

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 SHyNE Resource (NSF ECCS-2025633), the IIN, and Northwestern's MRSEC program (NSF DMR-1720139).”



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<http://www.nuance.northwestern.edu/epic/publication-acknowledgements/index.html>

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I. Policies and Introduction

Reservations

S-3400 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 3400, 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

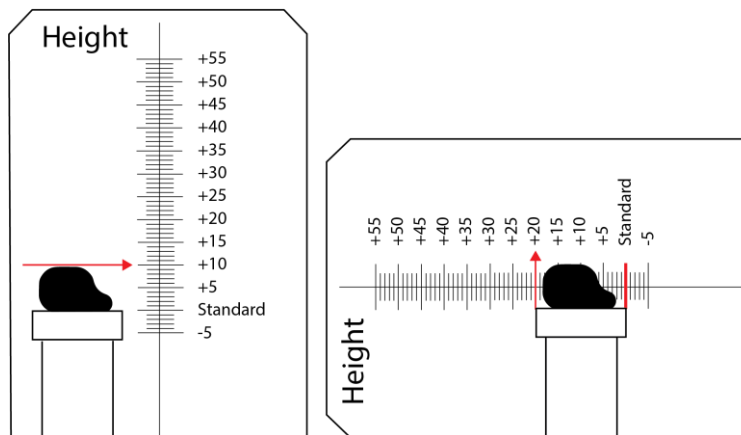
During your session, you may store image data to your own folder within the S3400 folder on the EPIC_SEM (S:) drive. The SEM server is accessible through a computer in the lab. EDS data must first be saved onto the AZtec projects folder on the (D:) and then transferred onto your folder on the S:\\ drive. 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

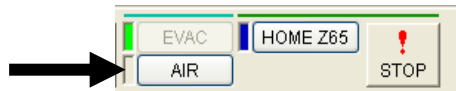
1. Please wear gloves when handling any compents and samples that will go into the SEM.
2. Do not install any software onto the SEM’s PC.
3. Do not insert any flash drives into any microscope computer!

II. Sample Loading

1. Log into the NUcore system
 - a. Under the “My Reservations” tab, click ‘begin reservation’ next to the appropriate session
2. Mount sample and prepare the sample holder
 - a. Place pin stubs in the holes of the round sample holders and tighten with the set screw
 - b. Mount larger samples directly on the top of the sample holder with carbon tape
3. Use the sample gauge to measure both the sample Height and Size (width).
 - a. Measure the width from the line labeled “standard.”
 - b. Put the sample at eye level to measure the height of the **highest portion** of the sample.



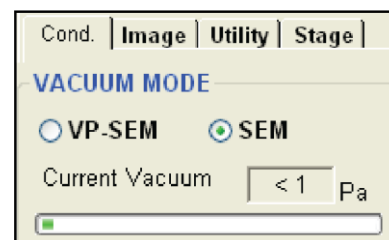
- c. Enter these values into the dialog box, rounding **up** to the next value if it is between two choices. ***This is critical so you don't crash your sample into fragile components around the objective lens.***
4. Vent the chamber by pressing the **AIR** button in the upper right corner of the PC-SEM interface.



- a. The stage will automatically be sent to the **Home Z65** position and the chamber will vent.
 - b. The chamber is vented when it beeps softly **three** times.
5. Carefully open the SEM door using handles on either side and load your sample holder onto the stage.
 6. Hold the chamber door shut and press the **EVAC** button on the front of the SEM.

7. Turn on Chamber Scope with the **Camera On/Off** button below the chamber scope monitor to the left of the PC monitors.
8. A dialog box will appear on the screen. Input the size (width) and height of the sample and click **OK**.
9. When the chamber reaches high vacuum (~90 seconds), the light by the EVAC button will stop blinking and the SEM will beep once loudly.
10. Ensure the proper Vacuum Mode is selected.

- a. **High Vacuum Users:** Ensure **SEM mode** is selected under the **COND** tab (right side of screen)
- b. **Low Vacuum Users:** Ensure **VP-SEM mode** is selected under the **COND** tab (right side of screen)

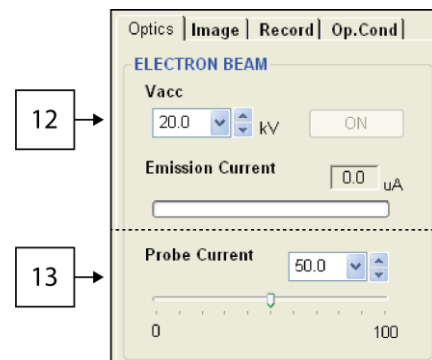


11. If the **Setup** tab on the left side of the screen is NOT open, select **Setup > Optics Setup**
12. Choose your accelerating voltage level (Vacc).

- a. If you are unsure what kV to use, start low (5kV). See section IX. (pg 10) for more information on the effects of accelerating voltage.

13. Set probe current to 50 to start. (This can be changed later.)

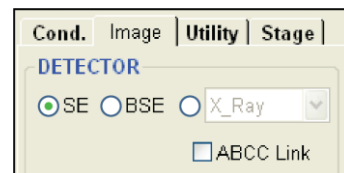
- a. The probe current controls the strength of the condenser lenses in the column.
 - i. Higher values of probe current will result in greater signal strength but poor resolution.
 - ii. Lower values will give greater resolution, but reduced signal strength and a noisier image.



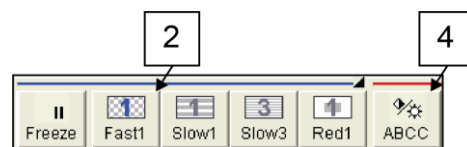
14. Turn the beam on by pressing **ON** in the upper left corner of the control screen.

III. Start Up

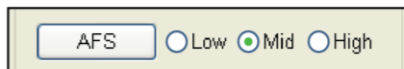
- Under the **Image** tab on the right side of the screen, ensure the **SE detector** is selected.
 - Low Vacuum Users:** choose either the **BSE** or **ESED** detector. The **Slow 1** scan speed will help when viewing the sample in these modes.



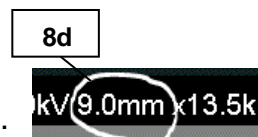
- Ensure the scan speed is set to **Fast 1** (in the top left part of the screen).



- Lower the magnification **all the way** (Mag knob **CCW** until it beeps).
- Adjust brightness and contrast (**ABCC** button).
- Use the trackball to move that stage X/Y and locate your sample.
- Focus on the surface of your sample.
- In the **Optics Setup** window, press **AFS** to auto-saturate the tungsten filament source.
 - IMPORTANT:** Whenever the **kV** is changed, **always** re-saturate the filament with something in focus by pressing **AFS**.



- For higher resolution imaging or EDS, you will need to move the sample closer to the lens.
 - From the Stage menu on the right of the screen, select the **Analysis** button to send the stage to a Working Distance of 10mm.
 - Hit the **STOP** button (upper right corner of the screen) to stop movement of the stage **if** your sample gets too close to the lens.
 - Focus on the tallest point of the sample.
 - If WD is **less than 10mm**, send sample back to **HOME Z65** position. Remove sample from chamber and measure it again. *Having a correct sample height will prevent you from crashing into the lens.*
 - If WD is **equal or greater than 10mm**, you may continue to decrease the WD by going to the **Stage** tab and adjusting the Z value (5 mm is the minimum.)

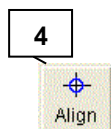


IV. Alignment

1. Insert the desired aperture by turning the aperture knob. (See section IX. for more information)
 - a. Turn knob **CW** for smaller aperture.
 - b. Turn knob **CCW** for larger aperture.
 - c. 1 is the largest aperture, 4 is the smallest.
 - d. Typically aperture 2 or 3 is used.

2. Increase magnification to at least **5,000x** and find a small round feature on your sample to use for alignment.

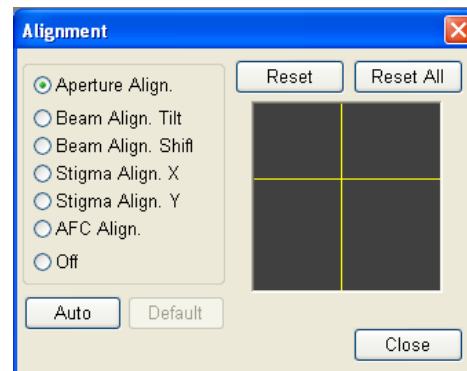
3. Focus your area of interest.



4. Click the **Alignment** button.

5. Select **Aperture Alignment**.

- a. The focus wobble will turn on automatically.
- b. Adjust the **X** and **Y Stigma/Alignment** knobs on the control surface to minimize translation of the image.



6. Select **Beam Align. Tilt** and **Beam Align. Shift**

- a. Adjust both the **X** and **Y** knobs, one at a time, until you find the brightest image possible.
- b. Repeat **a.** for **Beam Align. Shift**.

7. Select **Stigma Align. X** and use the **X** and **Y Stigmator/Alignment** knobs on the control surface to minimize translation of the image.

- a. Adjust **both X** and **Y** knobs to align each of the **X** and **Y** stigmators.

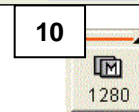
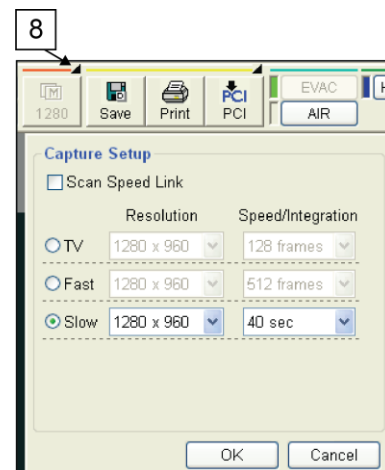
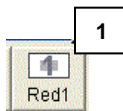
8. Select **Stigma Align. Y** and repeat step 7.

9. Do nothing for **AFC Align**.

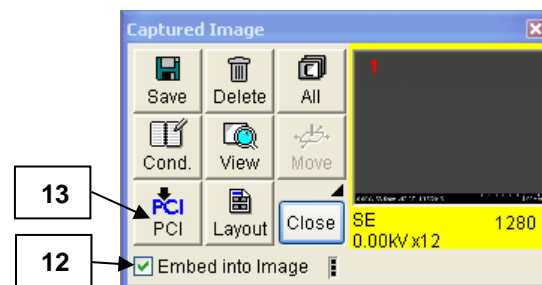
10. Close the alignment menu.

V. 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 steps 2 thru 4 as many times as necessary.
6. Return to a normal **Scan Speed** and reduce the **Magnification** to the 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. This is a good option for samples that are charging during a slow scan.
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 of the screen.
12. Ensure the **Embed into Image** box is checked.
13. Select images you want to save and click the **PCI** button. (If not open already, **Quartz PCI** will open automatically.)
14. 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**.

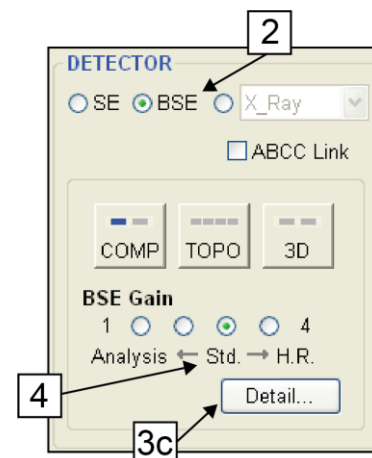


left of



VI. Backscatter Detector


1. Ensure the Chamber Scope is turned **OFF**.
2. Select the BSE detector by clicking on **BSE** in the **Image** tab.
3. The **BSE** detector has 5 segments, which can be turned on and off independently for different modes of operation.
 - a. **Comp** uses all 4 axially mounted segments for good compositional contrast.
 - b. **Topo** and **3D** have different configurations to enhance topographic contrast.
 - c. If you press the **Detail** button, you can fully configure the detector.
4. The **BSE Gain** setting is used to control the preamplifier gain on the detector, with the default setting in the third position.
 - a. For low signal applications (ie, low kV, low Probe Current, etc.) you may need to increase the gain to get a good image.
 - b. If the contrast is saturated (too bright), decrease the gain.



VII. EDS with AZtec

1. Turn **OFF** chamber camera.
2. Set the Accelerating Voltage to an appropriate level.
 - a. Usually **15-30keV**, but can be lower depending on your sample.
3. Select a larger Aperture to increase the current. (Usually #1 or #2, though no Aperture may be selected as well.)
4. Set the Area to be analyzed to **10mm**.
 - a. This **IS NOT** the **Z** height of the stage displayed on the right side of the screen under the **Stage** tab.
 - b. The **WD** should read ~10mm (+/- 0.2mm) when the area of analysis is in focus.
5. **Probe Current** may be increased to increase x-ray signal. Realign SEM after changing Probe Current.

VIII. Shut Down and Sample Removal

1. Click the **OFF** button in the upper left corner of the  screen.
2. Click the **AIR** button to automatically send the stage to the Home Z65 position and vent the chamber.
3. After the chamber vents (you will hear 3 quiet beeps), gently open the door and remove your sample.
4. **If you were using low vacuum mode**, switch the SEM is back to **SEM mode** under the **Conditions** tab.
5. While holding the door firmly shut, press the **EVAC** button to evacuate the sample chamber.
6. **Please dispose of all gloves, carbon tape, stubs, etc before leaving the SEM.**
7. Log out in the NUcore system.

IX. Additional Information

Probe Current	Resolution	Signal	Charging
Higher	▼	▲	▲
Lower	▲	▼	▼

kV	Resolution	Contrast	Charging	Edge Effects
Higher	▲	▼	▲	▲
Lower	▼	▲	▼	▼

Aperture	Resolution	Signal	Depth of Field
Larger	▼	▲	▼
Smaller	▲	▼	▲

X. Frequently Asked Questions

1. Login Usernames and Passwords

- a. SEM computer username/pass – **semuser**
 - b. EDS computer username/pass – **oxford**
 - c. NUANCE SEM File Server
 - i. Username: **NUANCE_User**
 - ii. Password: **Nuance**
-

2. **PROBLEM:** The INCA or AZtec EDS software has frozen up.

ANSWER:

- a. Close the EDS software and run Tidy Up (icon on desktop). Restart EDS software. If that does not work, go to next step.
 - b. Close all software on BOTH SEM and EDS computers and restart them both. Start the SEM computer first, then the EDS computer.
-

3. **PROBLEM:** I turn the HV on and can't see anything.

ANSWER: Try each of these in turn. If one doesn't work, go to the next one.

- a. Ensure **le** is reading something (other than 0)
 - i. If the **le** reads 0, the filament is blown OR there is a communication issue somewhere. Restart the SEM computer. If it recurs find lab staff.
- b. Turn down **Mag** all the way.
- c. Click **ABCC**
- d. Remove the Aperture all the way.
 - i. If you can see things now it means the Aperture alignment is way off, either physically or electronically. Find lab staff.
 - ii. Hit **AGA** if you still don't see an image with the aperture out.
 - iii. The EDS software may have control of the SEM. Run Tidy Up on the EDS computer.
 - iv. If Tidy Up doesn't work, close all software on BOTH SEM and EDS computers and restart them both. Start the SEM computer first, then the EDS computer.