Scanning Electron Microscopy (SEM)

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Basic Methods of Materials Analysis

- Look at the Material (Shiny or Dull?)
- Hold Material (Heavy or 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

Incident kV electron beam

Backscattered electrons (BSE)

Secondary electrons (SE)

Auger electrons

Characteristic x-rays

Visible light

‘Absorbed’ electrons

Electron hole pairs

Specimen
Scanning Electron Microscope (SEM)
What is an SEM?

- **Electron Gun**
- **Electron lens**
- **Apertures**
- **Scan Coils**
- **Sample on a Stage**
- **Vacuum System**
- **Magnification Control**
- **Scan Generator**
- **Detector**
- **Amp**
- **Display**
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 but centered

Underfocus & off center

Centered & focussed
## Characteristics of Sources

<table>
<thead>
<tr>
<th>Characteristics</th>
<th>Units</th>
<th>Tungsten</th>
<th>LaB$_6$</th>
<th>FEG</th>
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<tbody>
<tr>
<td>Operating Temperature</td>
<td>K</td>
<td>2700</td>
<td>1700</td>
<td>300</td>
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<tr>
<td>Current Density</td>
<td>A/m$^2$</td>
<td>5x10$^4$</td>
<td>10$^6$</td>
<td>10$^{10}$</td>
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<tr>
<td>Crossover size</td>
<td>µm</td>
<td>50</td>
<td>10</td>
<td>&lt;0.01</td>
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<tr>
<td>Energy spread</td>
<td>eV</td>
<td>3</td>
<td>1.5</td>
<td>0.3</td>
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<tr>
<td>Stability</td>
<td>% / hr</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>5</td>
</tr>
<tr>
<td>Vacuum</td>
<td>Pa</td>
<td>10$^{-2}$</td>
<td>10$^{-4}$</td>
<td>10$^{-8}$</td>
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<tr>
<td>Lifetime</td>
<td>hr</td>
<td>100</td>
<td>500</td>
<td>&gt;1000</td>
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</tbody>
</table>
Magnetic lens

- Power
- Copper coils
- Water
- Soft iron
- Pole pieces
- Water-cooled surface
- Gap
- Electron ray paths
- Bore
- Concentrate flux
- Field varies
- Zero force on axial electrons
Secondary Electron Detector

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

- **W Electron Gun**
- **Electron Beam**
- **Environmental Backing Valve**
- **Specimen Chamber**
- **PLA #1**
- **PLA #2**
- **Auxiliary Gas (CO₂, N₂, Ar)**
- **Sample**
- **Turbomolecular Pump**
- **Vacuum Pump**
- **Pressure (Torr):**
  - 10⁻⁷
  - 10⁻⁵
  - 10⁻³
  - 10
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

Conventional SEM

Focused Ion Beam Systems

E-Beam Lithography

Wafer Inspection Tools

Other 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 Moberly Chan
SEM Based Materials Analysis

- Energy Dispersive X-ray Spectroscopy (EDS or EDX)
- Wavelength Dispersive X-ray Spectroscopy (WDS)
- Electron Backscattered Diffraction (EBSD or EBSP)
- Cathodoluminescence (CL)
- Backscattered Electron Detector (BSD)
- And Others
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

Ni/Fe Meteorite

EDX Map

FeOx  Fe  Ni
Electron backscattered Diffraction (EBSP or EBSD)
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
• 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 \( \Rightarrow \) 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 hierarchy versus theoretical for potential match phases
- If match is made within acceptable error limits, store data & repeat for next point

Collected EBSP

Move beam or stage

Hough space

Save data to file

Indexed EBSP

Phase and orientation

Courtesy Tim Maitland, HKL Technology
EBSP: Visualization of Data

General Micro-structure

Deformed silica (quartz)

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

Courtesy of Tim Maitland, HKL Technology
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

- High resolution
- Unclear surface structures
  - More edge effect
  - More charge-up
  - More damage
- Clear surface structures
- Less damage
- Less charge-up
- Less edge effect
- Low resolution

Accelerating Voltage
Effects of Accelerating Voltage: Example
Sample Interaction Volume with Voltage

Incident electrons

[Low acceleration voltage]

[High acceleration voltage]

[Low atomic number]  [High atomic number]
Working Distance

![Diagram showing the relationship between working distance, resolution, and depth of field.]

- High resolution
- Smaller depth of field
- Low resolution
- Greater depth of field

The working distance affects the resolution and depth of field of a sample in a scanning electron microscope.
Working Distance: Example
Aperture Size

- Large current (BEI, X-ray analysis)
  - Low resolution
  - Smaller depth of field
- High resolution
  - Greater depth of field
  - Grainy image
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

Specimen

Courtesy of Anthony J. Garratt-Reed
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 Niagareae (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

Stress Fracture
Example: Chromite
Example: SnO on Sn
Example: Carbon Nanotube Spheres
Example: Nanotube Spheres

4µm 6000X

2µm 12000X
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
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