Scanning Electron Microscopy (SEM)
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Invented by Ernst Ruska, a German physicist (1933)

He was awarded the Nobel Prize for physics for his invention in 1986

History

First SEM – debuted in 1938 by Manfred Von Ardenne

In 1965, Cambridge Scientific Instrument (UK) & JOEL (Japan) first commercialized SEM individually
Light microscopes

Photons

Photons are substituted with electrons

Glass lenses are substituted with electromagnetic and electrostatic lenses

Electron microscope

Electrons

$e^-$
COMPARISON OF THE SIZE OF OBJECTS
Electron microscopes are scientific instruments that use a beam of highly energetic electrons to examine objects on a very fine scale.

Applications of SEM
1. Topography: the surface features of the sample
2. Morphology: the shape and size of the sample
3. Composition: the elements that the sample is composed of and their relative amount (％) – purity
4. Crystallographic info: how atoms are arrange in the sample
Budding yeast cell

Compound microscope image

TEM image

SEM image

E. coli bacteria

Compound microscope image

TEM image

SEM image
FEI Nova NanoSEM 230 FE-SEM

DETERCTORS: BSE, TLD, ETD, low vac, helix
EDX: Oxford
The instrument
PARTS OF SEM

1. Electron cannon.

2. Chamber of vacuum system

3. Vacuum pumps system

4. Magnet

5. Operation panel with focus, alignment and magnification tools and a joystick for positioning

6. Screen for menu and image display

7. Cryo-unit to prepare frozen material before insertion in the observation chamber in Cryo-SEM mode
- Electron gun consisting of cathode and anode.
- The condenser lens controls the amount of electrons travelling down the column.
- The objective lens focuses the beam into a spot on the sample.
- Deflection coil helps to deflect the electron beam.
- SED attracts the secondary electrons.
- Additional sensors detect backscattered electrons and X-rays.
Image formation

Electrons in

Electrons out

or: x-rays out

We shoot high-energy electrons and analyze the outgoing electrons/x-rays
How do we get an image?

Sample

Area of the sample scan

Signal/image formed
So how does a SEM change the magnification of an image?

- By reducing the size of the area scanned by the scan coils, the SEM changes the magnification of the image.
● This form of image processing is only in grayscale which is why SEM images are always in black and white.

● These images can be colorized through the use of feature-detection software, or simply by hand editing using a hand graphic editor.

● This is usually for aesthetic effects, for clarifying structure, or for adding a realistic effect to the sample.
SEM micrograph of a chloroplast in maize (Zea mays) showing thylakoids (green) and assimilation starch granules (grey). (Prepared by freeze fracturing; micrograph is pseudo-colored.) (Source: G. Wanner LMU)
SE = from the surface of the sample

BSE= from the surface of the sample and from deeper within the sample

BSE are reflected electron

The image in SEM is mapping out the density of the sample surface

BSE that get trapped in the sample caused the x-ray to be given off
Electron beam-sample interactions

- The incident electron beam is scattered in the sample, both elastically and inelastically.
- This gives rise to various signals that we can detect.
- Interaction volume increases with increasing acceleration voltage and decreases with increasing atomic number.
Backscattered electrons (BSE)

• A fraction of the incident electrons is retarded by the electro-magnetic field of the nucleus and if the scattering angle is greater than 180 °C the electron can escape from the surface
• High energy electrons (elastic scattering)
• Fewer BSE than SE
Factors that affect BSE emission

- Direction of the irritated surface
  - more electrons will hit the BSE detector when the surface is aligned towards the BSE detector

- Average atomic number (Z)

- When you want to study differences in atomic numbers the sample should be as levelled as possible (sample preparation is an issue!)
SE and BSE SEM images of triangular precipitates at grain boundary of SnMnNb 0.05 sample.

BSE can observe that the material consists of 2 distinct phases; the bright grains being composed by a phase with average atomic number higher than the dark triangular precipitates.
X-rays

- *Photons* not electrons
- Each element has a *fingerprint* X-ray signal
- Poorer spatial resolution than BSE and SE
- Relatively few X-ray signals are emitted and the detector is inefficient
  → relatively long signal collecting times are needed
X-rays

• Most common spectrometer: EDS (energy-dispersive spectrometer)
• Signal overlap *can* be a problem
• We can analyze our sample in different modes
  – spot analysis
  – line scan
  – chemical concentration map
    (elemental mapping)
Electron guns

• We want many electrons per time unit per area (high current density) and as small electron spot as possible

• Traditional guns: thermionic electron gun (electrons are emitted when a solid is heated)
  – Wewire, LaB$_6$-crystal

• Modern: field emission guns (FEG) (cold guns, a strong electric field is used to extract electrons)
  – Single crystal of W, etched to a thin tip

FEG – gives FESEM
Electron guns

• Using field emission guns (FEG) - we get a smaller spot and higher current densities compared to thermionic guns
• But we need better vacuum for a FEG
Detectors

Backscattered electron (BSE) detector (Solid-State Detector)

Secondary electron (SE) detector: (Everhart-Thornley)
Traditional detectors

- Secondary electrons (SE): Everhart-Thornley Detector (ETD)
- Backscattered electrons (BSE): Solid State Detector
- X-rays: Energy dispersive spectrometer (EDS)
Vacuum

• The electron beam generated by the electron gun would encounter interference from air particles in the atmosphere.

• these particles block the path of the electron beam, they would also be knocked out of the air and onto the specimen, which would distort the surface of the specimen.

Vacuum requirements is dependant of the type of detector

• Chemical (corrosion) and thermal stability is necessary for a well-functioning filament (gun pressure)
  – A field emission gun requires $\sim 10^{-10}$ Torr
  – LaB$_6$: $\sim 10^{-6}$ Torr
Charge-up is a phenomenon where the negative charge of the incident electron beam accumulates (charged up) on the surface of a non-conductive specimen and the potential in the beam-incident area changes to cause various image troubles. The charge-up phenomenon occurs when the total number of electrons emitted from a specimen, or backscattered electrons ($I_{BSE}$), absorbed electrons ($I_{ab}$) and secondary electrons ($I_{SE}$) is not equal to the number of incident electrons ($I_p$), as shown in Fig. 9.
Fig. 11  Example of Image Disturbance due to Charge-up (Photo a)
CHARGE-UP: can be prevented by coating the non-conductor sample with metal (conductor) – allowing the charge on the sample surface go to ground through the coated conductive film.
SEM SAMPLE PREPARATION

Coating the specimen

- To increase the conductivity of the specimen and to prevent the high voltage charge on the specimen.
- Coated with thin layer i.e., 20nm-30nm of conductive metal.
- All metals are conductive and require no preparation before being used.
SEM SAMPLE PREPARATION

Coating the specimen
- Non-metals need to be made conductive
- Done by using a device called a "sputter coater."

Conductive materials
- Gold
- Gold-palladium Alloy
- Platinum
- Osmium
- Iridium
- Tungsten
- Chromium
- Graphite
“Sputter Coater”
SEM SAMPLE PREPARATION

Appropriate size samples --- fit in the specimen chamber
Mounted rigidly on a specimen holder---specimen stub
SEM SAMPLE PREPARATION

For imaging in the SEM, specimens must be:

- Electrically conductive
- Electrically grounded
SEM SAMPLE PREPARATION

1. Cleaning the surface of the specimen
2. Stabilizing the specimen
3. Rinsing the specimen
4. Dehydrating the specimen
5. Drying the specimen
6. Mounting the specimen
7. Coating the specimen
SEM SAMPLE PREPARATION

Cleaning the surface of the specimen

- Very important
- Surface contains many unwanted deposits, such as dust, mud, soil etc
SEM SAMPLE PREPARATION

Dehydrating the specimen

- Water must be removed
- Air-drying causes collapse and shrinkage, this is commonly achieved by replacement of water in the cells with organic solvents such as ethanol or acetone.
- Dehydration -- performed with a graded series of ethanol or acetone.
SEM SAMPLE PREPARATION

Drying the specimen

- Specimen should be completely dry
- Otherwise the sample will be destroyed
SEM SAMPLE PREPARATION

Mounting the specimen

- Specimen has to be mounted on the holder
- Mounted rigidly on a specimen holder called a specimen stub
- Dry specimen -- mounted on a specimen stub using an adhesive such as epoxy resin or electrically conductive double-sided adhesive tape.
FESEM for samples prepared at various St/MMAs = 2-5

Topography and morphology
Shape-stabilised n-octadecane-AC-nanocomposites - 3D

Activated carbon derived from peat soil as a framework for the preparation of shape-stabilized phase change material, T Khadiran, MZ Hussein, Z Zainal, R Rusli, Energy 2015 (82), 468-478.
Shape-stabilised n-octadecane-AC-nanocomposites

Palm Kernel Shell Activated Carbon as an Inorganic Framework for Shape-Stabilized Phase Change Material
Nicholas et al., Nanomaterials 2018, 8, 689
Surface morphology of the samples before (left) and after (right) immersion in PBS for 14 days.

The surface of the nanocomposite was covered by an apatite layer.

Inset showing the surface of the nanocomposite covered by apatite layer, at a magnification of 2500×.
**SEM Advantages**

- Gives detailed 3D and topographical imaging and the versatile information garnered from different detectors.
- Works very fast.
- Modern SEMs allow for the generation of data in digital form.
- Most SEM samples require minimal preparation actions.
SEM Disadvantages

- SEMs are expensive and large.
- Special training is required to operate an SEM.
- Preparation of samples can result in artifacts.
- Limited to solid samples.
- Carry a small risk of radiation exposure associated with the electrons that scatter from beneath the sample surface.
Environmental SEM: ESEM

• Traditional SEM chamber pressure: ~ 10^{-6} Torr
• ESEM: 0.08 – 30 Torr
• Various gases can be used
• Requires different SE detector
Why ESEM?

• To image challenging samples such as:
  – insulating samples
  – vacuum-sensitive samples (e.g. biological samples)
  – irradiation-sensitive samples (e.g. thin organic films)
  – “wet” samples (oily, dirty, greasy)

• To study and image chemical and physical processes in-situ such as:
  – mechanical stress-testing
  – oxidation of metals
  – hydration/dehydration (e.g. watching paint dry)
Resolution

• Best resolution that can be obtained: size of the electron spot on the sample surface
  – The introduction of FEG has dramatically improved the resolution of SEM’s

• The volume from which the signal electrons are formed defines the resolution
  – SE image has higher resolution than a BSE image

• Scanning speed:
  – a weak signal requires slow speed to improve signal-to-noise ratio
  – when doing a slow scan drift in the electron beam can affect the accuracy of the analysis
Energy Dispersive X-Ray Microanalysis (EDX/EDS)
An EDX spectrum was collected at each point identified in the top secondary electron image to individually characterize the wires. The resulting spectra show that the largest diameter wire (spot 1) is Ni, the medium diameter wire (spot 2) is Al, and the thinnest diameter wire (spot 3) is Ti. Note that a small amount of Al smeared onto the other wire surfaces when these wires were wound together.
x-ray mapping of elements.

The original secondary electron image of the three twisted wires is shown again, this time overlaid with the color-coded element maps corresponding to the different wires shown in this field of view. The individual element maps are also shown.
Summary

• SEM is a versatile instrument for many applications especially for the study of surface morphology.
• To get a micrograph, an electron probe is scanned across the surface of the sample and detectors interpret the signal as a function of time.
• A resolution of 1 – 2 nm can be obtained when operated in a high resolution setup.
• The introduction of ESEM and the field emission gun have simplified the imaging of challenging samples.
Summary

- **Signals:**
  - **Secondary electrons (SE):** mainly topography
    - Low energy electrons, high resolution
    - Surface signal dependent on curvature
  - **Backscattered electrons (BSE):** mainly chemistry
    - High energy electrons
    - “Bulk” signal dependent on atomic number
  - **X-rays (EDX):** chemistry
    - microanalysis
Take home keywords

- SEM, ESEM and FESEM
- Topography, morphology, composition
- Instrumentation/components: Electron gun, lens (condenser and objective), deflection coil, sensors/detectors, TLD, ETD (Everhart-Thornley) for SE, BSE, etc.
- Formation of an image: scanning of electron
- Electron beam-sample interactions: SE, BSE and x-ray
- Energy Dispersive X-Ray Microanalysis (EDX / EDS), elemental mapping
- Resolution
- Sample preparation