Publications that made use of the EPIC facility instruments must include the following acknowledgements:

“This work made use of the EPIC facility of Northwestern University’s NUANCE Center, which has received support from the Soft and Hybrid Nanotechnology Experimental (SHyNE) Resource (NSF ECCS-1542205), the MRSEC program (NSF DMR-1720139) at the Materials Research Center, the International Institute for Nanotechnology (IIN), the Keck Foundation, and the State of Illinois, through the IIN”

Find this information online at http://www.nuance.northwestern.edu/epic/publication-acknowledgements/index.html
I. Policies and Introduction

Reservations

SU-8030 reservations are made using the NUCore online reservation system. Start your reservation before you begin using the instrument. When your session is complete, be sure to end your reservation in NUCore. If you need extra time on the microscope, we recommend ‘extending’ your original reservation, rather than making an additional reservation. There is a hardware control system on the SU-8030, so the system will not function unless you are logged in to NUcore. Tampering with or disabling the hardware control may result in revocation of your EPIC privileges.

Saving Your Data

During your session, you may store image data to your own folder within the SU-8030 folder on the EPIC_SEM (S:) drive. The SEM server is accessible through a computer in the lab. EDS data must first be saved onto the INCA projects folder on the desktop and then transferred onto your folder on the S:\ drive. You can transfer your data from the SEM server computer to a USB, etc. You should NEVER take your data directly from either the SEM or EDS computers.

SEM Rules

1. Please wear gloves when handling any components and samples that will go into the SEM.
2. Do not install any software onto the SEM’s PC.
3. Do not insert any flash drives into any microscope computer!
II. Sample Loading

1. Log into the NUcore system
   a. Under the “My Reservations” tab, click ‘begin reservation’ next to the appropriate session

2. Assemble the sample holder
   a. The base (A) should be placed so the RED side is DOWN. If the red side is not down, it will be difficult to remove your sample from the chamber at the end of your session.
   b. Screw the top onto the end of the screw with the smaller tread until it’s tight and does not rotate (D onto C)
   c. Before screwing the screw and top into the base, screw the tightening washer (B) onto the center screw.
   d. Screw the top (D), screw (C) and washer (B) assembly onto the base
      i. The screw should not go all the way through the base. The base must stay flush to the counter.
   e. Adjust the height of the sample by rotating the base on the screw.

3. Assemble the sample holder and load your sample stubs onto the holder. Verify the following:
   a. All samples fit under the standard height gauge. No part of any sample should EVER be taller than, or touching the gauge.

   b. Sample stubs are secured with the set screws around the sides of the holder.
4. Verify that the Hitachi PC-SEM software is running on the left-hand screen.
   a. **Note:** To log into the PC and PC-SEM software, *username and password are “semuser”.*

5. Press **AIR** button on load lock and wait for beep.

6. Activate Chamber Camera using the **Camera On/Off** button located below the chamber scope monitor if it is not already on.

7. Open load lock door using metal handle.

8. With exchange rod in **UNLOCK** position (turned **CW**), load sample holder onto rod.

9. Turn exchange rod **CCW** to **LOCK** position.

10. Close door and press **OPEN**. When the load lock is pumped and door is open, SEM will beep.

11. Slide exchange rod **ALL** the way into the chamber.
   a. **ATTENTION:** NOT inserting the rod in all the way may result in a stuck sample and/or damage to the stage.

12. Turn the exchange rod **CW** to the **UNLOCK** position and pull the rod **ALL** the way back until it snaps into place.

13. Press the **CLOSE** button.

14. Press the **HOME** button in the upper right corner of the PC-SEM interface.
III. Start Up

1. Ensure the SEM is in High-Mag Mode (there should be no LM in the Mag display.)
   a) Use the H/L button to the right of the Magnification display to toggle between high and low mag modes.

2. Once the stage is done moving to the Home position, Load Default Operating Conditions.
   a) Click on menu Setup > Condition Load …
   b) On the newly opened Setup window, select the Op. Cond tab
   c) Select Load Memorized Condition
   d) Select Default.pm1 and click Load at the bottom of the window.
   e) Click YES twice.
   f) Close the Setup window.

   Note: This will load a standard set of operating conditions that will be generally good for finding your sample and also good for viewing some samples. You will probably need to adjust at least one of the settings later, depending on your needs and your sample.

3. Turn on the High Voltage by pressing the red ON button in the upper-left corner of the software.

4. You will see a prompt that displays the sample dimensions. If correct, click OK.
   a) If these are NOT correct:
      i. Click cancel.
      ii. Select the Stage tab along the right side of screen.
      iii. Click the Set button under specimen size to enter the correct Size (width) of your sample.

   Note: Height should always be set to Standard height.
5. Ensure **scan speed** is set to **Fast 1**.

6. Ensure SEM is in **Low-Mag** Mode.
   a) Use the H/L button to the right of the Magnification display to toggle between high and low magnification modes.

   **NOTE:** **LM** will appear next to the magnification when the scope is in Low Mag Mode.

7. Reduce the magnification **ALL THE WAY** (Turn Mag knob on control panel **CCW** until it beeps).

8. Adjust the brightness and contrast (**ABC** button) to visualize the center of the sample holder.

9. Use joystick to find your sample.

10. Return to **High-Mag** mode by clicking the H/L button. (**LM** box should disappear from the Mag display.)

11. Focus on the **tallest point** on the sample to determine the working distance (**WD**). (The number in between **voltage** and **magnification** on the image display.)

12. Ensure the **WD** is equal to, or greater than the **Z height** (found under the **Stage** tab on the right side of the screen).
   a) If **WD** < **Z**, remove your sample from the SEM and recheck with height gauge.

13. To change the **Z height** of the stage, under the **Stage** tab on the right side of the screen type in the desired **Z** height and press **Go**.
   a) The allowable range will be displayed in blue. (i.e. **[1.5 ~ 40.0]**mm)
   b) **CAUTION:** In addition to checking the **WD** against the **Z** height, you should also watch the sample on the chamber scope as it moves closer to the lens.
   c) Very short **WD** (<4mm) for the high resolution imaging with the **Upper Detector**.
   d) Longer **WD** (>8mm) for using the **Lower Detector**.
   e) 15mm is the analytical working distance for doing EDS.

   **Note:** The actual **working distance** displayed on the image should be 15mm, this will differ slightly from the stage **Z** height.
14. For High-mag work, once the sample is in position, lock the stage by clicking the Lock button in the upper right corner of the screen. 

**NOTE:** This will need to be released before the Z or Tilt of the stage can be moved again.

15. **Flashing** – If a red/blinkng **Please Flash** appears across the top of your image, the tip must be flashed. **Do not flash the tip unless prompted.**
   a) Ensure the beam is off.
   b) In the HV menu on the left side of the screen click Flasing.
   c) Make sure Intensity is set to 2, click Execute.
   d) When complete turn the beam back on and redo the alignments.

   ![Graph showing reduction, stability, and instability phases](image)

   Flash 15 -45, 8-12 hours

   e) The “flash” procedure helps to establish stable operation of the cold field -emission electron gun by driving excess adsorbed gas molecules from the gun’s cathode, or electron emitter.
IV. Alignment

1. Initial alignments should be done starting at **at least 10,000x**.
   a) Alignment should always be done at a **higher** magnification than what you will be collecting images at.
   b) Focus and alignment is best obtained using a small **round** feature.

2. Click the **Align** button.

3. Select **Beam Align** and center the bright spot using the **X/Y Stigma/Align** knobs on the control surface.

4. Select **Aperture Align** and use the **X/Y Stigma/Align** knobs to minimize any image translation.
   a) You want the image to be pulsing in place and not moving on and off the edge of the screen.

5. Use the same method to minimize translation for **Stigma Align X** and **Stigma Align Y**.
   **Note:** **YES**, adjust both the X and Y knobs for each of these.

6. Exit the alignment menu.

7. Use Focus knobs to focus the image.

8. **EXTREMELY IMPORTANT:** Adjust the **X/Y Stigma/Align** knobs to correct for any astigmatism.
   a) To do this you can select **Red1** for the reduced size screen.

II. Image Capture

1. Select the Red 1 scan speed

2. Increase the Magnification on the area of the sample you want to image

3. Focus the image using the Focus knobs (Coarse, then Fine.)
   a) The image is in focus when no stretching of features is seen.

4. Adjust each Stigmator knob (individually, not simultaneously) until the image quality is improved.

5. Focus the image again.
   a) If stretching can still be seen, increase Magnification and repeat step 2 thru 4 as many times as necessary.

6. Reduce Magnification to desired level.

7. Adjust Brightness/Contrast using ABCC button and/or Brightness and Contrast knobs on the control surface.

8. To set the capture scan speed, right-click the Image Capture button.
   a) For slow scan image capture: select Slow, the resolution and the time of the scan. The default setting is shown in the example.
   b) For frame integration, select Fast scan mode and the number of frames to integrate.

9. Click OK.

10. Left-click the Image Capture button to acquire the image.

11. Images taken will appear in the Captured Images window at the bottom left of the screen.

12. Ensure the Embed box is checked.

13. Select images you want to save and click the PCI button.

14. If not open already, Quartz PCI will open automatically.
15. From PCI, you can save multiple ways.
   a) To save only a .TIF file, select **File > Export**.
   b) To save .PCI and .TIF files at the same time, select **File > Save As**.
   c) To Export all images at once, select **File > Export All** or **Save All**.

16. **Note**: PCI is available on other computers in the main lab. If you save the PCI version of the file you can edit your images or take measurements later.
17. **Extra Special Note**: If you are taking multiple images of the same sample and don’t want to name each image manually:
   a) Open the Quartz PCI software from the Windows Start Menu.
   b) From the PCI software File > Series Name and enter a project name. All images transferred to PCI from the SEM software will be named: series01, series02, etc.

III. **EDS Parameters**

1. Turn **OFF** chamber camera.
2. Accelerating Voltage set to appropriate level.
   a) Usually **15-30keV**, but can be lower depending on your sample.
3. On the **SEM** Tab on right side of screen
   a) **Probe Current** set to **HIGH**.
4. Area to be analyzed at **15mm** (+/- 0.2mm)
   a) This **IS NOT** the Z height of the stage displayed on the right side of the screen under the **Stage** tab.
   b) Your sample is at 15mm when the area of interest is in focus at 15mm. (Shown by the **WD**.) You will need to move the Z Height of the stage to get your sample to this position.
5. **Ie** may be increased to increase x-ray signal.
IV. Sample Specific Adjustments

Not every sample is best viewed under identical conditions. The default settings are generally good for many samples, but not everything. You’ll need to adjust the operating conditions to maximize the type of information you need from your sample. View charts on page 6 for more.

1. The HV menu (Can be opened by clicking the black box with the Vacc and Ie in it.)
   a) **Vacc**: Accelerating Voltage – variable from 500V-30kV. Change using the drop down menu.
      i. **Higher Voltage** = better signal, more beam penetration, smaller spot size.
      ii. **Lower Voltage** = lower signal, less beam penetration, better surface detail, lower resolution.
   b) **Ie**: Emission Current – typically set to 10-15uA for imaging.
      i. **Higher Current** = more signal, more charging. Needed for EDS.
      ii. **Lower Current** = less signal, less charging.
   c) **Emission Adjust** - checked by default.
   d) **Deceleration Mode** - unchecked by default.
      i. Deceleration mode allows the user to apply a negative bias to the sample stage which effectively decelerates the primary electron beam, allowing for landing energies as low as 100V. The beam can be decelerated up to 2.5kV. Ex: Setting Vacc to 3kV and V deceleration to 2.5kV will give a landing energy of 0.5kV.

   **IMPORTANT**: Sample MUST be well grounded.
2. The SEM tab

a) Detectors

i. (T)op Detector – High-Angle Backscatter and Beam Deceleration. Works best between 1.5-5mm WD.

ii. (U)pper Detector – High resolution SE signal and Low-Angle Backscatter. Works best between 1.5-15mm WD.

iii. (L)ower Detector – Directional SE. Lower resolution than Upper Detector, shows less charging. Works best >8mm WD.

b) Modes

i. SE – Secondary Electron Signal. Standard SEM signal. Any combination of the 3 detectors may be used.

ii. HA-BSE – High-Angle Backscatter. From Top Detector only. Can be used in conjunction with the Upper detector or Deceleration Mode.

iii. LA-BSE – Low-Angle Backscatter. From Upper detector only. Can be used in conjunction with the Lower detector or Deceleration Mode.

iv. SE(L) – Bias plates redirect signal only to the Lower detector.

c) Operating Condition

i. Probe Current

1. Norm – For normal SEM operation.

2. High – Causes the beam to avoid the obj. aperture. Gives high current necessary for EDS. Increases signal and charging, decreases resolution and noise.

ii. Cond. Lens 1 – Set to 5 by default.

1. Higher values increase resolution and decrease signal.

2. Lower values decrease resolution and increase signal.
V. Shut Down and Sample Removal

1. **Release** stage (if it was Locked).
2. Click the **EXC** button to send the stage to the exchange position.
3. Click Acceleration Voltage **OFF** button.
4. Turn Chamber Scope **ON** (if it was off).
5. When stage is done moving, press **OPEN** button on airlock.
6. When SEM beeps, push the exchange rod (in the **UNLOCK** position) **ALL THE WAY** into the chamber until the blue **XC** light comes on.
7. Turn the knob **CCW** to **LOCK**, pull the rod **ALL** the way back until it clicks in place.
8. Press **AIR** button on airlock.
9. When airlock beeps, airlock is at atmosphere, open door **with handle** **NOT THE ROD**.
10. Turn rod **CW** to the **UNLOCK** position and remove sample holder.
11. Ensure rod is pulled completely out and clicks in place.
12. Close door **with handle** and press **EVAC** button on airlock.
13. Turn Chamber Scope **OFF**.
14. Log out from the NUcore system.
VI. Additional Information

<table>
<thead>
<tr>
<th>Ie</th>
<th>Signal</th>
<th>Charging</th>
</tr>
</thead>
<tbody>
<tr>
<td>Higher</td>
<td>↑</td>
<td>↑</td>
</tr>
<tr>
<td>Lower</td>
<td>↓</td>
<td>↓</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>kV</th>
<th>Resolution</th>
<th>Contrast</th>
<th>Charging</th>
<th>Edge Effects</th>
</tr>
</thead>
<tbody>
<tr>
<td>Higher</td>
<td>↑</td>
<td>↓</td>
<td>↑</td>
<td>↑</td>
</tr>
<tr>
<td>Lower</td>
<td>↓</td>
<td>↑</td>
<td>↓</td>
<td>↓</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Z Height WD</th>
<th>Resolution</th>
<th>Upper SE Signals</th>
<th>Depth of Field</th>
</tr>
</thead>
<tbody>
<tr>
<td>Larger</td>
<td>↓</td>
<td>↓</td>
<td>↑</td>
</tr>
<tr>
<td>Smaller</td>
<td>↑</td>
<td>↑</td>
<td>↓</td>
</tr>
</tbody>
</table>
VII. Frequently Asked Questions

1. There is no image from the microscope.
   a. Someone hit Ctrl+Alt+Del which froze the video feed.
      i. Go to File > Password Lock
      ii. Enter password: semuser
   b. The Beam Align might be way off.
      i. Open the Align menu and check the beam is centered over the crosshairs.
      ii. If you can’t see the spot, turn up the contrast.

2. Before or after sample exchange, the SEM is making a loud, intermittent beeping.
   a. The exchange rod is probably not pulled all the way out. The inner airlock door will not open if the rod is not pulled all the way out.

3. The SEM is making a single long beeping noise.
   a. The Liquid Nitrogen dewar is low.
      i. If it is after hours or on the weekend, feel free to fill it.
   b. The airlock door was not shut properly.
      i. Hold the door in firmly to achieve a proper seal.

4. The scroll pump in the chase is making a lot of noise.
   a. The airlock door was not shut properly.
   b. Hold the door in firmly to achieve a proper seal.

5. The software is frozen / freaking out.
   a. Restart both computers. Left computer FIRST, then right.
      i. Left computer
         1. user: semsuer
         2. pass: semuser
      ii. Right computer
         1. user: INCA
         2. pass: INCA

6. A red/blinking Please Flash appears across the top of your image.
   a. The tip must be flashed. Do not flash the tip unless prompted.
      i. Ensure the beam is off.
      ii. In the HV menu click Flashing.
      iii. Make sure Intensity is set to 2, click Execute.
      iv. When complete turn the beam back on and re-do the alignments.