**MAEG5140** **Materials Characterization Techniques**

**The Chinese University of Hong Kong**

**Laboratory: Scanning Electron Microscope [SEM]**

**Objectives:**

In this laboratory, you will learn how to operate a scanning electron microscope (SEM) to view the microscopic world.

A scanning electron microscope (SEM) is a type of [electron microscope](http://en.wikipedia.org/wiki/Electron_microscope) that produces images of a sample by scanning it with a focused beam of [electrons](http://en.wikipedia.org/wiki/Electron).

The model used in this lab is JEOL7800F. You will examine the specifications and features of this SEM during the lab.

Note1: The first major part of this lab sheet is about the operation of the SEM, and there are many pictures describing how a particular button works, there is NO wired internet connection in the lab, so you may have to print this lab sheet or to view it in your own electronic devices.

Note2: To avoid computer virus infection the TA will send the measurement files to students’ representatives after the lab. NO USB DRIVE is allowed to be inserted into the equipment computer.**Introduction:**

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the sample's surface topography and composition. The electron beam is generally scanned in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer. Specimens can be observed in high vacuum, in low vacuum, in wet conditions (in environmental SEM), and at a wide range of cryogenic or elevated temperatures. (source: <https://en.wikipedia.org/wiki/Scanning_electron_microscope>)

**Why use electrons instead of light in a microscope?**

A modern light microscope has a maximum magnification of about 1000x. The resolving power of the microscope was not only limited by the number and quality of the lenses but also by the wavelength of the light used for illumination. White light has wavelengths from 400 to 700 nanometers (nm). The average wavelength is 550 nm which results in a theoretical limit of resolution (not visibility) of the light microscope in white light of about 200 – 250 nm.

**Compare an Optical Microscope vs a Scanning Electron Microscope:**

|  |  |
| --- | --- |
| PW_optical_image_of_nanofibers.png(a) Optical microscope image of nanofibers | PW_SEM_image_of_nanofibers_at_4000x.png(b) Scanning electron microscope image at 4000x magnification of same nanofibers |

Figure 1: Comparison between Optical Microscope and Electron Microscope at the same magnification factor. (source: <http://www.nanoscience.com/technology/sem-technology/>)

**The Main Equipment:**

|  |  |
| --- | --- |
| 1. Computer and monitor of SEM  2. Main machine and chamber of SEM  JSM-7800F Schottky Field Emission Scanning Electron Microscope | Products |  JEOL Ltd. |  |

**Knobset panel, specimen stage control panel and trackball:**



**Sample preparation tool:**

|  |  |
| --- | --- |
| https://www.rc-harwell.ac.uk/wp-content/uploads/2021/08/Sample-holders-e1629296195297-300x225.jpg  Sample holders |  |
| Conductive glues, stickers to fix samples onto the sample bases. | Tweezers (Upper: general purposes;  Lower: for sample bases) |

**Procedures:**

**A: Sample specimen preparation**

1. Carbon tape/copper tape is a fairly easy method to secure a sample to a sample mount. Using tweezers, center the double stick carbon tab on the sample mount. Slightly apply pressure around the edges of the tape.
2. Remove the cover from the other side of the double stick carbon tape/copper tape. Using tweezers align your sample with the carbon tab and gently place your sample on the sticky surface. Gently apply pressure with the tweezers to the edges of your sample. (Remember excessive pressure is not necessary and can cause more trouble trying to remove with sample with no residue.)
3. Align the top of the sample mount inside the JEOL adapter part. Use the screw on bottom to raise of lower the sample mount.



1. For non-conductive samples a coating is usually needed for observation in SEM. This can be easily performed by using sputter coating of gold, platinum, or gold-palladium.

**B: Inserting a sample**

1. Turn on both monitors and check that SEM software is running. Start them if necessary.
2. Prepare your sample.
3. Insert sample into microscope:
4. Press Exchange position.
5. Press and hold VENT for ca. 1 sec. Open securing latch. Wait.
6. Open chamber and insert holder along the direction of the arrows.
7. Close chamber.
8. Press and hold EVAC for ca. 1 sec. Wait until blinking stops.
9. Operate the rod to move the sample to the stage.

(\*\*This step can only be performed by Technician!)

1. Take out the rod.
2. Wait until vacuum level reaches less than 2.8x10-4 Pa.
3. Select Acceleration voltage.
4. Press Observation ON.



**C: Microscope operation**

1. Set your accelerating voltage either at the top of the screen, or the bottom panel.



1. Ensure that the LED (Lower Electron Detector) is selected by clicking the detector in the scanning window.



1. Select the ZFC button (if not already selected)



1. Select a WD of 10 mm.



(Note: Working distance (WD) value: sets the effective focal length of the objective lens.

Z height value: sets the distance of the (supposed) surface level of the sample from the objective lens.

If WD > Z, if your sample is lower than the correct level and vice versa.

If WD < Z you need to set the Sample Offset value accordingly. )

1. The Z height will change until it matches 10 mm.



(Note: The sample can be set also 0-40 mm higher than the nominal level, but then the Sample Offset value has to be set after inserting sample. It is located at the bottom of the sample holder select window, which pops up automatically after holder insert. )

1. Turn Observation ON if vacuum down to 2.8E-4.



1. Select a Quick 2 scan speed. Quick 1 is set for auto alignments and should never be changed. Quick 2 is faster than Quick 1.



1. Use the Navigator window to center over sample. Right click – Move stage to center.
2. Adjust brightness and contrast. Use knobs on knob set. Select ACB button on computer. Select ACB button on knob set.



1. Aligning

Usually the microscope is aligned well enough for micrometer scale operation. In this case, only focusing is necessary. For higher magnification work, the electron beam needs to be aligned and astigmatism of the objective lens has to be corrected.

1. Focus

The first level of aligning is always focusing. Focusing is done using the FOCUS knob on the operation console. Clockwise rotation is under focus (weaker lens) and counterclockwise is over focus (stronger lens).

If possible select some feature, which you can use in the magnification range from ca. 1000 to 20000.

Start from a low magnification and when you get good enough image move on to higher magnification for focusing. It the alignments are really off, you might not get a clear image at all.

1. Beam align

Press ALIGN on operation console. The image starts to move on the screen. Use the X and Y knobs to minimize the movement. Press ALIGN OFF (STIG) button when image has stopped. Repeat for magnifications up to ca. 20000. Focus the image whenever necessary.

1. Image capture
2. Select the camera button that says “Normal”. (To change image capture settings, select Setup (S) > Operation Settings > Scan Setting)



1. When prompted, save your images to the folder named with your folder name.
2. Unfreeze the image, if necessary, by clicking on the FREEZE button in the knobset panel.

**D: Taking out samples**

1. Click Observation OFF to turn off acceleration voltage.
2. Click Exchange Position to move the sample holder to the exchange position.

(\*\*Make sure that EXCH POSN is lit on the exchange compartment before proceeding.)



1. Bring the sample to the exchange compartment by operating the rod.
2. Pressurize the exchange compartment:
   1. Press and hold VENT for ca. 1 sec until it starts to blink.
   2. Open securing latch. Wait.
   3. Open chamber door.