

Basic SEM imaging

Overview: Provides basic instructions on how to image samples with the SEM and the three stationary electron detectors (InLens, EsB, and SESI).

Before you start:

- Make sure that the nitrogen tank is full, and valve is open. If the tank is empty, the load lock will not open

Log-In

- Click on the 'SmartSEM' icon on the desktop to open software
- If the ChamberScope does not open automatically, open it by clicking on the 'Chamber Scope' desktop icon

Sample Preparation

- Use a Zeiss stub to mount your sample. The Zeiss stubs are different than the stubs used with the FEI instrument- the height is shorter.

Sample loading via the load lock

- Press the **Vent** button. This will flush the chamber with nitrogen gas and after a few s, you can open the door
- Slide sample onto the dove tail receiver, with the three holes on the flat side of the sample holder pointed towards the load lock door/transfer rod. With one hand on the sample holder, attach the transfer rod by turning the black knob clockwise with your other hand. Close the load lock door.
- Check to make sure that the LED over Stage Ready is lit. This indicates that the stage is in a position inside the chamber to receive the sample. If it is not lit, move the stage to the Sample Exchange position. In the Stage Points List menu, double click \$exchange.
- Press **Transfer**. This will pump down the load lock and open the gate valve.
(-If sample holder not in the 'exchange' position, push the 'exchange' button on the keyboard.
 - Performs sample exchange routine in which the stage is lowered and sample moved to exchange position.)

- Transfer sample holder to stack dove tail with arm. Un-attach transfer rod to sample by turning black knob counter-clockwise.

- Fully retract transfer rod, the Rod Retracted LED should light up.

- Press **Store**

Stage Navigation

- Use joystick to move stage inside the chamber. The smaller joystick on the left controls **z** (up and down) and **t** (tilt). The larger joystick on the right controls **x**, **y**, and **r** (rotate). The two **m** buttons move your sample holder up and down relative to the stage.

- ***** **Break** is used to immediately stop stage movement.

- Make sure the sample will not collide with the SEM column when moved over for imaging, lower **z** if necessary.

- There are two IR cameras in the chamber to use for stage navigation. These can be selected from the 'Detectors' drop down menu in 'SEM Control'.

1. "TV". This is the main camera you should use. It is in the back of the instrument, pointing towards the front door.

2. "Aux 1". This camera points down at the sample from an angle. It is located near the FIB gun.

-Position your region of interested under the SEM column. This is easily accomplished with Stage Navigation.

>>Pull out menu >> Stage Navigation

Also accessed by the 'binoculars' icon on the Toolbar.

-In Stage Navigation, press the >> button on the lower right corner. Select your sample under the Sample Holder drop down menu.

-Click on the region of the sample holder that contains the region of interest, and the stage will automatically center this point underneath the SEM column.

Turn on the SEM

-Make sure that the chamber vacuum is sufficient.

>>SEM Control >> Vacuum

System Vacuum should read better than 1×10^{-5} mbar

-Open the Separation Valve

>>Pull out menu >> Airlock

-Click **Open Column Chamber Valve**

-In the lower right hand corner of the SmartSEM software, click on the **EHT button**, select **EHT On**. The SEM will run up to set potential within a few s.

SEM setup in >>SEM Control

1. Set the accelerating potential (EHT, extra high tension)

>>Gun

Set EHT in kV. Available range 0.1 to 30 kV

1. Set the current

>>Apertures

-Current is determined by diameter of aperture. Change Aperture Size from 7 μm to 120 μm .

-30 μm is standard for SEM imaging, offers best resolution as it is in the center of the aperture grid; in using any other apertures the beam must be deflected in 2 dimensions to access it. The 60 and 120 μm work well for simultaneous FIB/SEM work.

-Check 'High Current' if you would like to double the current at a given aperture size.

-'OptiProbe' can be used to fine tune current at a given aperture

>>Gun

Select OptiProbe, choose current with slider

2. Choose a detector

>>Detectors

-Select 1 of 3 standard electron detectors:

1. InLens

-Detects SE with high efficiency, in column

-Potential range of 100 V to 20 kV (max is 20 kV)

-Works best at short WD (3-6 mm) and low tilt angle

2. SESI (Secondary Electron, Secondary Ion)

-Detects SE and SI, Everhart-Thornly type detector positioned away from sample

-Collector voltage can be modified to discriminate between ions and electrons

- SE mode: +400 V
- SI mode: -4 kV
- Potential range 1 to 30 kV. Typical WD for imaging in 4-6 mm range, but can be used at long WD (>10 mm)
- 3. EsB (Energy Selective Backscattered)
 - Detects backscattered electrons and SE, in column
 - Grid potential can be used to select BSE from SE
 - > 800 V: BSE
 - < 800 V: BSE + SE
 - Usually set grid potential to ~1/2 of EHT
 - Typical potential range of 1 to 5 kV, WD from 1 to 4 mm
- 3. Set up scan
 - >>Scanning
 - Choose the Store resolution of your images
 - Select a Scan Speed. Range from 1 to 15, lower is faster.
 - 'Cycle Time' is the time to acquire 1 screen.
 - 'Noise Reduction' is available to improve image quality by averaging or integrating n pixels, lines, or frames together. Choose higher values of n for more noise reduction.
 - Pre-set scanning conditions can be selected from the numbered buttons on the Toolbar.

Focus/Imaging Procedure:

- **For noise reduction, enable 'Quiet Mode'. This will turn off the scroll pump. Go to >>SEM Control >> Vacuum. Check 'Quiet Mode'.
- **Note that 'Tab' button on keyboard alternates between coarse and fine control.
- **Procedure below uses the keyboard buttons to optimize the image. However, each of these parameters can also be controlled in the software via >>SEM Control >> Apertures
- 1. Adjust contrast and brightness
 - Use the **Brightness** and **Contrast** keyboard buttons. If image is totally black, turn up Brightness until screen turns gray, then increase Contrast until image forms.
- 2. Rough Focus
 - Bring sample into rough focus with the **Magnification** and **Focus** knobs. Establish acceptable working distance (WD) for potential and detector. WD is on the Data Zone, and is only accurate if the beam is focused at the sample surface.
 - The + and – buttons by the Focus knob change the scan speed.
 - Check 'Track Z' on the Data Zone if you want the software to update WD with Z stage movement.
 - 5 mm WD recommended for most imaging
- 3. Wobble
 - Press **Wobble** on keyboard. Adjust the **Aperture X** and **Aperture Y** knobs so that image goes in and out of focus, instead of moving in x and y. Adjust one axis at a time.
 - Wobble amplitude can be adjusted in >>SEM Control.
- 4. Focus and Stigmation
 - Bring image into good focus, keep increasing magnification and adjust focus to bring the ever smaller features into focus

-Find small feature on surface (μm to nm range), and press **Reduced** to use reduced area scanning over feature over interest and slow down scan speed. On keyboard, adjust **Stigmator X** and **Stigmator Y** to eliminate stigmatism in image.
-Go back and forth between stigmatism and focus to obtain sharpest possible image at highest magnification.

5. Other Keyboard Features

-To center feature in image, press "Ctrl + Tab", then click the feature to center.
-At low mag, stage is moved. At high mag, image beam shifted
-**Shift X** and **Shift Y** on the keyboard can be used to navigate at high magnification.
-**Scan Rotate** will digitally rotate the image. Press the knob to activate.
-**Camera** switches between the TV camera and the electron detector
-**Freeze** stops the image. In >>SEM Control >> Scanning, you can select Freeze on End Frame or Freeze on Command.
-**Exchange** runs the Exchange macro. By default, it turns the beams off and moves the stage to the \$exchange position.
-**Resume** runs the Resume macro. By default, it just opens the Column Chamber valve.

6. Take Image

-Adjust scan speed, line/frame/pixel averaging can to improve image.
-**Freeze** button to pause image.
-Export image as TIFF. This can be done with the 'Camera' button on the toolbar.

Dual Detectors, Dual Mag, Signal Mixing

-To simultaneously image a single feature with 2 detectors in 2 windows:
-Right click on image >> Send to >> 2nd Image Window
-A green anchor shows which window is active. Choose detector for each image.
-Dual Mag allows a single region to be imaged at two different magnifications simultaneously.
>> SEM Control >> Scanning
Click 'Dual Mag'.
-To mix signal from two different electron detectors:
-Click 'Mixing' option in >> SEM control >> Detectors
-Choose mixing ratio with the 'Signal' slider.

Shut Down Procedure

1. Turn off EHT. Click the **All** button (has a green check mark) on the lower right hand of screen, select **EHT Off**.
***Never shutdown the gun.
2. Close the Column chamber valve.
>>Airlock, click **Close Column Chamber Valve**
3. Go to TV detector for easier navigation. Move stage to a safe distance from the pole piece.
4. Go to the sample exchange position.
>>Stage Points List, click \$exchange
5. Use airlock to retrieve sample.
Push **Transfer**, attached rod to sample.
Transfer sample to Load Lock, push **Vent**.
Open door, unscrew transfer rod, remove sample.
Push **Store** to pump down.

6. Log off software by clicking red X on window. Be sure to choose to log out.
7. If last user before weekend or holiday, shut down the server.
8. Record relevant information in notebook.