Scanning Electron Microscope (SEM)

Danny Porath 2003
Scanning Electron Microscope (SEM)

Radiolarian (in Plankton) x 750

(From IOWA U. web site)
With the help of........

1. Bruce Kahn - RIT
2. Yossi Rosenwacks
3. Yosi Shacam – TAU
4. JEOL guide for SEM
6. IOWA state U., Dept. of Material Science & Engineering site
7. …
Internet Sites

http://www.rit.edu/~bekpph/
http://www.rit.edu/~bekpph/sem/ARS/sem.htm
http://www.unl.edu/CMRAcfem/em.htm
http://www.jeol.com/sem_gde/tbcontd.html
http://mse.iastate.edu/microscopy/home.html
http://laser.phys.ualberta.ca/~egerton/SEM/sem.htm
http://acept.la.asu.edu/PiN/rdg/elmicr/elmicr.shtml
http://www.mos.org/sln/sem/seminfo.html
http://www.mih.unibas.ch/Booklet/Lecture/Chapter1/Chapter1.html
Homework 2

1. Find on the web, in a paper or in a book the 3 most impressive SEM and TEM images:
   a. 1 - Technically
   b. 1 - Scientifically
   c. 1 - Aesthetically

   Explain your choice. If needed compare with additional images.

3. Which types of analysis can be done by SEM/TEM beyond imaging. Explain shortly.

4. Be prepared to present each one of them to the class in 5 minutes.
Outline SEM/TEM:

1. Links and examples

2. Optics
   a. Ray diagrams
   b. Resolution
   c. Magnification

3. SEM/TEM structure

4. Electrons-surface interactions and signals

5. Types of disturbances
High resolution image of a frozen, hydrated yeast

Uncoated chromite
Transmission Electron Microscope (TEM) Image (Leo 922 OMEGA)

Tunnelling device on the basis of a Si/Ge heterostructure

Si[110] taken on LEO 922 Lattic spacings: [111] = 0.31nm, [200] = 0.27nm
**SEM Images** *(Leo 1530 and JEOL Guide to SEM)*

- **Toner x2,500**
- **Gold particles x36,000**
- **Integrated Circuit x720**
- **Eye of a fly x100**
- **Kosher Salt x75**
- **Toilet Paper x500**
SEM Images (Leo 1530 and JEOL Guide to SEM)

Black widow spider x500  Cucumber skin x350  Staple in paper x35

Big Radiolarian x500 and x2,000  Ceropia moth x350 and 15,000
SEM Imaging

Before Au$_{55}$ trapping

After Au$_{55}$ trapping

~4 nm gap
1611 Kepler suggested a way of making a compound microscope.

1655 Hooke used a compound microscope to describe small pores in sections of cork that he called "cells".

1674 Leeuwenhoek reported his discovery of protozoa. He saw bacteria for the first time 9 years later.

1833 Brown published his microscopic observations of orchids, clearly describing the cell nucleus.

1838 Schleiden and Schwann proposed the cell theory, stating that the nucleated cell is the unit of structure and function in plants and animals.

1857 Kolliker described the mitochondria in muscle cells.

1876 Abbé analyzed the effects of diffraction on image formation in the microscope and showed how to optimize microscope design.

1879 Flemming described with great clarity chromosome behavior during mitosis in animal cells.

1881 Retzius described many animal tissues with a detail that has not been surpassed by any other light microscopist. In the next two decades he, Cajal, and other histologists developed staining methods and laid the foundations of microscopic anatomy.
Some (more) History....

1882 Koch used aniline dyes to stain microorganisms and identified the bacteria that cause tuberculosis and cholera. In the following two decades, other bacteriologists, such as Klebs ans Pasteur, identified the causative agents of many other diseases by examining stained preparations under the microscope.

1886 Zeiss made a series of lenses, to the design of Abbé, that enabled microscopists to resolve structures at the theoretical limits of visible light.

1898 Golgi first saw and described the Golgi apparatus by staining cells with silver nitrate.

1924 Lacassagne and collaborators developed the first autoradiographic method to localize radioactive polonium in biological specimens.

1930 Lebedeff designed and built the first interference microscope.

1932 Zernike invented the phase-contrast microscope. These two developments allowed unstained living cells to be seen in detail for the first time.

1941 Coons used antibodies coupled to fluorescent dyes to detect cellular antigens.

1952 Nomarski devised and patented the system of differential interference contrast for the light microscope that still bears his name.

Optics
Image Through a Thin Lens

$\begin{align*}
d_1 &= \text{object distance} \\
d_2 &= \text{image distance} \\
f_1, f_2 &= \text{focal lengths} \\
e_1, e_2 &= \text{extrafocal distances} \\
h_1, h_2 &= \text{object/image heights}
\end{align*}$
Various Optical Ray Diagrams

1/f = 1/u + 1/v
M = f/(u-f) = (v-f)/f
Two lens System and Magnification

Objective

Projector

\[ M_1 = \frac{(v_1 - f_1)}{f_1} \]

\[ M_2 = \frac{(v_2 - f_2)}{f_2} \]

\[ M = \frac{(v_1 - f_1)(v_2 - f_2)}{f_1 f_2} \]
**Light Sources**

**Transmission illumination**
- TEM

**Reflected illumination**
- SEM
Spectral range
Resolution

The resolution depends on the lens ability to collect light (~1/f#) and inverse to the aperture number (NA)

\[
f/# = \frac{f}{D}
\]

\[n - \text{refractive index}\]

\[NA = n \sin(\alpha)\]

\[NA = \frac{1}{2 \times f/#}\]

\[\text{Resolution} = k_1 \frac{\lambda}{NA}\]
Resolution ... Airy Discs

Laser beam Diffraction through a pinhole

Rayleigh Resolution Criterion

\[ R_1 = \frac{d_1}{2} = 0.61\lambda / \text{nsin}(\alpha) = 0.61\lambda / \text{NA} \]
**Diffraction limited Resolution**

Thus the smallest separation is determined by the N.A. \((1/2f\#)\)

Typically the best objective has \(N.A \approx 1.6 \Rightarrow \text{resolution} \approx 170 \text{ nm}\)

For \(\lambda \sim 400 \text{ nm} \) (green light)

\(\Rightarrow \text{Decrease } \lambda\)
Electron Microscopy - Decreasing The Wavelength

\[ E = \frac{p^2}{2m} = eV \quad \text{Energy Conservation} \]

\[ p = \sqrt{2meV} \]

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

\[ \lambda = \frac{h}{p} = \frac{h}{\sqrt{2meV}} = \frac{6.6 \cdot 10^{-34}}{\sqrt{2 \cdot 9.1 \cdot 10^{-31} \cdot 1.6 \cdot 10^{-19} \cdot 50000}} \approx 0.05 \text{ Å} \]

Resolution (50 kV): \[ R_1 = 0.61\lambda/\text{NA} \sim (0.6 \cdot 0.05)/1.6 \sim 0.2 \text{ Å} \]
The Evolution of Resolution
Magnifications (YBCO)

- **SEM**
  - x70
  - x300
  - x1400
  - x2800

- **Optical**
**Depth of Focus**

Depth of focus, $h$, is the distance from the plane of optimum focus in which the beam diverges by no more than the spot diameter $d_1$.

$$h = 0.61\lambda/[n \sin(\alpha) \tan(\alpha)]$$

Depth of Field - the range of positions for the object for which our eye can detect no change in the sharpness of the image.
SEM Structure
SEM Operation

Magnification = length of TV screen/Scanning length
**SEM Operation**

![Diagram of SEM Operation]

*Fig. 1 Schematic illustration of a SEM. Each of the components shown on the figure is understood in some detail if optimum operation of the instrument is to be achieved.*
JEOL Optical System

Fig. 1 Schematic of JWS-7515 electron optical system.
SEM Ray Diagrams

Large WD:

- Demagnification decreases
- Spot size increases
- Divergence angle $\alpha$ decreased

The decrease in demagnification is obtained when the lens current is decreased, which in turn increases the focal length $f$ of the lens. The resolution of the specimen is decreased with an increased working distance, because the spot size is increased. Conversely, the depth of field is increased with an increased working distance, because the divergence angle is smaller.
Beam's Path through the Column
Light vs. Electron Microscopes

Light Microscope
- Light source
- Condenser lens
- Specimen
- Objective lens
- Eyepiece lens
- Image viewed directly

Transmission Electron Microscope
- Heated filament (source of electrons)
- Specimen
- Lens
- Image on fluorescent screen

Scanning Electron Microscope
- Beam deflector
- Detector
- Image on viewing screen
The Electron Source

The electron source:

Filament: Tungsten

This filament is a loop of tungsten which functions as the cathode. A voltage is applied to the loop, causing it to heat up. The anode, which is positive with respect to the filament, forms powerful attractive forces for electrons.
The Potential Distribution in the Tungsten Gun
**LaB$_6$ Gun**

- **LaB$_6$ cathodes**
  - Broers
    - resistive heating wire
    - cooled structural mount
  - Vogel
    - structural and direct electrical contact mounting
  - Ferris
    - structural and resistive heating ribbon

Heating current path through precision-machined, single-piece carbon rod and mounting strips; sub-base provides rigidity and easier mounting.
Field Emission Gun
Field Emission Gun

- Field Emission Tip
- First Anode
- Second Anode
- First Cross-Over

Diagram showing the components:

- Cold Cathode
- Extractive Voltage ($V_e$)
- Acceleration Voltage ($V_a$)
- First Anode
- Second Anode
- Electron Beam 100 Å
## Gun Types

### SEM Cathode Comparison

<table>
<thead>
<tr>
<th></th>
<th>Tungsten filament</th>
<th>LaB₆</th>
<th>Schottky (TF)</th>
<th>Field Emission</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Apparent Source Size</strong></td>
<td>100 micrometers</td>
<td>5 micrometers</td>
<td>&lt;100 Angstroms</td>
<td>&lt;100 Angstroms</td>
</tr>
<tr>
<td><strong>Brightness</strong></td>
<td>1 A/cm² steradian</td>
<td>20-50 A/cm² steradian</td>
<td>100-500 A/cm² steradian</td>
<td>100-1000 A/cm² steradian</td>
</tr>
<tr>
<td><strong>Vacuum Required</strong></td>
<td>10⁻⁵ Torr</td>
<td>10⁻⁶ Torr</td>
<td>10⁻⁸ Torr</td>
<td>10⁻⁹ Torr</td>
</tr>
</tbody>
</table>
Thermionic Emitter Materials

Relation between Heating Temperature and Electron Emission Density of Thermionic Cathode Materials

- LaB₆
- BaO
- ThO₂
- W
- Cb
- Ta
- Mo
- Th-W

Electron emission density (A/cm²)

Relation between Life and Brightness

- 1600°C
- 1500°C
- 1400°C
- 2500°C
- 2400°C
- 2300°C

Brightness (A/cm²·str)

JEOL Probe Current Control

Fig. 2 Wide-range probe current control.
The Magnetic Coils

Electron Beam

θ is a direction in the plane, $v_L$ is $\perp$ to the plane.

Nonaxial electrons will experience a force both down the axis and one radial to it. Only electrons traveling down the axis feel equal radial forces from all sides of the lens. The unequal force felt by the off-axis electrons causes spiralling about the optic axis.

Two components to the $B$ field:

- $B_L =$ longitudinal component (down the axis)
- $B_R =$ radial component (perpendicular to axis)

(Loretto H.M., "Electron Beam Analysis of Materials")
The Magnetic Lenses

coil
iron shroud

Electro-Magnetic Lens

Field Intensity

$H_z$ parallel to axis
$H_r$ perpendicular to axis

(“)???
The Scanning Coils

- Scan the sample (raster)
- Synchronized with CRT
- One pair of coils for X and one for Y
JEOL Heat Conductive System – TO Avoid Thermal Drift

Fig. 3 Schematic of a heat conductive system.

Fig. 5 Temperature step response at the objective lens cone tip and drift speed of secondary electron image when switching from 1 kV to 12 kV.
e-Surface interactions and Signals
Collected Signals in SEM

- Secondary electrons (SE)
- Backscattered electrons (BSE)
- X-rays
- Cathodoluminescence (CL)

Sample

Courtesy Z. Barkay
While all these signals are present in the SEM, not all of them are detected and used for information. The signals most commonly used are the **Secondary Electrons**, the **Backscattered Electrons** and **X-rays**.
Signal Emission from Interaction Volume
## Basic SEM Modes of Operation - Summary

<table>
<thead>
<tr>
<th>Signal/Mode</th>
<th>Information</th>
<th>Material</th>
<th>Resolution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Secondary electrons (SE)</td>
<td>Morphology</td>
<td>All (*)</td>
<td>~1nm</td>
</tr>
<tr>
<td>Backscattered electrons (BSE)</td>
<td>Atomic number</td>
<td>All (*)</td>
<td>0.1-0.5µm(**)</td>
</tr>
<tr>
<td>X-ray (EDS or WDS)</td>
<td>Atomic composition</td>
<td>All (flat)</td>
<td>~1µm</td>
</tr>
<tr>
<td>(CL)Cathodoluminescence</td>
<td>Bandgap, impurities, lifetimes</td>
<td>Insulators and semiconductors</td>
<td>~ 1µm</td>
</tr>
</tbody>
</table>

(*) usually sizes of 1cm, dependent on SEM configuration

(**) voltage and Z dependent

Additional modes: Voltage contrast (VC) and EBIC - usually used in devices and p-n junctions.

*Courtesy Z. Barkay*
Inelastic Scattering

During inelastic scattering, energy is transferred to the electrons surrounding the atoms and the kinetic energy of the energetic electron involved decreases. A single inelastic event can transfer a various amount of energy from the beam electron ranging from a fraction to many kilo-electron volts. The main processes include phonon excitation, plasmon excitation, secondary electron excitation, continuum X-ray generation, and ionization of inner shells. In all processes of inelastic scattering, energy is lost, though different processes lose energy at varying rates.
Secondary Electrons

1. Secondary electrons are predominantly produced by the interactions between energetic beam electrons and weakly bonded conduction-band electrons in metals or the valence electrons of insulators and semiconductors.

2. There is a great difference between the amount of energy contained by beam electrons compared to the specimen electrons and because of this, only a small amount of kinetic energy can be transferred to the secondary electrons.
Secondary Electrons and Detection

SE are specimen electrons that obtain energy by inelastic collisions with beam electrons. They are defined as electrons emitted from the specimen with energy less than 50 eV.

A broken surface of a piece of metal, formed using SE imaging.
Elastic Scattering – Backscattering

As the name implies, elastic scattering results in little (<1eV) or no change in energy of the scattered electron, although there is a change in momentum. Since momentum, \( p=mv \), and \( m \) doesn't change, the direction of the velocity vector must change. The angle of scattering can range from 0-180 degrees, with a typical value being about 5 degrees.

Elastic scattering occurs between the negative electron and the positive nucleus. This is essentially Rutherford scattering. Sometimes the angle is such that the electron comes back out of the sample. These are backscattered electrons.
Backscattering Detector

Elastic scattering occurs between the negative electron and the positive nucleus. This is essentially Rutherford scattering. Sometimes the angle is such that the electron comes back out of the sample. These are backscattered electrons.

Aluminum copper alloy formed using backscattered electron imaging.

The light area is mostly aluminum and the dark area is mostly copper.
Energy distribution of SE and BSE

Courtesy Z. Barkay
Detection

An electron detector is used with the SEM to convert the radiation of interest into an electrical signal for manipulation and display by signal processing electronics, which is much like a television. Most SEM's are equipped with an Everhart-Thornley (E-T) detector. It works in the following manner:

The scintillator material is struck by an energetic electron. This collision produces photons which are conducted by total internal reflection in a light guide to a photomultiplier. These photons are now in the form of light so they can pass through a vacuum environment and a quartz glass window. The photon is then converted back into an electron current where a positive bias can attract the electrons and collect them so that they will be detected.
E-T Electron Detector
When a SEM is used, the column must always be at a vacuum. There are many reasons for this. If the sample is in a gas filled environment, an electron beam cannot be generated or maintained because of a high instability in the beam. Gases could react with the electron source, causing it to burn out, or cause electrons in the beam to ionize, which produces random discharges and leads to instability in the beam. The transmission of the beam through the electron optic column would also be hindered by the presence of other molecules. Those other molecules, which could come from the sample or the microscope itself, could form compounds and condense on the sample. This would lower the contrast and obscure detail in the image.

A vacuum environment is also necessary in part of the sample preparation. One such example is the sputter coater. If the chamber isn't at vacuum before the sample is coated, gas molecules would get in the way of the argon and gold. This could lead to uneven coating, or no coating at all.
The Objective Lens

- The lens that focuses the beam of electrons towards the sample.
- The SE detector produces a clear and focused topographical image of the sample.
- The BSE detector is used to determine the composition of the sample. Each element in the sample appears as a different shade, from almost white to black.
The left photo shows the sample chamber located at the base of the column. The right photo shows the lens and detectors located inside the sample chamber.
A prepared sample is mounted on a specimen stub and placed on the stage.
The Sputter Coater

The sputter coater is used to coat non-metallic samples (bugs, plants, human hair, etc.) with a thin layer of gold. This makes them conductive, and ready to be viewed by the SEM. If the samples are metallic, they can simply be mounted and placed in the SEM.
Environmental SEM

ESEM enables to view specimens and processes in their natural state in a gaseous environment.
How Does ESEM Work?

The primary electron beam hits the specimen which causes the specimen to emit secondary electrons. The electrons are attracted to the positively charged detector electrode. As they travel through the gaseous environment, collisions occur between an electron and a gas particle results in emission of more electrons and ionization of the gas molecules. This increase in the amount of electrons effectively amplifies the original secondary electron signal. The positively charged gas ions are attracted to the negatively biased specimen and offset charging takes effect.

As the number of secondary electrons varies the amplification effect of the gas varies. If a large number of electrons are emitted from a position on the specimen during a scan, there is a high signal. If only a small amount of electrons are emitted the signal is less intense. The difference in signal intensity from different locations on the specimen allows an image to be formed.
How Does ESEM Work?
Disturbances
Types of Imaging Disturbances

Image disturbances can be classified by the following expressions:

- Chromatic aberrations
- Images lacking sharpness and contrast
- Unstable images
- Generally poor-quality images
- Noisy images
- Images showing jagged edges
- Unusual-contrast images
- Distorted or deformed images.
**Aberrations**

**Chromatic**

\[
\frac{1}{r_{opt}} = 0.67 \frac{\lambda}{\alpha^2}
\]

**Spherical**

\[
\begin{align*}
0.61 & = \frac{\lambda}{\alpha} \quad r_1 = \frac{\lambda}{\alpha} \\
C_s \alpha^3 & = r_2 \\
\Rightarrow r = r_1 + r_2
\end{align*}
\]

**Optimized**

\[
\begin{align*}
\alpha_{opt} & = 0.67 \frac{\lambda^{1/4} C_s^{-1/4}}{} \\
r_{opt} & = 1.21 \frac{\lambda^{3/4} C_s^{1/4}}{} \\
h & = \frac{0.61}{\alpha^2}
\end{align*}
\]
Image Disturbances and Their Causes

Lack of sharpness
- Improper accelerating voltage setting
- Instability of gun emission caused by insufficient heating of filament
- Improper electron probe diameter
- Improper setting and incorrect centering of objective aperture
- Insufficient astigmatism correction
- Improper focal depth
- Too large magnification
- Specimen charge-up and magnetization
- Defocus of camera system

Low image quality
- Improper accelerating voltage setting
- Improper probe current setting
- Incorrect astigmatism correction
- Noise caused by excessive photomultiplier (PMT) gain
- Improper contrast and brightness
- Improper specimen preparation process
- Improper photographic material
- Improper positional relation between specimen and detector
- No specimen tilting

Noises
- Instability of accelerating voltage and gun emission
- Discharge of detector
- Charge-up of specimen surface
- Burnt CRT or dusty CRT screen
- External stray magnetic field
- Mechanical vibration

Image distortion and deformation
- Specimen charge-up
- External stray magnetic field
- Electron beam damage
- Deformation of specimen itself during its preparation
- Image drift caused by column interior charge-up
- Specimen drift on heating and cooling stages
Image Changes Caused by Interactions Between Electron Probe and Specimen

Influence of accelerating voltage on image quality:

Fig. 1  Diffusion of incident electrons (after Ducumb and Shields).

Fig. 2  Effect of accelerating voltage.
When high accelerating voltage is used as at (a), it is hard to obtain the contrast of the specimen surface structure. Besides, the specimen surface is easily charged up. The surface microstructures are easily seen at (b).
LEFT: The image sharpness and resolution are better at the higher accelerating voltage, 25 kV.

RIGHT: At 5 kV, the microstructures of the specimen surface are clearly seen as the penetration and diffusion area of incident electrons is shallow.
The smaller the electron probe diameter on the specimen, the higher the magnification and resolution. However, the image smoothness, namely, the S/N ratio depends on the probe current. Namely, as the probe diameter is reduced, the probe current is reduced.

It is therefore necessary to select a probe current suited for the magnification and observation conditions (accelerating voltage, specimen tilt, etc.) and the specimen.
The smaller the probe current, the sharper is the image, but the surface smoothness is lost.
The higher the accelerating voltage, the greater is the edge effect, making the edges brighter.
Specimen Tilt

Specimen tilt is aimed at:

• Improving the quality of secondary electron images

• Obtaining information different from that obtained when the specimen is not tilted, that is, observing topographic features and observing specimen sides.

• Obtaining stereo micrographs.
Detector position and specimen direction

Fiber

Specimen chamber

Secondary electron detector
Composition of Signals

Fig. 17 Secondary electron detector

Fig. 18. Backscattered electron detector

Fig. 19. Principles of composition image and topography image
Composition of Signals - Slug 20 kV, x1,100

- BSE
- SE
- Topography
- X-ray (Si)
- X-ray (Al)
Influence of Charge-Up on Image Quality

1) Probe current, 2) Accelerating voltage, 3) Tilting angle
Contamination

1) Dry and clean sample, 2) Low temp, 3) Small samples
Effect of Working Distance and Aperture Size

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

Working Distance

- Small
- Large

Large current (BEI X-ray analysis) → Low resolution
- Smaller depth of field

Aperature size

- Large
- Small

Grainy image
- Greater depth of field
Formation of **astigmatism** for a lens with slightly different optical properties in the horizontal and vertical directions
Astigmatism

Wrong

Correct

(a) Shape changes in electron beam when there is astigmatism

(b) Shape changes in electron beam when astigmatism is corrected
Brightness and Contrast

- **Contrast**
  - (1) Excessive contrast
  - (2) Insufficient brightness
  - (3) Optimum contrast and brightness
  - (4) Excessive brightness
  - (5) Insufficient contrast

- **Brightness**
  - Pollen of marigold x360
X-ray Exposure and Composition

How to decide exposure time

\[
\text{Exposure time (s)} = 2500 \text{ (C/cm}^2\text{)} \times \frac{\text{Picture elements (cm}^2\text{)}}{\text{X-ray count rate (CPS)}}
\]

(a) Composition image (COMPO)

(b) X-ray image CA 50 seconds

(c) X-ray image Ca 300 seconds

50 sec

300 sec
External Disturbances

Magnetic Field

(a) Influenced by external magnetic field.
(b) Influenced by external magnetic field

Mechanical vibration

(a) Influenced by mechanical vibration
(b) Uninfluenced by mechanical vibration
**Image Distortion**

Barrel – Magnification decreases with distance from optical axis

Pin-cushion – Magnification increases with distance from optical axis
Image Distortion

Latex particles

(a) Normal Image

(b) Horizontally distorted image
Summary

What you see.....

Is not necessarily what you get!!!

Be careful with images...