Applied Physics 298r

Scanning Electron Microscopy (SEM)

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April 26, 2004

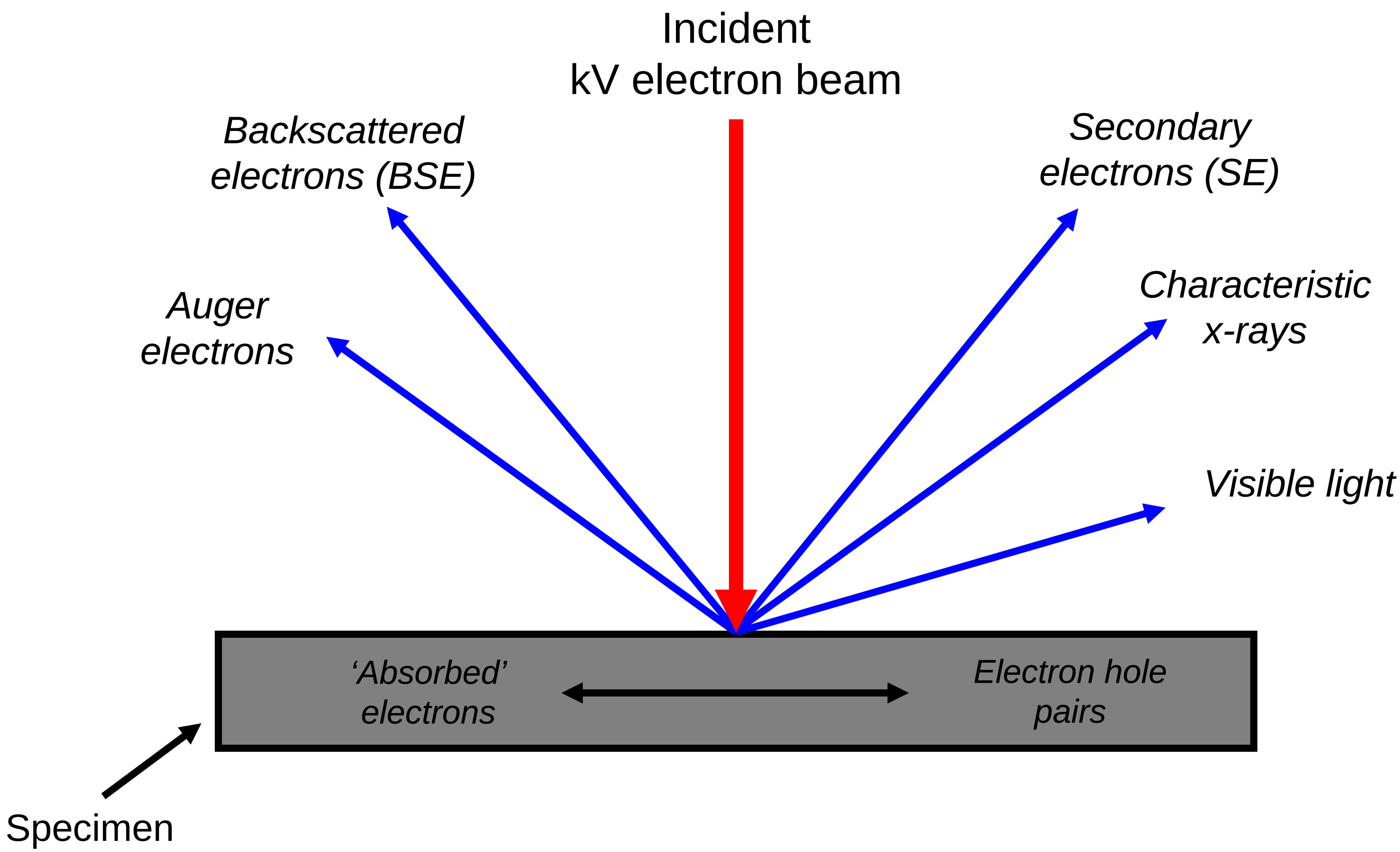
Basic Methods of Materials Analysis

- Look at the Material (Shiny or Dull?)
- Hold Material (Heavy of Light?)
- Taste Material (Surface Chemistry?)
- Bite Material (Ductile ?)
- Drop Test (Brittle ?)

- Put it in a Scanning Electron Microscope (SEM) !

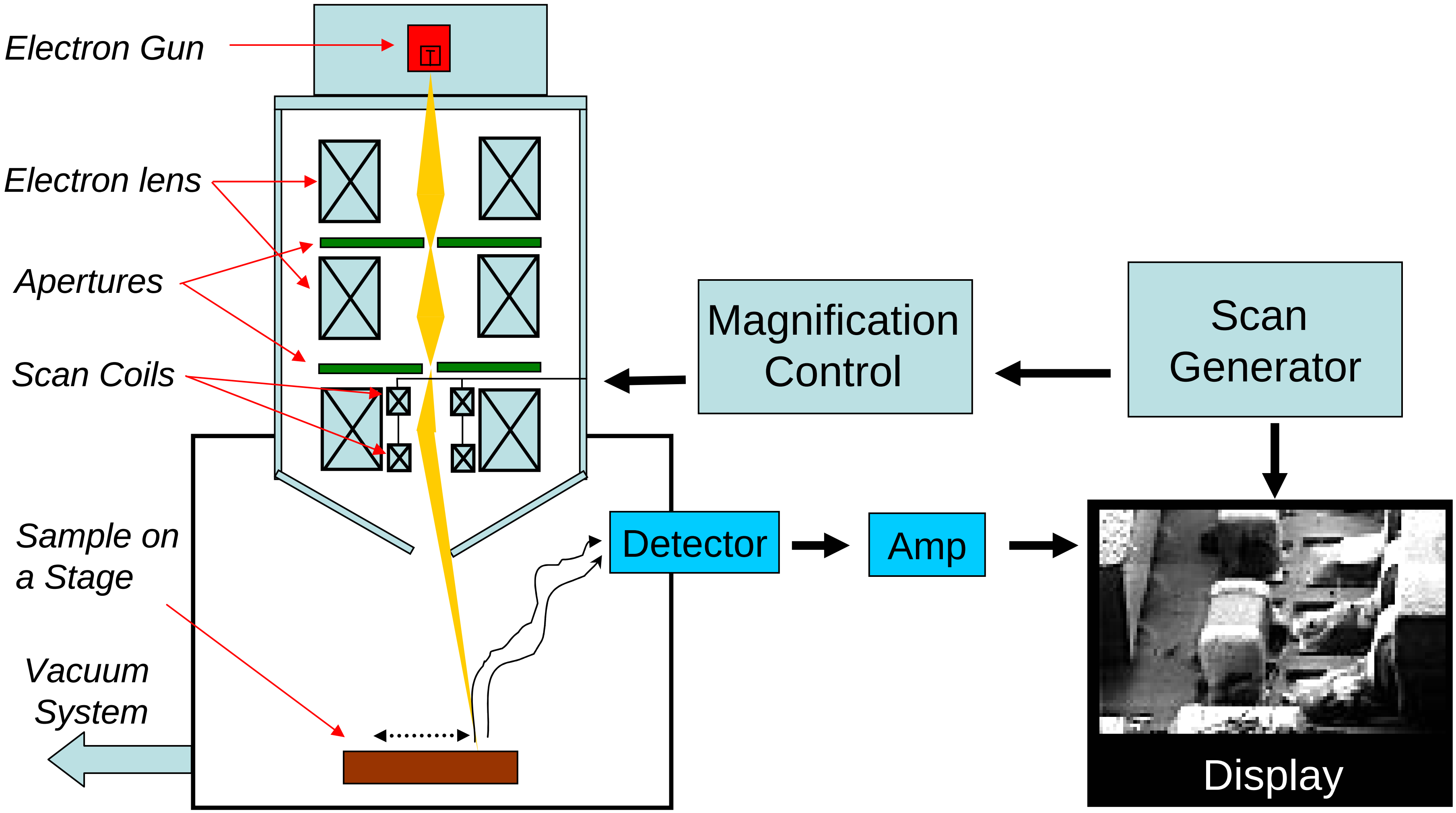
- Other Methods

Large Number of Signals



Scanning Electron Microscope (SEM)

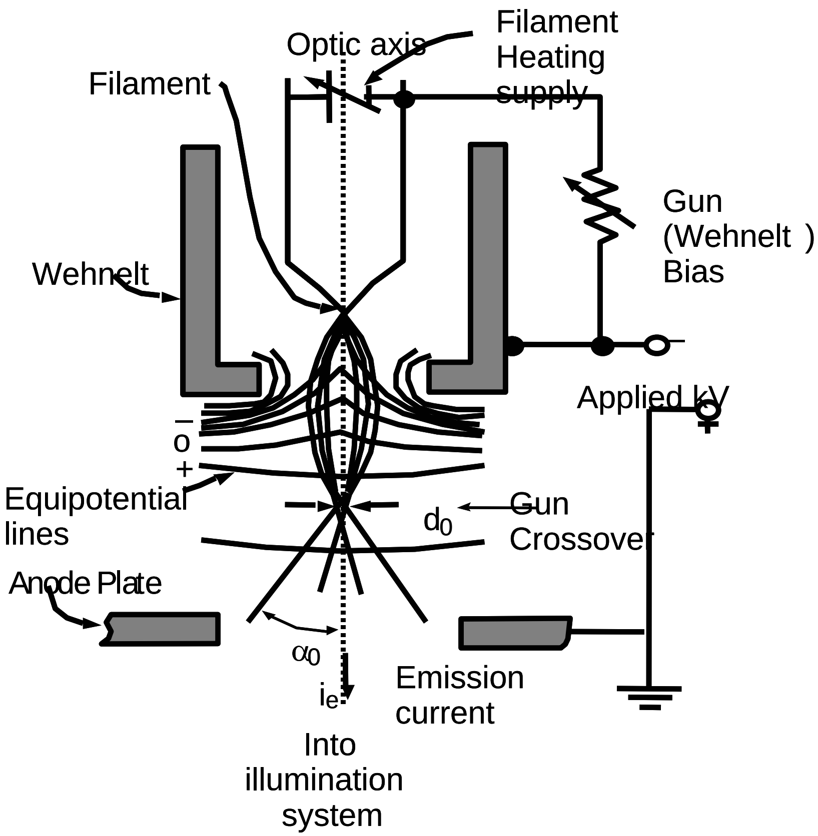
What is an SEM?



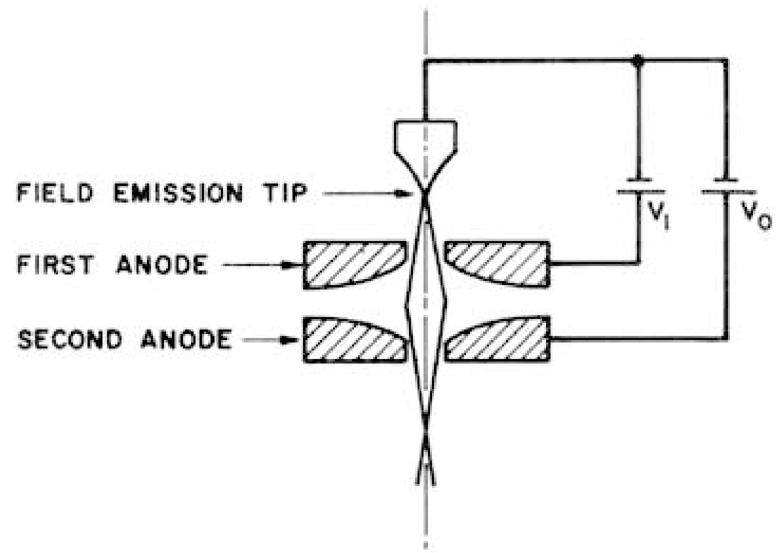
Types of Electron Gun

- Two main types;
  - Thermal
    - Tungsten Filament
    - LaB_6 Filament
  - Field Emission
    - Cold Field Emission
    - Schottky Field emission gun

The Electron Gun



Thermal Emission

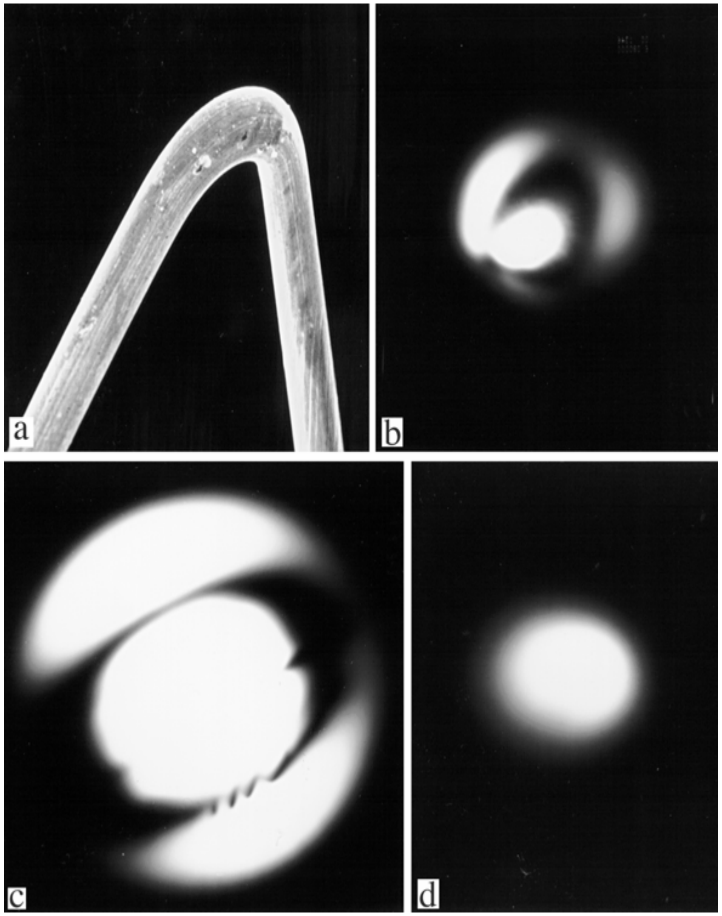


Field Emission

Hairpin Tungsten Filament

Light bulb

Underfocus
& off center



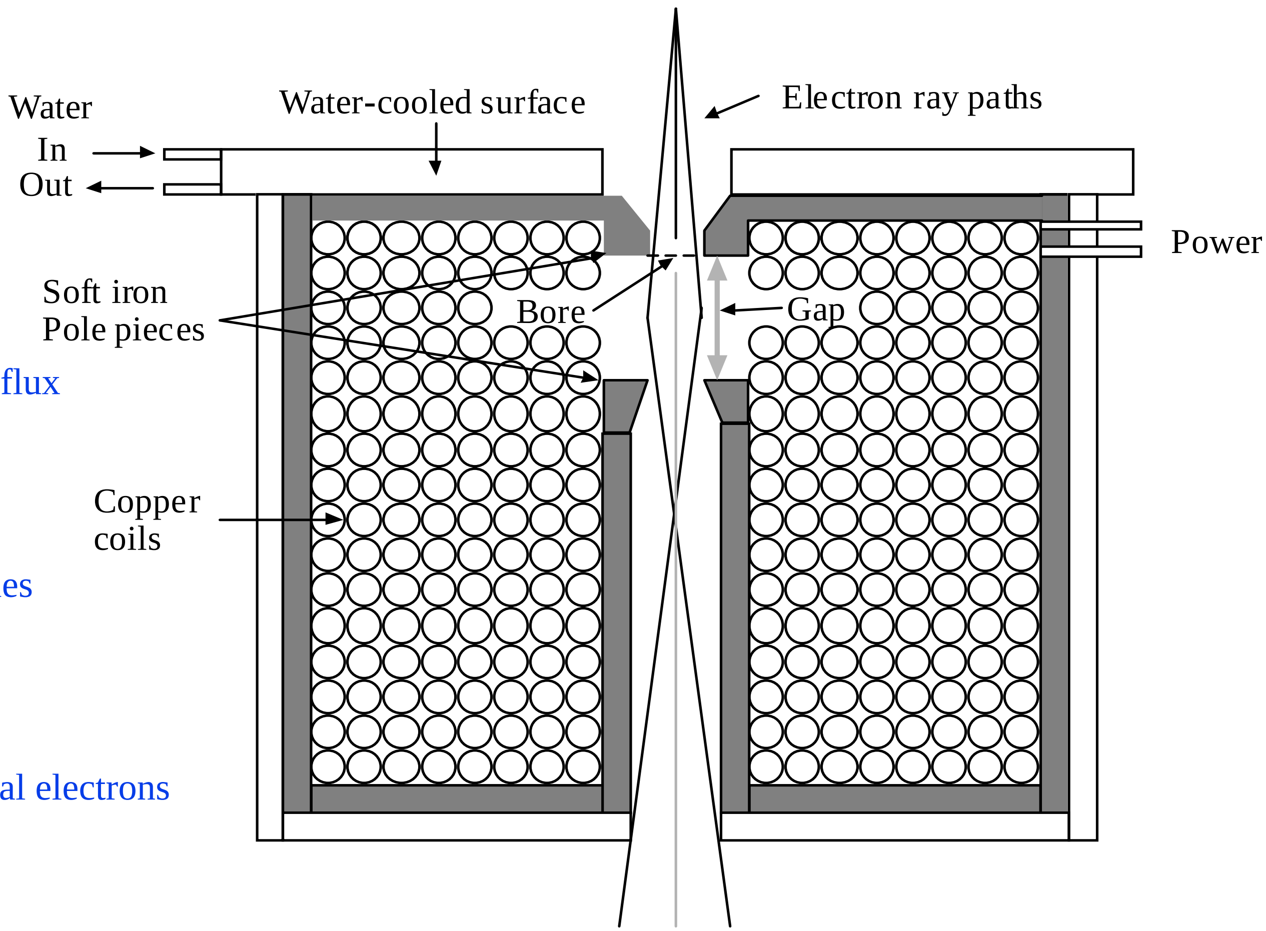
Underfocus
but centered

Centered &
focussed

Characteristics of Sources

	Units	Tungsten	LaB_6	FEG
Operating Temperature	K	2700	1700	300
Current Density	A/m^2	$5x10^4$	10^6	10^{10}
Crossover size	µm	50	10	<0.01
Energy spread	eV	3	1.5	0.3
Stability	% / hr	<1	<1	5
Vacuum	Pa	10^{-2}	10^{-4}	10^{-8}
Lifetime	hr	100	500	>1000

Magnetic lens

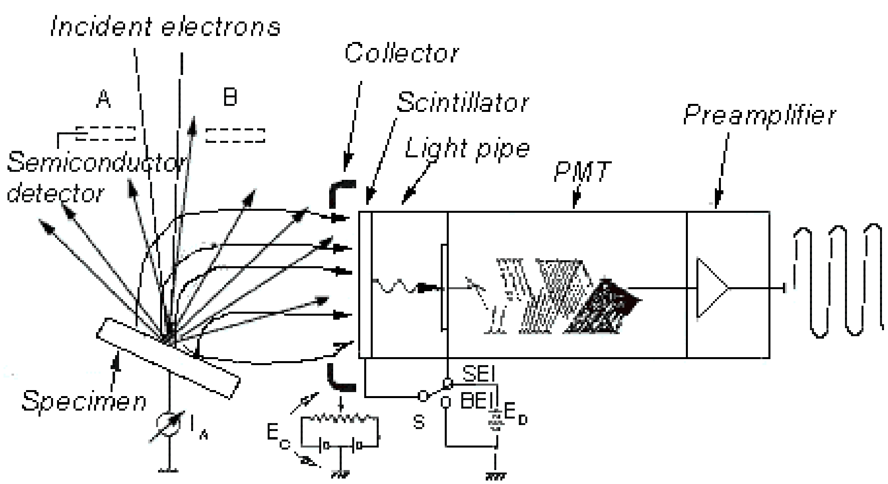


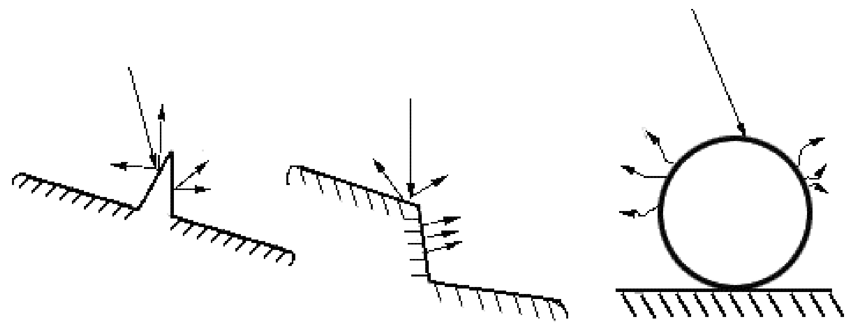
Concentrate flux

Field varies

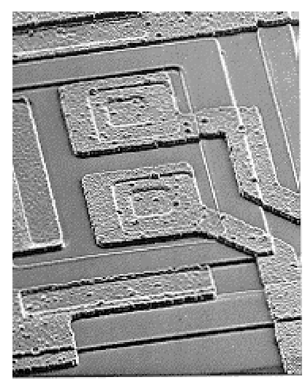
Zero force on axial electrons

Secondary Electron Detector

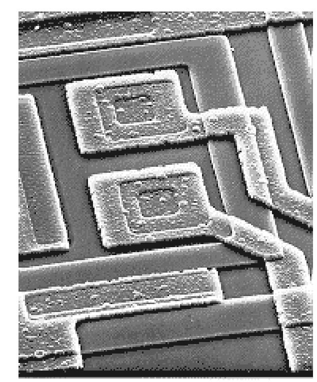




(a) Protrusion (b) Edge (c) Circumference

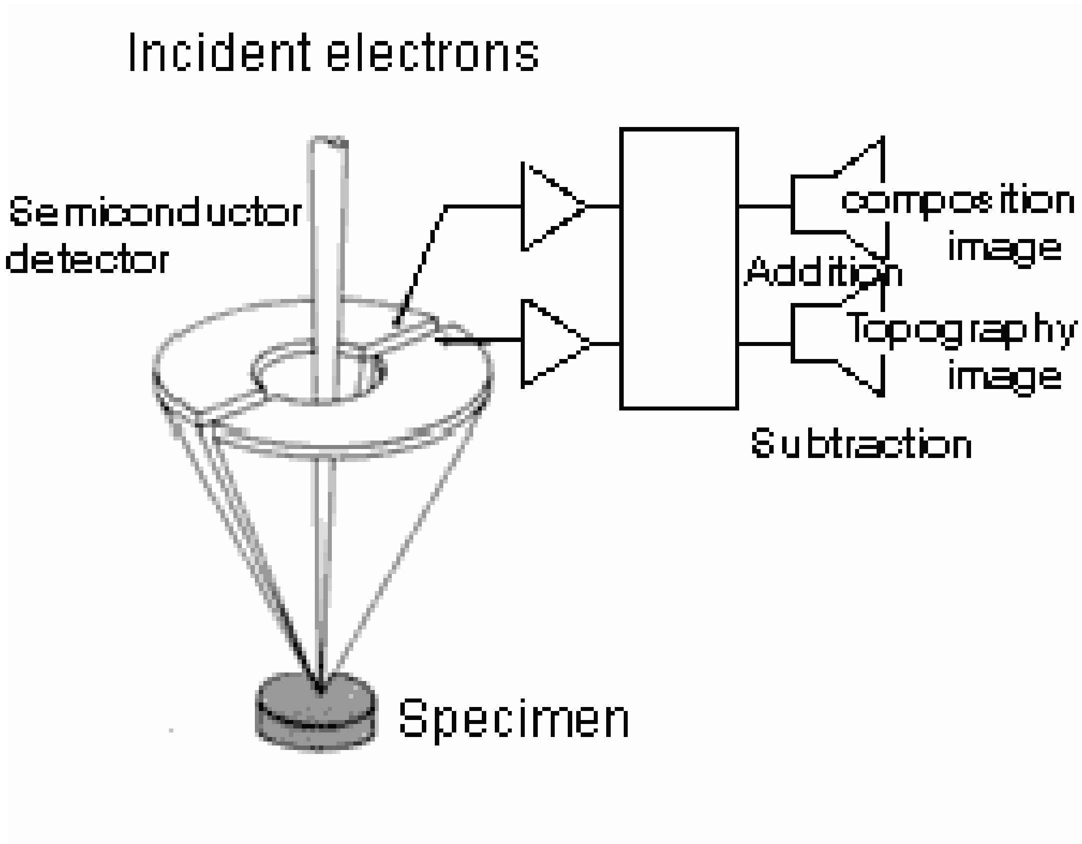


(a) 5 kV x720
Tilt angle: 50°

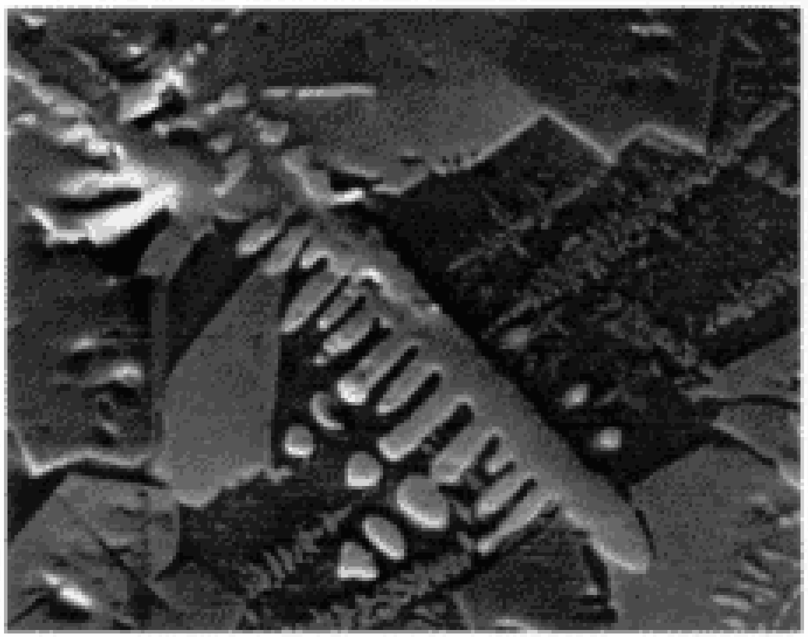


(b) 25 kV x720
Tilt angle: 50°

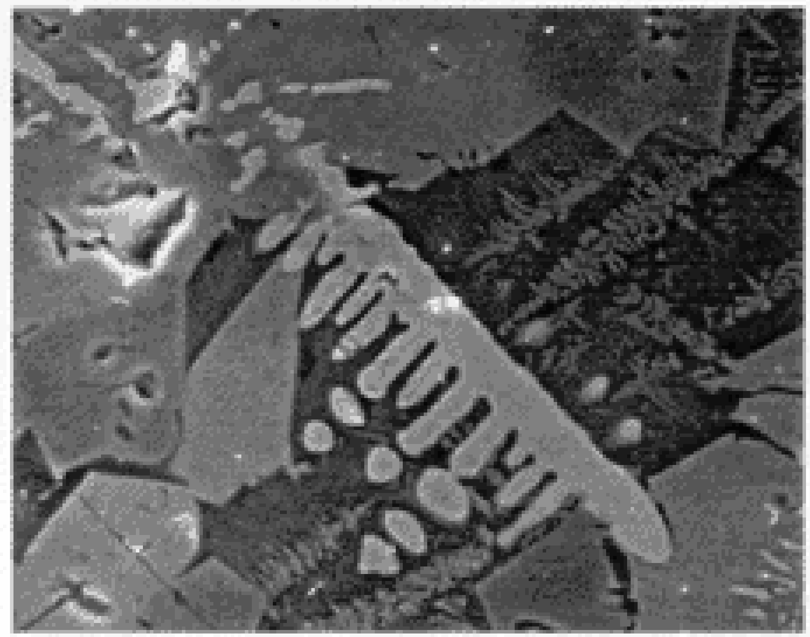
Backscattered Electron Detector



SEM: Electron Detectors



Backscattered Electron Image



Secondary Electron Image

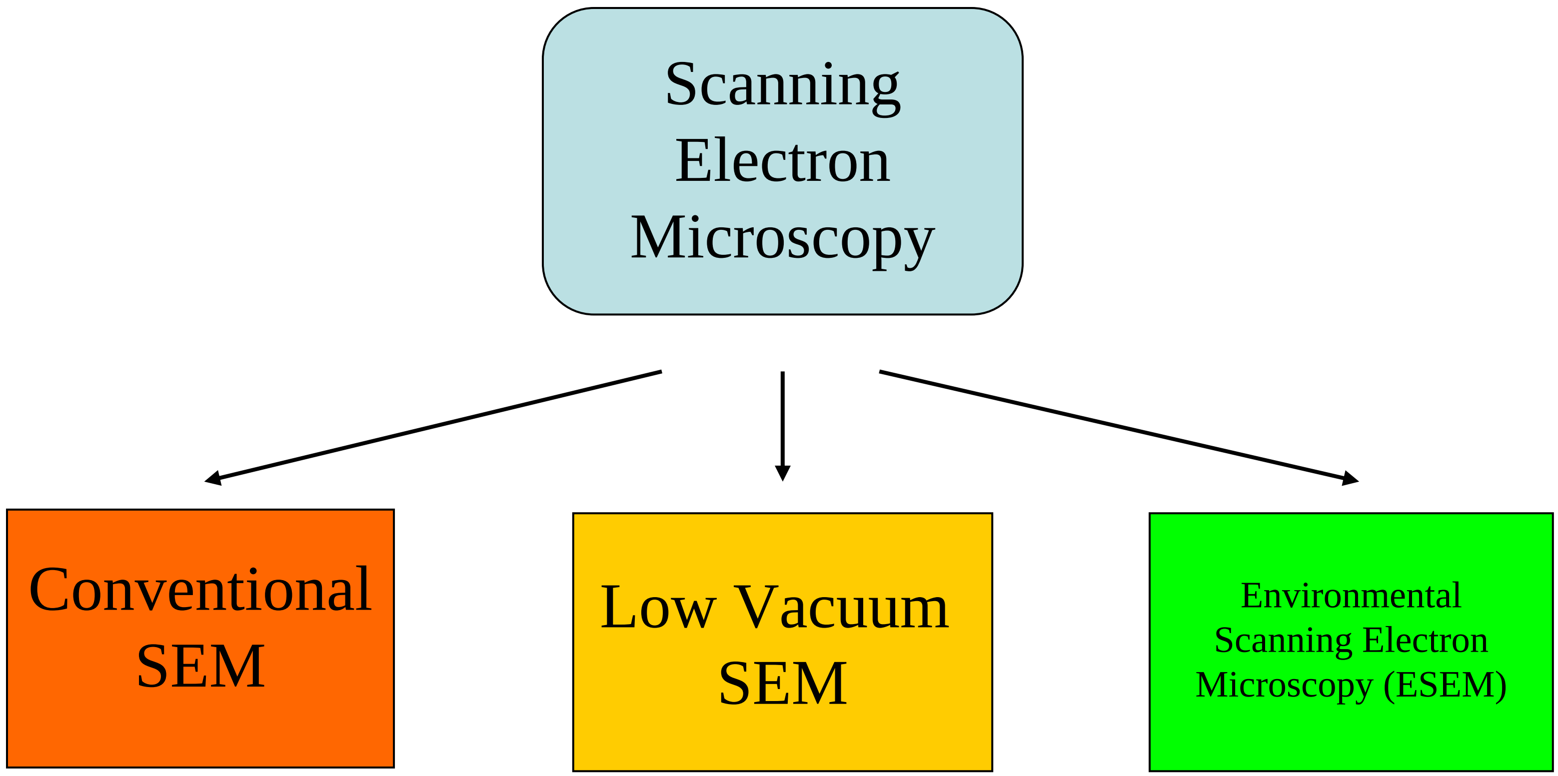
Brief History of SEM

- 1935 Knoll Scanning TEM
- 1942 RCA Labs, Zworykin
- 1948 Cambridge University C. W. Oatley
- 1963 Pease, SEM V became Cambridge Scientific Instruments Mark 1 Stereoscan
- 1960 Everhart and Thornley : development of the SE Detector

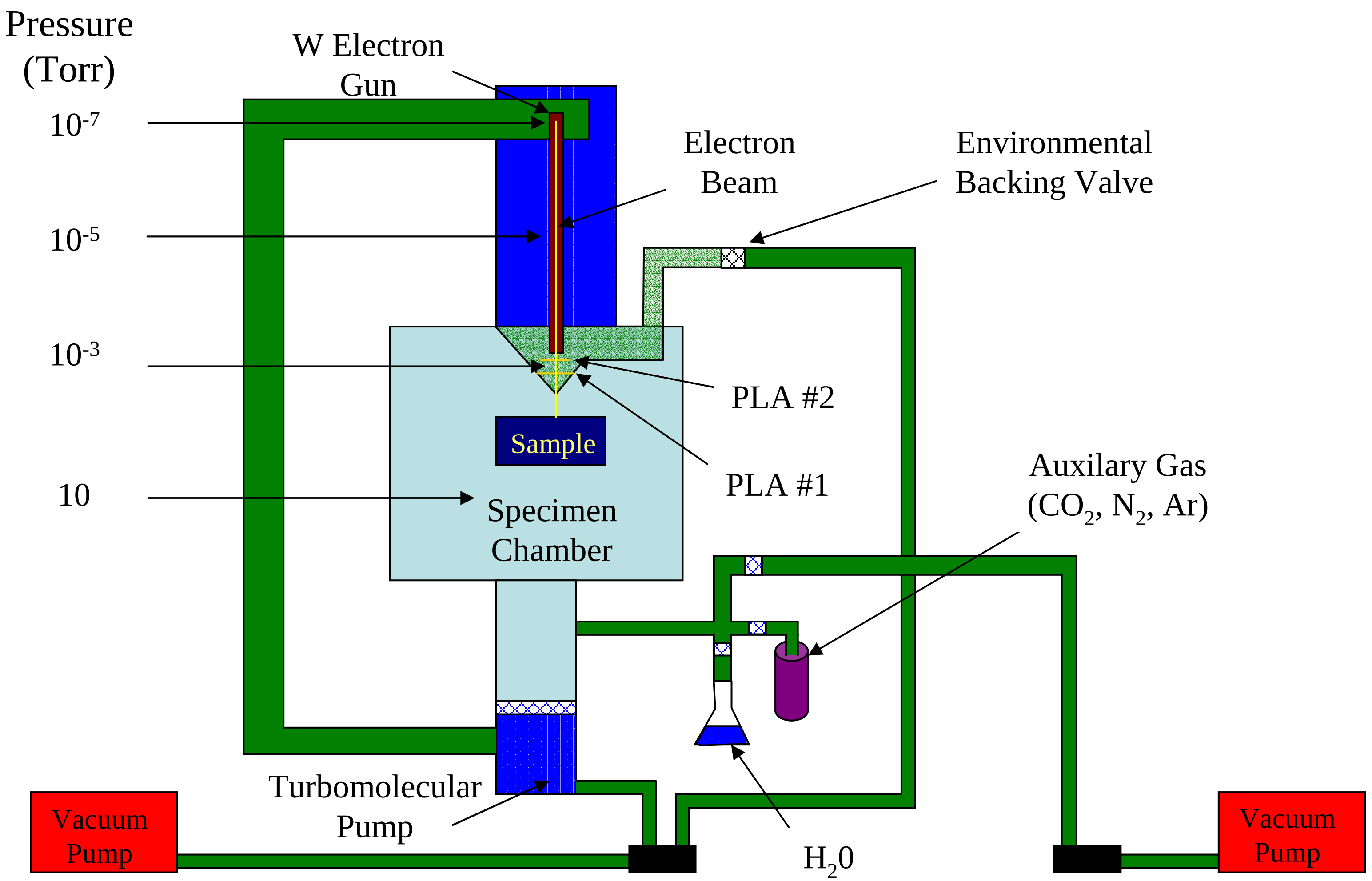
Why SEM ?

- Good resolution
- Large magnification range 20x – 200000x or more
- Depth of field
- 3-D Information (perspective)
- Easy to use
- Easy sample preparation
- Lots of uses and applications
- Great deal of information obtainable on one instrument
- Reasonable cost (purchase and operating)
- Ubiquitous

SEM Flavors


Scanning Electron Microscopy
Conventional SEM
Low Vacuum SEM
Environmental Scanning Electron Microscopy (ESEM)

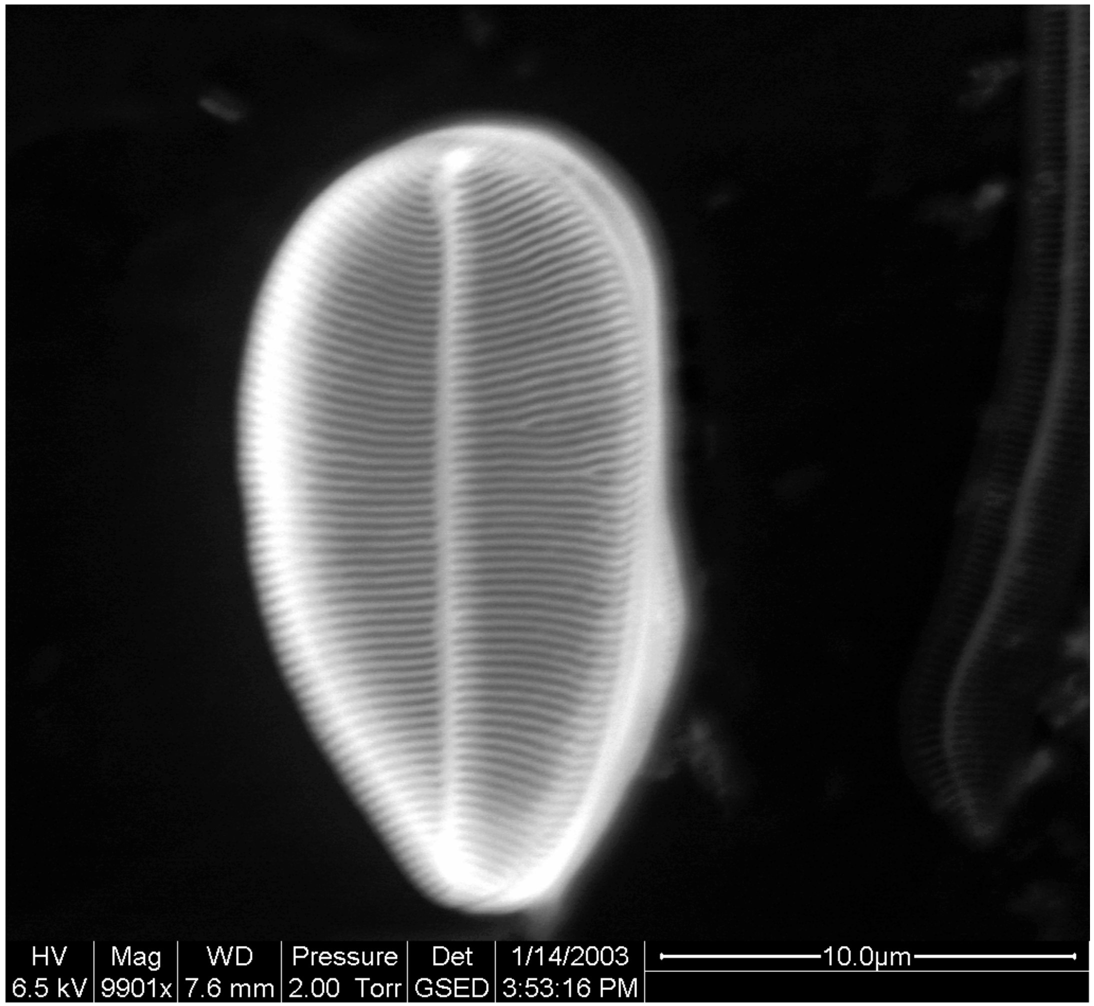
Environmental Scanning Electron Microscope (ESEM)



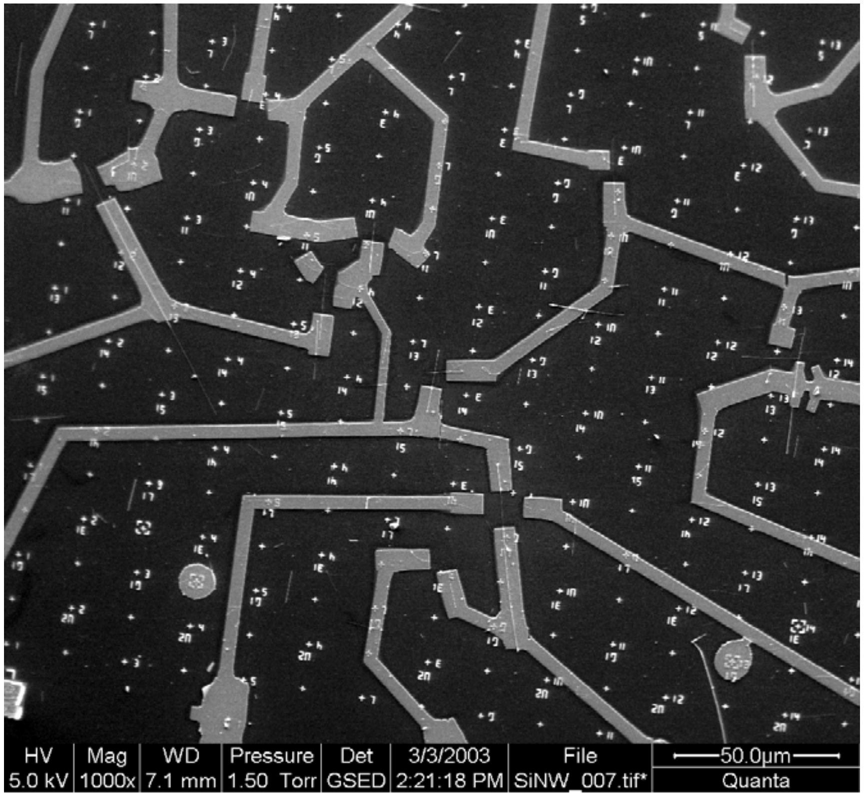
ESEM : Quanta 200

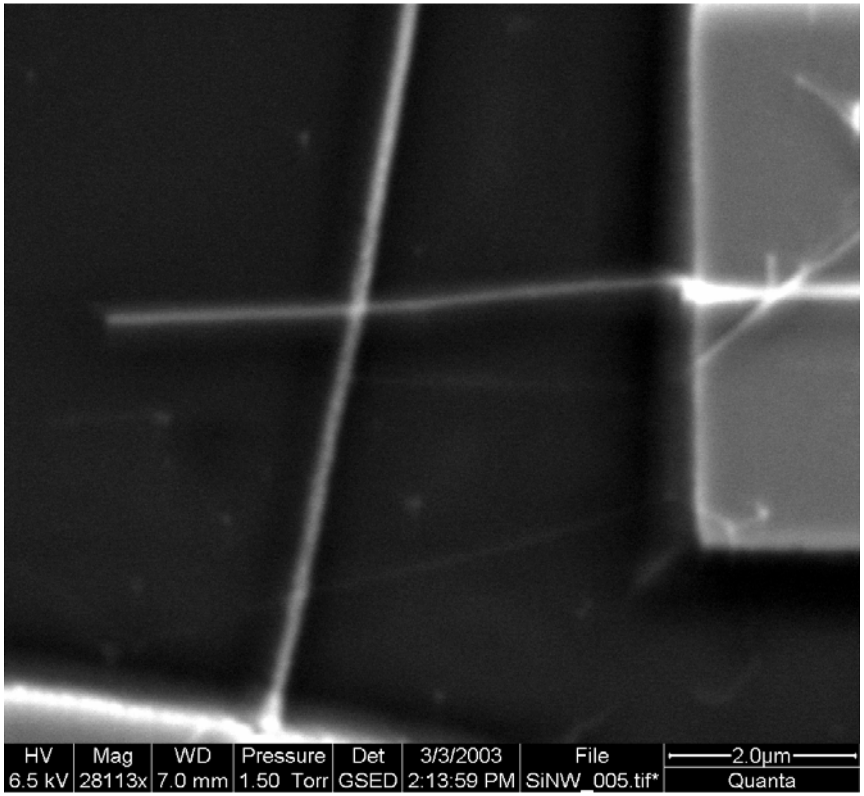
•CIMS Contact : Dr. Richard Schalek

ESEM Example: Salt Water Diatom



ESEM Example: Silicon Nanowires

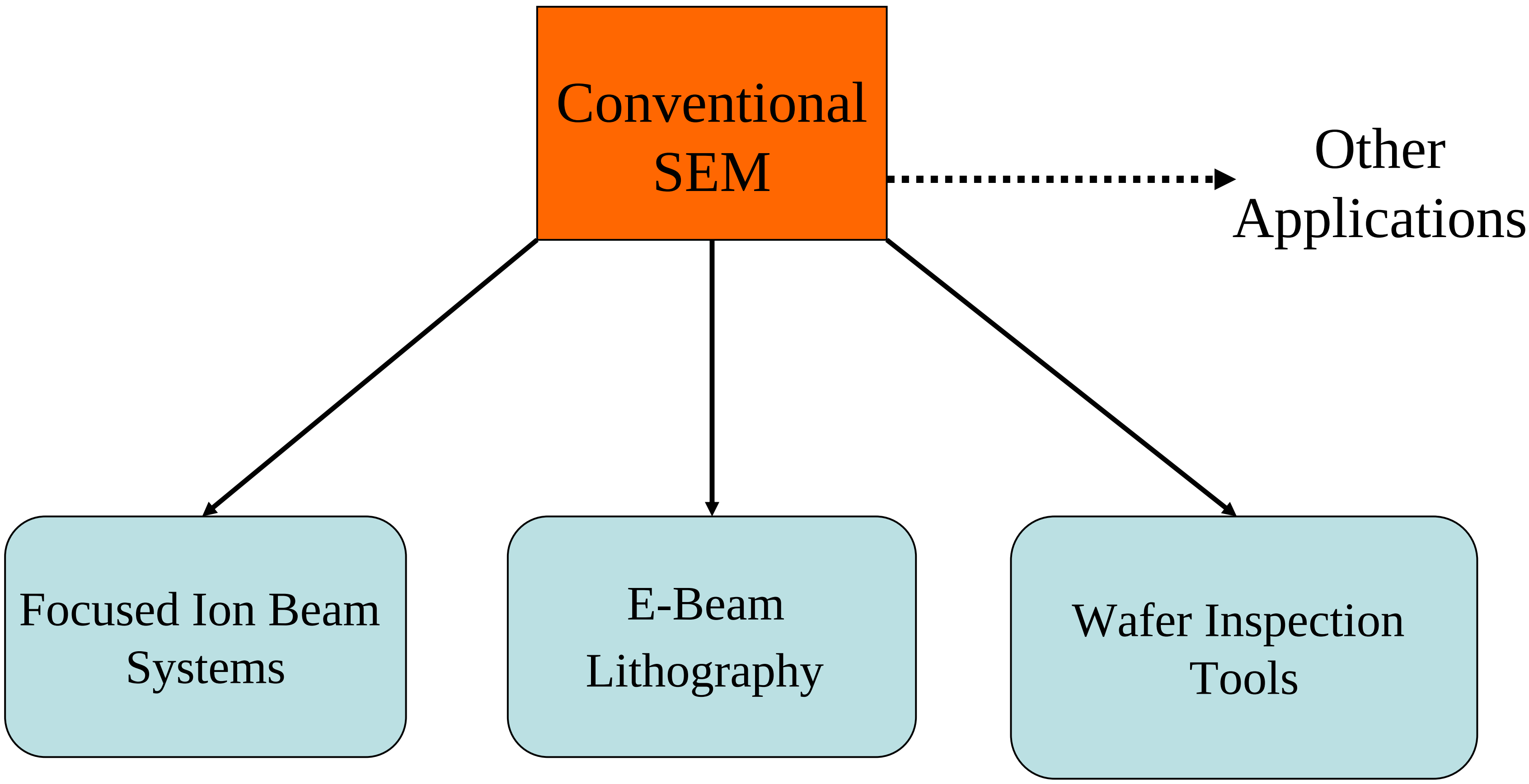




ESEM: In situ Experiments

Naptha

SEM FAB Applications

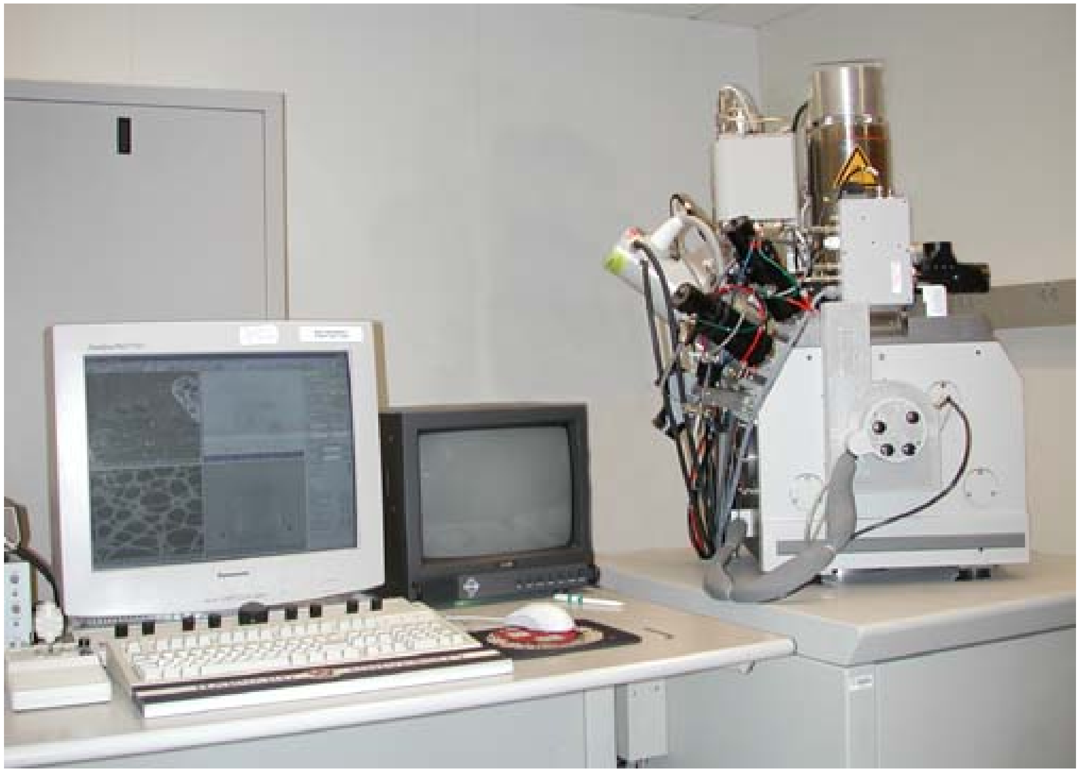


SEM FAB Applications

Hitachi RS-3000 Defect Review SEM

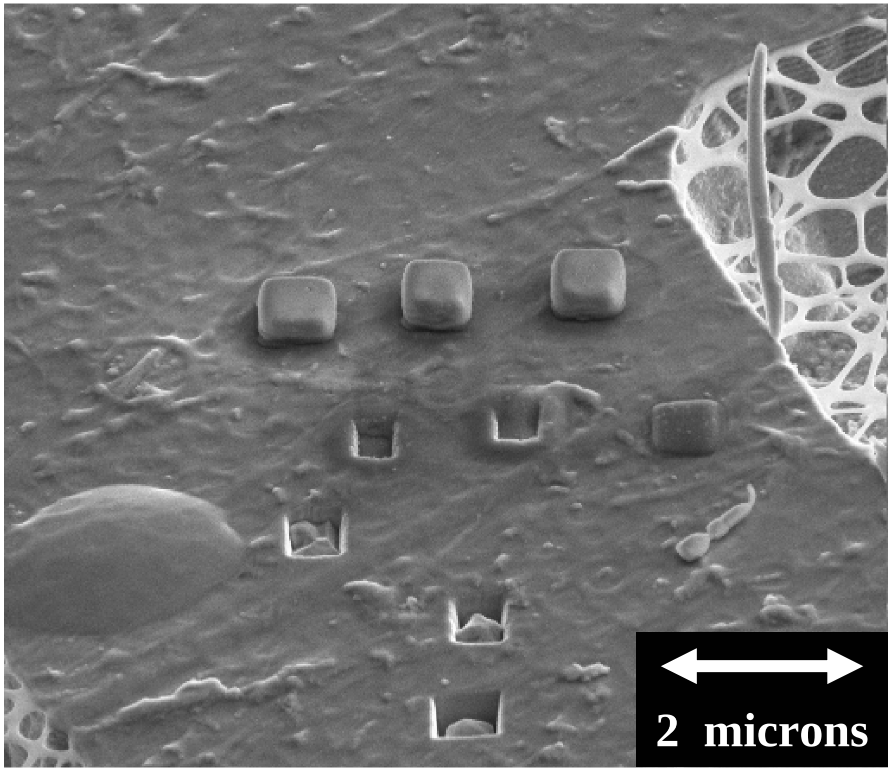
Raith 150 Ultra High Precision E-Beam Lithography and Metrology System

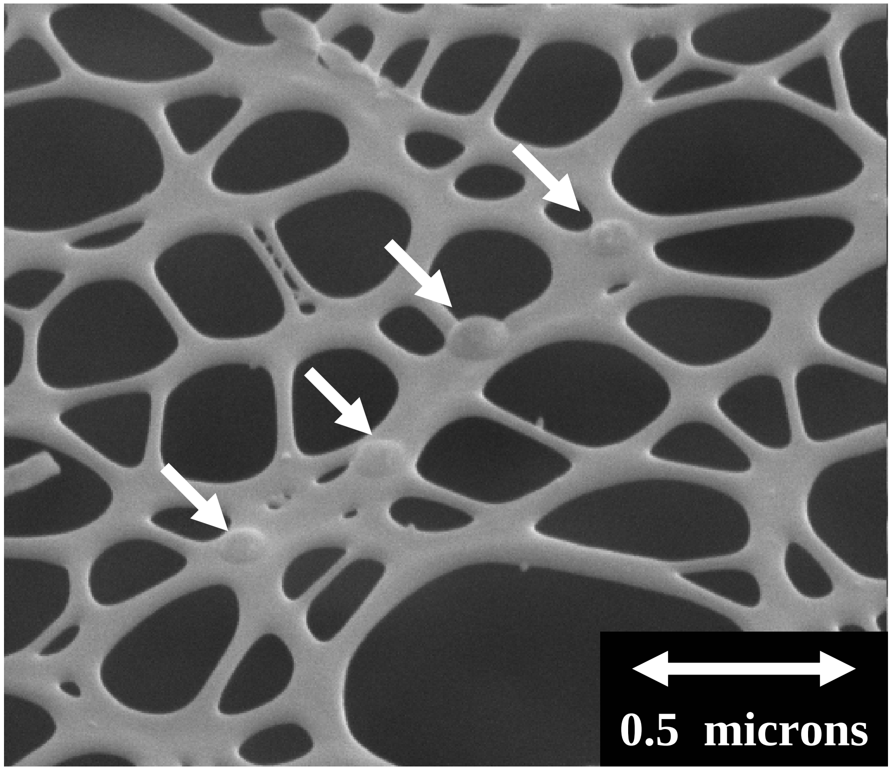
Focused Ion Beam System



FEI D235 DualBeam FIB/SEM

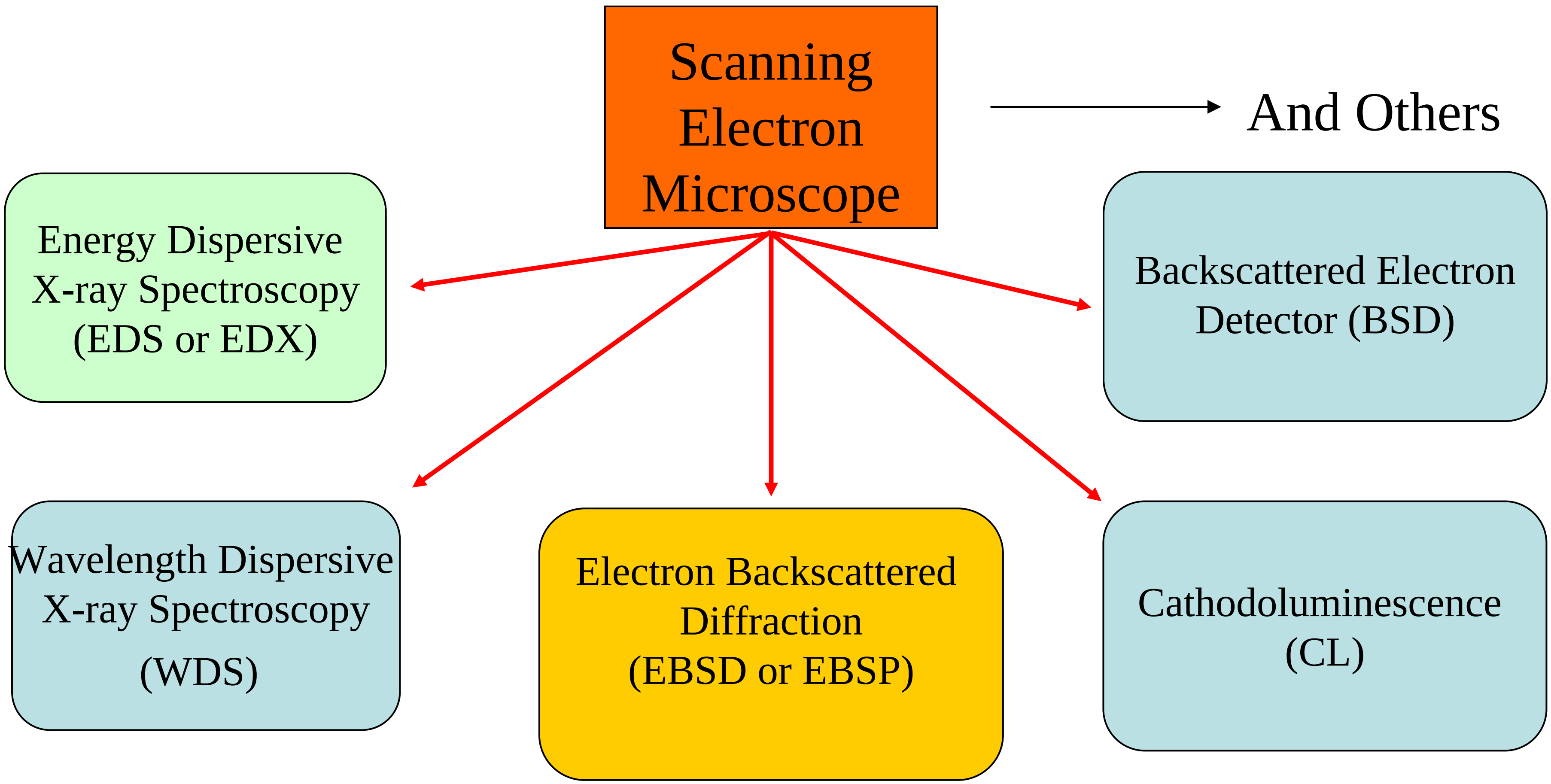
FEI D235 DualBeam FIB/SEM





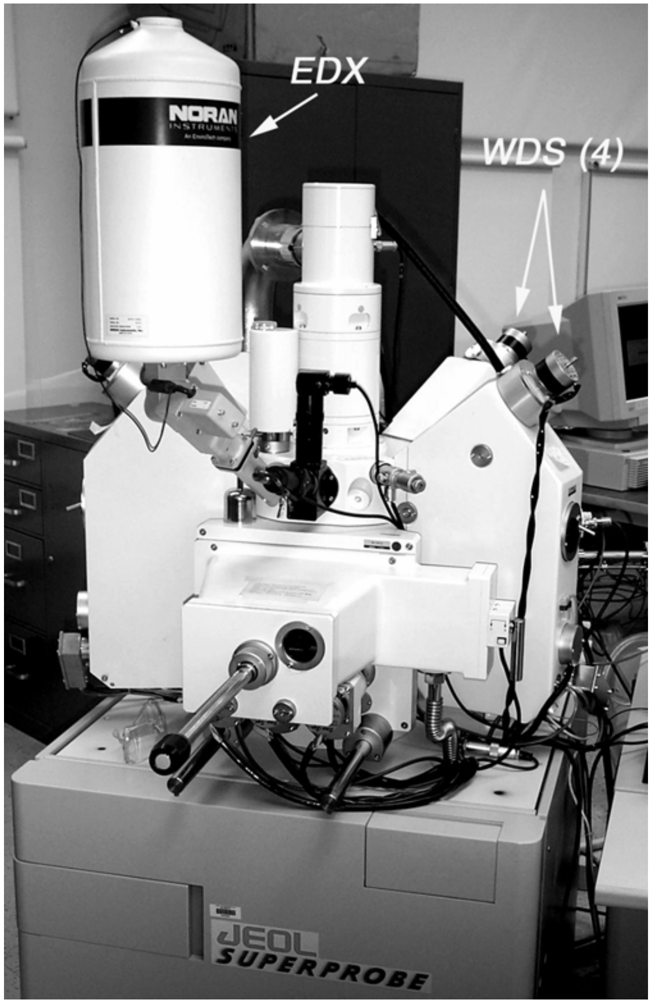
- Focused Ion Beam (with liquid Ga source) can scan and etch
- Or with injected gas cause deposition (metals, dielectrics)
- CIMS Contact : Dr. Warren MoberlyChan

SEM Based Materials Analysis

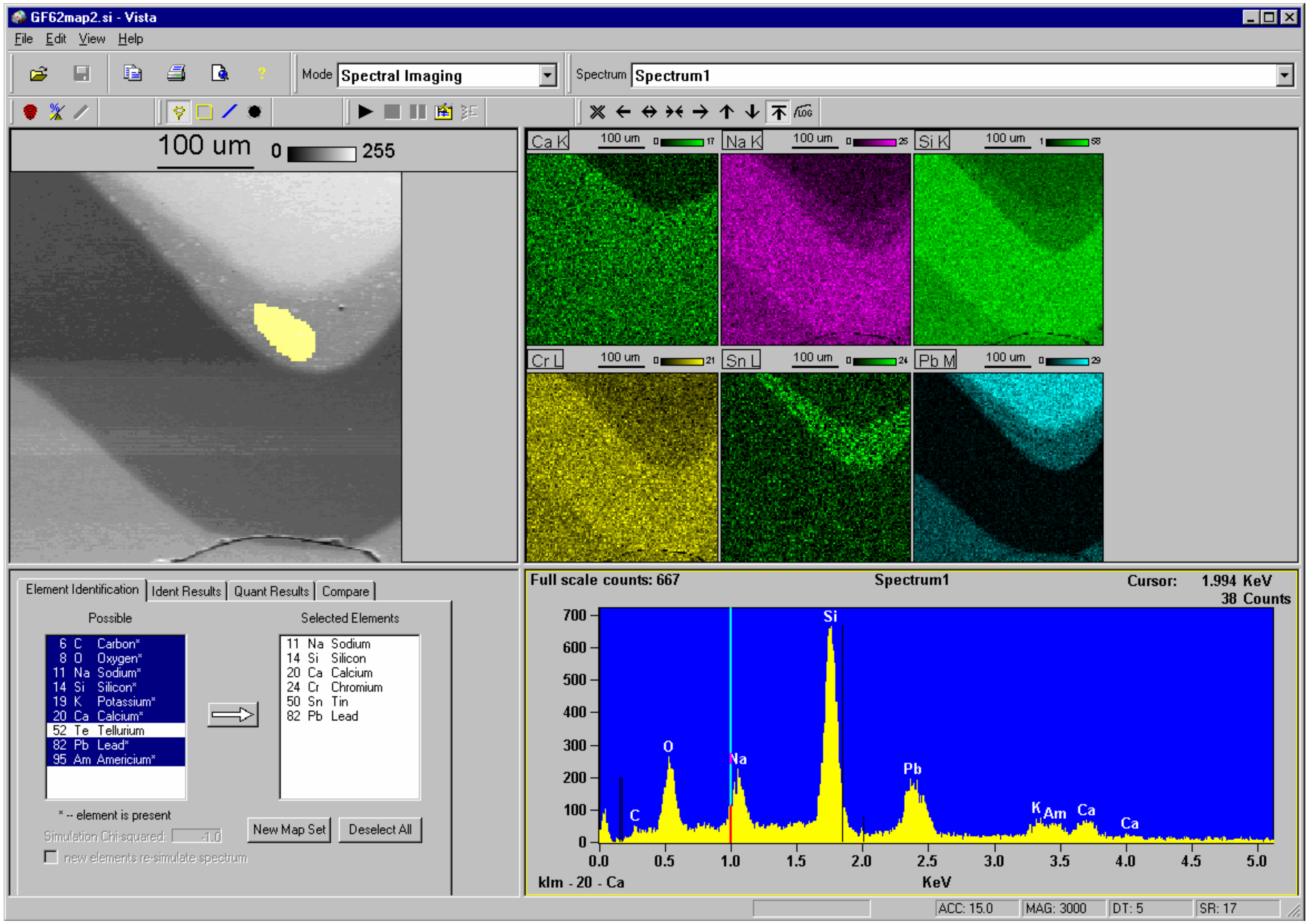


EDX and WDS analysis

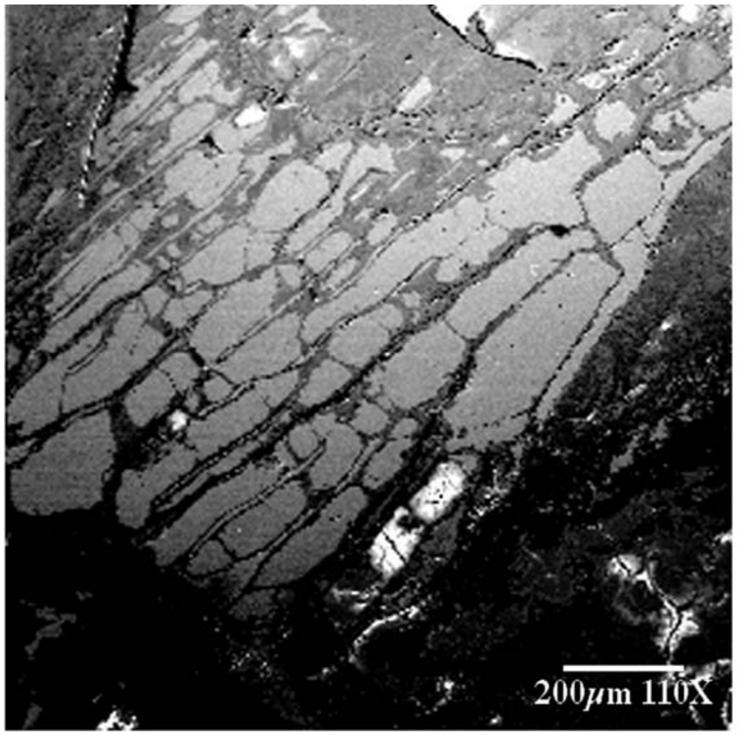
- Electron Microprobe
  - Combines EDX and WDS analysis
  - SEM Column
  - Typically Tungsten Filament
  - Optimized for microanalysis



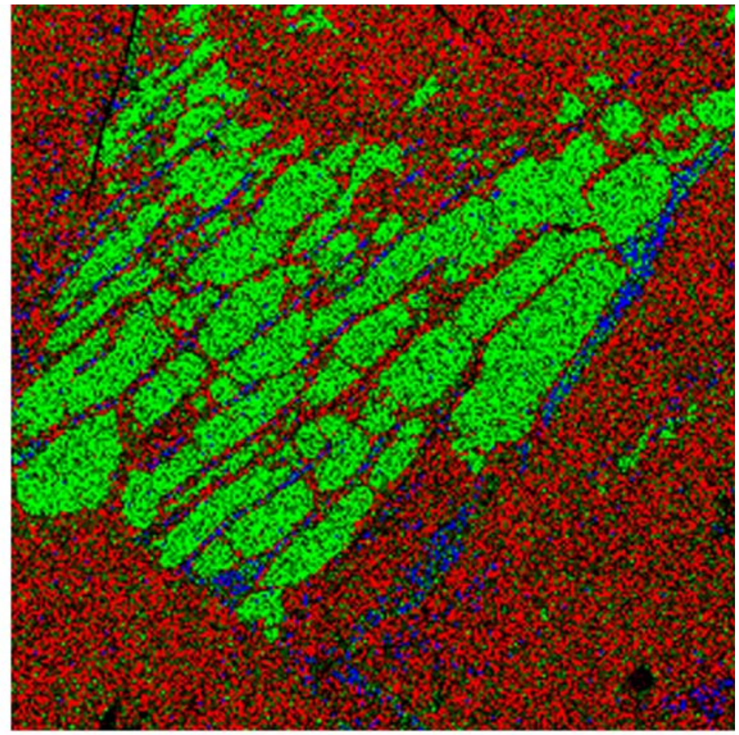
EDX Spectrum and Mapping



EDX Analysis Mapping



SEM Mapping Image



EDX Map

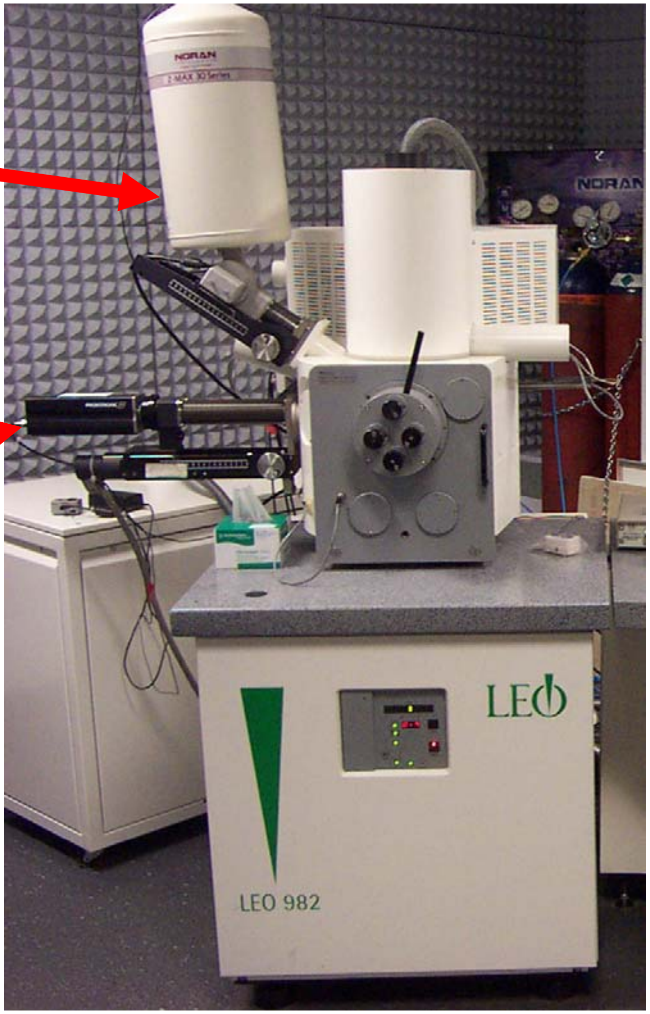
Ni/Fe Meteorite

FeOx *Fe* *Ni*

Electron backscattered Diffraction (EBSP or EBSD)

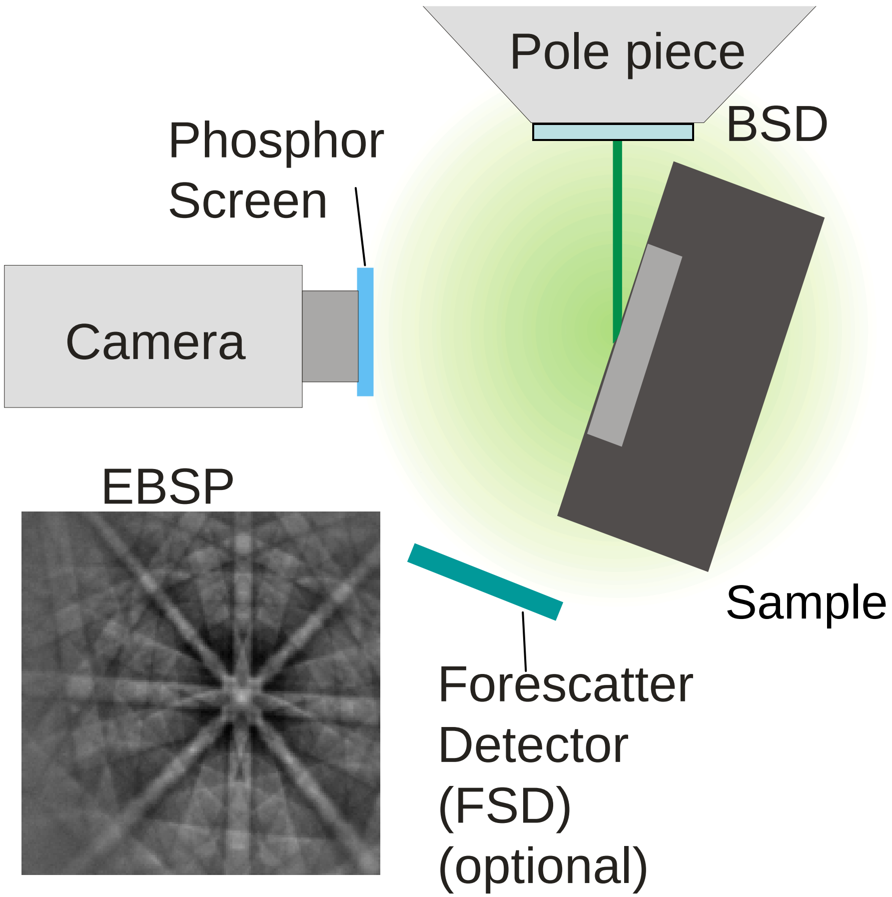
EDX System

EBSP System



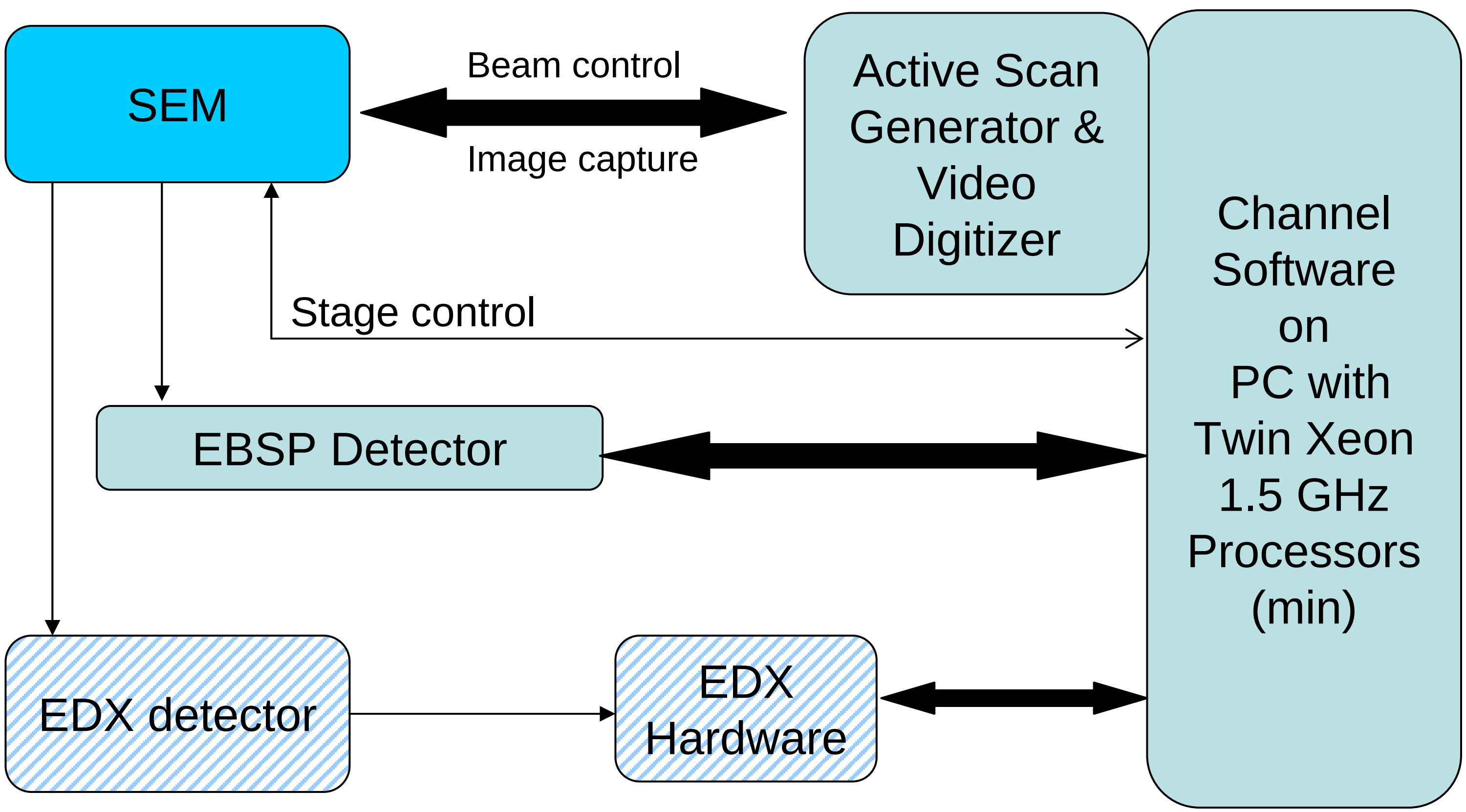
EBSP – Fundamentals

- **SEM based technique**
  - 70° tilted specimen
  - 1-10 nA , ~20 kV
- **Detector**
  - Phosphor + CCD camera
- **EBSP**
  - Kikuchi bands (planes)
  - Zones (directions)
- **Orientation**
  - Sub-micron resolution
  - ~0.5deg angular resolution
- **Surface Effect**
  - Sampling upper 30-50nm
  - Surface prep important!



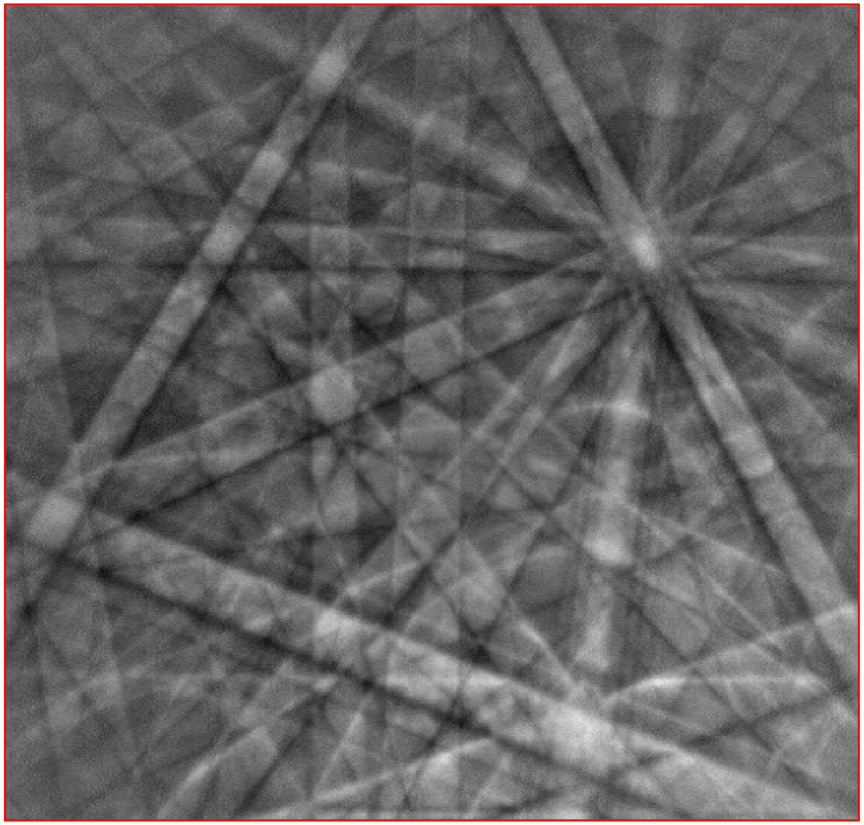
Courtesy Tim Maitland, HKL Technology

EBSP: System Diagram



Courtesy Tim Maitland, HKL Technology

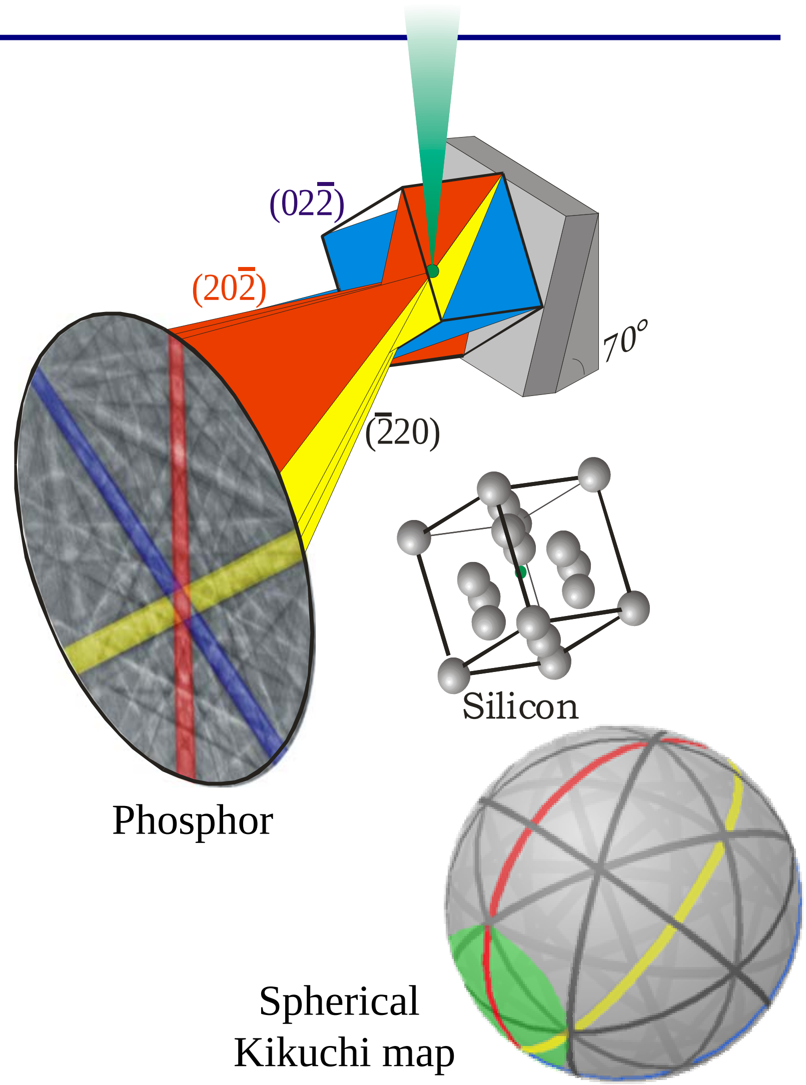
What does an EBSP look like?



Silicon at 20kV Courtesy Tim Maitland, HKL Technology

How it works - EBSP formation

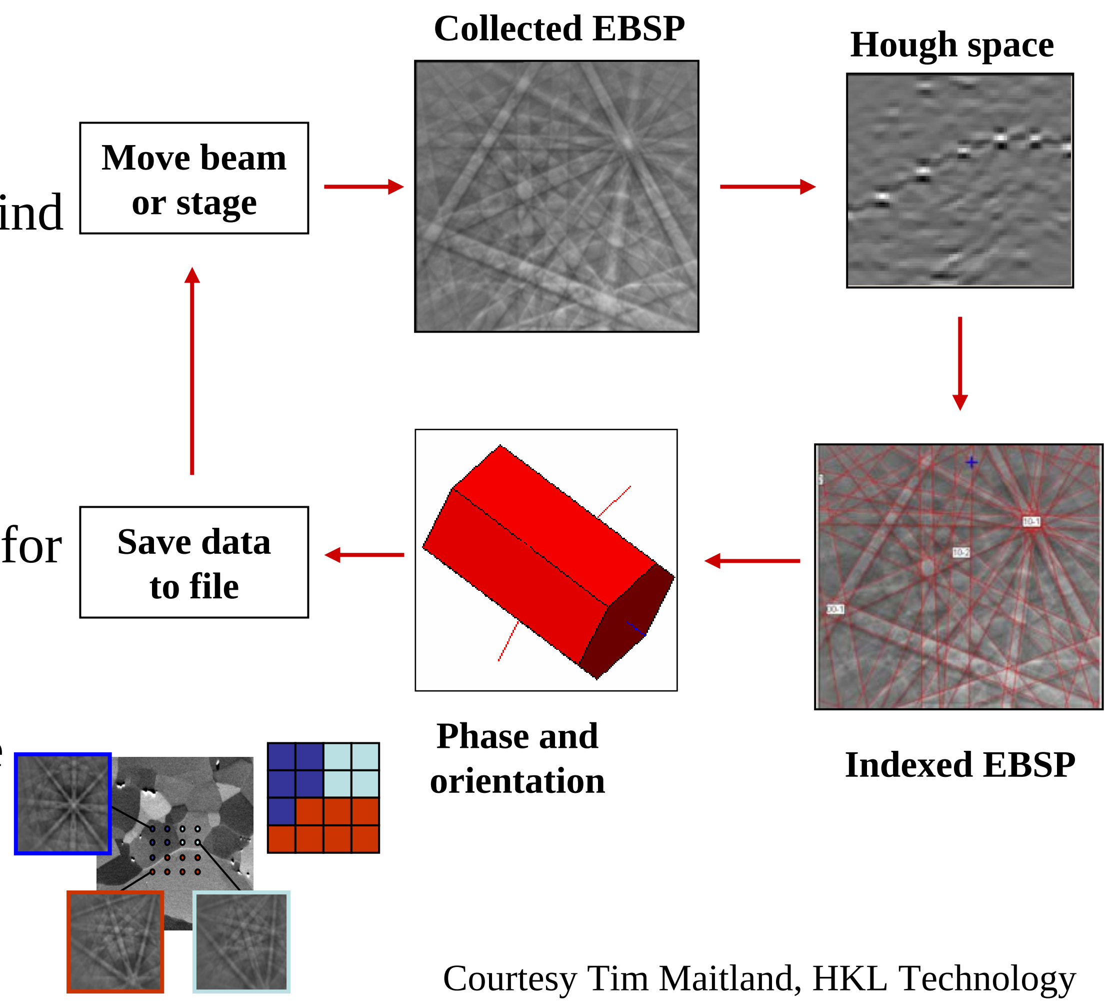
- The Electron beam strikes the specimen
- Scattering produces electrons travelling in all directions in a small volume (the excitation volume)
- Electrons that travel in a direction that satisfies the Bragg condition ($n\lambda=2d_{hkl}.\sin\theta$) for a plane (hkl) are channeled ⇒ Kikuchi bands
- The electrons hit the imaging phosphor and produce light
- The light is detected by a CCD camera and converted to an image
- Which is indexed...



Courtesy Tim Maitland, HKL Technology

EBSP: Indexing Cycle

- Position electron beam
- Capture EBSP
- Perform Hough transform, find peaks (= band position & orientation)
- Compare to possible peak positions and intensity heirarchy versus theoretical for potential match phases
- If match is made within acceptable error limits, store data & repeat for next point

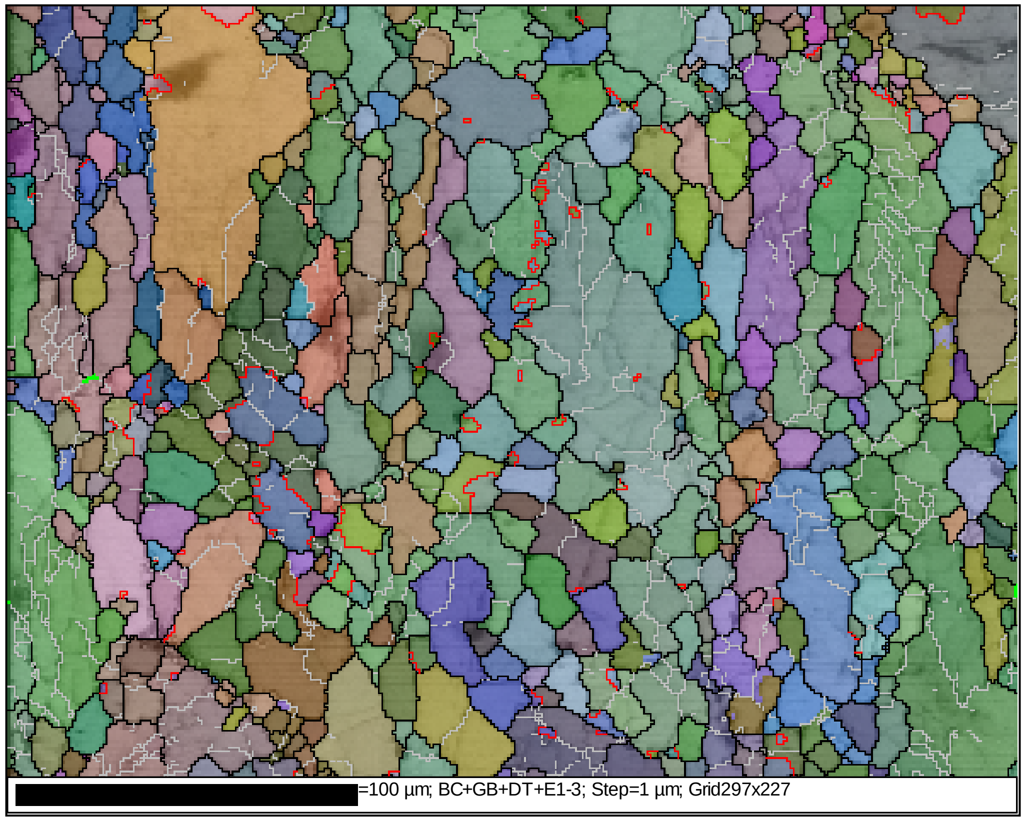


Courtesy Tim Maitland, HKL Technology

EBSP: Visualization of Data

General Micro-structure

Deformed silica (quartz)



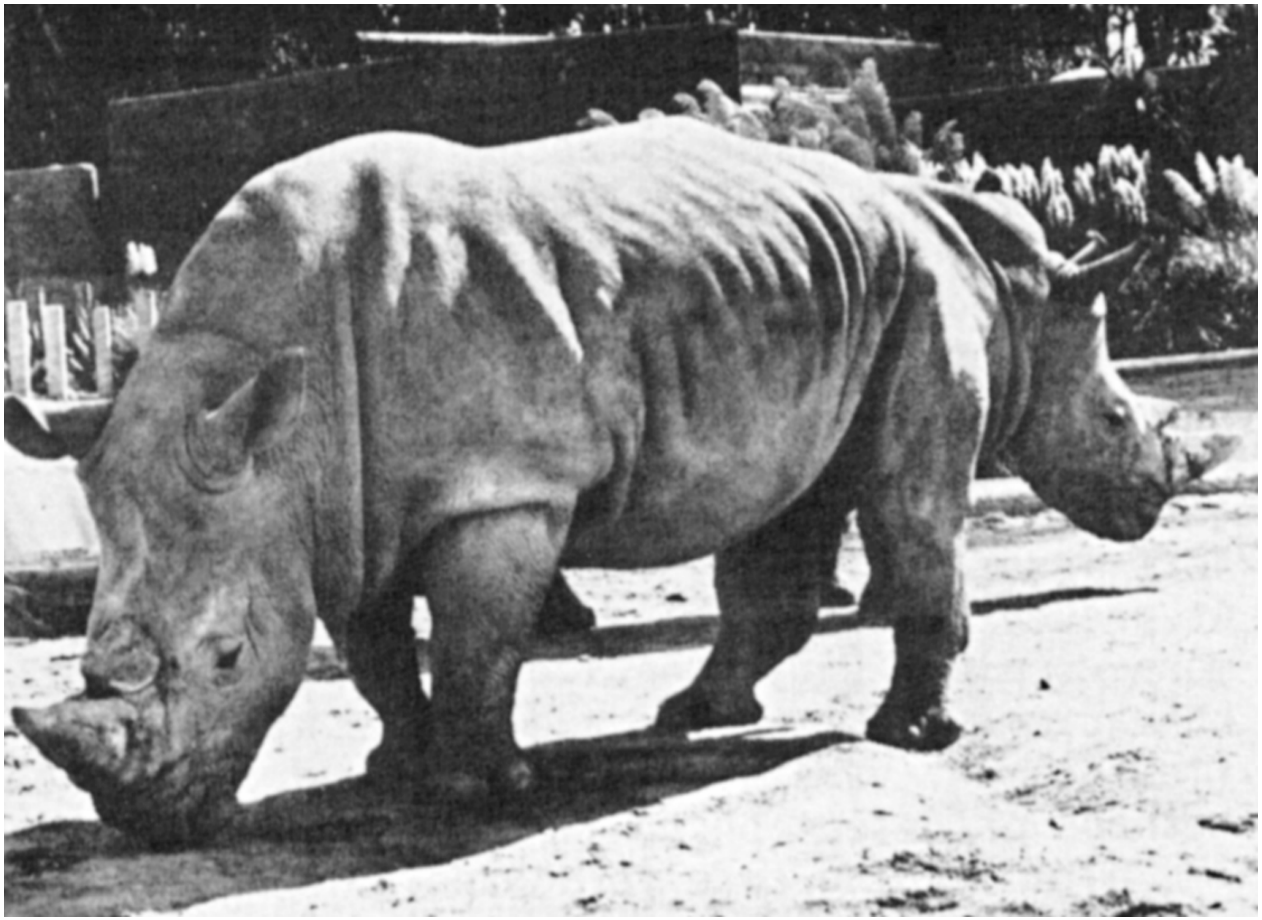
Courtesy of Tim Maitland, HKL Technology

Pixel map of pattern quality + crystal orientation + grain boundary location and character

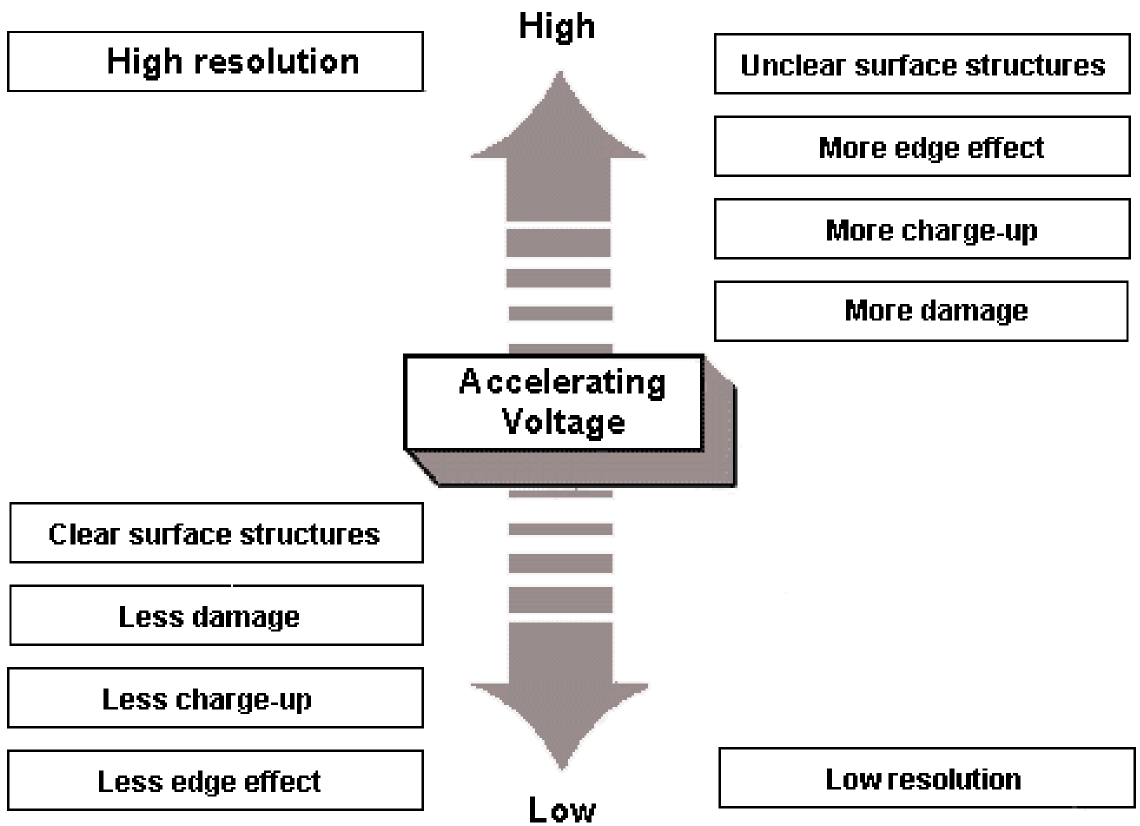
SEM Operating Considerations

- Effects of Accelerating Voltage
  - Sample charging
  - Resolution / Image quality
- Effects of aperture size
  - Depth of Field and Resolution
- Working distance
  - Depth of Field and Resolution
- Tilting the sample
  - Understanding the Geometry
- Effect of Probe size/current
- Astigmatism

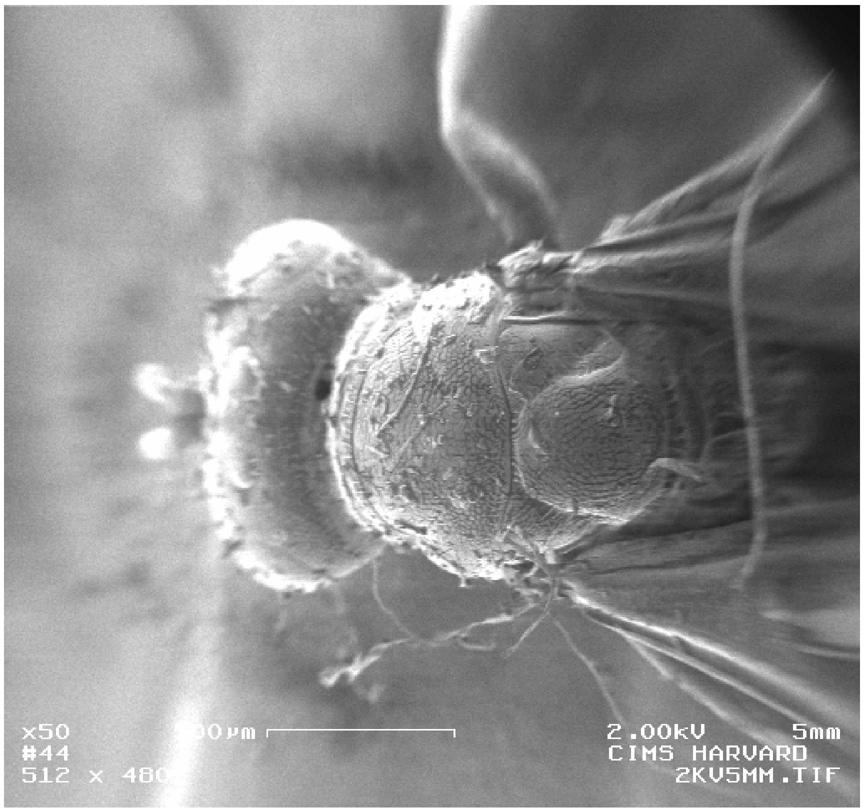
Imaging

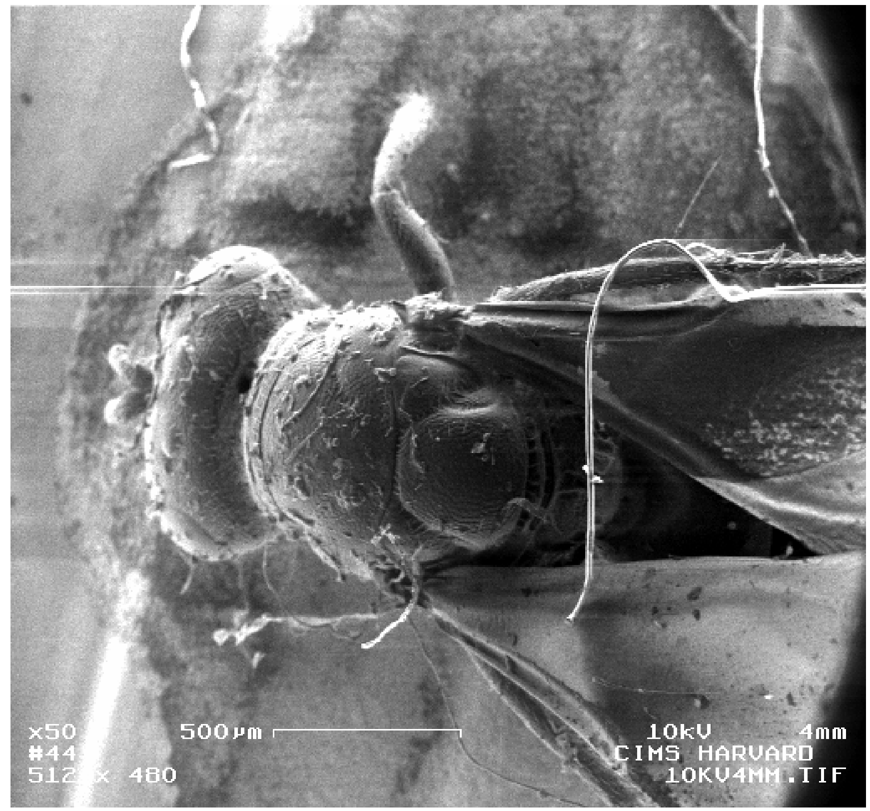


Effects of Accelerating Voltage

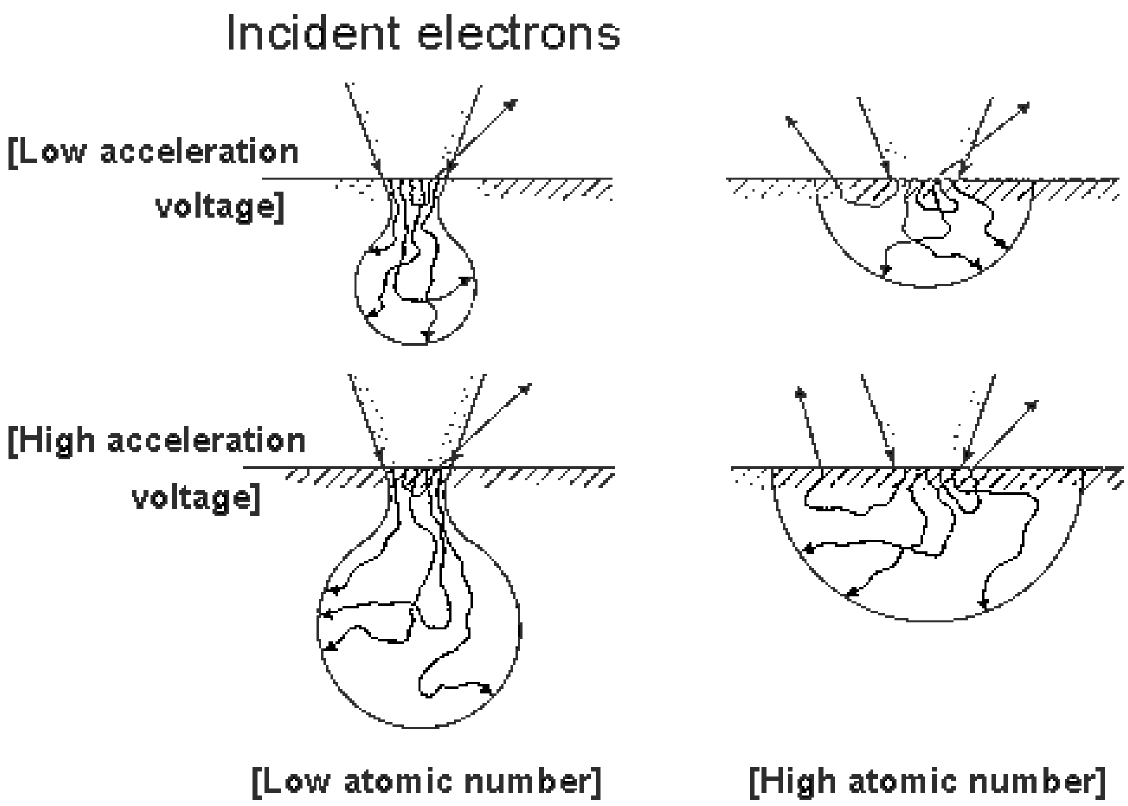


Effects of Accelerating Voltage: Example

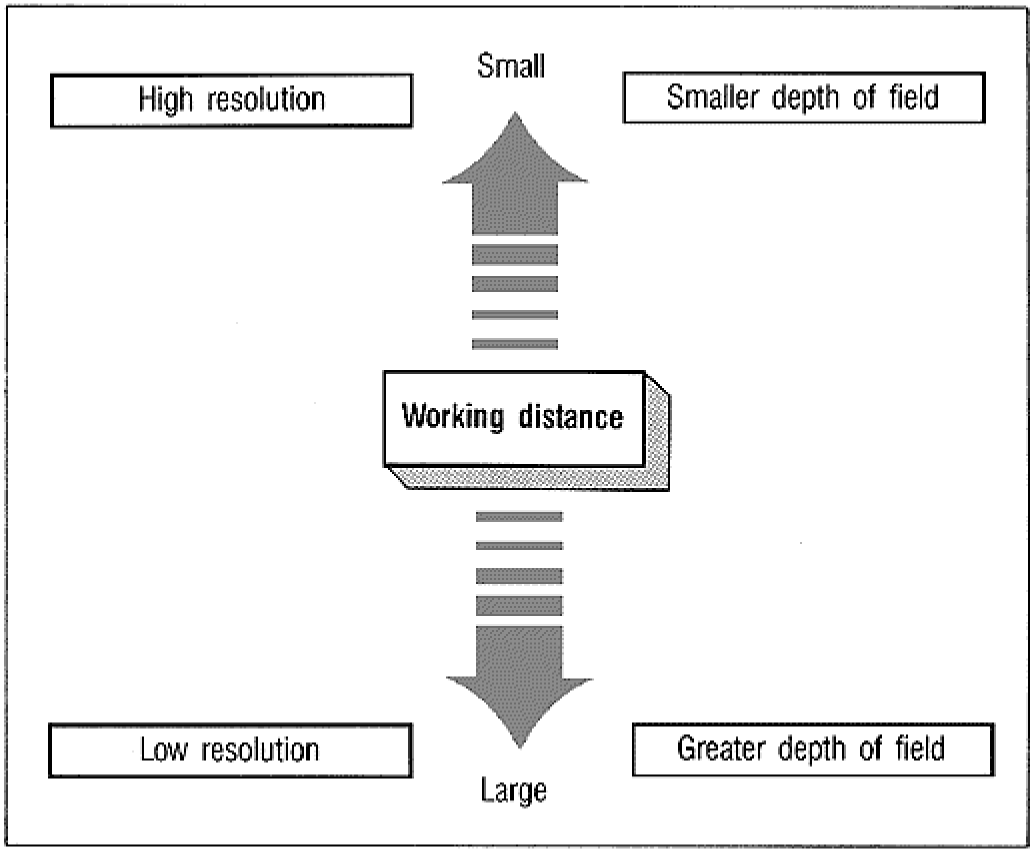




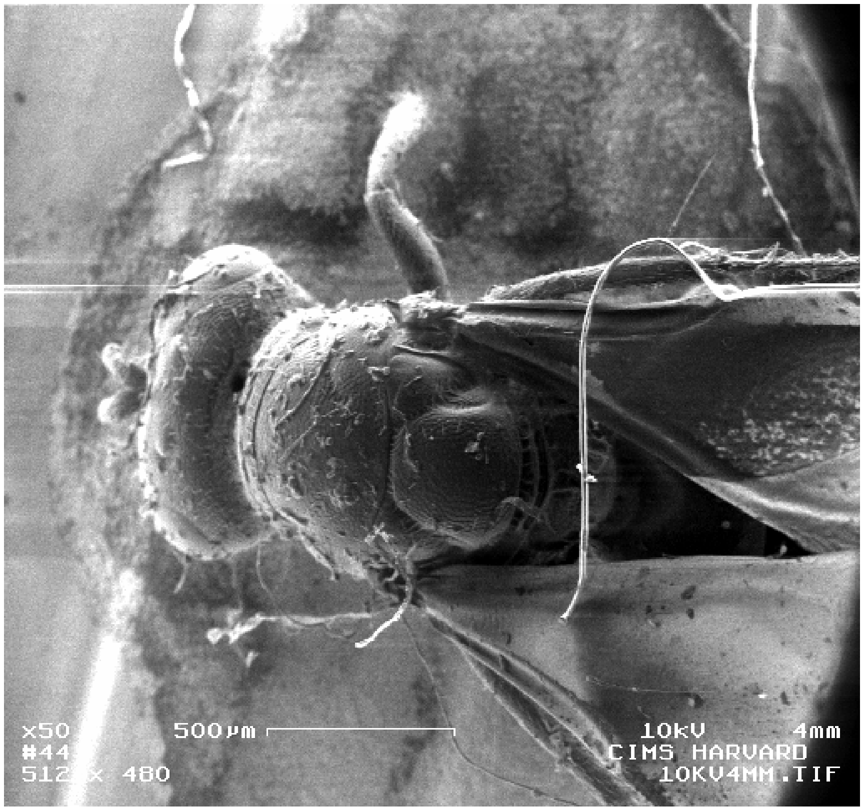
Sample Interaction Volume with Voltage

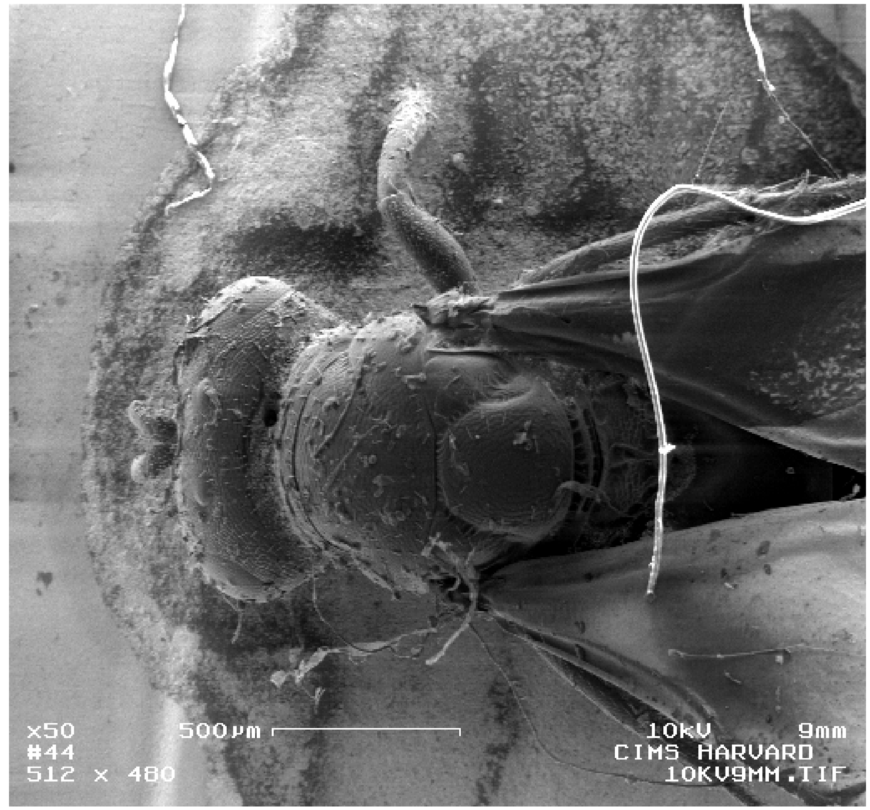


Working Distance

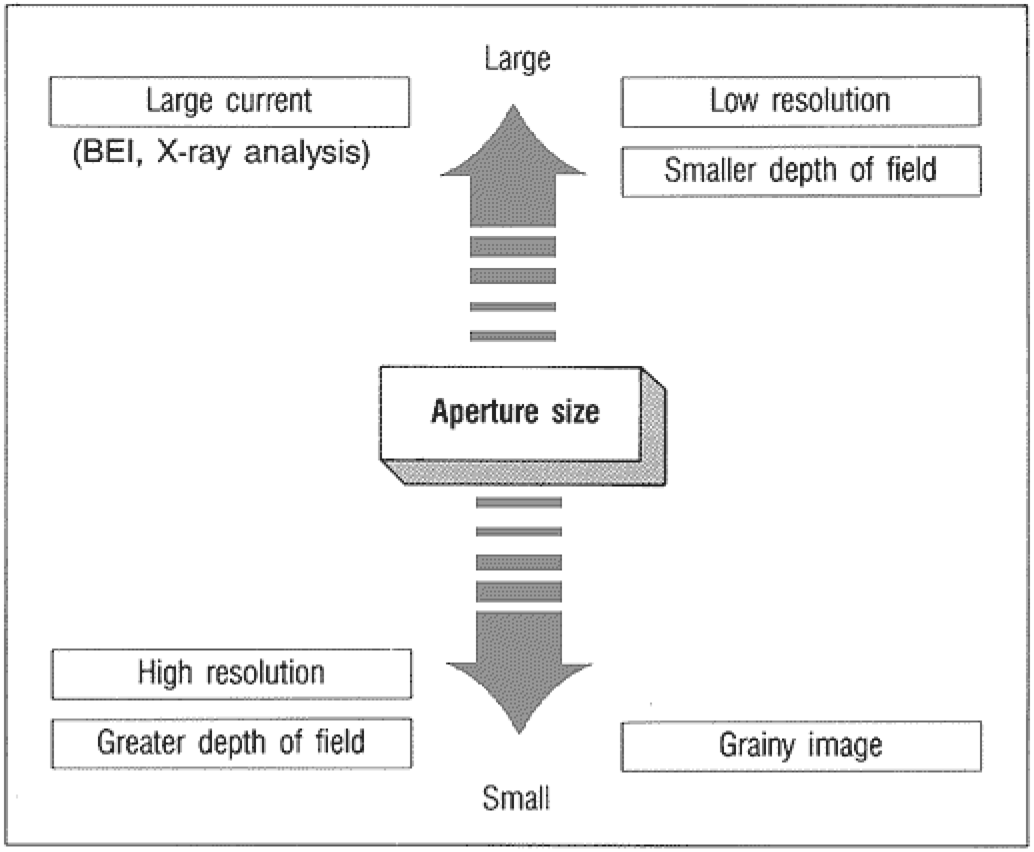


Working Distance: Example

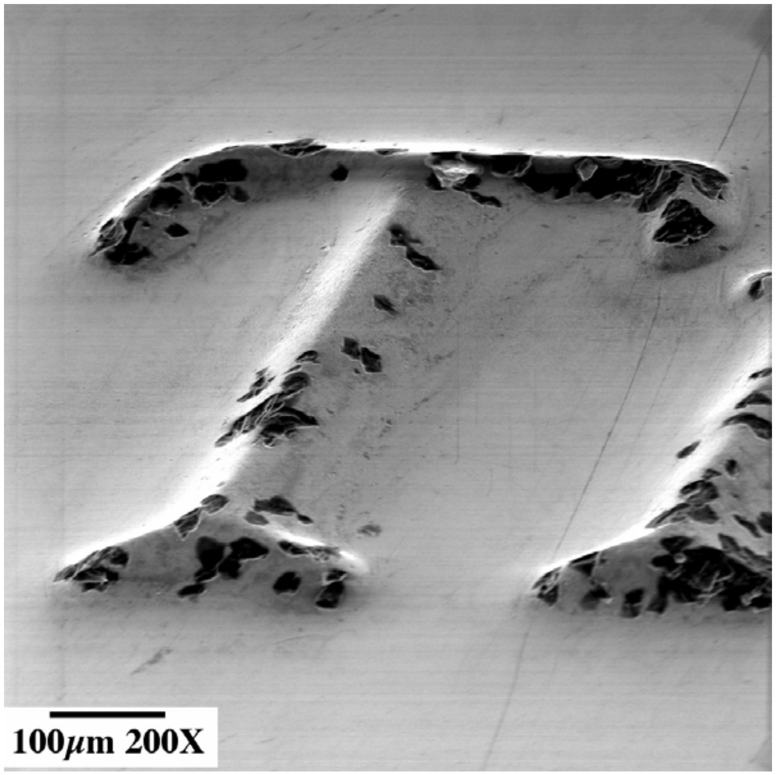




Aperture Size

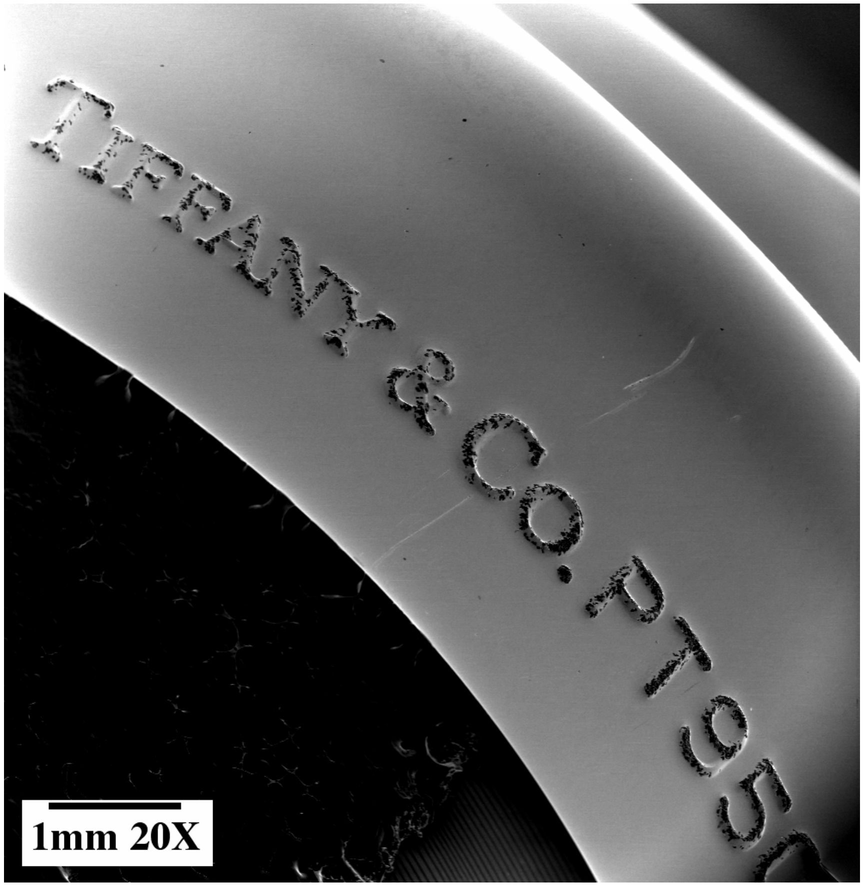


Tilting the Sample



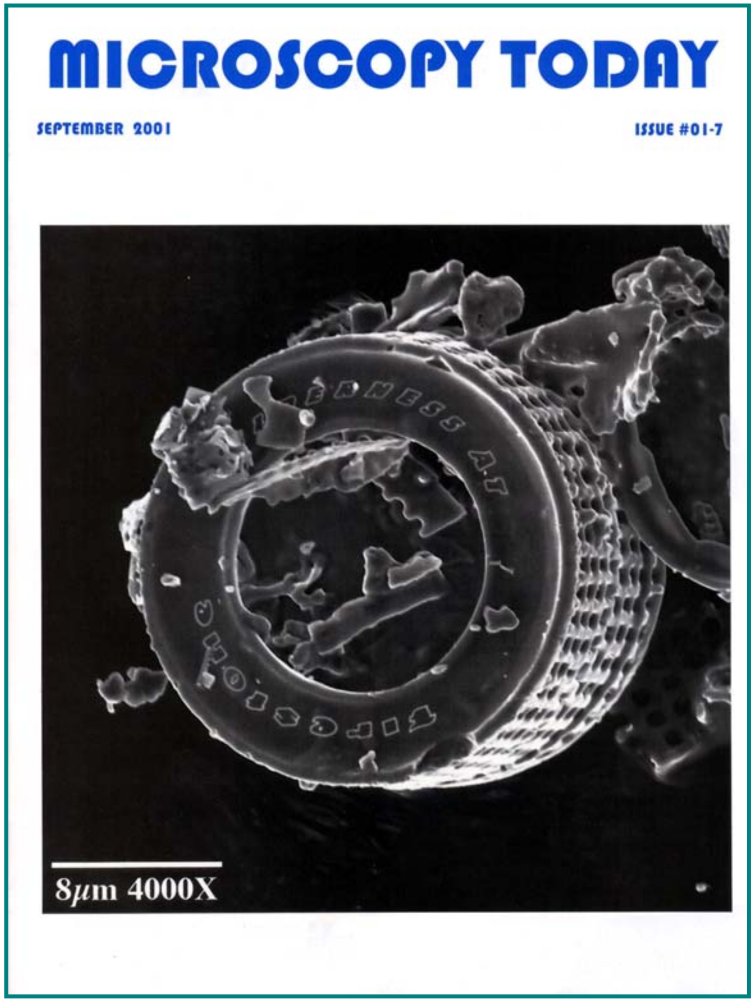
- Important
- Understand the geometry of the sample
- Understand the geometry of the detector

Tilting the Sample / Geometry



- Tilting is important
- Two or Three different tilt angles
- Pull back to capture overall view and to understand geometry

Imaging Considerations



- Seeing is not believing
- Image interpretation is important
- Beware of image artifacts
- Beware of sample/detector geometry effects
- Beware !!!

Demonstration: Using Web SEM

JEOL 5910 SEM running
Web SEM from CMSE MIT

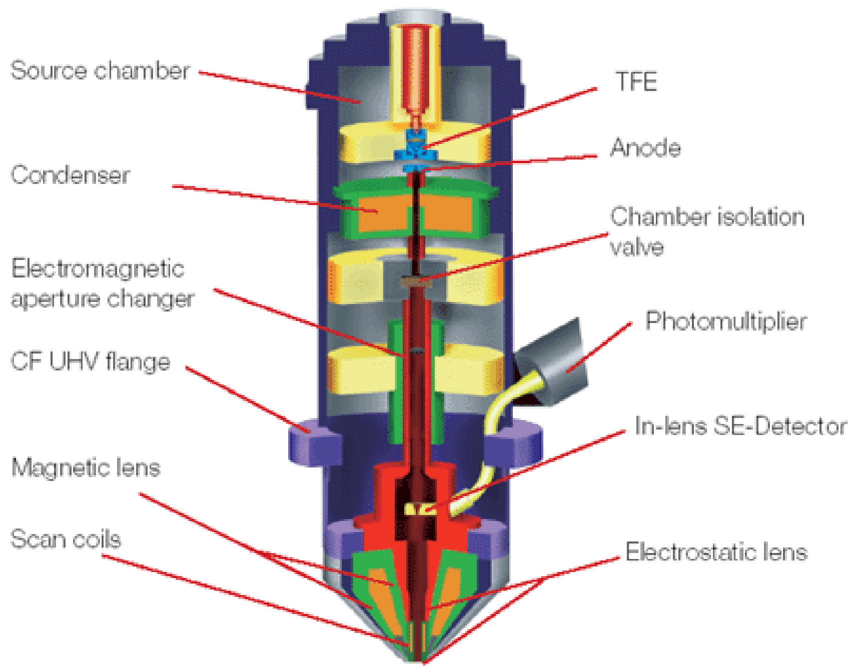
Courtesy of Anthony J. Garratt-Reed

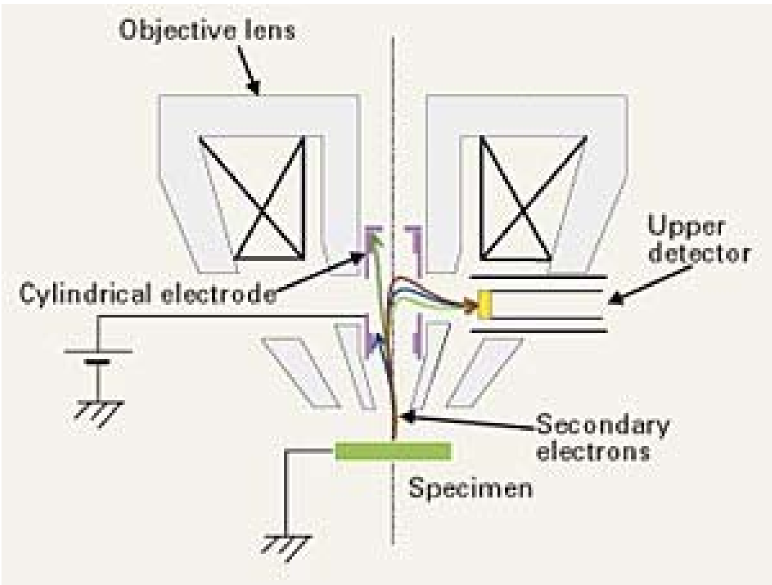
Specimen

SEM Instrument Considerations for Nanostructure Imaging

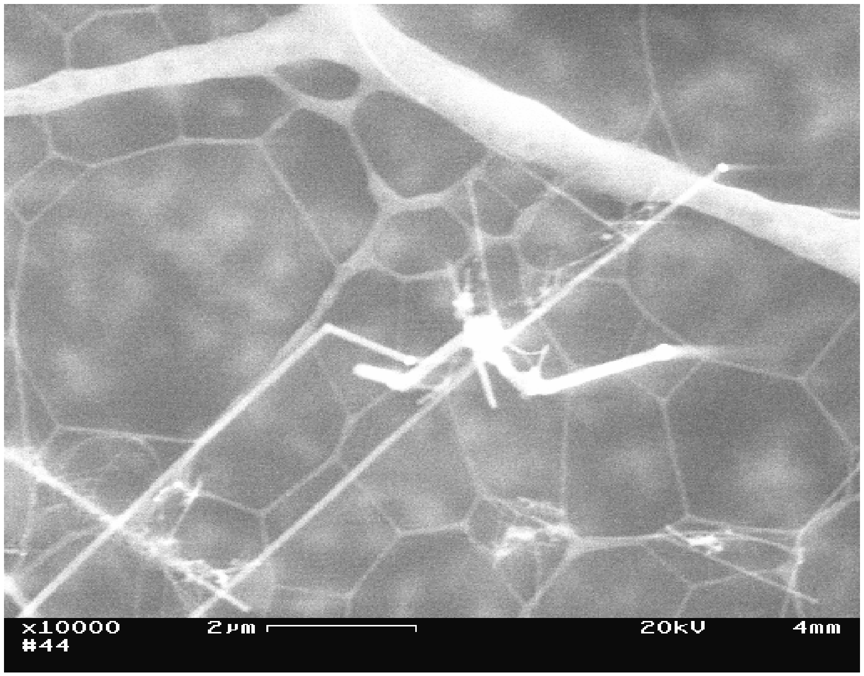
- In the lens secondary electron detector
- Field emission electron gun
  - Cold emission (Better ?)
- High vacuum or UHV system
  - Minimize contamination

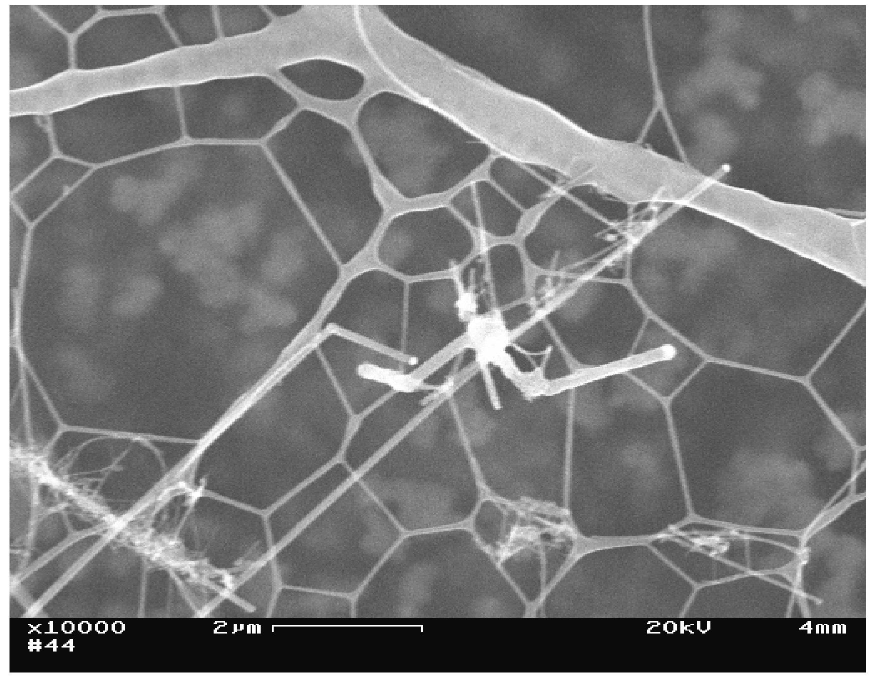
In the Lens SE Detector





In the Lens SE Detector Example

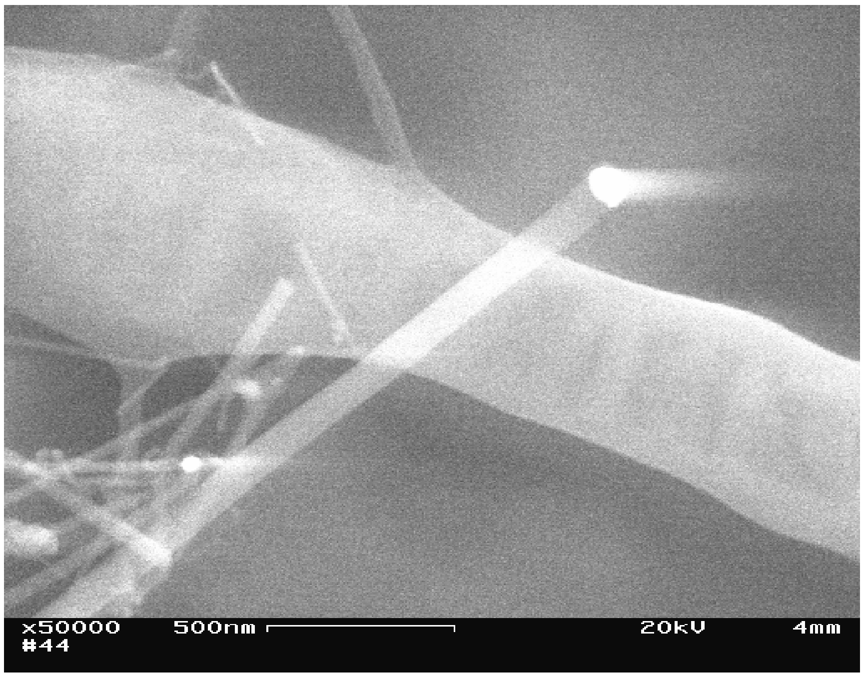


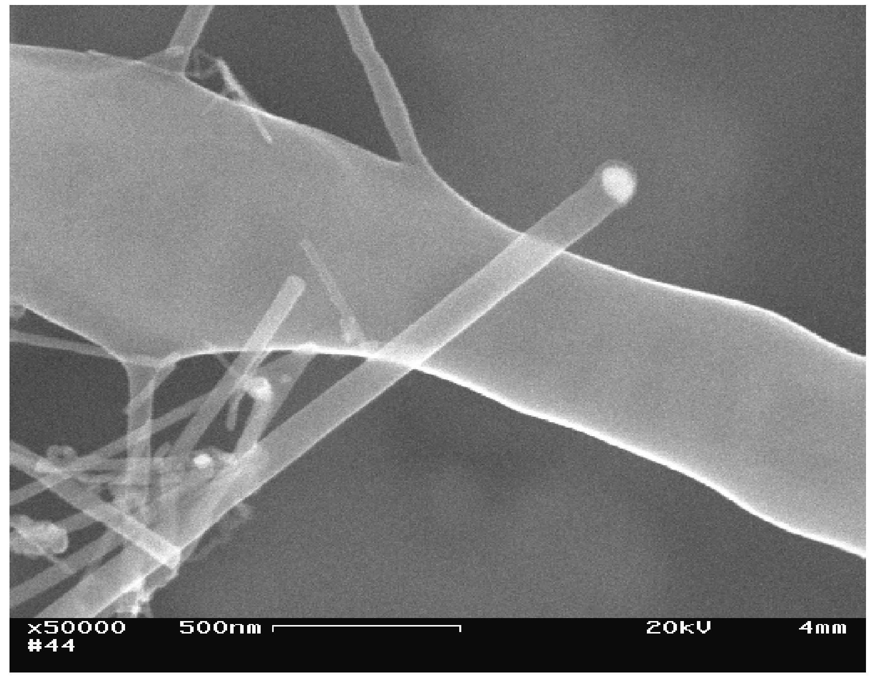


Standard SE Detector

In Lens SE Detector

In the Lens SE Detector Example

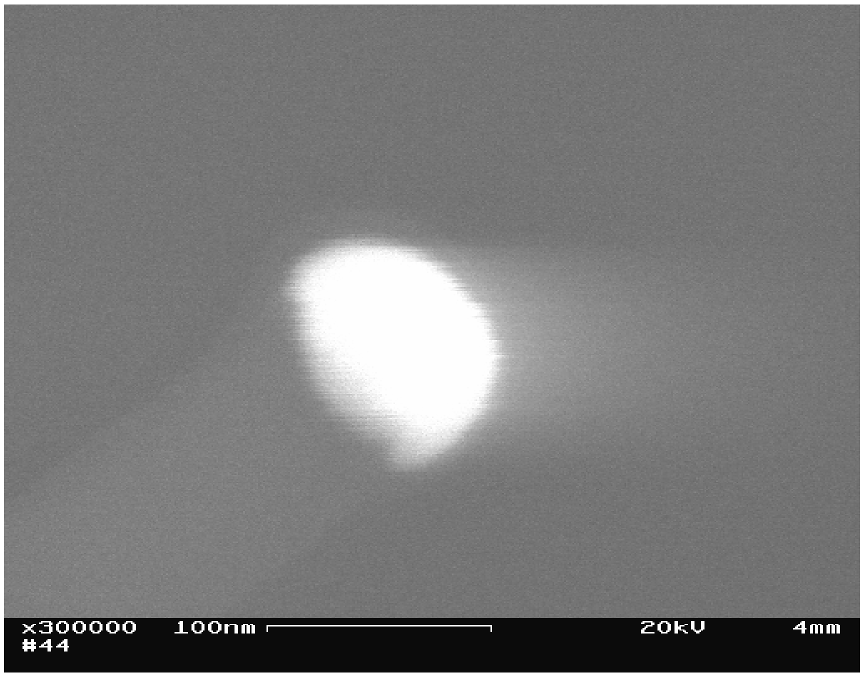


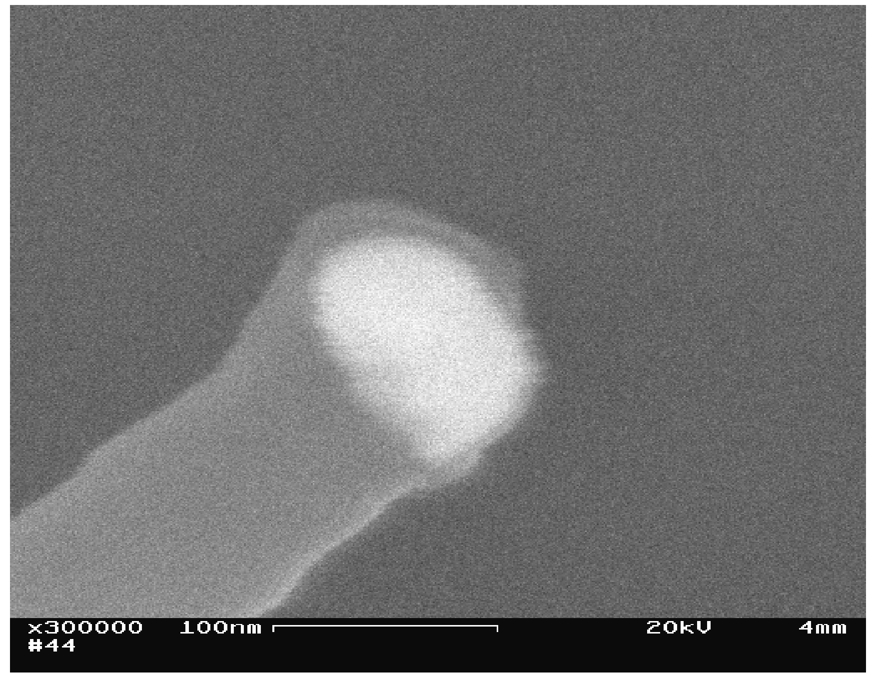


Standard SE Detector

In Lens SE Detector

In-Lens SE Detector Example





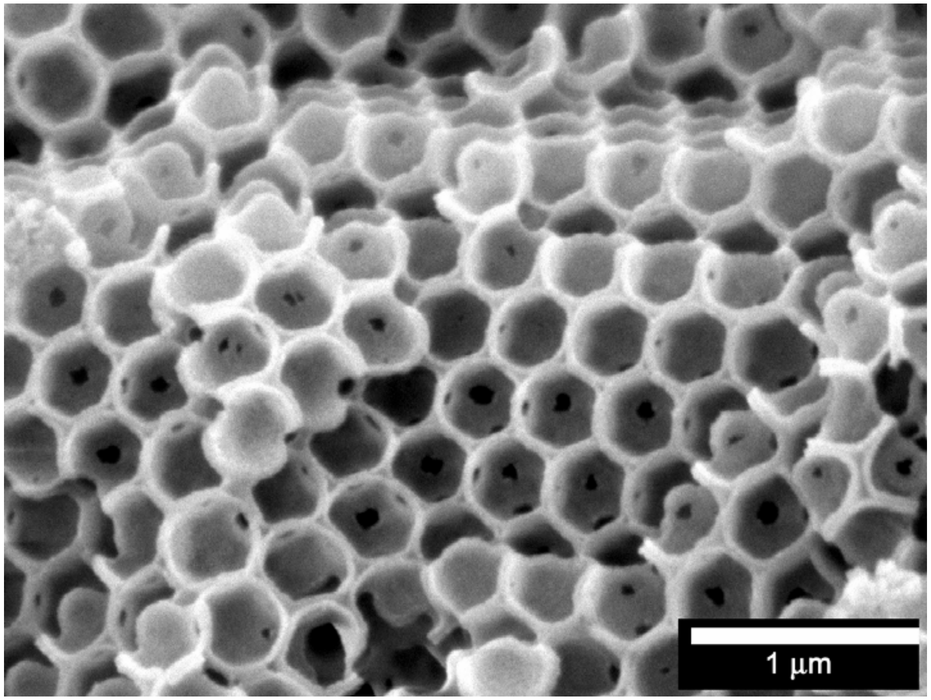
Standard SE Detector

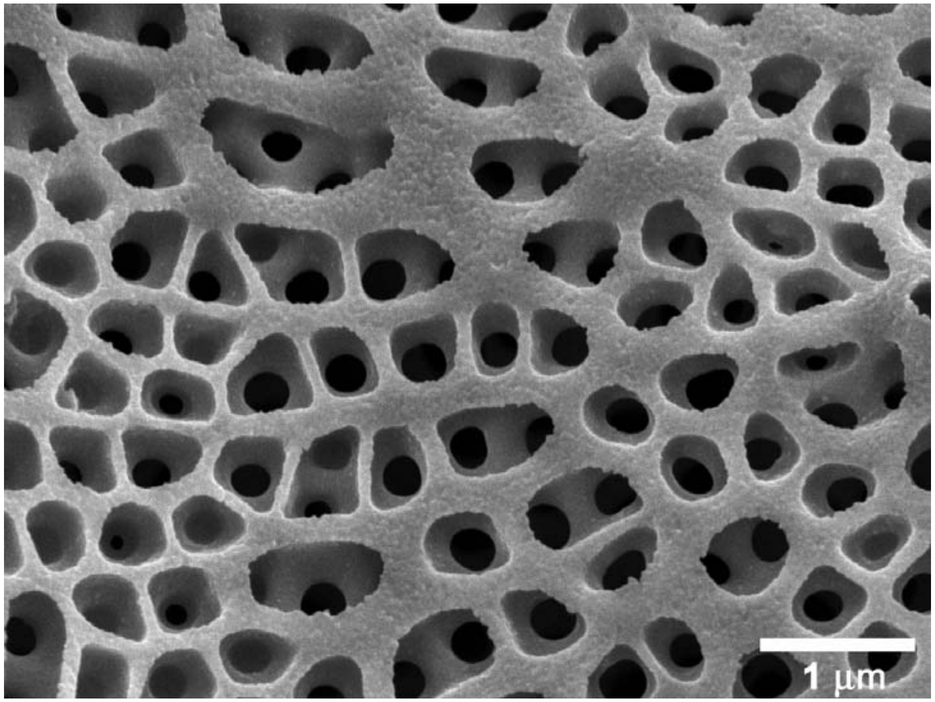
In Lens SE Detector

Some Examples SEM Imaged Nanostructures

- Materials Comparisons
- Structure Examination
- Silicon Nanowires
- Coatings
- Fabrication
- Nano Machines
- Nano Arrays
- Carbon Nanotubes

Example: Materials Comparisons





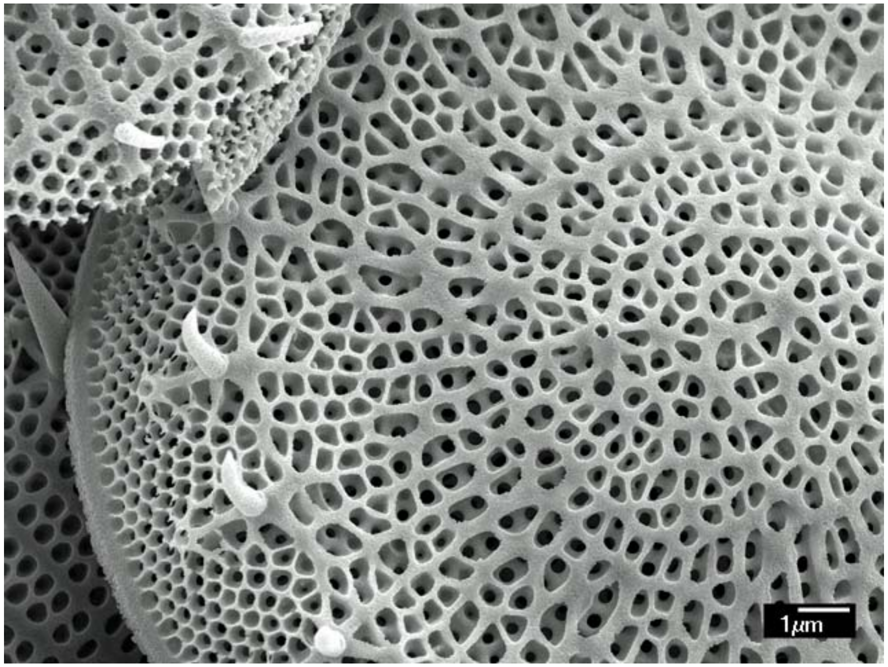
3DOM:
Composition: CaO (20 mol%) - P2O5 (4 mol%) - SiO2 (76 mol%)

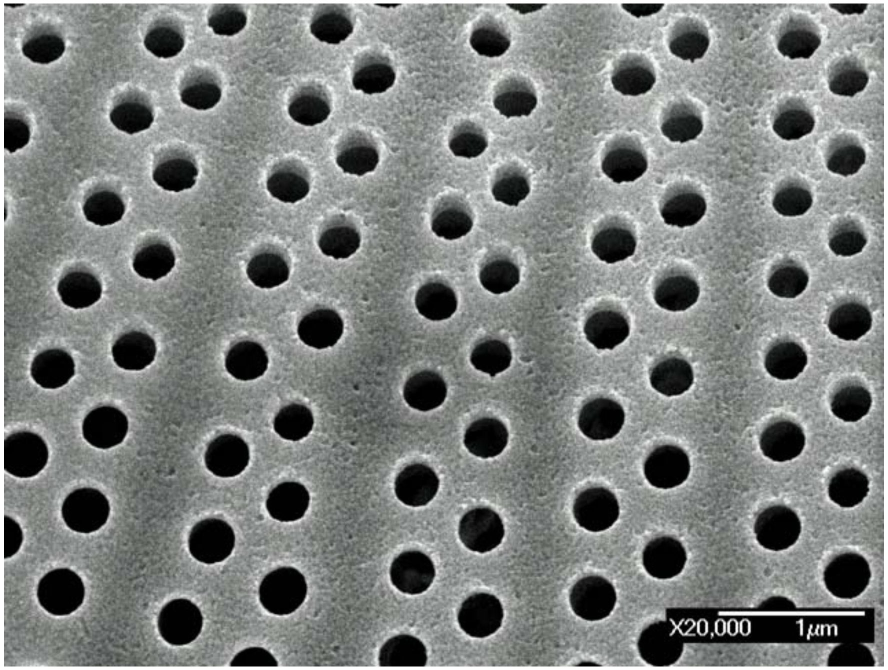
Sol-Gel Process

Diatom:
Stephanodiscus Niagarae (ME184)
Composition: SiO2 + small Ca

Phase Separation Process

Example: Diatoms

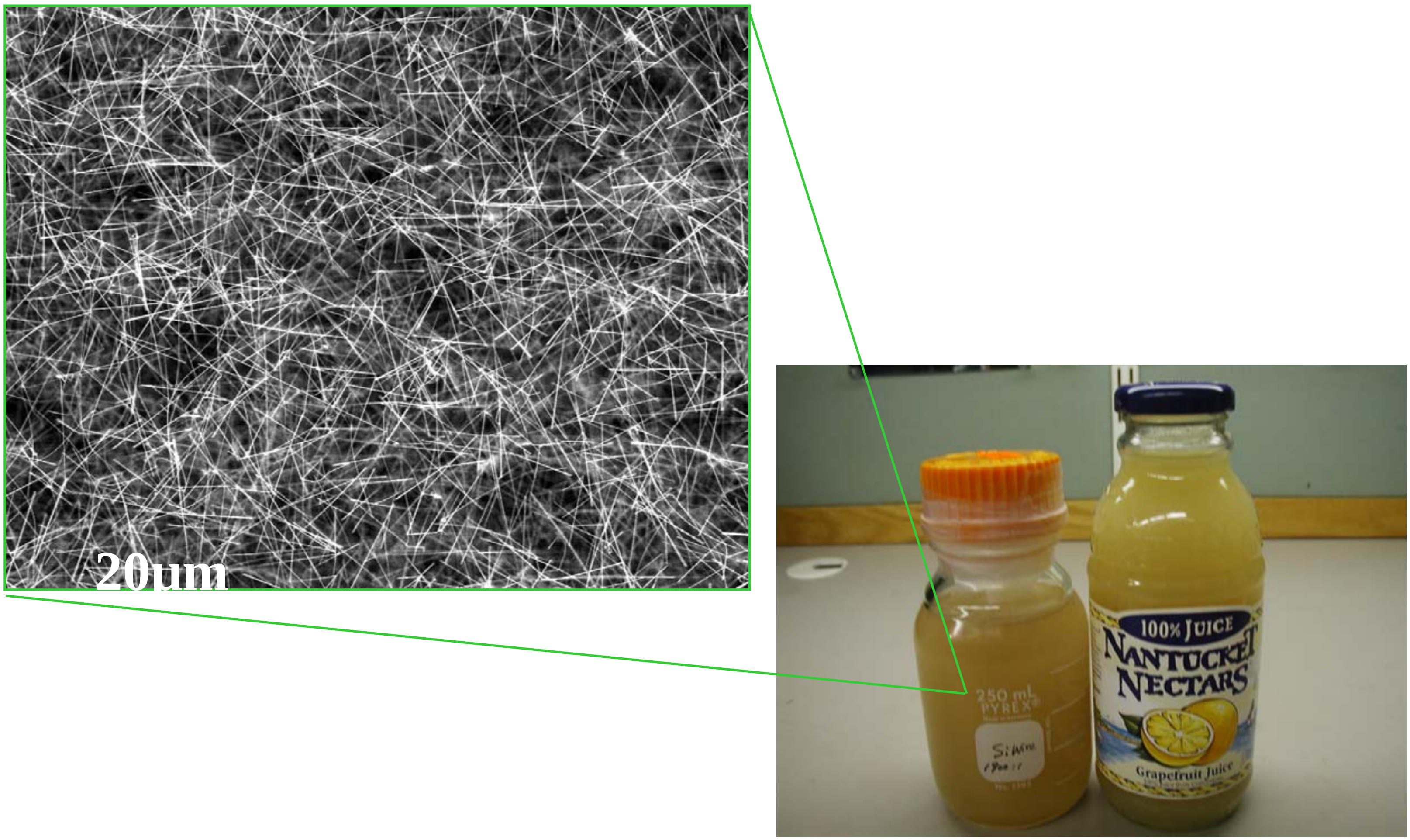




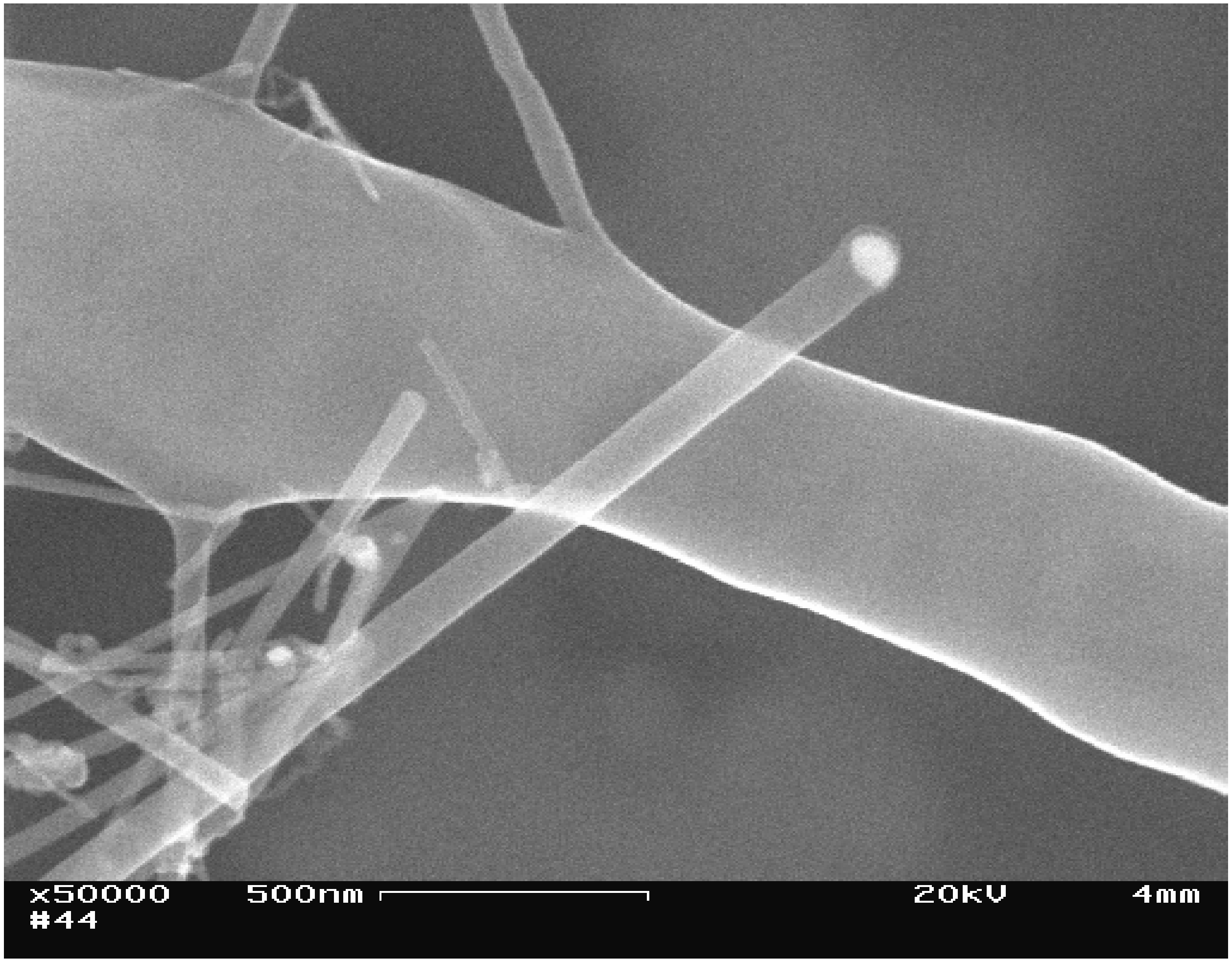
Stephanodiscus Niagarae (ME184)

Sample Courtesy of Mark Edlund, St. Croix Watershed Research Station

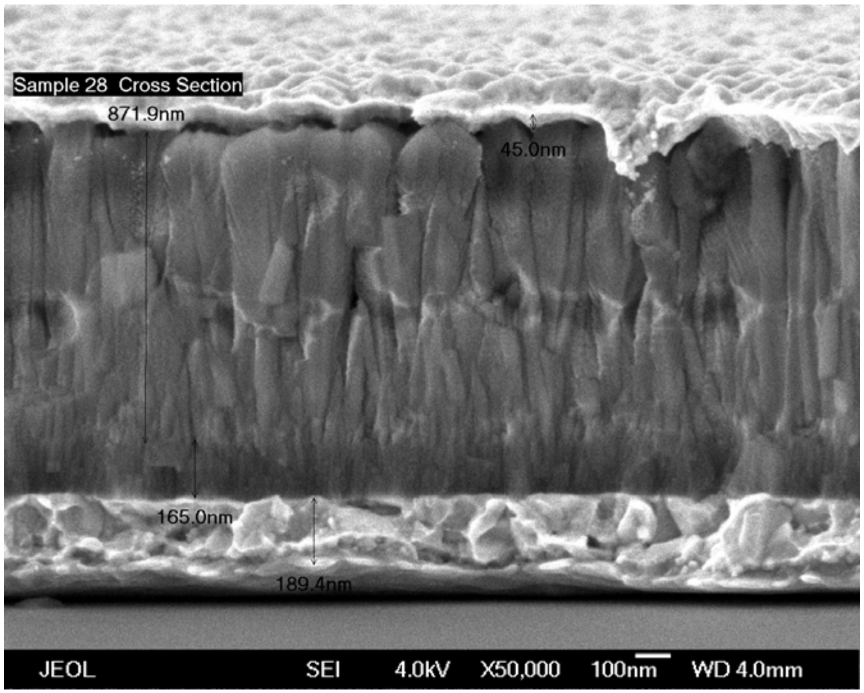
Example: Si Nanowires

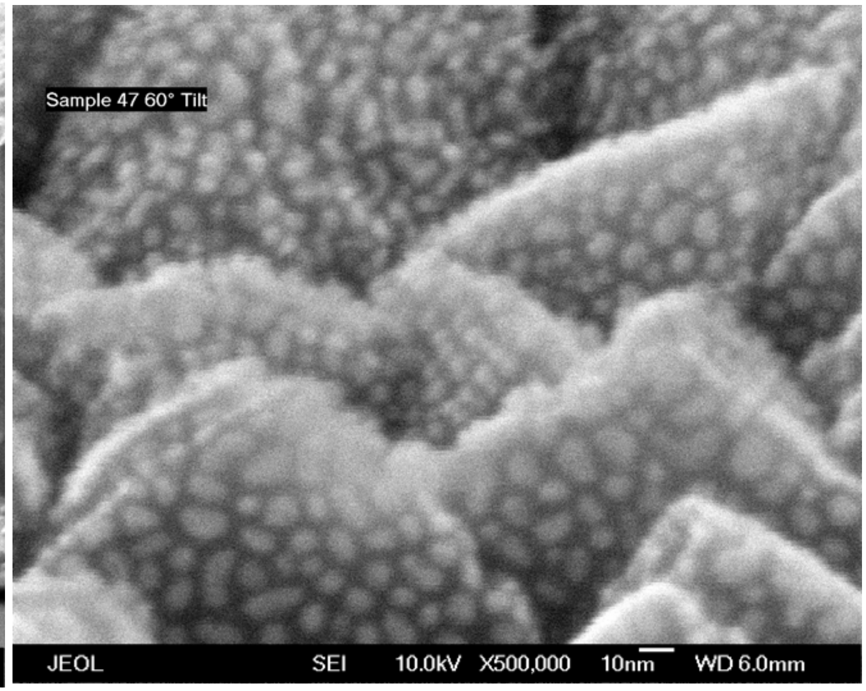


Example: Si Nanowires



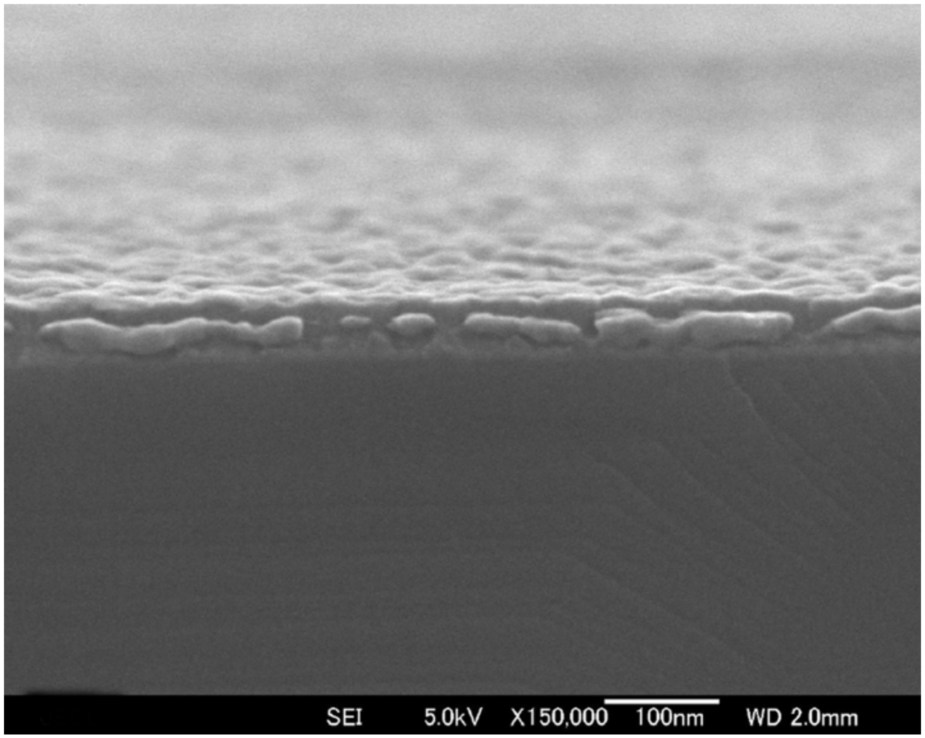
Example: Optical Coatings Au on SiC

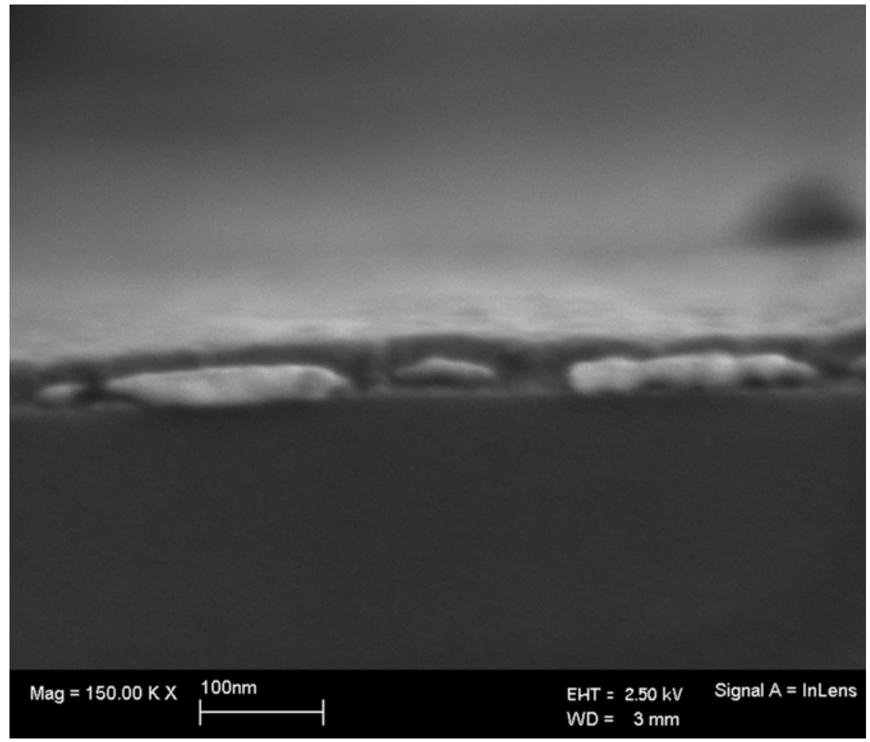




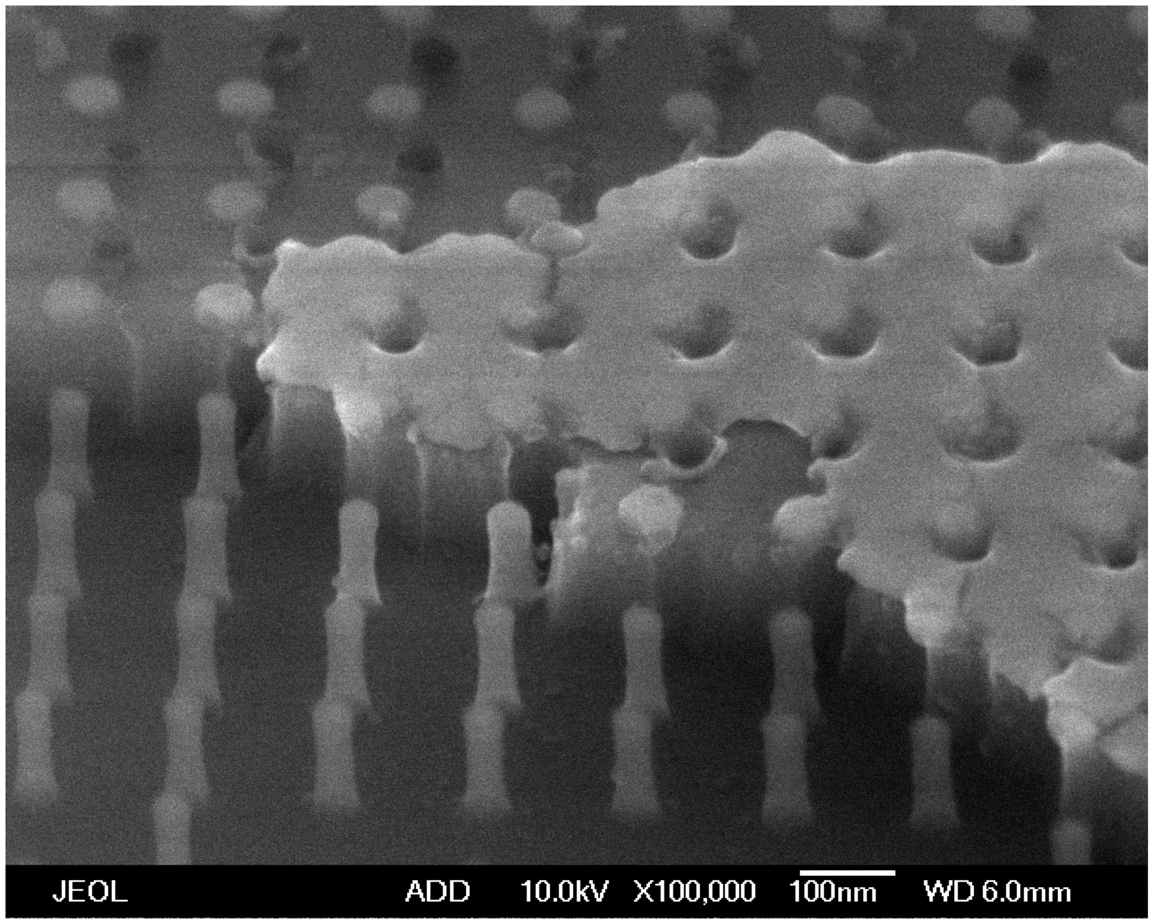
Courtesy Mike Coy JEOL

Example: Cross Sections



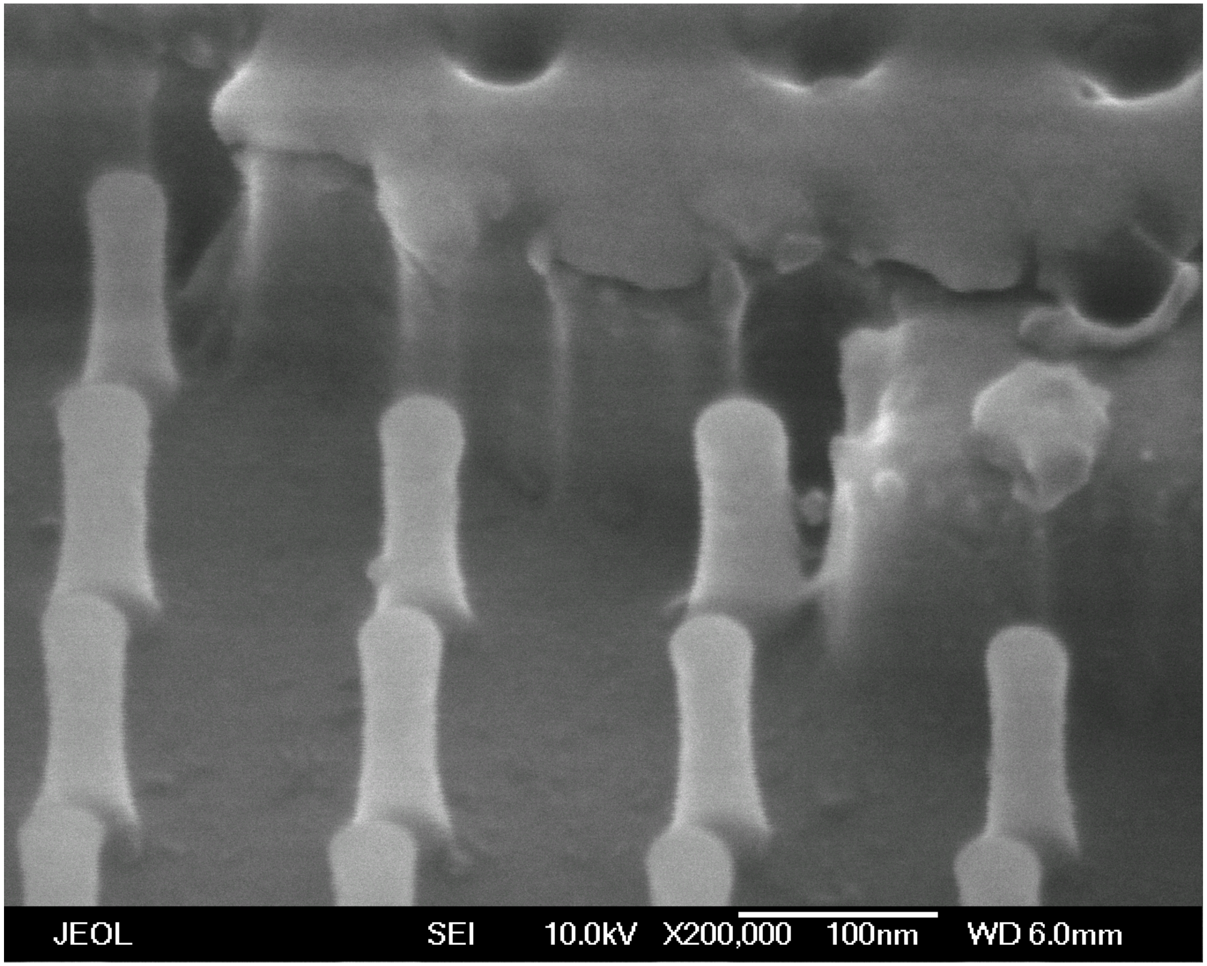


Example: Nano Array



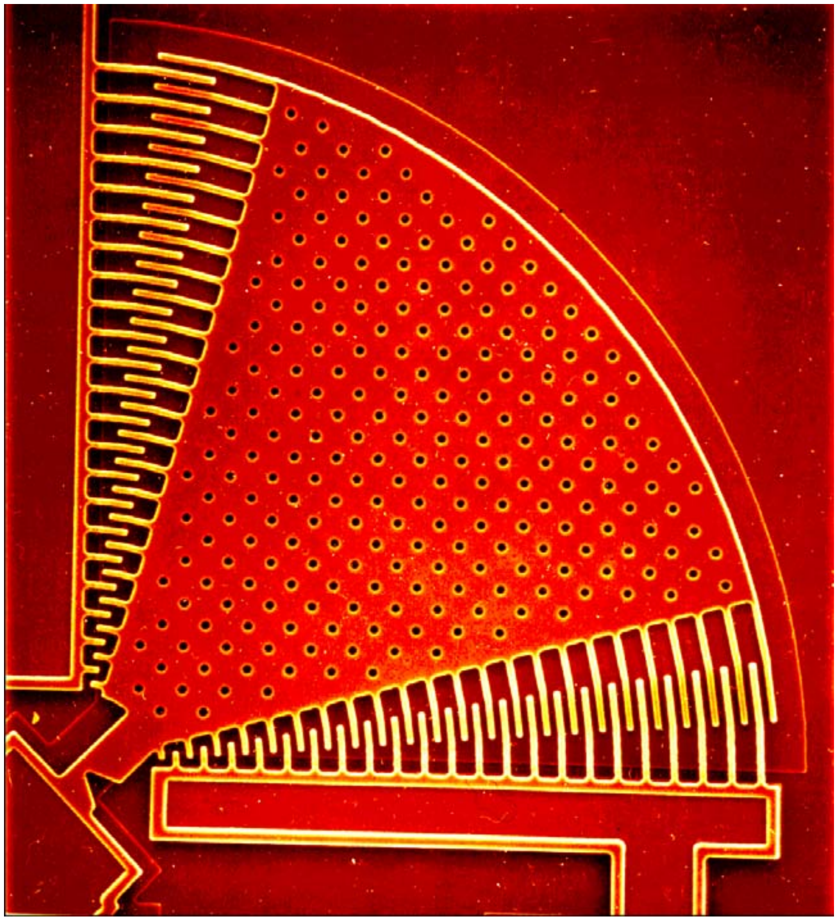
Courtesy Mike Coy JEOL

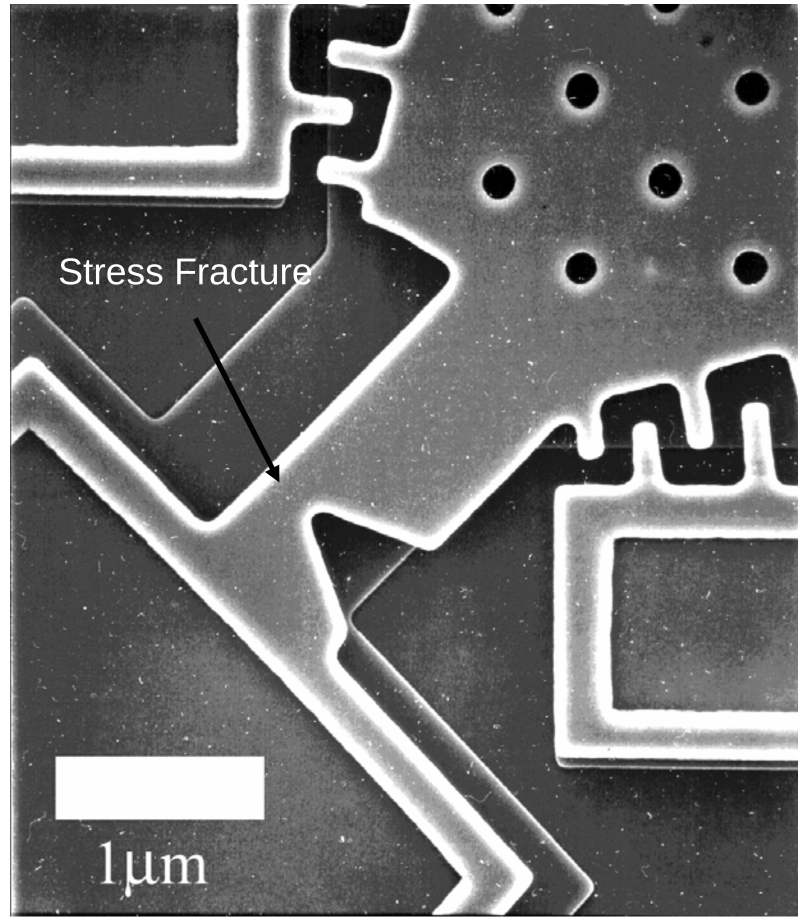
Example: Nano Array



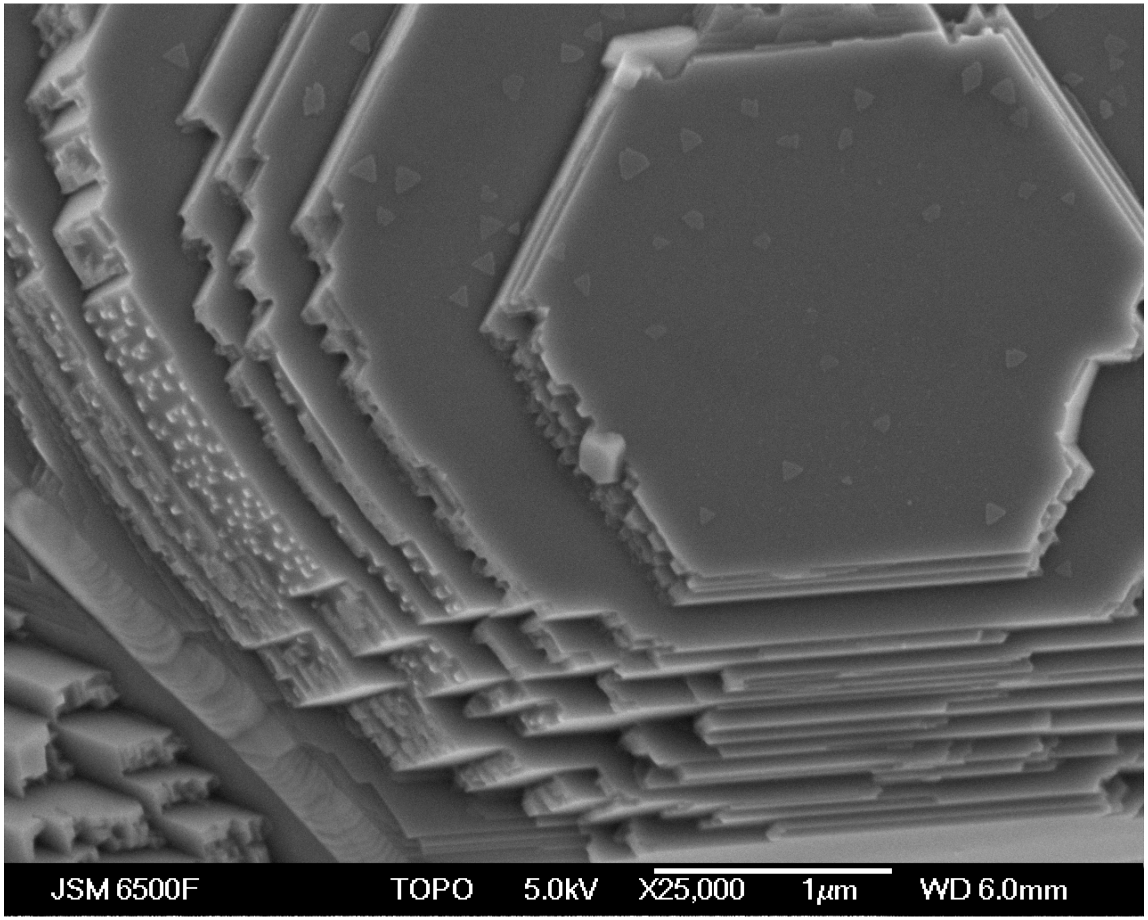
Courtesy Mike Coy JEOL

Example: Nano Machines Failure Mode Analysis

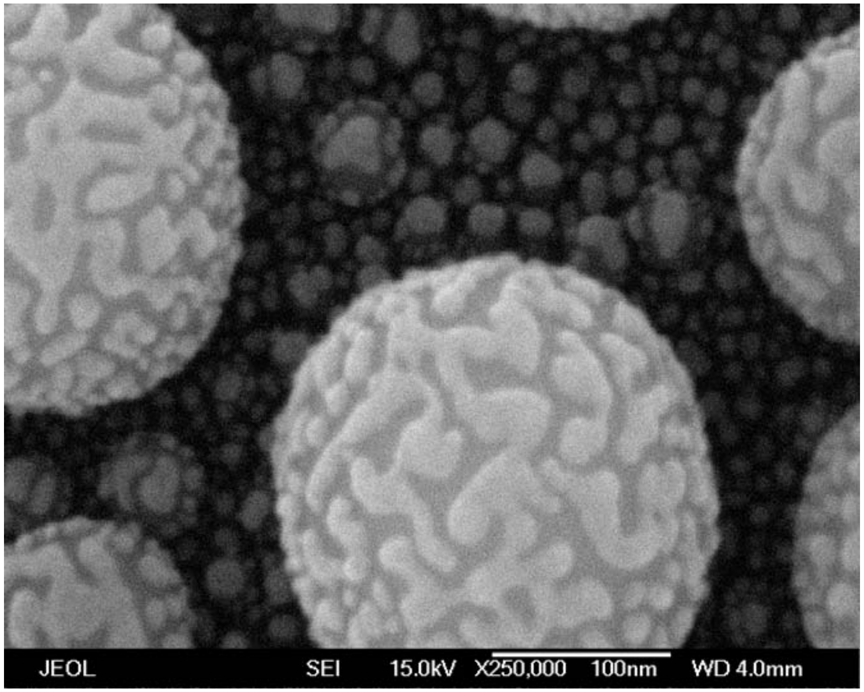


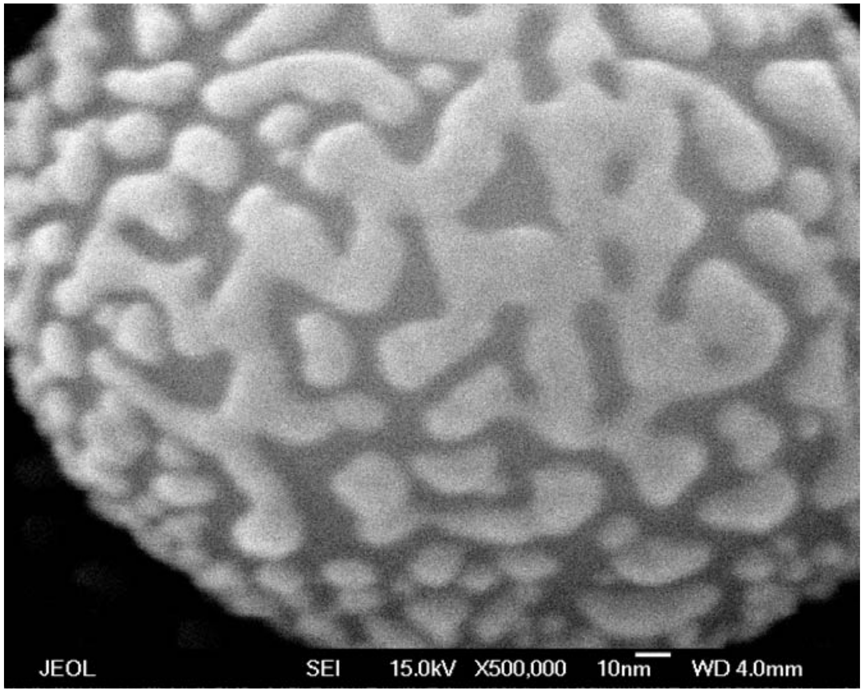


Example: Chromite

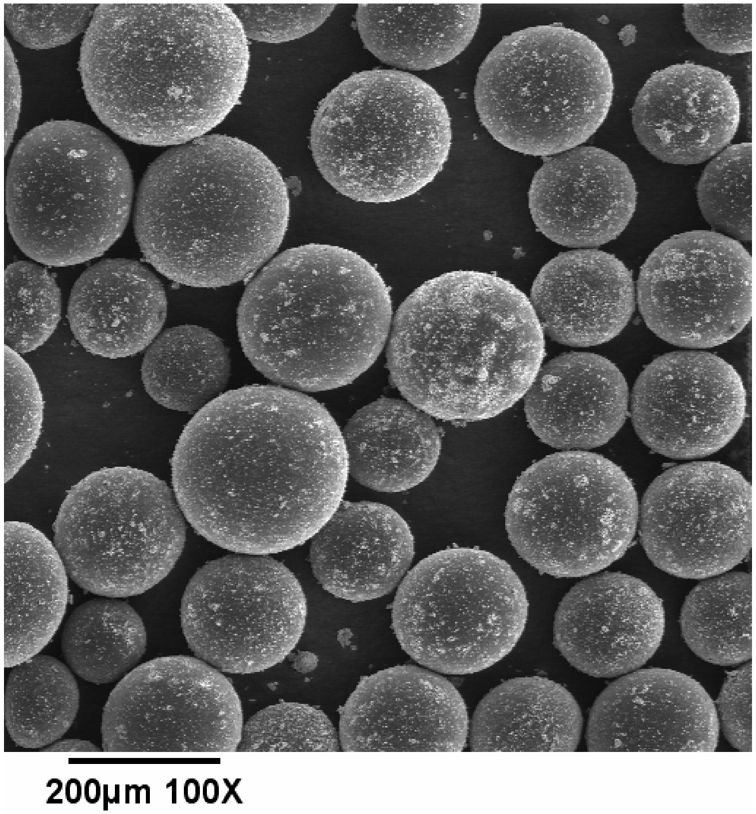


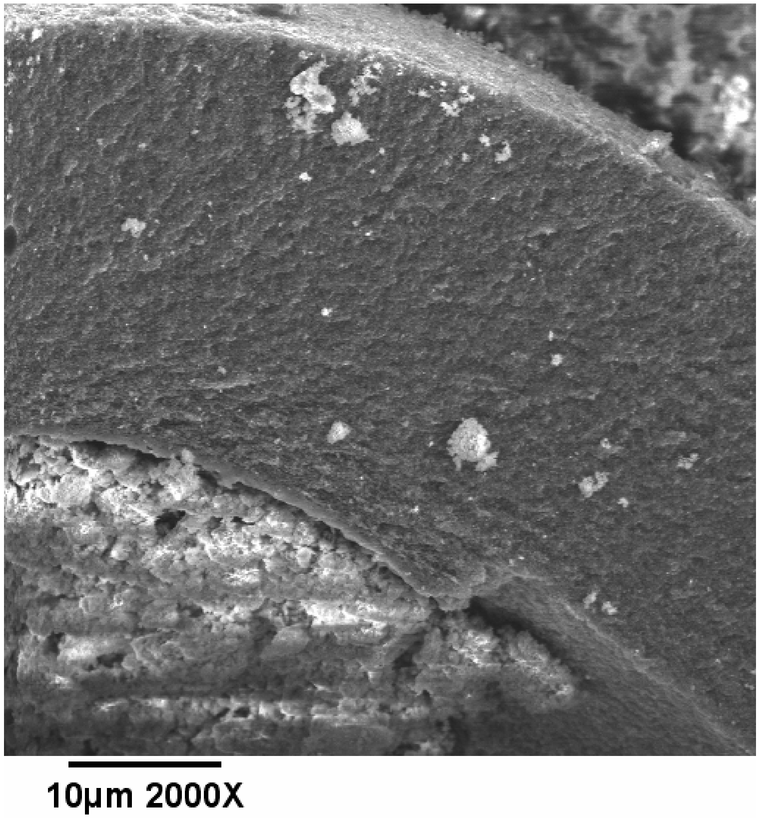
Example: SnO on Sn



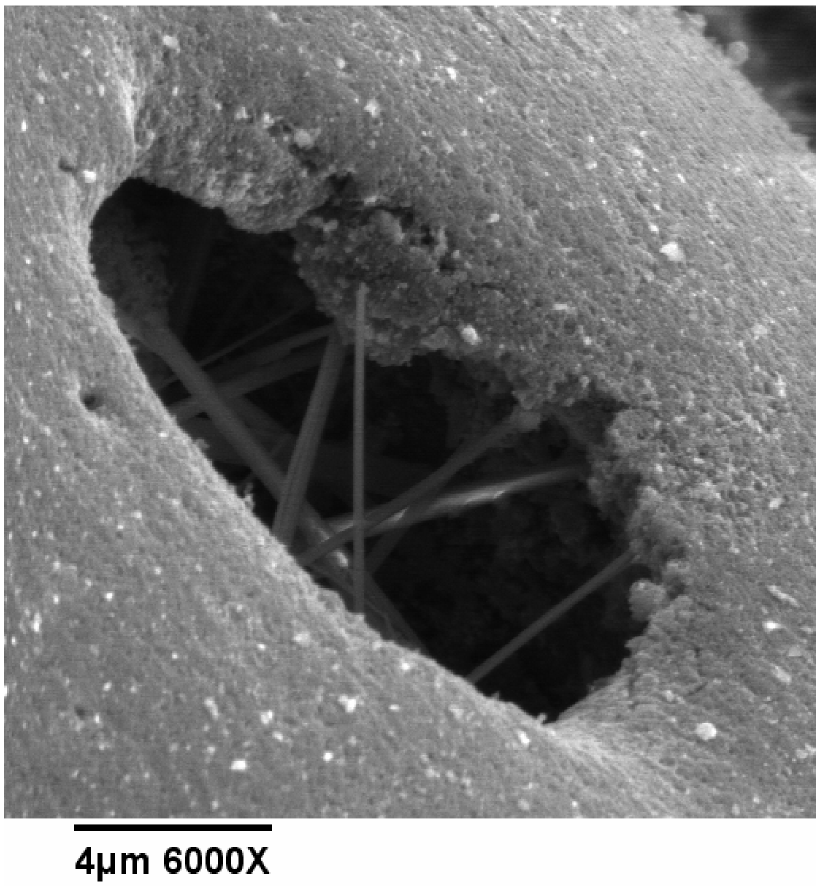


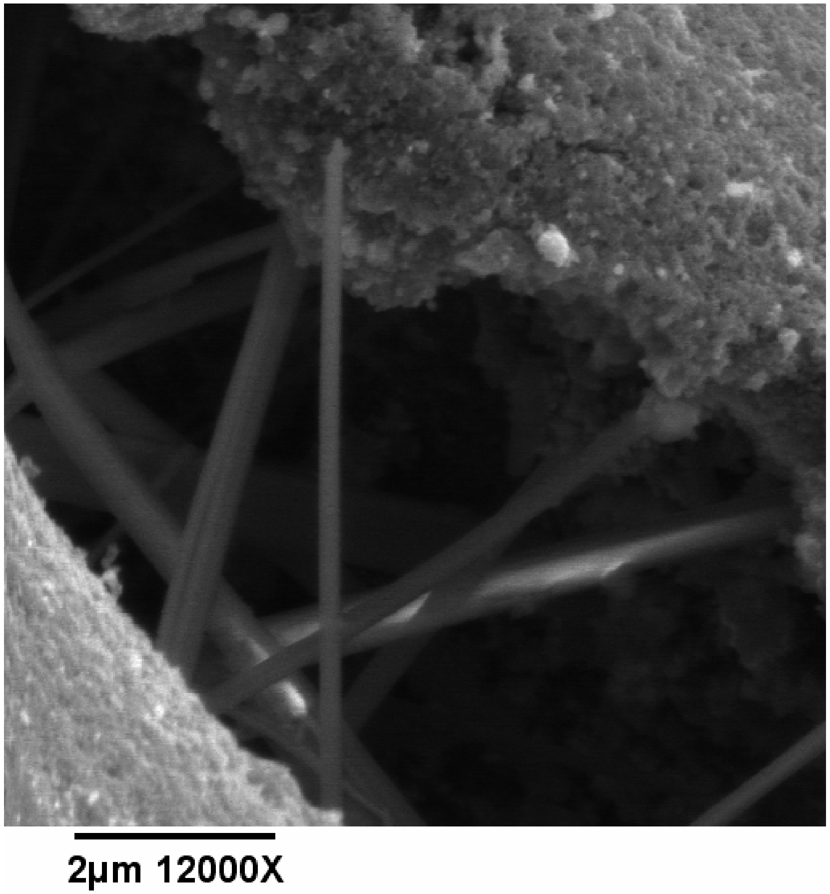
Example: Carbon Nanotube Spheres



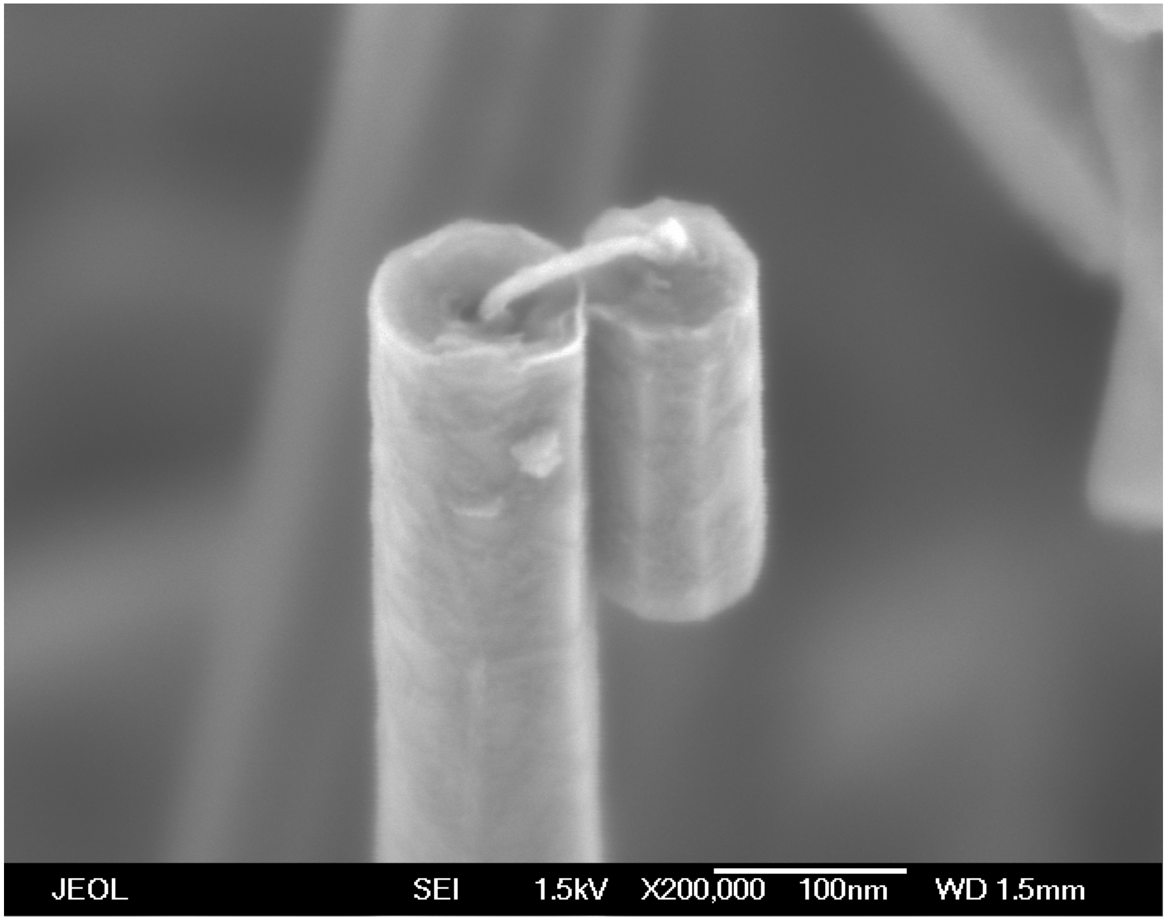


Example: Nanotube Spheres



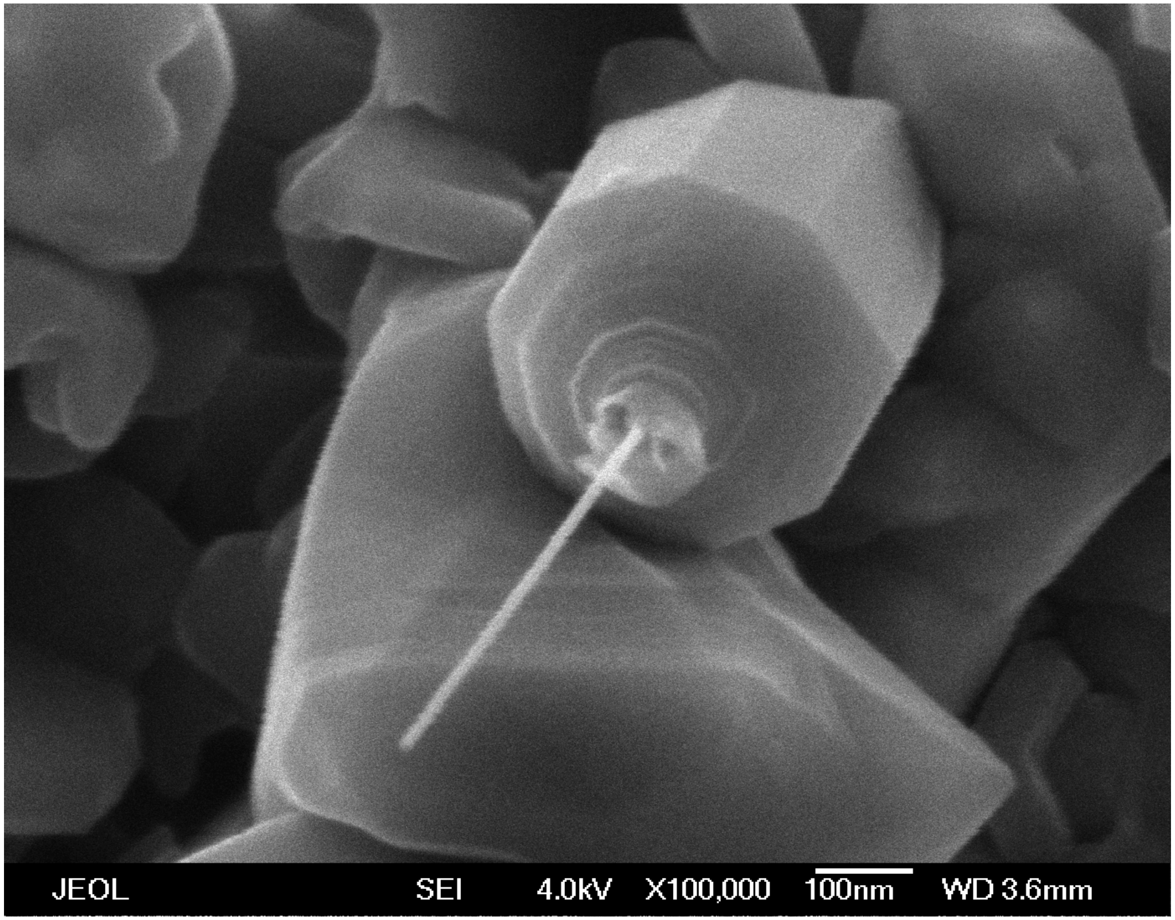


Example: Carbon Nanotubes



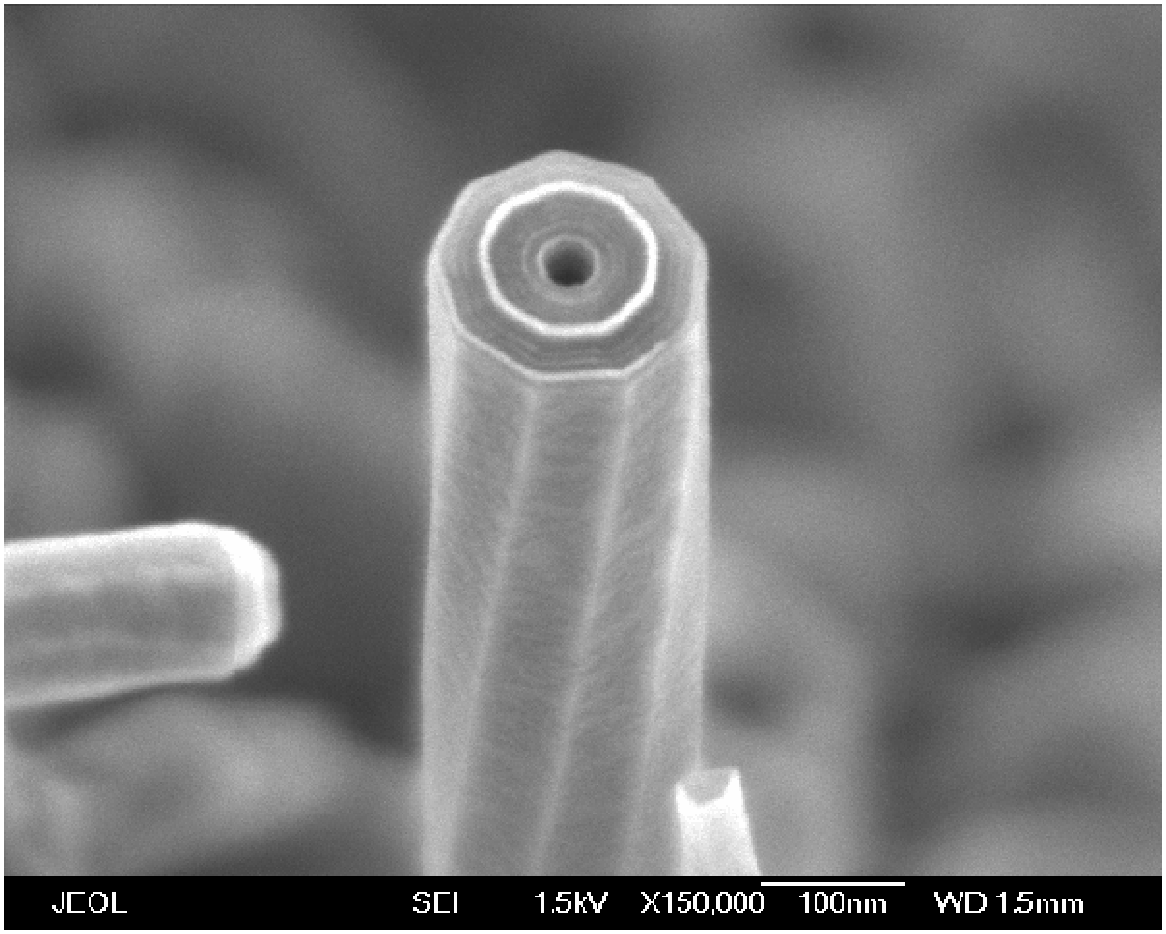
Courtesy Mike Coy JEOL

Example: Carbon Nanotubes



Courtesy Mike Coy JEOL

Example: Carbon Nanotubes



Courtesy Mike Coy JEOL

SEM : Nanostructures Imaging Checklist

- General SEM Considerations
  - Low beam voltage
  - Small apertures
  - Small working distance
  - Minimal sample coatings (if any)
  - Precise adjustment of astigmatism

- Instrument Considerations
  - In the lens SE Detector
  - Cold Field Emission Electron Gun
  - High Vacuum or UHV System

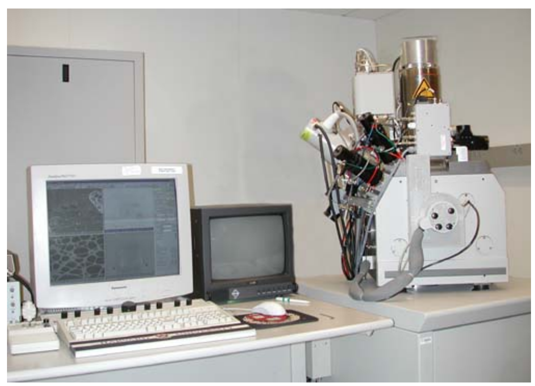
CIMS SEM Instruments

LEO A SEM

LEO B SEM

Cameca Microprobe

Quanta 200 ESEM



FEI D235 Dual Beam
FIB/SEM

Acknowledgements

- Richard Schalek , CIMS
- Warren MoberlyChan, CIMS
- Dave Lange, CIMS, EPS
- Yue Wu and Carl Barrelet , Charles Leiber Group
- Anthony J. Garratt-Reed, MIT
- Mike Coy, Verne Robinson, JEOL
- Tim Maitland, HKL Technologies