Scanning Electron Microscopy & Ancillary Techniques

By Pablo G. Caceres-Valencia

The prototype of the first Stereoscan supplied by the Cambridge Instrument Company to the duPont Company, U.S.A. (1965)
The SEM permits the observation and characterization of heterogeneous organic and inorganic materials on a nanometer (nm) to micrometer (μm) scale.

The SEM is one of the most versatile instruments available for the examination and analysis of the microstructural characteristics of solid objects.

There more than 50,000 SEM world-wide.

The JSM (now known as the JSM-1) was JEOL's first commercially produced Scanning Electron Microscope. The JSM -1 was made commercially available in 1966. Among its advanced features was a Eucentric Stage.

Resolution: 250Å (at 25kV)  Magnification: 100 - 30,000  Scan area: 1x1 mm (at 25kV)
Some Micro-Analytical Techniques

**SEM/EDS** = scanning electron microscope/energy-dispersive spectrometer
**EPMA/WDS** = electron probe microanalyzer/wavelength-dispersive spectrometer
**AEM/EDS** = analytical transmission electron microscope/energy-dispersive spectrometer
**AEM/EELS** = analytical transmission electron microscope/parallel collection energy loss spectrometer
**AES/SAM** = Auger electron spectrometer/Scanning Auger microscope
**SIMS** = Secondary ion mass spectrometer
**PIXE** = proton induced X-ray emission
**RBS** = Rutherford backscattering
<table>
<thead>
<tr>
<th>Technique Name</th>
<th>Input Beam</th>
<th>Output Signal</th>
<th>Lateral Resol.</th>
<th>Depth Resol.</th>
<th>Detection Limit</th>
<th>LEA*/ Imaging</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>SEM/ EDS</td>
<td>Electrons (0.1-30keV)</td>
<td>X-rays (&gt;100eV)</td>
<td>~1μm</td>
<td>~1μm</td>
<td>1000ppm</td>
<td>Be/Yes</td>
<td>Routine specimen preparation, rapid.</td>
</tr>
<tr>
<td>EPMA/WDS</td>
<td>Electrons (0.5-50keV)</td>
<td>X-rays (&gt;100eV)</td>
<td>~1μm</td>
<td>~1μm</td>
<td>100ppm</td>
<td>Be/Yes</td>
<td>Quantitative analysis</td>
</tr>
<tr>
<td>AEM/ EDS</td>
<td>Electrons (100-400keV)</td>
<td>X-rays (&gt;100eV)</td>
<td>~5-10nm</td>
<td>10-100nm</td>
<td>1000ppm</td>
<td>B/Yes</td>
<td>Thickness TEM specimens, high spatial resolution</td>
</tr>
<tr>
<td>AES/ PEELS</td>
<td>Electrons (100-400keV)</td>
<td>Electrons (50eV-Eo)</td>
<td>~1nm</td>
<td>1-20nm</td>
<td>10-100ppm</td>
<td>Li/Yes</td>
<td>Very thin specimens, light element analysis</td>
</tr>
<tr>
<td>AES/ SAM</td>
<td>Electrons (1-3keV)</td>
<td>Electrons (&lt;200eV)</td>
<td>~50nm</td>
<td>~3nm</td>
<td>0.1-1at%</td>
<td>Li/yes</td>
<td>Quantitative surface analysis, depth profile</td>
</tr>
<tr>
<td>XPS</td>
<td>X-rays (1-1.5keV)</td>
<td>Electrons (&lt;10eV)</td>
<td>~1000μm</td>
<td>&lt;3nm</td>
<td>1000ppm</td>
<td>He/No</td>
<td>Chemical shift</td>
</tr>
<tr>
<td>SIMS</td>
<td>Ions (4-15keV)</td>
<td>Ions</td>
<td>~1μm</td>
<td>0.1nm</td>
<td>1ppb</td>
<td>H/Yes</td>
<td>Depth profile, best element sensitivity</td>
</tr>
<tr>
<td>PIXE</td>
<td>H⁺, H⁺⁺ (2-15meV)</td>
<td>X-rays</td>
<td>~2μm</td>
<td>10μm</td>
<td>1-100ppm</td>
<td>Na/Yes</td>
<td>Analytical sensitivity, depth profile</td>
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<tr>
<td>RBS</td>
<td>H⁺, H⁺⁺ (2-15meV)</td>
<td>H⁺, H⁺⁺</td>
<td>~1mm</td>
<td>10nm</td>
<td>1-1000ppm</td>
<td>Li/No</td>
<td>Non-destructive, depth profile</td>
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<tr>
<td>Micro IR</td>
<td>Infrared Light</td>
<td>IR light</td>
<td>~10μm</td>
<td>N/A</td>
<td>N/A</td>
<td>Yes</td>
<td>Molecular spectroscopy</td>
</tr>
</tbody>
</table>

(*) LEA (lightest element analyzed)
<table>
<thead>
<tr>
<th>Year</th>
<th>Event</th>
</tr>
</thead>
<tbody>
<tr>
<td>1931</td>
<td>The TEM (Transmission Electron Microscope) was the first type of electron microscope developed. It was developed by Max Knoll and Ernst Ruska in Germany. The “late” development of the SEM was due to the electronics involved in scanning the beam of electrons across the sample. Earliest recognized work describing the concept of an SEM: Knoll (1935) and von Ardenne (1938).</td>
</tr>
<tr>
<td>1960</td>
<td>Everhart and Thornley developed an improved SE detector.</td>
</tr>
<tr>
<td>1965</td>
<td>First commercial instrument came out.</td>
</tr>
<tr>
<td>1967</td>
<td>Electron-channeling contrast produced by crystal orientation and lattice interactions was observed.</td>
</tr>
</tbody>
</table>
Magnification = \frac{\text{Image Size}}{\text{Object Size}}

**Resolution:** The fineness of detail that can be distinguished in an image.

*Image Size* is the image of the screen (constant).

![SEM Image of Al₂O₃ / Ni Composite](image)

Al₂O₃ / Ni composite (by Dr. Sekino, Osaka Univ.)
Optical Microscopy

Image formation by an optical lens

\[ \text{Magnification}(M) = \frac{v}{u} \]

\[ \frac{1}{f} = \frac{1}{u} + \frac{1}{v} \]

The limits of observation by human eye are the following:

• Type of electromagnetic radiations that can be detected (visible light 0.4-0.7mm; maximum sensitivity is for green at 0.56mm).

• Minimum signal intensity required for recognition (within the integration time of 0.1sec). At least ~ 100 photons per picture element or pixel within the 0.1 sec time is required. Optical systems can integrate the image over a much longer period of time and can operate at much lower light levels.

• Minimum spatial separation which can be resolved ~ 0.2mm called the resolving power of our eye.

Figure 3.7 A simple ray diagram relates the distance of the lens from the object, \( u \), to both the focal length of the lens, \( f \), and the position of the image plane, \( v \), and determines the magnification in the image, \( M = v/u \)
Any instrument capable of revealing details finer than 0.2 mm is called a microscope. (Microscopes using electrons needs a viewing screen which translates electron intensity to light intensity.)

**Resolution**

Resolution of a lens is defined in terms of the spatial distribution of intensity which is observed through the lens at its focus for a point source at infinity. It is the smallest distance that can be resolved.

The width ($\delta$) of the first intensity peak for the image of a point object at infinity in terms of the angular aperture of the lens $\alpha$, refractive index $\mu$ and the wavelength of the radiation $\lambda$ is given by the **Abbe equation**

$$\delta = 0.61 \frac{\lambda}{\mu \cdot \sin \alpha}$$

For the cylindrically symmetric case, the ratio of the peak intensities for the primary and secondary peaks in the intensity distribution is 9:1

*Figure 3.8*  The Abbe equation gives the width of the first intensity peak for the image of a point object at infinity in terms of the angular aperture of the lens $\alpha$ and the wavelength of the radiation $\lambda$. 
• $\mu \cdot \sin \alpha$ is called \textit{numerical aperture (NA)}. Maximum values for a lens are \(~ 1.3\) for immersion lens and \(0.95\) for those operating in air.

• Larger aperture of the lens a will maximize $\sin \alpha$ and will improve resolution. This will collect much more information in the image.

• Resolution is also the ability of a lens to distinguish between two point sources at infinity when they are viewed in the image plane.

• Raleigh criterion for resolution was that the angular separation of the two sources of equal intensity should ensure that maximum of the primary image peak of one source should fall on the first minimum of the image of the second source. This will result in an intensity minimum of \(13\%\) at the center.

• A lens of magnification $M$ and resolution $\delta$ will produce images with resolvable features whose separation is of the order of $M\delta$. If $M\delta < \delta_{\text{eye}}$, human eye cannot resolve all the features.
The best possible resolution $\delta$ using an optical lens is 0.5 $\mu$m. Assuming a 0.2mm resolution for human eye, the maximum ‘useful’ magnification $M$ is only 400 (or in general, in the $10^3$ range). To see ultra small objects, more magnification $M$ is required – but for human eye to resolve all the features of the image, $\delta$ should be adequate small to keep $M\delta > \delta_{\text{eye}}$.

To improve resolution two options are possible:

1) Reduce $\lambda$: Use of shorter wavelength (higher energy) electromagnetic radiation is difficult due to strong absorption by the lenses, specimen and other components especially when $\lambda < 300 \text{nm}$. Image must be viewed on a screen, not directly with your eye! X-ray microscope remains as an unsuccessful goal.

$$\delta = 0.61 \frac{\lambda}{\mu \cdot \sin \alpha}$$

2) Increase $\mu$: For example, an oil immersion lens has an inert high refractive index liquid between the sample and the lens to increase the resolution.
**Optical Microscopy - Limitations**

The wave nature of light imposes fundamental limitations on the resolution of an optical system. In visible light optical microscopy, the resolving power of the optical system (the ability of the system to show points or edges clearly) is often described by the **Rayleigh criterion**.

A point source can be just resolved from a neighboring points source when it is separated from the other by the radius of the first zero of the diffraction limited Airy disk. For a self-luminous body (as in fluorescence microscopy) this distance, \( r \) is given by: (\( \mu \) is the index of refraction of the embedded medium and \( \theta \) is the acceptance angle of the objective lens, \( N.A. \) is the numerical aperture)

\[
r = 0.61 \frac{\lambda}{N.A.}
\]

\[
N.A. = \mu \times \sin \alpha
\]
Using a N.A. = 0.95 (highest in an optical system under immersion oil is 1.6)
The maximum resolution is 0.2 μm.
Decreasing the wavelength will improve the resolution: *electrons*

**Electron Beams**
Aperture \( \alpha \sim 10^{-1} – 10^{-3} \) degrees
Wavelength for 200 kV, \( \lambda \sim 0.0025 \text{ nm} \)
Refractive index \( \mu = 1 \) for vacuum
Resolution \( \delta \sim 0.02 \text{ nm}, 1/10\text{th of lattice spacing}!! \)
In principle, atomic level resolution is achievable.
**Depth of Field / Depth of Focus**

The *depth of field* \( (d) \) is a measure of how much of the object we are looking at remains ‘in focus’ at the same time. The distance over which the image remains in focus is called *depth of focus* \( (D) \). It is the *distance normal to the specimen* surface that is within acceptable focus when the microscope is precisely focused on the specimen surface.

\[ D = M^2 d = \pm \frac{\mu \lambda}{(NA)^2} \]

\( D \) is important when imaging non-flat samples. Example: Fractography

![Diagram](image)

**Figure 3.12** Since the resolution is finite, the object need not be in the exact object-plane position in order to remain in focus, and there is an allowed depth of field \( d \). Similarly, the image may be observed without loss of resolution if the image plane is slightly displaced, so that there is an allowed depth of focus \( D \).
Comparison: Wavelength of Photon vs. Electron

Say you have a photon and an electron, both with 1 eV of energy. Find the de Broglie wavelength of each.

- Photon with 1 eV energy:
  \[ E = \frac{hc}{\lambda} \Rightarrow \lambda = \frac{hc}{E} = \frac{1240 \text{ eV nm}}{1 \text{ eV}} = 1240 \text{ nm} \]

- Electron with 1 eV kinetic energy:
  \[ \text{KE} = \frac{1}{2}mv^2 \text{ and } p = mv, \text{ so } \text{KE} = \frac{p^2}{2m} \]
  \[ \lambda = \frac{h}{\sqrt{2m(\text{KE})}} = \frac{hc}{\sqrt{2mc^2}(\text{KE})} = \frac{1240 \text{ eV nm}}{\sqrt{2(511,000 \text{ eV})(1 \text{ eV})}} = 1.23 \text{ nm} \]

Equations are different - be careful!
Wavelength of Electrons

The wavelength of the electron can be tuned by changing the accelerating voltage.

de Broglie:

\[ \lambda = \frac{h}{mv} \]

\( \lambda \): wavelength associated with the particle
\( h \): Plank’s constant \( 6.63 \times 10^{-34} \) Js;
\( mv \): momentum of the particle
\( m_e = 9.1 \times 10^{-31} \) kg; \( e = 1.6 \times 10^{-19} \) coulomb

(for \( V \) in KV, \( \lambda \) in Å)

\( V \) of 1 eV, \( \lambda = 12.3 \) Å
\( V \) of 60 kV, \( \lambda = 0.05 \) Å \( \Rightarrow \Delta x \approx 2.5 \) Å
\( V \) of 100 kV, \( \lambda = 0.039 \) Å

Microscopes using electrons as illuminating radiation

TEM (commercially available up to 400 kV)
SEM (usually up to 40 kV)
<table>
<thead>
<tr>
<th>Optical Instrument</th>
<th>Resolving Power</th>
<th>RP in Angstroms</th>
</tr>
</thead>
<tbody>
<tr>
<td>Human eye</td>
<td>0.2 millimeters (mm)</td>
<td>2,000,000 A</td>
</tr>
<tr>
<td>Light microscope</td>
<td>0.20 micrometers (µm)</td>
<td>2000 A</td>
</tr>
<tr>
<td>Scanning electron microscope</td>
<td>5-10 nanometers (nm)</td>
<td>50-100 A</td>
</tr>
<tr>
<td>Transmission electron microscope</td>
<td>0.5 nanometers (nm)</td>
<td>5 A</td>
</tr>
</tbody>
</table>

**What are Electron Microscopes?** They are microscopes that uses electrons instead of light to form an image and to examine objects on a very fine scale.

**What is a Scanning Electron Microscope?** A microscope that uses electrons to examine the sample surface. This examination can yield the following information:

- **Topographical:** surface features
- **Morphology:** size and shape of surface features
- **Composition:** elements and compounds and the relative amount of them.
- **Crystallographic:** how the atoms are arranged in the sample.
Basic steps involved in all electron microscopes:

- A beam of electrons is formed (electron gun) and accelerated toward the specimen using a positive electrical potential.
- This beam is confined and focused using metal apertures and magnetic lenses into a thin, focused, monochromatic (all e-s have the same energy/wavelength) beam.
- This beam is focused onto the sample using a magnetic lens.
- Interactions occur between the beam of electrons and the sample inside the interaction volume.
- These interactions are detected and transformed into an image.

### Table 4.1. Area Sampled as a Function of Magnification$^a$

<table>
<thead>
<tr>
<th>Magnification</th>
<th>Area on sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>10X</td>
<td>(1 cm)$^2$</td>
</tr>
<tr>
<td>100X</td>
<td>(1 mm)$^2$</td>
</tr>
<tr>
<td>1,000X</td>
<td>(100 μm)$^2$</td>
</tr>
<tr>
<td>10,000X</td>
<td>(10 μm)$^2$</td>
</tr>
<tr>
<td>100,000X</td>
<td>(1 μm)$^2$</td>
</tr>
<tr>
<td>1,000,000X</td>
<td>(100 nm)$^2$</td>
</tr>
</tbody>
</table>

$^a$ Assumes CRT screen measures 10 cm × 10 cm.
### SEM vs. TEM

<table>
<thead>
<tr>
<th>SEM</th>
<th>TEM</th>
</tr>
</thead>
<tbody>
<tr>
<td>High resolution (1-10nm in SE mode and 10-1000nm in BSE mode).</td>
<td>High resolution (capable of atomic imaging 1.2-1.5 Angstroms)</td>
</tr>
<tr>
<td>Bulk specimens</td>
<td>Thin specimens</td>
</tr>
<tr>
<td>Large depth of field. 3-D appearance</td>
<td>Only 2-D appearance</td>
</tr>
<tr>
<td>Capable of low magnifications (complementary to optical microscopy)</td>
<td>Provides crystallographic and structural information (visualization of defects, lattice arrangements, orientation relationships, etc.)</td>
</tr>
</tbody>
</table>

#### SEM

<table>
<thead>
<tr>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Better resolution and depth of field than optical microscopes</td>
<td>Specimen under vacuum (it must be stable under vacuum)</td>
</tr>
<tr>
<td>Provides morphological and compositional information in small areas</td>
<td>Specimen must be conductive</td>
</tr>
<tr>
<td>Semi or non destructive technique</td>
<td>Specimen preparation can introduce artifacts</td>
</tr>
<tr>
<td>Relatively easy to use</td>
<td>Some visualization problems (up or down)</td>
</tr>
</tbody>
</table>
Electron microscopes were developed due to the limitation of the light microscopes. The physics of light limits the magnifications to x500 x1000 and the resolution of 0.2μm. Additionally in light microscopy high magnifications are accompanied by very low depth of field, i.e. 2-D images (flat samples).

**Characteristic Information obtained from a SEM**

**Topographic:** Surface features of an object, “how it looks”.

**Morphological:** The shape and size of the particles making up the object.

**Compositional:** Elements and compounds that the object is made of.

**Crystallographic:** How the atoms are arranged in 3-D.
The above information can be used to relate the properties of the materials with its microstructure.

Example: Fracture mode

The combination of large depth of field, higher magnification, greater resolution and compositional and crystallographic information makes the SEM one of the most heavily used instrument in research and semiconductor industry.
Basic Scanning Electron Microscope

1. Electron column
   - Electron gun
   - First condenser lens, C1
   - Second condenser lens, C2
   - Objective lens, O
   - Final aperture
   - X-ray detector (WDS or EDS)

2. Specimen chamber
   - Specimen

3. Vacuum pumping system
   - Diffusion or turbo-molecular pumps

4. Electronic control and imaging system
   - High voltage $V_0 \approx 20$ kV
   - Scan generator
   - Magnification control
   - Display CRT
   - Record CRT
   - Backscattered preamp
   - Secondary preamp
   - Specimen current preamp
   - Selection switch
   - Video amplifier
   - Camera
**Electron Column:** consists of an electron gun and two or more electron lenses, operating in a vacuum.

**Electron Gun:** produces a source of electrons and accelerates these electrons to an energy in the range 1-40 keV.

**Electron lenses** are used to reduce the diameter of this source of electrons and place a small, focused electron beam on the specimen and scan it.

**Working Distance (W):** The distance between the lower surface of the objective lens and the surface of the specimen is called working distance.

**Depth-of-Focus:** The capability of focusing features at different depth within the same image.

**Secondary Electron:** are electrons of the specimen ejected during inelastic scattering of the energetic beam electrons. It is used to produce topographical images with a high depth of focus.

**Backscattering Detector:** Collects the elastically scattered electrons and produces topographical and compositional images (contrast due to surface height changes and atomic number differences respectively).

**EDS and WDS Detector:** Collects the x-ray radiation emitted from the specimen. It gives the chemical composition of the specimen.

**Vacuum System:** produces a vacuum level acceptable for the operation of the electron gun and sample.
The image mode most commonly used is the "secondary electron image". Changes in grey intensity over the screen suggest the presence of "hills" and "valley" on the surface sample and these changes do correspond to the topography of the surface.
<table>
<thead>
<tr>
<th>Signal Type</th>
<th>Type of Signal Detected</th>
<th>Image Information</th>
<th>Image Resolution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Secondary</td>
<td>Secondary electrons</td>
<td>Topography</td>
<td>10nm</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Voltage Contrast</td>
<td>100nm</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Magnetic and electric fields</td>
<td>1μm</td>
</tr>
<tr>
<td>Backscattered</td>
<td>Backscattered electrons</td>
<td>Chemical</td>
<td>100nm</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Topographical</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Crystallographic</td>
<td></td>
</tr>
<tr>
<td>Cathodo-luminescence</td>
<td>Photons</td>
<td>Chemical</td>
<td>100nm</td>
</tr>
<tr>
<td>Absorption</td>
<td>Absorbed Current</td>
<td>Topography</td>
<td>1μm</td>
</tr>
<tr>
<td>X-Rays</td>
<td>Characteristic X-Rays</td>
<td>Chemical</td>
<td>1μm</td>
</tr>
<tr>
<td>Auger</td>
<td>Auger emitted electrons</td>
<td>Chemical</td>
<td>1μm</td>
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<tr>
<td>Transmission</td>
<td>Transmitted electrons</td>
<td>Crystallographic</td>
<td>1 – 10nm</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Internal defects</td>
<td></td>
</tr>
<tr>
<td>Conductive Current</td>
<td>Sample Induced Current</td>
<td>Induced current</td>
<td>100nm</td>
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