A Bridging Course (ABC)

Electron Microscopy (SEM & TEM)

presented by

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Research Scientists
FACTS
Basic Introduction to

Scanning Electron Microscopy (SEM)

presented by
Derrick Ang, Dr
(D.A.D.)
**Audience today?**

<table>
<thead>
<tr>
<th>A) Little or no knowledge of SEM</th>
</tr>
</thead>
<tbody>
<tr>
<td>B) Current SEM users</td>
</tr>
<tr>
<td>C) Needs my “autograph”</td>
</tr>
</tbody>
</table>

**Target Audience:**

A) Potential users who do not have any knowledge about Scanning Electron Microscopy.
Objective:

Aims to introduce new users to the basics of SEM, its capabilities and limitations.

Users will have an EEE (Easier, Enjoyable & Enlightening) time during the practical hands-on training/usage.

“Know yourself & know your enemies.”
Objective:

Aims to introduce potential users to the basics of SEM, its capabilities and limitations.

time is precious, so waste it wisely!
1. Why we need SEM and what is it?
2. SEM Instrumentation.
3. Jargon & Machine Variables in SEM.
4. Other Capabilities of SEM.
5. Sample Preparation for SEM.
6. SEMs available in FACTS.
1. Why need SEM?

Because my boss asks me to learn!
1. What is SEM?

Scanning Electron Microscope (SEM)

An instrument which **scans/rasters** a fine **electron probe/beam** over a material, and using a variety of detectors to reconstruct an image from the **signals** generated within the sample.
1. What is SEM?
An Analytical Tool (EDX).

EDX elemental mapping

Cu-Au-Ni-Ca-Sn
Let’s have a feel... Size & Cost

- Electron Microscopes
  - Scanning Electron Microscope (SEM)
  - Transmission Electron Microscope (TEM)

Scale bar
SEM (~ $ HDB flat)

TEM (~ $ Condominium)
2. SEM Instrumentation
2. SEM Instrumentation
2. SEM Instrumentation
Signals emitted when a beam of electrons hits a specimen.

Many of these signals are used in SEM or other techniques.

In TEM, they are concerned with the transmitted and diffracted electrons.

**Elastic scattering**: no energy lost, no other radiation emitted.

**Inelastic scattering**: some energy lost, usually other radiation emitted (e.g. X-rays, secondary electrons).
Beam-specimen interactions: **SEM**

- **Primary Electron Beam**
  - Inelastic scattering resulting in ejection of loosely bound electrons from sample. Shallow depth of production.
  - Elastic scattering resulting in primary electron being re-emitted from sample. Backscattering proportional to average atomic number.

**Secondary Electrons** (nm range)

**Backscattered Electrons** (several 10’s of nm to 100 nm)

**Samples Surface**

**Characteristic X-rays**

1-3 μm Analysis Depth

Volume of Primary Excitation

1-3 μm
To obtain the correct images efficiently & safely.

Winning images from FACTS Art of Science Competitions
3. Jargon & Machine Variables

**SEM/FESEM Settings**

1. Resolution
2. Accelerating Voltage
3. Probe current/spot size
4. Working distance

**Playground Merry-go-round**

5. Focus
6. Astigmatism
7. Brightness
8. Contrast
1. Resolution:
The smallest separation at which two points can be seen as distinct entities.
2. **Acceleration Voltage** ($V_A$):
   The voltage applied to the electron microscope that accelerates the electron beam down the column.
Beam-specimen interactions: **SEM**
2. Acceleration Voltage ($V_A$)
   The voltage applied to the electron microscope that accelerates the electron beam down the column.

   **For SEI:**

   - **Low $V_A < 5$ kV**
   - **High $V_A (30$ kV)**

   *Different modes (e.g. EDX, BEI) require different $V_A$*
An Imaging Tool

JEOL 7600F
STEM Au@Cu₂O

SEI Octahedron Oxides

SEI Nanospherical Oxides

JEOL 5410 - Pollen
JEOL 5410 - Human Hair
JEOL 5310 - Macroporous TiO₂
3. Jargon & Machine Variables

3. Probe current/Spot size (SS):
The size of the electron beam cross-section at the surface of the sample.

For SEI:
Smaller SS, Better Resolution
3. Jargon & Machine Variables

4. Working Distance (WD):
The distance from the underside of the objective lens to the surface of the sample.

For SEI:
Smaller WD, Better Resolution
Sample Requirements implemented in...
5. **Focus:**

Bringing the electron beam to its smallest diameter cross-section at the sample surface to produce a sharp, well-defined image.
6. **Astigmatism:**

Asymmetrical beam distortions cause blurring of an image so that it appears out of focus even when focus is correctly set. It can be corrected by stigmator lens (STIG: X and Y knobs).

Streaking/stretching of image.
3. Jargon & Machine Variables

7. Brightness:

Nothing simple enough to say.

Very Good 😊
8. Contrast:
Extent to which various parts of an image differ in brightness.
3. Jargon & Machine Variables

**SEM/FESEM**

1. Resolution
2. Accelerating Voltage
3. Probe current/spot size
4. Working distance

**Settings**

5. Focus
6. Astigmatism
7. Brightness
8. Contrast

Play around! Don’t AUTO.
## 4. Other Capabilities of SEM

<table>
<thead>
<tr>
<th>Type</th>
<th>Capability</th>
<th>Species</th>
<th>Purpose</th>
</tr>
</thead>
<tbody>
<tr>
<td>Imaging</td>
<td>SEI (Secondary Electron Image)</td>
<td>Secondary electrons</td>
<td><strong>Topographical and morphological observation</strong></td>
</tr>
<tr>
<td>Imaging</td>
<td>BEI (Backscattered Electron Image)</td>
<td>Backscattered electrons</td>
<td><strong>Compositional observation</strong></td>
</tr>
<tr>
<td>Imaging</td>
<td>STEM (Scanning Transmission Electron Microscopy)</td>
<td>Transmitted electrons</td>
<td><strong>2D morphological observation / Internal structure</strong></td>
</tr>
<tr>
<td>Analytical</td>
<td>EDX (Energy Dispersive X-ray spectroscopy)</td>
<td>X-rays</td>
<td><strong>Elemental analysis</strong></td>
</tr>
<tr>
<td>Analytical</td>
<td>EBSD (Electron Backscattered Diffraction)</td>
<td>Backscattered electrons</td>
<td><strong>Crystallographical and phase analysis</strong></td>
</tr>
<tr>
<td>Patterning</td>
<td>EBL (Electron Beam Lithography)</td>
<td>Primary beam</td>
<td><strong>Custom patterns</strong></td>
</tr>
</tbody>
</table>
Beam-specimen interactions: **SEM**

- **Inelastic scattering**: Resulting in ejection of loosely bound electrons from sample. Shallow depth of production.
- **Elastic scattering**: Resulting in primary electron being re-emitted from sample. Backscattering proportional to average atomic number.
4. Secondary Electron Micrographs

- Topographical/Morphological Observation
4. Backscattered Electron Micrograph

• Compositional Observation

SEI

Sn-Ca
Ni
Cu

15kV X2,000 10μm 111118

BEI

Sn-Ca
Ni
Cu

15kV X2,000 10μm 111118
4. Transmission Electron Micrograph

- 2-dimensional Morphological Observation/Internal structure Observation

JEOL 7600F
STEM Au@Cu$_2$O
4. Energy Dispersive X-ray Spectroscopy

- Elemental Analysis

**Sn-Ca-Ni-Cu**

**Area Scan**

**Elemental mapping**

Sn  Ca  Ni  Cu
• Crystallographical and Phase Analysis

BEI

Shows secondary phase

Different crystallographic orientations

Primary phase

Presence of secondary phase
4. Electron Beam Lithography

Electron beam lithography (often abbreviated as e-beam lithography) is the practice of emitting a beam of electrons in a patterned fashion across a surface covered with a film (called the resist), ("exposing" the resist) and of selectively removing either exposed or non-exposed regions of the resist ("developing").

The purpose, as with photolithography, is to create very small structures in the resist that can subsequently be transferred to the substrate material, often by etching. It was developed for manufacturing integrated circuits, and is also used for creating nanotechnology architectures.
## 5. Sample Preparation

### Sample requirements implemented in FACTS

<table>
<thead>
<tr>
<th>Constraint</th>
<th>Why?</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample size, DH (&lt; 25\text{mm x 5mm})</td>
<td>Chamber size, detectors</td>
</tr>
<tr>
<td><strong>Dry</strong> sample</td>
<td>High vacuum required</td>
</tr>
<tr>
<td><strong>NO BULK magnetic</strong> samples (*\text{except on 7800F})</td>
<td>Electromagnetic lens inside SEM</td>
</tr>
<tr>
<td>Electrically conductive</td>
<td>Using electron as a probe</td>
</tr>
</tbody>
</table>
Sample Requirements implemented in

Sample Size (< 20mm x 5mm)
Sample Requirements implemented in

Dry Sample

Macroporous TiO₂

Concrete slabs

Pellet
Sample Requirements implemented in

**NOOO Bulk Magnetic Samples!!!**

- Stainless Steel
- Nickel Foam
- AFM Tip
Sample Requirements implemented in Electrically Conductive

White streaks are electron charging effects.

Drifting too!
5. Sample Preparation

Sample holder

- Copper Tape
- Carbon Tape

Pt/Au Sputter Coater
5. Sample Preparation

Pt/Au Sputter Coater

Use as little “make-up” as possible!
SEM is just like Food!

Fresh sample → Correct Preparation → Easier life → Best SEM results.
6. Scanning Electron Microscopes in FACTS (Facility for Analysis Characterisation Testing & Simulation)

- 2x JEOL 7600F
- JEOL 6340F
- JEOL 6360
- JEOL 5500, 5410
2 NEW FAMILY MEMBERS!

JEOL 7800F Prime
Thermo Scientific Quattro S
# Specifications

<table>
<thead>
<tr>
<th>Model</th>
<th>TS Quattro S (ESEM)</th>
<th>JEOL 7800F Prime (with low vacumm)</th>
<th>2x JEOL 7600F</th>
<th>JEOL 6340F</th>
<th>JEOL 6360</th>
<th>JEOL 5500</th>
<th>JEOL 5410</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resolution</td>
<td>1 nm (high vac)</td>
<td>0.7 nm</td>
<td>1.5 nm</td>
<td>3 nm</td>
<td>4 nm</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.3 nm (esem)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Accelerating Voltage</td>
<td>0.2 - 30 kV</td>
<td>0.01 - 30 kV</td>
<td>0.1 - 30 kV</td>
<td>0.5 - 30 kV</td>
<td>0.5 - 30 kV</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Magnification</td>
<td>25x to 1,000,000x</td>
<td>25x to 1,000,000x</td>
<td>25x to 1,000,000x</td>
<td>25x to 600,000x</td>
<td>30x to 300,000x</td>
<td></td>
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<tr>
<td>Electron Source</td>
<td>Thermal FEG</td>
<td>Thermal FEG</td>
<td>Thermal FEG</td>
<td>Cold FEG</td>
<td>Tungsten</td>
<td></td>
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<tr>
<td>Other Attachments</td>
<td>- BEI</td>
<td>- BEI</td>
<td>- BEI</td>
<td>- BEI</td>
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<td>- BEI</td>
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<tr>
<td></td>
<td>- EDX</td>
<td>- TED</td>
<td>- TED*</td>
<td>- EDX</td>
<td></td>
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<tr>
<td></td>
<td>- WDX</td>
<td>- EDX (170mm²)</td>
<td>- EDX (10 &amp; 50mm²)</td>
<td>- EBL*</td>
<td></td>
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<tr>
<td></td>
<td># Environmental up to 3500 Pa</td>
<td># Accept bulk magnetic samples.</td>
<td># Accept bulk magnetic samples.</td>
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<tr>
<td></td>
<td># Heating stage up to 1400°C</td>
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Software – For Training

Online Micro and Nano Characterisation Instruction (OMNI)

https://web.mse.ntu.edu.sg/omni/
About FACTS is a central facility for the advanced application of electron microscopy & X-Ray diffraction to research in NTU and is also open to scientists and engineers from NUS, A*STAR RIs and private companies.

Equipment

Scanning Electron Microscopes
- 3 SEMs, 4 FESEMs, 1 ESEM, 1 EPMA, 1 AES, 1 FIB & 1 cryo-FIB (NISB)
- Capabilities:
  - High Resolution Secondary Electron Imaging (SEI)
  - Backscattered Electron Imaging (BEI)
  - Energy Dispersive Spectroscopy (EDS) (Point, Line and Mapping)
  - Scanning Transmission Electron Microscopy (STEM)
  - Electron Backscattered Diffraction (EBSD)
  - Electron Probe Micro Analysis (EPMA)
  - Scanning Auger Electron Microscopy (SAM/AES)
  - Focused Ion Beam (FIB)
  - Electron Beam Lithography (EBL)

Transmission Electron Microscopes
- 6 TEMs & 3 cryo-TEMs (NISB)
- Capabilities:
  - Aberration-corrected Atomic Resolution Imaging
  - Selected Area Electron Diffraction (SAED)
  - Convergent Beam Electron Diffraction (CBED)
  - Nanobeam Electron Diffraction (NBED)
  - Bright and Dark Field Images
  - Energy Dispersive Spectroscopy (EDS) (Point, Line and Mapping)
  - Scanning Transmission Electron Microscopy (STEM)
  - Electron Energy Loss Spectroscopy (EELS)
  - Cryo-TEM

X-ray Instruments
- 8 XRDs, 2 SAXS/WAXS & 1 XPS
- Capabilities:
  - Powder XRD
  - Grazing Incidence XRD – Thin film In-situ High Temperature XRD
  - Single Crystal XRD
  - Reciprocal Space Mapping
  - X-ray Reflectometry
  - High Resolution XRD
  - Small/Wide Angle X-ray Scattering (SAXS/WAXS)
  - Residual Stress, Texture and Pole Figure Investigations
  - X-ray Photoelectron Spectroscopy (XPS)
  - Ultraviolet Photoelectron Spectroscopy (UPS)
  - High flux XRD for in-situ measurement
Becoming a FACTS user

FACTS facilities are open to faculty, postgraduate students and research staff to use. Training is provided for users after which instrument can be booked online (https://cebs.ntu.edu.sg/fom/welcome). Please see our policy for more information (http://research.ntu.edu.sg/facts/Pages/Policies.aspx). The hourly usage rates can be found at http://research.ntu.edu.sg/facts/Pages/Charges.aspx. For more information, please contact our facility personnel. Tel: (65) 6790 6158/6592 1813.

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