Advanced Scanning Electron Microscopy (SEM)

By: Yusuf Umardani M.Eng

Outline

- Apa yg bisa dilakukan dg SEM?
- Bagaimana kita mendapatkan gambar (image)?
- Interaksi Electron beam dg sample
- Signal yg dapat digunakan untuk mengkaraterisasi microstructure:
  - Secondary electrons
  - Backscattered electrons
  - X-rays
- Components dari SEM
- Beberapa hal yg berkaitan dg resolusi
- Preparasi Sample
- Beberapa aplikasi dari SEM

Apa instrument yg paling serbaguna bagi ilmuwan material?

Apa yg dapat kita pelajari dengan SEM?

- Topography and morphology
- Chemistry
- Crystallography
- Orientation of grains
- In-situ experiments:
  - Reactions with atmosphere
  - Effects of temperature

"Easy" sample preparation!!

"Big" samples!

Topography and morphology

- High depth of focus

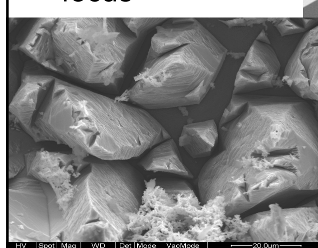


Image: Christian Kjølseth, UiO

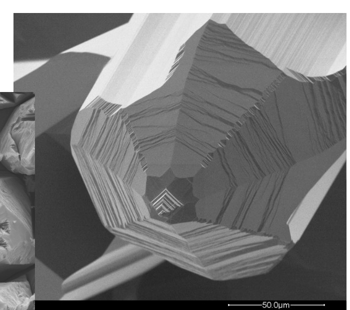
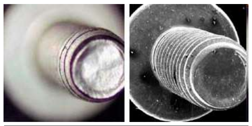


Image: Camilla Kongshaug, UiO

Depth of focus

Optical microscopy vs SEM

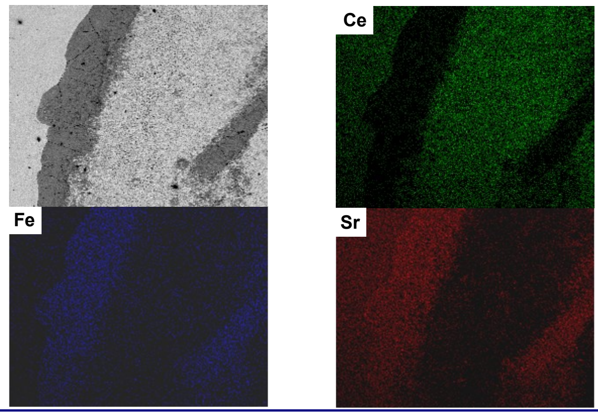


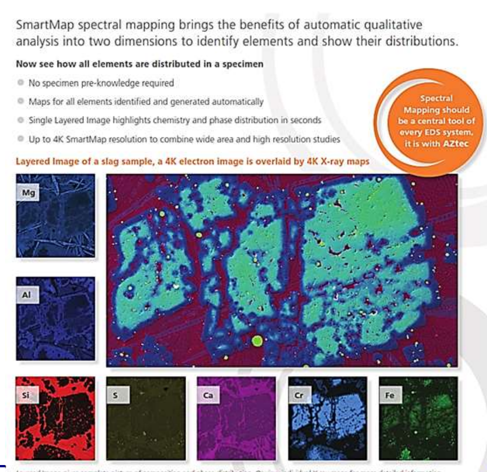
Screw length: ~ 0.6 cm

Images: the A to Z of Materials

- SEM memiliki tingkat kedalaman fokus yang lebih baik daripada mikroskop optic, sehingga SEM cocok untuk mempelajari permukaan kasar
- Makin tinggi perbesaran, makin rendah depth of focus-nya

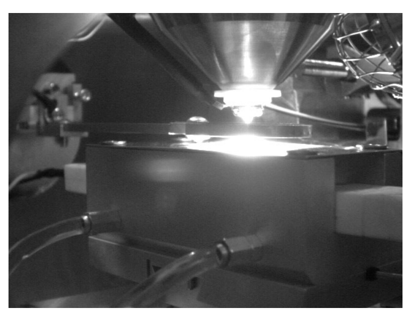
Chemistry





In-situ imaging

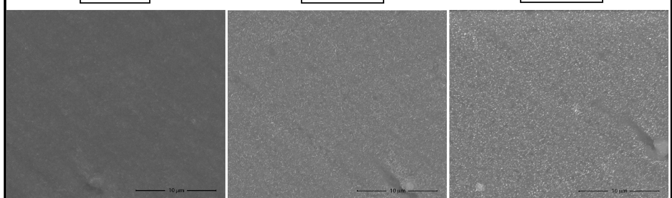
- A modern SEM can be equipped with various accessories, e.g. a hot stage



In-situ imaging: oxidation of steel at high temperatures

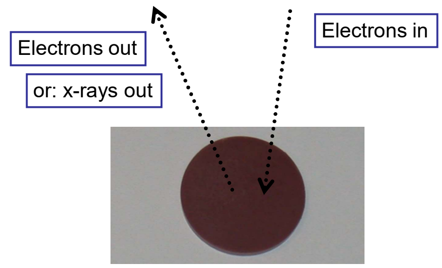
- 800 °C, pH_2O = 667 Pa
- Formation of Cr_2O_3

2 min	10 min	90 min



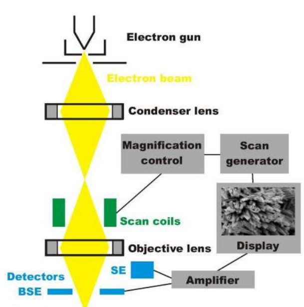
Images: Anders W. B. Skilbred, UiO

Bagaimana kita mendapatkan gambar?

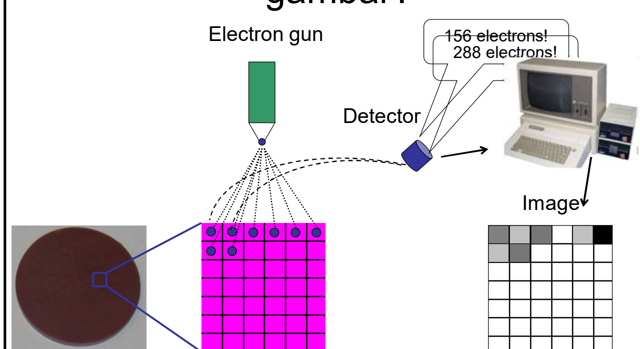


- Singkatnya: kita memotret elektron berenergi tinggi dan menganalisa elektron / sinar-x yang akan datang

instrument SEM

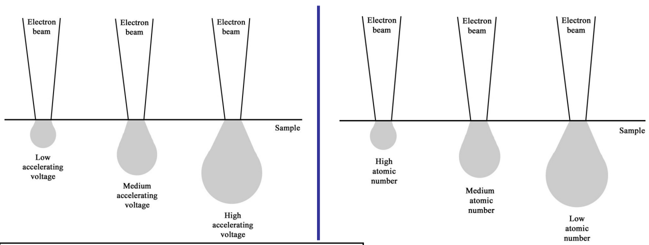


Bagaimana kita mendapatkan gambar?

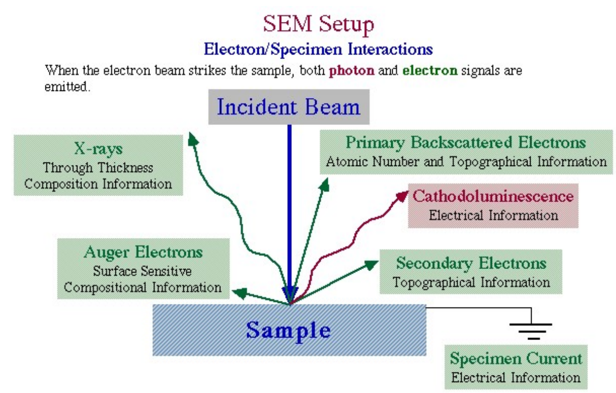


Interaksi electron beam dan sample

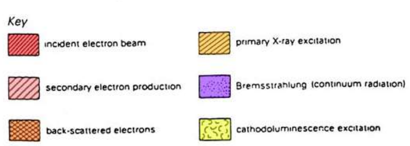
- Kejadian sinar elektron yg tersebar pd sampel, baik elastik maupun inelastic, menimbulkan berbagai sinyal yang bisa kita deteksi (lebih lanjut slide berikutnya)
- Volume interaksi meningkat dengan meningkatnya voltase akselerasi dan menurun seiring bertambahnya jumlah atom

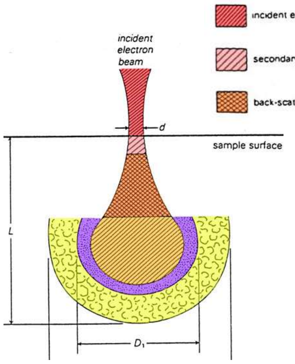


Images: Smith College Northampton, Massachusetts



Where does the signals come from?

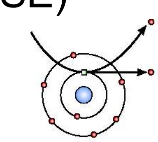


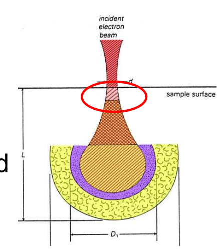


- Diameter of the interaction volume is larger than the electron spot
→ resolution is poorer than the size of the electron spot

Secondary electrons (SE)

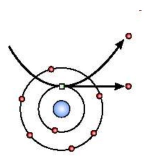
- Generated from the collision between the incoming electrons and the loosely bonded outer electrons
- Low energy electrons (~10-50 eV)
- Only SE generated close to surface escape (topographic information is obtained)
- Number of SE is greater than the number of incoming electrons
- We differentiate between SE1 and SE2





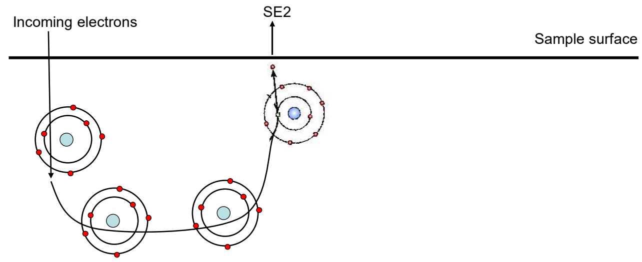
SE1

- The secondary electrons that are generated by the incoming electron beam as they enter the surface
- High resolution signal with a resolution which is only limited by the electron beam diameter



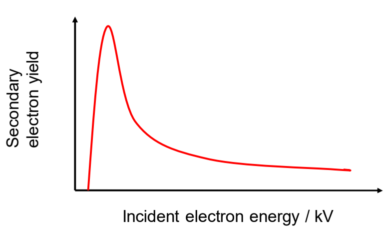
SE2

- The secondary electrons that are generated by the backscattered electrons that have returned to the surface after several inelastic scattering events
- SE2 come from a surface area that is bigger than the spot from the incoming electrons → resolution is poorer than for SE1 exclusively



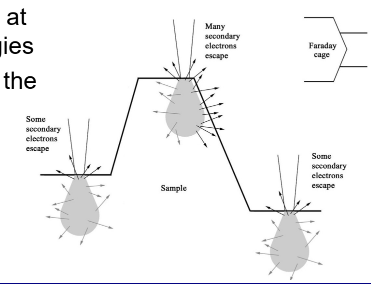
Factors that affect SE emission

1. Work function of the surface
2. Beam energy and beam current
   - Electron yield goes through a maximum at low acc. voltage, then decreases with increasing acc. voltage



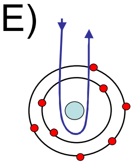
Factors that affect SE emission

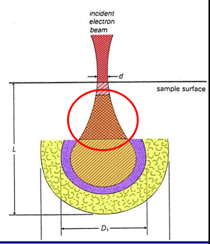
3. Atomic number (Z)
   - More SE2 are created with increasing Z
   - The Z-dependence is more pronounced at lower beam energies
4. The local curvature of the surface (the most important factor)



Backscattered electrons (BSE)

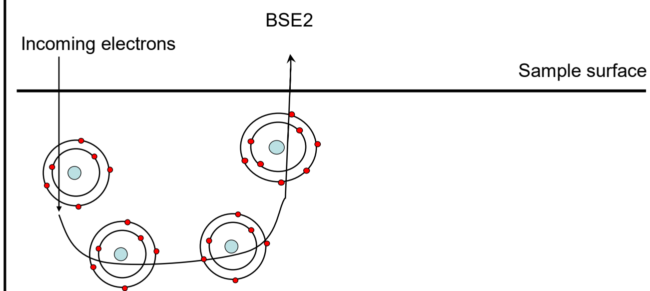
- A fraction of the incident electrons is retarded by the electro-magnetic field of the nucleus and if the scattering angle is greater than 180 ° the electron can escape from the surface





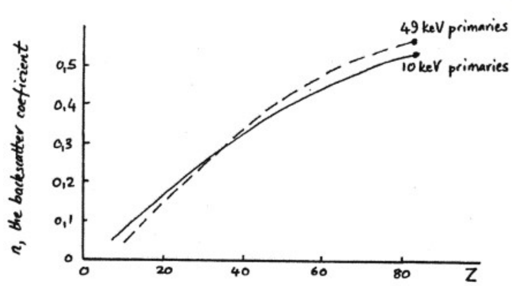
BSE2

- Most BSE are of BSE2 type



BSE as a function of atomic number

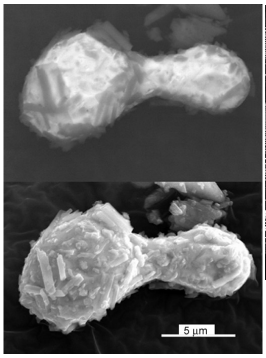
- For phases containing more than one element, it is the average atomic number that determines the backscatter coefficient η

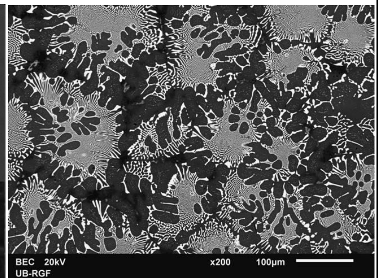


Factors that affect BSE emission

- Direction of the irritated surface
  - more electrons will hit the BSE detector when the surface is aligned towards the BSE detector
- Average atomic number
- When you want to study differences in atomic numbers the sample should be as levelled as possible (sample preparation is an issue!)

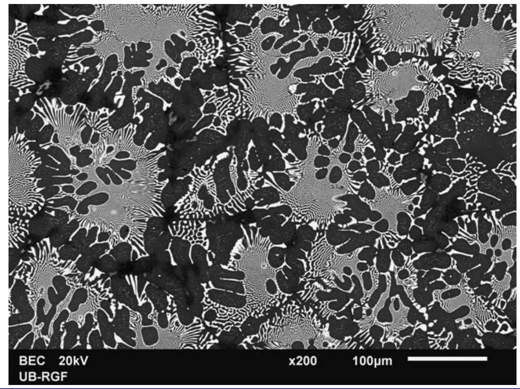
BSE vs SE

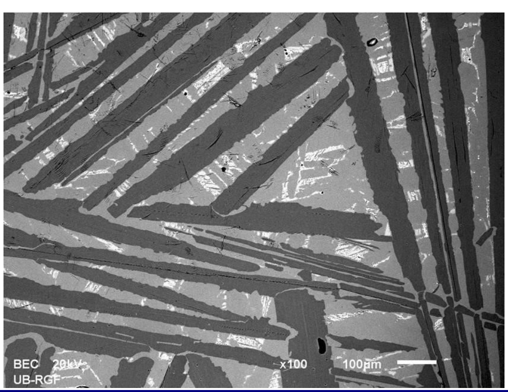




Images: Greg Meeker, USGS

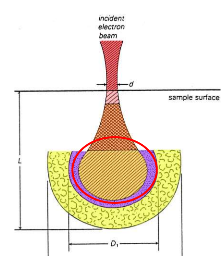
SEM-EDS application: Metallurgy





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)

Considerations when using EDS

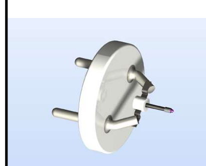
- Dead time
  - some twenty-thirty percent is ok
- Statistics
  - Signal-to-noise ratio
- Drift in electron beam with time
- Build-up of a carbonaceous contamination film after extended periods of electron probe irradiation

Electron guns

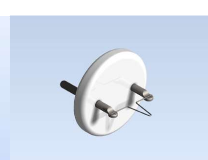
- We want many electrons per time unit per area (high current density) and as small electron spot as possible
- Traditional guns: thermionic electron gun (electrons are emitted when a solid is heated)
  - W-wire, LaB_6-crystal
- Modern: field emission guns (FEG) (cold guns, a strong electric field is used to extract electrons)
  - Single crystal of W, etched to a thin tip

Electron guns

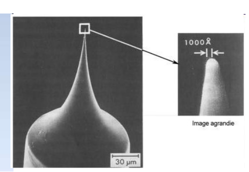
- With field emission guns we get a smaller spot and higher current densities compared to thermionic guns
- Vacuum requirements are tougher for a field emission guns



Single crystal of LaB_6



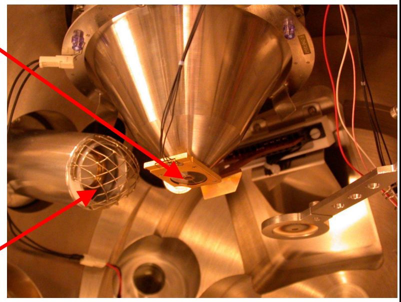
Tungsten wire



Field emission tip

Detectors

Backscattered electron detector: (Solid-State Detector)



Secondary electron detector: (Everhart-Thornley)

Image: Anders W. B. Skilbred, UiO

Traditional detectors

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

Why do we need vacuum?

- Chemical (corrosion!!) and thermal stability is necessary for a well-functioning filament (gun pressure)
  - A field emission gun requires ~ 10^{-10} Torr
  - LaB_6: ~ 10^{-6} Torr
- The signal electrons must travel from the sample to the detector (chamber pressure)
  - Vacuum requirements is dependant of the type of detector

Environmental SEM: ESEM

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

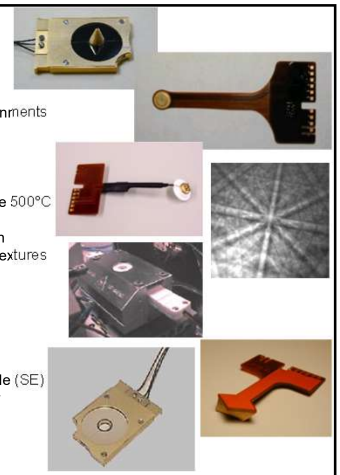
Why ESEM?

- 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)

Accessories ESEM:

- GAD – Gaseous Analytical Detector
  → for X-ray analysis in gaseous environments
- GSED – Gaseous Secondary Electron Detector
  → 500 μm aperture, allowing 20 Torr chamber pressure
- Hot stage GSED
  → Must be used at temperatures above 500°C
- EBSD – Electron Backscatter Diffraction
  → Grain orientation, grain and subgrain structures, phase identification, micro textures
- Hot stages – 1000°C and 1500°C

- ETD – Everhart-Thornley Detector
  → Secondary electron detector
- LFD – Large Field Detector
  → used in low vacuum and ESEM mode (SE)
- SSD-BSD – Solid State Backscattered Detector
  → Backscatter electrons
- EDS – Energy dispersive spectroscopy
  → X-ray analysis

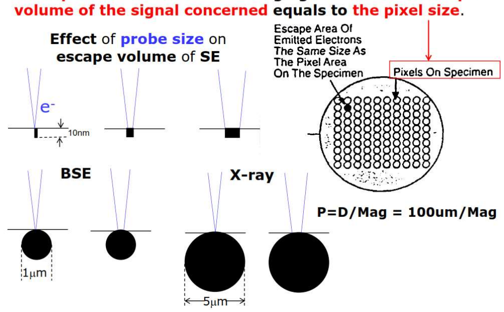


Resolution of Images

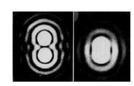
The **resolution** is the **pixel diameter** on **specimen surface**.

The optimum condition for imaging is when the escape volume of the signal concerned equals to the pixel size.

Effect of **probe size** on **escape volume** of **SE**



Depth of Field



Depth of Field

$$D = \frac{4 \times 10^5 W}{AM} \ (\mu m)$$

$A/2 = \alpha$

To increase **D**
Decrease aperture size, A
Decrease magnification, M
Increase working distance, W (mm)

$\alpha = 5 \times 10^{-3} = 0.005$
($1^{o} = 0.0175$)

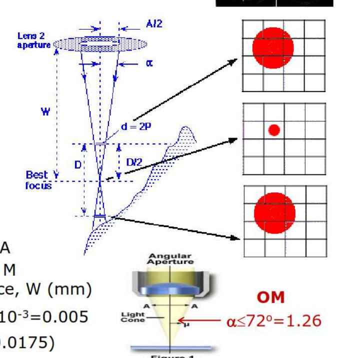


Image Contrast

SE Images

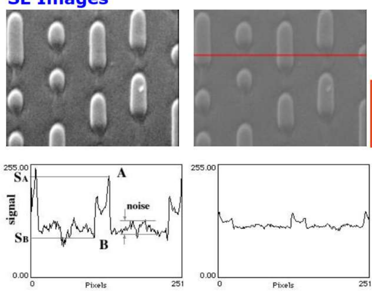


Image contrast, C is defined by

$$C = \frac{S_A - S_B}{S_A} = \frac{\Delta S}{S_A}$$

S_A, S_B Represent signals generated from two points, e.g., A and B, in the scanned area.

In order to detect objects of small size and low contrast in an SEM it is necessary to use a high beam current and a slow scan speed (i.e., improve signal to noise ratio).

SE-topographic and BSE-atomic number contrast

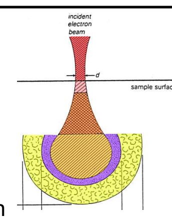
Some comments on resolution

- 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

Summary

- The scanning electron microscope is a versatile instrument that can be used for many purposes and can be equipped with various accessories
- 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: chemistry
    - Longer recording times are needed

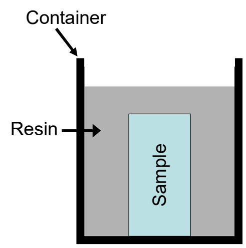
Preparasi sampel

Diskusi

Mount Sample

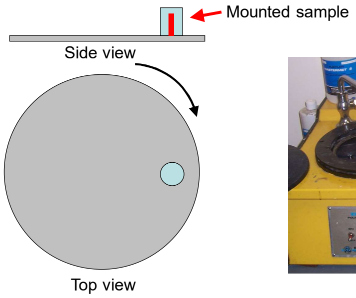
Sample cut up if too large to fit inside container

Containers 25 to 50 mm (1 to 2 inches) diameter



Grind and Polish

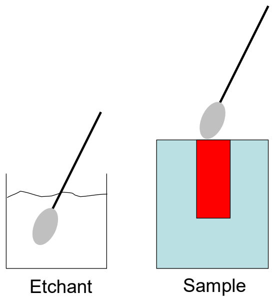
1. Sample surface ground
2. Sample polished to mirror finish
   - Polishing media is paste or slurry



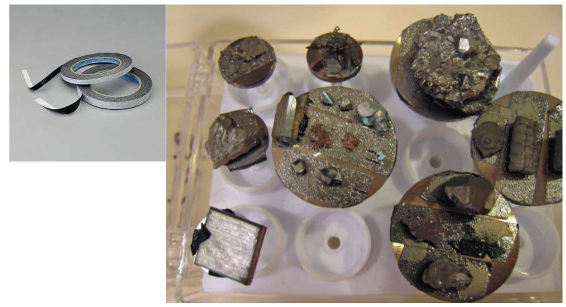
Polishing Wheels

Etch to reveal microstructure features

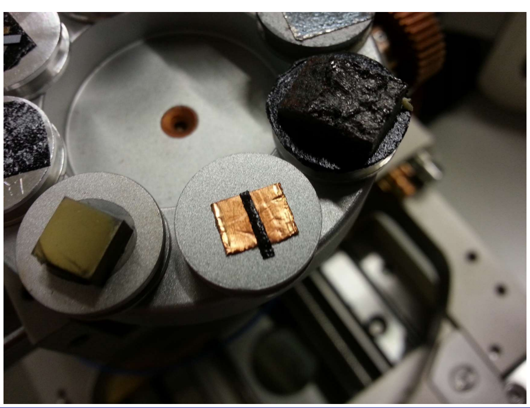
Etchant chemical used depends on the metal



Memasang sample SEM



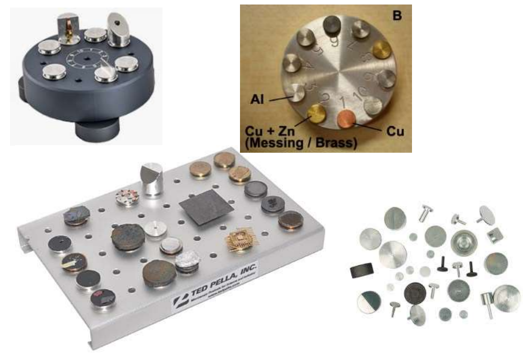
Berbagai bentuk sample



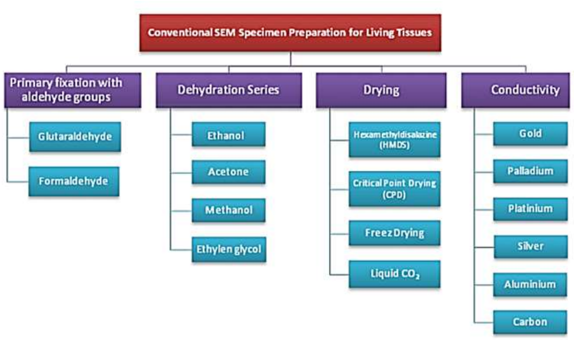
Pengaturan posisi sample

Tilting sample

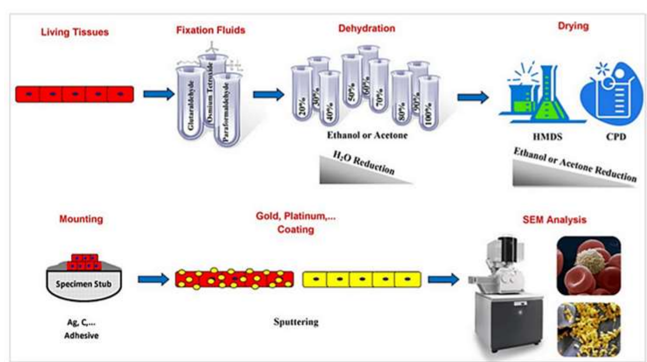
Sample holder

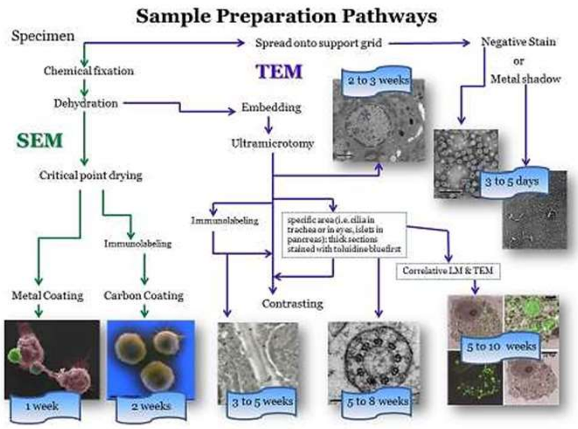


Operational Flowchart to Prepare Living Tissues for SEM Characterization

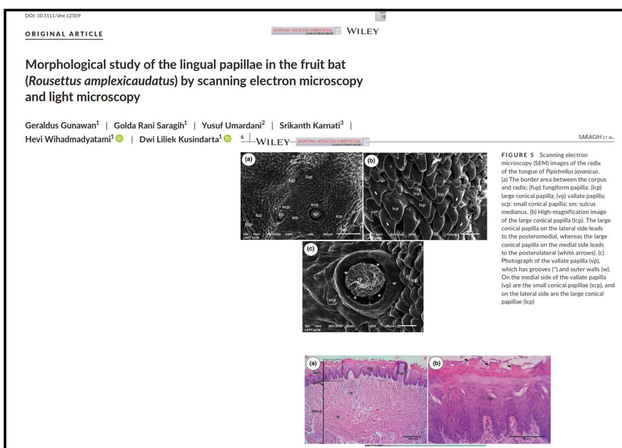


Preparation processes biological sample for SEM imaging





Bebarapa Aplikasi dari SEM


DOI: 10.1111/ahe.12509

ORIGINAL ARTICLE

ANATOMIA HISTOLOGIA EMBRYOLOGIA WILEY

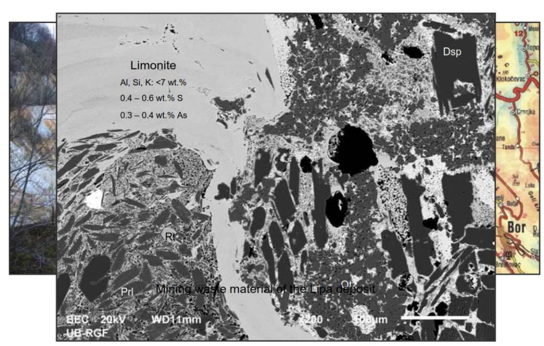
Morphological study of the lingual papillae in the fruit bat (*Rousettus amplexicaudatus*) by scanning electron microscopy and light microscopy

Geraldus Gunawan[1] | Golda Rani Saragih[1] | Yusuf Umardani[2] | Srikanth Karnati[3] | Hevi Wihadmadyatami[1] | Dwi Liliek Kusindarta[1]

6 WILEY ANATOMIA HISTOLOGIA EMBRYOLOGIA SARAGIH ET AL.

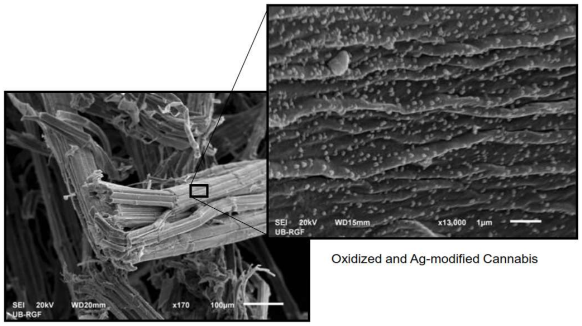
FIGURE 5 Scanning electron microscopy (SEM) images of the radix of the tongue of *Pipistrellus javanicus*. (a) The border area between the corpus and radix; (fup) fungiform papilla; (lcp) large conical papilla; (vp) vallate papilla; scp: small conical papilla; sm: sulcus medianus. (b) High-magnification image of the large conical papilla (lcp). The large conical papilla on the lateral side leads to the posteromedial, whereas the large conical papilla on the medial side leads to the posterolateral (white arrows). (c) Photograph of the vallate papilla (vp), which has grooves (*) and outer walls (w). On the medial side of the vallate papilla (vp) are the small conical papillae (scp), and on the lateral side are the large conical papillae (lcp)

SEM-EDS: Environmental



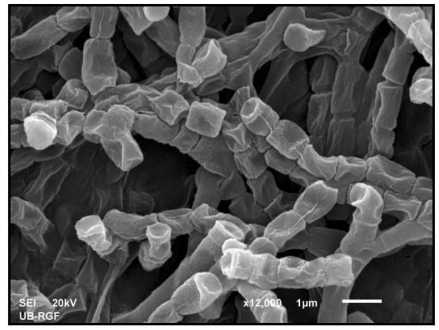
Polished section of the mining waste material from the Lipa deposit

SEM: Material Science and Technology (Textile Engineering)



Oxidized and Ag-modified Cannabis

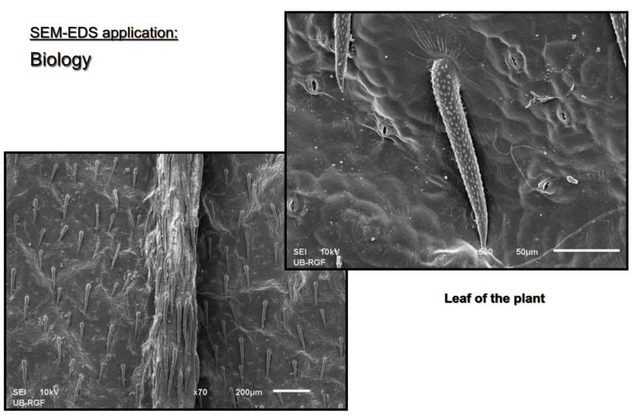
SEM: Biology



Bacteria *Streptomycetes*

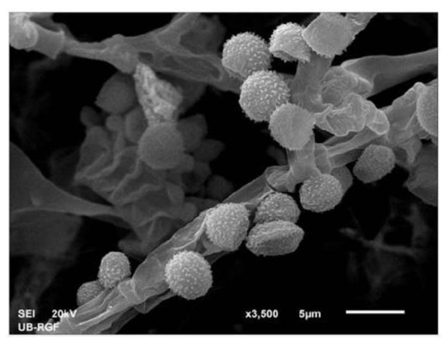
SEM-EDS application:

Biology

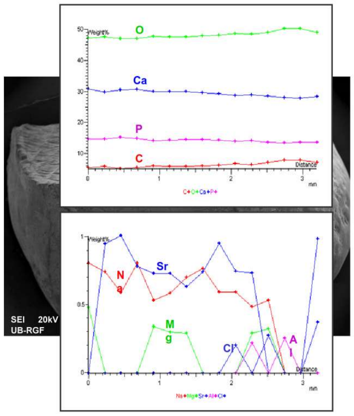


Leaf of the plant

SEM: Biology

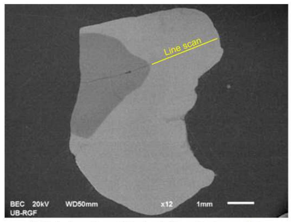


Fungi's spores on the painting



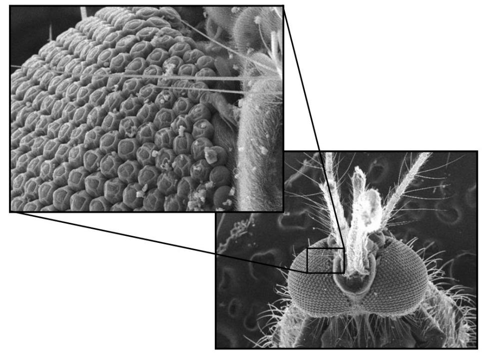
SEM-EDS application:

Medicine - Dentistry

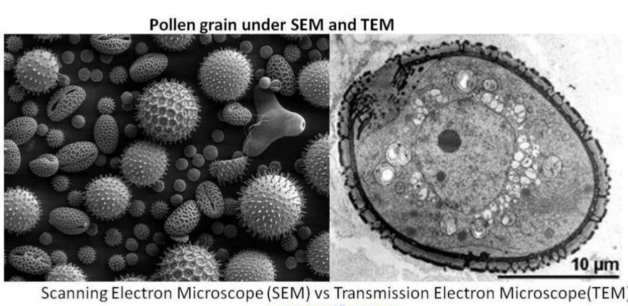


Polished section of the tooth from the image left

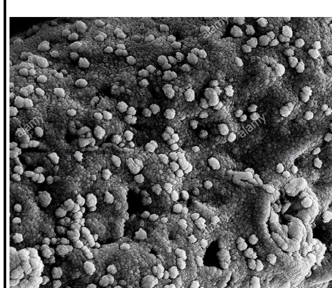
SEM: Mata Nyamuk

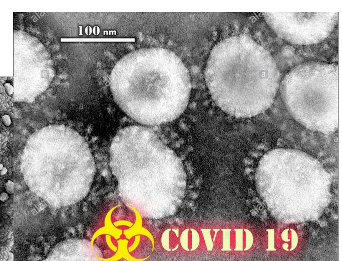


SEM vs TEM



SEM vs TEM





After edited