

SCANNING ELECTRON MICROSCOPY (SEM)

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Introduction to Electron Microscopy; Scanning Electron Microscopy (SEM)



OUTLINE

1. BRIEF INTRODUCTION TO ELECTRON MICROSCOPY

- 2. THEORY OF SCANNING ELECTRON MICROSCOPE (SEM)
- **3. SEM SPECIMEN PREPARATIONS**
- 4. CHARACTERIZATION AND INTERPRETATION OF SEM AND SEM-EDS RESULTS

Why should we use electron microscope?

Wavelength of light and electron

	Species of light	Wavelength (λ , nm)				
	Electric wave	10 ⁶ ~10 ¹²				
LIGHT	Ultra red light	10 ³ ~10 ⁵				
	Visible light	390~760				
	Ultraviolet light	13~390				
ELECTROMAGNETIC	X-ray	0.05~10				
WAVE	γ -ray	0.005~0.1				
	Accelerating voltage	Wavelength of electron (λ, nm)				
ELECTRON is	20 kV	0.00859				
dependent on the	120 kV	0.00335				
accelerating voltage	200 kV	0.00251				
	1000 kV	0 00087				

The difference between optical and electron microscope



Examples



Martensite structures



Small depth of field Low resolution Large depth of field High resolution

Images of bacteria

SEM AND TEM INSTRUMENS



Typical Conventional SEM







Scanning Electron Microscope









Typical TEM



HVEM JEM-1300NEF





An electron microscope cannot see the wood for the tree.

An electron microscope is just for seeing trees.

Images

BFI (bright field image) DFI (dark field image) <u>Magnified image</u> Observation microstructure/ nanostructure (morphology of grain, grain size and distribution, the presence of second phase, dislocations, stacking fault etc.), high resolution image/lattice image.

Electron diffraction patterns

- Crystal structure

TEM

- Crystalline or amorphous
- Monocrystalline or polycrystalline
- One phase or more present in the specimen
- Orientation of specimen or individual grain, twin grains, epitaxial relationship





Electron microscopes have a range of disadvantages :

- 1. They are costly.
- 2. Sample preparation is often much more elaborate. It is often necessary to coat the specimen with a very thin layer of metal, such as gold (Au) and Platinum (Pt). The metal can reflect the electrons.
- 3. The sample must be completely dry. This makes it impossible to observe living specimens.
- 4. It is impossible to observe moving specimens (they are dead).
- 5. It is impossible to observe the color. Electrons do not possess a color.
- 6. The image is only black/white. Sometimes the image is colored artificially to give a better visual impression.
- 7. They require more training and experience in identifying artifacts that may have been introduced during the sample preparation process.
- 8. The energy of the electron beam is very high. Therefore, the sample is exposed to high radiation and cannot be used for living organisms.
- 9. The space requirements are high. They may need a whole room.
- 10. Maintenance costs are high.

SEM (Theory and Analysis)

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens.

The signals that derive from **electron and specimen interactions** reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample.



Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM techniques (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm).

The SEM is also capable of performing analyses of selected point locations on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions (using **EDS**), crystalline structure, and crystal orientations (using **EBSD**, **electron backscatter diffraction**). The design and function of the SEM is very similar to the **EPMA (electron probe microanalysis)** and considerable overlap in capabilities exists between the two instruments.



The penetration of the beam into the mass of the specimen is determined by basically 4 parameters: beam current, spot size, <u>accelerating voltage</u> and <u>atomic number of the specimen</u>.



Fundamental Principles of Scanning Electron Microscopy (SEM)

Secondary electrons and backscattered electrons are commonly used for imaging samples. **Secondary electrons** are most valuable for showing morphology and topography of the specimen. **Backscattered electrons** are most valuable for illustrating contrasts in composition in multiphase samples (i.e., rapid phase discrimination).

Emission of secondary electrons occurs when incident electrons penetrate a specimen and are then inelastically scattered.

A free electron in the specimen may receive an electrostatic force (Coulomb force) from the incident electron or scattered electron and thereby part of the latter's energy and then jump out into the vacuum. This electron is called **a secondary electron**.

However, the energy received by free electron is as small as a few ten eV maxima. Therefore, it has been thought that only the secondary electron produced within a depth range about 10 nm below the specimen surface can escape into the vacuum.



Electron scattering in a specimen is classified into two types;

1. **elastic scattering** in which incident electron is scattered at a large angle with almost no loss in energy.

2. **Inelastic scattering** in which incident electron loose energy but are scattered at a small angle. The elastically scattered electrons having approximately the same energy as the incident electrons are scattered from the vicinity of specimen surface into the vacuum, while inelastically scattered electrons which have lost energy substantially is scattered from a comparatively deep location in the specimen into the vacuum.



Inelastic collisions of the incident electrons produce <u>X-ray generation</u> with electrons in discrete orbitals (shells) of atoms in the sample. As the excited electrons return to lower energy states, they yield X-rays of a fixed wavelength (related to the difference in energy levels of electrons in different shells for a given element).

Thus, characteristic X-rays are produced for each element in a mineral "excited" by the electron beam. SEM analysis is considered "non-destructive"; that is, x-rays generated by electron interactions do not lead to volume loss of the sample, so it is possible to analyze the same materials repeatedly.









EDS Principle

Energy Dispersive X-ray Spectroscopy (EDS or EDX) is a qualitative and quantitative X-ray microanalytical technique that provides information on the chemical composition of a sample for elements with atomic number (Z)

Characteristic X-ray Generation

The atoms are ionized by the primary electron beam leading to holes generated on the core shells; following ionization the electrons from outer shells fill the holes and cause the emission of X-ray fluorescence lines.

<u>The characteristic X-ray</u> lines are named according to the shell in which the initial vacancy occurs and the shell from which an electron drops to fill that vacancy.

For instance, if the initial vacancy occurs in the K shell and the vacancy filling electron drops from the adjacent (*L*) shell, a K_{α} x-ray is emitted. If the electron drops from the *M* shell (two shells away), the emitted x-ray is a K_{β} x-ray. Similarly, if an *L*-shell electron is ejected and an electron from the *M*-shell fills the vacancy, L_{α} radiation will be emitted.

Light Elements

Light Elements are difficult to measure as valence electrons are involved in characteristic x-ray production and chemistry.

For heavy elements (>= Na) the Ka x-ray is not associated with chemical bonds.



Light elements (Z < 11) cannot be routinely analysed by EDS.

Hydrogen (Z = 1) and He (Z = 2) do not have Characteristic X-rays, and the Li (Z = 3) K X-rays are of too low energy to be detected by EDS.

H	Periodic Table of the Elements © www.elementsdatabase.com											2 He					
Li 3	Be	 hydrogen alkali metals alkali earth metals 					 poor metals nonmetals noble gases 					B	C	N ⁷	08	F	¹⁰ Ne
11 Na	12 Mg	12 Itransition metals Tare earth metals							13 Al	14 Si	15 P	16 S	17 Cl	18 Ar			
19 K	20 Ca	SC SC	22 Ti	V ²³	Cr ²⁴	25 Mn	²⁵ 26 27 28 29 30 n Fe Co Ni Cu Zn					Ga 31	Ge ³²	33 As	34 Se	35 Br	36 Kr
87 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 TC	44 Ru	⁴⁵ Rh	Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	Te Te	53 	Xe
Cs	Ba	57 La	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	Pt	79 Au	Hg	81 TI	⁸² Pb	83 Bi	⁸⁴ Po	At 85	86 Rn
B7 Fr	⁸⁸ Ra	AC	Unq	Unp	Unh	107 Uns	108 Uno	Une	Unn								

Ce	Pr	Nd	Pm	62 Sm	Eu	Gd ⁶⁴	Tb	66 Dy	67 Ho	Er	69 Tm	Yb	⁷¹ Lu
90	91	92	93	94	Am	96	97	98	99	100	101	102	103
Th	Pa	U	Np	Pu		Cm	Bk	Cf	Es	Fm	Md	No	Lr

Examples

BSE analysis: COMPOSITION

SE analysis: TOPOGRAPHY



Heavy elements (high atomic number) \rightarrow backscatter electrons more strongly than light elements (low atomic number) \rightarrow appear brighter in the image



All the elements have different-sized nuclei. As the size of the atom nucleus increases, the number of BSE increases. Thus, BSE can be used to get an image showing the different elements present in a sample.



Schematic cross-section of SEM

Components of the SEM



Electrons emitted by the gun are accelerated typically by 20 kV.

They pass through condenser and objective lenses, and then through a set of scan coils and an aperture. A scan is simultaneously generated on a computer monitor.

Electrons emitted by the specimen are detected, amplified and the signal is then used to produce an image.

ELECTRON GUN



Schematic illustration of electron paths from a thermionic electron gun

Thermionic source (from heating)



Tungsten filament



LaB₆ filament tip



W field-emission 200 µm filament

Schematic illustration of electron paths from a field emission gun

Field-emission source (from electric field)

Comparison of three types of source operating at 100 kV

	Units	Tungsten	LaB ₆	Field Emission
Work function, Φ	eV	4.5	2.4	4.5
Richardson's constant	A/m ² K ²	6×10^{5}	4×10^{5}	
Operating temperature	K	2700	1700	300
Current density	A/m ²	5×10^{4}	106	1010
Crossover size	μm	50	10	<0.01
Brightness	A/m ² sr	109	5×10^{10}	1013 -
Energy spread	eV	3	1.5	0.3
Emission current stability	%/hr	<1	<1	5
Vacuum	Pa	10-2	10-4	10-8
Lifetime	hr	100	500	>1000









Vacuum system

Category of vacuum (1 Torr \approx 130 Pa, 1 Pa= 7.5x10⁻³ Torr)

- Rough vacuum: 100 to 0.1 Pa (~1 to 10⁻³ Torr)
- Low vacuum: 0.1 10⁻⁴ Pa (~10⁻³ 10⁻⁶ Torr)
- High vacuum: 10-4 10-7 Pa (~10-6 10-9 Torr)
- Ultra High vacuum: if the pressure is lower than 10⁻⁷ Pa (~ 10⁻⁹ Torr)

All electron microscopes are operated under vacuum:

-To keep specimen clean (less contamination)
-To not reduce lifetime of filament
-To avoid contaminant in the column
-To improve image resolution

Resolution in the SEM

- The resolution of the SEM is determined by the size of the incident beam. It can be reduced by introducing an aperture into the beam path and reducing the probe size using the condenser lens. Note that reducing the probe size using the condenser lens reduces the beam current. Therefore, as you reduce the probe size, you eventually reach a point where imaging is impossible. For a typical SEM operating at 20 kV, the minimum usable probe size is 1 3 nm.
 - The resolution also depends on accelerating voltage. Higher energy electrons experience less spherical aberration when they pass through the lenses. It is also improved by reducing the working distance, up to a certain point. Beyond that point, the lenses may not be able to focus the beam on the sample.

As already noted, <u>images obtained with backscattered electrons have a</u> <u>lower resolution than images obtained with secondary electrons, because</u> <u>they originate from deeper within the specimen</u>.

Depth of field 1

Depth of field is the distance above and below the plane of optimum focus within which the image is in focus.

In the diagram on the right, *d* represents the diameter of the electron beam at the specimen. The depth of field is *h* since it makes no difference to the sharpness of the image if the object is anywhere within the range *h*.



Reducing the angle α increases the depth of field. It can be achieved by using a smaller aperture or increasing the working distance.

Depth of field 2

These diagrams illustrate the effect of the convergence angle α on the depth of field.

Because of the geometry of the imaging system, scanning electron microscopes have a much greater depth of field than optical microscopes.



Astigmatism 1

Astigmatism is a problem that is commonly encountered in SEM (and TEM). The aberration of lenses causes rays in a plane parallel to the optical axis to be focused at a different focal point from rays in a plane at 90° to it. The effect of astigmatism is that objects in the image generally appear "stretched" in one direction and then in the other direction as you go through focus.

All electron microscopes are equipped with stigmators that allow the user to correct astigmatism, as shown in the next slide. Properly corrected astigmatism is essential in achieving high-resolution images.




Astigmatism 2

The SEM images shown below left illustrate how astigmatism affects the image as you go through focus. On the right is shown the image following correction with the stigmators.



Corrected

What are the differences between SEM from a normal optical microscope/magnifier?

Image-formation:

	Optical Microscope	Scanning electron Microscope
Image-formation	Image formed by lenses	Fine probe scanning
Image contrast	The brightness of reflected light colors of reflected light	The intensity of emitted electrons energy of emitted electrons

Good, Sharp and Informative Images:

	Optical Microscope	Scanning electron Microscope
In order to take good images	Good focusing: Stigmatism	Fine probe Stigmatism
Image contrast	The brightness of reflected light colors of reflected light	The intensity of emitted electrons of a particular energy. Changeable with the conditions: The energy of an incident beam The direction of emitted electrons etc



In order to get a small electron probe

Small electron source => FE-SEM
Small probe => High performance lenses,
Stability=> Vibration, Temperature, Magnetic field
Small diffuseness of beam; Stigmatism and Focus

Influence of astigmatism to the sharpness in images



Influence of Accelerating Voltage

(See-Through Effect)

5 kV





図6 試料:焼結体

Fine morphology becomes unclear with the accelerating voltage.



図7 試料:塗膜

Image contrasts inside the sample come up.

25 kV

Influence of Accelerating Voltage

Importance of low landing energy



Image Contrasts

Basically:

Brightness of electrons emitted from a sample

The behavior of electrons inside the sample

The behavior of electrons emitted selective detection of electrons

Behavior of electrons inside a sample



量子発生の深さと空間分解能(Goldsteinによる)



Where were electrons generated

Primary electrons diffuse in the sample.

Low-energy electrons :

They can come out just a narrow area near the top surface. => SE-Image = High-resolution

High-energy electrons :

They can come from a wide area deep inside of sample. => BSE-Image = Not High-resolution

They come out after reactions with the constituent atoms => Contain information on atoms in the sample.

Energy Distribution of Emitted Electrons

Contrasts in SEM-Image

SEM Image - SE-image: Morphology, High-resolution •BSE-image: Composition + Morphology, Structural Image





ET: Everhart-Thornley Scintillation SE detctor



BSE-Detector in SEM

Primary electrons



BSE Detector in SEM

BSE is decomposed into the compositional part and topological part.



Five detectors in SEM



Changes in Image Contrasts taken with TTL Detector (metallic particles : E=1.27 kV)



Slopes are bright. High resolution

Slopes are still bright. Low contrast Low resolution

Slopes are still bright. Higher contrast Low resolution

composition. Slopes are dark. Much higher contrast Lower resolution

SEM SPECIMEN PREPARATION

SPECIMENS:

- 1. Powder
- 2. Bulk Specimens: Metals/alloys Ceramics Minerals Natural fibers Nanofiber membranes etc.
- 3. Biological materials
- 4. Specimens containing oil

SPECIMEN PREPARATION

1. For small particles (powder, <100 nm)



2. For small particles (powder, >100 nm)



MOST IMPORTANT: DISPERSION !!





📥 The Scanning Electron Microscope.mp4 - VLC media player



09:38

 \Box





Example:

Nanocrystalline Cellulose Studied with a Conventional SEM

(Sosiati, International Conference on Physics, ICP 2014)



Figure 3. SEM image of nanocrystalline cellulose prepared on a C-tape which dried imperfectly (a) and TEM image of the same specimen (b).

Figure 4. SEM images of nanocrystalline cellulose prepared on perfectly dry of a C-tape (a) and a Si plate (b) substrates.



Figure 5. SEM images of nanocrystalline cellulose prepared on a carbon coated Cu-grid with different dispersion time; (a) 10 min and (b) 30 min.

For conventional imaging in the SEM, the specimen must be electrically conductive at least at the specimen surface and electrically grounded to prevent electrostatic charge accumulation at the surface during electron irradiation.

For the specimen with enough conductivity, two crucial reasons for coating are maximizing signal and improving the spatial resolution.





Target: Au, Au-Pd alloy, Pt, W, C



Charge-up

- No dried in an oven (120°C, 1 h)
- C-coating

- dried in an oven (120°C, 1 h)
- C-coating

Zeolites



Specimens with non-conducting features

What is the charge-up phenomenon ?

Charge-up occurs during observation of non-conductive specimens





The charge-up phenomenon can be overcome as follows.

- 1. Reduce the accelerating voltage
- 2. Reduce the specimen irradiating current
- 3. Apply the metal coating
- 4. Observe image in low vacuum mode
- 5. Utilize a low-acceleration BSE signal.

What is contamination?

Specimen contamination remains one of the major SEM issues to be overcome. Contamination can be resulted from many places:

- Vacuum system
- Specimen surface
- Specimen handling
- Processing chemistry
- Specimen itself

Example of contaminated specimens





The phenomenon by which gas molecules of hydrocarbons existing around the specimen due to electron beam irradiation then bond together and adhere to the specimen surface is <u>contamination</u>.

With the electron beam irradiating the sample, the clarity of the image in that area decreases and becomes darker due to the matter accumulated on the specimen surface suppressing the discharge of secondary electrons from the specimen.



Contamination can be reduced by the following methods:

- Reduction of residual gas molecules in the specimen chamber
- Reduction of gas molecules derived from specimen.

Metals/alloys



600, 800, 1000, 1500, 2000# Emery papers



Alumina or diamond paste (30, 15, 5, 1, 0.1 μm)



Etching



Mirror surface Scratch free

CLEAN !!

General preparation for biological specimens

Such specimens are usually observed in dried condition since the interior of the electron microscope is evacuated.



Oily specimens

These are observed with a low vacuum SEM. In some cases, a cool stage may be used for cooling the sample for observation.



Apply a little paste or adhesive to the specimen stub and fix the sample (cut to a small size) onto this.

CHARACTERIZATION AND INTERPRETATION OF SEM AND SEM-EDS RESULTS



Some examples and discussion



SEM images of various nanoparticle morphologies

SEM-EDS Results?

Qualitative and quantitative analyses



Typical SEM images after welding (a) and EDX results (b).



0.78 1.55 0.17

26.02

3125 3.58

10000

Yes

25.92

0.54

10.19





SEM images of melting zone welded with AA6061-TiC filler. (a) TiC nanoparticles within Mg_2Si secondary phase. (b) TiC cluster at the center of an aluminum alpha grain. c EDS mappings of Fig. 3a where red represents Al, yellow represents Ti and green represents Si.

EDS elemental mapping of quartz surface at pH 5, indicating Al accumulations in the fractures and crevices but absent on the smooth surface

Characterization of Tensile Properties of Alkali-treated Kenaf/Polypropylene Composites



Tensile strength and modulus (a) and elongation (b) versus duration of alkali treatment. 20% fiber loading

(Sosiati et al., 2019)

ATU.

SEM observation



SEM images of tensile fracture surface obtained from the composite specimens with different alkali treatment durations of kenaf fibers. (a) Untreated, (b) 4 h, (c) 10 h, (d) 24 h, and (e) 36 h.

The Influence of Carbon Fiber Content on the Tensile, Flexural, and Thermal Properties of the Sisal/PMMA Composites



SEM images of the tensile fracture surface of US/TC/PMMA composites without TC (a) and with US/TC ratios of 2:1 (b), 1:1 (c), and 1:2 (d).



(Sosiati et al., 2022)
PREPARATION AND CHARACTERIZATION OF NON-WOVEN NANOFIBROUS MEMBRANES OF CHITOSAN AND POLY(ETHYLENE OXIDE) IN A HIGH ETHANOL CONCENTRATION



(Sosiati et al., 2022)





CSNPs: chitosan nanoparticles



Tensile properties of CS/PEO and CSNPs/PEO nanofiber membranes

Specimen	Tensile	Modulus	Tensile
	Strength	Elasticity	Strain
	(MPa)	(MPa)	(%)
CS/PEO (CS/PEO (10/90)			
CS-0/PEO	2.98	10.34	434
(Neat PEO)			
CS-0.5/PEO	2.86	9.70	338
CS-1.0/PEO	2.46	12.52	318
CS-1.5/PEO	3.26	16.22	214
CSNPs/PEO			
CSNPs-	3.96	18.92	137.60
0.5/PEO			
CSNPs-	6.06	15.96	121.50
1.0/PEO			

