
# An Introduction to Scanning Electron Microscopy and Focused Ion Beam

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## Part I: SEM

- What is SEM & How does it work
  - Electron Guns (Electron Sources)
  - Electron Lenses and Lens Aberrations/Corrections
  - Major electron beam parameters and how to control them
- Applications
  - SE & BSE imaging and detectors
  - Analytical techniques: EDS, EBSD, CL, ECCI

## Part II: FIB

- How does FIB work
- Common applications

# What is SEM?

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

## Scanning Electron Microscope

- An instrument for observing and analyzing the surface microstructure of a bulk sample by using a finely focused beam of electrons
- SEMs can achieve 1-4 nm imaging resolution depending on the instrument
- The probe scans across the surface of the sample, in a raster scanning pattern

## Primary Applications

- **Surface topography/morphology**
- **Composition analysis**
- Crystallography
- Optical / Electronic properties
- and more …

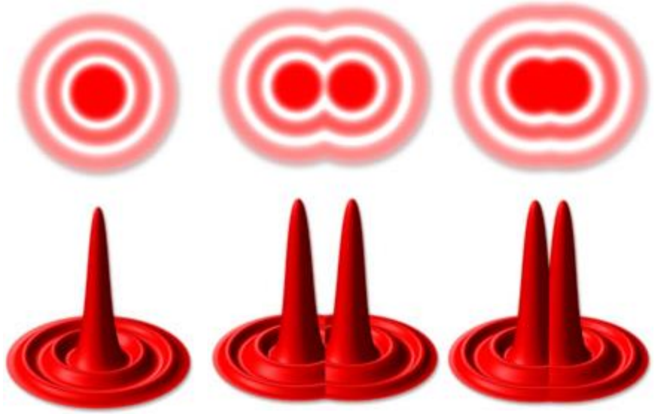
# Why Use Electrons Instead of Visible Light in a Microscope

## Resolution

- The ability to distinguish two very small and closely-spaced objects as separate entities
- It is determined primarily by wavelength of the light source according to Abbe's equation:

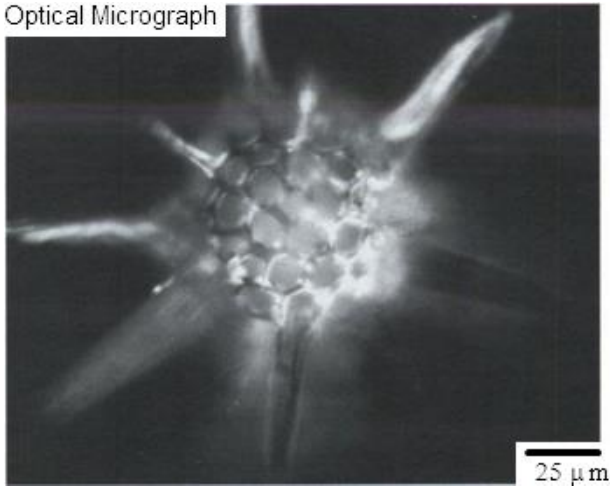
$$d = \frac{0.61\lambda}{n sin\alpha}$$

d – resolution (electron ~nm ; light 0.2um)
$\lambda$– wavelength of (electron $\lambda$=8.7pm@20KV; light spectrum 400-700nm)
n – refractive index relative to free space
$\alpha$– semi-angle in radians

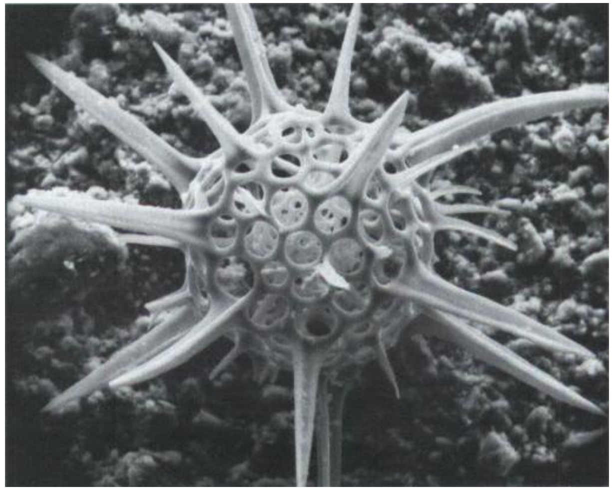


Airy Disk

zeiss-campus.magnet.fsu.edu/articles/basics/imageformation.html




Optical



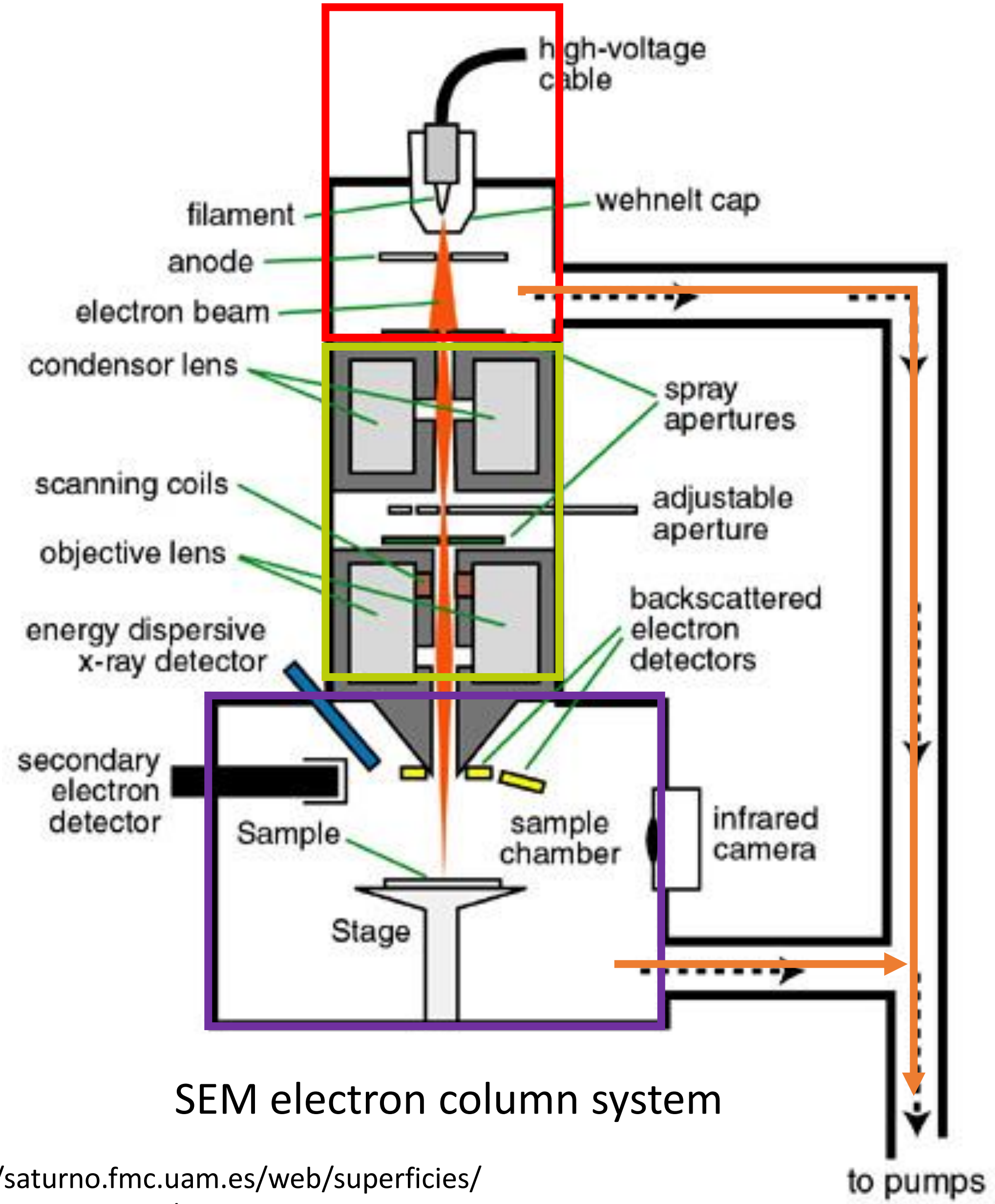
SEM (secondary electron)

**Advantages of using SEM over optical microscopes**

- High resolution (~ nm)
- Large depth of focus (how much is in focus)
- Wide range of magnification

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

# How does an SEM work? SEM Component Breakdown




SEM electron column system

http://saturno.fmc.uam.es/web/superficies/instrumentacion/Instrumentation.htm

- **Electron Source (Gun):** emits electrons and accelerates them in a strong electric field.
- **Electron Optics:** form and control the electron beam
  - Condenser lenses : converge the electron beam emitted from the gun into a fine beam, change probe current or spot size.
  - Apertures: stop electrons that are off-axis or off-energy from coming down the column
  - Deflectors: deflect the electron beam in the X and Y directions.
  - Stigmators: correct astigmatism
  - Objective lens: converges the electron beam into a fine beam and focuses it onto the sample surface.
- **Detectors:** detect different signals
- **Vacuum system:** This is required by the electron source and is essential for getting clean signals

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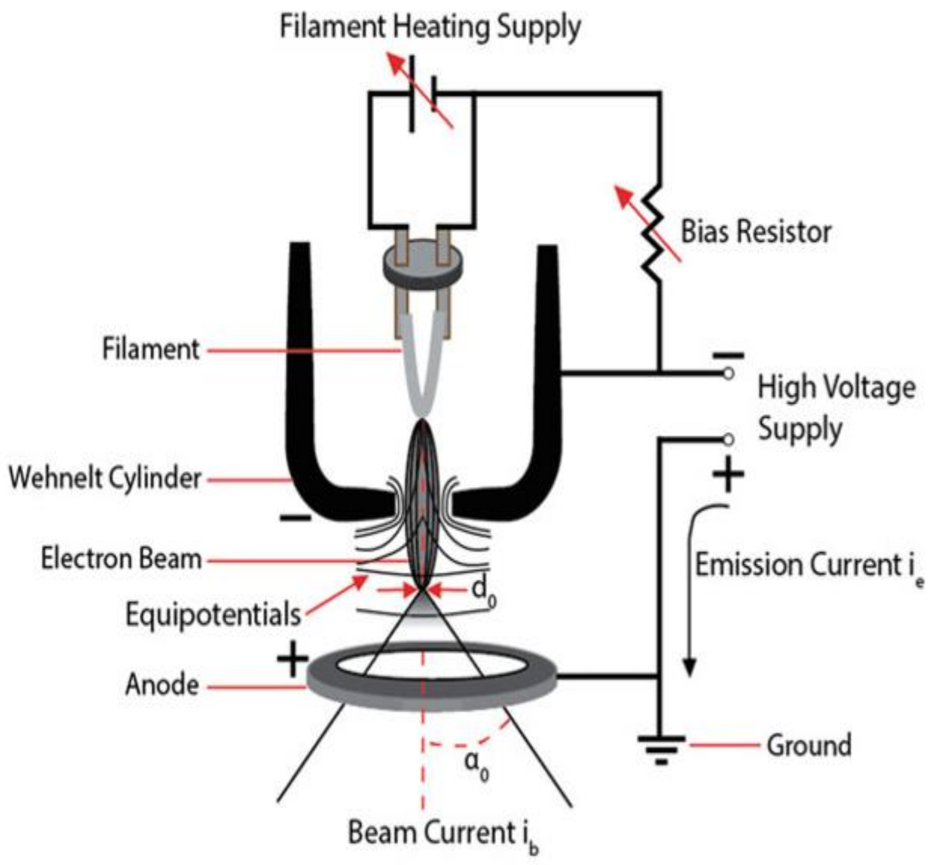
# A General Look at Electron Sources

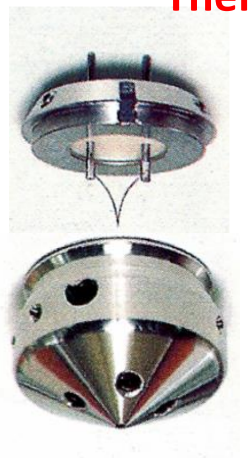
- Purpose:
  - Produce electrons,
  - Roughly shape the beam
  - Set the electron energy (Accelerating Voltage)
- Three types of electron sources
  - Thermionic e- Gun(heat)
  - Cold Field Emission Gun (electric field)
  - Schottky Field Emission Gun (a hybrid source that combines heating and electric field)
- Each source (different principles) has advantages and disadvantages




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

# Electron Gun: Thermionic Gun & Cold Field Emission Gun

## Thermionic Gun

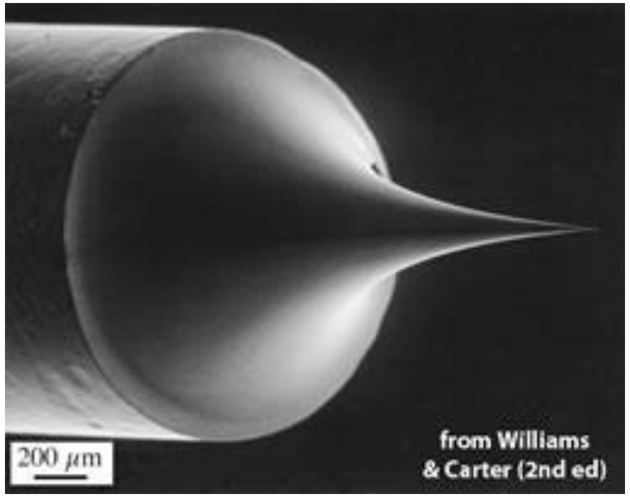


W~ 2700 K

$LaB_6$ ~ 1800 K

- Filament (W or $LaB_6$) emits electrons as a result of heating
- Major Advantages:
  - Very high probe current
  - Stable probe current
  - Less complex vacuum system
- Disadvantages:
  - Lower resolution
  - Relatively short lifetimes

## Cold Field Emission Gun

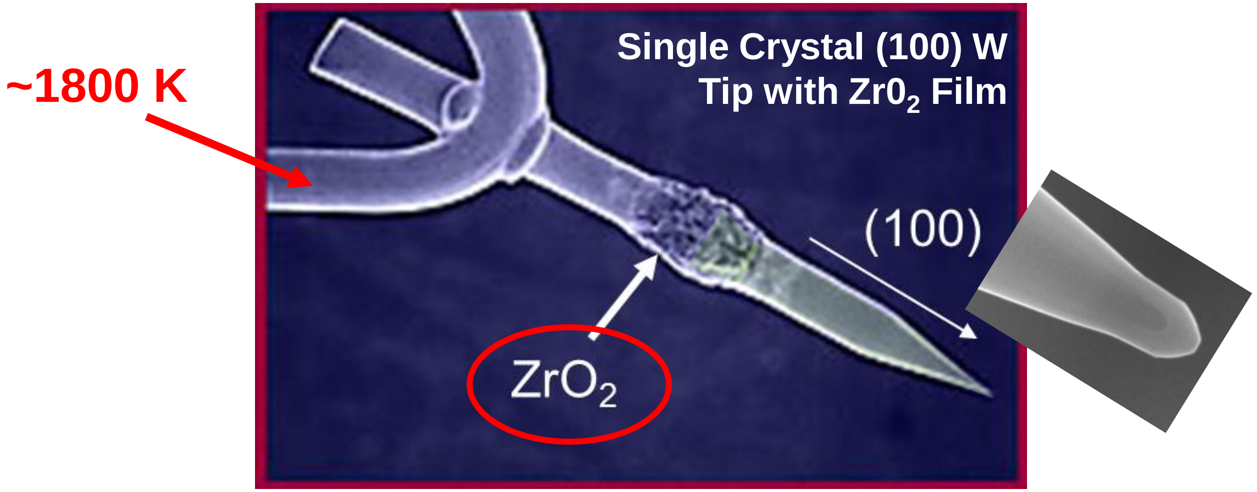



- Single-crystal Tungsten fashioned into a sharp tip (~100 nm) allows the applied electric field to be highly concentrated, and purer energy electrons are emitted.
- Major Advantages:
  - High imaging resolution (best)
  - Very long potential source lifetime
- Disadvantages:
  - Lowest maximum probe current
  - Poor probe current stability
  - Needs periodic "flashing" (300K room temperature)
  - Requires ultra-high vacuum in gun area

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

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

# Electron Gun – Schottky (TFE) Thermal Field Emission Gun




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

- Electric field assisted thermionic emission gun
- Single-crystal Tungsten wire fashioned into a tip R~500 nm, allows the applied electric field to be concentrated. Heated to~ 1800K. Purer high energy e- are emitted.
- ZrO2 coats further lower the work function

- Major Advantages:
  - High imaging resolution
  - High probe currents obtainable
  - Excellent current stability
  - Long potential source lifetime
- 
- Disadvantages:
  - Requires ultra-high vacuum in gun area
  - Finite lifetime

*A Beginner Guild to Scanning Electron Microscopy* by Anwar Ul-Hamid, Springer, 2018

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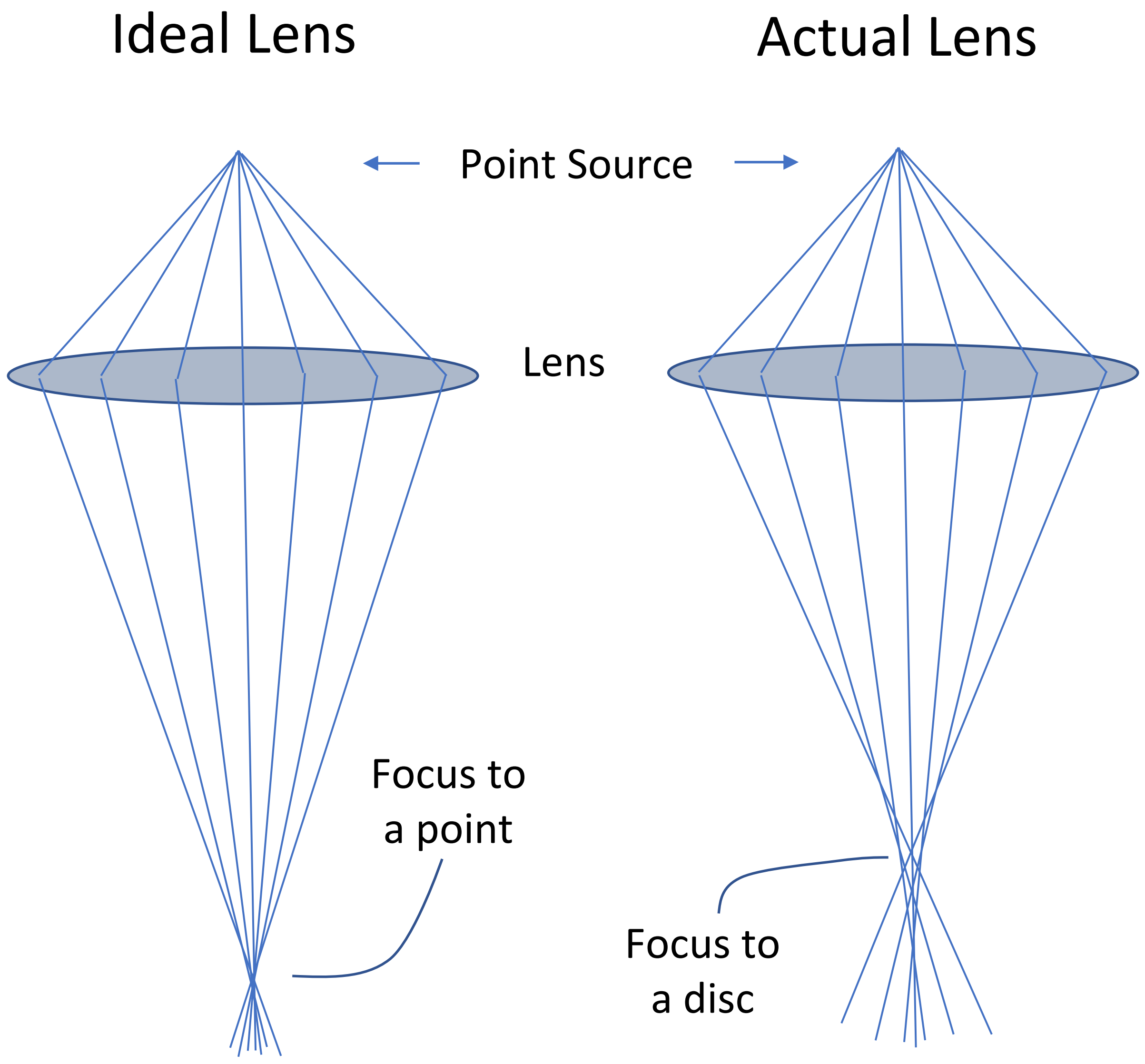
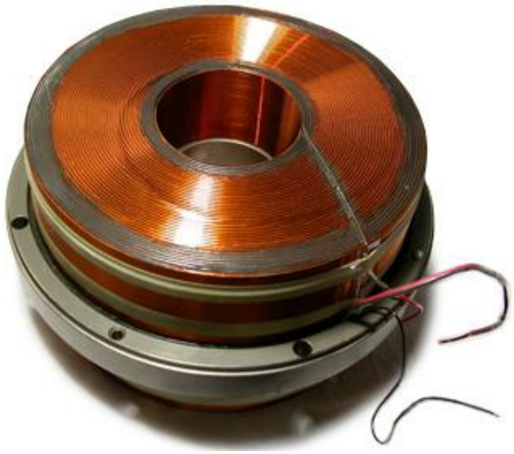
# The Electromagnetic Lens & Lens Aberration



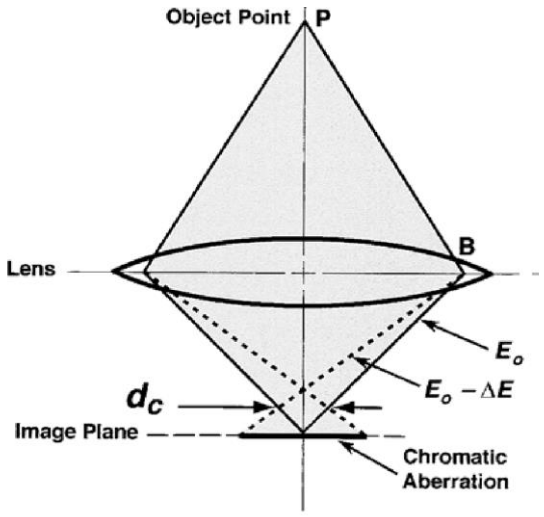



**An electromagnetic lens**

**Aberration effects:**

- Spherical Aberration
- Chromatic Aberration
- Astigmatism

# Aberration from Electron Source



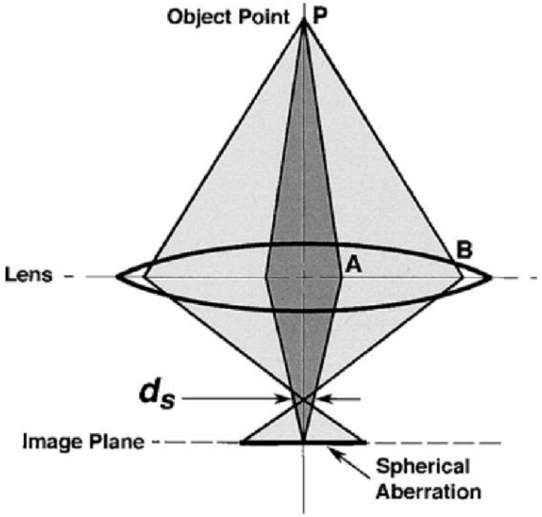
(a)

$$d_c = C_c \alpha \left( \frac{\Delta E}{E_0} \right)$$

*C*c : the chromatic aberration coefficient
*α* : the convergence Angle
*ΔE/E*$_0$ : the fractional variation in the electron beam energy

## Chromatic Aberration

- Electrons with different energy (velocity) are focused on different points in the image plane
- Increases with source **energy spread** (heated sources!)
- Decreases with increasing electron energy ($E_0$)



(a)

$$d_s = \frac{1}{2} C_s \alpha^3$$

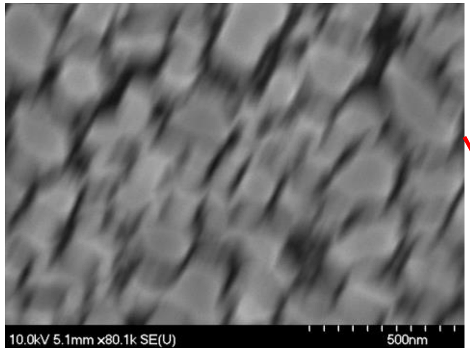
*C*s :the spherical aberration coefficient
*α*: the convergence Angle

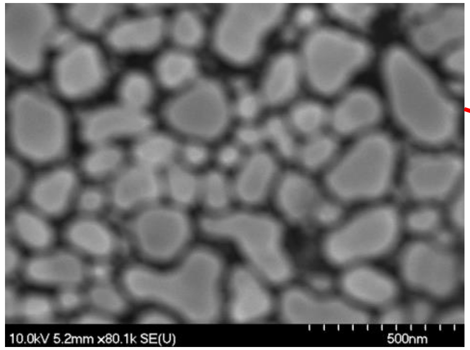
## Spherical Aberration

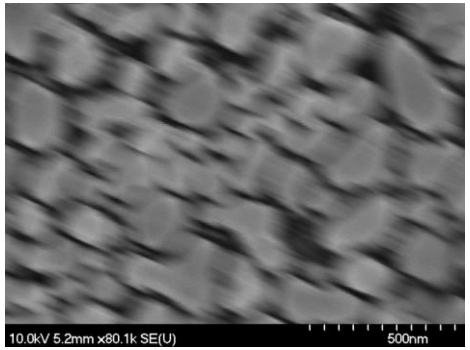
- Electromagnetic field of the lenses is not uniform
  - Stronger towards the outside
  - Weaker towards the center
- Off-axis beams are overfocused
- Solution! Add a limiting aperture

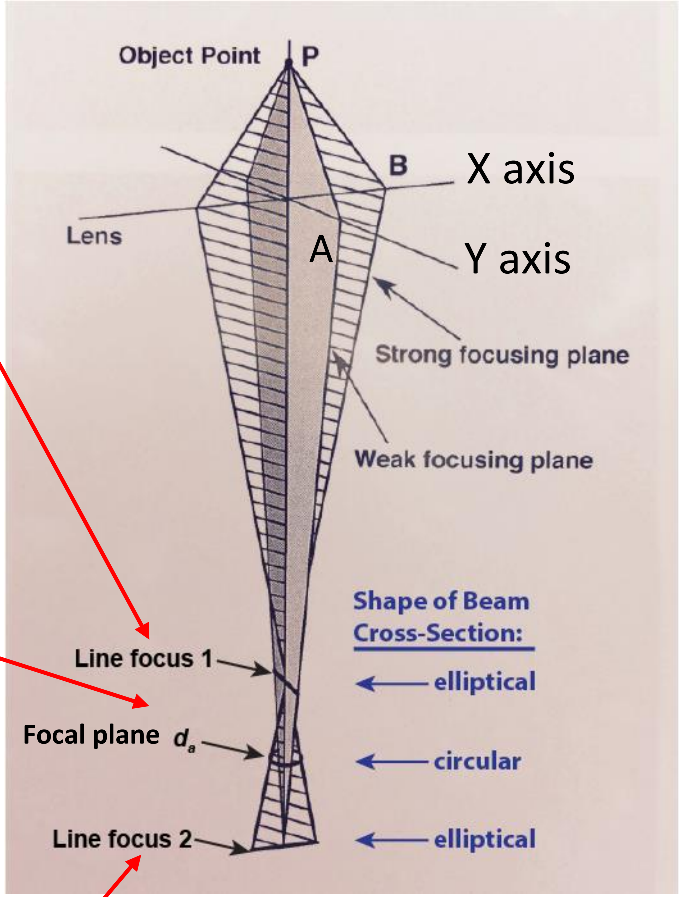
Adapted from *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press

# Last Major Aberration - Astigmatism



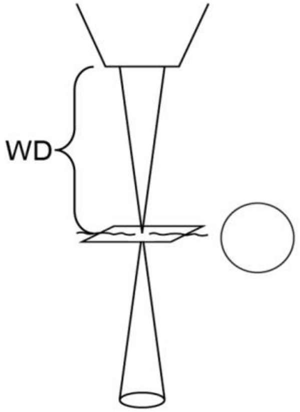



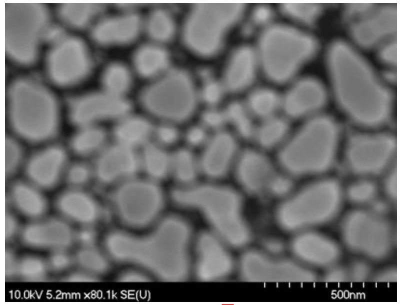



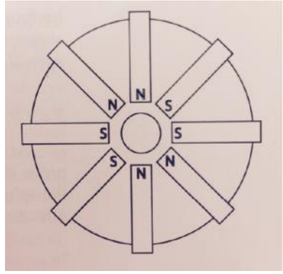




- Caused by asymmetry in the lens, one axis is stronger than the other axis
- Images will appear 'stretched' as the operator changes focus

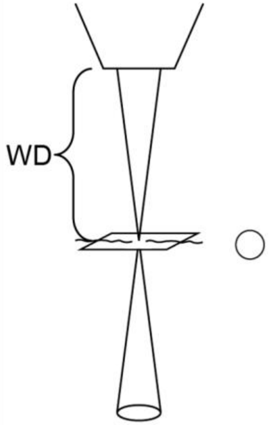
In focus Bad Astigmatism

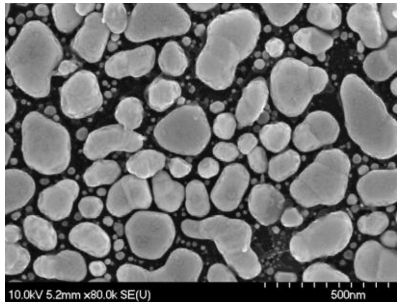



In focus Corrected Astigmatism




Octupole stigmator with four sets of opposing magnetic poles, used to force the beam into a small circular shape.

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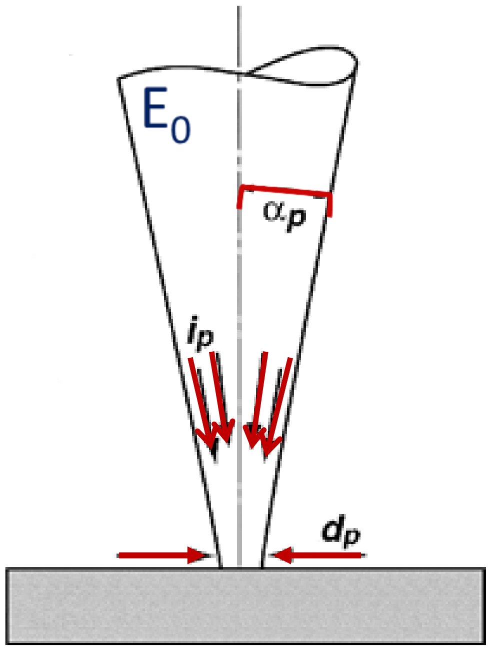
# Major Electron Beam Parameters

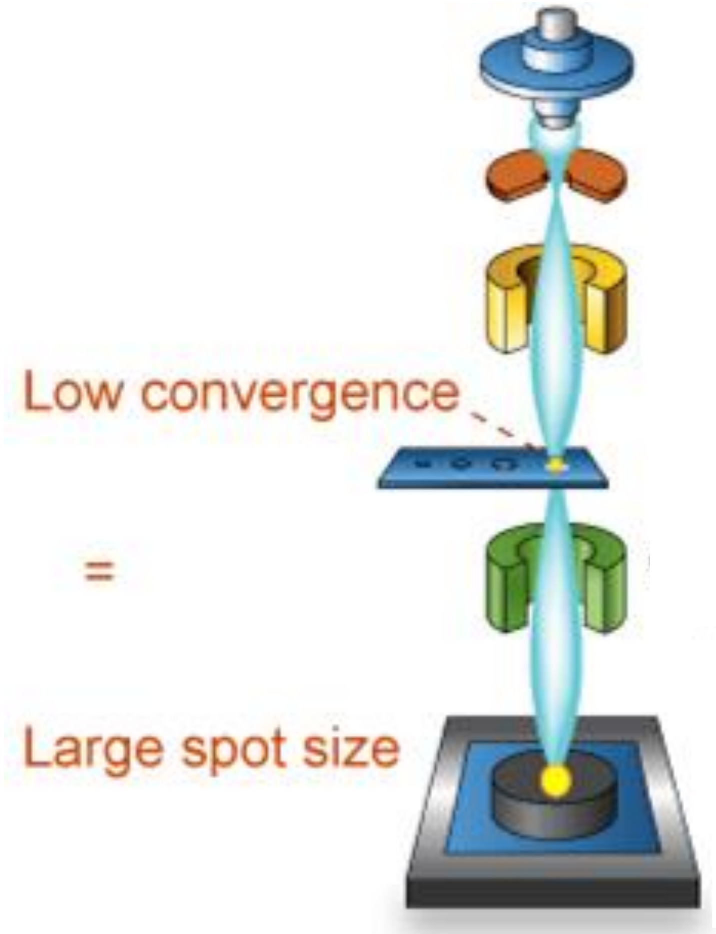
- Four electron beam parameters define the probe:
  - Spot size – dp
  - Probe current – Ip
  - Probe convergence angle – αp
  - Accelerating Voltage – $E_0$

- 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




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

# Function of the Condenser Lens: Spot Size/Probe Current



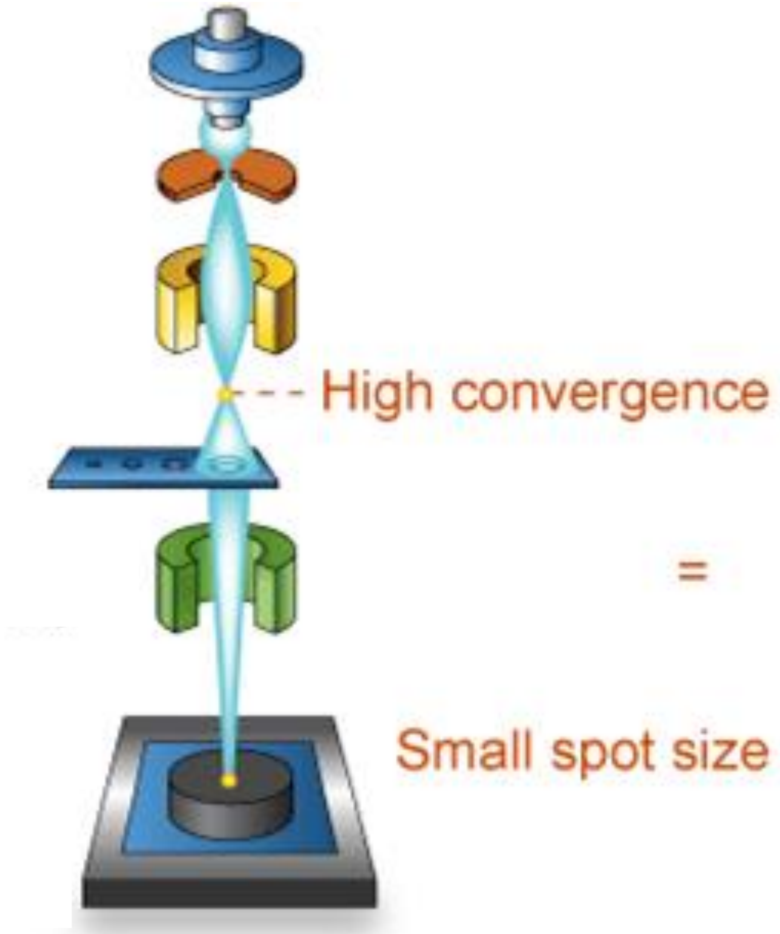

Electron
source

Condenser lens(es)
**Lens current**

Objective
lens (focus)

Sample

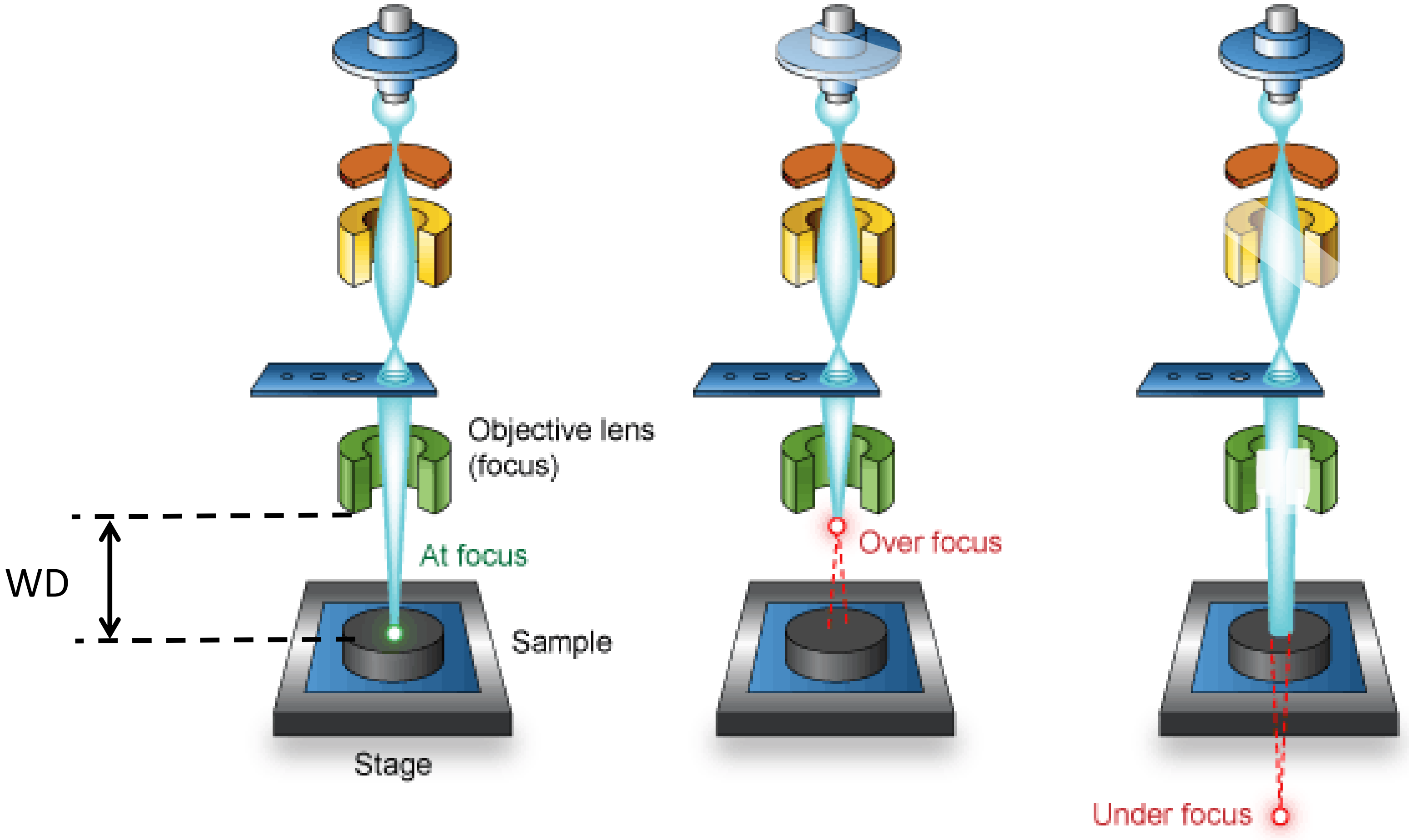
stage




- Low convergence – more beam accepted by aperture
  - More current: smoother but less resolution
- High convergence – less beam accepted by aperture
  - Less current: rougher but higher resolution

http://www.ammrf.org.au/myscope/sem/practice/principles/lenses.php

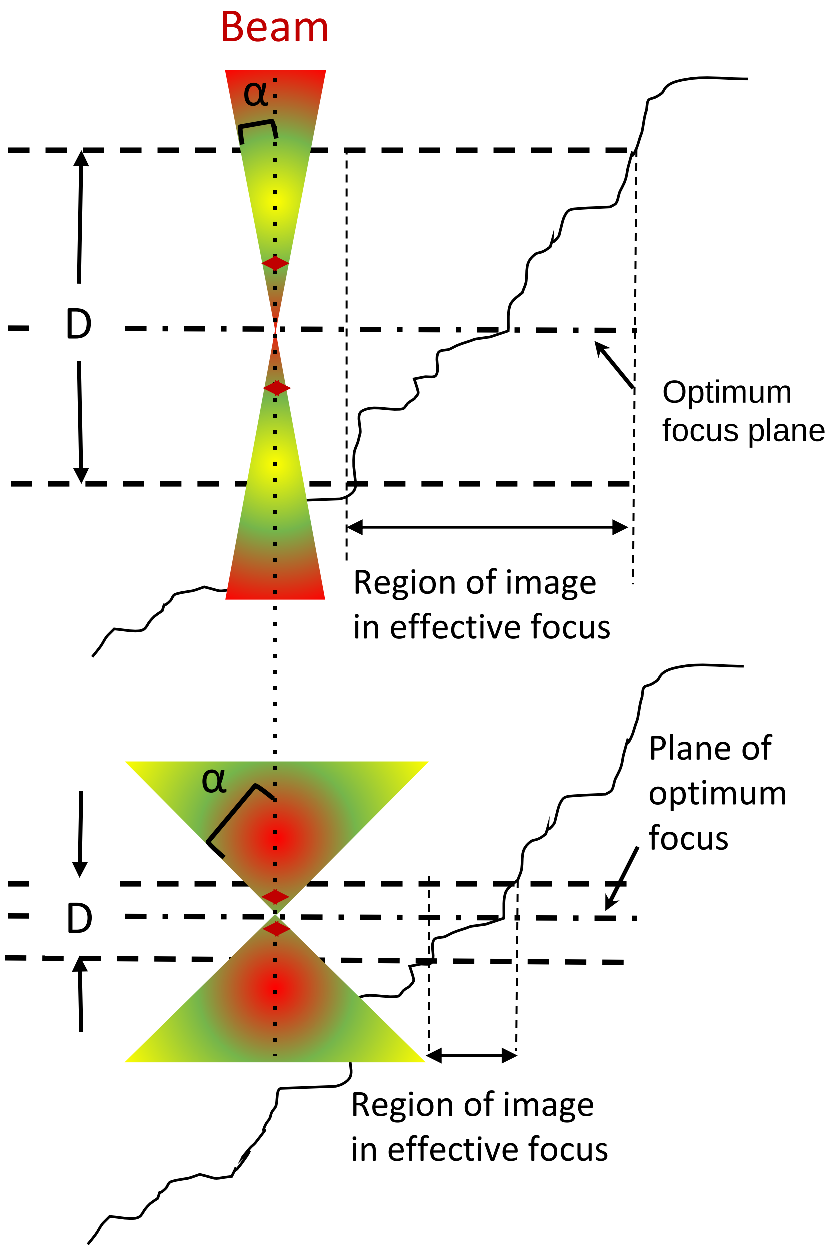
# Function of the Objective Lens: Focus!




http://www.ammrf.org.au/myscope/sem/practice/principles/lenses.php

- Change the lens current to change the focal distance
- Commonly called the Working Distance (WD)

# Depth of Focus




**Depth of focus** is the distance above and below the **focus plane** that beam becomes broadened to a noticeable size "blurring" the image

**Quantifying Depth of Focus**

The depth of focus can be described by:

$D \approx 0.2 \text{ mm} / \alpha M$ (1)

$\alpha = Rap/WD$ (2)

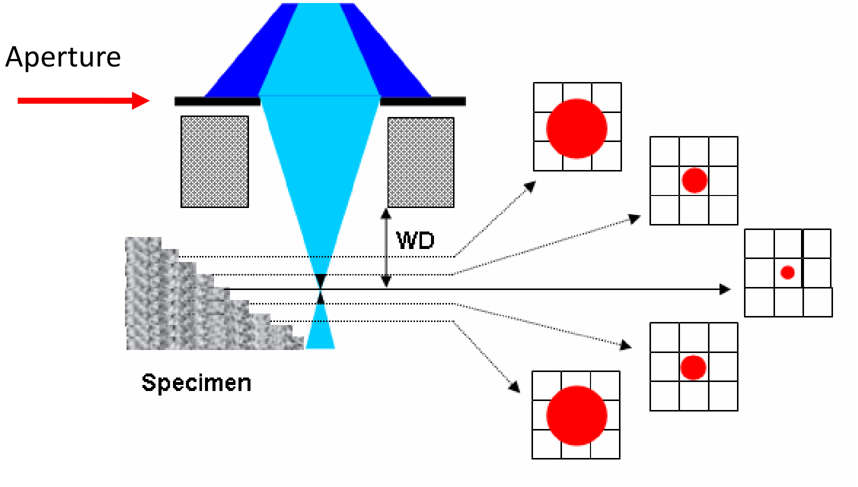
α – probe convergence angle
M – magnification
Rap – radius of the aperture
WD – working distance

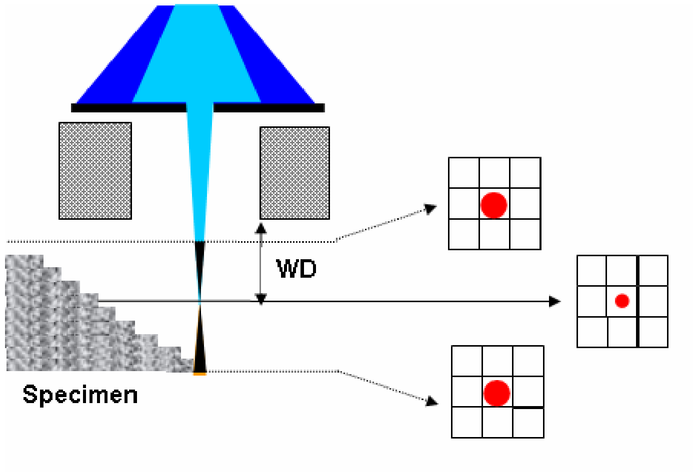
Equation 1: Depth of focus is inversely proportional to convergence angle α and M.

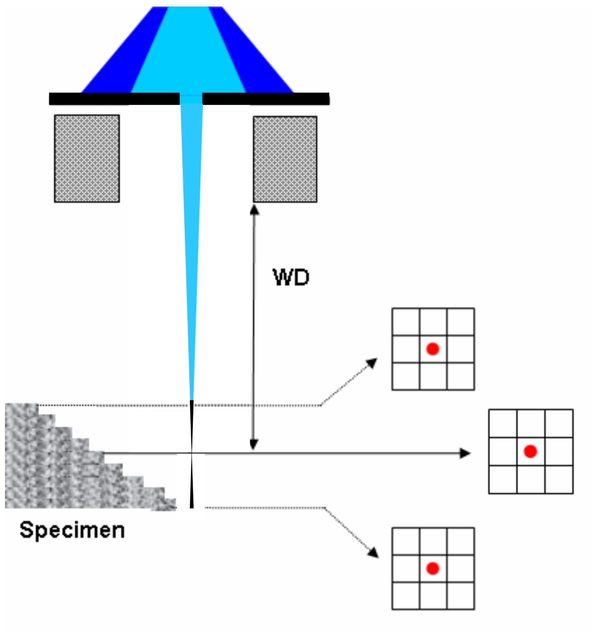
Equation 2: Decreasing Rap and increasing WD both increase the depth of focus.

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

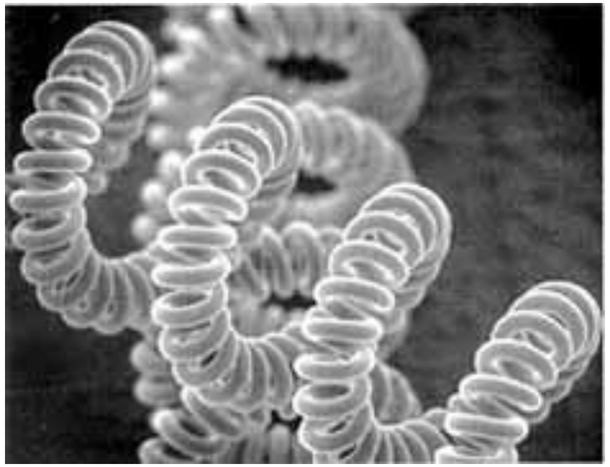
# Depth of Focus (Convergence Angle) : WD & Objective Aperture



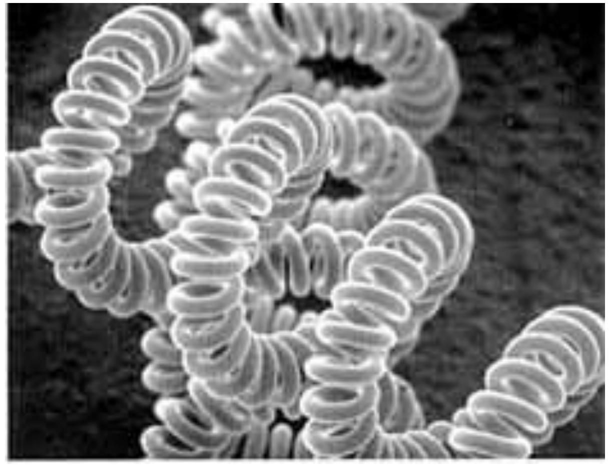



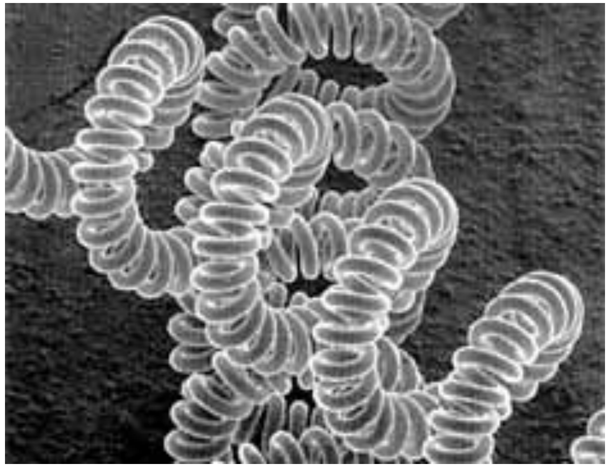

light bulb coil



600 um aperture and 10 mm WD.



200 um aperture and 10 mm WD.



JEOL guide

100 um aperture and 38 mm WD.

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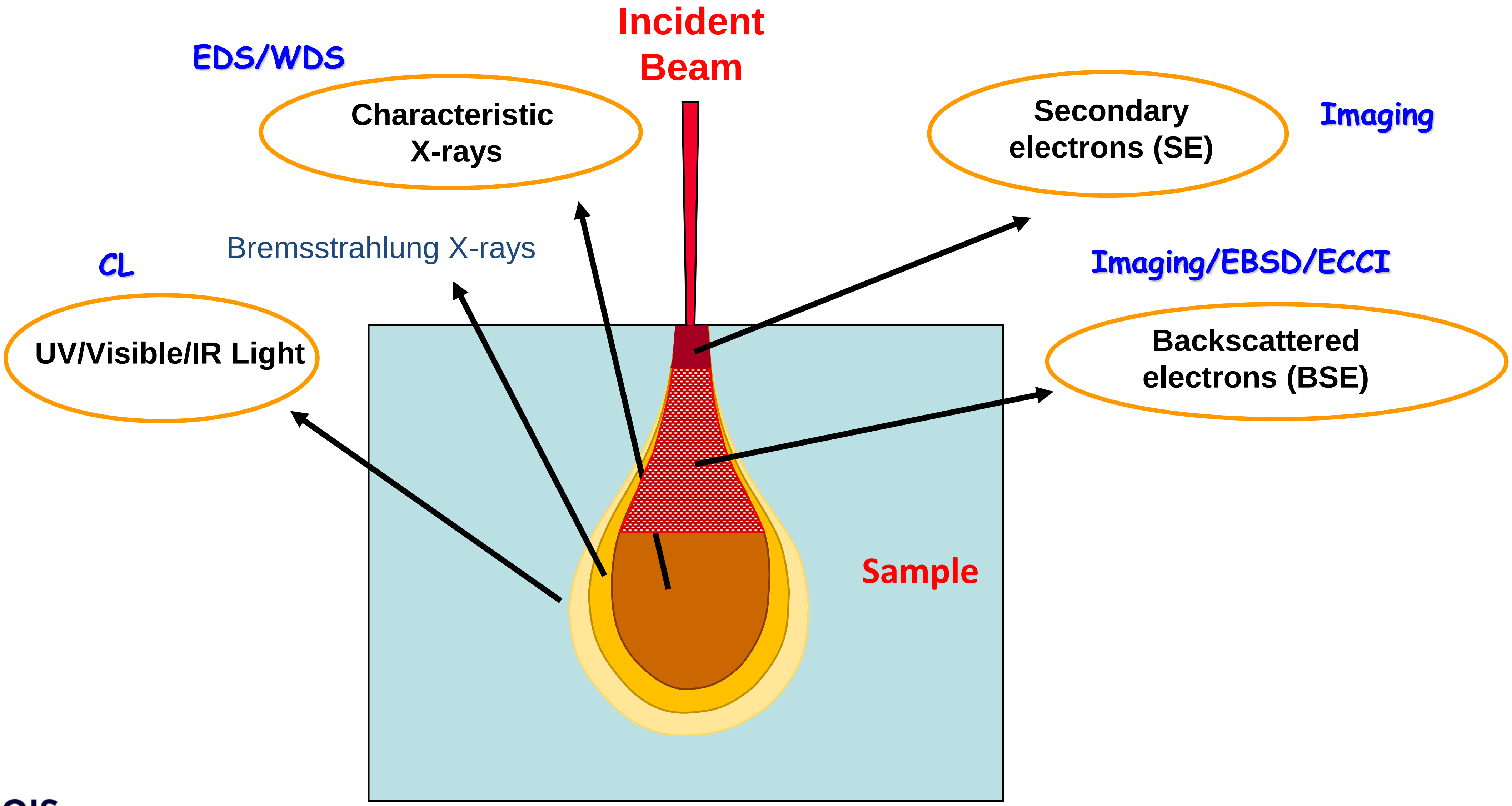
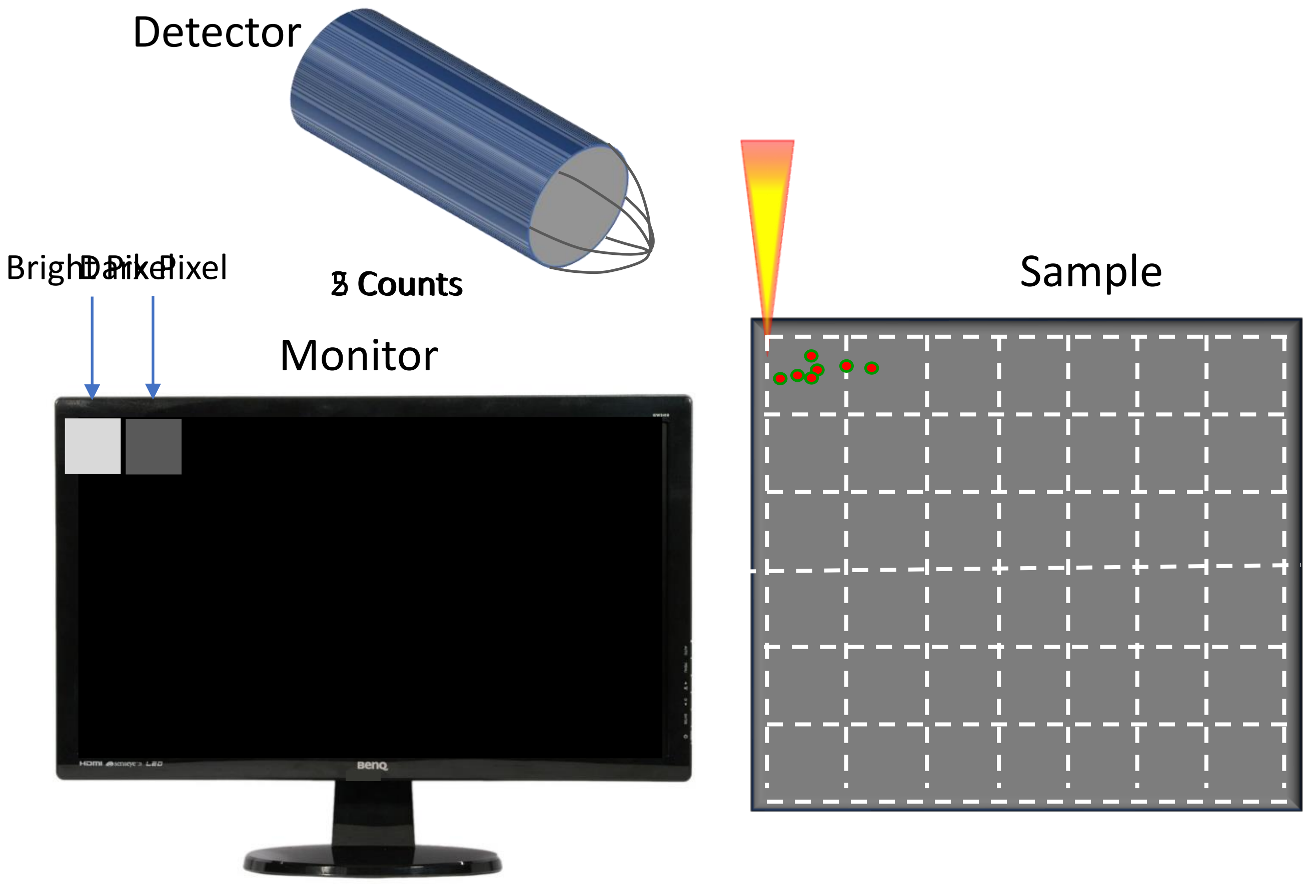
# Interactions between Electron Beam and Specimen



# Contrast Mechanism: How is the image generated?




***Contrast*** represents the difference of signals detected at any two chosen points of interest in the scan raster.

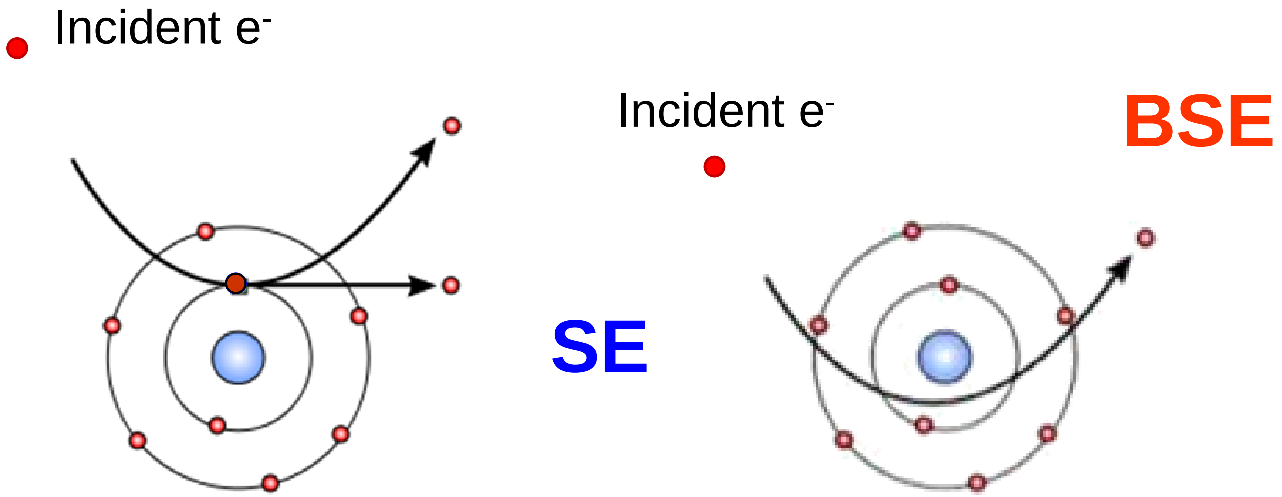
**Contrast Definition:**

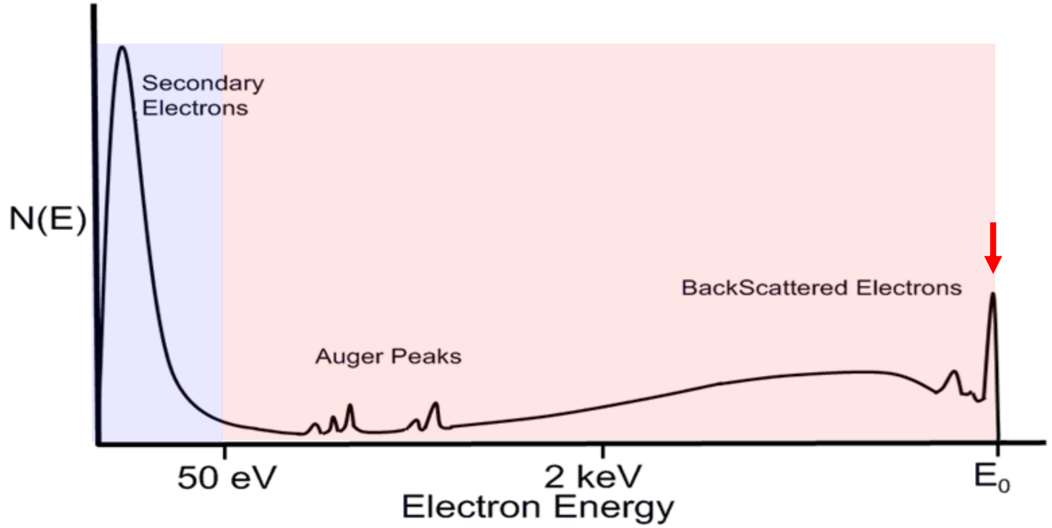
$$C = (S2 - S1)/S1,\ S2 > S1$$

- S2 is **the signal** from the feature of interest;
- S1 is the background signal

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

# Secondary Electrons (SE) & Backscattered Electrons (BSE) Imaging




SE/ BSE energy range

## Secondary electrons (SE)

- Ejected as a result of the interaction between the primary beam electrons and the specimen
- From a few nm below the sample surface
- low energy electrons (< 50 eV)

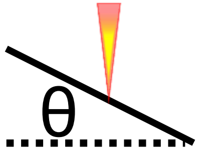
## Backscattered electrons (BSE)

- Primary beam electrons scattered back out of the sample.
- Approximately 20~50nm spatial resolution
- High energy electrons (up to the primary energy $E_0$)

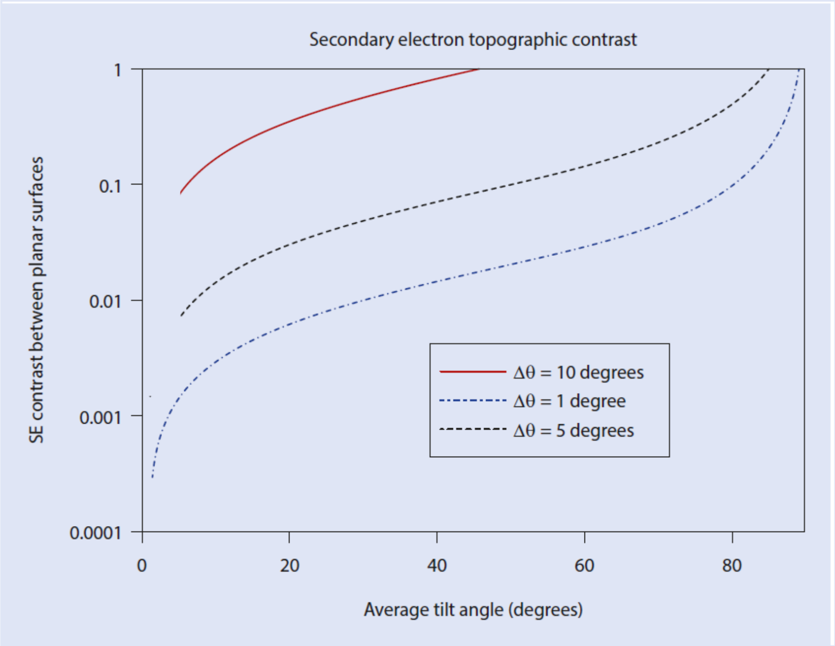
# Secondary Electrons Topographic Contrast

- SE yield is strongly dependent on incident angle of the beam with the sample surface.

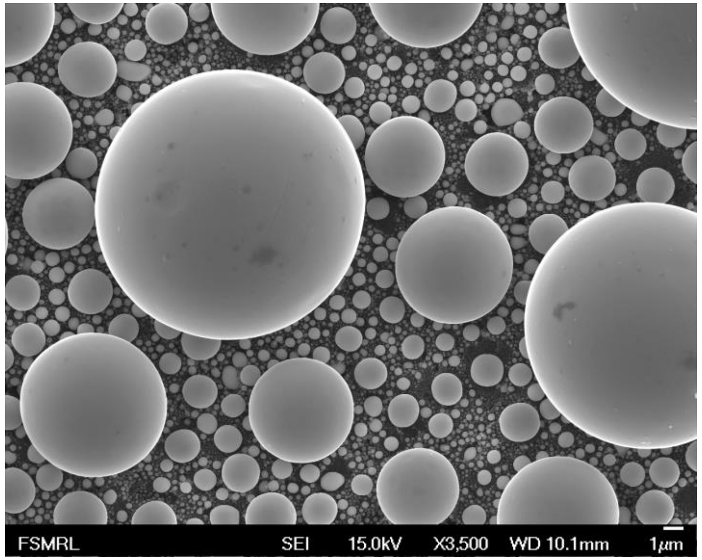
$$\delta(\theta) = \delta_0 \sec\theta$$

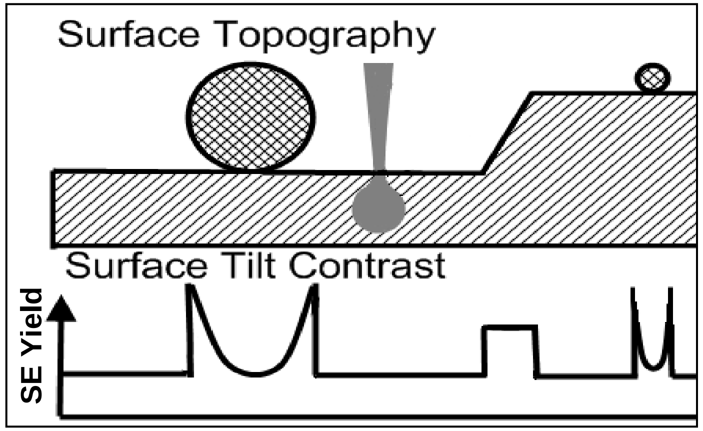


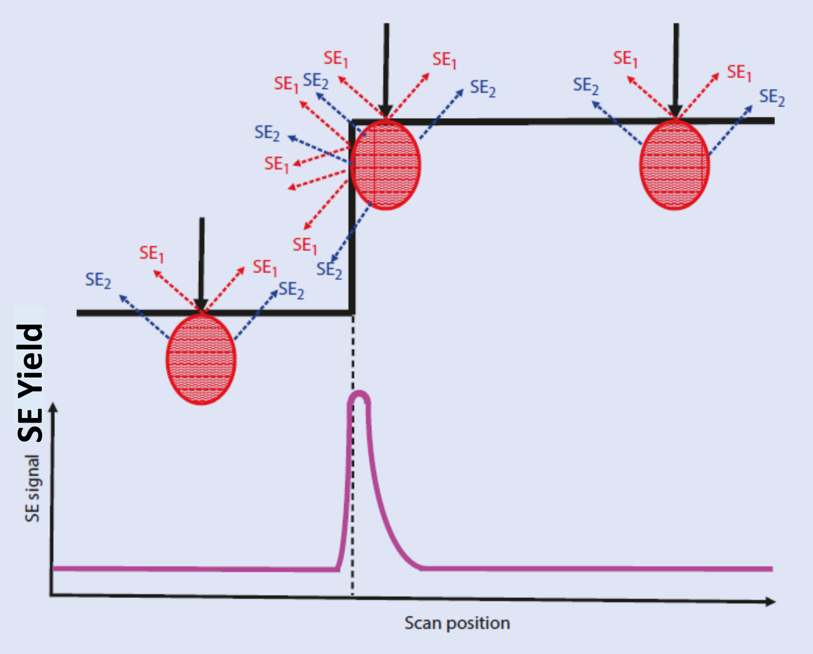

$\delta(\theta)$ : the SE yield coefficient
$\theta$ : the sample tilt angle




- Edge effect: Edges and ridges of the sample emit more SEs and thus appear brighter in the image.



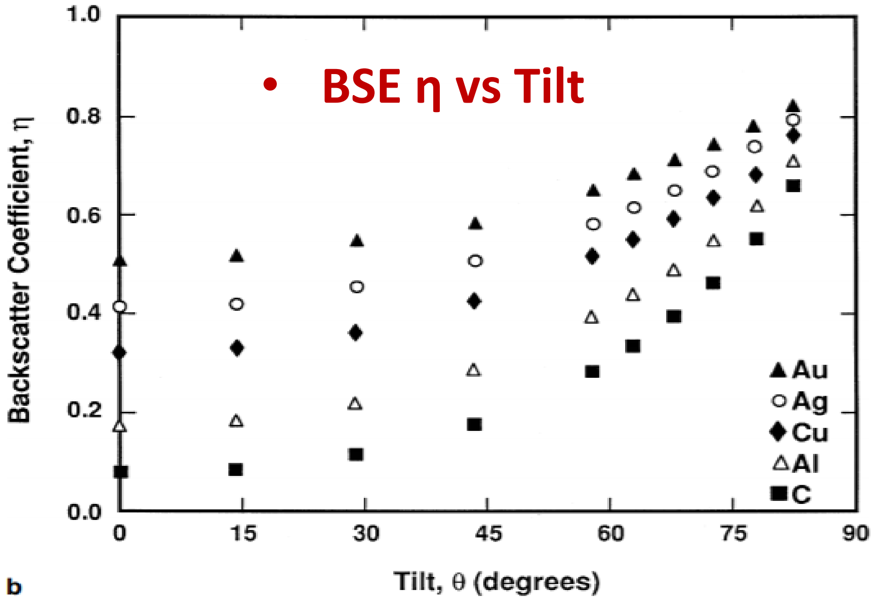




From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press

## BSE Yield coefficient ( η )

$$\eta(Z,\theta)=(1+cos\theta)^{-\frac{9}{\sqrt{Z}}}$$




## Compositional Contrast

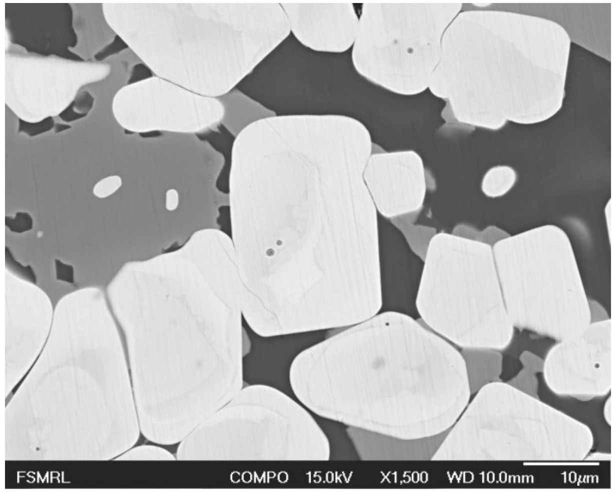
- increases monotonically with average **atomic number Z**

## Topographic contrast

- increases monotonically with the **specimen tilt angle θ**

## Channeling contrast

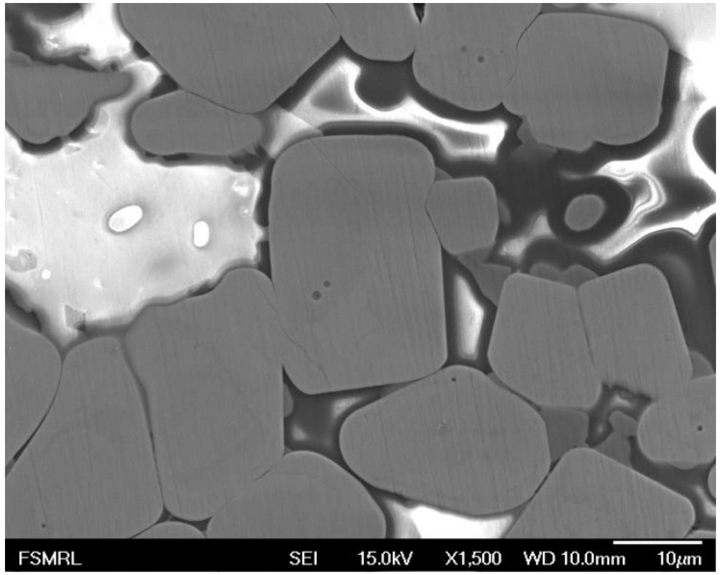
- crystal orientation and structure



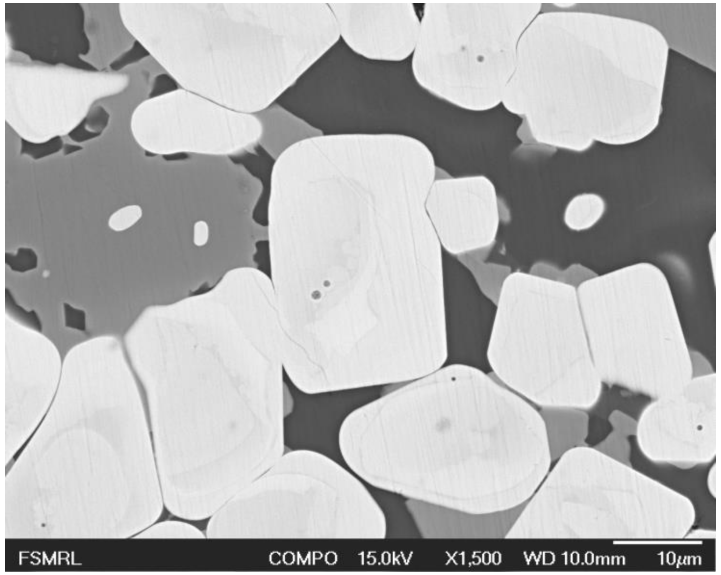

*Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press

# Contrast Comparison of Images Taken at Different Imaging Modes

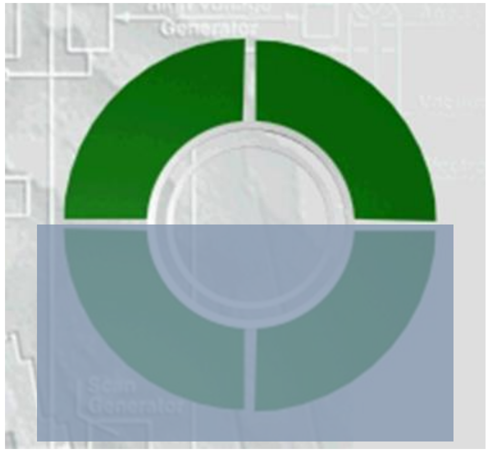
**SE image**
**(Topographic Contrast)**



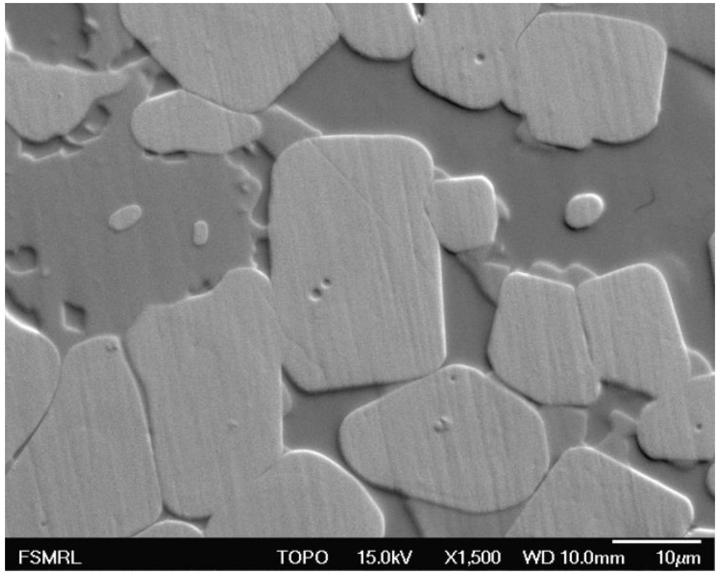

**BSE COMPO mode Image**
**(Compositional Contrast)**




**Segmented BSE Detector**
**Four quadrants**



**BSE Topo mode Image**
**(Topographic Contrast)**




**La, Mn, Ca, Al oxides Multiple phases**

# Outline

## Part I: SEM

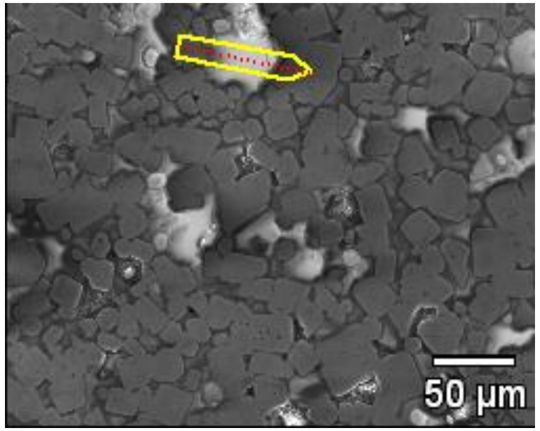
- ❑ What is SEM & How does it work
  - Electron Guns (Electron Sources)
  - Electron Lenses and Lens Aberrations/Corrections
  - Major electron beam parameters and how to control them
- ❑ Applications
  - SE & BSE imaging and detectors
  - Analytical techniques: EDS, EBSD, CL, ECCI

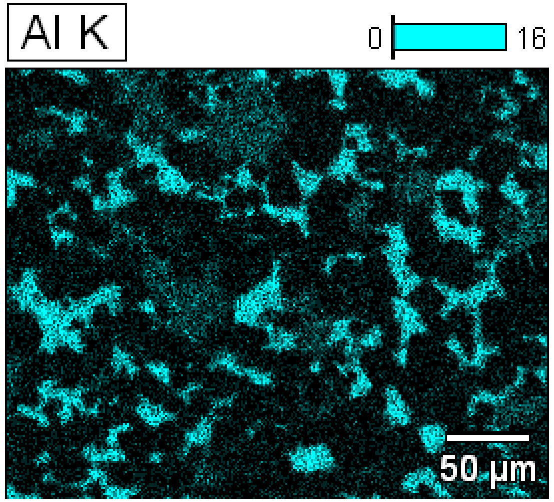
## Part II: FIB

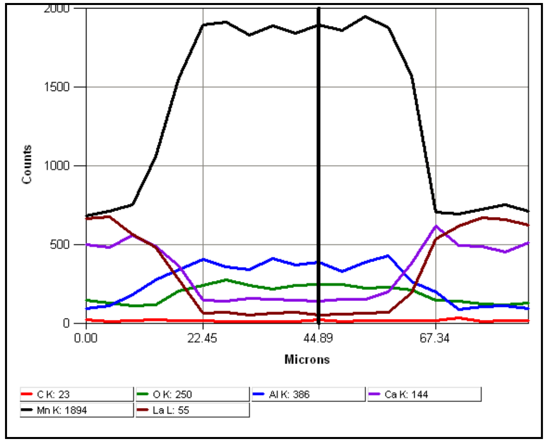
- ❑ How does FIB work
- ❑ Common applications

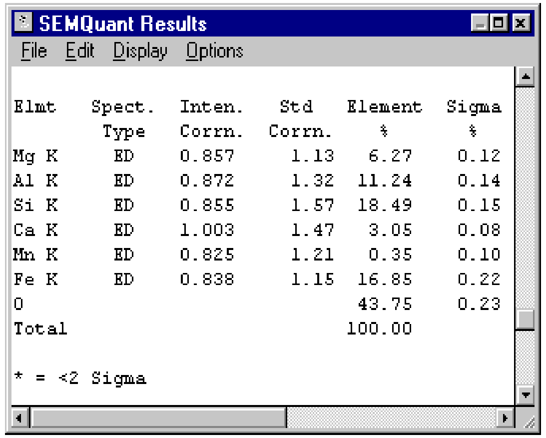
# EDS Microanalysis in the SEM

- EDS : Energy Dispersive Spectroscopy.
- Powerful technique for Chemical composition analysis:
  - ○ **What elements**: Elements identification
    - Qualitative analysis
  - ○ **Where**: Elements distribution
    - Point/region analysis, line scans, full-spectrum imaging
  - ○ **How much**: Concentration
    - Quantitative analysis



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

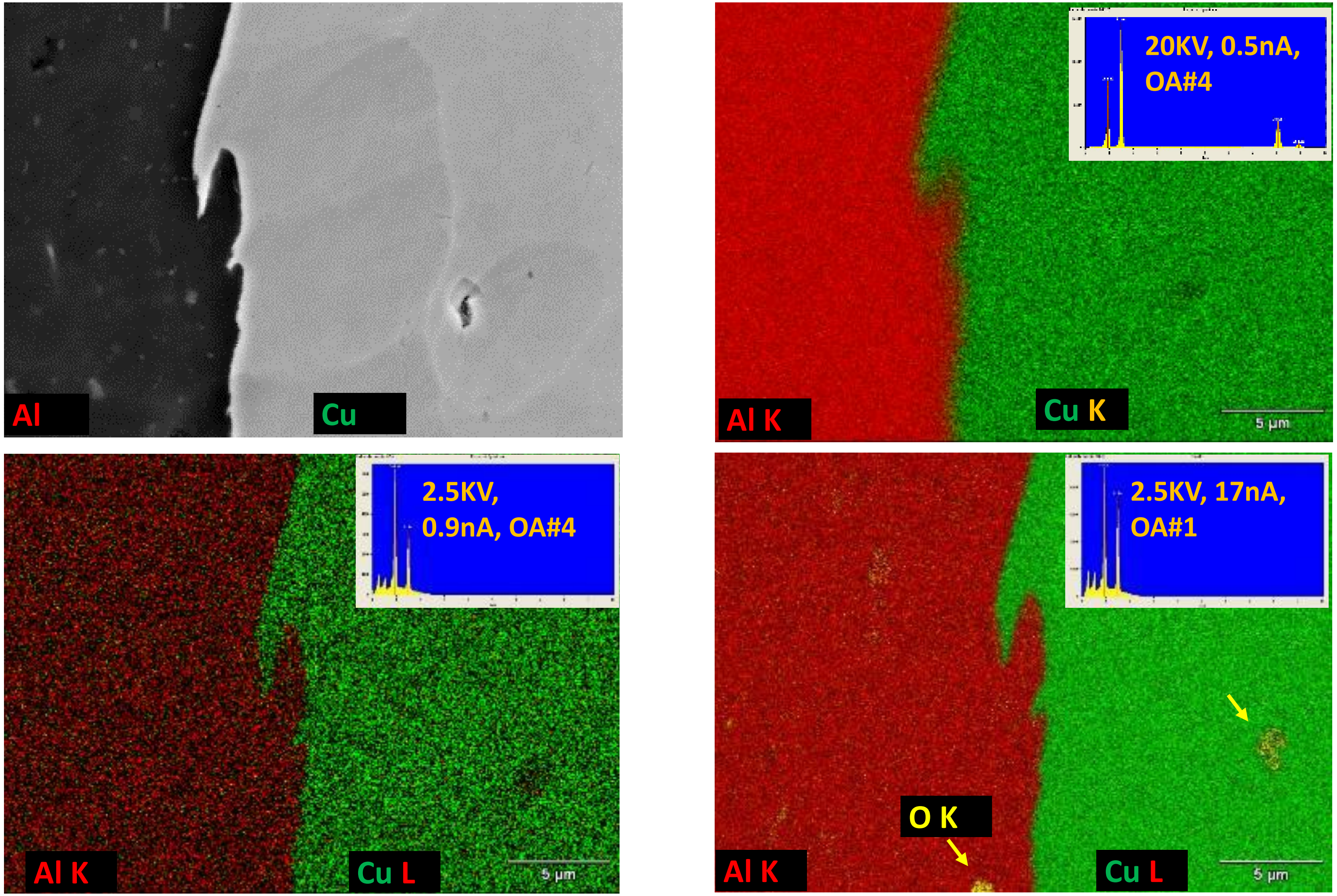
# EDS mapping

*Sample courtesy Prof. Chenhui Shao*

Low Beam Energy Analysis:

- Improve Spatial Resolution
- Visualize Light elements(**Oxide inclusion**)
- Alternative for overlapping peaks
- Reduce Charging for insulator /non-conducting materials

Cu Kα=8.0 KeV
Cu Lα=0.936 KeV

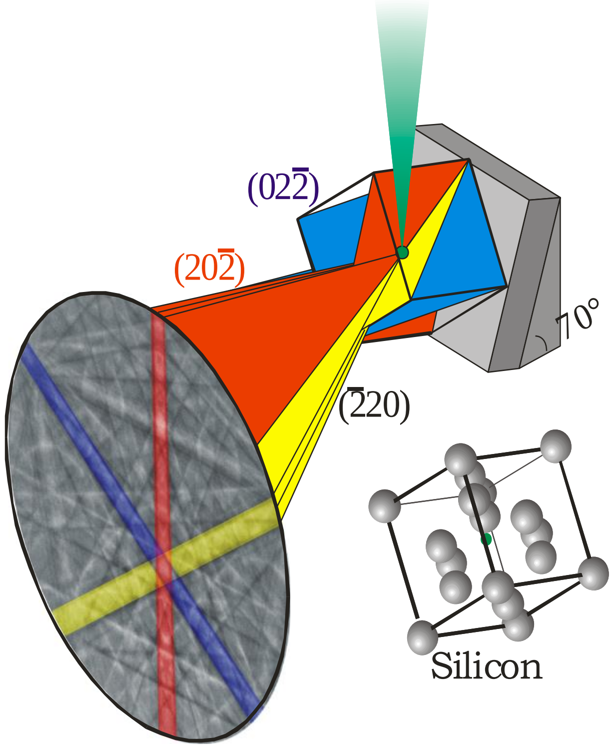


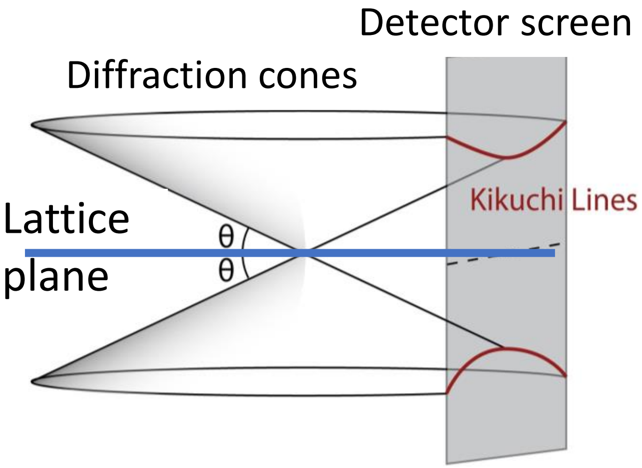

Low beam energy, high probe current gives sharp interface and clear map

ILLINOIS
Materials Research Laboratory
GRAINGER COLLEGE OF ENGINEERING

# EBSD: Electron Backscatter Diffraction

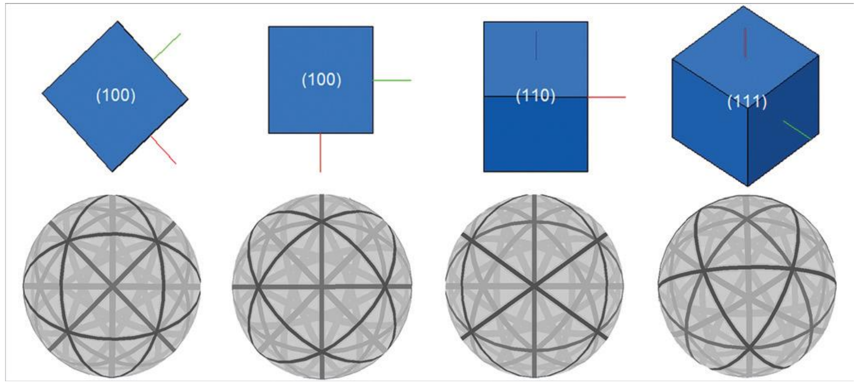
- Major functions: Crystal orientation determination and phase identification
- **Crystalline Sample** high tilted 70º
- Inside the sample, electrons **scatter** in all directions. When electrons that satisfy Bragg's law with a **lattice plane**, they will be strongly scattered to form a diffraction cone.
- The cones **intersect** the detector screen to form a **Kikuchi band**.
- Superposed multiple Kikuchi bands to create the complete **Kikuchi pattern**.
- Analysis and indexing of the pattern to extract crystallographic information




$2d \sin \theta = n\lambda$

$\lambda$=8.7pm@20KV; d=sub nm;
Bragg's angel $\theta < 1°$

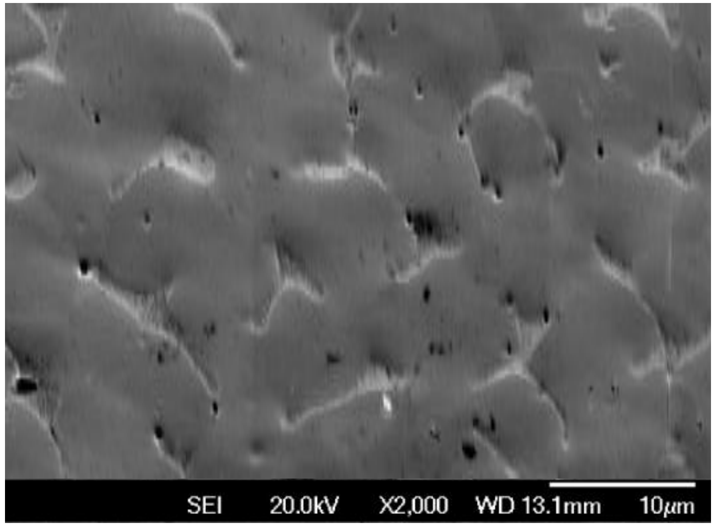


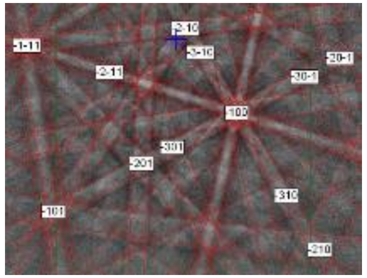

Kikuchi Patterns

Courtesy HKL Technology (Oxford Instruments Microanalysis Group) Oxford – EBSD Explained
**MethodsX** 5 (2018) 1187-1203

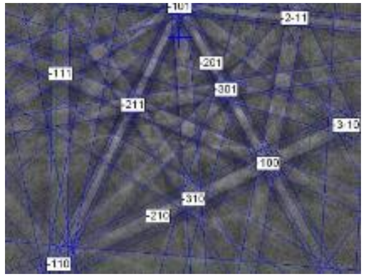
# EBSD: Electron Backscatter Diffraction

*Sample courtesy Prof. Brent Heuser*



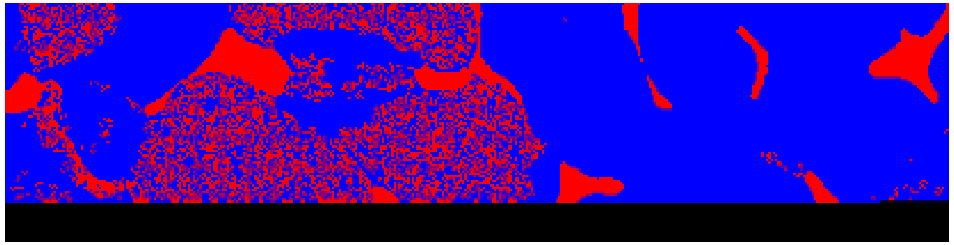



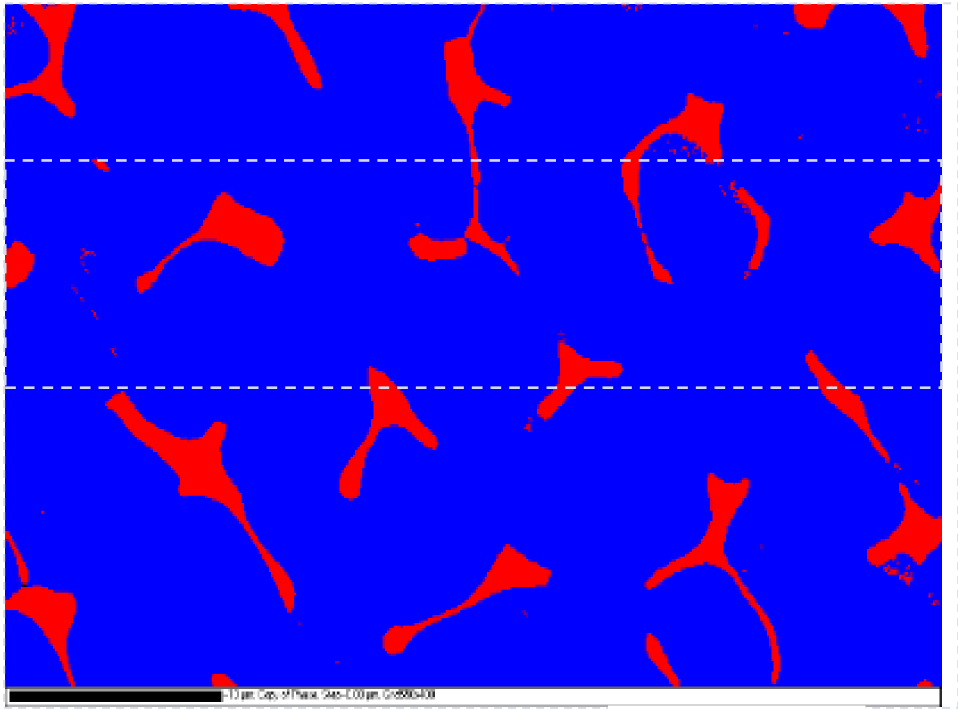
bcc:
ferrite

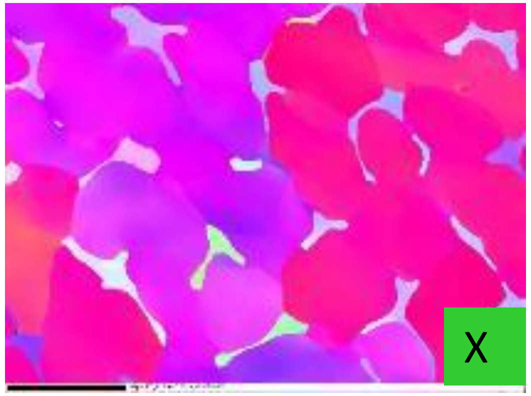


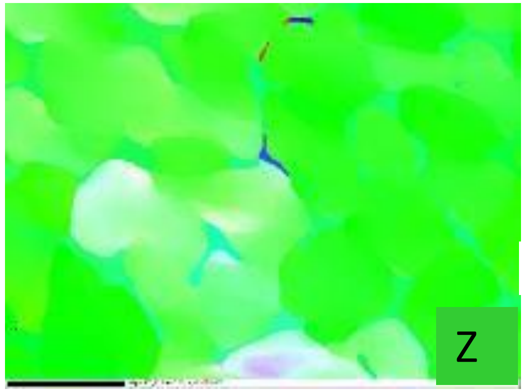
fcc:
austenite

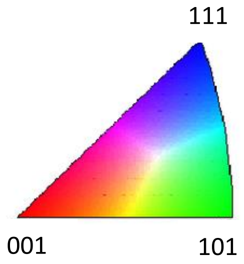
Phase map:
indexing





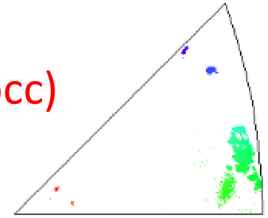


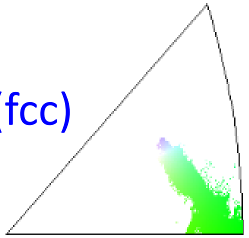




Crystal Orientation maps: Preferential orientation for both phases

IPF Z (bcc)



IPF Z (fcc)

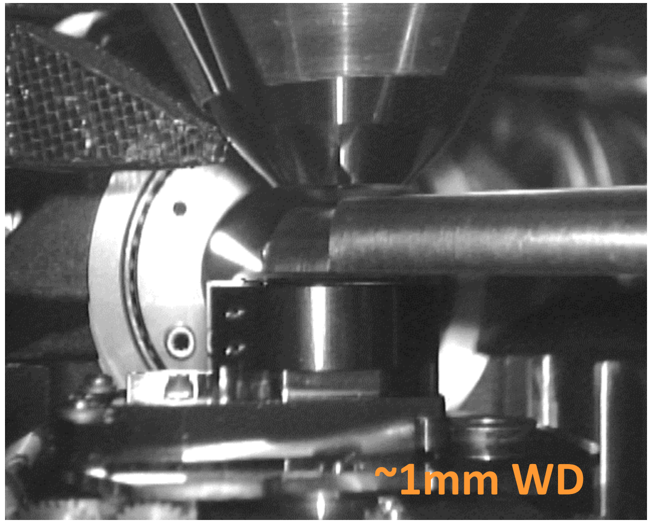


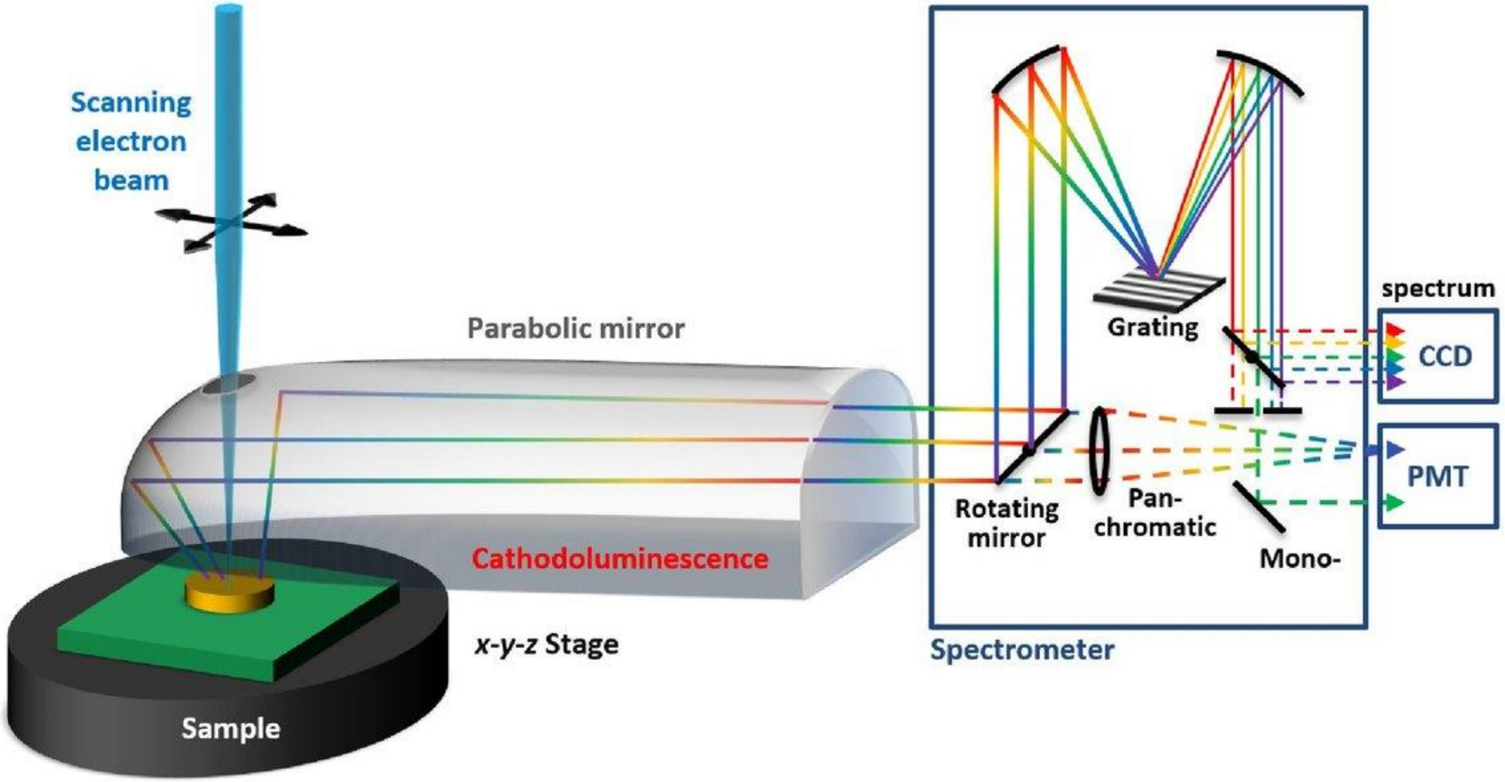
The inverse pole figures: The crystal orientation distribution

# CL: Cathodoluminescence

**Luminescent materials**
(Phosphorus, direct bandgap semiconductors, organic molecules, Rocks, minerals, etc., **structural and functional properties** )

- Optical properties,
- Electronic states,
- Electronic properties,
- Structure,
- Defects




- Generate and emit photons of various wavelengths
- Keep beam moving in the same direction/continuous beam
- Change beam direction/reflection
- Grating: separate full wavelength spectrum of UV/Vis/IR light into monochromatic lights

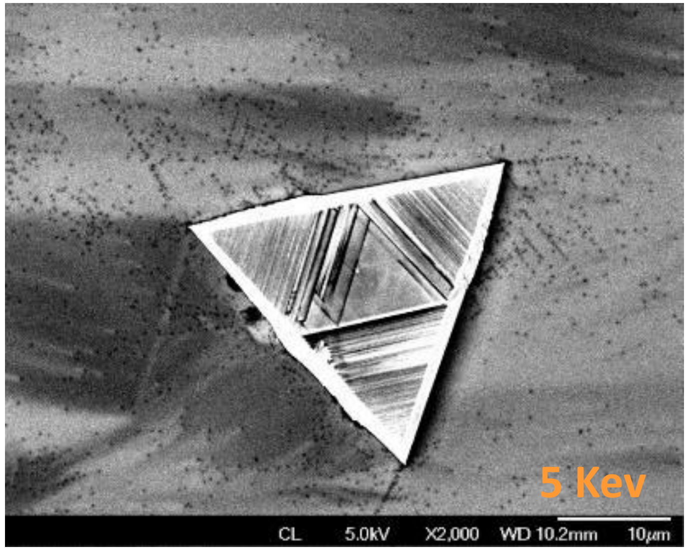
www.spiedigitallibrary.org

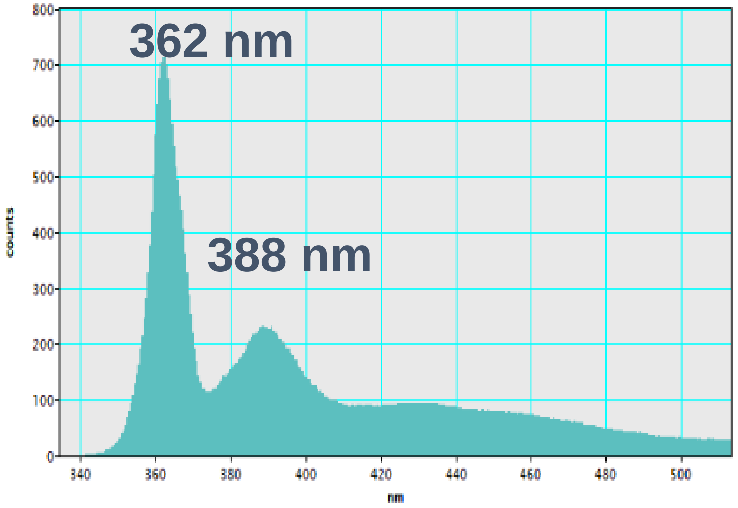
# CL Imaging of GaN Film with Triangular Islands

*Sample courtesy Prof. David Cahill*

**GaN thin film on the sapphire substrate**

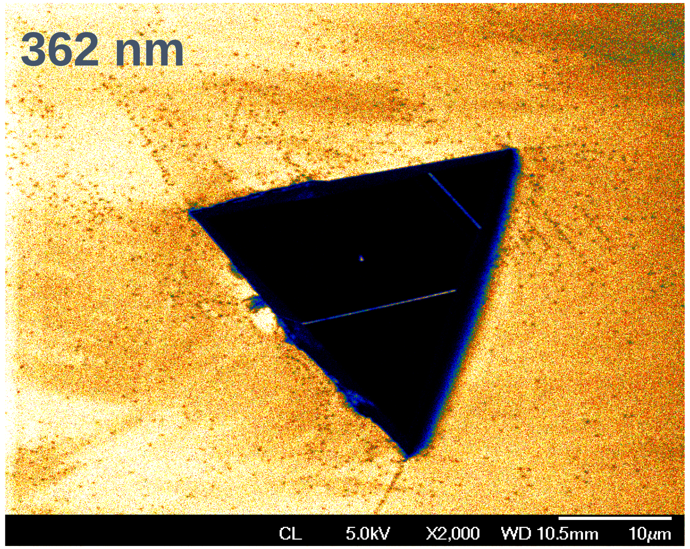
**PanCL imaging**

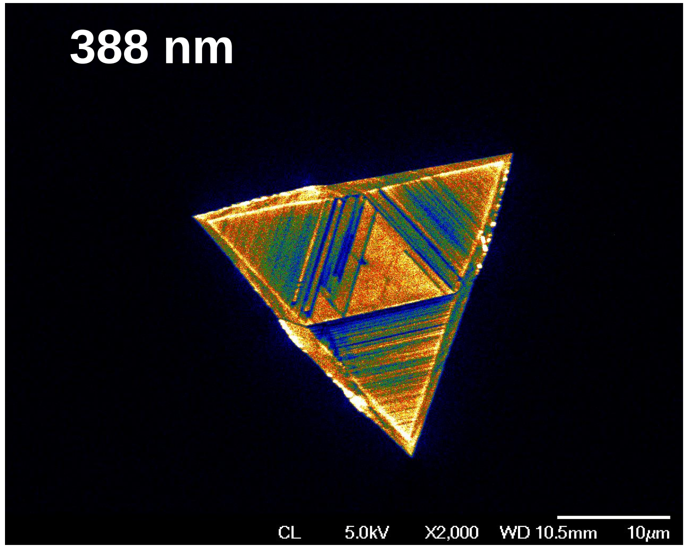



**MonoCL spectroscopy spectrum**

**MonoCL imaging at 362 nm**



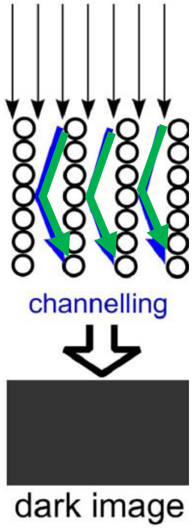

**MonoCL imaging at 388 nm**

Different optical properties

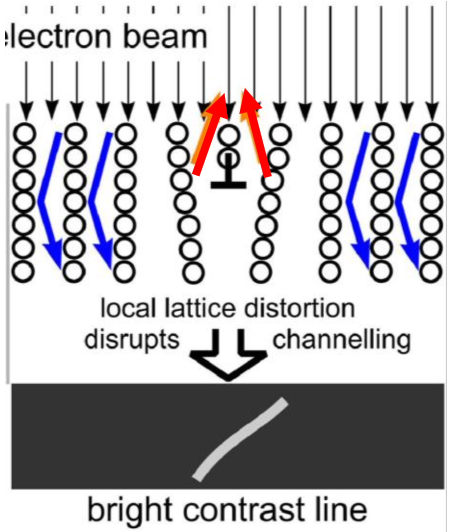
***Pseudo* color, true color UV**

# ECCI: Electron Channeling Contrast Imaging

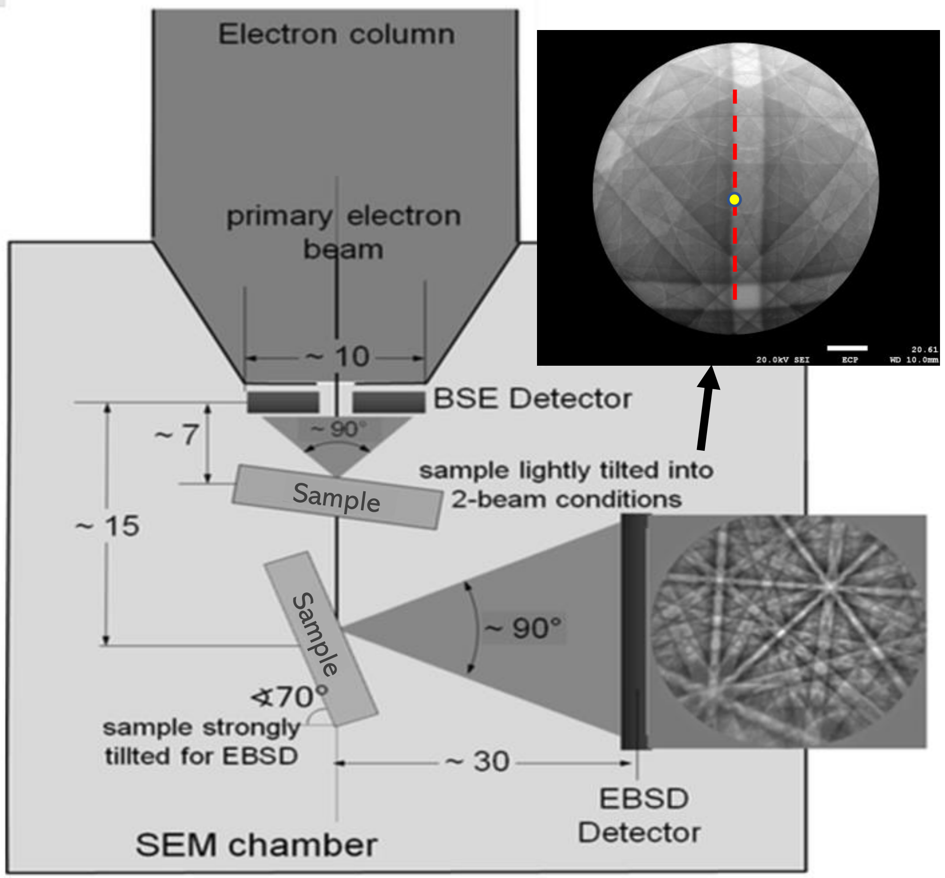
- Characterization of **crystalline** materials: metals, ceramics, semiconductors, etc.
- For imaging crystal defects: dislocations or stacking faults.
- Complementary to TEM (rapid, non-destructive, good for single crystal or big grain in polycrystal, due to **rocking** without pivot point)
- BSE, Electron channeling effect: It happens strongly when a single set of **atomic plane** satisfies Bragg's law with the incident beam, which is called two-beam diffraction condition.
- Channeling contrast: Orientation induced contrast




1) Without defects, most beam electrons penetrate along the **crystal planes**, dark image




2) Defects prevent the channeling, generating more BSE signals, and therefore a bright contrast.



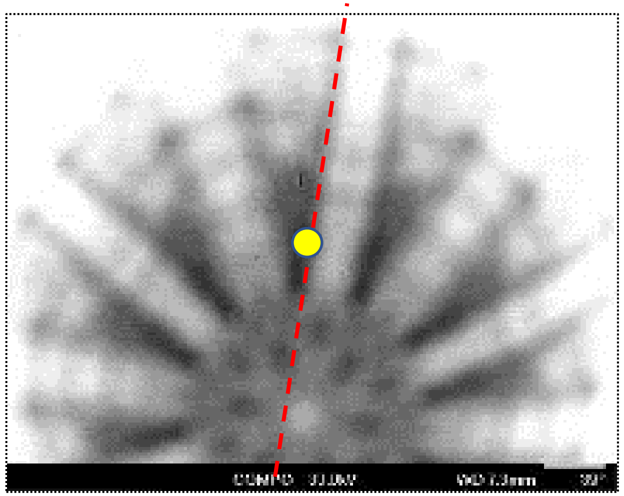

$2d \sin \theta = n\lambda$

Acta Materialia 75 (2014) 20-50

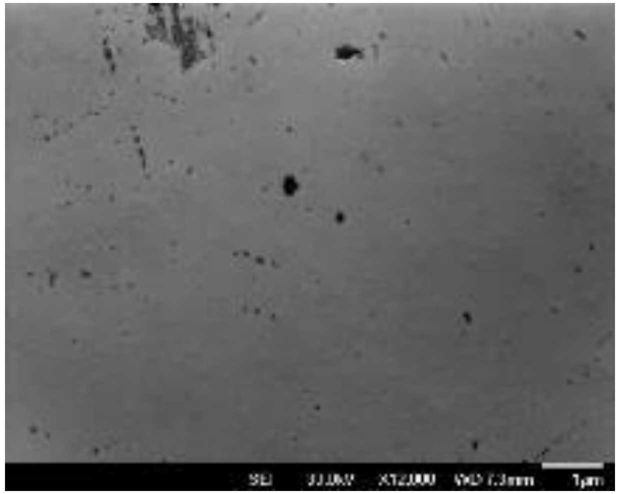
Journal of Non-Crystalline Solids 570 (2021) 121019

# ECCI: Electron Channeling Contrast Imaging

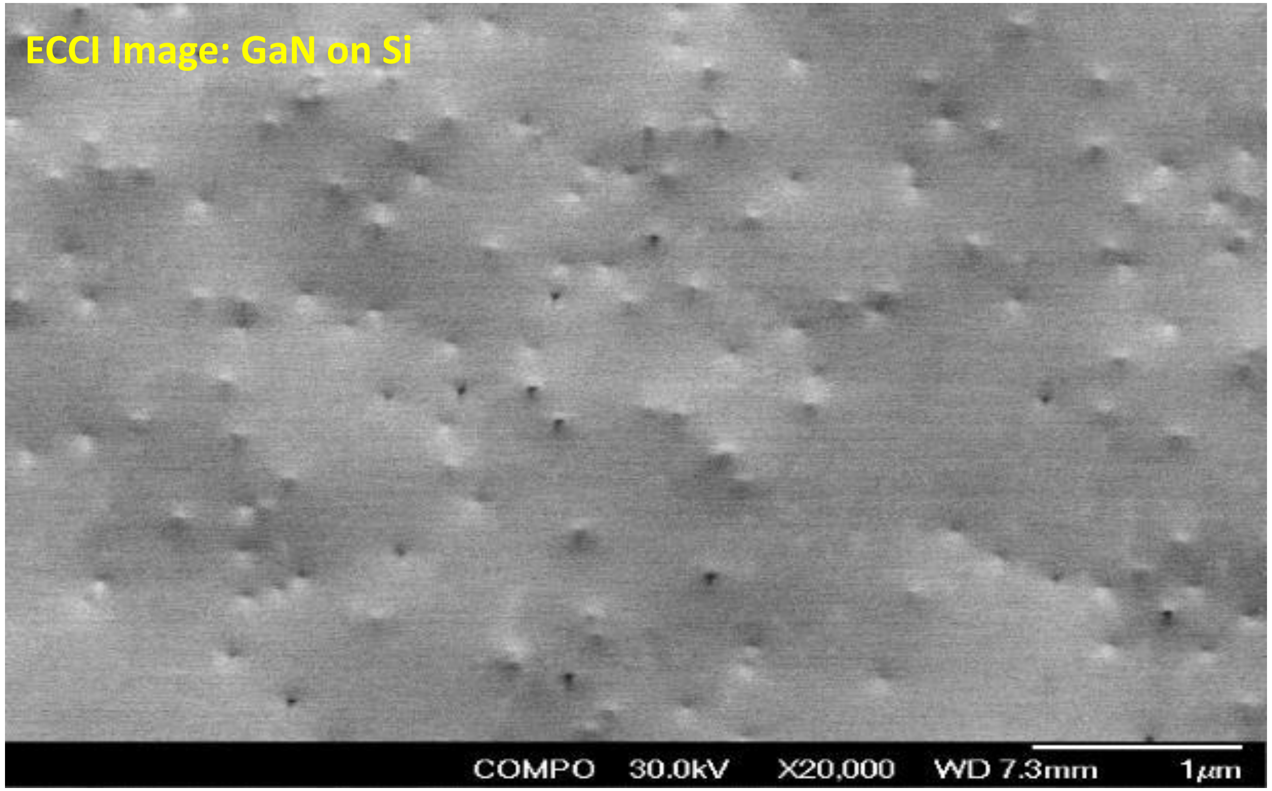
*Sample courtesy Prof. Can Bayram*



**ECP (Electron Channeling pattern)**
**BSE, Rocking beam**



**SE Imaging**




- Each dot with **B-W** (black-white) contrast is a threading dislocation
- **B-W** contrast corresponds to the direction of dislocation
- **Calculate dislocation density or do more detailed dislocation analysis**

# Outline

## Part I: SEM

- What is SEM & How does it work
  - Electron Guns (Electron Sources)
  - Electron Lenses and Lens Aberrations/Corrections
  - Major electron beam parameters and how to control them
- Applications
  - SE & BSE imaging and detectors
  - Analytical techniques: EDS, EBSD, CL, ECCI

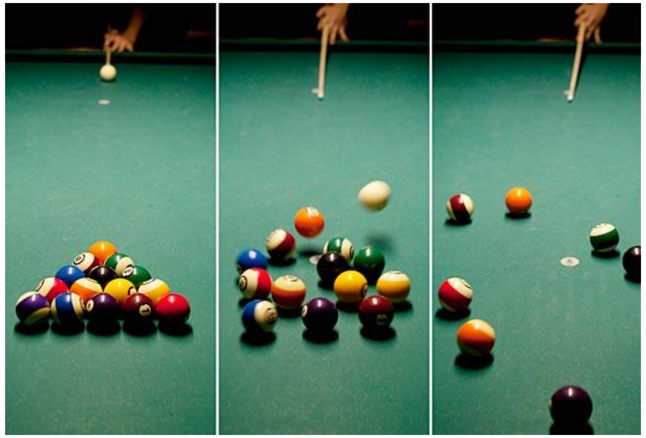
## Part II: FIB (Focused Ion Beams)

- How does FIB work
- Common applications

# The FIB: How Does it Work?

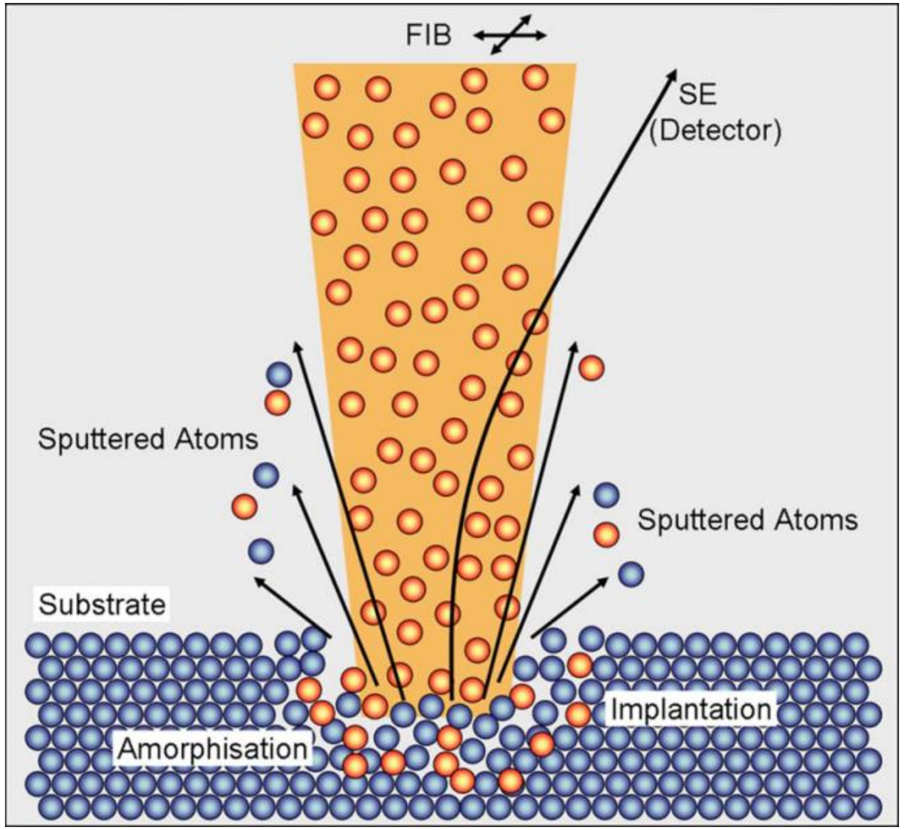
Think of the FIB like this:

Fire the projectile



Apply to this:

The sputtered atoms will go flying all over the place.




- Mill the sample, cut it into pieces;
- As a cutting tool or as an imaging tool

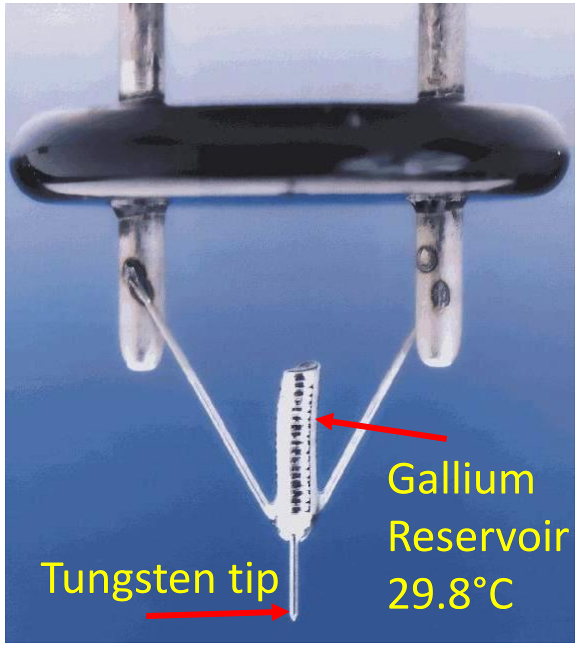
I. Utke, *et al.* J. Vac. Sci. Technol. B 26(4) 2008

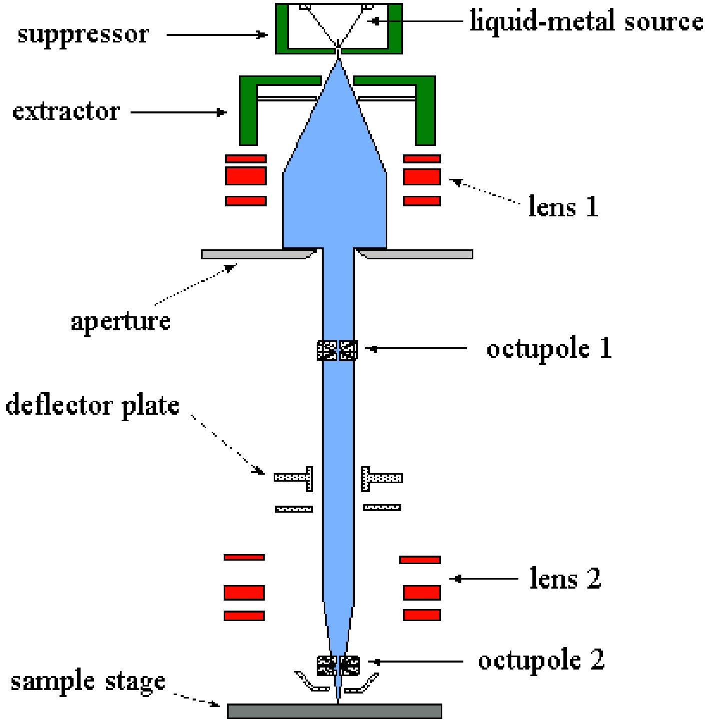
http://www.bedrockbilliards.com/wp-content/uploads/bedrock-pool-break.jpg

http://www.themaneater.com/media/2008/1106/photos/bowling021-e_jpg_900x800_q85.jpg

# FIB System – Source & Components

LMIS

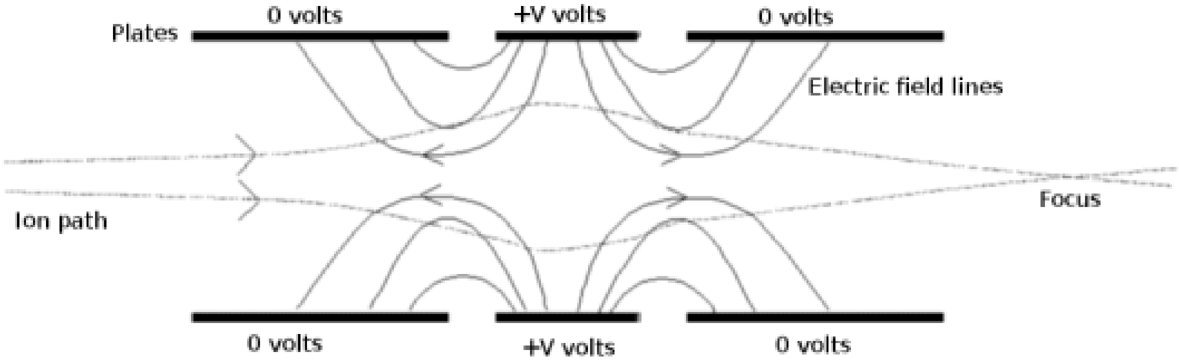



*Kinetic Energy = 1/2 **m** v²*
- ***m** is the mass measured in kilograms*
- ***v** is the velocity of meters per second*

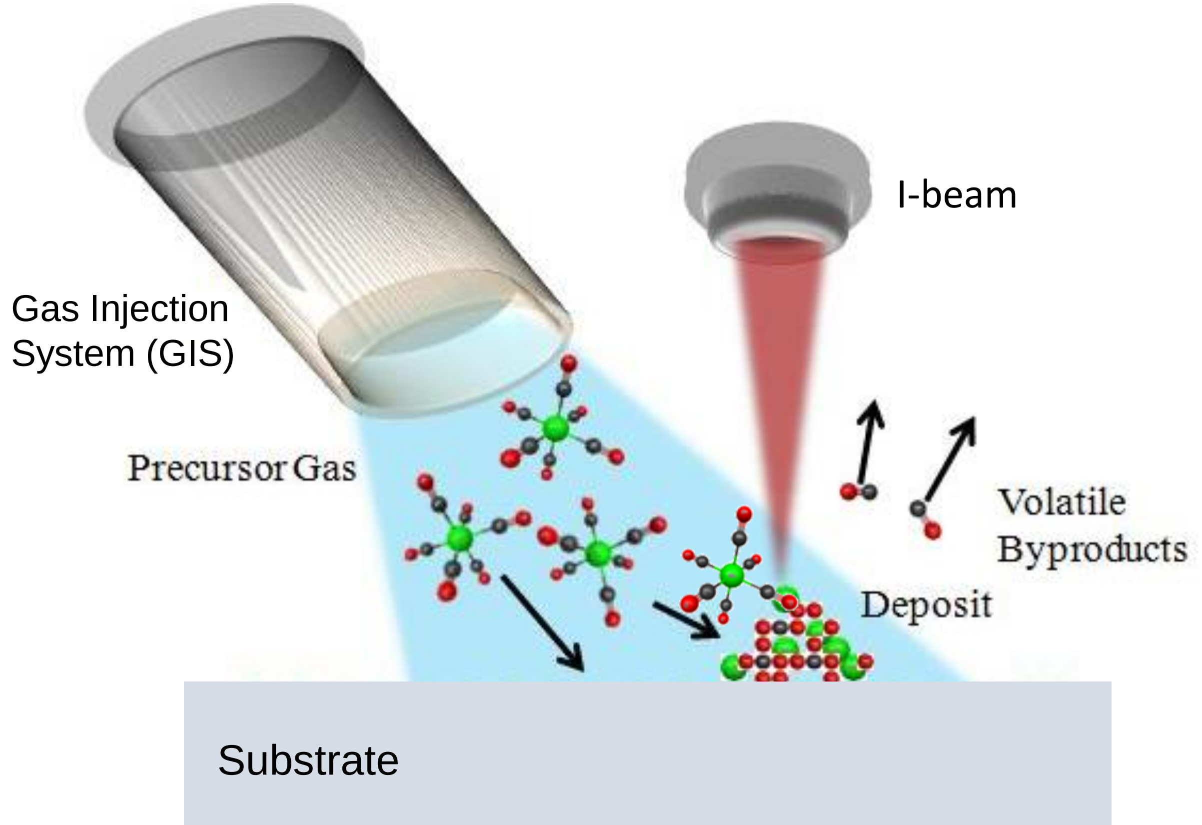
Einzel Lens

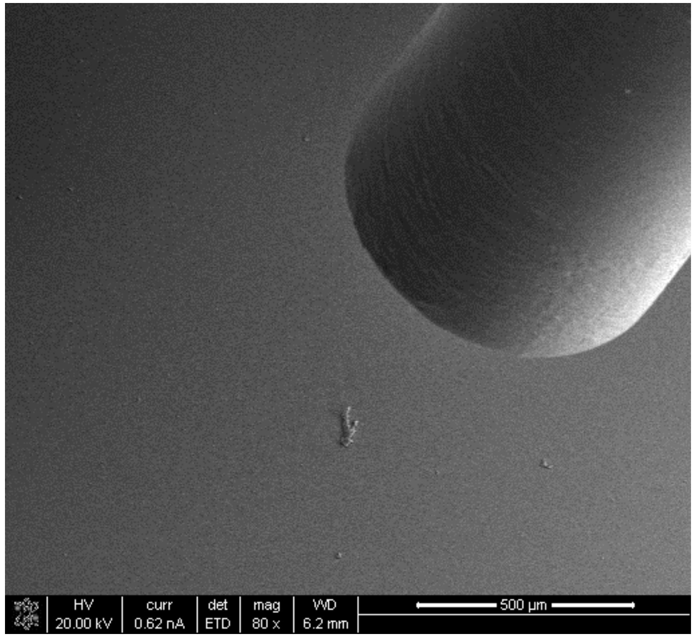
- Liquid Metal Ion Source: Gallium reservoir
  - Other types of sources: Au, Si, Ne, He
- Electrostatic lenses: extract, shape
  - Ions react weakly to electromagnetic lenses
  - Electrostatic lenses act against the charged particle, ions, which is caused by creating a high-electric field between the electrodes.




https://www.researchgate.net/figure/A-typical-gallium-liquid-metal-ion-source-A-current-passed-through-the-hairpin-melts_fig21_339830750

# Beam Induced Deposition

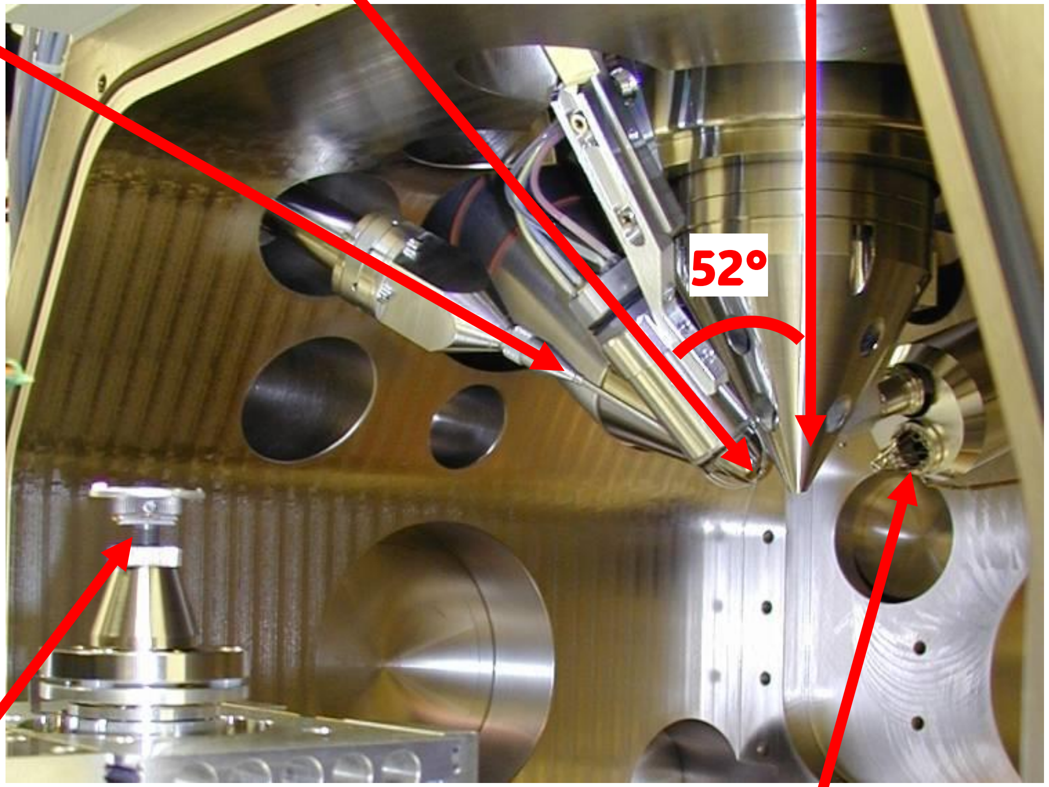



SEM image of inserted GIS
~50-200um distance

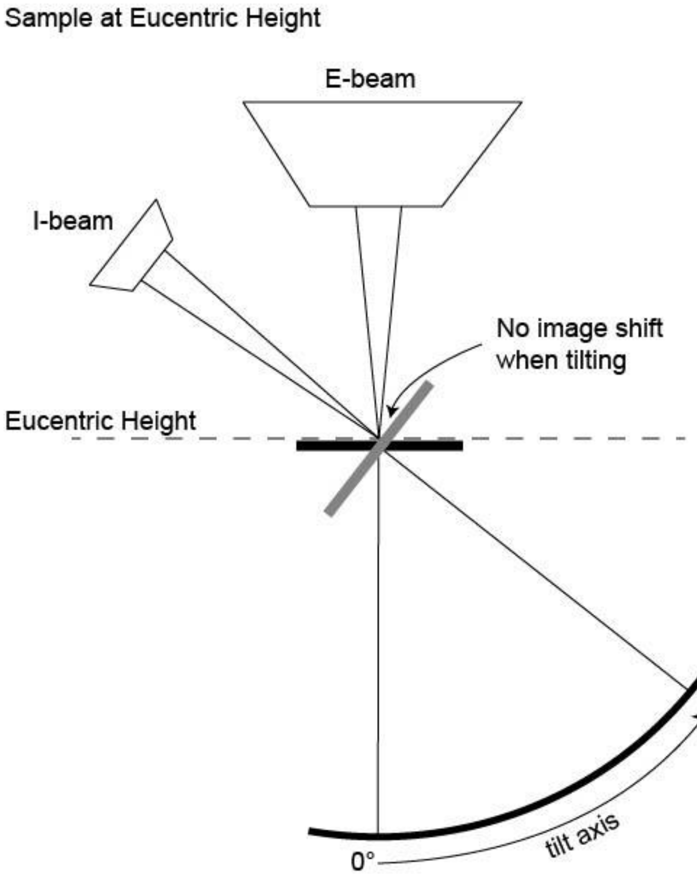
- Precursor gas flows through the needle
- Precursor gas can be a range of materials, such as Pt-rich, W-rich, or C.
- Beam-induced deposition, structures are deposited near the beam:
  - The precursor gas is released onto the sample, then cracked by the ion beam.
  - The non-volatile products (here Pt-rich solid) adsorb on the sample surface.
  - The volatile products are removed by the pumps.

**Pt Injection Needle-GIS** **FIB Column** **Electron Column**




**Specimen Stage (chamber door open)**

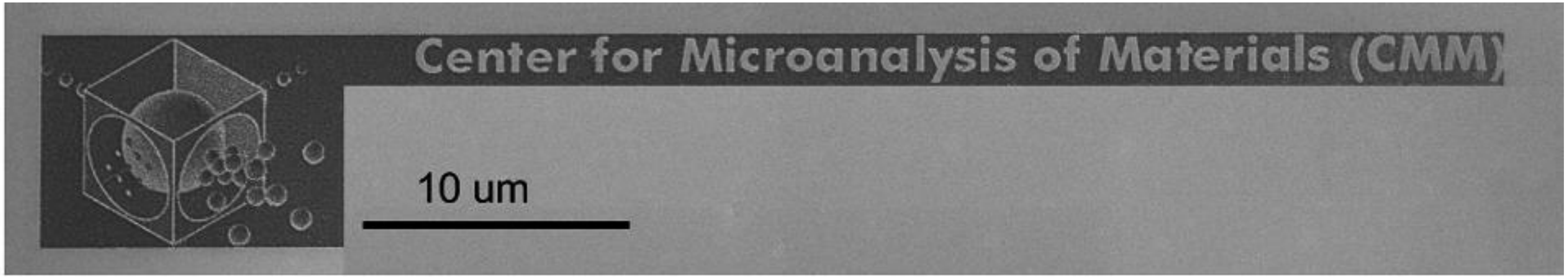
**ET & TLD electron detectors**

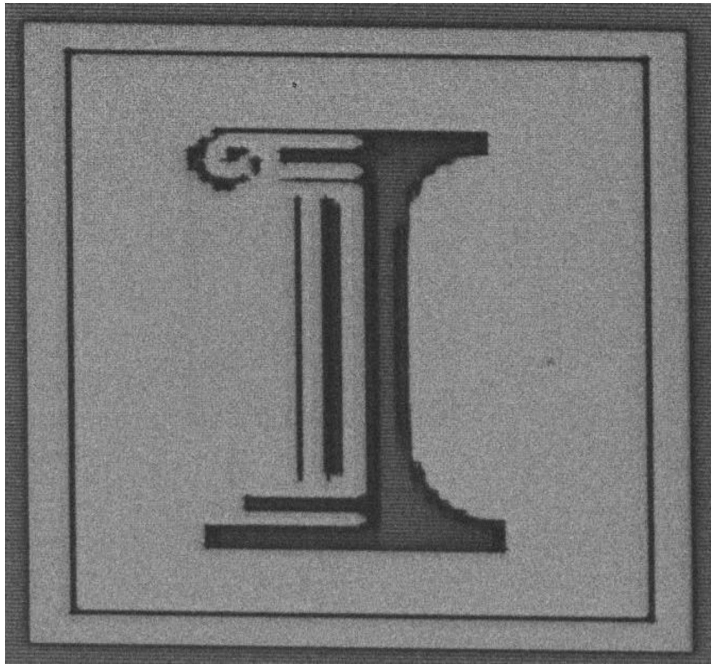


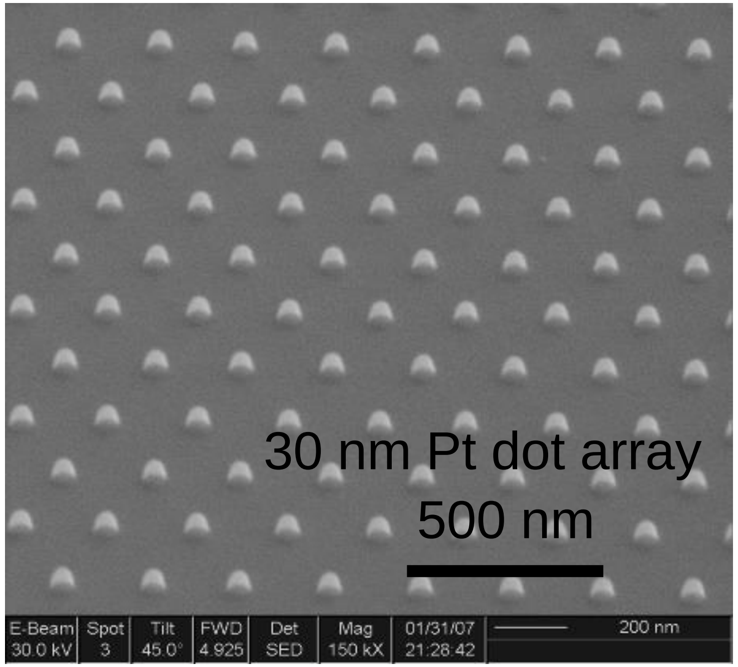

"**Eucentric**" is a point in the stage height control ("Z") where the sample's image does not move/shift when the sample is tilted.

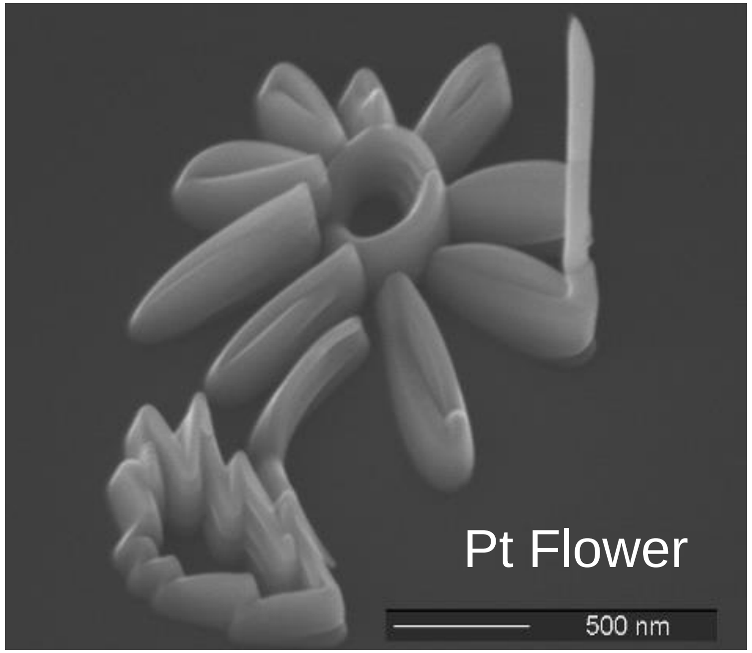
# FIB Applications: Controlled Etching & Deposition

Etched or deposited structures using **grey-scale bitmaps** or **pattern object scripting**.



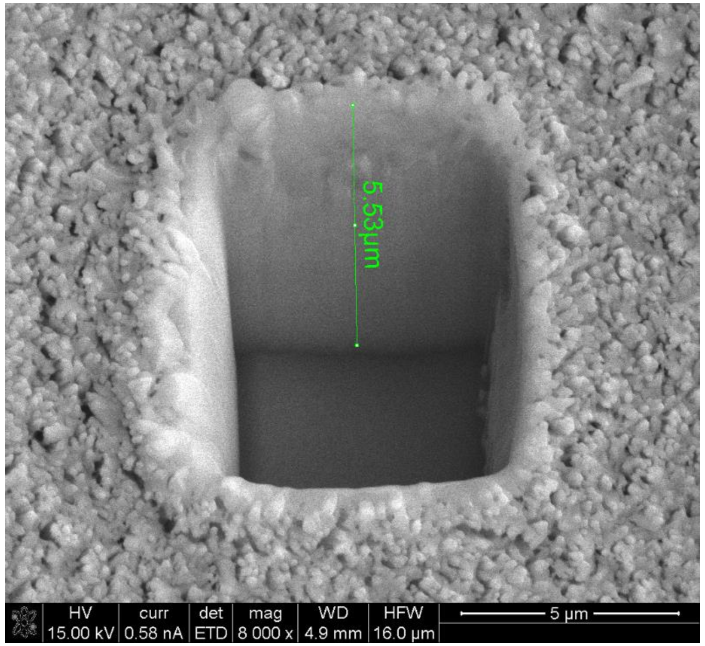


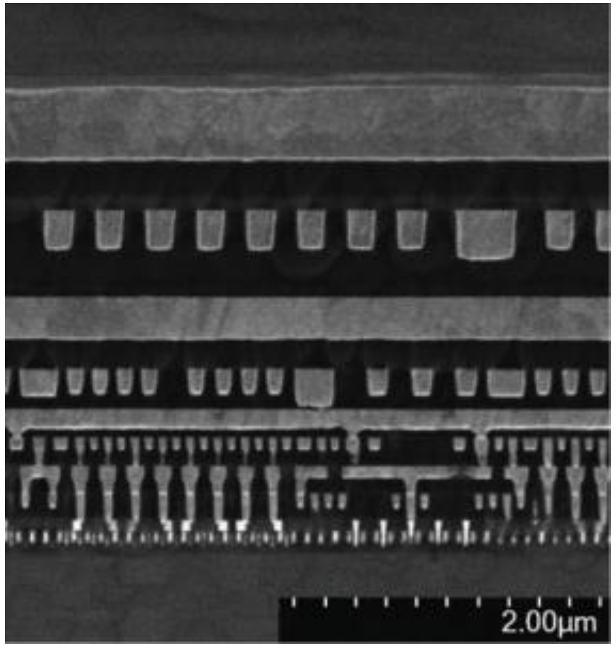




Used as masks or emitters

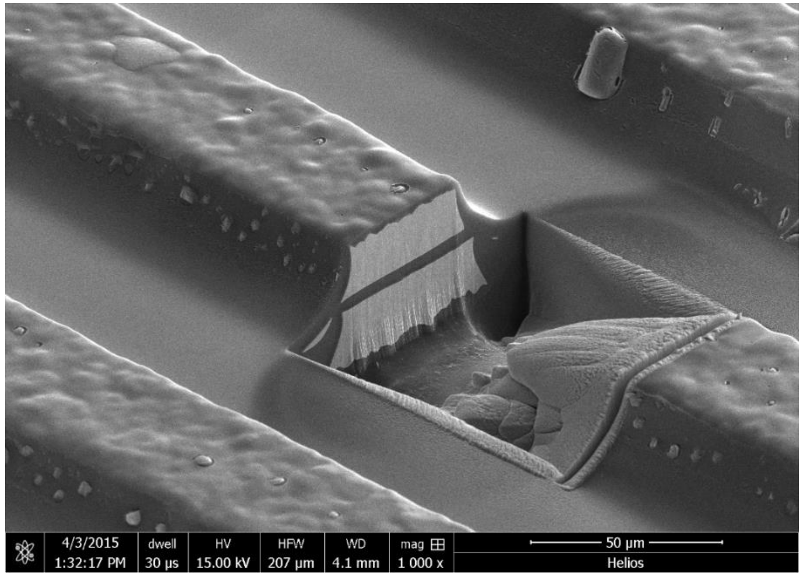
# Cutting through materials & seeing Cross-sections: FIB Specialty

The thickness of a deposited layer




In the IC (integrated circuit) industry: cut through the chips, for semiconductor and chip analysis




***Sample courtesy Prof. John Rogers***

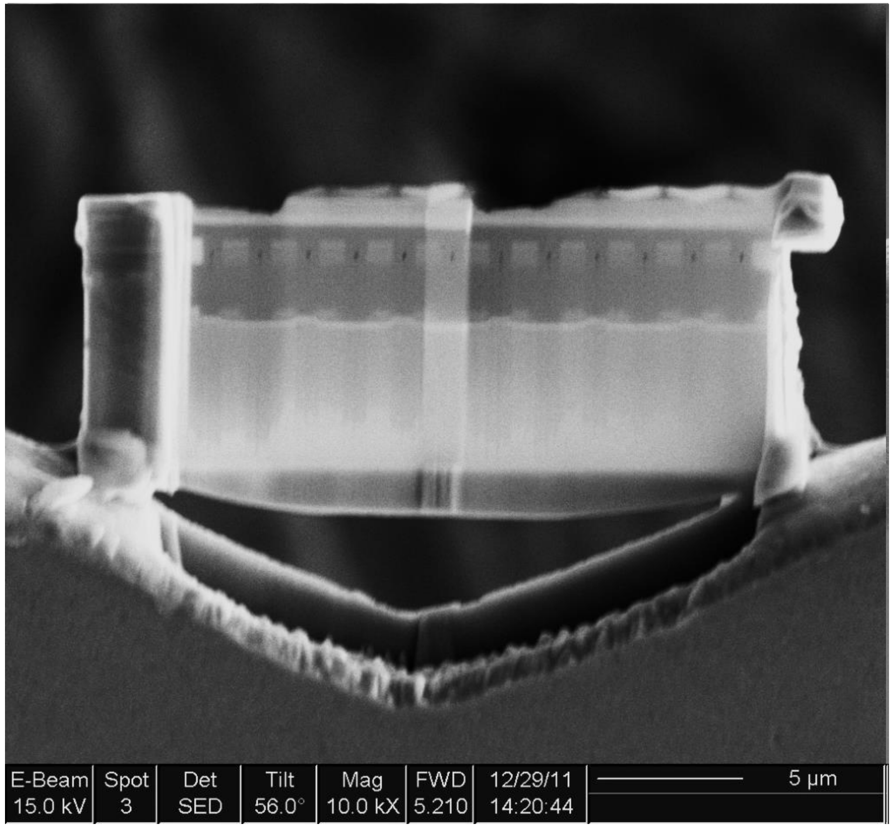
Make big trenches to expose the internal features

UR - Chip x-section: http://electroiq.com/blog/2014/12/from-transistors-to-bumps-preparing-sem-cross-sections-by-combining-site-specific-cleaving-and-broad-ion-milling/

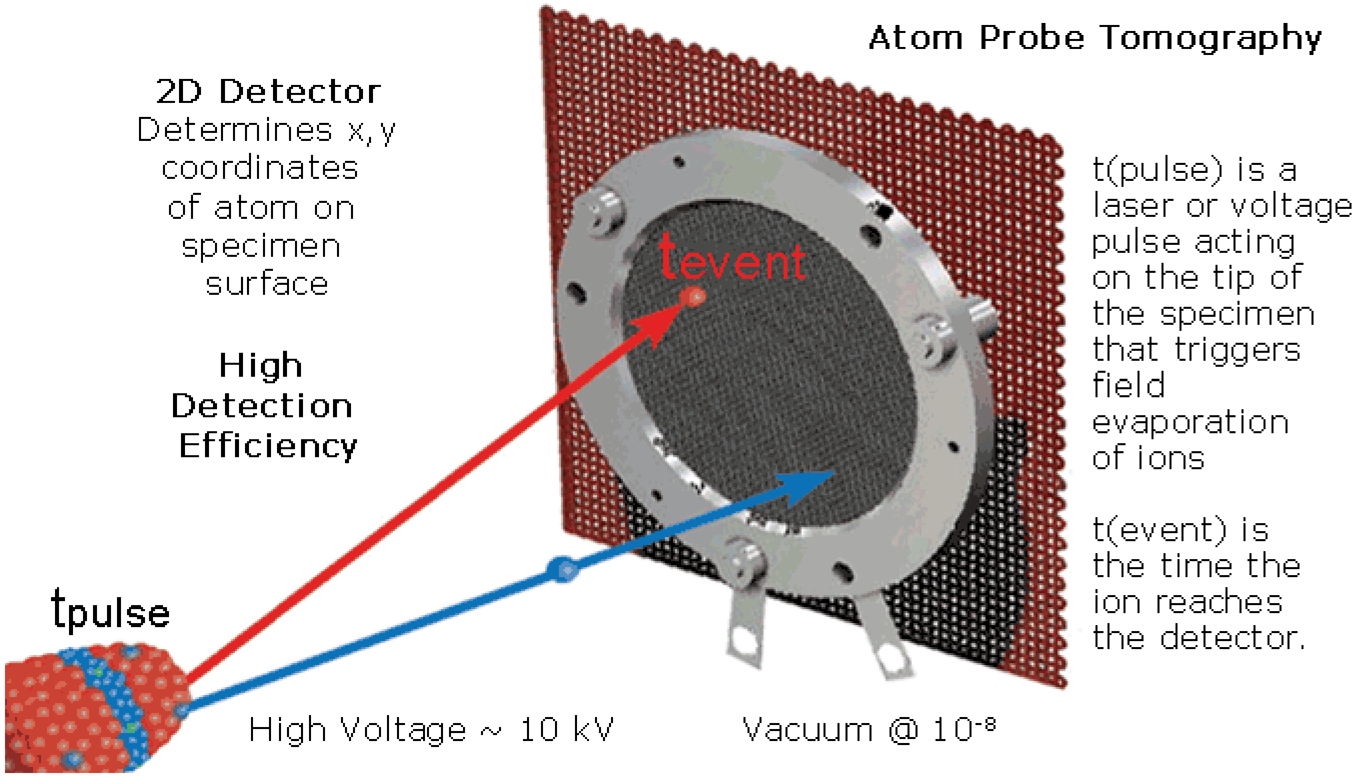
# Site-specific TEM Sample Prep with FIB & Omni probe

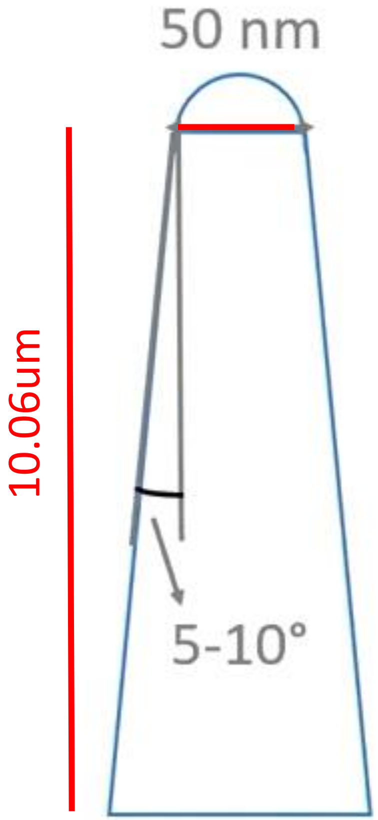
The major steps that we need to make a TEM sample:

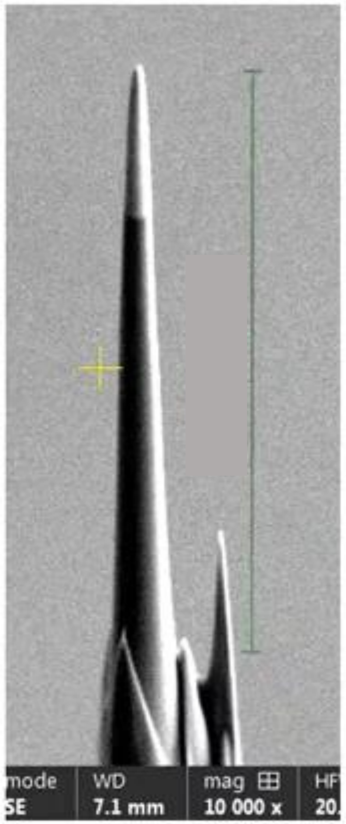
- Step 1 - Locate the area of interest
- Step 2 - Deposit a **protective** platinum or Carbon layer on the spot
- Step 3 – Mill out the outsides around the protection layer, two **trenches** milled on both sides of the area
- Step 4 – Thinning the wedge sample
- Step 5 - Perform “under **J cut**“ and weld the needle to the sample with Pt
- Step 6 – Mill to release the sample from the substrate, **lift it out**, and transfer it to the grid
- Step 7 – "Weld" the sample to a Cu TEM half-grid and cut the needle free
- Step 8 - ion beam **thin and polish** the sample to electron transparency (<100nm thickness)



# Atom Probe Tomography Sample Preparation



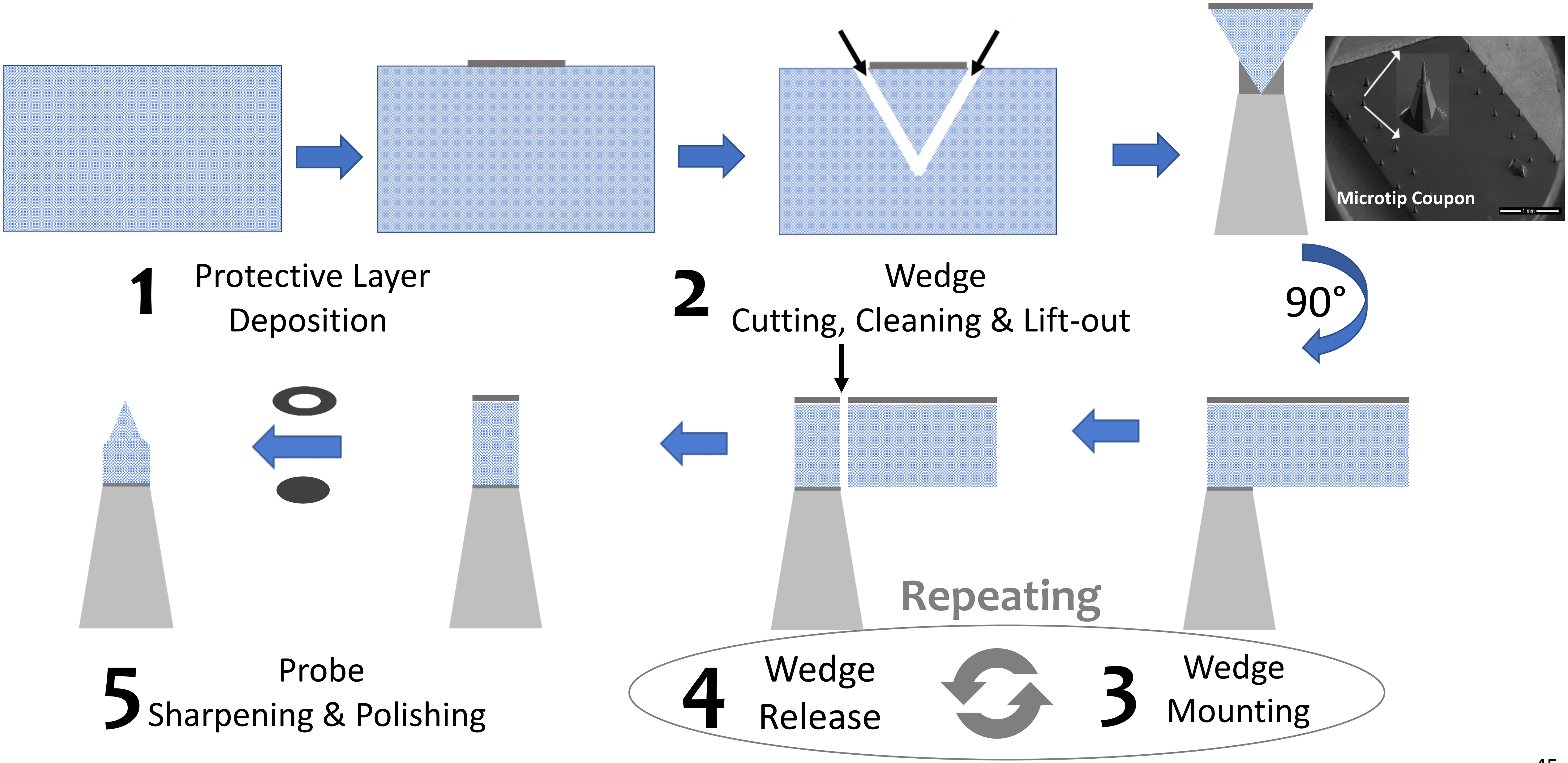




## The ideal shape of an atom probe

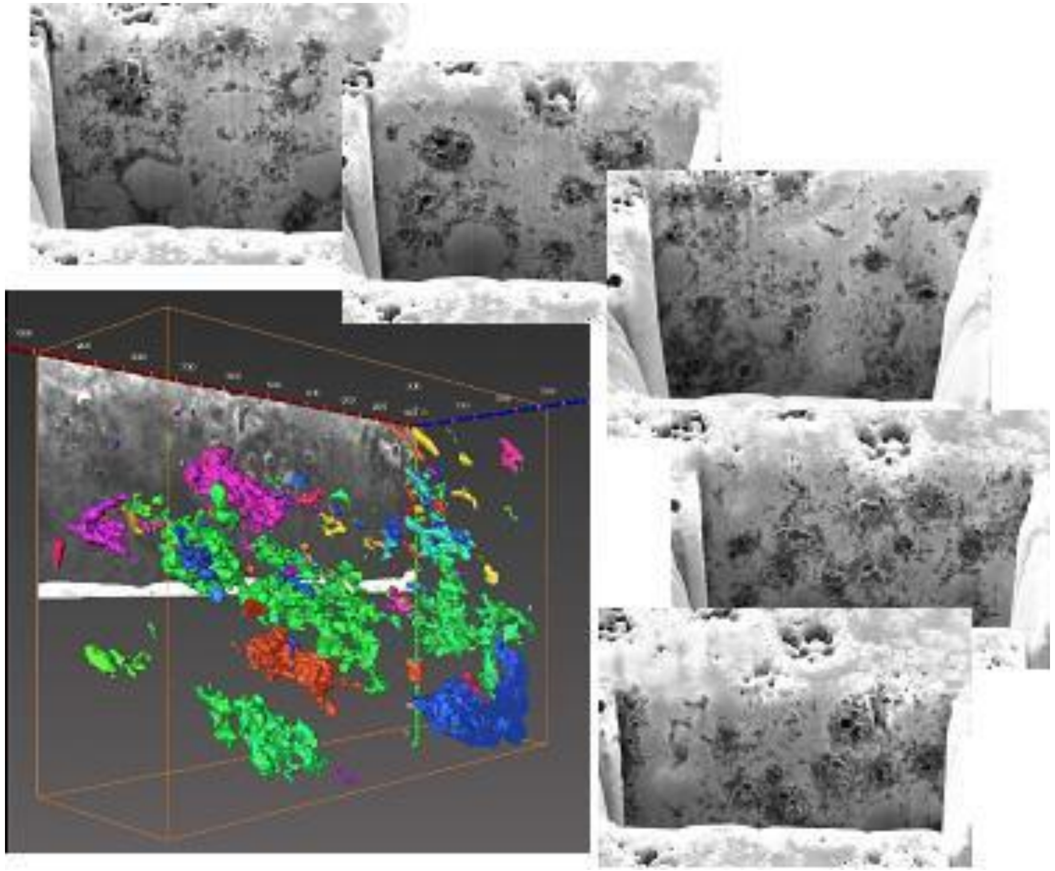
- Hemispherical cap with a diameter of ~ 50 nm
- On a truncated cone with a half shank angle between 5° to 10 °
- No parasitic spikes at least 10 µm away from the ROI in the tip

https://www.cameca.com/products/apt/technique

# Working Flow: Atom Probe Sample Preparation



# Serial Sectioning and 3D Reconstruction



FIB Sectioning of **Shale** and Resulting 3D Reconstruction Showing Pore Structure

- FIB was used to slice out the individual sections
- Image each section and reconstruct a 3D model of the sample
- Choice of detectors for more analysis

http://www.jeolusa.com/RESOURCES/ElectronOptics/ImageGallery/tabid/323/galleryType/SlideShow/ItemID/219/AlbumID/26/Default.aspx

# FIB Pro’s and Con’s

- Excellent for:
  - Patterning small areas (Processing area 10s~100s μm per scan)
  - Cross-sections
  - Precision TEM (10~20 μm in length) and APT sample preparation
  - Serial sectioning and 3D reconstruction

- Is not good for:
  - Replace e-beam or optical lithography! (Throughput)
  - Make large (100’s of μm) TEM samples

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