Frederick Seitz Materials Research Laboratory – University of Illinois at Urbana-Champaign

6th Advanced Materials Characterization Workshop

June 6 and 7, 2012

Scanning Electron Microscopy (SEM) and Focused Ion Beams (FIB) in Materials Research

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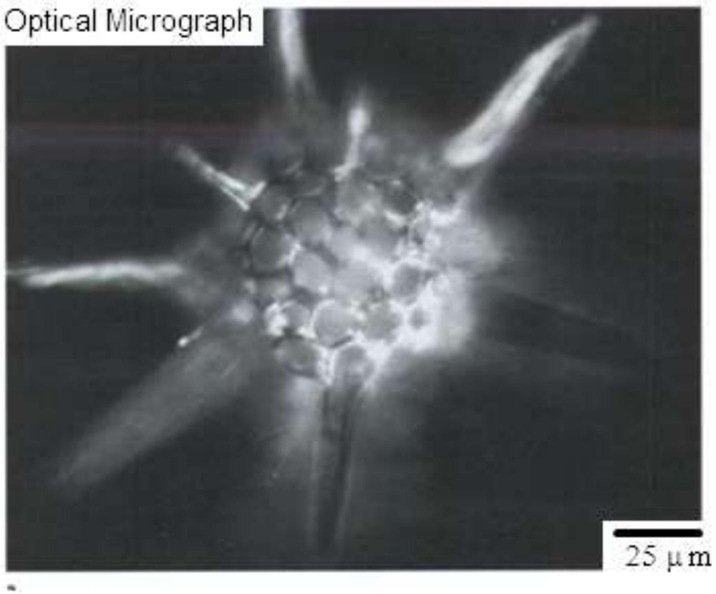
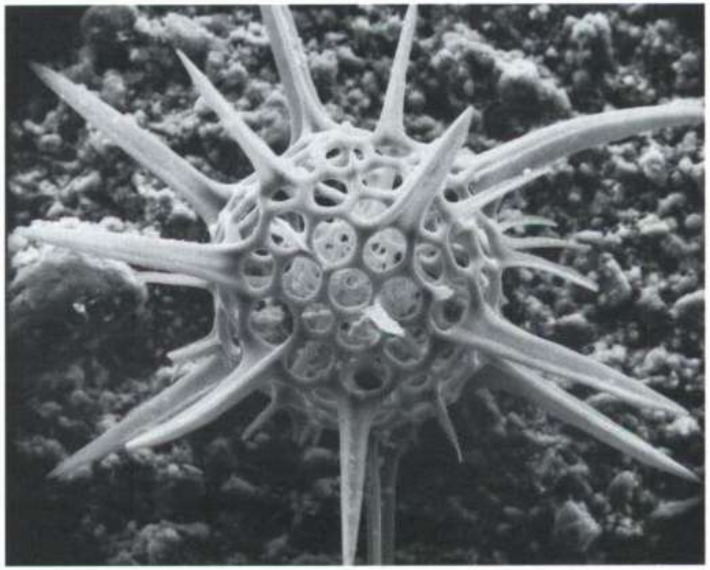
University of Illinois at Urbana-Champaign

Comparison to Optical Microscopy



Optical



SEM (secondary electron)

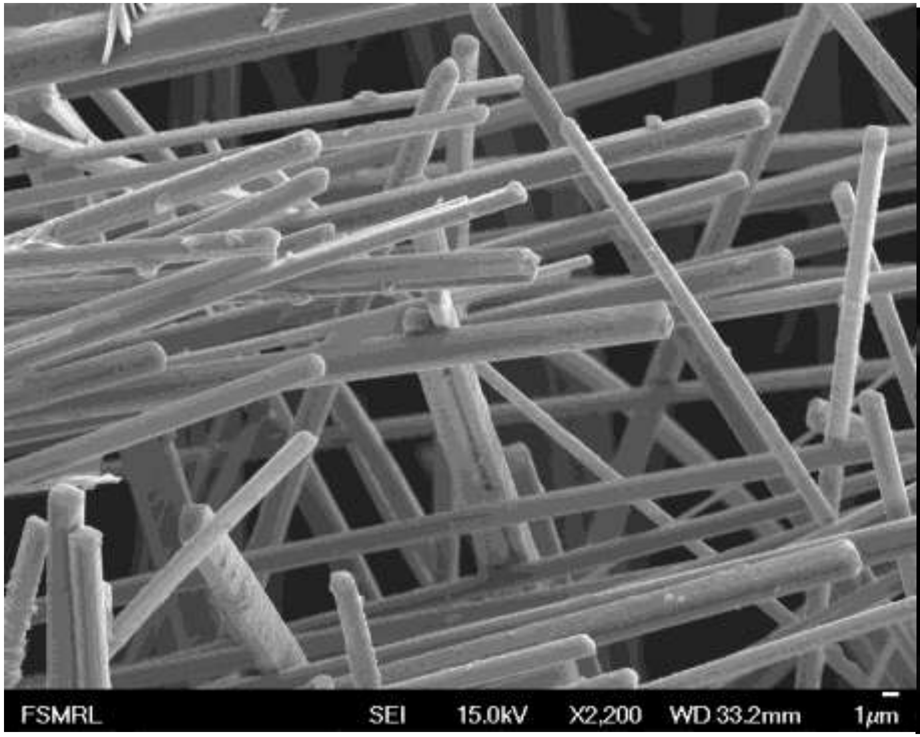
The higher resolution and depth of focus available with the SEM are clearly observed, The SEM also provides a very wide, easily adjustable range of magnifications. For most imaging applications minimal or no sample preparation is required.

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.

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

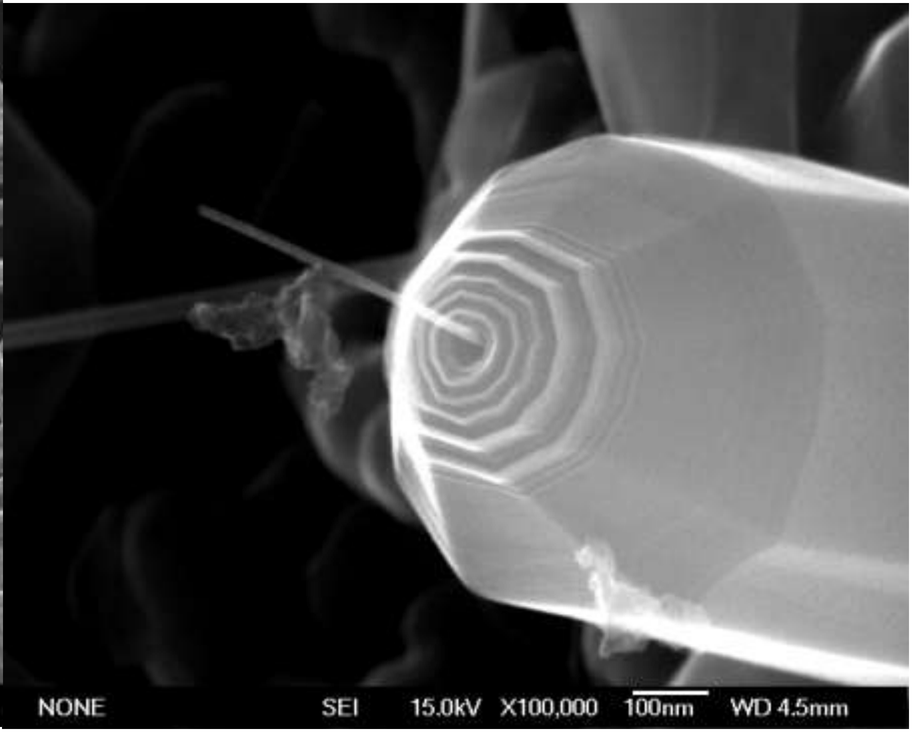
Exceptionally High depth of Focus

2,200X original magnification



“rat’s hair” psilomolene (Mn, Ba oxide natural mineral sample)

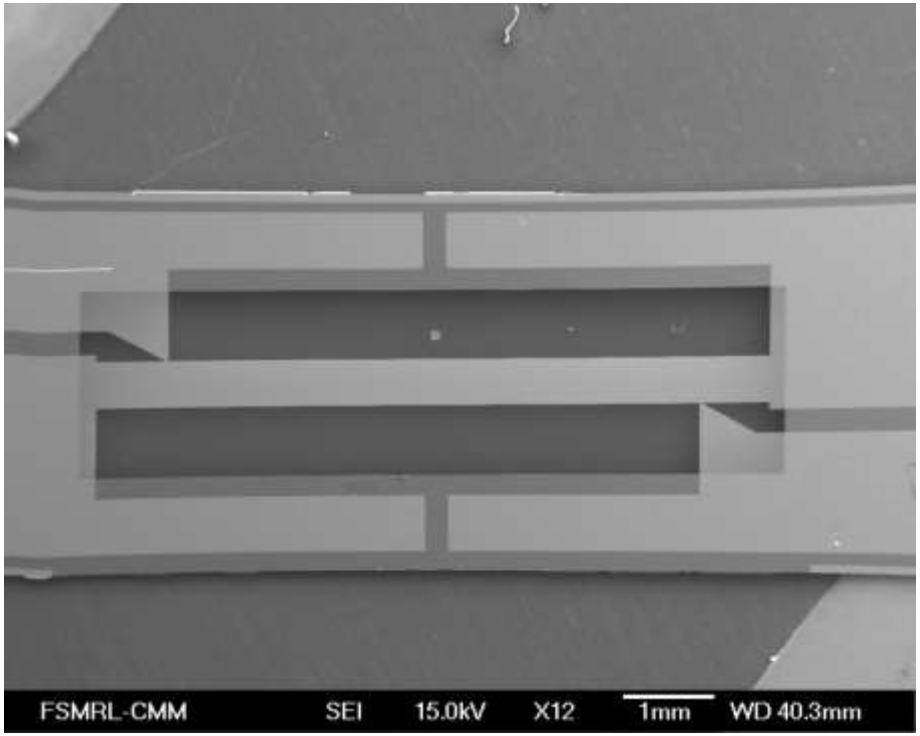
100,000X original magnification



Carbon Nanotube

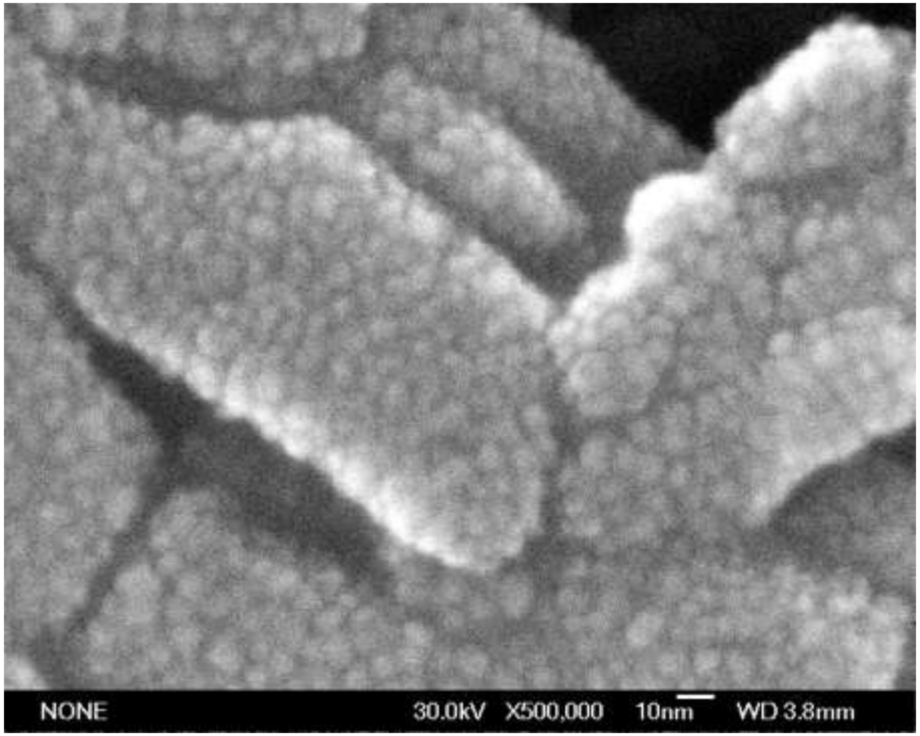
Extremely Wide Range of Magnifications

12X original magnification



Miniature Sensor Device - Calorimeter

500,000X original magnification



Sputtered Au-Pd on Magnetic Tape

What does the SEM do?

Many Applications:

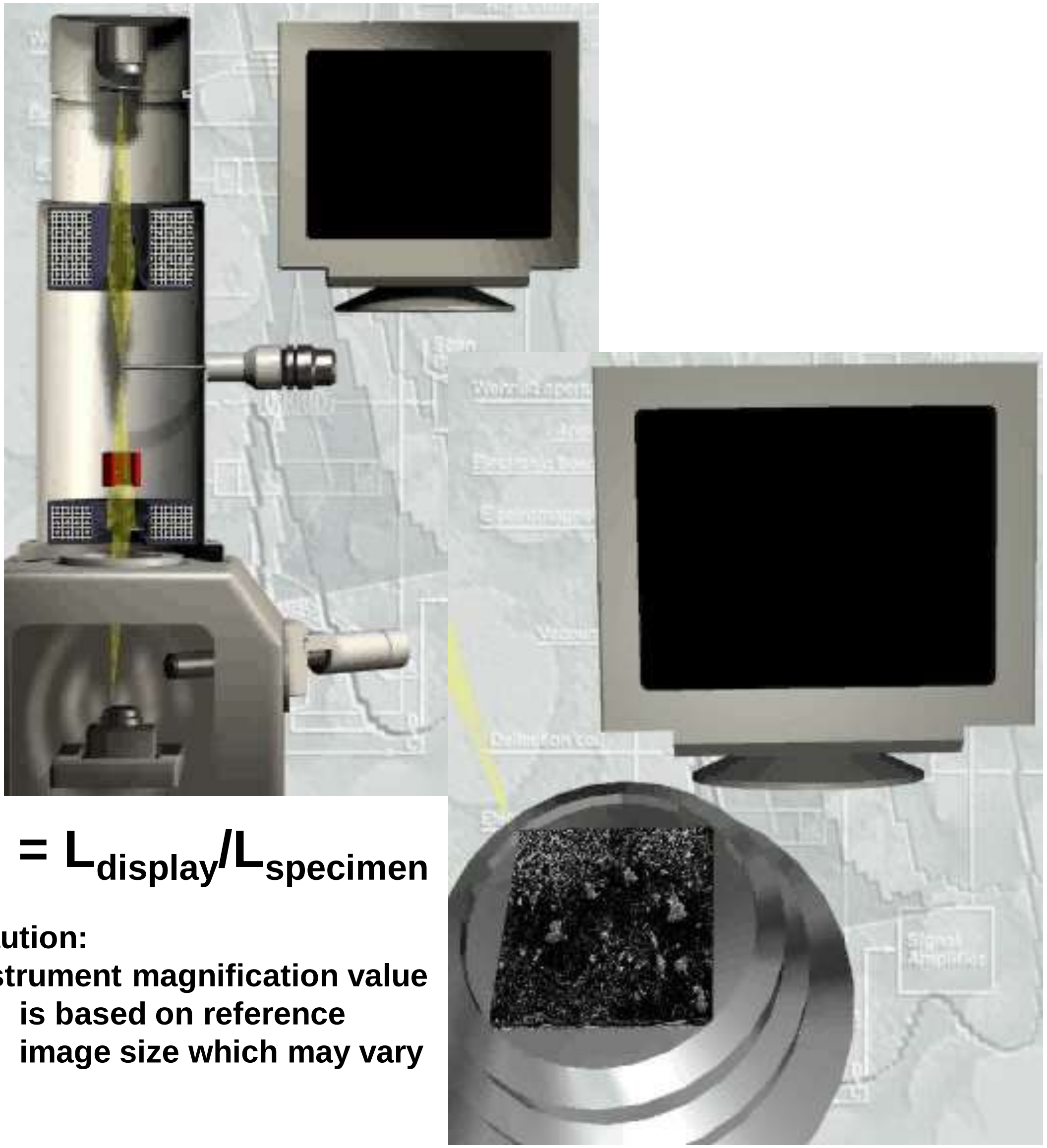
One of most widely employed microscopy techniques other than optical microscopy.

<u>Surface topography / morphology</u>
<u>Composition analysis</u>
Crystallography (electron diffraction and channeling techniques)
Optical/Electronic properties (cathodoluminescence, EBIC)
Many other more specialized applications

- A Scanning Electron Microscope is an instrument for observing and analyzing the surface microstructure of a bulk sample using a <u>finely focused beam</u> 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.

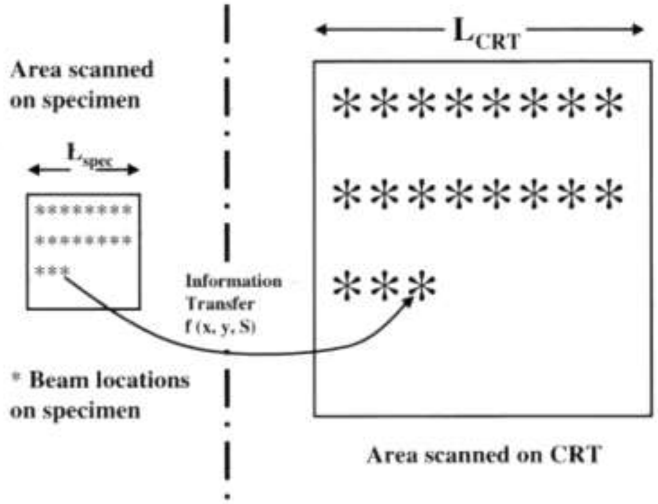
Animation from *A Guide to X-Ray Microanalysis*, Oxford Microanalytical Instruments

Sequential Image Acquisition in SEM



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

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

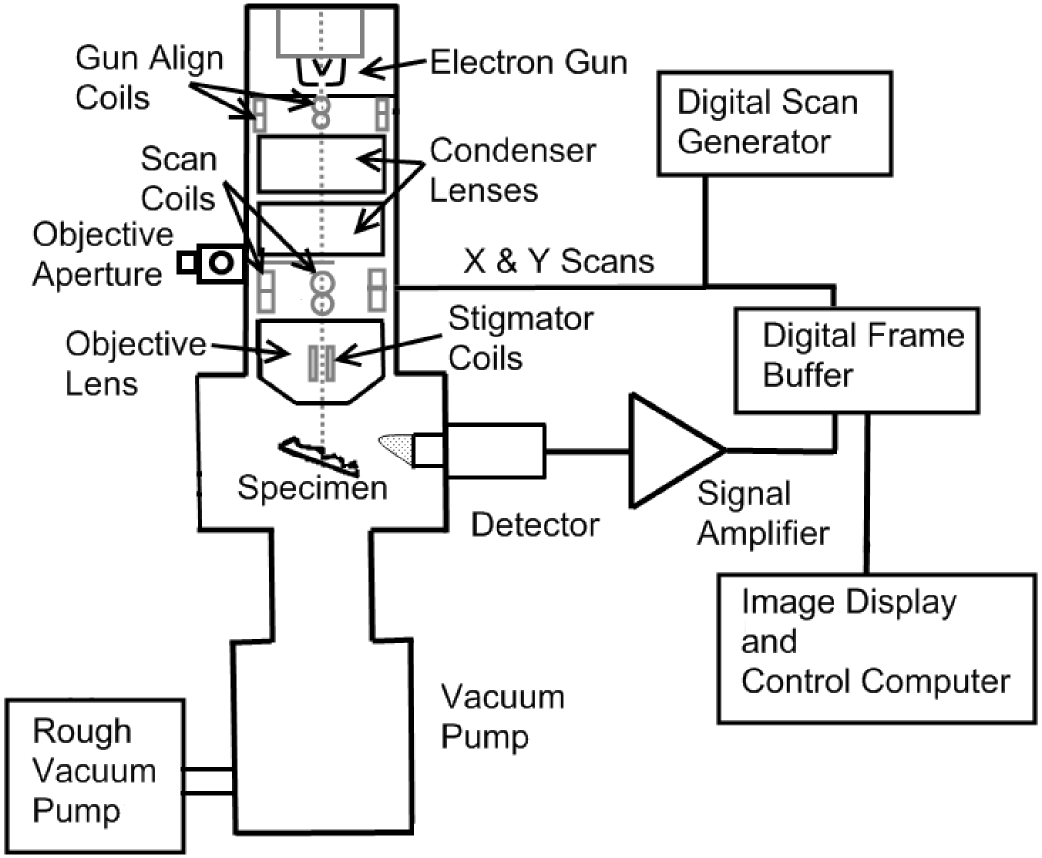


- **The scan of the electron beam and the digitization of the image pixel value are synchronized with intensity proportional to the collected signal.**
- **Typically electrons emitted from the sample are detected to assemble the image.**
- **Magnification is given by the ratio of the length of the line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

Animations from, *The Oxford Guide to X-Ray Microanalysis*, Oxford Instruments Microanalysis Group

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

Generalized Construction of an 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

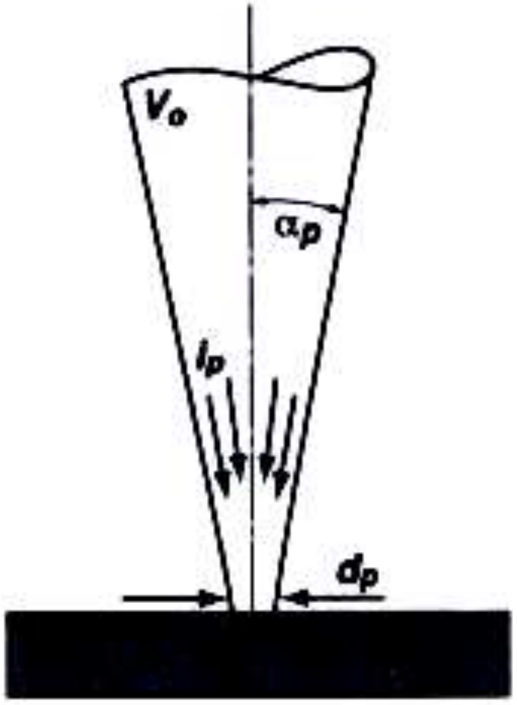
Major Electron Beam Parameters

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 optical brightness, β, of the probe is essentially equal to the brightness of the source, thus is a very important electron source parameter.

$$\beta = \frac{\text{current}}{\text{area} \cdot \text{solid angle}} = \frac{i_p}{\left(\frac{\pi d_p^2}{4}\right) \cdot \pi \alpha_p^2} = \frac{4 i_p}{\pi^2 d_p^2 \alpha_p^2}.$$

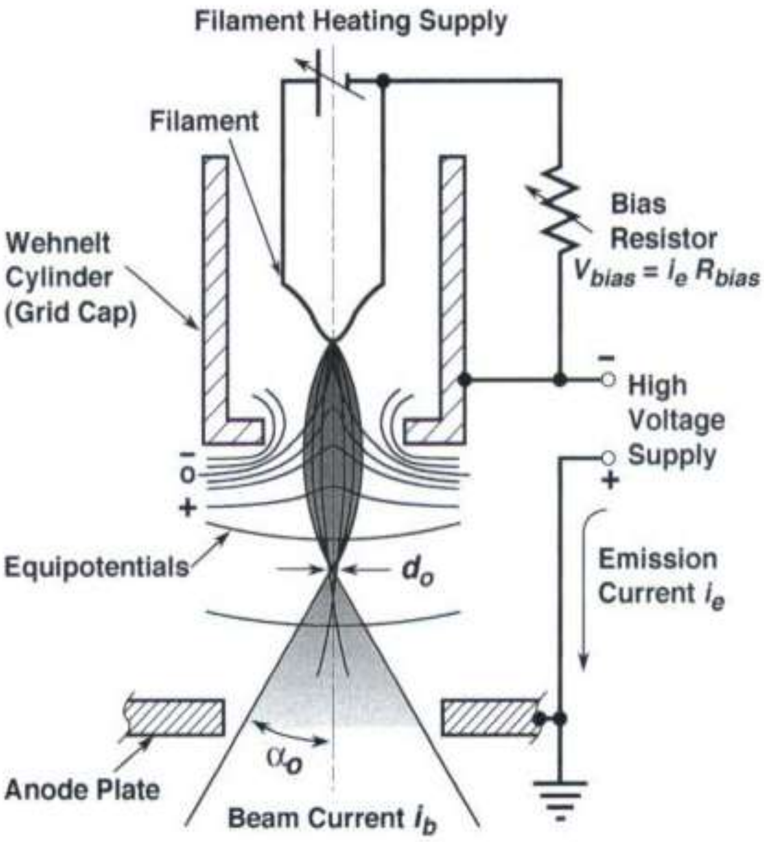
From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press

Electron Sources - Thermionic Sources

Tungsten Filament

LaB$_6$

http://www.a-p-tech.com/lab6.htm



Schematic of a generalized thermionic electron source for electron microscopy.

Major Advantages:

- **Very high probe currents obtainable**
- **Stable probe, especially W**
- **Less complex vacuum system**
- **Lowest overall cost / easy to maintain**

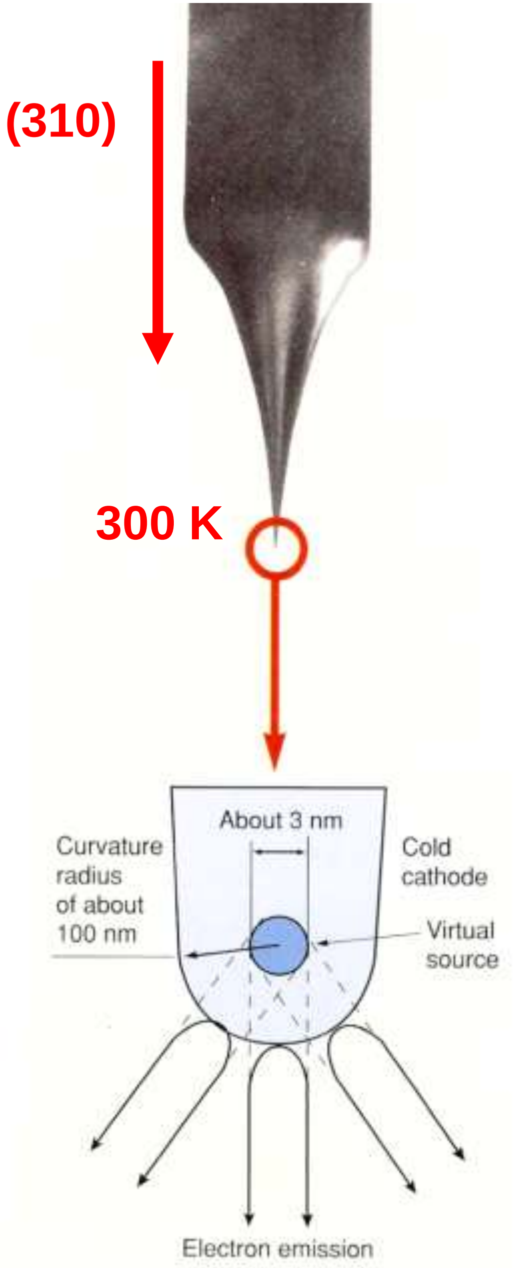
Disadvantages:

- **Lower brightness**
- **Relatively short lifetimes**

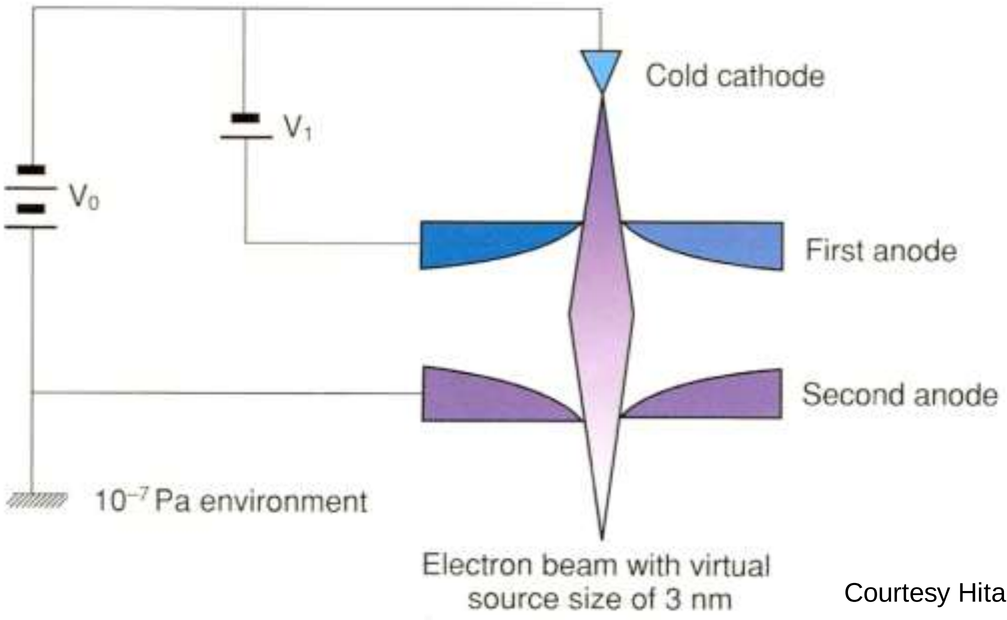
From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press

Electron Sources -Cold Field Emission

Sharp Single Crystal (310) Tungsten Tip



Courtesy Hitachi Instruments



Courtesy Hitachi Instruments

Major Advantages:

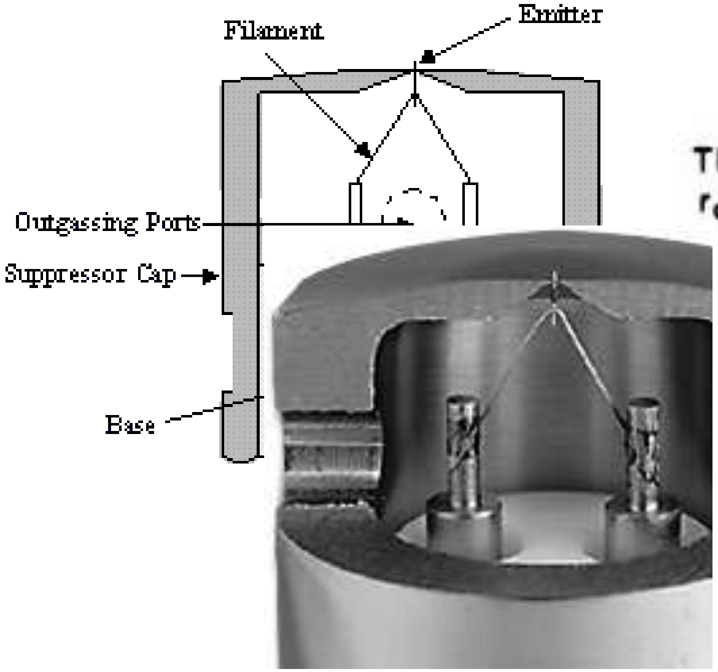
- **Highest brightness SEM source available**
- **Very long potential source lifetime – many years**

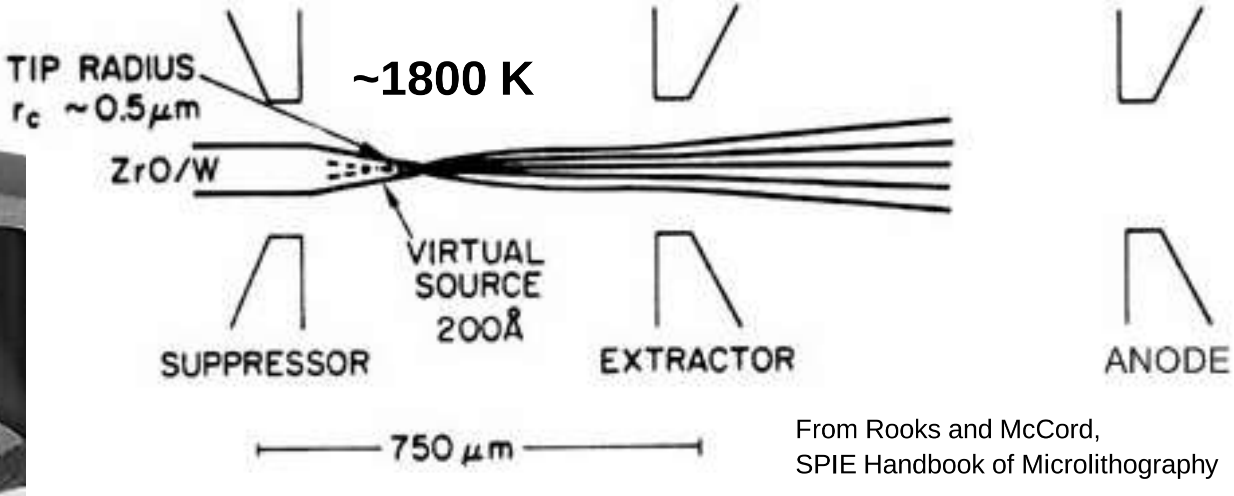
Disadvantages:

- **Lowest maximum probe current**
- **Poor short and long term probe current stability**
- **Requires ultra-high vacuum in gun area**
- **Cost (initial)**

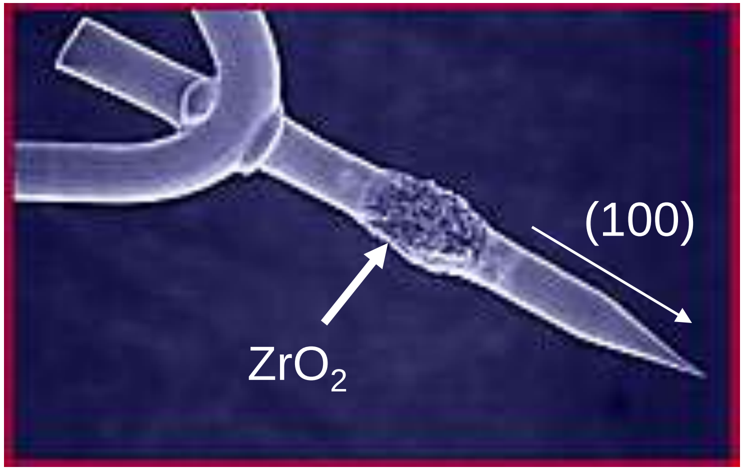
Electron Sources - Thermal-Field (Schottky) Emission

Sharp Single Crystal (100) Tungsten Tip with ZrO_2 Film





From Rooks and McCord,
SPIE Handbook of Microlithography



http://www.fei.com/products/components/electron-ion-sources.aspx

Major Advantages:

- **Very high brightness source**
- **High probe currents obtainable (few hundred nA)**
- **Long potential source lifetime (few years)**
- **Excellent short and long term stability**

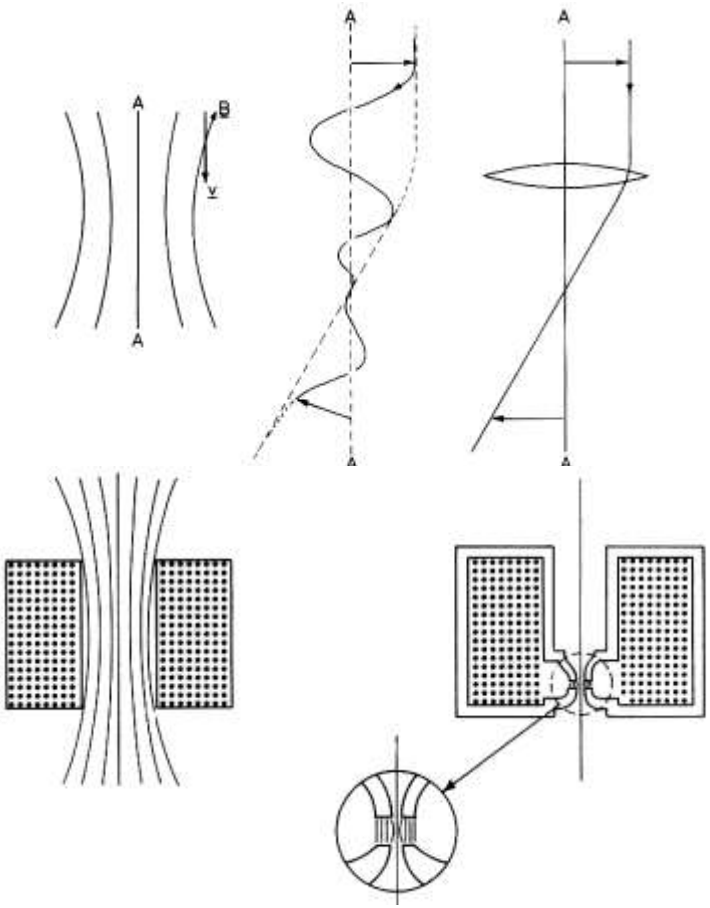
Disadvantages:

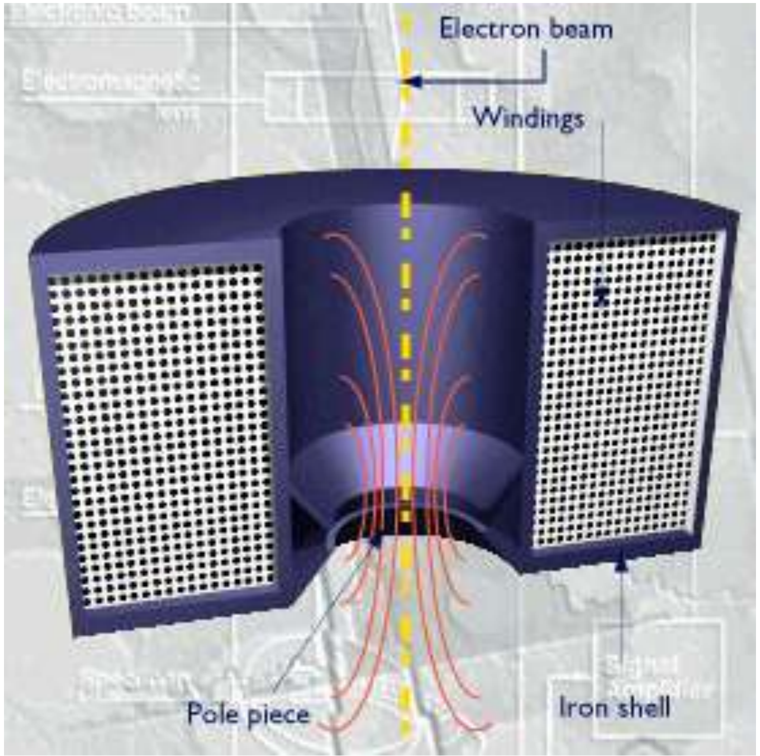
- **Requires ultra-high vacuum in gun area**
- **Source heating is continuous, 24/7 (finite life)**
- **Cost (initial and maintenance)**

Comparison of Electron Sources

	TUNGSTEN	LaB_6	SCHOTTKY	COLD FIELD
Effective Source Size (nm)	15,000	5000	15	3
Brightness (A/cm^2SR)	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	<150µA	<100µA	<150 µA	<20µA
Maximum Probe Current (SEM)	1000+ nA	<1000 nA	10-500 nA	<2.0 nA
SEM resolution (typical specs range)	3.0 -4.0 nm	2.0-3.0nm	<1.0 – 2.0 nm	<1.0-1.5 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 hrs	1000 hrs	>>1 year	>>>1 year
Cost	$	$$	$$$	$$$

Lenses in Electron-Optical Columns





Graphic from *A Guide to X-Ray Microanalysis*, Oxford Microanalytical Instruments

Magnetic Lens

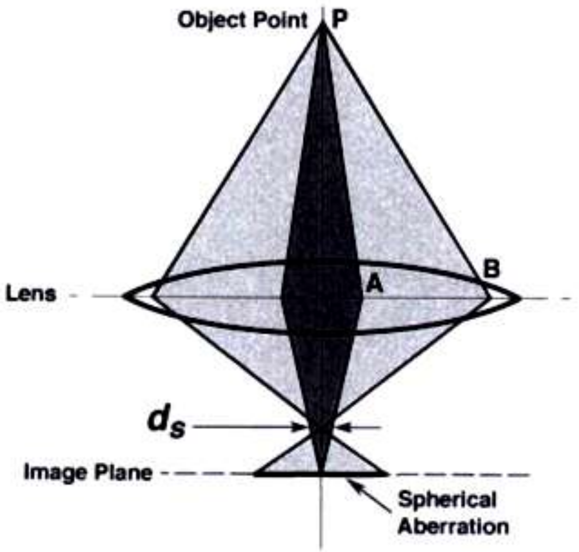
- Electromagnet coil
- Precision machined soft iron "pole piece"

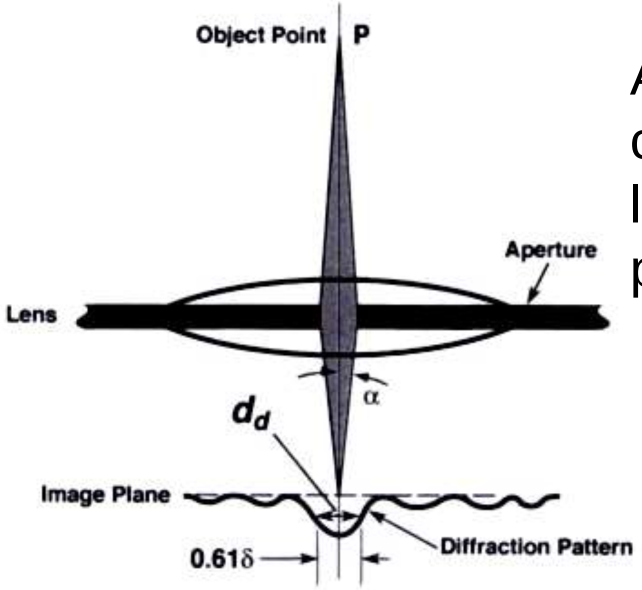
Limiting Parameters

- Spherical Aberration
- Chromatic Aberration
- Astigmatism
- Aperture diffraction

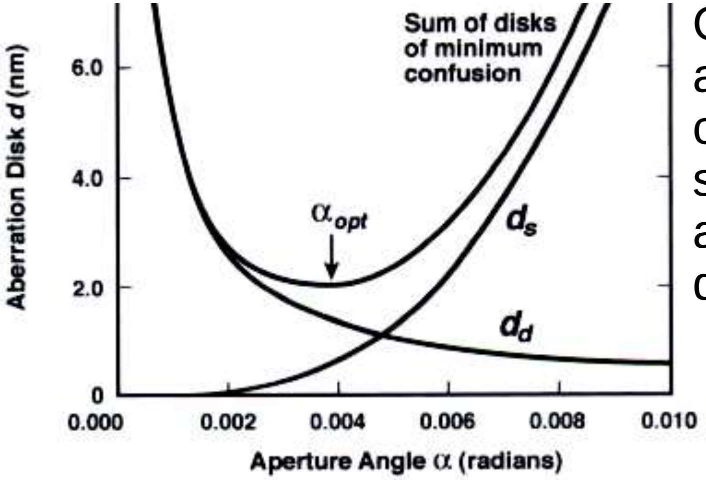
Lens Aberrations / Optimum Aperture Angle

Spherical Aberration

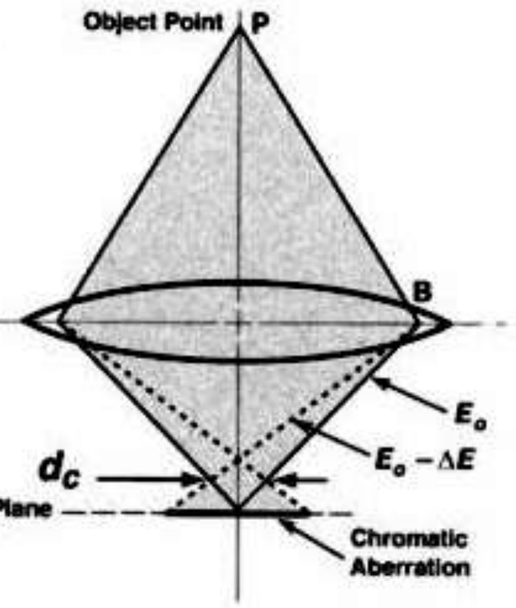




Aperture Diffraction causes a fundamental limit to the achievable probe size.



Optimum aperture angle determined by combined effect of spherical aberration and aperture diffraction.

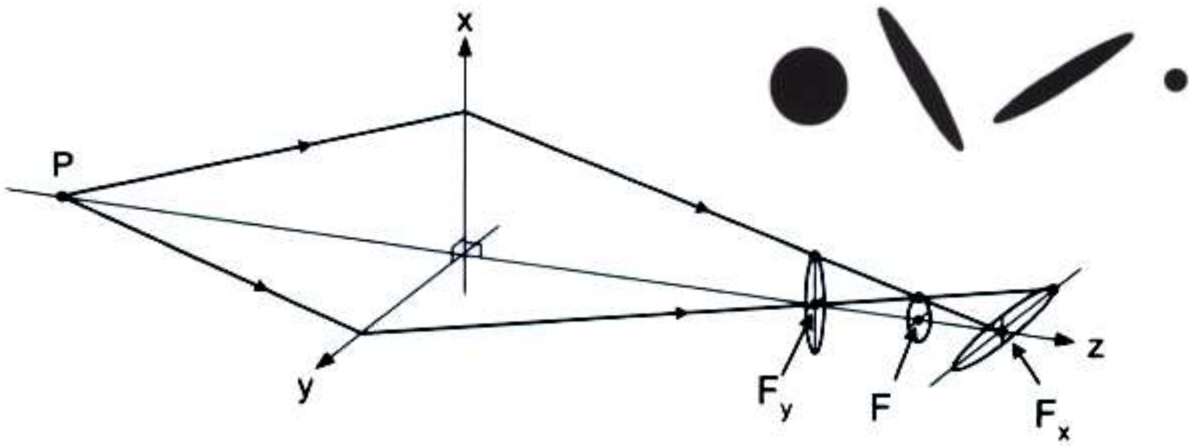


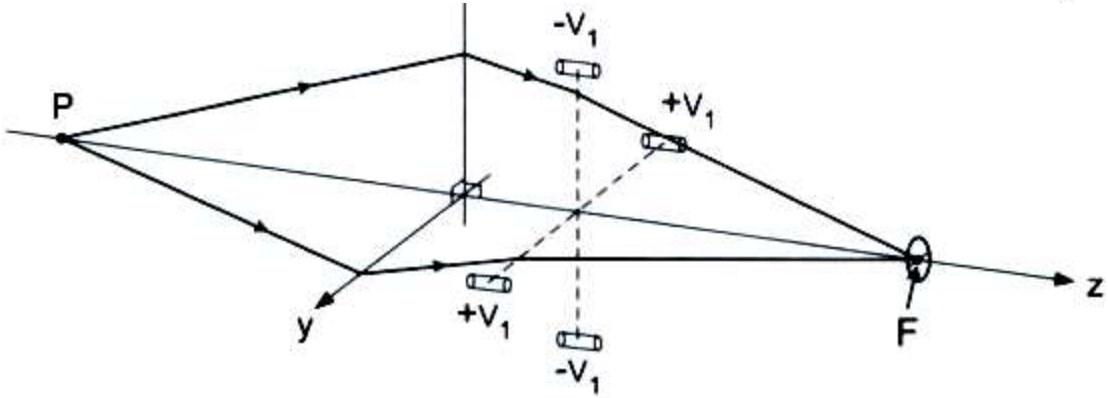
Chromatic Aberration

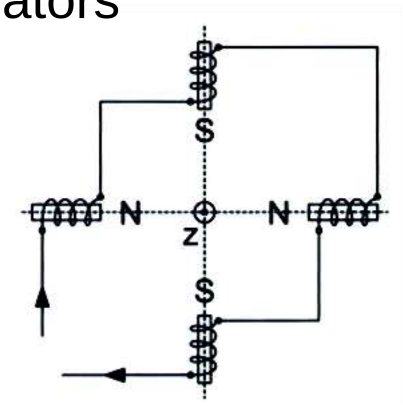
Adapted from *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press

Lens Aberrations / Astigmatism

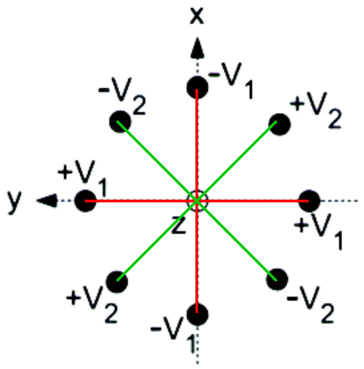
Astigmatism is caused by imperfections in the lens or other interference. It can be corrected using additional elements called stigmators contained inside the objective lens.







Magnetostatic quadrupole lens is basis of a stigmator



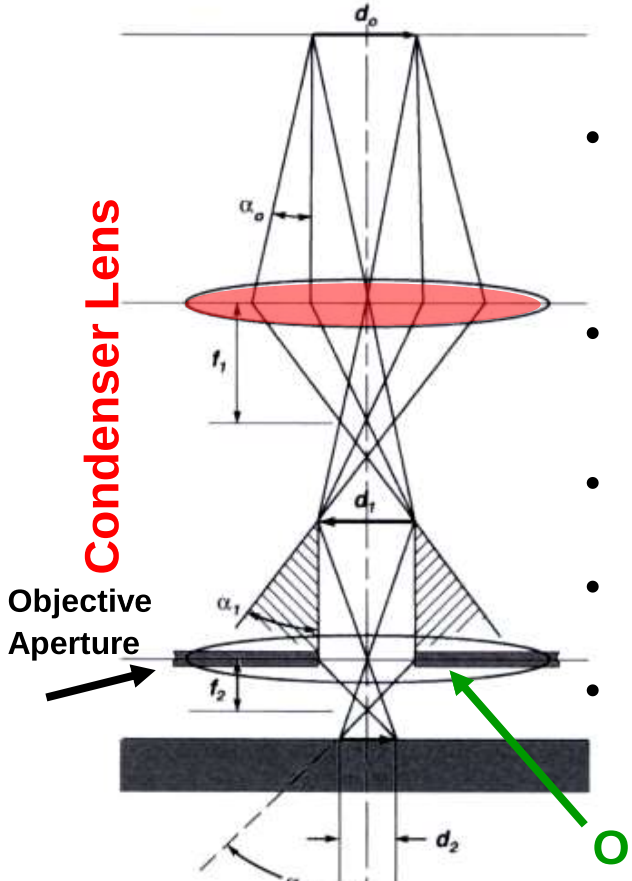
Octupole lens stigmator

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

Function of Condenser Lens(es)

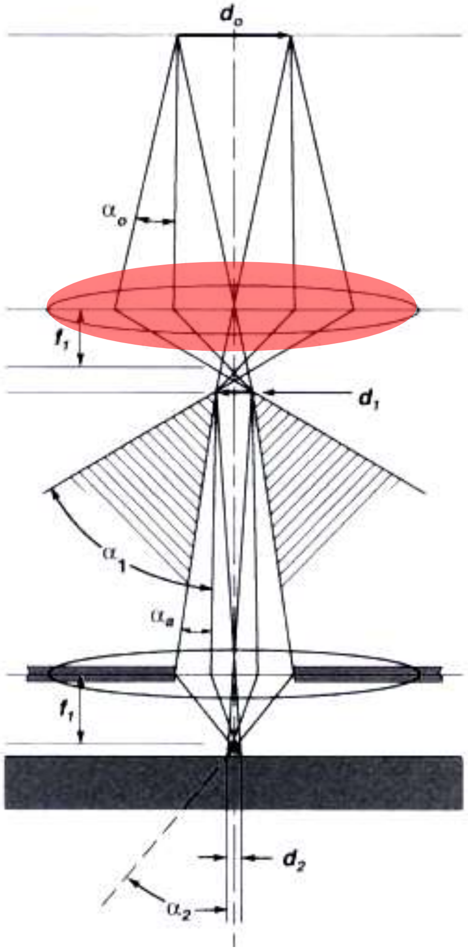
De-magnify the beam extracted from the source to enable a small spot to be obtained on the sample. Multiple lenses may be used in the condenser lens system.

Weak Lens

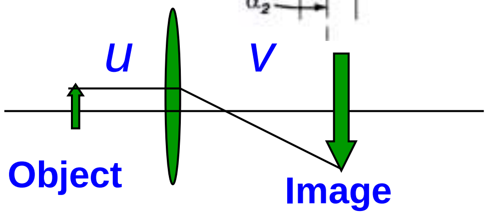


- Longer focal length, Small α_1, Larger d_1
- More beam accepted into objective aperture
- Higher probe current at specimen
- Larger focal spot at specimen
- Lower resolution
- Higher Signal Levels

Strong Lens



- Short focal length, Larger α_1, Smaller d_1
- Less beam accepted into objective aperture
- Lower probe current at specimen
- Smaller focal spot at specimen
- Higher Resolution
- Lower Signal Levels

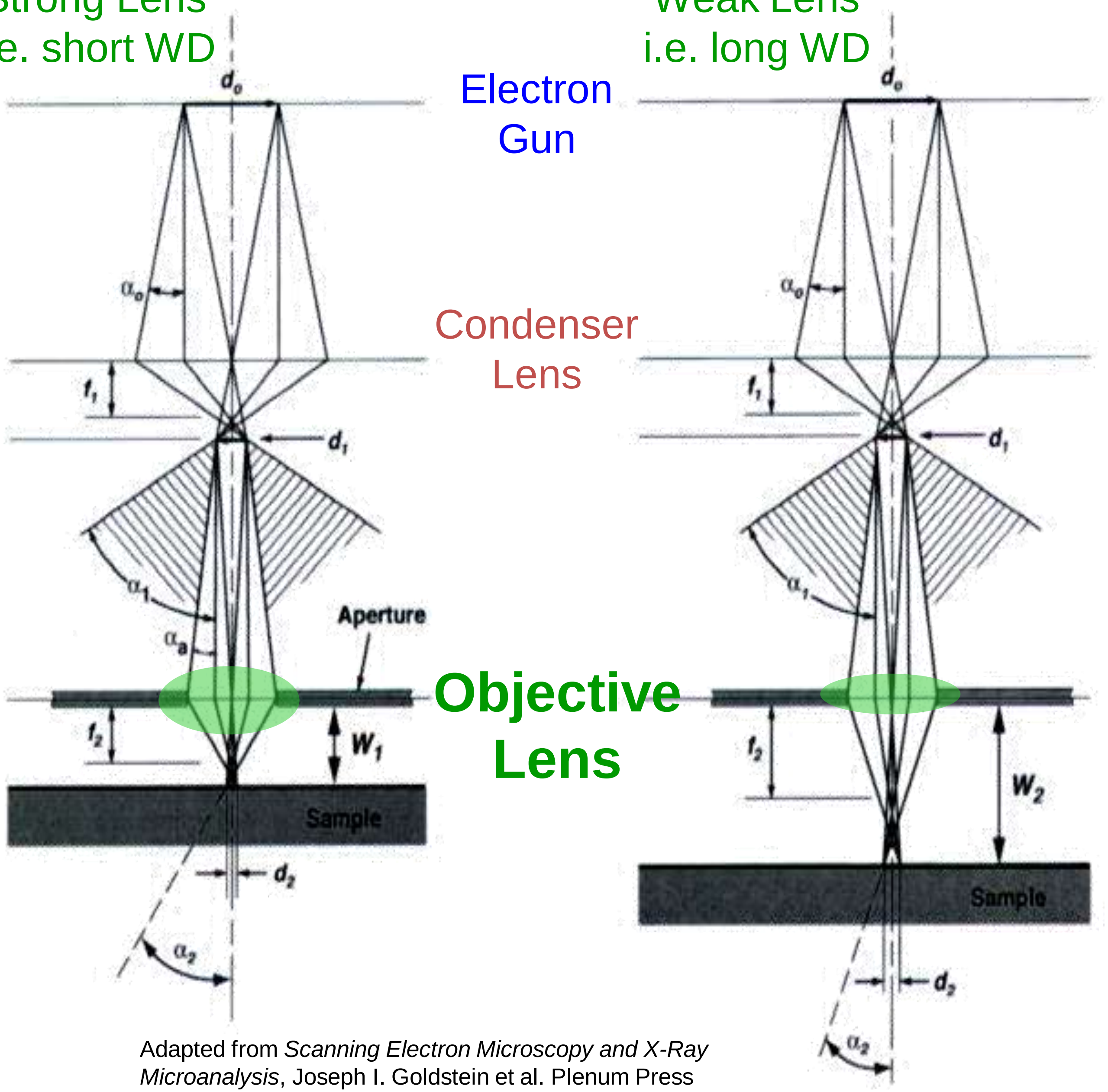


$$\frac{1}{u}+\frac{1}{v}=\frac{1}{f} \qquad M=v/u$$

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

Objective Lens / Working Distance

- **Focus the electron beam on the specimen with minimal lens aberrations, astigmatism is corrected**
- **Short Focal Lengths(W_1) –> smaller d_2, larger α_2 -> better resolution, poorer depth of focus**
- **Longer Focal Lengths(W_2) –> larger d_2, smaller α_2 -> better depth of focus, poorer resolution**
- **Smaller Apertures –> smaller d_2, smaller α_2 -> better resolution & better depth of focus, limited by aperture diffraction or S/N**



Strong Lens
i.e. short WD

Weak Lens
i.e. long WD

Electron Gun

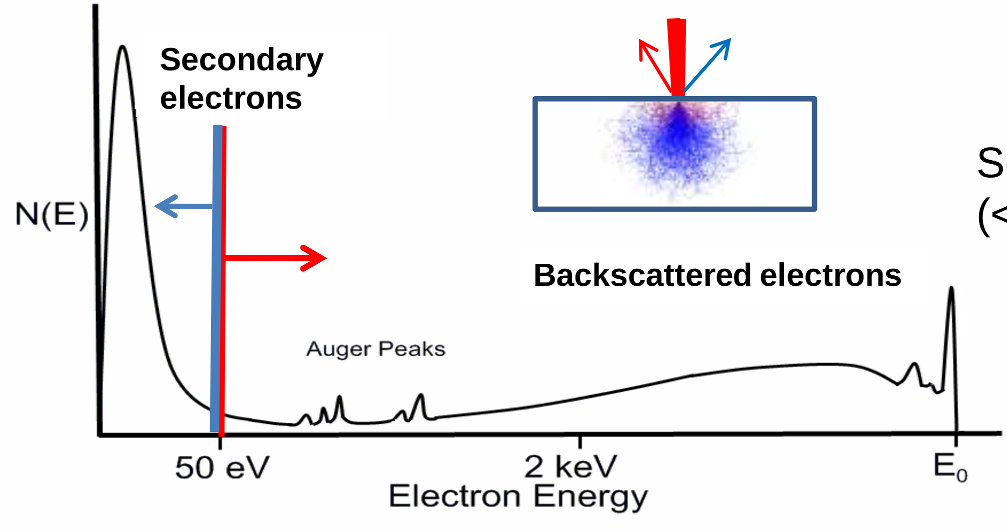
Condenser Lens

Objective Lens

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

Secondary and Backscattered Electrons

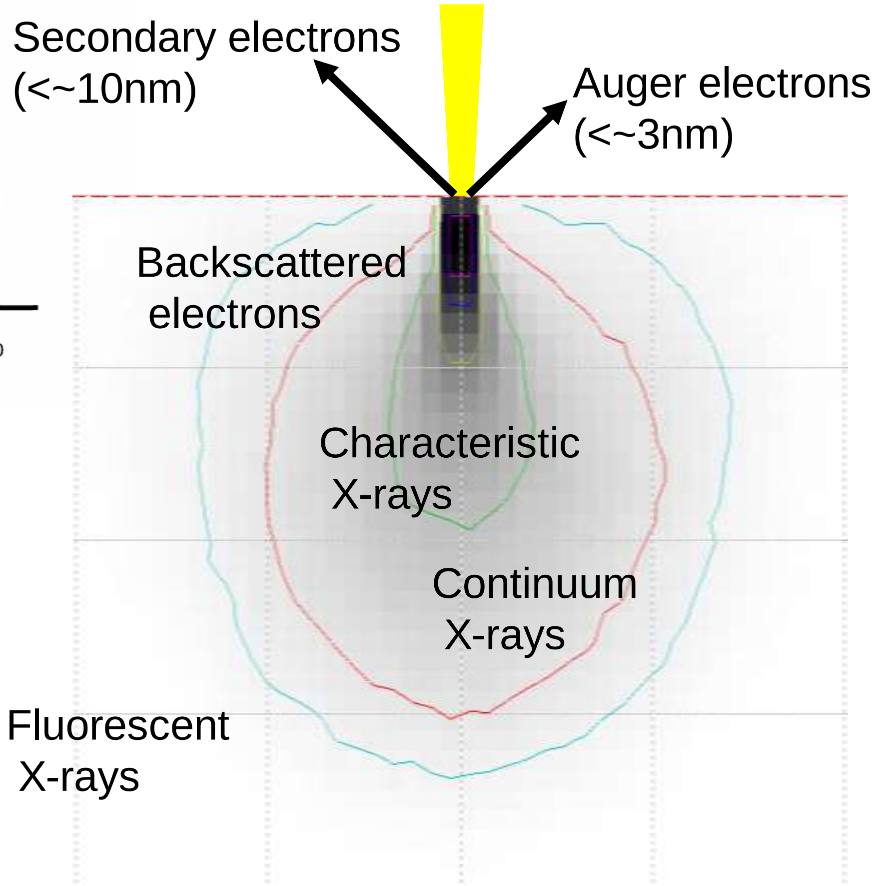
Energy Distribution of Emitted Electrons



Secondary electrons are low energy electrons (<50eV) ejected from the specimen atoms by the energetic primary beam.

Backscattered electrons are primary beam electrons scattered back out of the sample.
There is a purely elastic peak and a continuum of BSE that have lost energy.

Depth/Lateral Distribution of Emitted Electrons (and other emissions)



Secondary electrons (<~10nm)

Auger electrons (<~3nm)

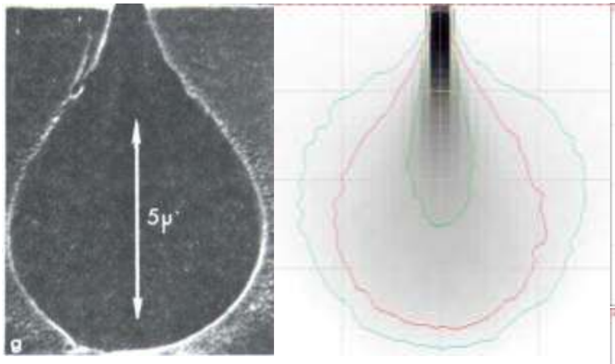
Backscattered electrons

Characteristic X-rays

Continuum X-rays

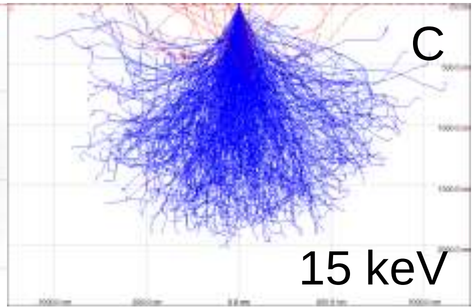
Fluorescent X-rays

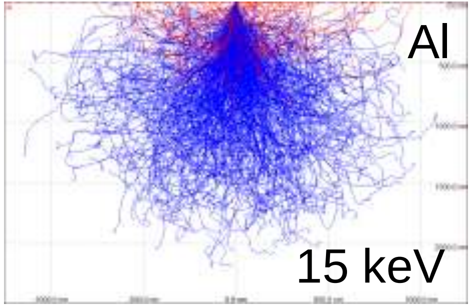
Monte-Carlo simulations of electron scattering

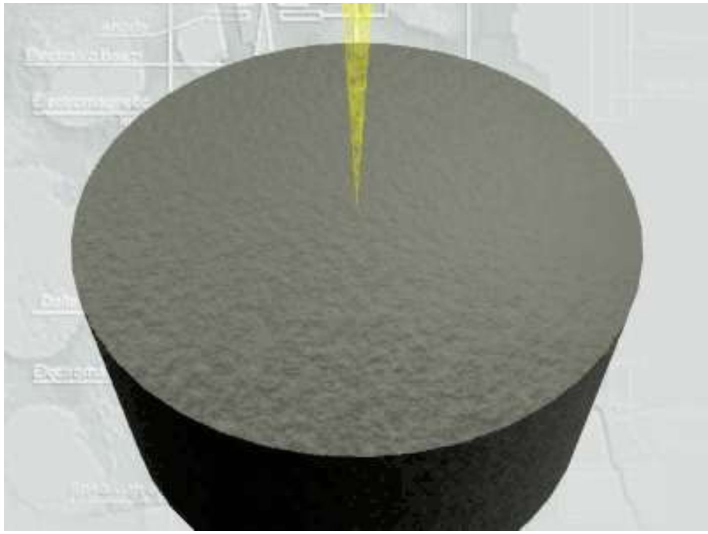


PMMA @ 20kV

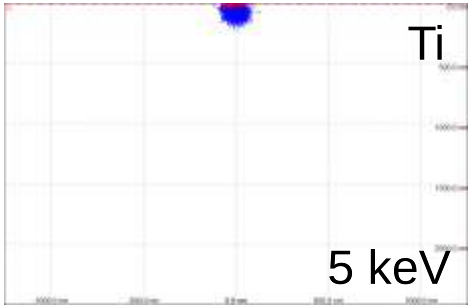
Everhart et.al. (1972)

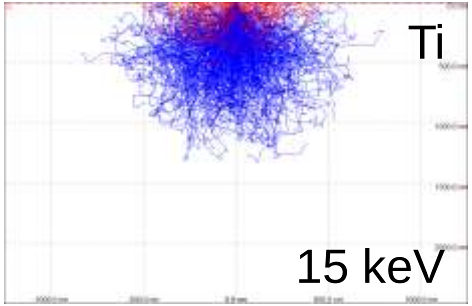


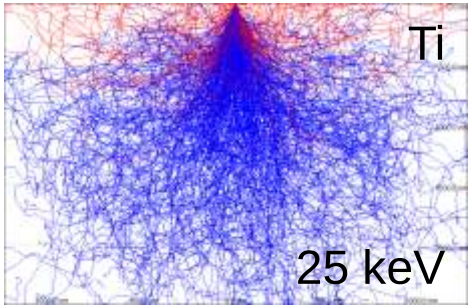




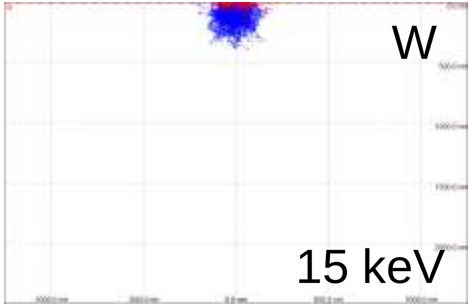
1 µm







- Determine effective lateral or depth resolution for a particular signal in a defined sample.
- Simulate X-ray generation / X-ray spectra in a defined sample.
- Simulate image contrast/ images.

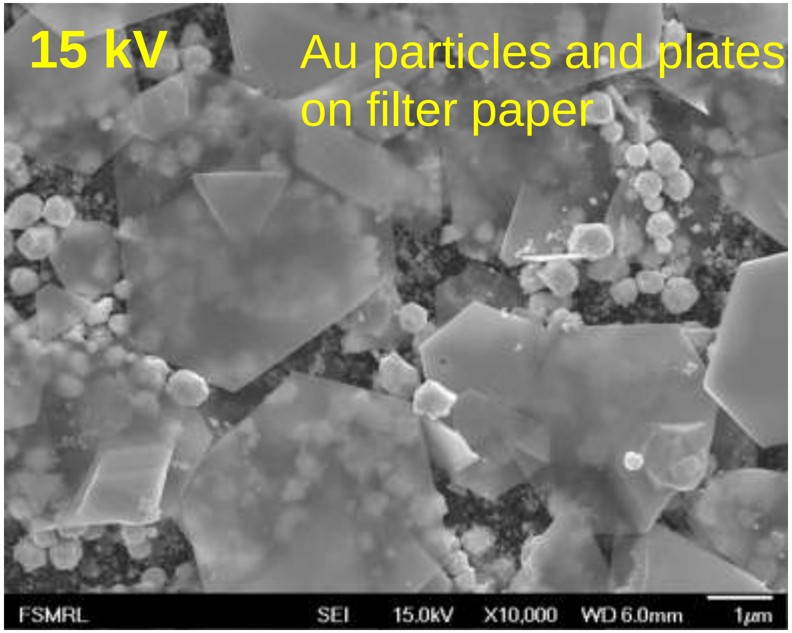


Simulations are very useful for testing if a measurement is possible or interpreting results.

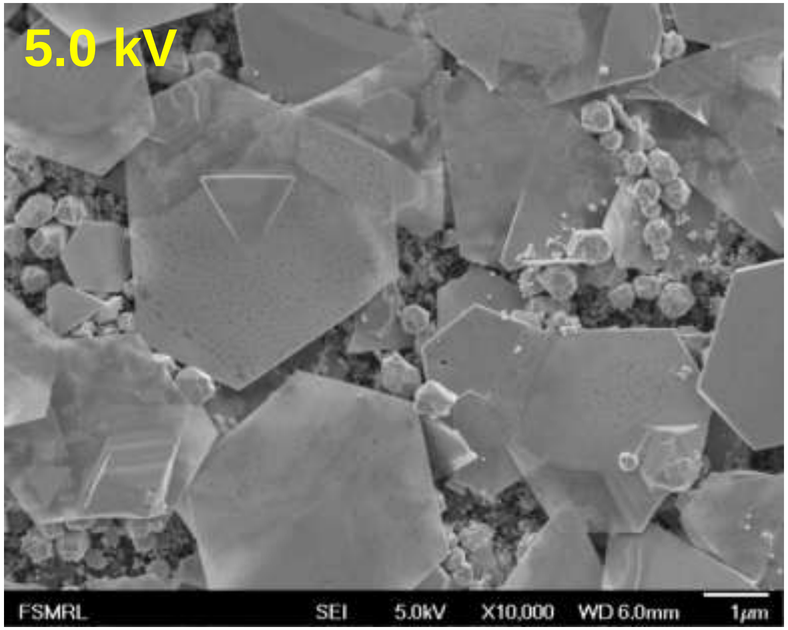
Animation from *A Guide to X-Ray Microanalysis*, Oxford Microanalytical Instruments

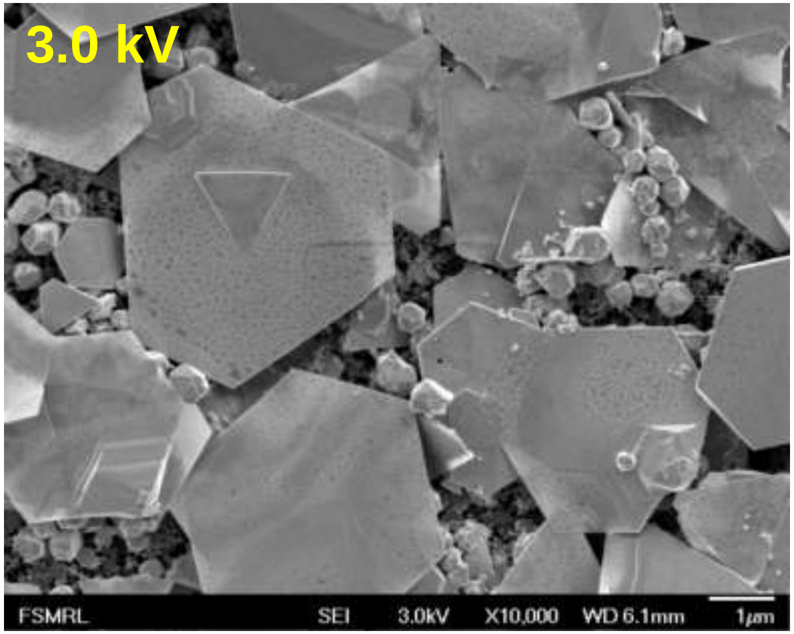
Monte Carlo Calculations, CASINO

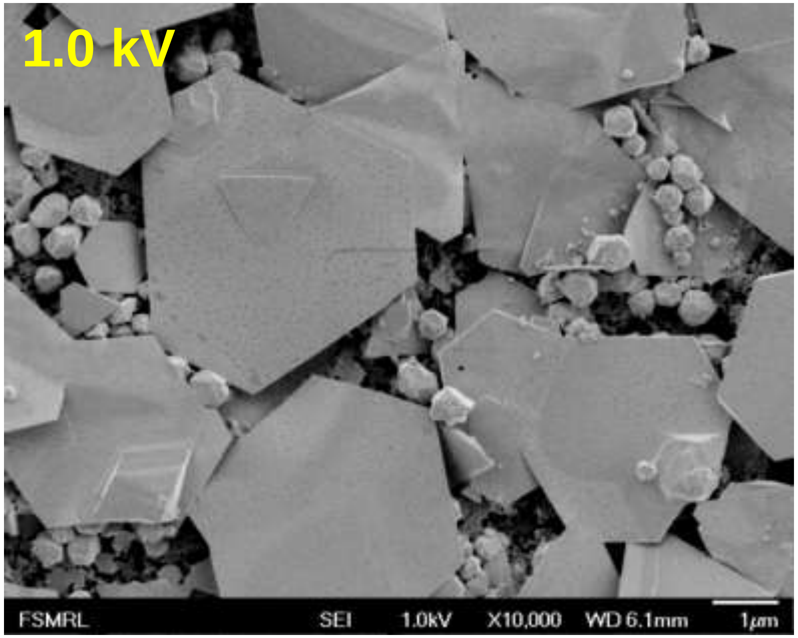
Effect of Beam Energy on SE Imaging



Au particles and plates on filter paper

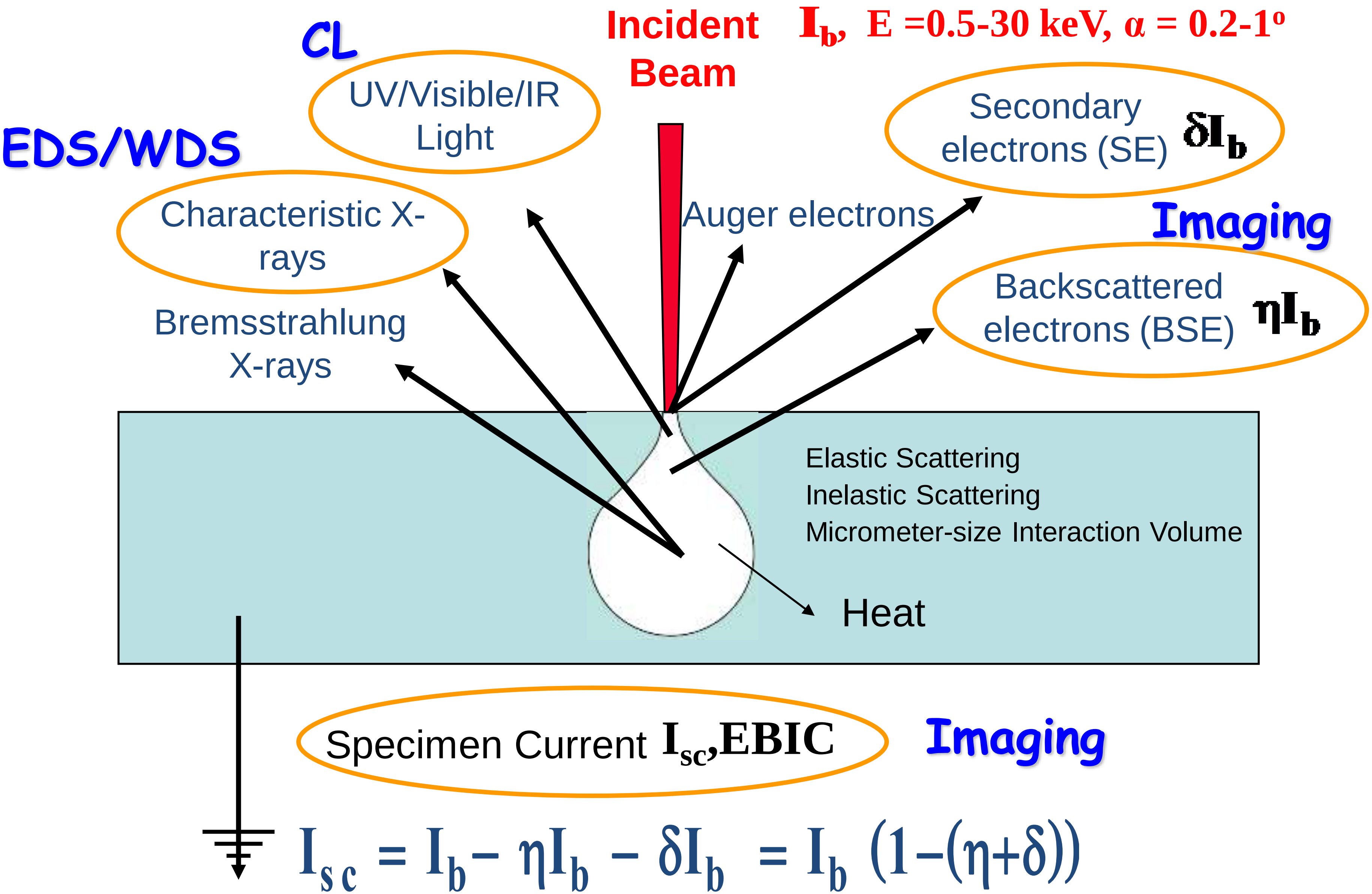






Sample courtesy Tom Bassett (P. Kenis group)

Electron Beam / Specimen Interactions



Incident Beam I_b, E =0.5-30 keV, α = 0.2-1º

CL

UV/Visible/IR Light

EDS/WDS

Characteristic X-rays

Bremsstrahlung X-rays

Auger electrons

Secondary electrons (SE) δI_b

Imaging

Backscattered electrons (BSE) ηI_b

Elastic Scattering
Inelastic Scattering
Micrometer-size Interaction Volume

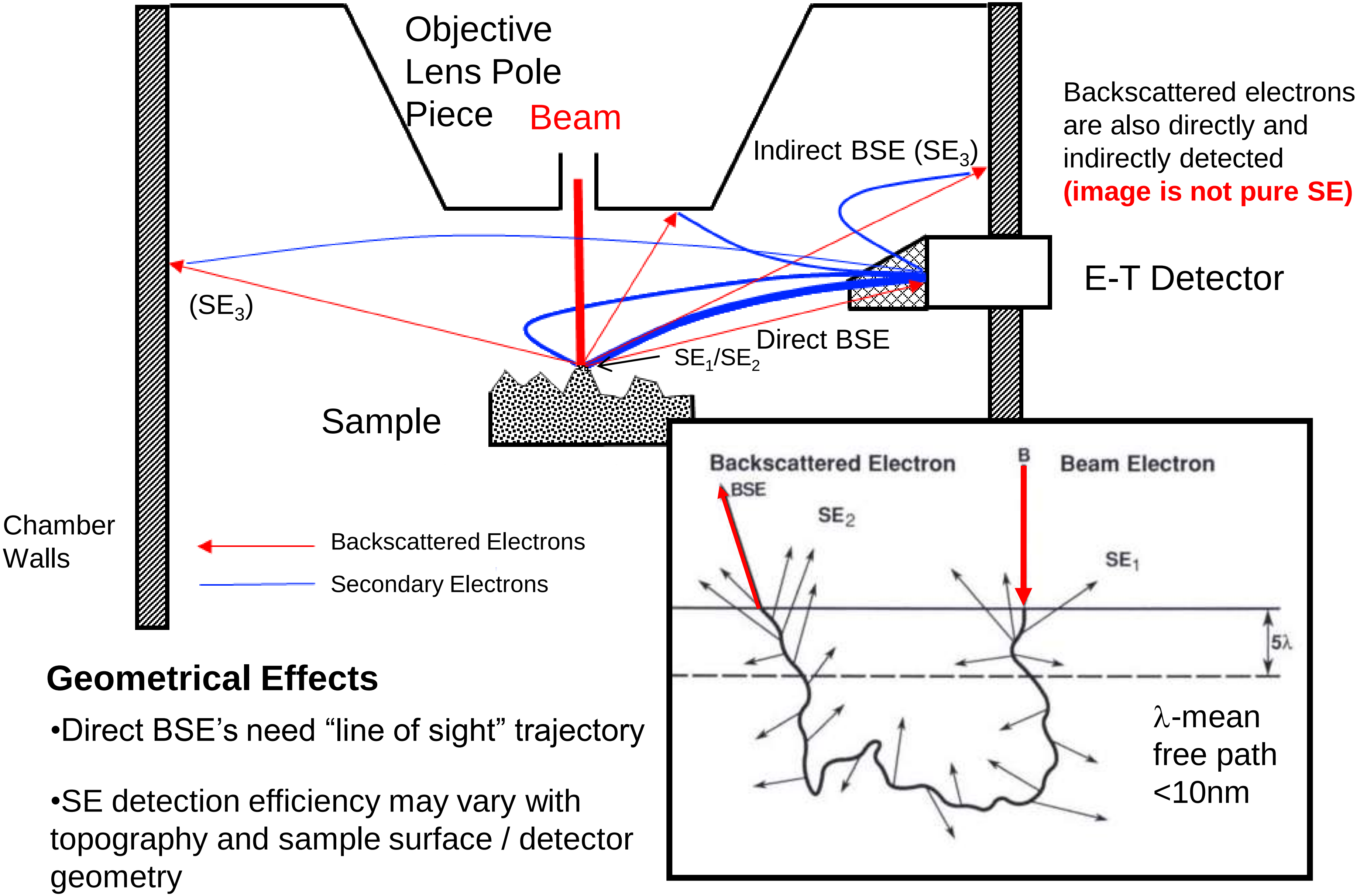
Heat

Specimen Current I_{sc},EBIC

Imaging

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

Sources of Electrons Detected By E-T Detector



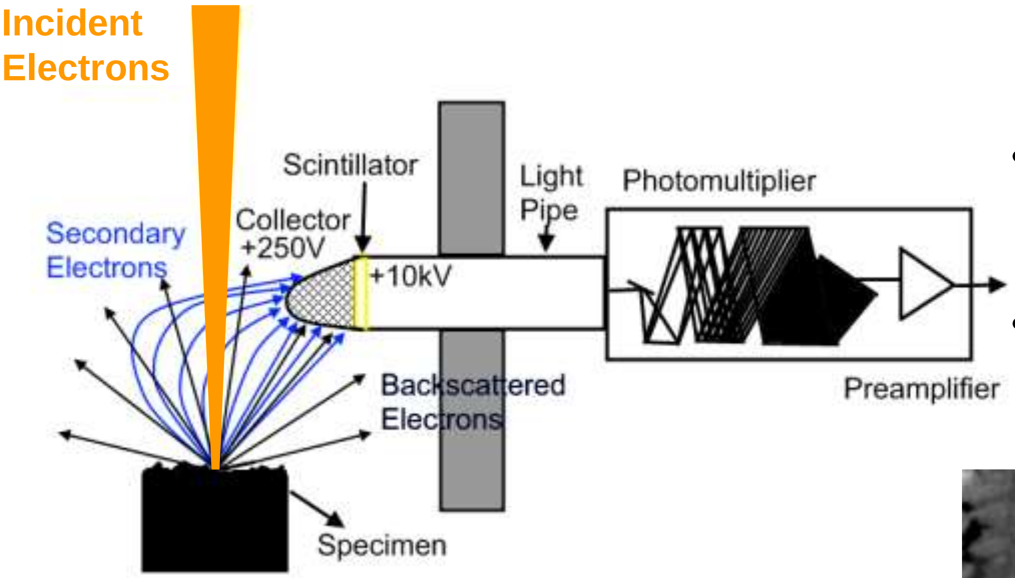
Backscattered electrons are also directly and indirectly detected **(image is not pure SE)**

λ-mean free path <10nm

Geometrical Effects

- Direct BSE’s need “line of sight” trajectory
- SE detection efficiency may vary with topography and sample surface / detector geometry

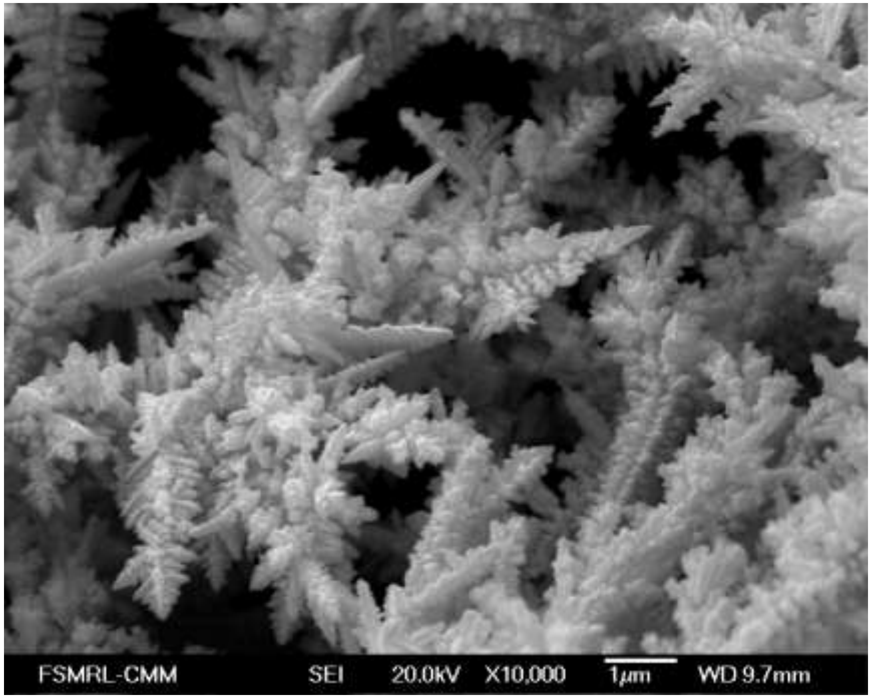
"Secondary Electron Detector" / Imaging



Everhart-Thornley SE detector

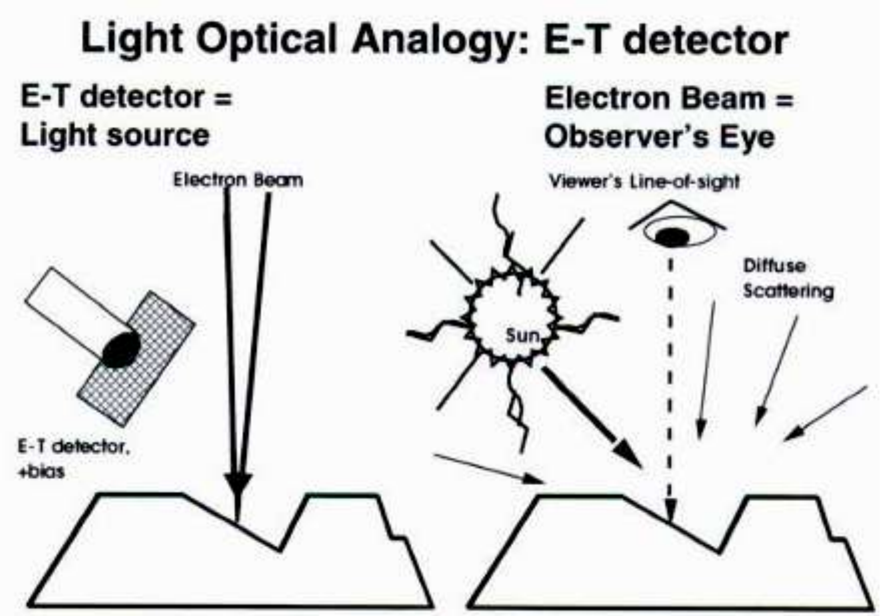
- Faraday Cage (collector) is usually biased a few hundred volts positive (for collection efficiency).
- Scintillator is biased +10kV to accelerate electrons to sufficient energy to efficiently excite scintillating material.
- Amplified output level is directly used to set brightness (offset) and contrast (gain) in corresponding pixel in image.

Contrast results from **topographical dependence** of the secondary electron yield, other detected electrons (BSE), edge effects, and geometrical collection effects.



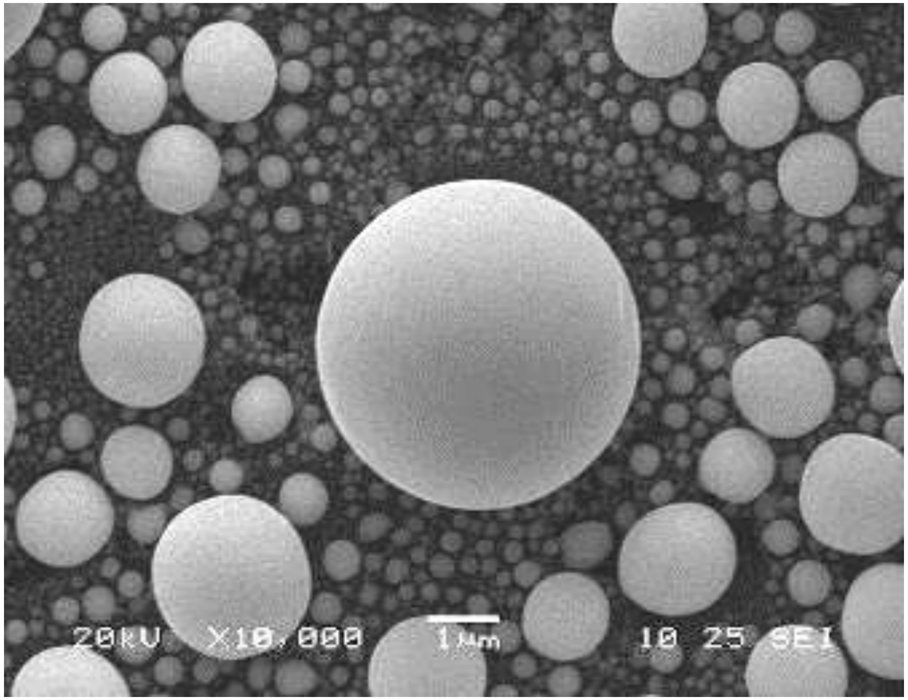
Electrodeposited Gold Dendritic Structure

Analogy to Oblique and Diffuse Optical Illumination



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

Detector Position

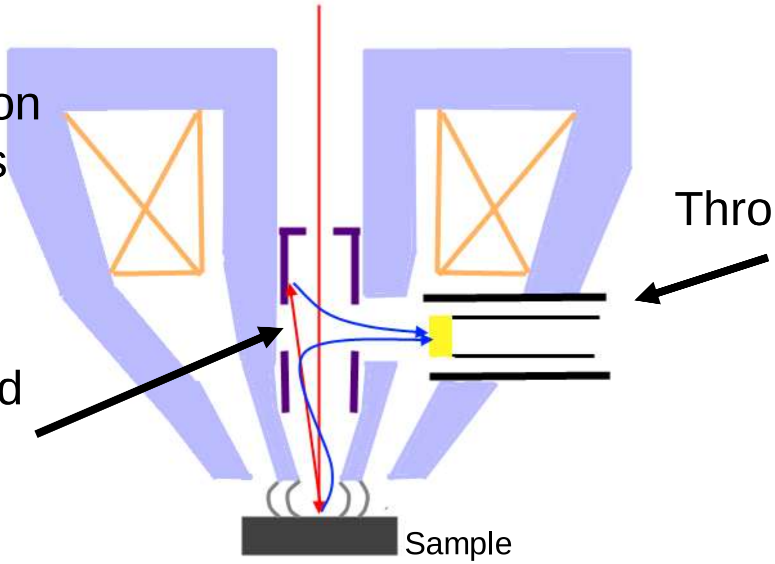


These dependencies on electron yield and detection in combination with the high depth of focus of the SEM, gives the familiar SEM images with a good perceptive sense of surface topography.

Through The Lens Detector

Highest Resolution SEM’s use a semi-immersion type objective lens to improve resolution, especially at low beam energies and short WD.

Low voltage performance can be further improved along with extremely low “landing energies” made accessible by biasing sample ($V_{landing} = V_{beam} - V_{bias}$).



Semi-Immersion Objective Lens

Through-The-Lens-Detector

In-chamber E-T detector can still detect direct and indirect BSE’s → signal mixing with through-the-lens-detector

Arrangement of biased electrodes and BSE conversion plates to provide selection or filtering of detected energy range:
Detect only SE’s to only Low Angle BSE’s to various mixtures.

At short WD, SE’s are confined by lens field and travel up bore of lens.
Low angle BSE’s also travel up bore of lens

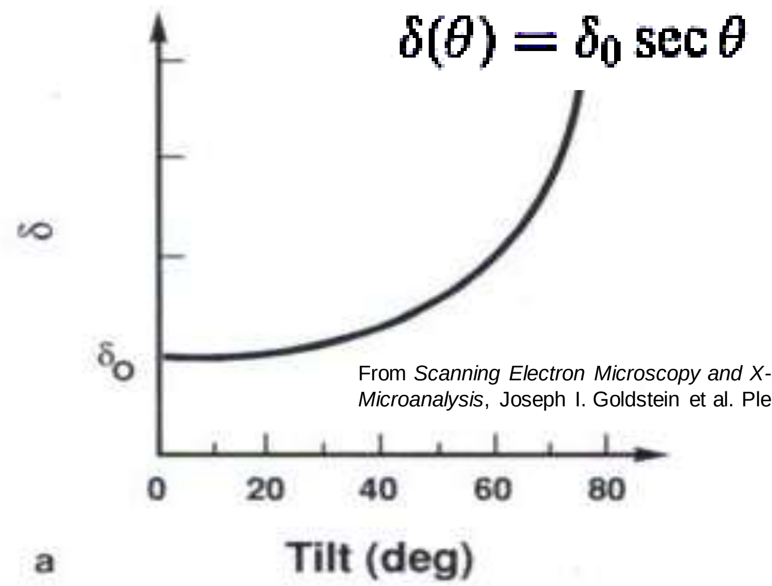
– LOSS of Oblique Illumination effect

Secondary Electron Yield

SE yield is strongly dependent with angle (local) of incidence of the beam with the sample surface.

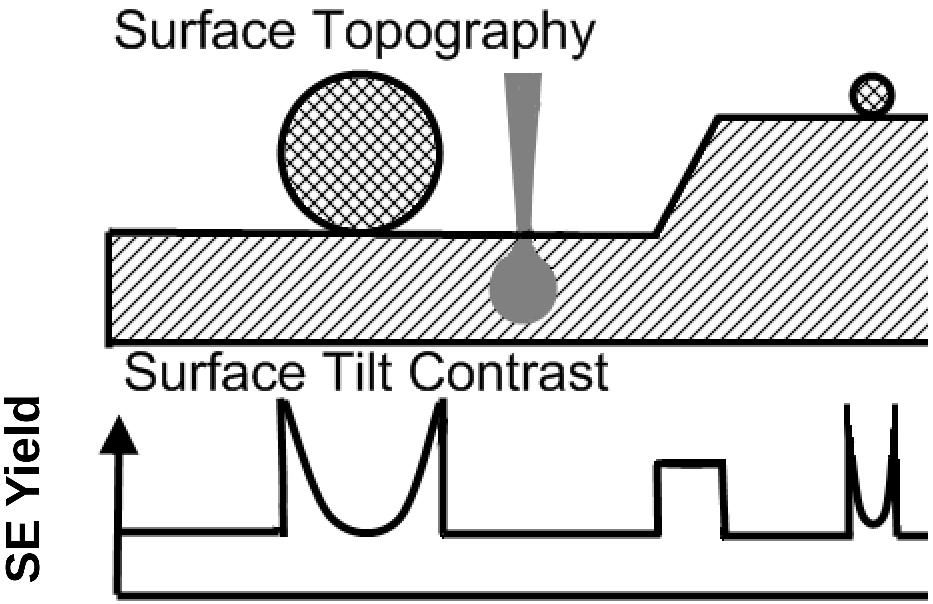
Geometrically the volume of material from which secondary electrons escape increases proportionally with the secant of the tilt angle.

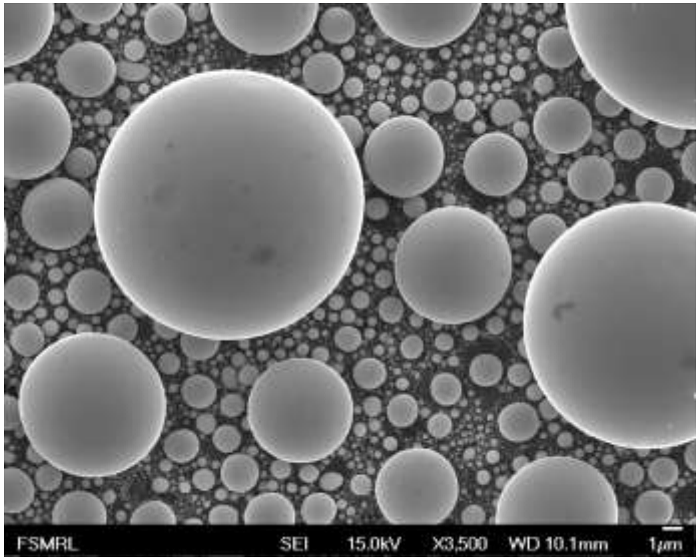
Note: There is also some material (atomic number) dependence of SE yield.



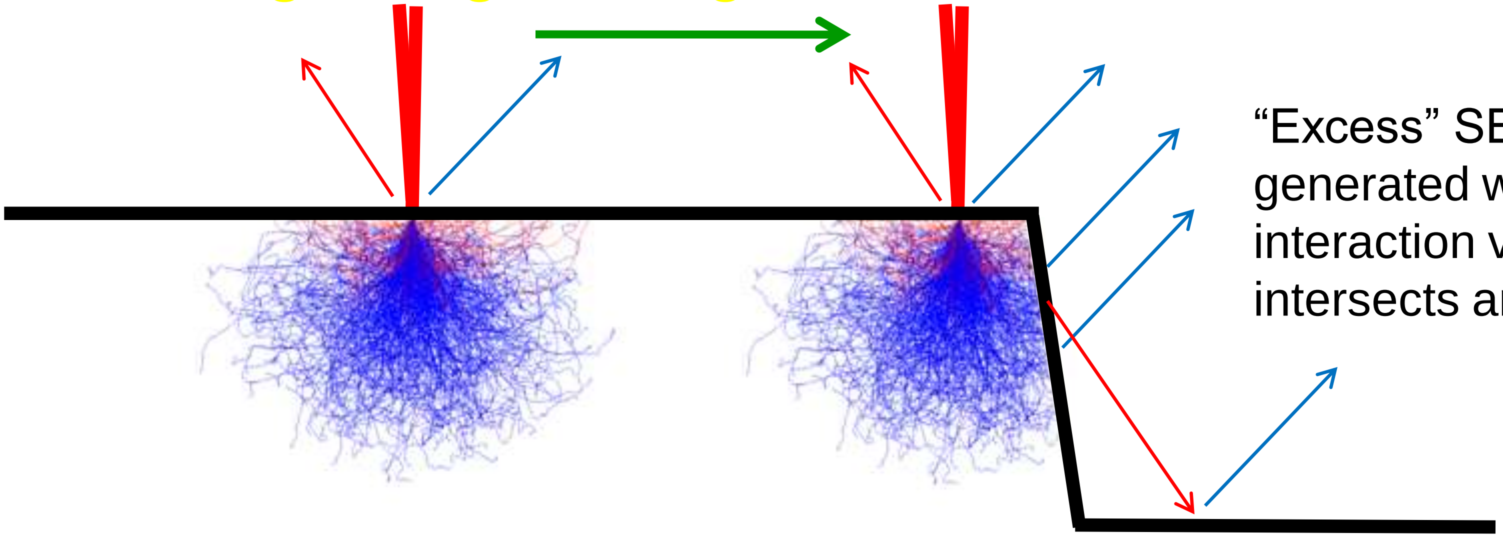
$$\delta(\theta) = \delta_0 \sec\theta$$

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

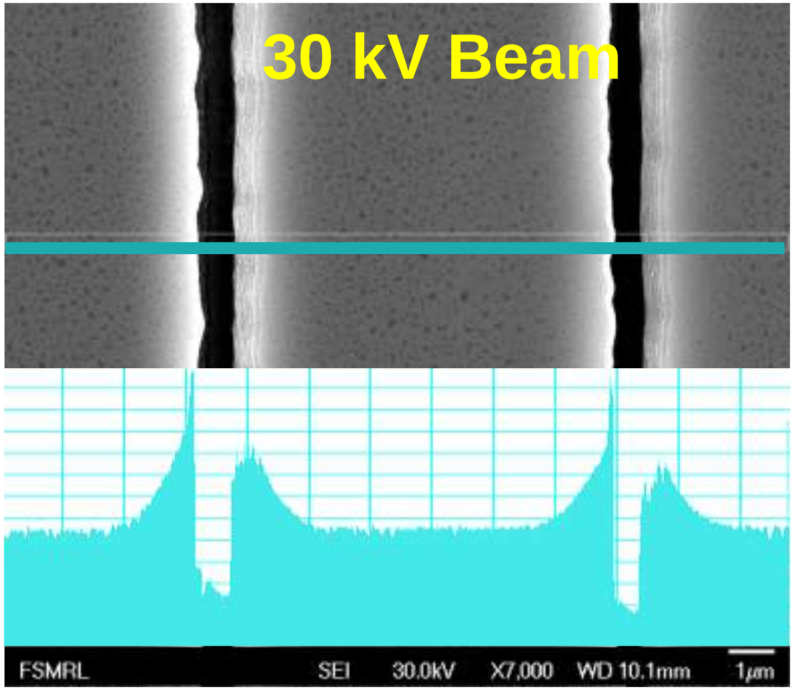




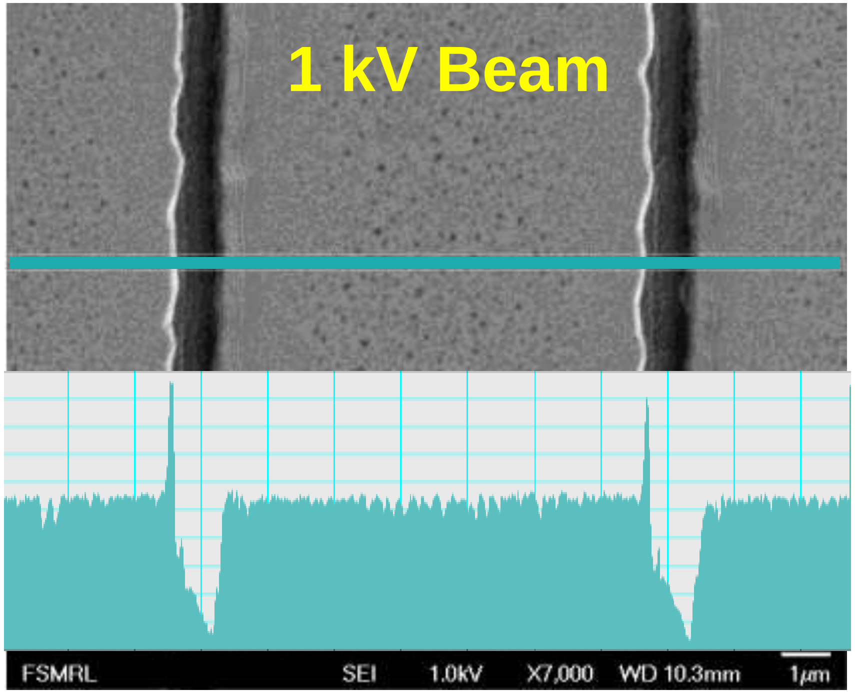
Edge Brightening Effect on Contrast



“Excess” SE’s generated when interaction volume intersects an edge.

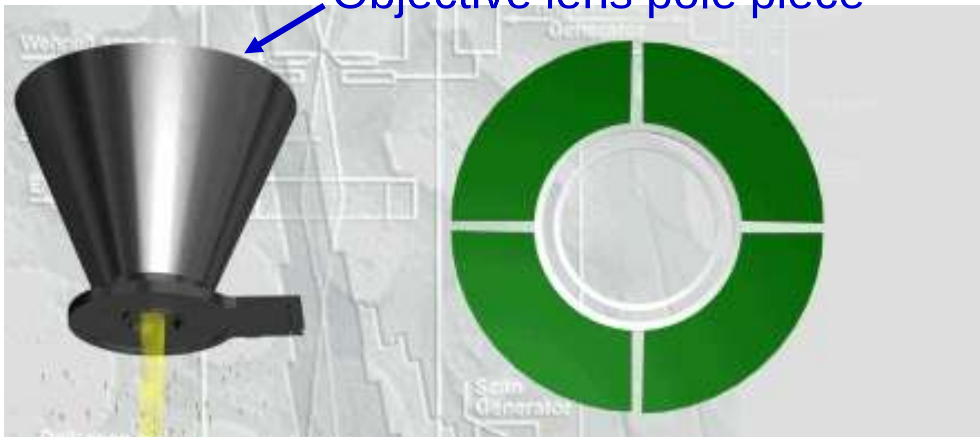


30 kV Beam



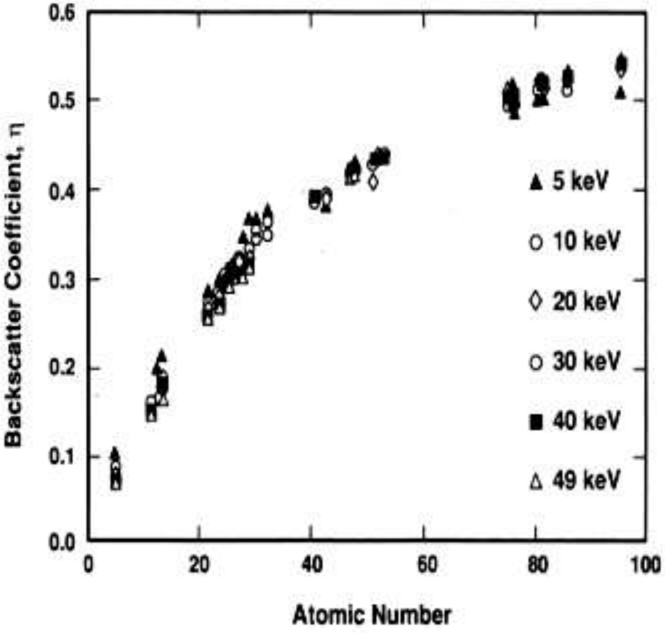
1 kV Beam

Backscattered Electron Detectors and Yield



Objective lens pole piece

Typical 4 quadrant solid state BSE detector



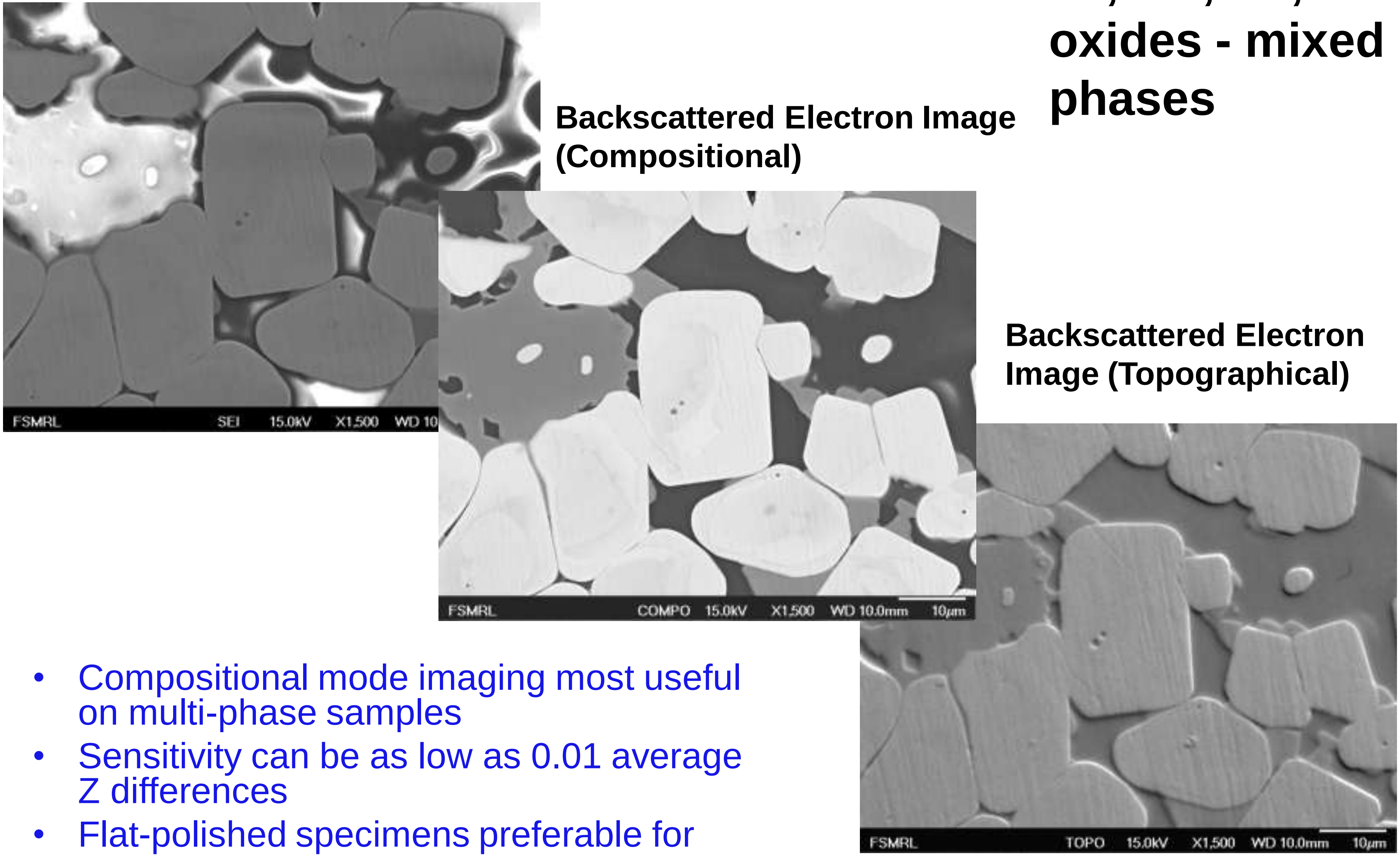
Backscattered electron yield is a strongly dependent on sample mean atomic number.

- Solid State (often 4 quadrant) Backscattered Electron detector placed annularly to bottom of objective lens (electron sensitive large area photodiode).
- Scintillator/PMT type detectors are also available.
- **Composition image** – electronically sum signal from all 4 quadrants.
- BSE **topographic images** – differencing various detector quadrants.
- Also can provide electron channeling (crystallographic orientation) contrast in suitable samples.

Graphic from, *The Oxford Guide to X-Ray Microanalysis*, Oxford Instruments Microanalysis Group

Backscattered Compositional Contrast

La, Mn, Ca, Al oxides - mixed phases



Secondary Electron Image

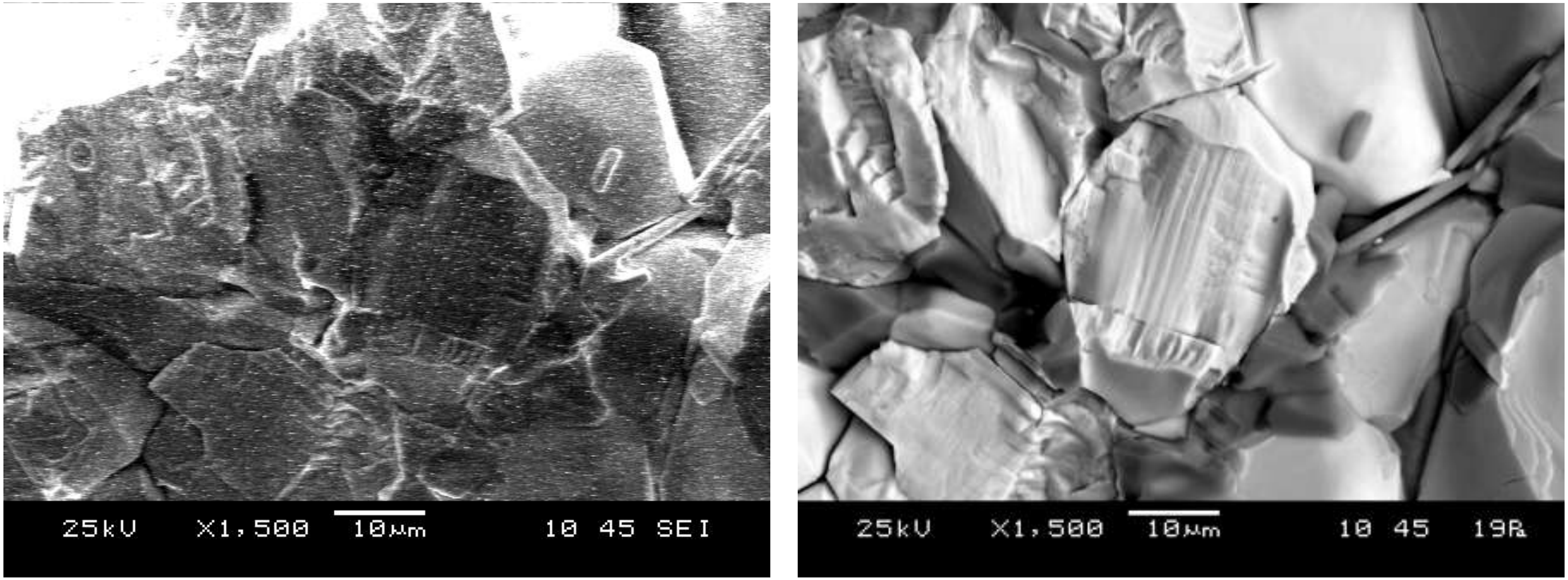
Backscattered Electron Image (Compositional)

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

Variable Pressure (VP / LV / Environmental) SEM

- **A solution to specimen charging of un-coated non-conductive samples is to introduce a gas (air, etc.) into the specimen chamber.**
- **The high energy electrons ionize the gas, thus positive ions are available to dynamically neutralize any charge on the sample.**
- **Available in both Schottky FEG and Thermionic (Tungsten) instruments.**

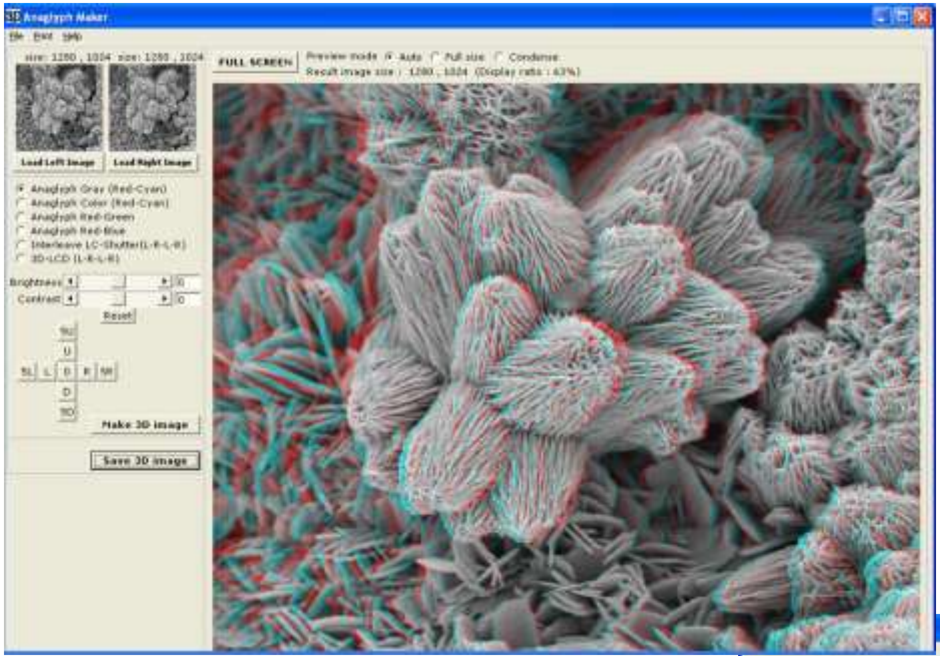


@ high vacuum

Uncoated Dysprosium Niobium Oxide Ceramic

@ 20 Pa air

Stereo-microscopy (Qualitative/Quantitative)



size: 1280 , 1024 size: 1280 , 1024

Preview mode ● Auto ○ Full size ○ Condense

Result image size : 1280 , 1024 (Display ratio : 63%)

● Anaglyph Gray (Red-Cyan)
○ Anaglyph Color (Red-Cyan)
○ Anaglyph Red-Green
○ Anaglyph Red-Blue
○ Interleave LC-Shutter(L-R-L-R)
○ 3D-LCD (L-R-L-R)

Brightness 0
Contrast 0

Qualitative (Visual)

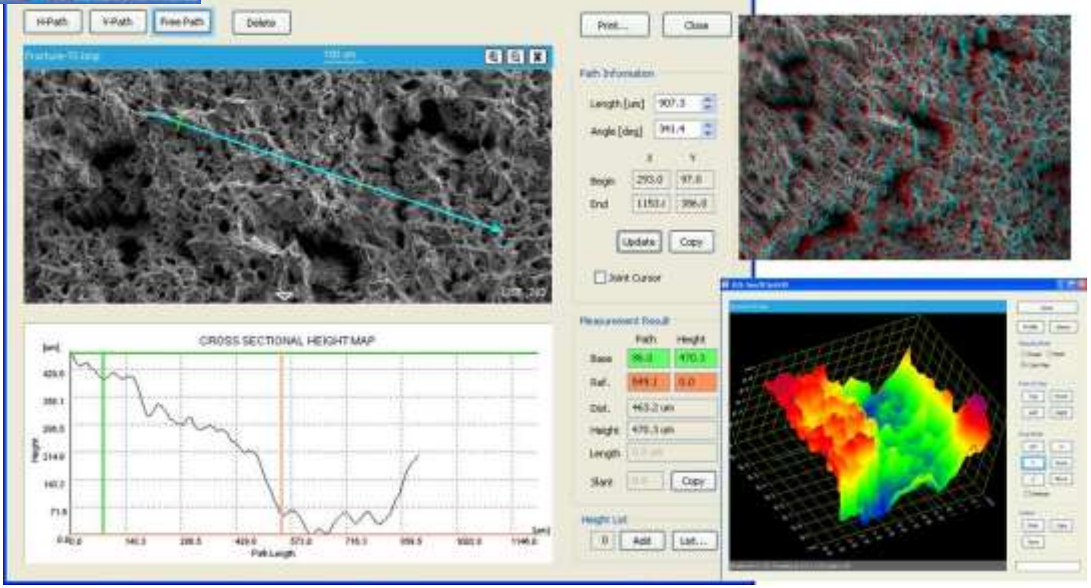
- Anaglyph (shown here in freeware software)
- Other Methods (same as in movies, TV, scientific visualization Labs)
- Stereo Image pair is very easy to obtain and make anaglyph (essentially any SEM, image mode)

Anaglyph Freeware:http://www.stereoeye.jp/software/index_e.html

Quantitative Height Maps/Profiles

Geometrical assessment of parallax shifts in **eucentrically tilted** image pair, triplet, etc. to reconstruct a height map.

Manually – possible for limited number of points
Automated – software*
* now several commercial software solutions available



Path Information
Length [um] 907.3
Angle [deg] 161.4
X Y
Begin 293.0 97.0
End 1150.0 396.0
☐ Joint Cursor

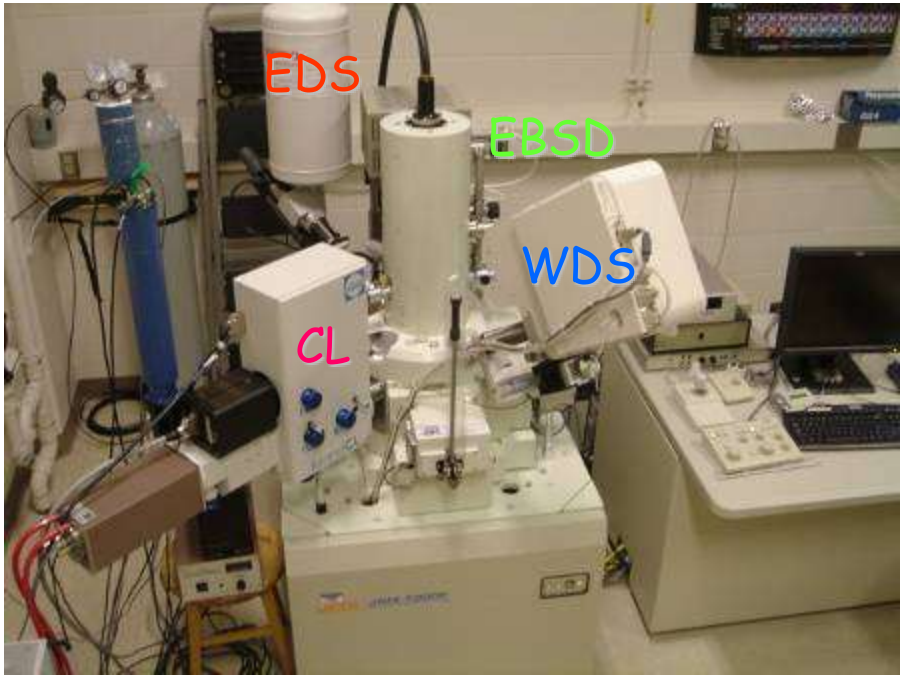
Measurement Result
	Path	Height
Base	96.0	470.3
Ref.	549.1	0.0

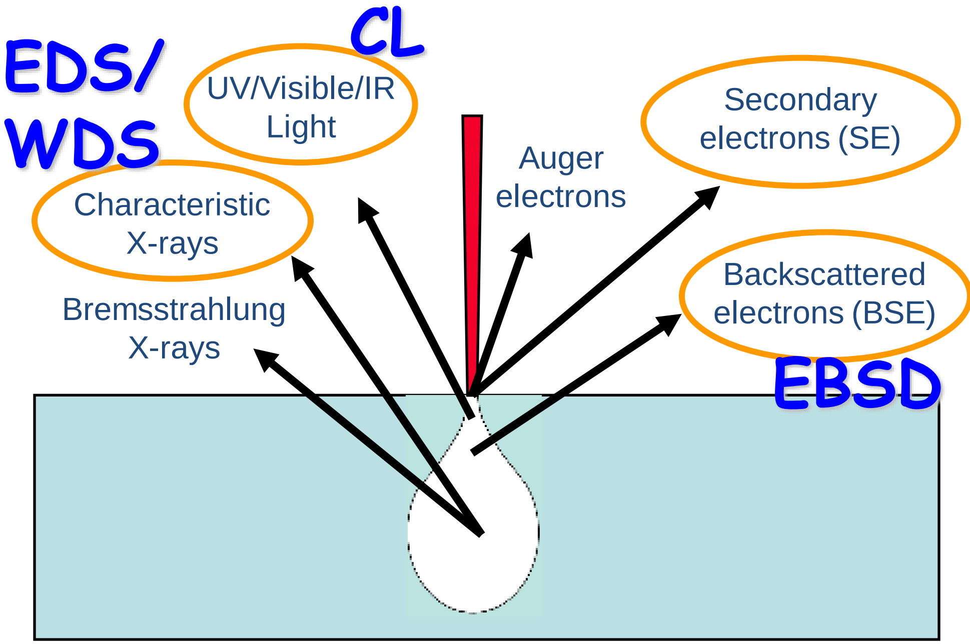
Dist. 463.2 um
Height 470.3 um
Length 0.0 um
Slant 0.0

Height List 0

For a review and assessment of the technique see:
F. Marinello et.al., Critical factors in SEM 3D stereo microscopy, Meas. Sci. Technol. **19 (2008) 065705 (12pp)**

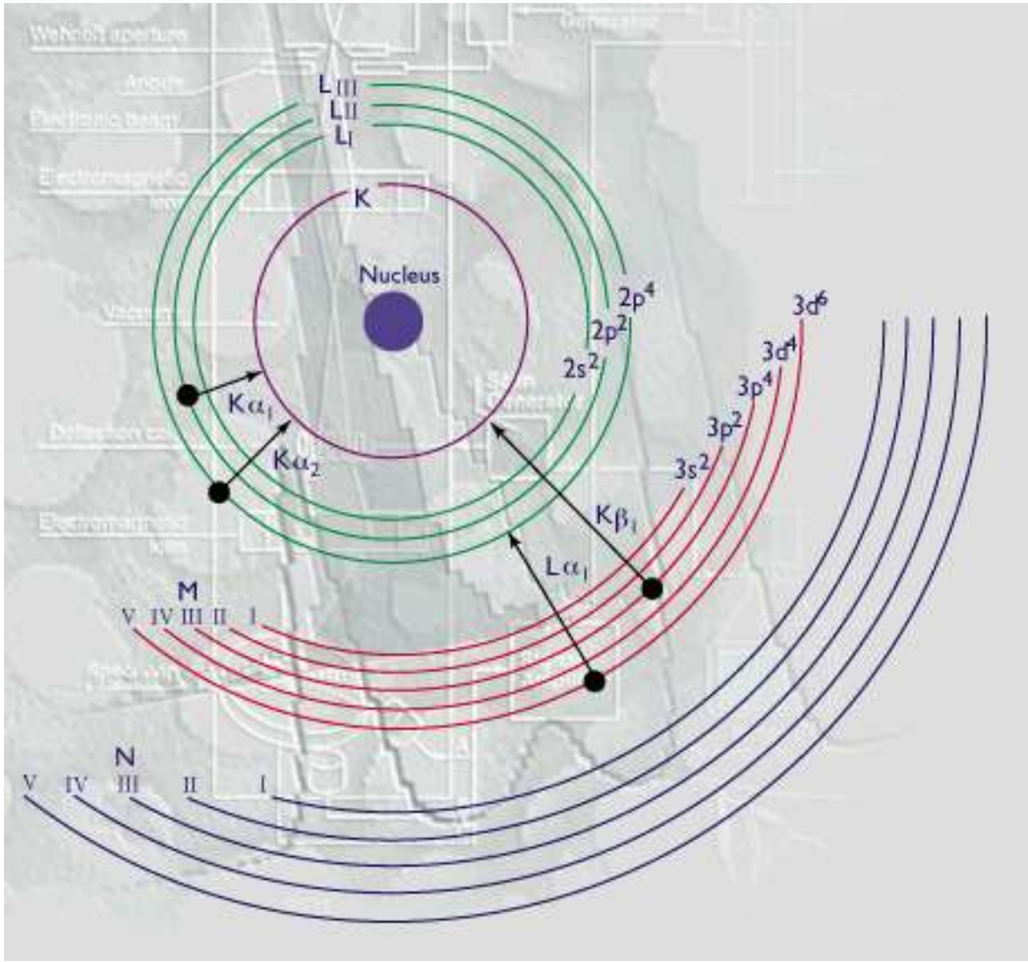
JEOL JSM-7000F Analytical Scanning EM





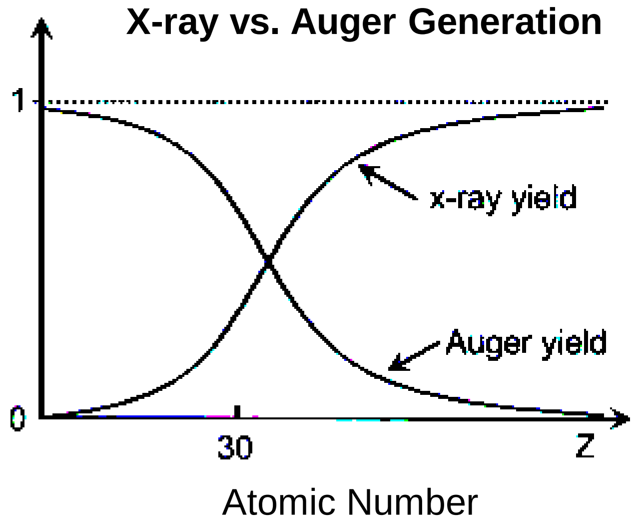
- Energy-Dispersive Spectroscopy (EDS) – solid state detector simultaneously measures all energies of X-ray photons.
- Wavelength Dispersive Spectroscopy (WDS) – sequentially measures intensity *vs* X-ray wavelength (energy). Superior energy resolution and detection limits (P/B ratio).
- Electron Backscattered Diffraction (EBSD) – acquires electron diffraction information from surface of highly tilted bulk sample with lateral resolution of low 10’s of nm.
- Cathodoluminesence (CL) – optical emission spectrometer and imaging system for 300-1,700nm. Liquid He cooled stage module.

Characteristic X-ray Generation

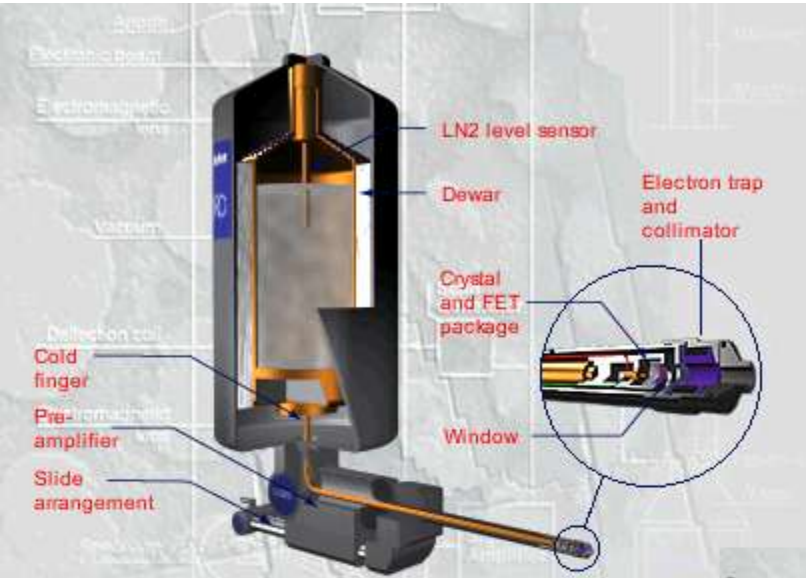


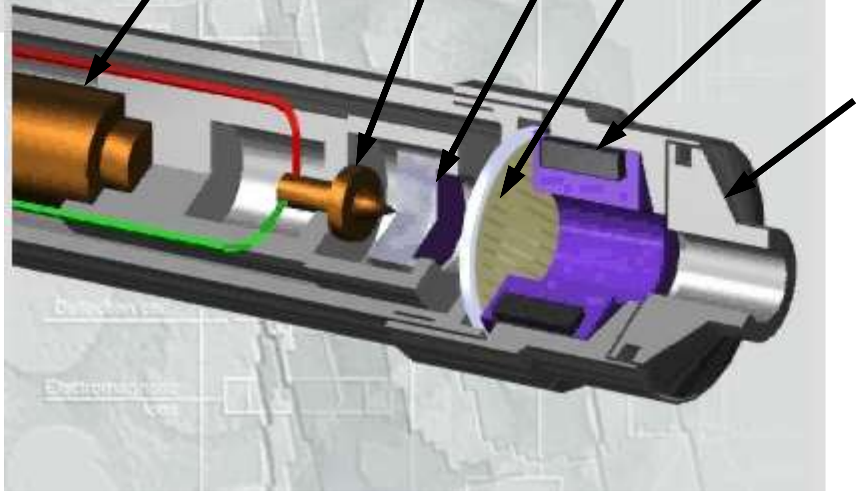
Graphic from, *The Oxford Guide to X-Ray Microanalysis*, Oxford Instruments Microanalysis Group

- 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.



Energy Dispersive X-ray Detector: Si(Li)





Copper Rod (at Liq. N_2 Temperature)

FET charge sensitive amplifier

Si(Li) Detector

X-ray Window

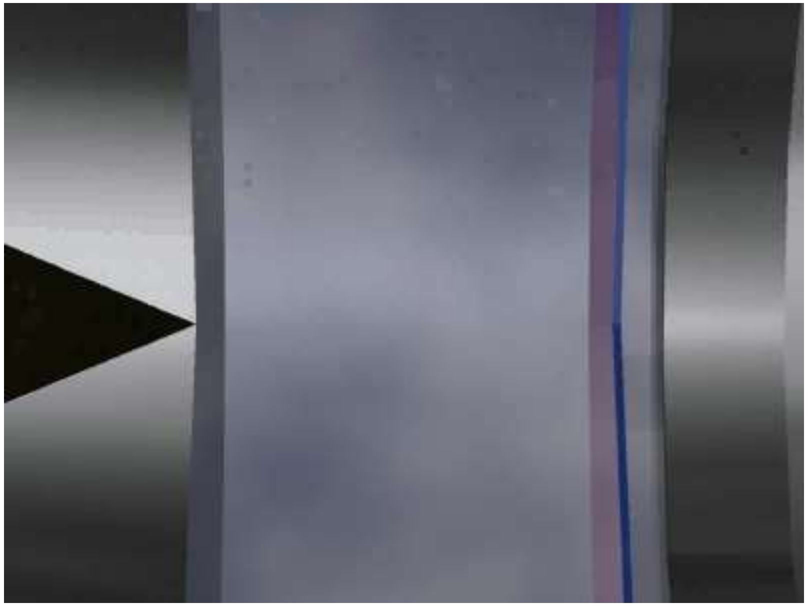
Magnetic Electron Trap

Collimator

Other technologies now available: Si drift (SDD) and microcalorimeter detectors

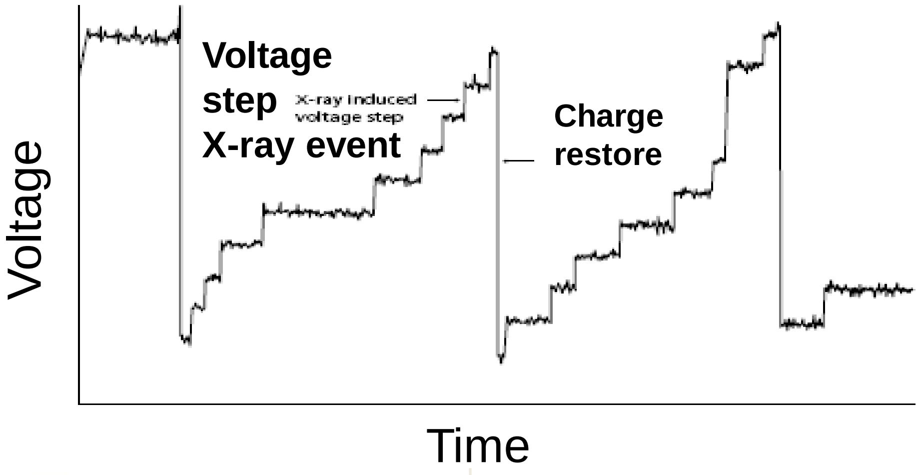
Graphics from *A Guide to X-Ray Microanalysis*, Oxford Microanalytical Instruments

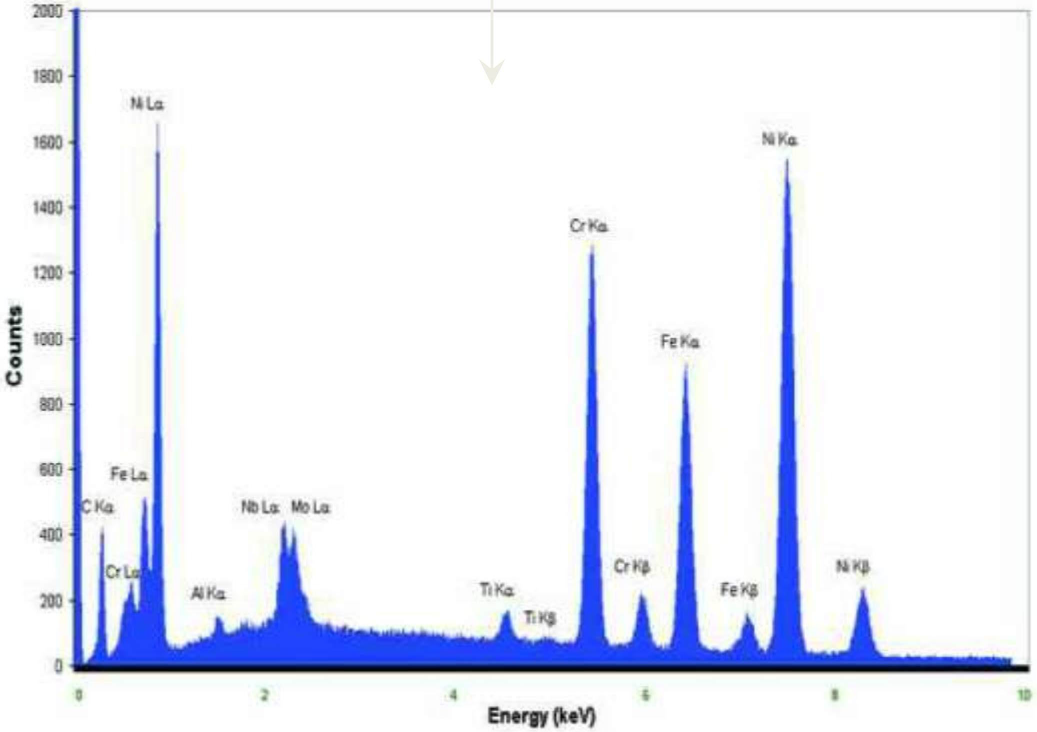
Mechanism of X-ray Energy Determination



Animations from, *The Oxford Guide to X-Ray Microanalysis*, Oxford Instruments Microanalysis Group

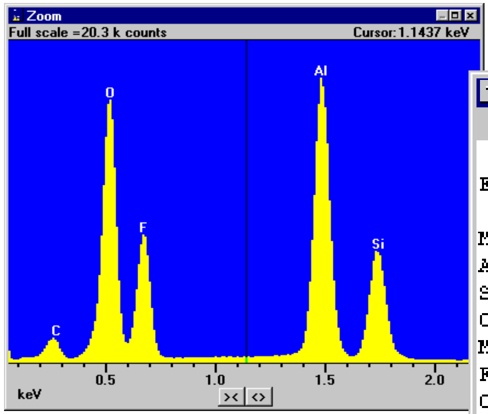
- **X-ray loses energy through inelastic scattering events creating electron / hole pairs**
- **High voltage bias keeps generated pairs from re-combining**
- **Charge sensitive amplifier "counts" pairs generated by X-ray**
- **Spectrometer calibration effectively multiplies by energy/pair (3.8 eV) to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

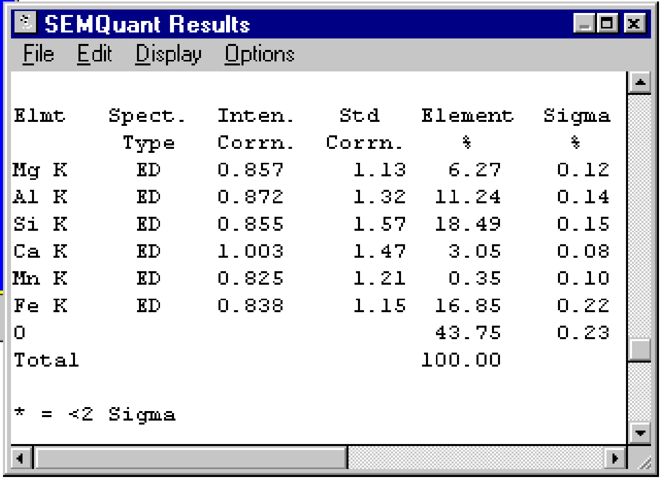




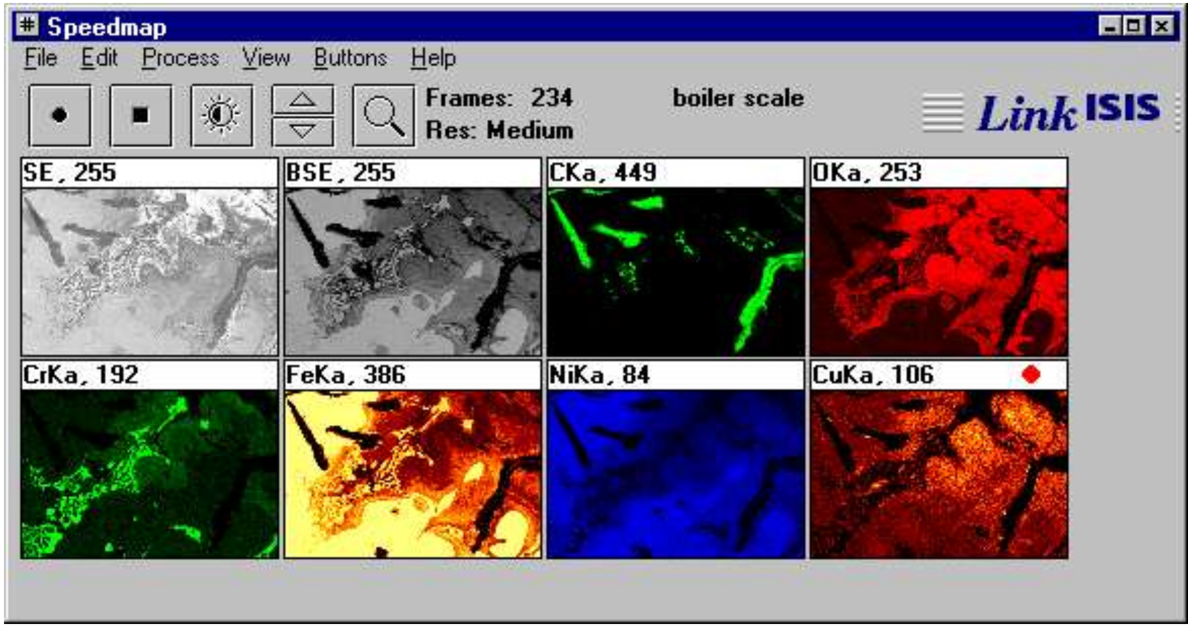
X-ray EDS Microanalysis in the SEM

- **Fast Parallel Detection**
- **Qualitative elemental analysis**
  - **From Beryllium up on periodic table**
  - **Sensitivities to <0.1 wt.% depending on matrix and composition**
- **Quantitative analysis**
  - **Many requirements / Limitations**
- **Digital elemental distribution imaging and line-scans, full spectrum imaging**
- **Analysis of small volumes, from order of μm^3 to << 1 μm^3 depending on accelerating voltage, element analyzed, and matrix**
- **~130 eV Mn K-alpha resolution typical for Si(Li) detector**





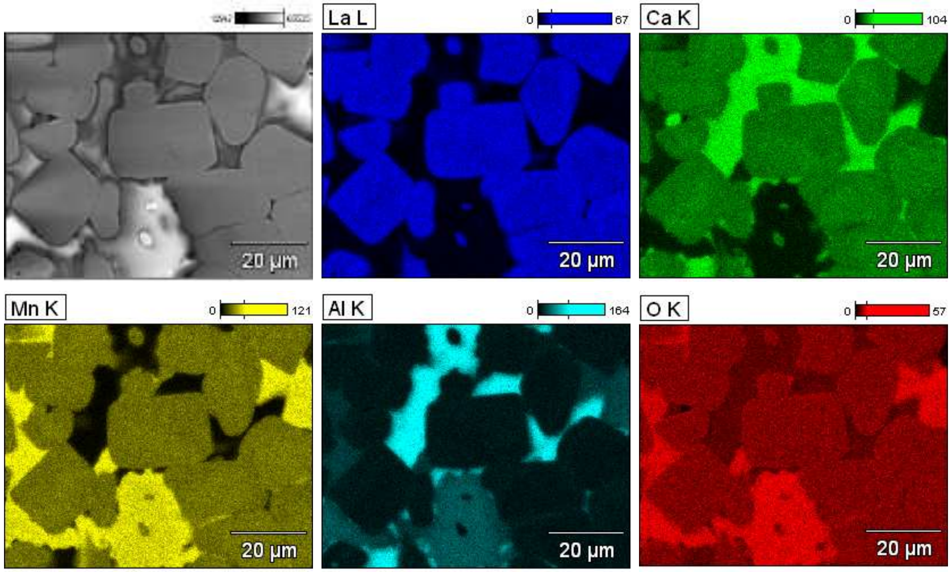
```
Elmt    Spect.   Inten.    Std    Element   Sigma
         Type    Corrn.   Corrn.     %        %
Mg K      ED     0.857     1.13     6.27     0.12
Al K      ED     0.872     1.32    11.24     0.14
Si K      ED     0.855     1.57    18.49     0.15
Ca K      ED     1.003     1.47     3.05     0.08
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
```

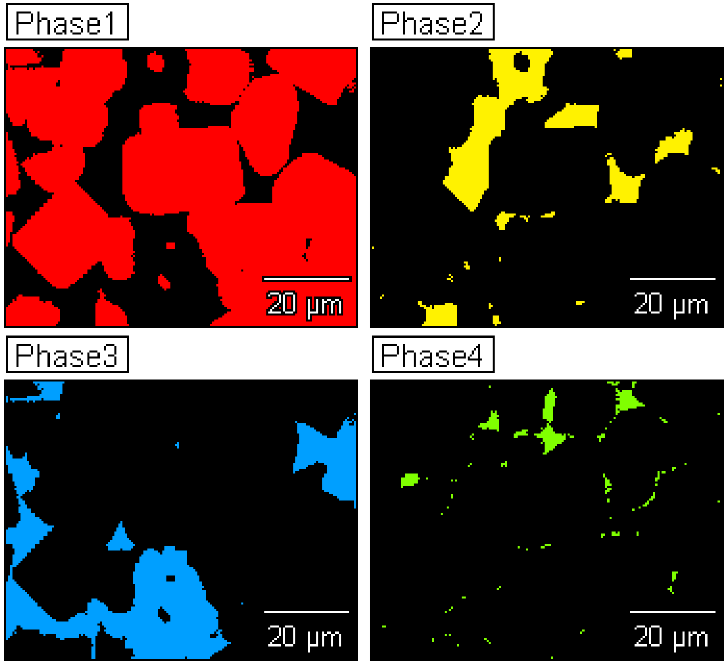


Frames: 234
Res: Medium
boiler scale

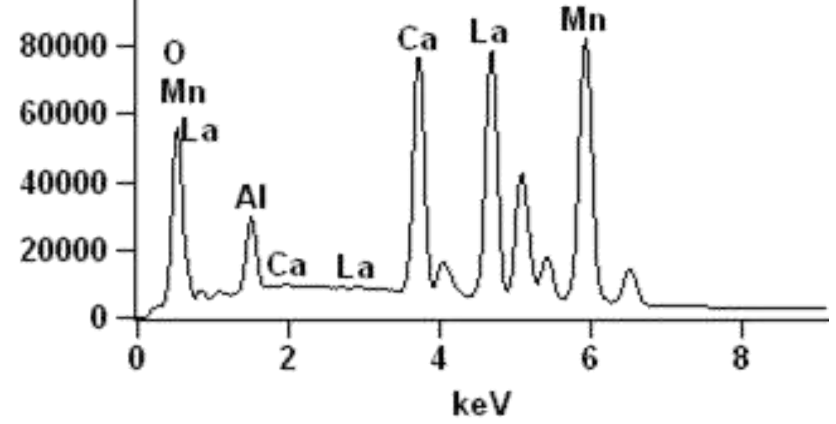
EDS Full Spectrum Imaging

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)

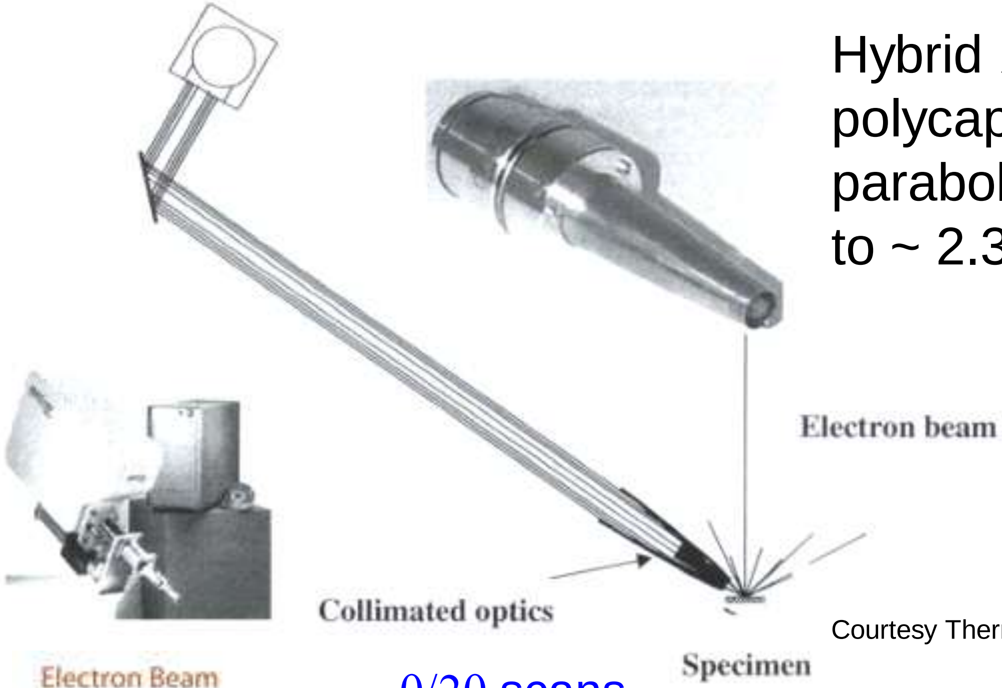


Element Line	*Weight %*	*Weight % Error*
O K	22.13 Stoichiometry	---
Al K	3.81	+/- 0.02
Ca K	10.26	+/- 0.04
Mn K	30.57	+/- 0.07
La L	33.23	+/- 0.09

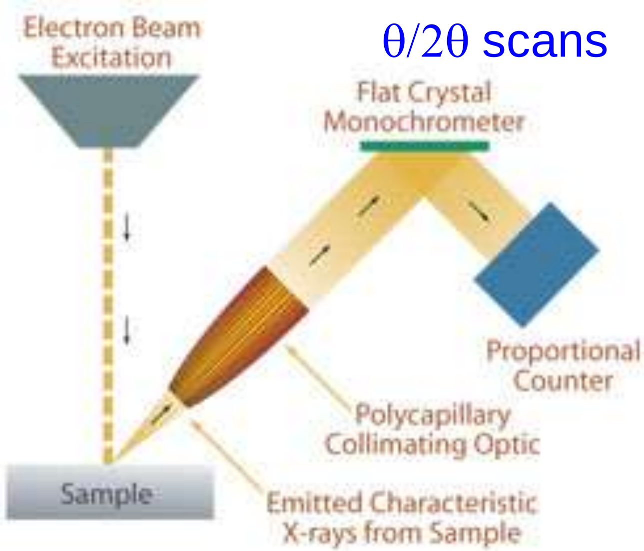
Thermo Instr.: Noran System Six

Parallel Beam Wavelength Dispersive Spectroscopy

Hybrid X-ray optics containing both a polycapillary optics (up to ~12 keV) and a paraboloidal grazing incidence optics (up to ~ 2.3 keV).



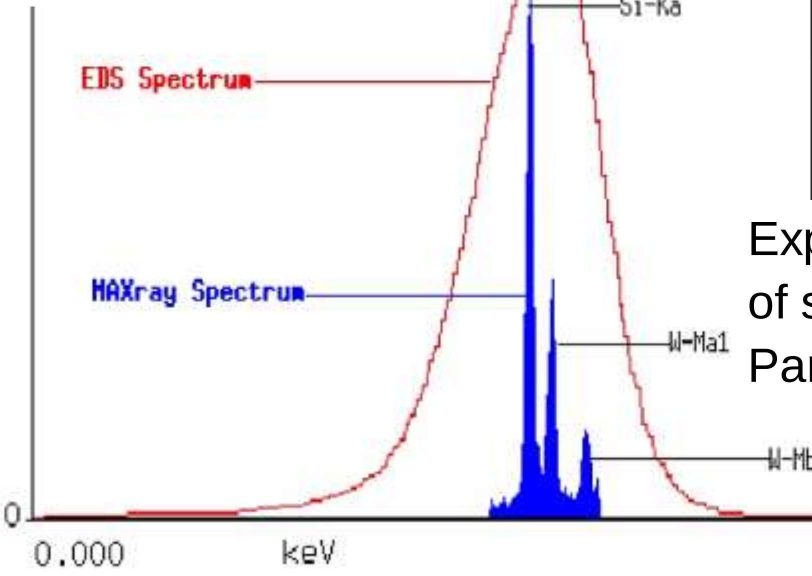
Comparison of EDS (SiLi) to Parallel Beam WDS (Thermo Instruments MaxRay)

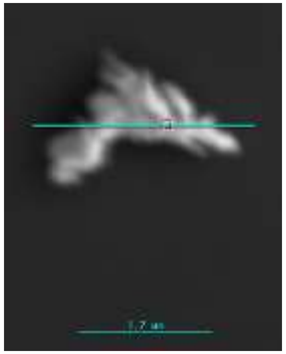


θ/2θ scans

X-ray Optical Systems, Inc.
http://www.xos.com/index.php/?page_id=71&m=2&sm=3

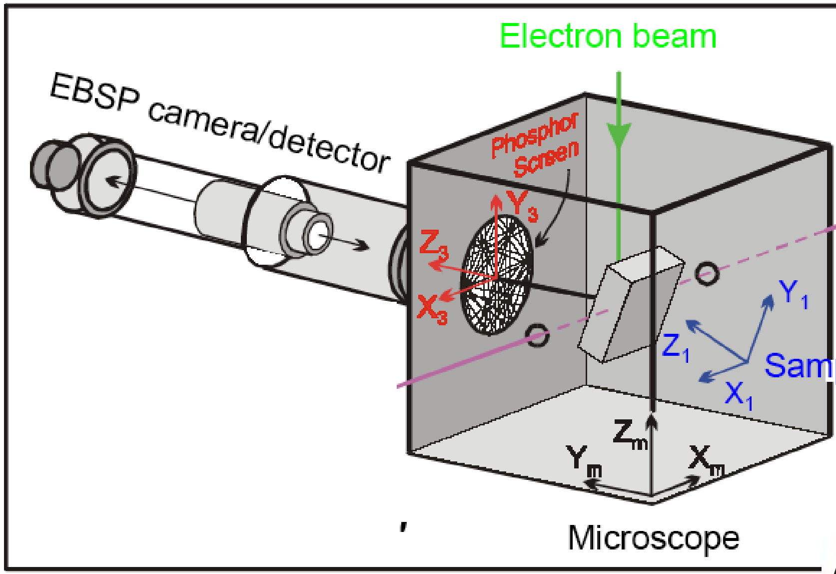
Courtesy Thermo Instruments

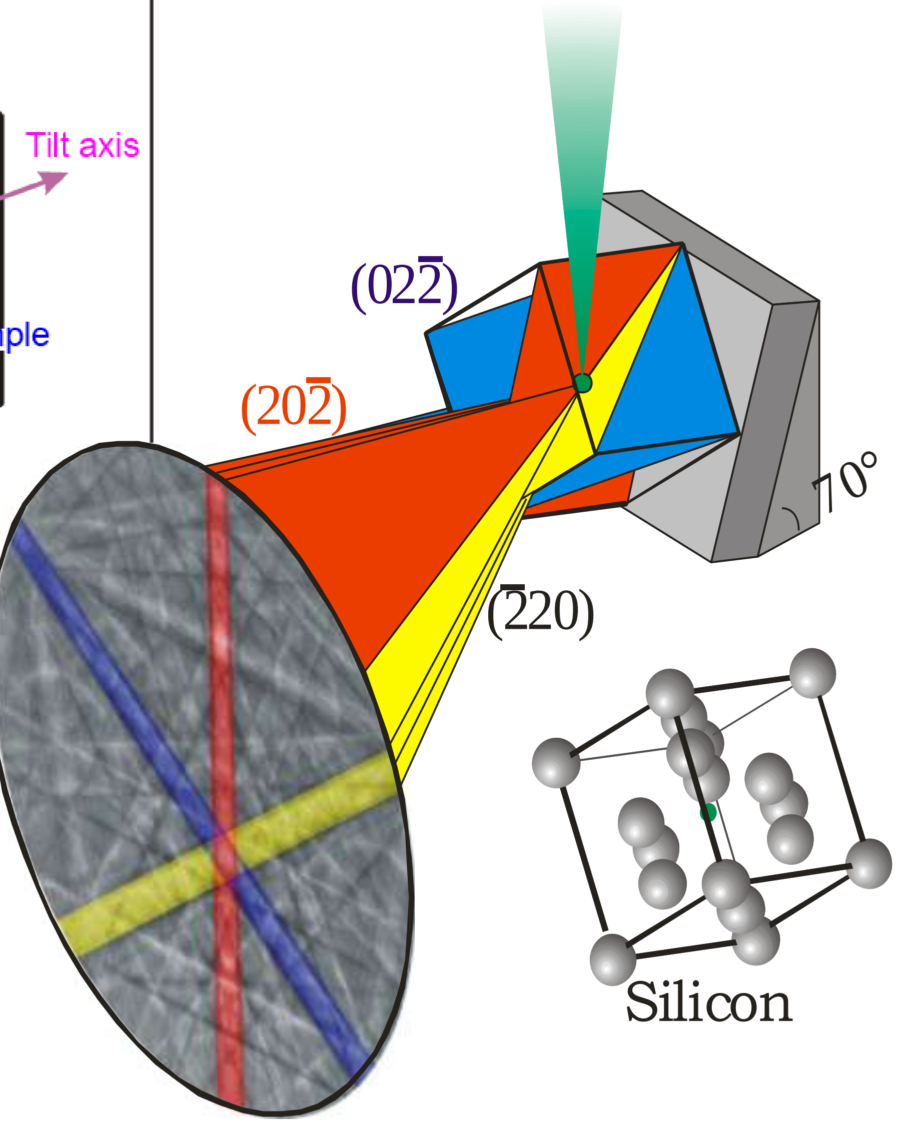




Expl.: Identification of sub-micron W Particle on Si

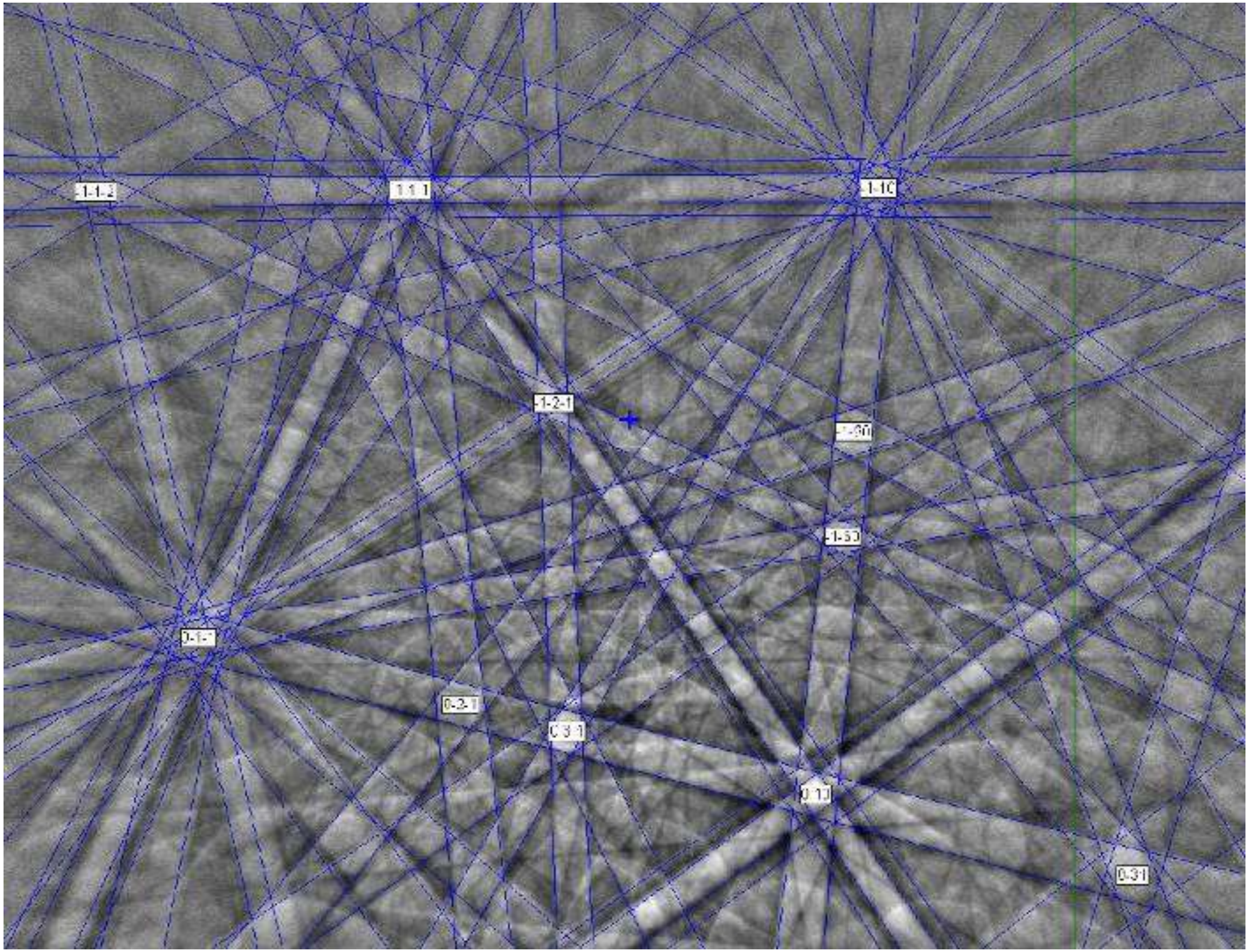
Electron Backscattered Diffraction in the SEM (EBSD)





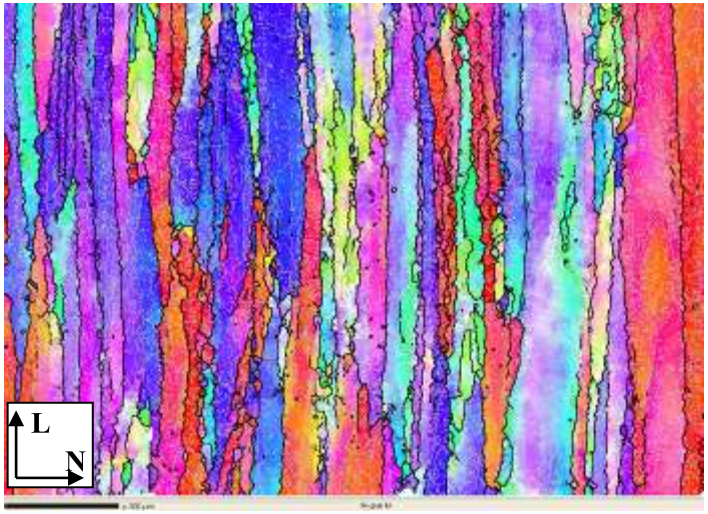
Courtesy HKL Technology (Oxford Instruments Microanalysis Group)

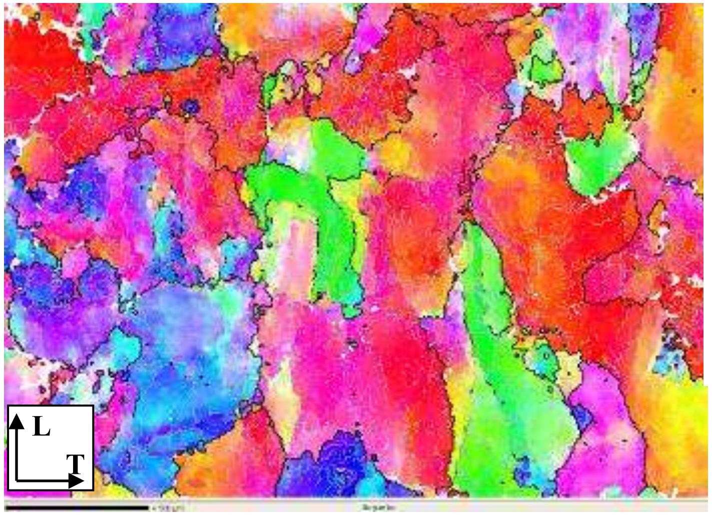
EBSD of GaAs Wafer

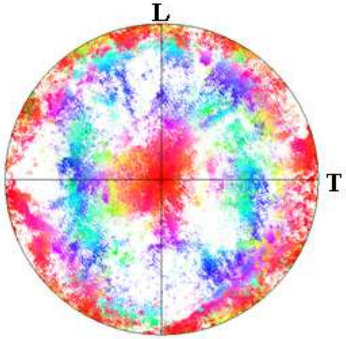


Microtexture in Al-Li Alloys for Future Aerospace Applications

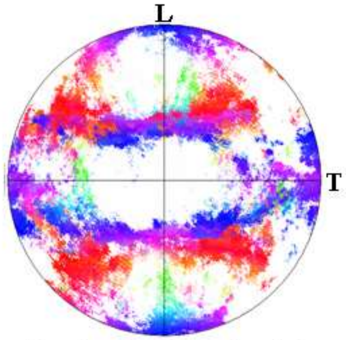
Investigation of crystallographic aspects grain morphology and delaminations



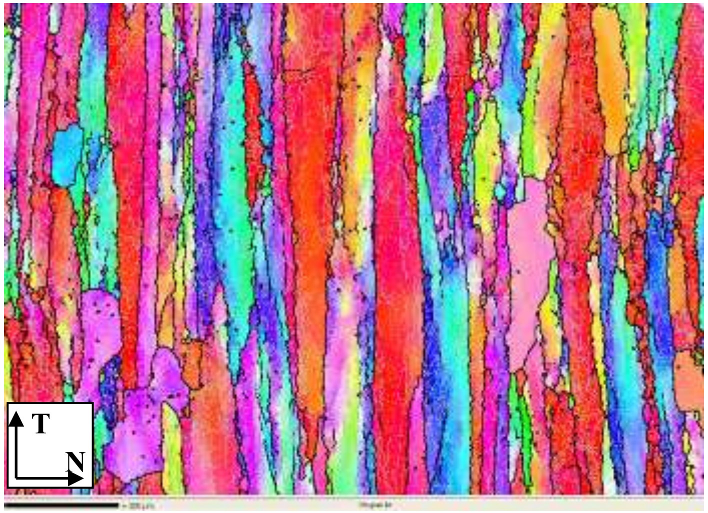


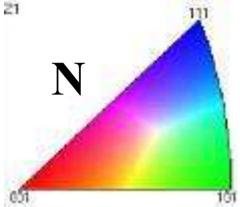


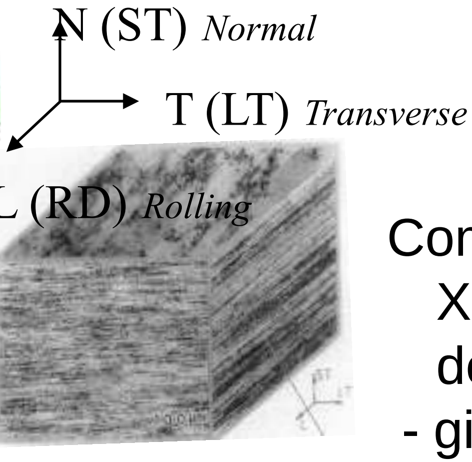
[100] Pole Figure (T/8)



[111] Pole Figure (3T/8)



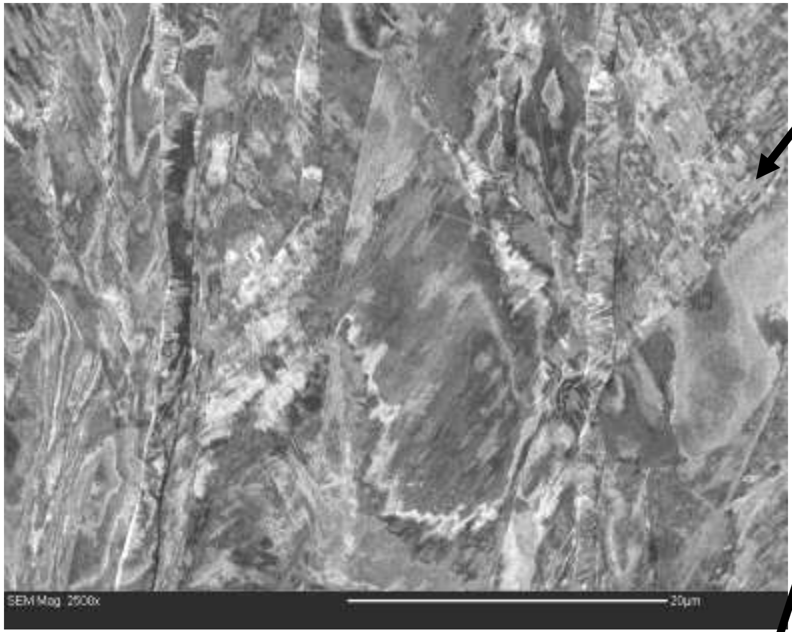




Complimentary to XRD texture determination
- gives **local texture** & **misorientations**

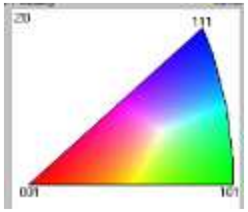
True Grain ID, Size and Shape Determination

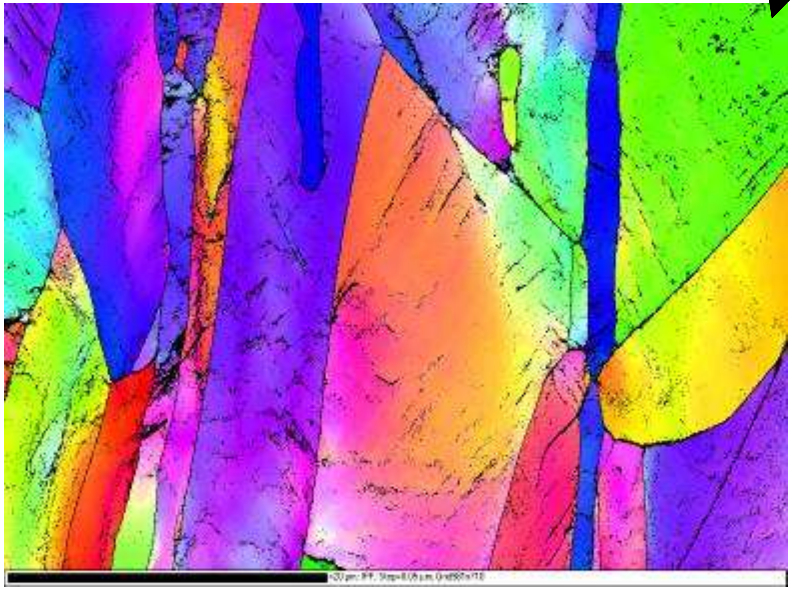
Phase Discrimination by EBSD



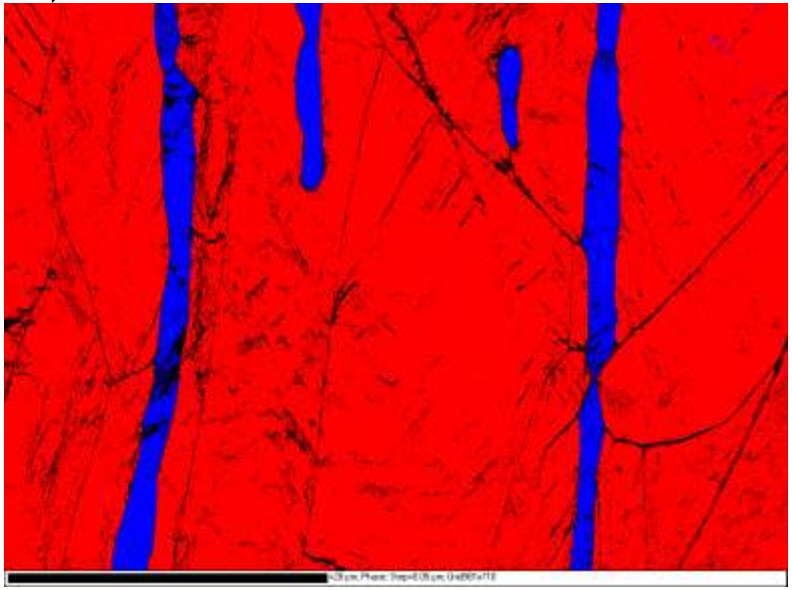
Forward Scatter image

Z-projected Inverse Pole Figure Image



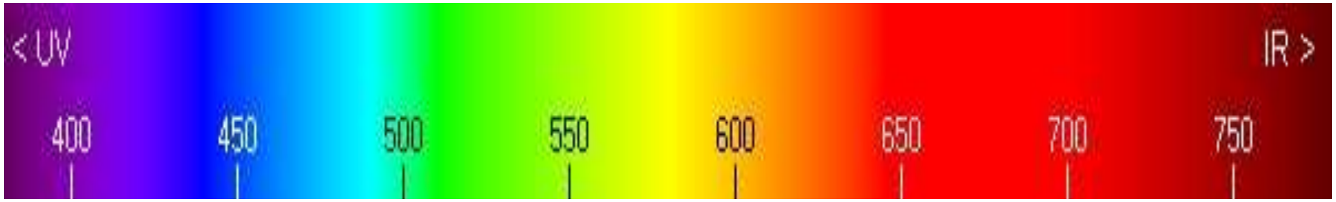


Phase Image (Stainless Steel)
Red = FCC iron
Blue = BCC iron



Cathodoluminesence (CL)

Emission of light from a material during irradiation by an energetic beam of electrons.

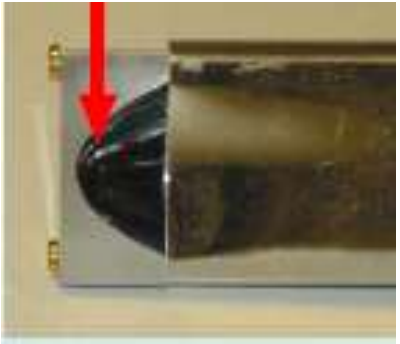


Wavelength (nm)

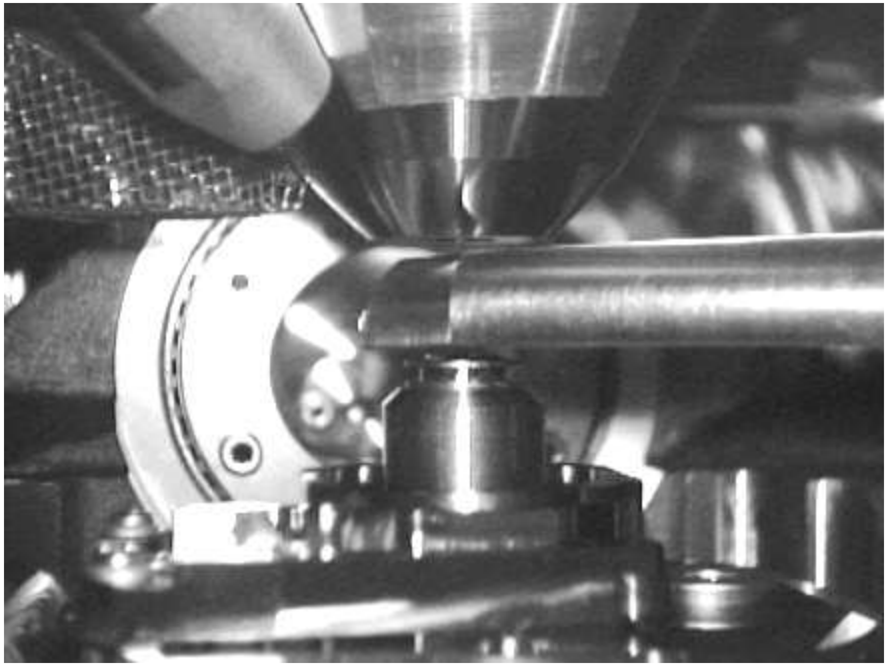
Collection of emitted light

Parabaloidal mirror placed immediately above sample (sample surface at focal point)

Aperture for electron beam

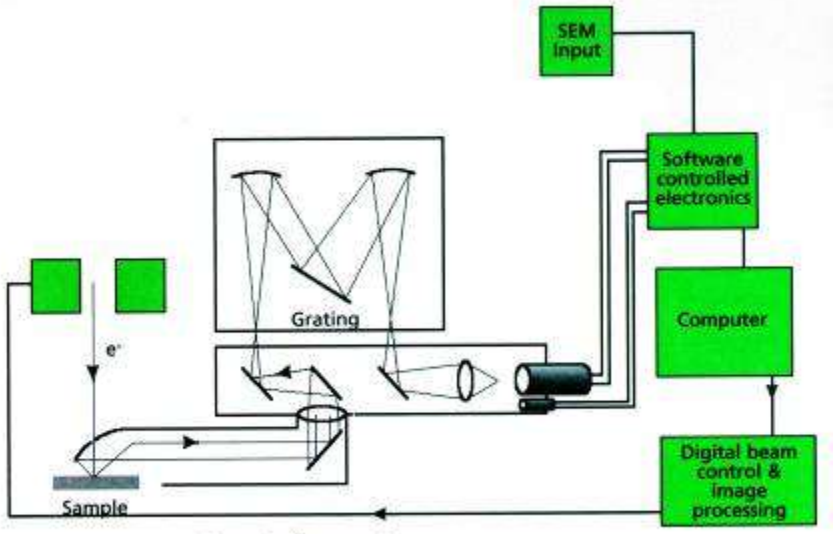


Collection optic in position between OL lens and sample



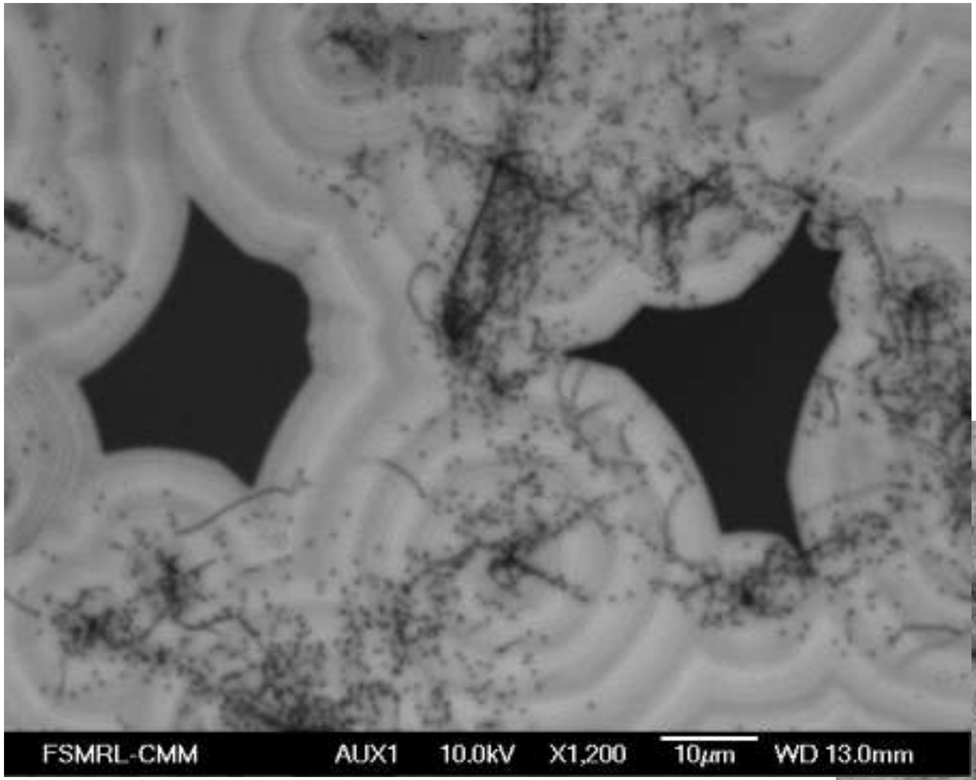
Cathodoluminesence Imaging and Spectroscopy

- **Optical spectroscopy from 300 to 1700 nm**
- **Panchromatic and monochromatic imaging** (spatial resolution - 0.1 to 1 micrometer)
- **Parallel Spectroscopy** (CCD) and full spectrum imaging
- **Enhanced spectroscopy and/or imaging with cooled samples (liq. He)**
- **Applications include:**
  - Semiconductor bulk materials
  - Semiconductor epitaxial layers
  - Quantum wells, dots, wires
  - Opto-electronic materials
  - Phosphors
  - Diamond and diamond films
  - Ceramics
  - Geological materials
  - Biological applications
  - Plasmonic Structures

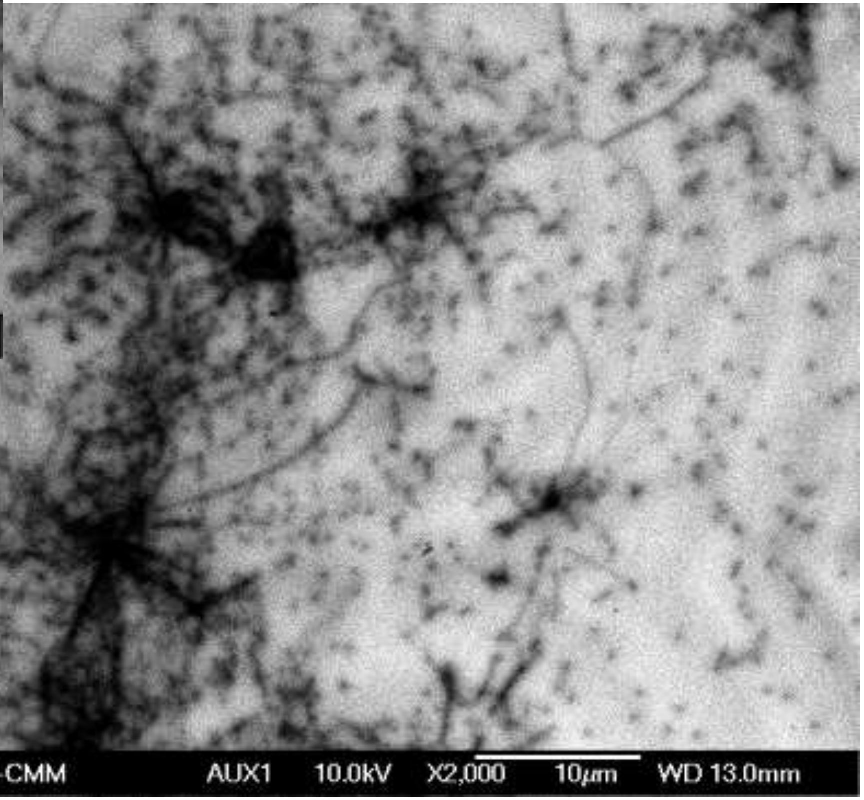


Schematic diagram of MonoCL 2

Pan-Chromatic CL imaging of GaN

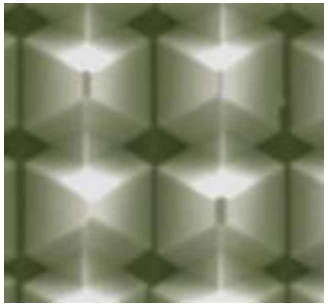


Defects (dislocations) are observed as points or lines of reduced emission; act as e-h pair traps with non-radiative recombination.

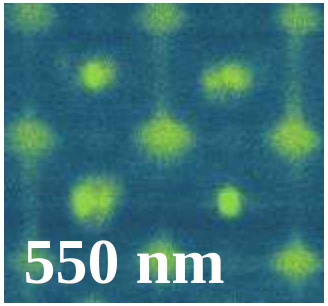


Monochromatic CL imaging of GaN Pyramids

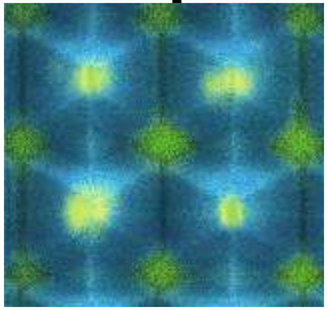
SEM

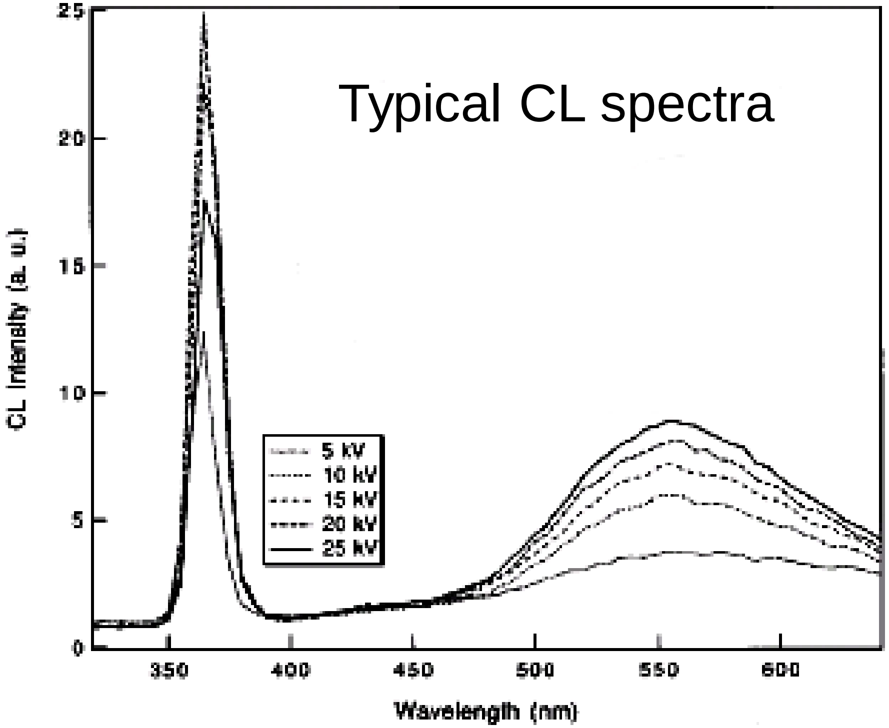


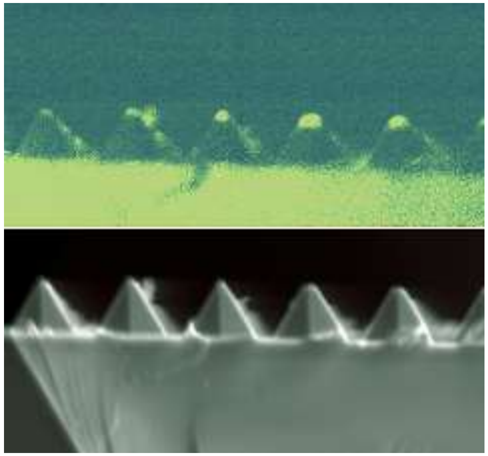
CL Image



composite







CL imaging of cross-sectional view

SEM

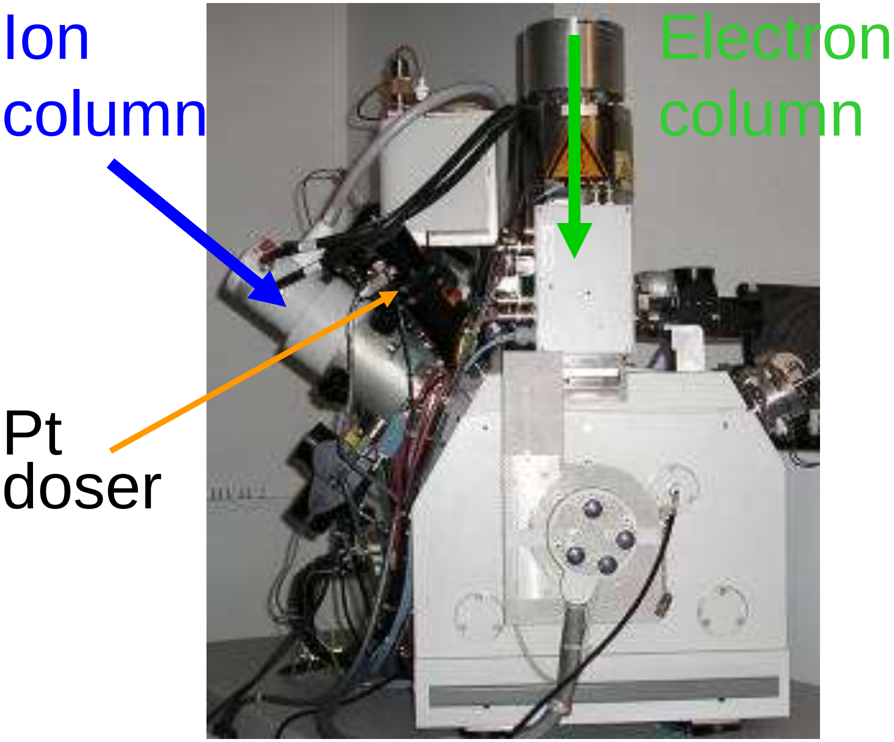
The strongest yellow emission comes from the apex of the elongated hexagonal structure.

Results courtesy of Xiuling Li , Paul W. Bohn, and J. J. Coleman, UIUC

Summary: Scanning Electron Microscopy

- Remarkable depth of focus
- Imaging from millimeters to a sub-nanometer
- Chemical composition with 0.1-1 µm resolution
- Crystallography using electron EBSD
- Optical properties on a micrometer scale (via CL)

Dual-Beam Focused Ion Beam (DB235-FIB)

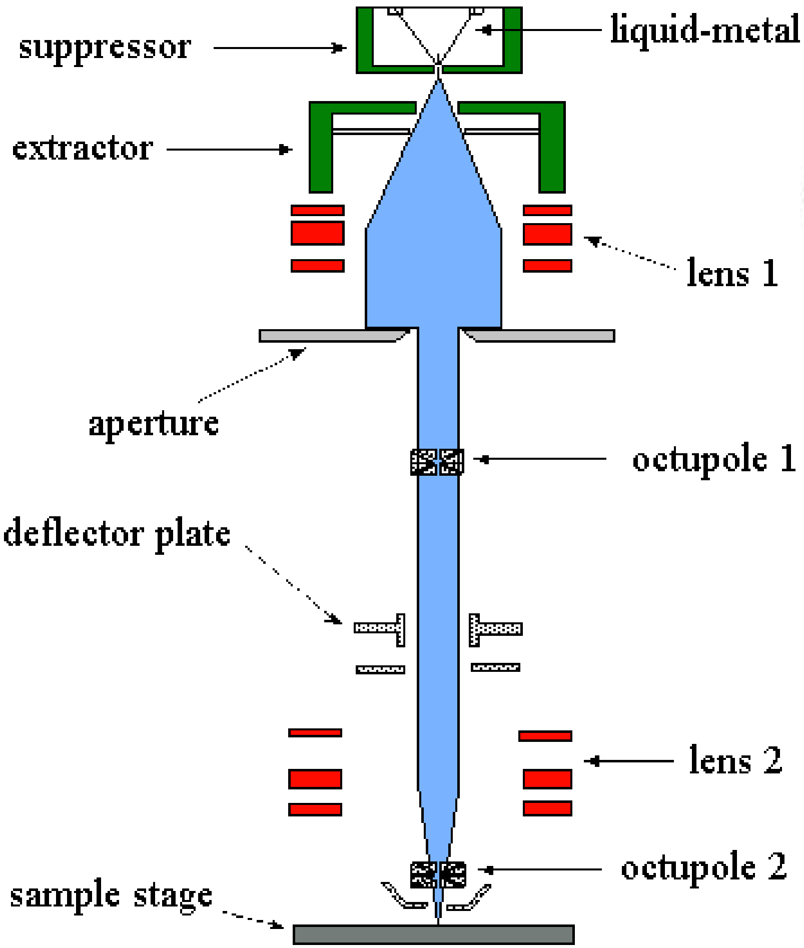


The **FEI Dual-Beam DB-235 Focused Ion Beam and FEG-SEM** has a high resolution imaging (7nm) **Ga$^+$ ion column** for site-specific cross-sectioning, TEM sample preparation, and nano-fabrication. The **Scanning Electron Microscope** (SEM) **column** provides high resolution (2.5 nm) imaging prior to, during, and after milling with the ion beam. The instrument is equipped with beam activated Pt deposition and 2 *in-situ* nano-manipulators: **Omniprobe** for TEM sample preparation and **Zyvex** for multiprobe experiments.

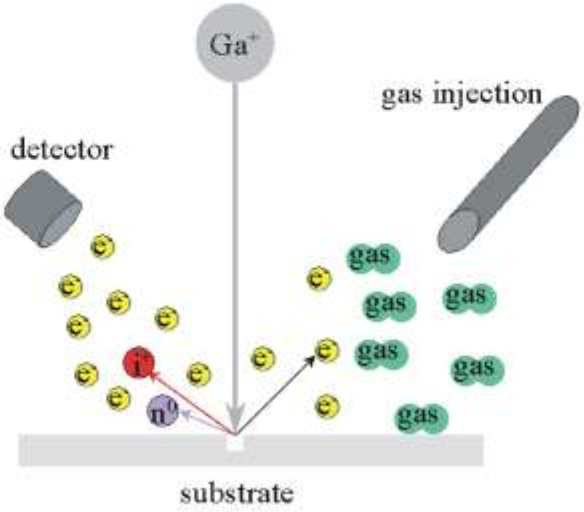
- site –specific cross-sectioning and imaging
  - * Serial sections and 3-D reconstruction are an extension of this method
- site –specific preparation of specimens for Transmission Electron Microscopy (TEM)
- site –specific preparation of specimens for EBSD
- nano-fabrication (micro-machining and beam-induced deposition)
- modification of electrical routing on semiconductor devices
- failure analysis
- mask repair

The Focused Ion Beam (FIB) Column

Layout of the
Focused Ion Beam column



Liquid Metal Ion Source



The gallium ion beam hits the specimen thereby releasing secondary electrons, secondary ions and neutral particles (e.g. milling).

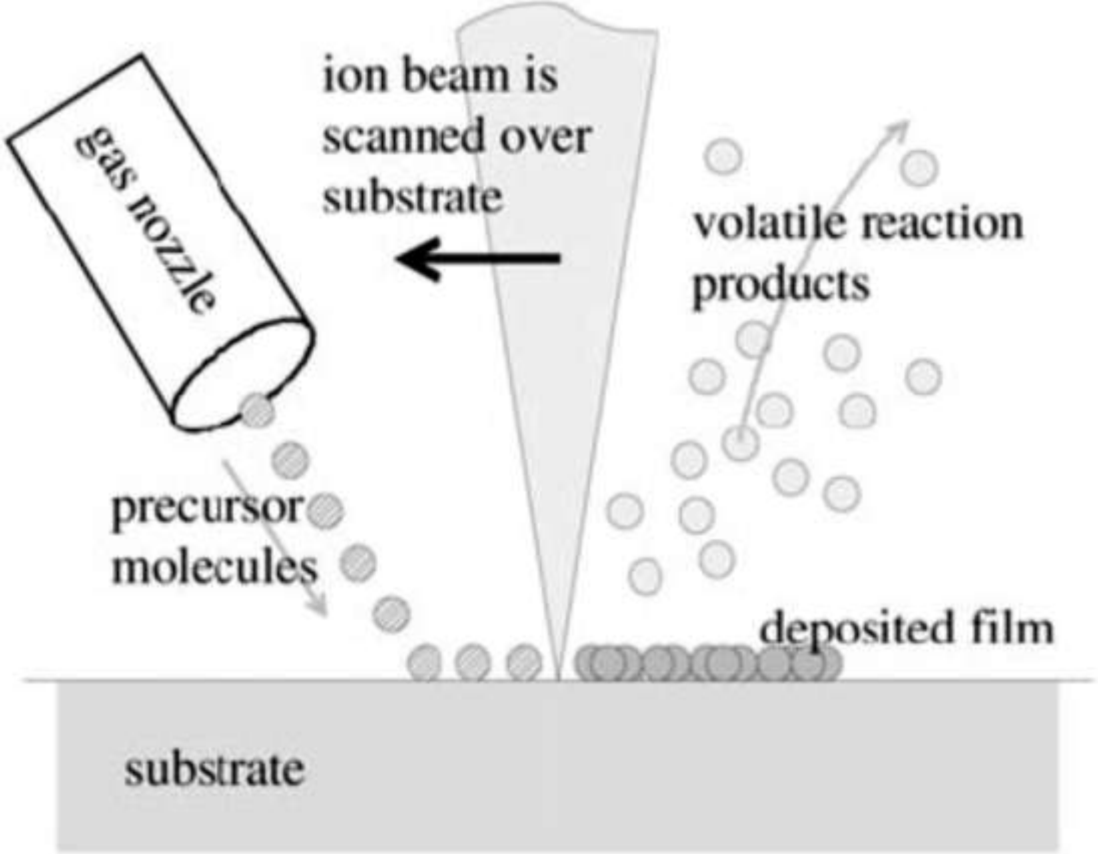
The detector collects secondary electrons or ions to form an image.

For deposition and enhanced etching: gases can be injected to the system.

Ion/Electron Beam Induced Deposition

IBID and EBID

- Precursor molecules adsorb on surface.
- Precursor is decomposed by ion or electron beam impinging on surface.
- Deposited film is left on surface.
- Volatile reaction products are released.

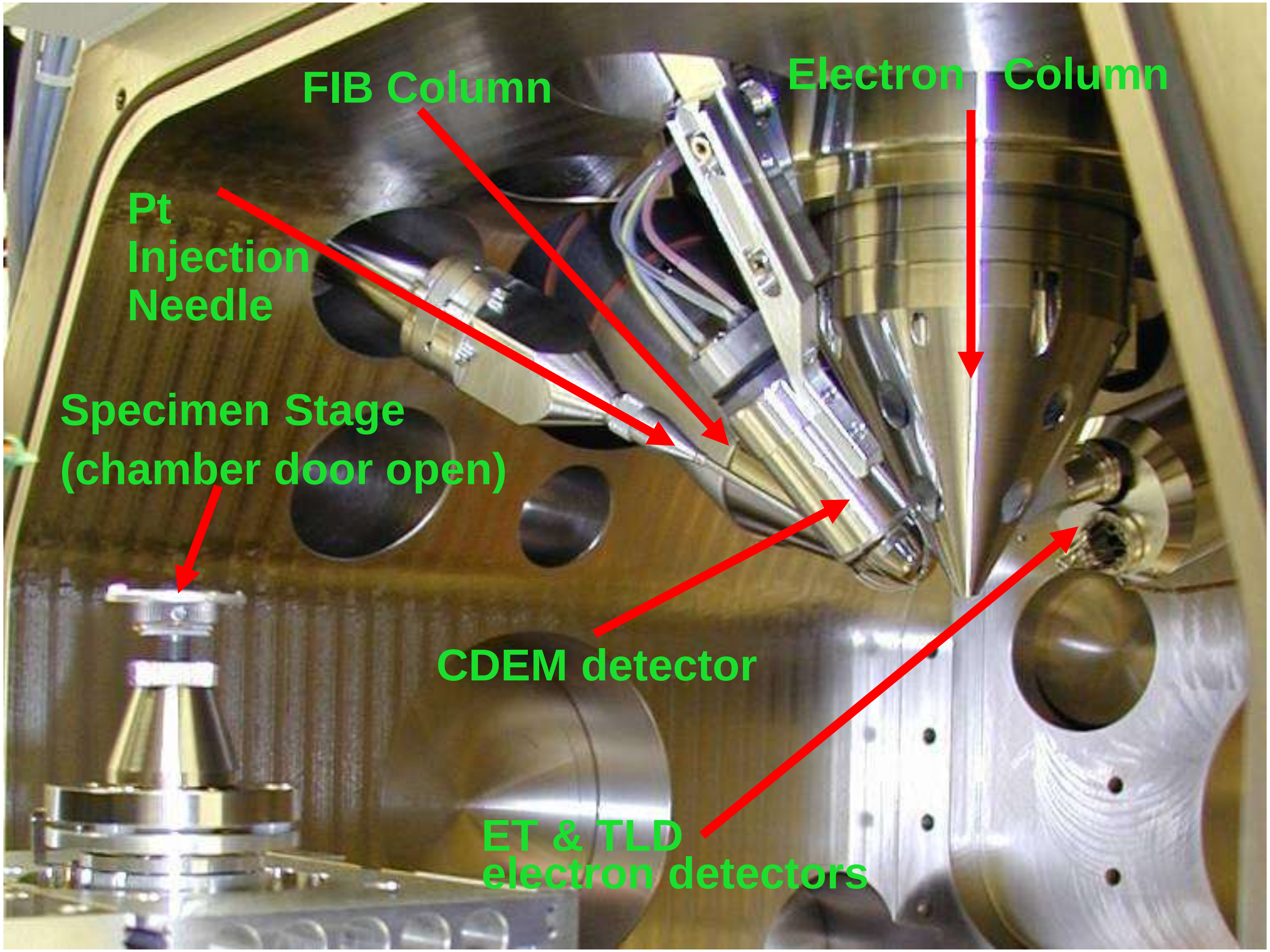


Pt, W, and Au are common metals

SiO_x can be deposited as an insulator

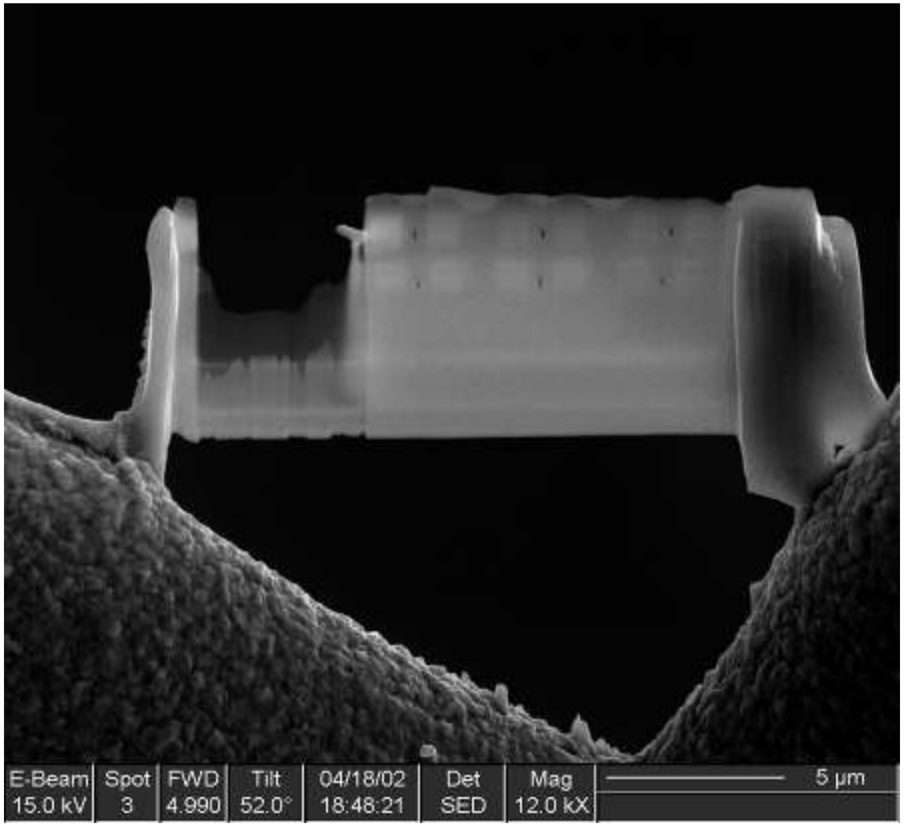
Similarly, reactive gases, can be injected for enhanced etching in milling and improving aspect ratio for milled features.

FEI DB235 - FIB Specimen Chamber

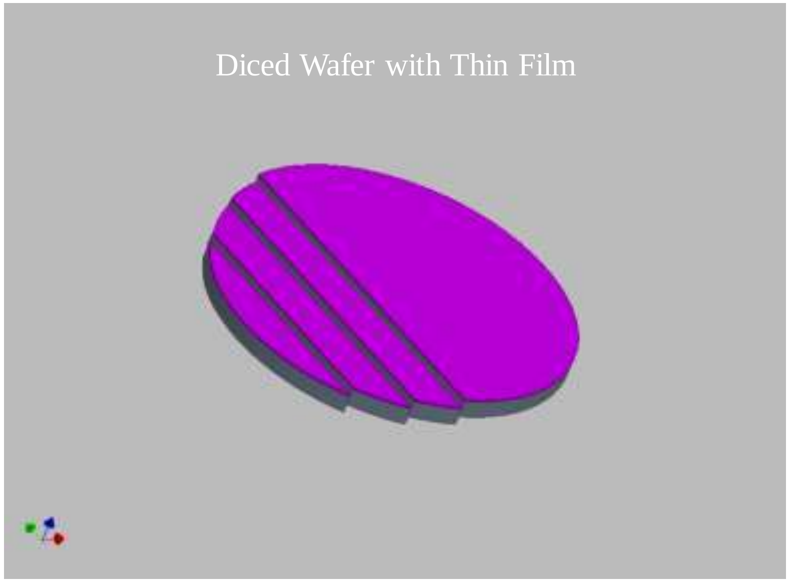


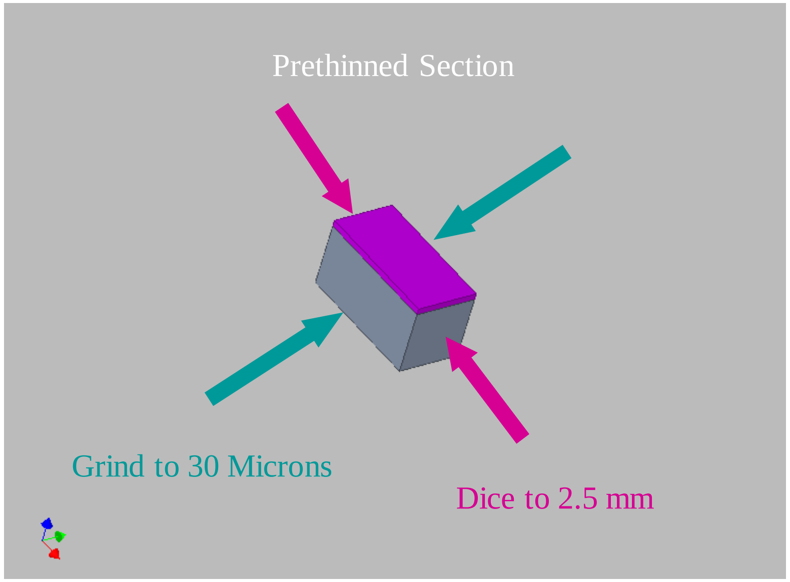
TEM Sample Preparation w/ Omniprobe

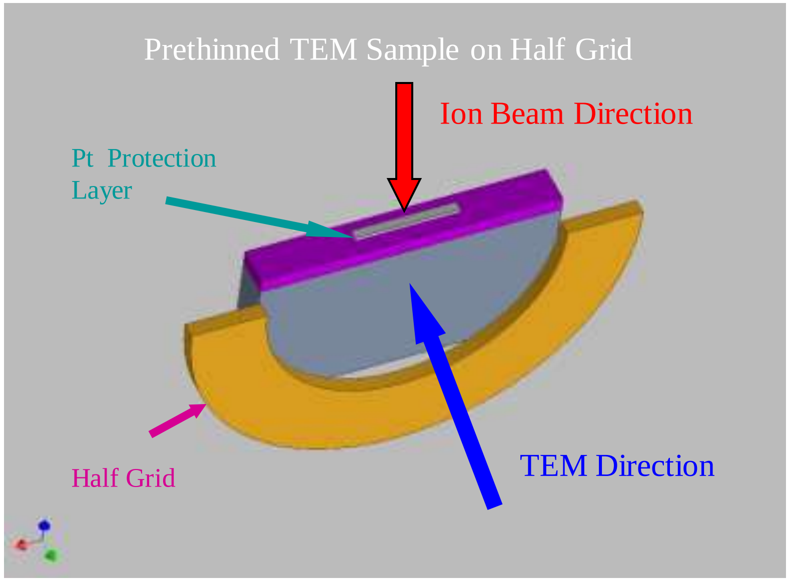
- Step 1 - Locate the area of interest (site – specific)
- Step 2 - Deposit a protective platinum layer
- Step 3 - Mill initial trenches
- – e-beam view after Step 3
- Step 5 - Perform “frame cuts“ and “weld" manipulator needle to sample
- Step 6 – Mill to release from substrate and transfer to grid
- Step 7 – "Weld" sample to a Cu TEM half-grid and FIB cut manipulator needle free
- Step 8 - FIB ion polish to electron transparency

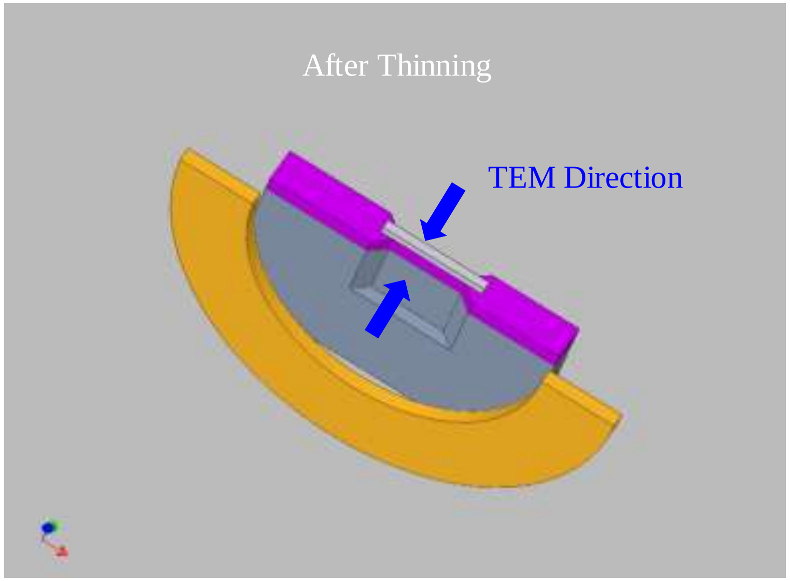


“Pre-Thinned” TEM Sample prepared by FIB



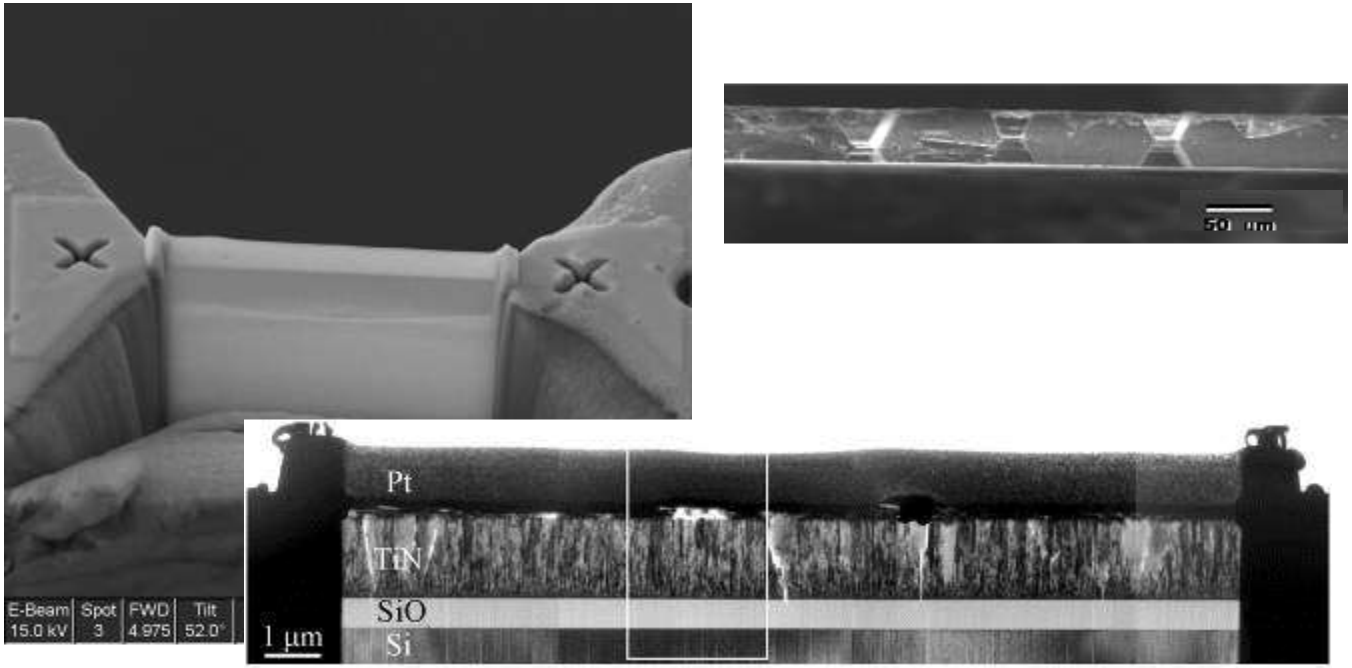


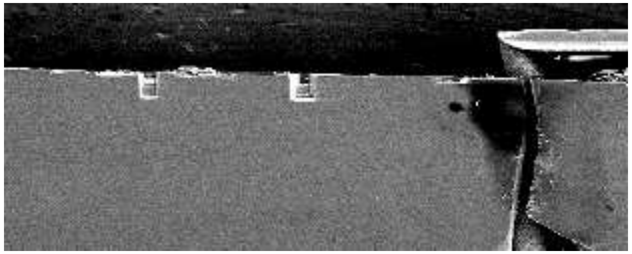


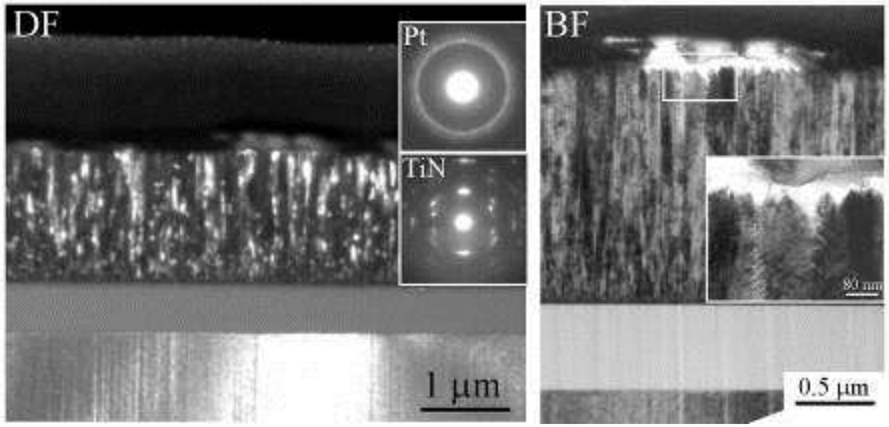


“Pre-Thinned” TEM Sample prepared by FIB

Drawing of typical “pre- thinned” specimen for FIB TEM sample preparation

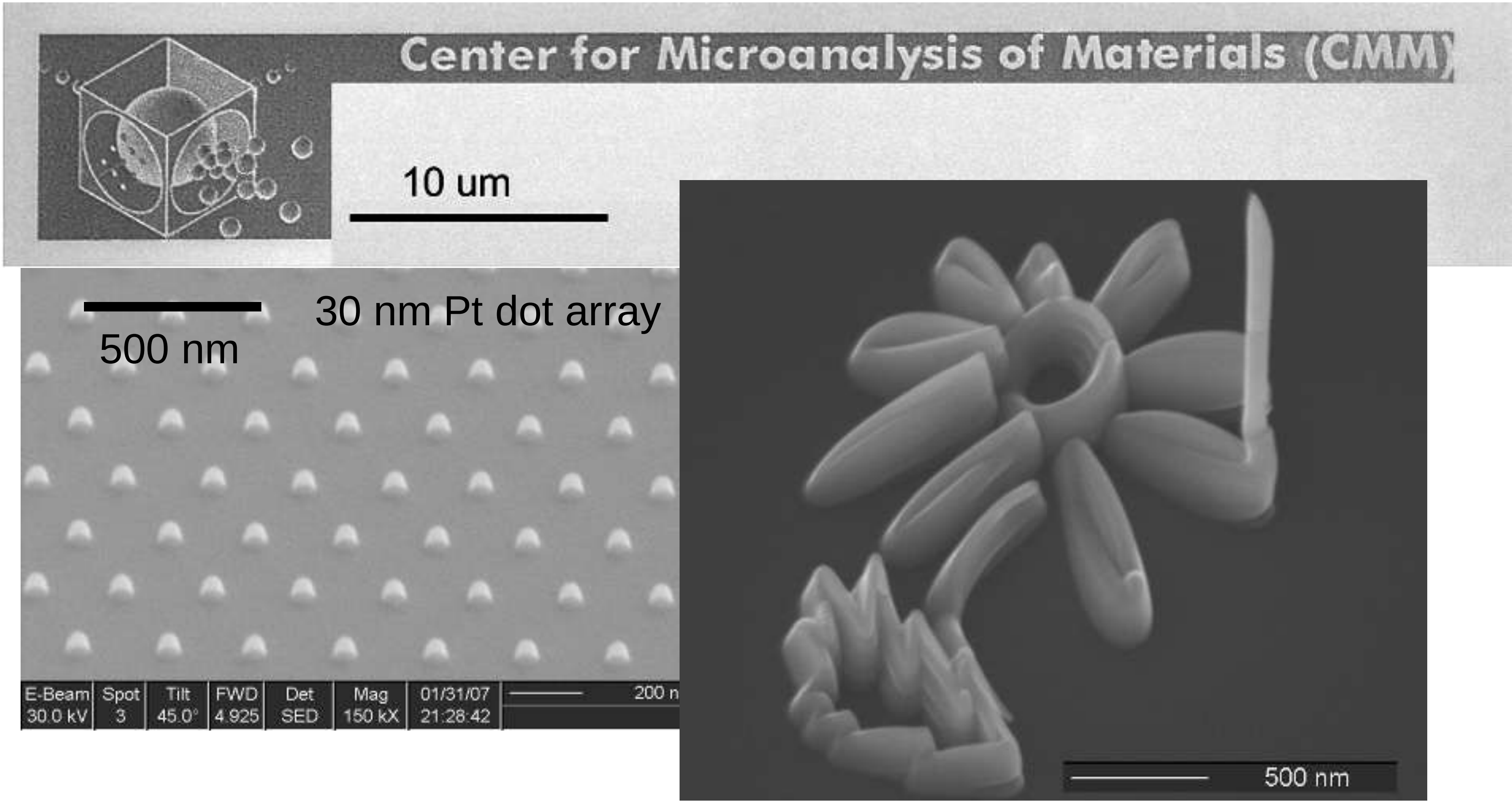




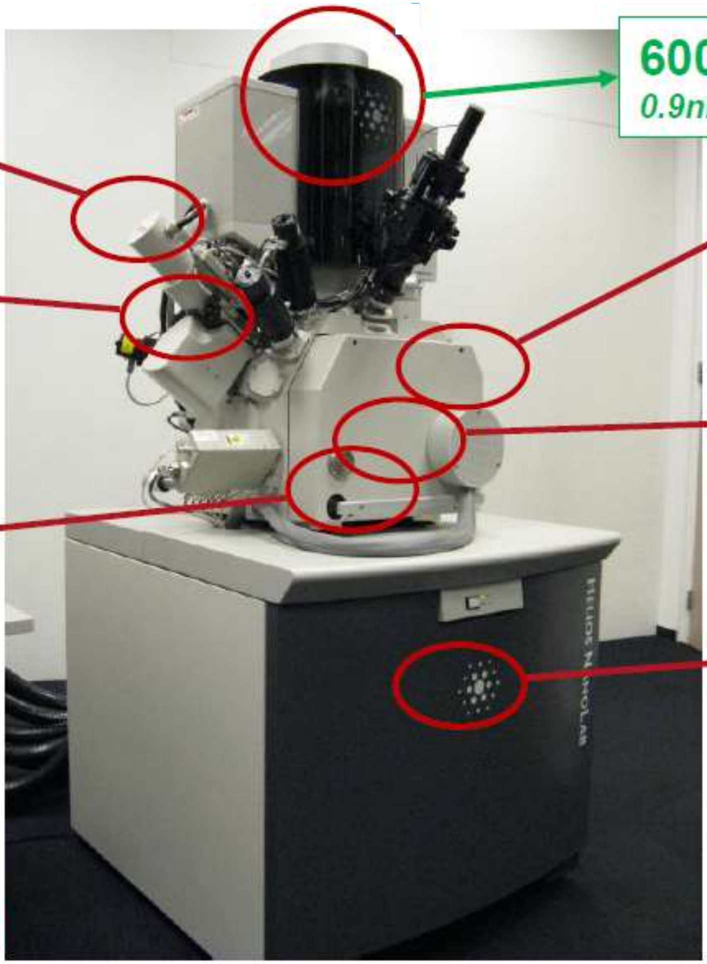


Precisely controlled etching and deposition

Etched or deposited structures using grey-scale bitmaps (more complex, parallel process) or scripting language (sequential, unlimited # of points).



Newly acquired DB-FIB: FEI Helios NanoLab 600i



Tomahawk FIB
2.5nm@30kV, 1pA-65nA
Differential Pumping &TOF

600i→Elstar Column
0.9nm @15kV, 1,4nm @1kV

ICE detector
Improved FIB imaging
Secondary I^+ or e^- Contrast

Advanced sample cleaning
Plasma Cleaner, CryoCleaner

Beam Deceleration

150mm high precision piezo stage

Standard 16bit scan/ pattern engine

+ new features:
- detection / scan strategies
- gas injection processes
- 3D analysis
- integrated CAD prototyping
- cryo

Summary: Focus Ion Beam Microscopy

- Site –specific cross-sectioning imaging and EBSD sample preparation
  - Serial sections and 3-D reconstruction are an extension of this method
- Site –specific preparation of specimens for transmission electron microscopy (TEM)
- Nano-fabrication (micro-machining and beam-induced deposition)
- Modification of the electrical routing on semiconductor devices

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