SIGMA 500 FESEM Training
Practical Electron Microscopy

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Introduction to Electron Microscopy
Course Aim

To provide the SEM user with an understanding of the operational principles, procedures and practical experiences to enable good quality micrographs to be achieved.

- Combination of theory and practical
- Theoretical background of SEM
- Practical aspects of SEM operation
- Hands on experience of SEM using real specimens
Some SEM History & Origin of Zeiss SEMs

• 1847  Carl Zeiss begins making optical microscopes
• 1881  Cambridge Scientific Instruments founded by Horace Darwin
• 1931  Germans Max Knoll & Ernst Ruska invent the TEM and later STEM
• 1938  Von Ardenne publishes the seminal theoretical and experimental work on the SEM. Von Ardenne is largely credited as being the true father of the SEM
• 1942  Zworykin independent work at RCA in the USA advances the technology but still not high resolution except in STEM mode
• 1953  Dan McMullan, the first of three research students (later also K.C.A. Smith & O. Wells) under the direction of Sir Charles Oatley at Cambridge University builds the first viable high resolution SEMs. This leads directly to the first commercial SEM.
• 1965  First commercial SEM *(Cambridge Instruments Stereoscan Mark I)*
Early Scanning Electron Microscopes
Some SEM History & Origin of Zeiss SEMs

• 1985   First all digital SEM: ZEISS DSM 950
• 1986   First software controlled SEM: Cambridge Stereoscan 360
• 1990   Leica Electron optics formed, a merger between Cambridge Instruments and Wild Leitz
• 1992   Second generation software controlled SEM (*Leica 400*)
• 1996   LEO Electron Microscopy formed as a joint venture between Leica and Carl Zeiss
• 1999   Third generation software controlled *SEM (LEO 1400)*
• 2001   Carl Zeiss acquires all of LEO and SEMs are branded ZEISS
Scanning Electron Microscope

General Operating Principle of a SEM
Electron Optics

Operating principle of the **Gemini** column

### Features

- **highly stable thermal FEG**
  - < 0.5 % /h variation

- **low beam noise**
  - < 1 %

- **cross over free beam path**
  - No significant Boersch effect, high depth of field

- **beam booster**
  - Superb image resolution throughout the whole beam energy range, particularly down to 100 eV.
  - High resistance to ambient magnetic stray fields
Sigma 500
Specification & Option Overview

**Sigma 500**

| Res         | 0.8 nm @ 15 kV  
|             | 1.6 nm @ 1 kV   |
| Clear View Detection Suite |
| Inlens SE or Inlens Duo |
| ETSE        |
| VPSE-G4     |
| C²D         |
| AsB / HDBSD / SenseBSD |
| CL          |
| YAG Detector |
| BSD4        |
| aSTEM       |

**Detection**

**Other**

32 x 24 k image store
Plasma Cleaner
Eucentric or Cartesian stage
80 mm or 200 mm Airlock
What is the meaning of Resolution?

**What is Resolution?** Resolution is defined as the smallest distance between two particles that can be distinguished.

**Resolution measurement**
Resolution is not only dependent on the SEM, but on the sample itself and operating conditions used in its observation.
✓ In the SEM, the magnification is the ratio of the area scanned on the sample to the output device.

✓ The smaller the area scanned, the higher the magnification and visa versa.

✓ Historically, the output device was the Polaroid film (4”x5”)

For example, if $L_V = 500$ pixels, $L_{R1} = 5$ pixels, and $L_{R2} = 50$ pixels, then

\[ l_1 = \frac{500}{5} = 100 \times \]
\[ l_2 = \frac{500}{50} = 10 \times \]
Many signals are produced when an electron beam interacts with a sample.

- Incident Electron Beam
- Specimen
  - Absorbed Electrons
  - Elastically Scattered Electrons
  - Transmitted and in-elastically scattered Electrons
  - Backscattered Electrons
  - Secondary Electrons
  - Auger Electrons
  - Visible light (Cathode Luminescence)
  - Bremsstrahlung
  - Characteristic X-Ray photons
Beam Interaction Diagram

TEM
Electron beam
Cutaway section
SEM
Electron beam
Sample surface
Whole sample

Incident electrons (electron probe)
Secondary Electrons
Auger electrons
Backscattered Electrons
Continuum X-rays
Characteristic X-rays
Fluorescent X-rays

Electron Beam Interaction Diagram
## Electron Gun Types

<table>
<thead>
<tr>
<th></th>
<th>W</th>
<th>LaB6</th>
<th>Schottky FE</th>
<th>Cold FE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beam diameter</td>
<td>1 - 2 μm</td>
<td>1 - 2 μm</td>
<td>10 - 25 nm</td>
<td>3 - 5 nm</td>
</tr>
<tr>
<td>Temperature</td>
<td>2300° C</td>
<td>1500° C</td>
<td>1500° C</td>
<td>Room temp</td>
</tr>
<tr>
<td>Brightness</td>
<td>1</td>
<td>10</td>
<td>500</td>
<td>1000</td>
</tr>
<tr>
<td>Energy spread, ΔE</td>
<td>2.0 eV</td>
<td>1.5 eV</td>
<td>0.5 eV</td>
<td>0.2 eV</td>
</tr>
<tr>
<td>Stability, %/h</td>
<td>0.1 %</td>
<td>0.2 %</td>
<td>0.1 %</td>
<td>5 %</td>
</tr>
<tr>
<td>Probe current</td>
<td>1 μA</td>
<td>1 μA</td>
<td>&gt;100 nA</td>
<td>10 nA</td>
</tr>
<tr>
<td>Life time</td>
<td>1 month</td>
<td>6 months</td>
<td>18 months</td>
<td>5 years</td>
</tr>
<tr>
<td>Gun vacuum</td>
<td>10⁻⁵ Torr</td>
<td>10⁻⁷ Torr</td>
<td>10⁻⁹ Torr</td>
<td>10⁻¹¹ Torr</td>
</tr>
</tbody>
</table>

⭐⭐ Major Benefits of Schottky FE
The Schottky Emitter

✓ The tip is welded to a filament and is then centered mechanically in the Suppressor electrode which prevents stray thermionic emission from passing down the column.

✓ The voltage on the extractor electrode controls the emission current from the gun.

✓ The Anode is at ground potential (positive).
Schottky FE Emitter

Filament tip with collar-like ZrO reservoir (left), ZrO depleted tungsten filament (right).
Detectors Types

✓ Secondary Electron Detector (SED)
  • In-chamber Everhart Thornley
    • Best for surface topography
  • In-column (in-lens) or InLens Duo (backscatter, energy filtered)
    • Best for surface information (low energy)

✓ Backscattered Detector (BSE)
  • AsB solid state (below objective lens)
    • Best for channeling contrast (3kV and above)
  • EsB in-column
    • Best for Z contrast (3kV and below)
  • NTSBSD (4 or 5 Segment)
    • Suited for Z contrast and topographic imaging
  • SenseBSD - TEM-like images at low kV (7kV and below)

✓ Scanning Transmitted Electron Detector - aSTEM

✓ Variable Pressure Detector – VPSE G4
  • Ionization Detectors used at 1Pa and higher

✓ Energy Dispersive Spectrometer (EDS)

✓ Wavelength Dispersive Spectrometer (WDS)

✓ Electron Backscattered Diffraction Pattern (EBSD or EBSP)

✓ Cathodoluminescence Detector (CL)
In-Chamber Everhart Thornley Secondary Electron Detector

Requires High Vacuum
In-Lens Everhart Thornley Secondary Electron Detector

- Excite scintillator material (e.g. ZnS, NaI) (approx. 10kV acceleration voltage at crystal)
  - De-excitation by emission of light
  - Emission of electrons due to photoelectric effect in photomultiplier
  - Avalanche charge (signal enhancement)
  - Amount of charge ~ primary signal

Advantage: fast, best image quality
Disadvantage: scintillator voltage is limiting use to high vacuum
## Secondary Electron Detection

**InLens:**
- detail surface information
- edge effects
- SE1 detection
- sensitive to charging
- highest Resolution possible
- shorter working distance (WD) – better resolution
- optimum WD-range: <5mm (for beam booster to be efficient)

**Everhart-Thornley:**
- topographic surface information
- edge effects
- distinct shadows due to geometry
- SE2 detection
- insensitive to charging
- optimum WD-range: 5-12mm

![InLens](image1.png) ![SE2 (topography)](image2.png)
Solid State BSD Principle

- Backscattered electrons and secondary electrons collide with the introduced gas molecules and produce ions that dissipate the charge on the sample surface.

- Higher energy electrons (BSE) travel upwards.

- Backscattered electrons are detected by QBSD and converted to electrical signal.

- Amplified signal by head amplifier to signal processor; used to form an image.
Uncoated glass shard – VP mode, 15 kV
<table>
<thead>
<tr>
<th>Detector</th>
<th>Information</th>
<th>Opt. Working Distance</th>
<th>Energy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Everhart-Thornley (high vacuum)</td>
<td>Topographic surface information</td>
<td>5-12mm</td>
<td>0.02-30kV</td>
</tr>
<tr>
<td>VPSE-G4 (VP/high pressure)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>InLens/Duo</td>
<td>Surface details, High Resolution Imaging</td>
<td>&lt;5mm</td>
<td>0.02-20kV</td>
</tr>
<tr>
<td>AsB/CZ NTS BSD/Z</td>
<td>Z-Contrast</td>
<td>5-10mm</td>
<td>&gt;5kV</td>
</tr>
<tr>
<td></td>
<td>Channeling contrast (crystallographic information, strain, deformation)</td>
<td>2-5mm (material dependent)</td>
<td></td>
</tr>
<tr>
<td>InLens Duo</td>
<td>Z-Contrast (sharp due to energy filter)</td>
<td>&lt;5mm</td>
<td>&lt;1.5kV</td>
</tr>
<tr>
<td></td>
<td>Low Loss BSE: Compositions, Bondings</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SenseBSD</td>
<td>Low kV high contrast TEM-like imaging</td>
<td>4-6mm</td>
<td>&lt;=7kV (absolute)</td>
</tr>
</tbody>
</table>
The Three Basic Parameters
The Three Basic Parameters

✓ **Accelerating Voltage** (EHT)

✓ **Aperture Size** (diameter) which controls **Spot Size** (beam or probe current)

✓ **Working Distance** (sample-to-lens distance)
### Accelerating Voltage

<table>
<thead>
<tr>
<th></th>
<th>High kV</th>
<th>Low kV</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Higher resolution</td>
<td>Lower resolution</td>
</tr>
<tr>
<td></td>
<td>Greater beam penetration</td>
<td>Less beam penetration</td>
</tr>
<tr>
<td></td>
<td>Higher beam current</td>
<td>Lower beam current</td>
</tr>
<tr>
<td></td>
<td>Higher signal to noise</td>
<td>Lower signal to noise</td>
</tr>
<tr>
<td></td>
<td>More x-ray yield</td>
<td>Lower x-ray yield</td>
</tr>
<tr>
<td></td>
<td>Good for VP</td>
<td>Not so good for VP</td>
</tr>
</tbody>
</table>
Beam Sample Interaction – Influence of Beam Energy

*Platinum Rhodium Alloy Crystals at 1kV (left) and at 20kV (right)*
Aperture Size (changes Spot Size)

**Small Aperture**
- Higher resolution
- Higher depth of focus
- Lower beam current
- Lower signal to noise
- Less x-ray yield
- May have smaller field of view (aperture cut-off)

**Large Aperture**
- Lower resolution
- Less depth of focus
- Higher beam current
- Higher signal to noise
- Higher x-ray yield
Spot Size

• The aperture diameter controls the final spot size on the specimen.

• The smaller the spot size, the better the resolution; however, this smaller probe comes at the sake of losing beam current.

• With a small spot size, the area between the two blocks can be distinguished; with the larger spot size, it cannot.

• OptiProbe adjusts spot size.
Working Distance

**Short Working Distance**
- Higher resolution
- Less Depth of Focus
- Better collection efficiency for In-Lens/Duo, AsB, NTSBSD, SenseBSD, and EsB detectors

**Long Working Distance**
- Lower resolution
- Greater Depth of Focus
- Better collection efficiency for In-Chamber SE
Variables Involved in Image Formation

- Acceleration Voltage (kV)
- Final Aperture (size)
- Probe Current (I probe)
- Working Distance (WD)
- Specimen
- Column/Aperture Alignment

- Detector
- Specimen geometry
- Scanning speed
- Signal processing
- Noise reduction
- Contrast / brightness
- Chamber pressure
- System cleanliness
Questions to Ask Yourself

What is information is required from the specimen

- High Magnification
- Low Magnification
- Compositional information
- Topographical information
- X-ray analysis

Other Factors

- Stability
- Beam sensitivity
- Electrical conductivity
Charging and Contamination
Choosing a detector

- The choice of detector can have a significant effect on the apparent severity of charging.

- The conventional ET (Everhart - Thornley) detector is much less sensitive to charging than...

Individual polymer macro-molecules on Si at 1.5keV - Lower (ET) detector
Upper detector

- …the InLens detector. This is because these systems act as simple SE spectrometers and preferentially select low energy electrons.
- Note however that charging can be a useful form of contrast mechanism when properly employed.
- Backscattered electrons are less affected by charging and offer the same resolution at LV.

Same area as before using InLens detector.
If all else fails…..coat the sample

- **Coatings do not make the sample a conductor**
- They form a ground plane - i.e. the free electrons in the metal move so as to eliminate the external field
- The charge is not eliminated but the disruptive field is removed
- Successful coating means paying attention to the details...

Courtesy Dr. David Joy – ORNL / UTK
Software
SmartSEM® software

- **Dual Channel:**
  The possibility of detecting and displaying image information simultaneously from two different detectors allows users to quickly distinguish small differences on the sample.

- **Control Panel:**
  An optional hardware control panel is available with rotary control for improved tactile feedback.
Recipes
Using the recipes option, users can easily define and store protocols. The recipes enable the users to run the entire imaging process at a click of the mouse.

Macros
Creating and running macros make routine procedures to be performed easily and quickly. Various macros in the software are easily accessible and linked to the icons in the toolbar. Using the assigned macros reduces the imaging time, aids reproducibility and accuracy.
Image Navigation

➢ Locating the region of interest on a specimen in the SEM might sometimes be challenging, especially when identifying small features on large specimens.

➢ SmartSEM® graphical user interface facilitates easy and fast navigation using built-in camera as well as images from other sources as below

➢ Images can be imported from:

• A live, or stored, SEM image
• A digital camera, webcam or light microscope
• A CAD package
ZEISS Sample Holders

Carousel 9 stubs  |  Carousel 8 stubs  |  Single stubs  |  Single stubs  |  12 x single stubs

Universal 45° holder  |  Universal 75° angled  |  Large multi purpose  |  Multi purpose variable  |  70° angled Multi slice wafer section

Flat cleaved: clips for wafer sections  |  60° angled Multi slice wafer section  |  wafer sections  |  20° angled wafer section  |  4” or 5” wafer holder
SmartSEM® software

➢ Image store:
  Image resolutions up to 32k x 24k pixels images can be acquired and stored as TIF, BMP or JPG files.
  ATLAS (optional) 32k x 32k pixel images

➢ Annotation and measurements:
  A paint style control panel enables point to point, line width, line height, radial, and angle measurements on the image.
  Annotation of images with text, bitmaps, and micron markers is possible using all Windows™ fonts and colors.
  Insertion/recall of TIFF data from saved images is also possible.
SmartSEM® software

➢ Administrator function:

New users accounts can be easily created with various levels of access to the functionality of the instrument.

The usage of the system can also be monitored for individual users.
Image Optimization
Image Optimization

✓ What information and magnification will provide the data you are seeking
✓ Start off with low voltage and increase as resolution requires – never start at high voltage unless you know your sample
✓ Know the principles of "the three parameters"
   • Accelerating voltage
   • Aperture/Spot Size
   • Working Distance
✓ Make sure your sample is securely mounted to the specimen stub
✓ Make alignment and image adjustments at highest possible magnification
✓ Check aperture alignment
✓ Find center of focus (very important) before adjusting the stigmators – avoid edges or straight lines
✓ Repeat focus and stigmation
✓ Lower magnification, adjust C&B and select an appropriate scan mode
Noise Reduction

• The line and frame averaging options for the framestore can greatly reduce noise

• This should not used as an excuse to use beam currents that are too low!

• Whenever possible take a single slow speed scan rather than accumulating multiple high speed scans

• This eliminates blurring due to drift, and distortions in the video amplifier chain and usually produces a higher signal to noise ratio and better contrast

• Higher pixel resolution images require longer acquisition times compared to low pixel resolution images – dwell time should remain the same

Effect of the averaging on noise

Courtesy Dr. David Joy – ORNL / UTK
Hands On Operation
We make it visible.