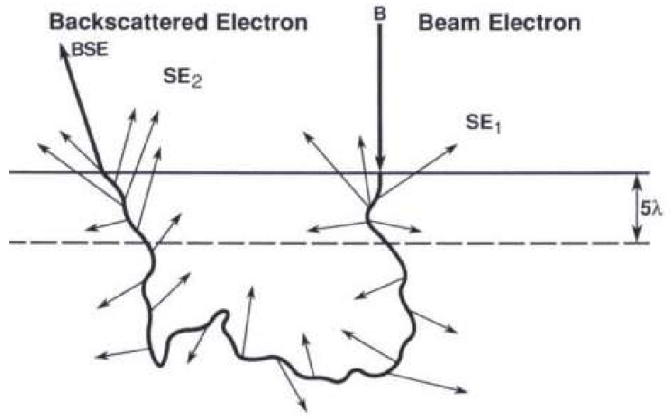
ELECTRON BEAM / SPECIMEN INTERACTIONS



- Energy-Dispersive Spectroscopy (EDS) /(EDX)– solid state detector simultaneously measures all energies of X-ray photons.
- Wavelength Dispersive Spectroscopy (WDS) – sequentially measures intensity *vs* X-ray wavelength (energy). Superior energy resolution and detection limits (P/B ratio).
- Cathode luminescence (CL) – optical emission spectrometer and imaging system for 300-1,700nm. Liquid He cooled stage module.

Sources of electrons detected by E-T Detector



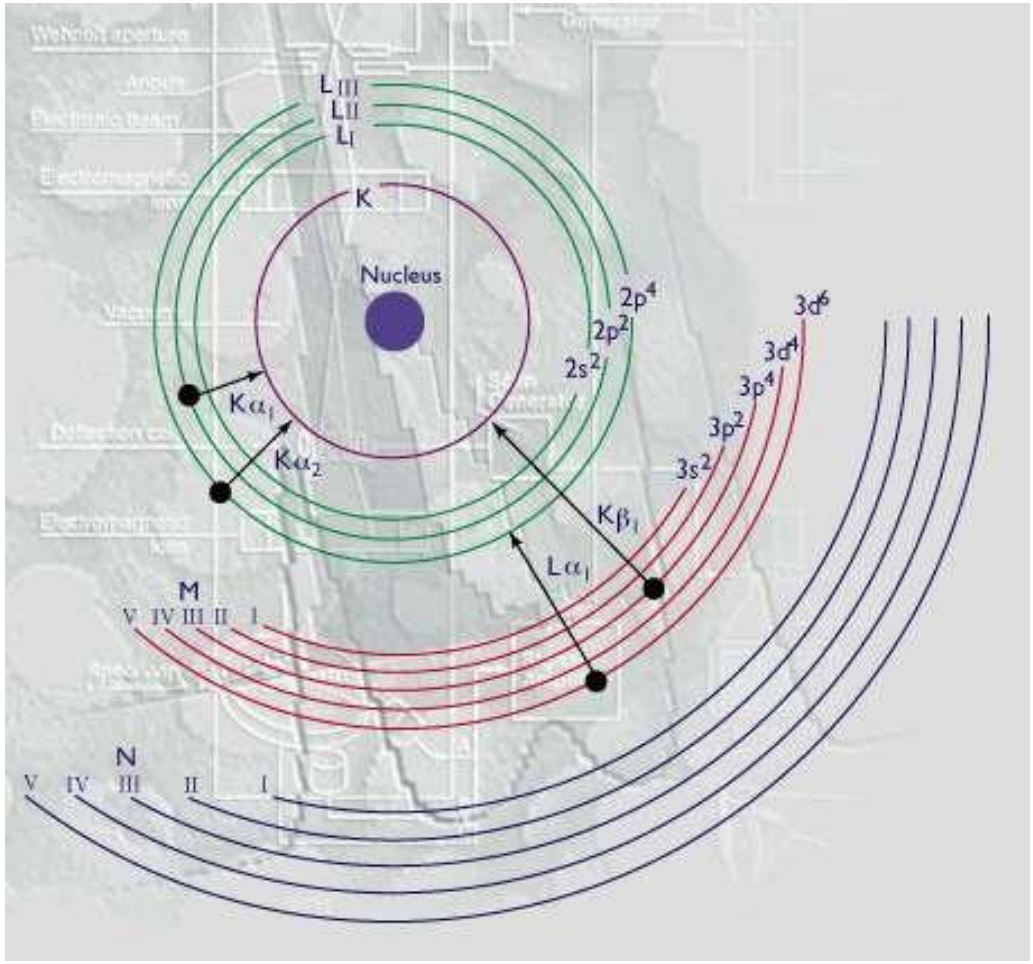


Geometrical Effects

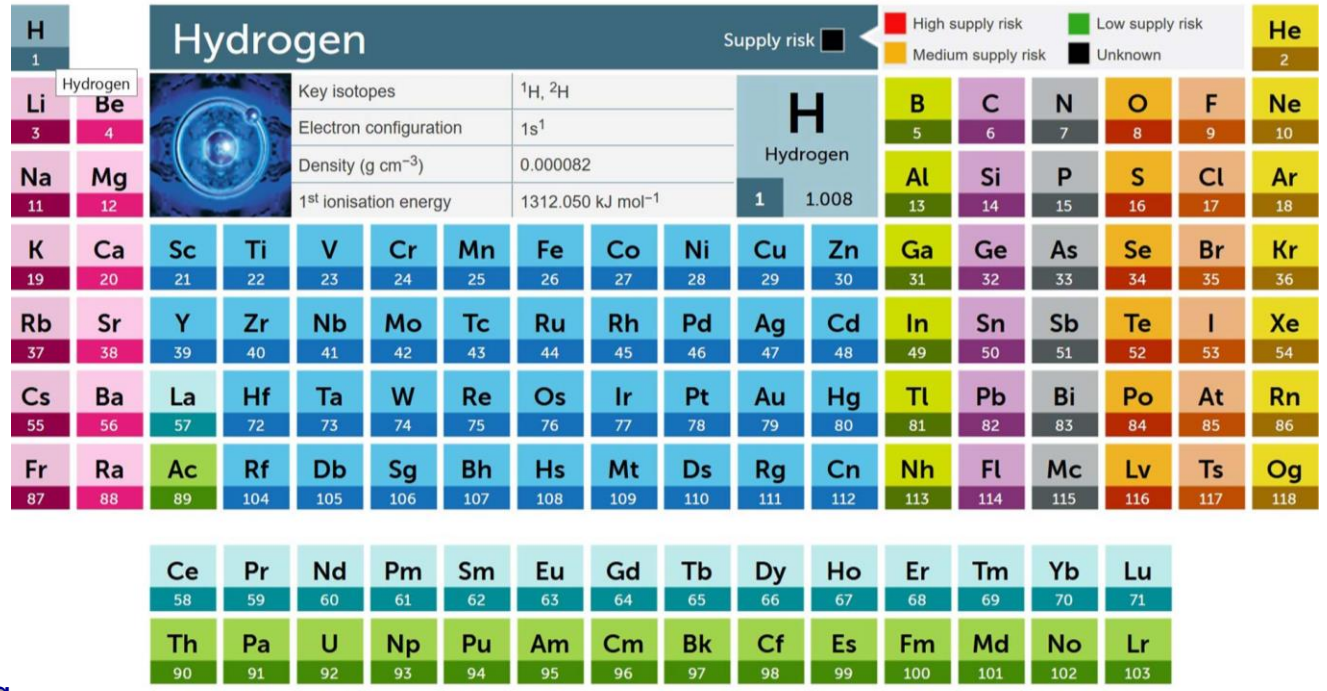
- Direct BSE's need "line of sight" trajectory.
- SE detection efficiency may vary with topography and sample surface / detector geometry.

Backscattered electrons are also directly and indirectly detected **(image is not pure SE)**

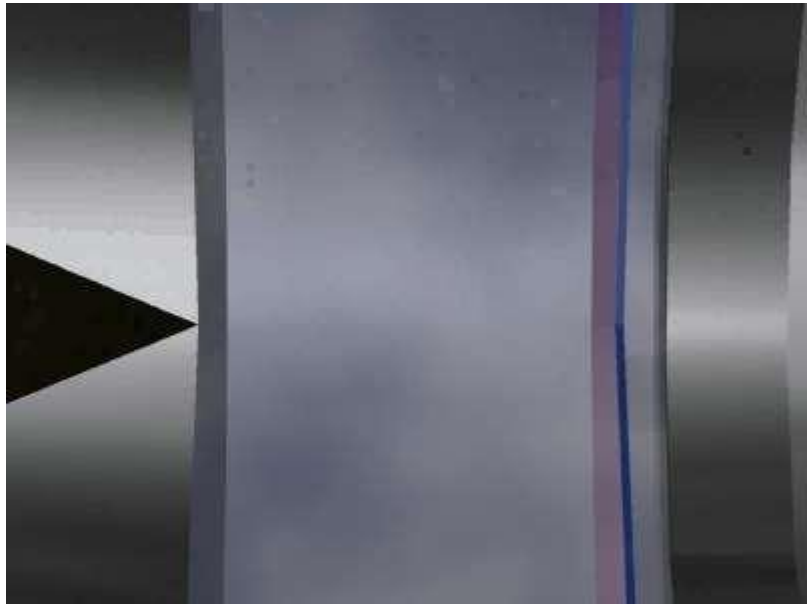
Characteristic X-Ray Generation

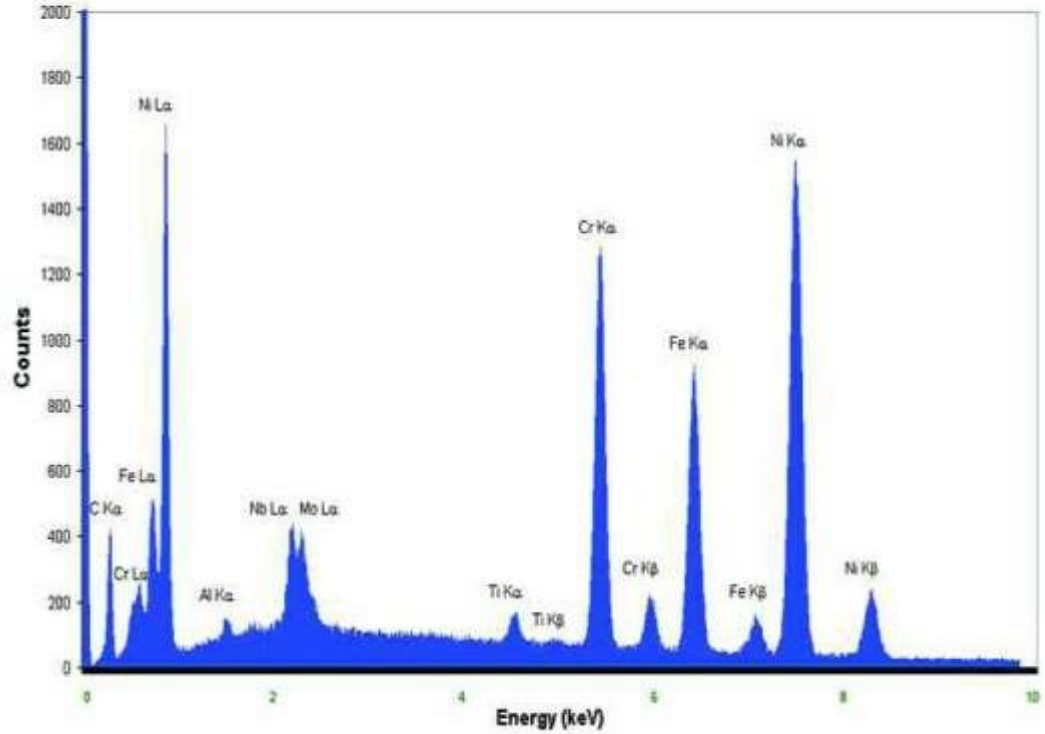


- A scattering event kicks out an electron from K,L,M, or N shell of atom in specimen.
- An electron from an outer shell falls to fill in the vacancy.
- Energy difference results in release of an x-ray of characteristic energy/wavelength or an auger electron.



Mechanism of X-Ray Energy Determination

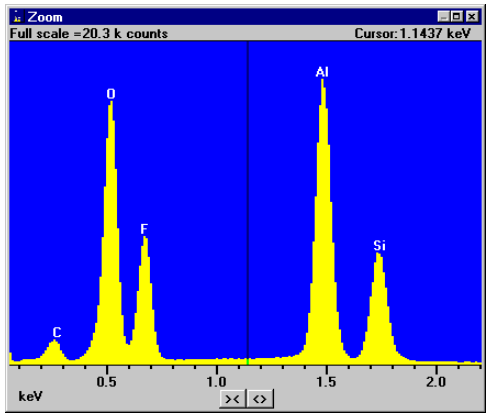




- X-ray loses energy through inelastic scattering events creating electron / hole pairs.
- High voltage bias keeps generated pairs from re-combining.
- Charge sensitive amplifier “counts” pairs generated by X-ray.
- Spectrometer calibration effectively multiplies by energy/pair (3.8 eV) to determine X-ray energy.

X-Ray EDS Microanalysis in the SEM

- Fast Parallel Detection
- Qualitative elemental analysis
  - From Beryllium up on periodic table
  - Sensitivities to <0.1 wt.% depending on matrix and composition.
- Quantitative analysis.
- Digital elemental distribution imaging and line-scans, full spectrum imaging.
- Analysis of small volumes, from order of um^3 to << 1 um^3 depending on accelerating voltage, element analyzed, and matrix.

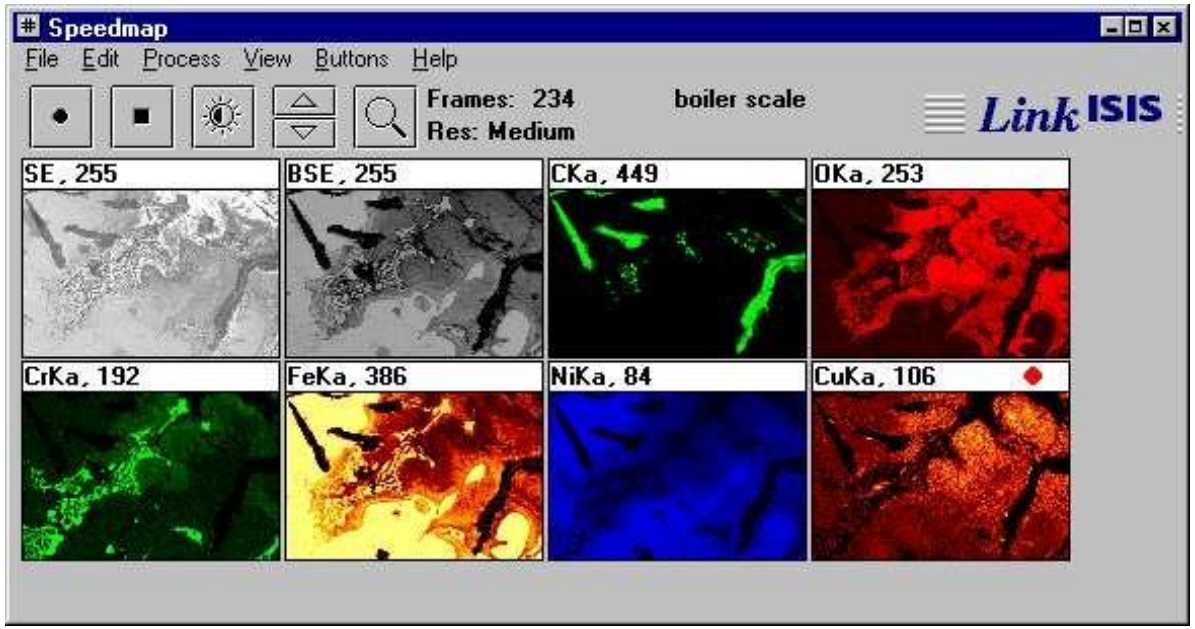


SEMQuant Results

File Edit Display Options

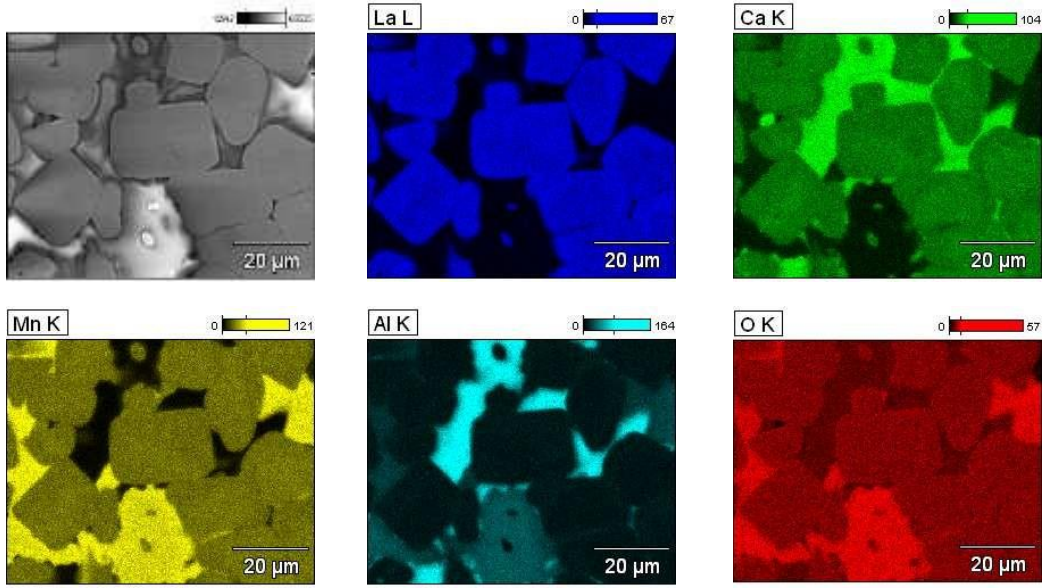
Elmt	Spect. Type	Inten. Corrn.	Std Corrn.	Element %	Sigma %
Mg K	ED	0.857	1.13	6.27	0.12
Al K	ED	0.872	1.32	11.24	0.14
Si K	ED	0.855	1.57	18.49	0.15
Ca K	ED	1.003	1.47	3.05	0.08
Mn K	ED	0.825	1.21	0.35	0.10
Fe K	ED	0.838	1.15	16.85	0.22
O				43.75	0.23
Total				100.00	

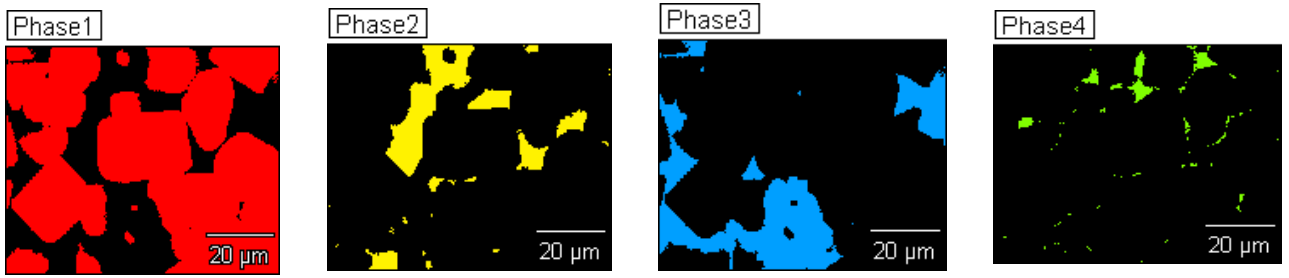
* = <2 Sigma

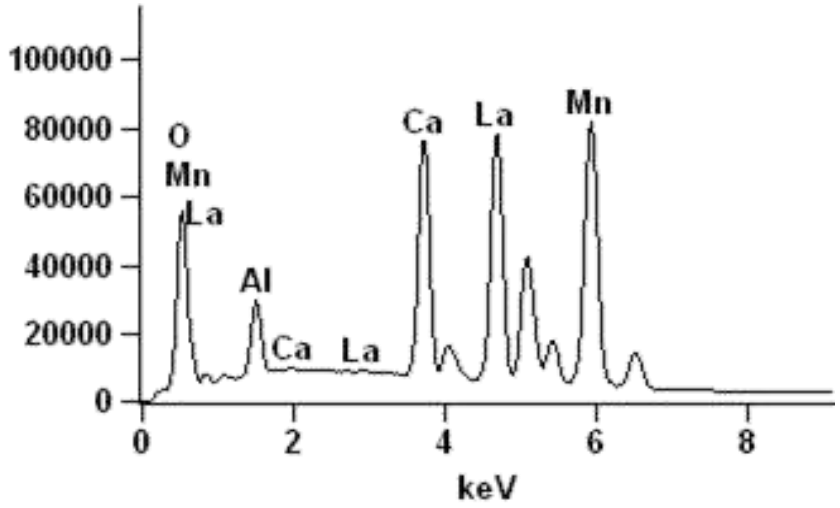


EDS FULL SPECTRUM IMAGING

In this case ,a full X-ray spectrum collected for each pixel. X-ray elemental maps, phase maps, spectra, and quantitative analysis extracted from full spectrum images.



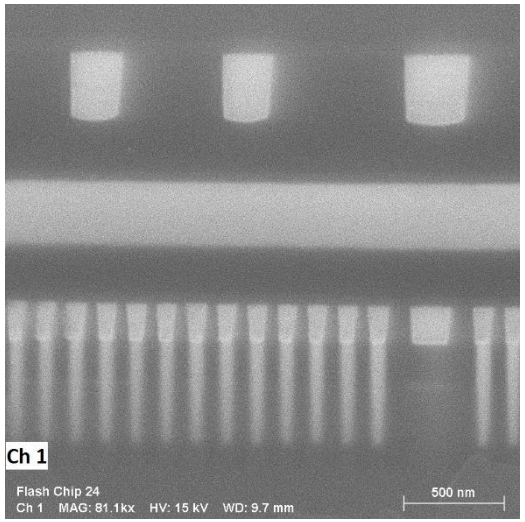


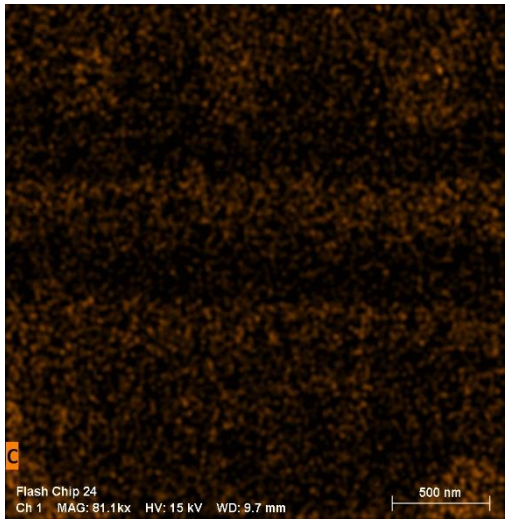


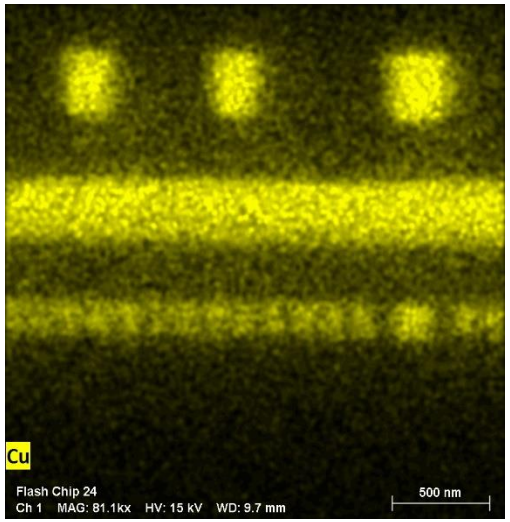
Cumulative Spectra and Quantitative Analysis for each extracted phase (ex. Phase 1)

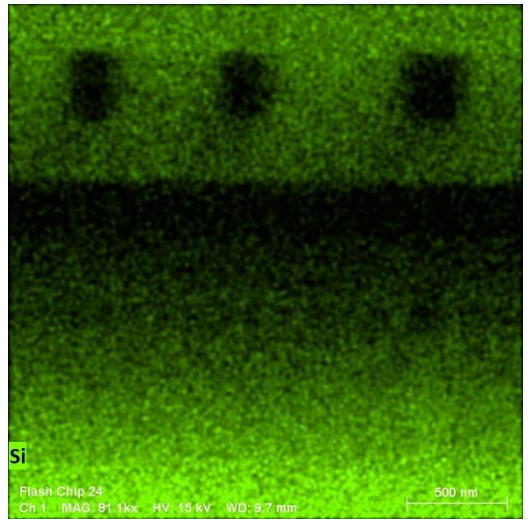
Element Line	Weight %	Weight %Error
O K	22.13 Stoichiometry	---
Al K	3.81	+/- 0.02
Ca K	10.26	+/- 0.04
Mn K	30.57	+/- 0.07
La L	33.23	+/- 0.09

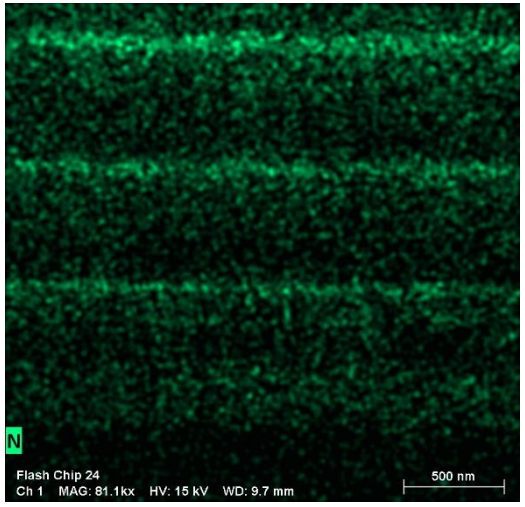
EDS FULL SPECTRUM IMAGING OF FLASH CHIP

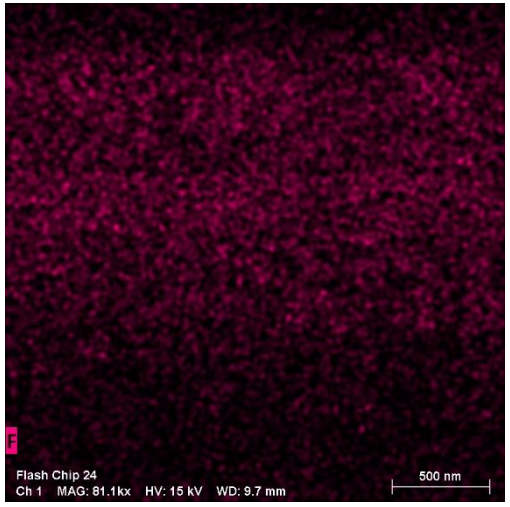


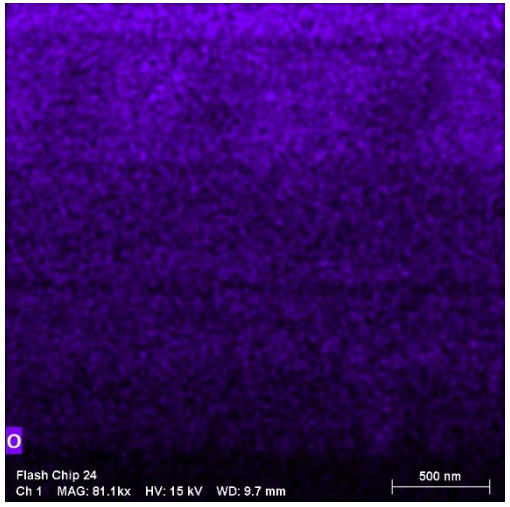


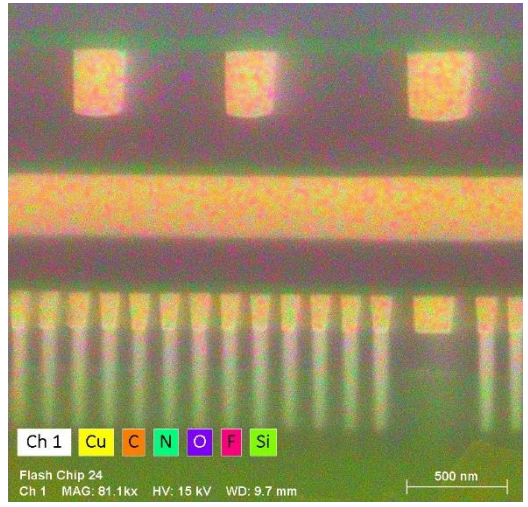












Different Types Of SEM

- Conventional (High Vacuum) SEM: It requires a dry, conductive sample and the sample must be able to withstand a high vacuum. This type of machine is used for routine imaging, using either secondary electrons(SE) or backscattered electrons(BSE).

- Variable Pressure or Low Vacuum SEM :This type of machine is basically like a conventional SEM but has the advantage in low vacuum (LV) mode that the pressure can be adjusted in the sample chamber until the artefact of "electron charging" is removed from images.

- CRYO-SEM:A Cyro-SEM is a conventional SEM that has been fitted with specific equipment that allows samples to be viewed in the frozen state. This is particularly useful for directly viewing hydrated (wet) samples, suspensions, pharmaceuticals and nanoparticles.

- Environmental SEM :This machine is designed to view a sample in its natural state, without the need for desiccation. Sample temperature and specimen chamber vapor pressure can both be controlled, allowing samples to be heated, cooled, wetted or dried.

- Focused Ion- Beam: This technology involves using an ion beam (typically gallium ions) directed onto a hard sample. The beam is focused to an extremely fine probe size(<10 nm) onto the surface of a specimen. The sample can be sectioned or shaped with the ion beam while it is being monitored by SEM.

- Electron Beam (E-BEAM) Lithography: EBL is a maskless lithography technique used for patterning of computer generated layout structures on photoresists on Si wafers. Upon irradiation of focused electron beam, electron-sensitive resists undergo chain-scission or crosslinking, resulting in solubility switch of materials during the subsequent development process.

Companies / Vendors of Scanning Electron Microscope

1.TESCAN
2.ZEISS
3.FEI COMPANY
4.HITACHI
5.Raith GmbH
6.JEOL