

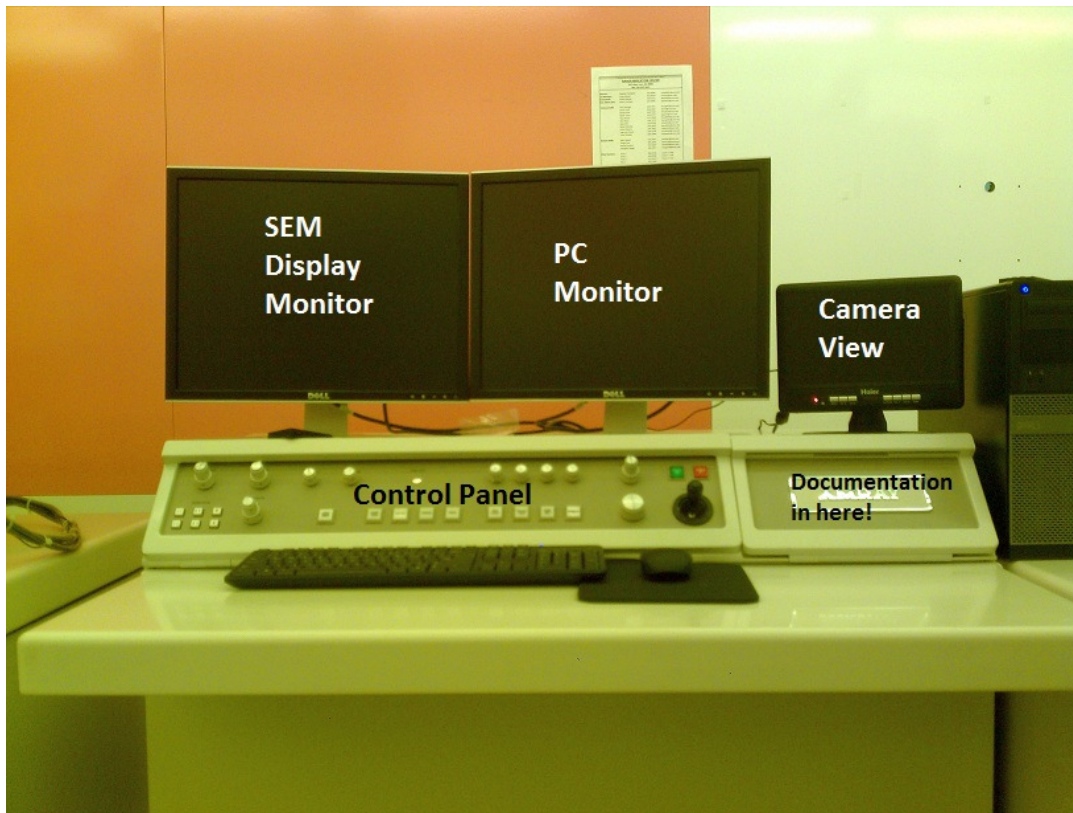
Amray 3600 FESEM

Standard Operating Procedure

v2.2 – modified 5.13.13 by Bryan Cord

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General Notes

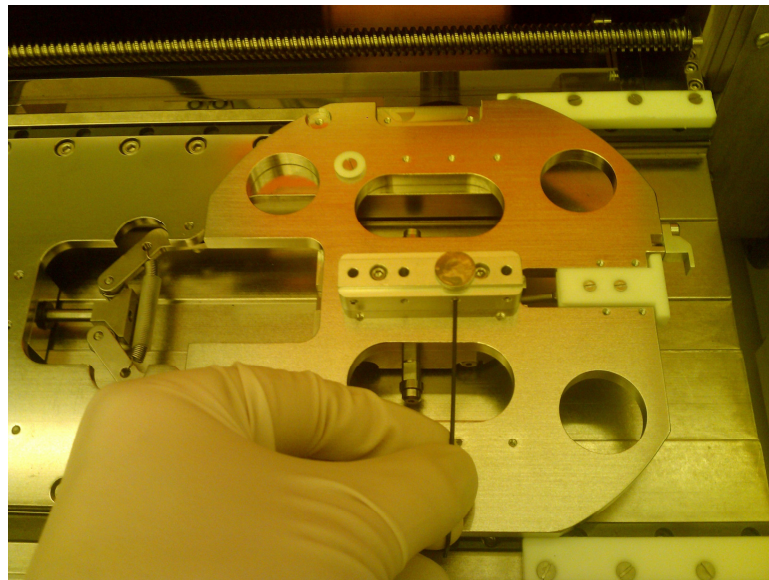
- This guide is intended as a quick reference for doing basic imaging on the Amray 3600. It assumes you're familiar with the system and is NOT a substitute for training. Contact Bryan Cord (bcord@umn.edu) for training if you need it.
- The system has a lot of advanced capabilities that aren't covered here. Fortunately, it also has a comprehensive help file. The "help" menu will give you a detailed explanation of every element in each of the different control windows. There's also a manual (linked on the desktop) that goes over everything in a more "how-to" format.
- Due to low-level incompatibility between Windows 7 and the 35-year-old SEM control hardware, moving the mouse from the Windows PC screen to the SEM display screen will cause the mouse to "double" across both screens. The mouse pointer on the PC screen is just a ghost and won't affect anything; move the pointer off the SEM display to get the Windows pointer back.
- Good SEM imaging takes practice, especially on this system. If you're not getting the resolution you feel like you should be getting, experiment with different settings or ask for help.

Sample Loading

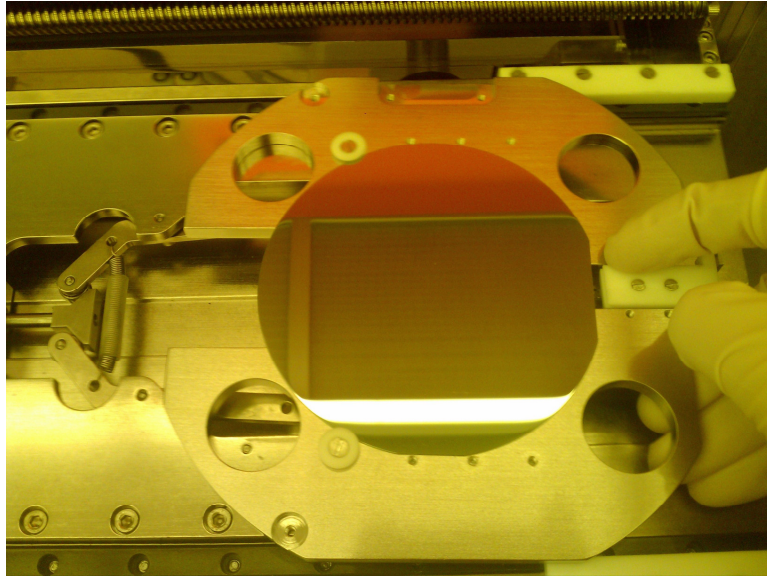
1. For small pieces, mount your samples on one of the provided stubs using carbon tape discs. Please do not tape samples directly to the block!
 - a. To make life easier, you may want to put some gold nanoparticles near the part of the sample you want to image to make it easier to locate in the SEM. Use the syringe in the toolbox to deposit $\sim 5\ \mu\text{L}$ of particles from the bottle on your sample. Make sure you put the syringe back in its protective tube when finished.



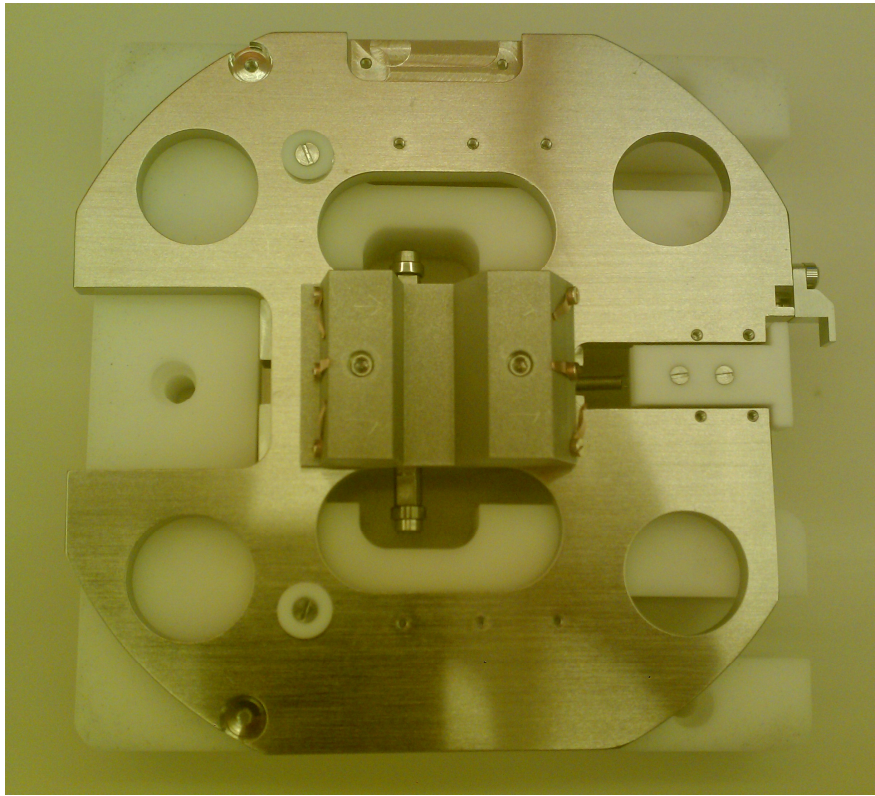
- b. If you're using nanoparticles, make sure you give the drop time to dry before pumping down the airlock! Even sucking up a small amount of water will shorten the lifetime of the airlock pump.
2. Place the stem of your sample stub in one of the holes on the raised block in the center of the chuck. Use the small allen wrench near the load lock to tighten the set screw that holds it in place.



3. For full wafers, remove the piece-part block from the chuck using the larger allen wrench in the toolbox and put it in a safe place. Clamp your wafer into the chuck using the spring clips. Be sure to replace the piece-part block when finished!

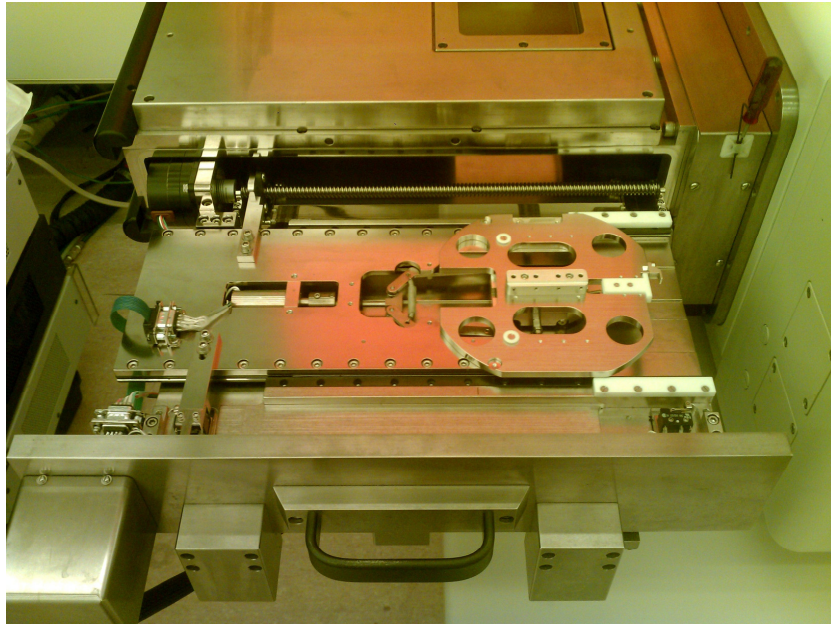


4. To view samples at an angle (for cross-sections and the like), use the provided angle chuck. Mount it on the wafer holder using the same two screws as the piece-part chuck. The arrows scribed on top of the block should point toward the SEM chamber when the chuck is attached properly. Attach your sample to either the 45 or 85 degree tilt using the copper clips. If the clips are in the way feel free to take some of them off while you're working as long as you put them back on when you finish.

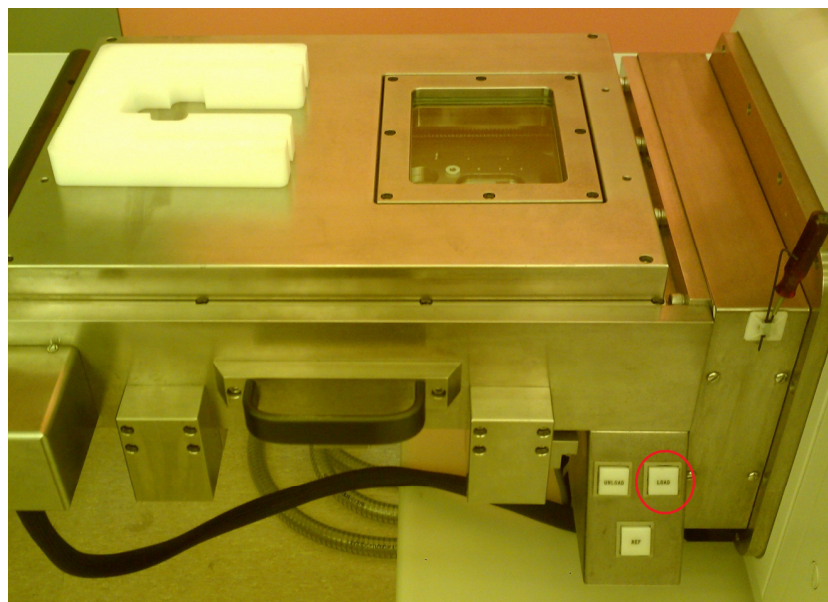


System Loading

1. Slide the chuck, with your sample mounted on it, into the load lock per the picture below.



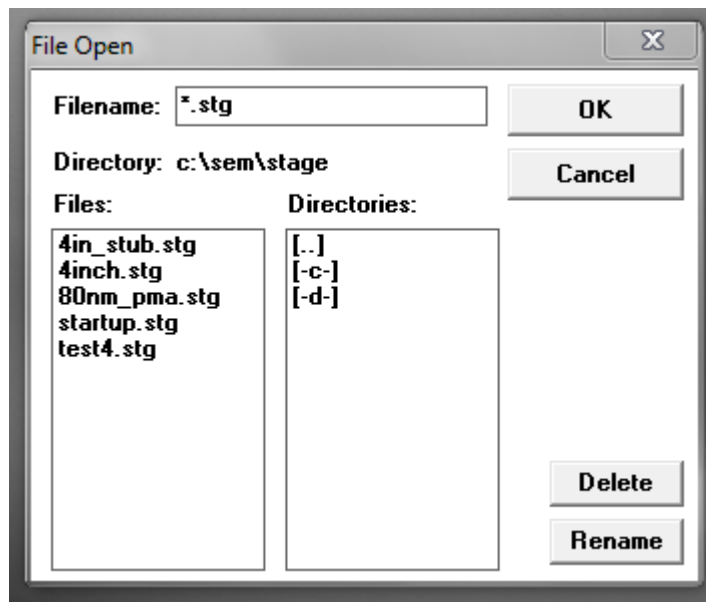
2. Close the load lock door and press the “Load” button on the nearby panel



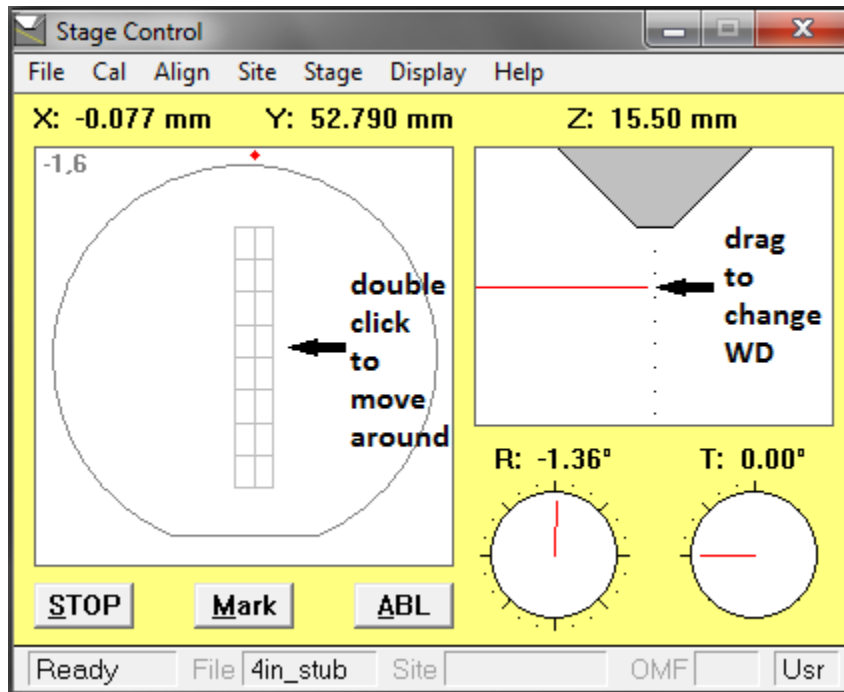
- a. You can also use the “Sample load” command in the “Vac” menu on the “SEM Control” software panel to do this. If you’re getting errors about stage referencing, this way usually works better.
3. If the SEM software isn’t already on, start it using the “Start SEM” icon on the desktop. If the PC is logged out for some reason, use the username **3600** without a password to log

into it.

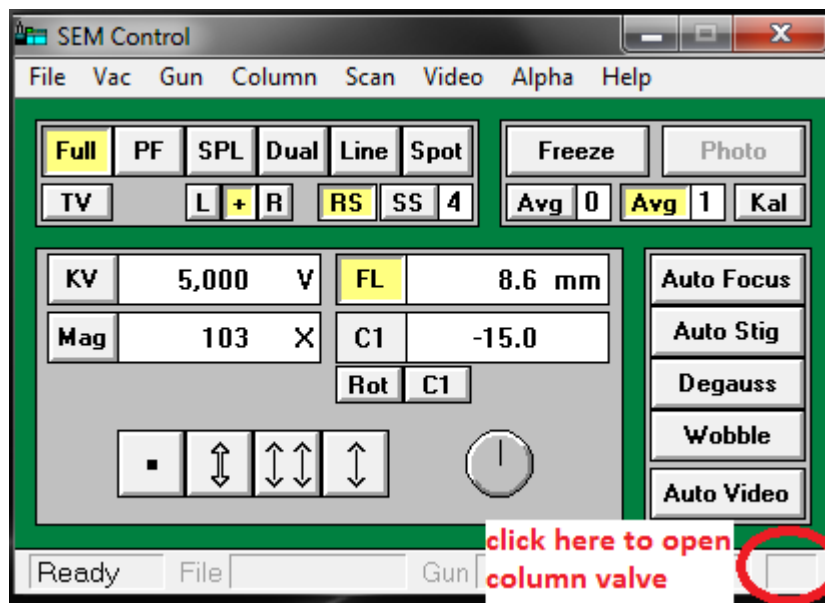
4. Wait a few minutes while the system evacuates the load lock and transfers the chuck into the chamber. When you see the gate valve door close on the camera display screen, the sample is loaded.
5. Load the correct holder profile for the holder with the File/Open command in the “Stage Control” window. If the piece-part block is attached, use “4in_stub.stg”; use “4inch.stg” if you’re imaging a full wafer and “angle.stg” if you’re using the angle chuck. The holder profile is what keeps the stage from crashing into the final lens, so don’t forget to do this!



6. Double-click on the stage map in “SEM Control” to move the stage to the approximate location of your sample. If using the piece-part stub, the crosshairs represent the different mounting sites along the stub.
7. Set the z-axis to the approximate working distance you’d like to use (6-8mm is good) by dragging the line in the Z window of the Stage Control window up and down. Make sure you set the working distance in the SEM Control window too (the “FL” setting) so the system knows where to focus.

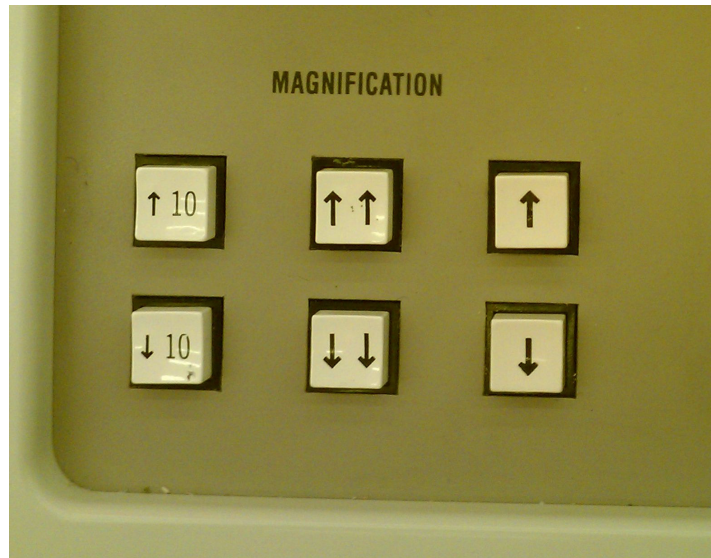


8. Set the operating voltage in the SEM Control window (the “KV” box) to the desired value. 5000V is fine for most imaging, but when imaging surfaces or thin features it may be useful to go to 1-2 kV.
9. Set the probe current by adjusting the “C1” value in the SEM Control window. A higher (larger negative) current will give you better signal to noise but increase charging and contamination. -12 is a good starting point with this setting; you can go to -15 or higher if the image is too noisy, or -10 or even lower if charging is a major issue.
10. Open the column valve, either by pressing the “column isolation valve” button on the column controls or by double-clicking the blank box in the lower-right corner of the SEM Control window.

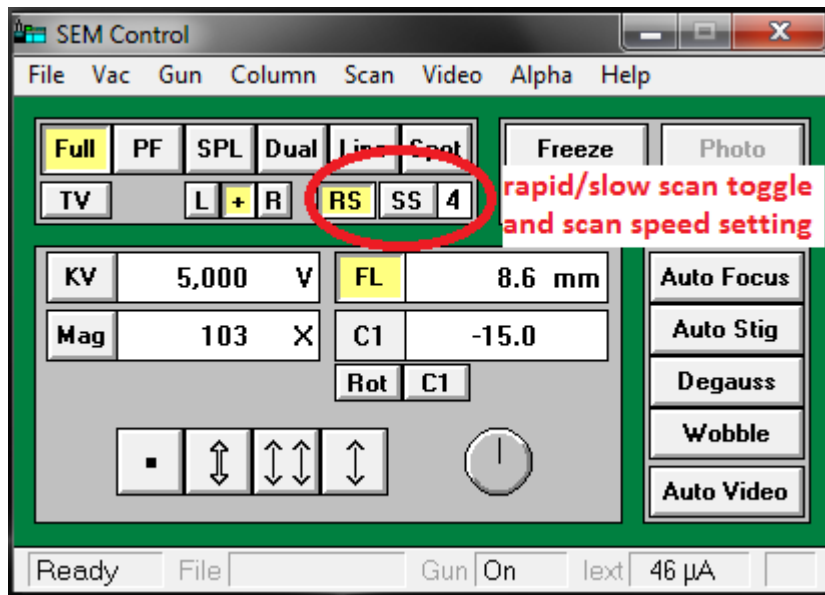


Imaging

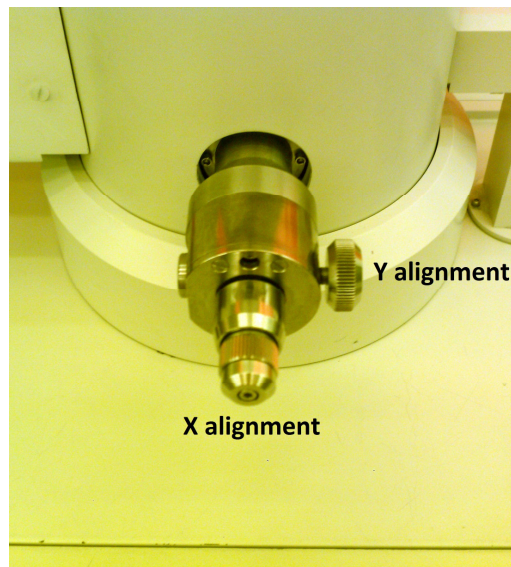
1. To change magnification, use the six buttons on the lower-left corner of the console. You can also type new magnifications in SEM control or use the labeled function keys to get to pre-set magnification levels. Moving around can be accomplished with the joystick, the stage control software, or the beam shift knob.



2. Brightness and contrast can be adjusted using the knobs of the same name on the console. The “auto video” setting will automatically optimize brightness and contrast.
3. Focus can be changed using the large knob under the joystick on the console. Note that the autofocus/auto-stigmation capabilities don’t really work well and are not recommended.
4. The easiest way to calibrate your imaging settings is to zoom out to the lowest mag and find an easily-visible “garbage” feature like a scratch, dust speck, etc. Try not to use the thing you’re actually imaging to calibrate your settings, as charging and/or contamination could ruin it before you get a chance to actually image it.
 - a. Moving around can be done with the joystick, the SEM display screen (right-click to change the mouse icon into a target, then double-click the target to bring the double-click site to the screen center), the Stage Control window, or the X and Y beam shift knobs.
 - b. This is much easier to do with a fast scan speed. Turn off “slow scan” on the console and adjust the “RS” value to 1 or 2 in the SEM Control window (click on “RS”, then right-click to lower the value or left-click to raise it) for best results.



5. Once you've found a feature to use for calibration, zoom in a bit and twist the focus knob to bring it into focus. Check the working distance (FL) setting in the SEM Control window and make sure it's approximately what you were looking for; drag the z-axis up or down accordingly (in the Stage Control window) if it isn't and refocus.
6. With the feature in focus, degauss the detector using the "Degauss" button in the SEM Control window. This will cause the image to jump out of focus. Refocus the image and degauss again. The detector is fully de-magnetized when degaussing doesn't affect the focus. Degaussing is critical if you're doing quantitative imaging, as a magnetized detector can cause spatial distortions in your image.
7. Zoom in on your calibration feature until you have problems getting it into focus. At this point, you'll need to align the aperture. Press the "wobble" button on the console (or use the equivalent software setting/hotkey). You should see the image wobbling in the x and/or y axes. Use the X and Y aperture alignment knobs on the column (see below) to adjust the aperture until the feature stops wobbling and just "breathes" in and out of focus in one place. Turn off the wobble when finished.



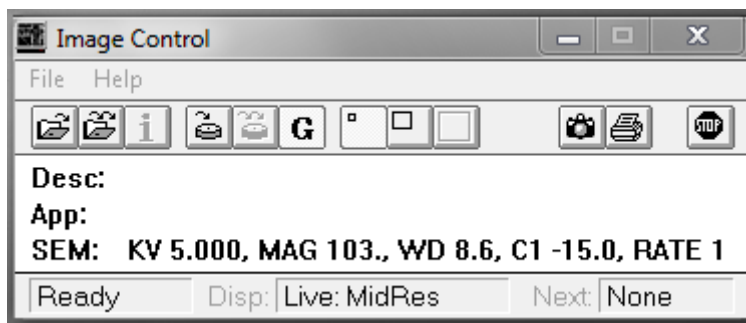
- a. If adjusting the apertures is causing the screen to go black, the beam is out of alignment. Stop the focus wobble and twist the x and y axes of the “beam alignment” knob until the image is as bright as possible, then continue.
 - b. Try not to lean on the column while adjusting the aperture knobs, as this will make aligning them even harder.
8. With the aperture aligned, continue to zoom in and focus until you’re again having problems focusing on a feature. Now focus as much as possible and adjust the x and y stigmators until the image comes fully into focus.
 - a. There are coarse and fine stigmator knobs on the console. For best results, move both fine stigmator knobs to the 12 o’clock position and use the coarse stigmator knobs to get as close as possible to the optimum setting. Then use the fine knobs to fine-tune, as well as compensate for any stigmatism drift that occurs while imaging.



- b. Continue to refocus while adjusting stigmatism.
 - c. Using a reduced scan field (pressing the “reduced field” button on the console) will let you use a much smaller image to adjust stigmatism. Since this image refreshes much more quickly than a full-screen image, you’ll get more immediate visual feedback that can make your life much easier. You can adjust the sized and position of the reduced field using the mouse on the display monitor.
9. Aperture adjustment, focus, and stigmatism are an iterative process. Continue to zoom in and adjust all three settings (using the focus wobble for aperture alignment) until you have a clear image at the desired magnification.

Saving Data

1. Once you have a good image with optimized settings, you'll want to save a high-quality scan of it. The best way to do this is to use a slow scan ("Slow Scan" button on the console) setting between 1 and 4 (click "SS" then left/right click to raise and lower the scan speed in SEM Control). Press "Freeze" during the scan to freeze the scan at the end of the frame.
 - a. It should be mentioned that the "Photo" button, which was originally connected to an old-timey film camera, doesn't do anything anymore.
2. You can add annotations if you want (see appendix) at this point. For measurement, you can simply draw a line/box on the image with the mouse on the SEM display monitor. To add extra text, use the "Alpha" menu in the SEM Control window.
3. To save images, use the "Image Control" window. You can change the resolution of the saved images here, as well as click or unclick the "G" icon to either save or not save (respectively) annotations and the data zone at the bottom of the image.



- a. If you're planning to publish an image, it's a good idea to save a high-res version without annotations and a low-res version with annotations. That way you'll know all the dimensions, but you can add your own (better-looking) decorations to the high-res image later.
4. Once you've got your save settings the way you want them, click the fourth icon from the left (or File/Save) to save your image. Because this is an old SEM, you're restricted to 8-character filenames. Live with it.
 - a. Try to save in the c:\Userdata directory, after creating a subdirectory with your name on it, just for the sake of organization.
 5. You can upload saved images to your UMN Netfiles locker for download later. Open Google Chrome (on the desktop) and log in using your X.500 username.
 - a. PLEASE DO NOT USE USB STICKS TO TRANSFER FILES! USB sticks are major virus-transmission vectors on Windows machines. Netfiles is fast and

convenient. You can access it from any PC at <http://netfiles.umn.edu>

- b. Anything left on the hard drive is subject to deletion at any time, so make sure anything important is backed up somewhere else.

Unloading

1. Close the column valve with either the button on the column or the control in the corner of the “SEM Control” window
2. Unload your sample by pressing the “Unload” button on the load lock or doing Vac/Unload in the SEM Control menu. The system will pump the airlock, unload your sample, and vent automatically. When the load lock button stops flashing, your sample can be removed.
3. Remove your sample from the stage and clean off any carbon tape or other residue you may have left in the process
4. The load lock stays vented when the system isn’t actively loading and unloading, so make sure the door is closed to minimize contamination.

Troubleshooting

Problem	Solution
Stage has crashed into the final lens; alarm is beeping.	Find the BNC cable plugged into a port with the ground symbol near it on the side of the main SEM chamber. Unplug that cable to stop the touch alarm and unlock the stage. Reference the stage with the “reference” command in the “stage” menu of the Stage Control window to drop it away from the final lens. Confirm that the lens isn’t damaged by focusing on something and make a note in CORAL that a stage crash occurred.
Stage has crashed into electron detector	Crashing into the electron detector is more problematic than crashing into the lens, since it doesn’t trigger the touch alarm. If the stage looks like it’s contacting the detector (check the IR cam) IMMEDIATELY hit the STOP button in the Stage Control. Re-reference the stage if necessary (see final lens crash procedure above) and drop the z-axis to 20mm or some other large working distance. Make a note in CORAL that a detector crash occurred.
Stage won’t move	The stage is probably out of reference. The lower-right corner of the Stage Control window will say UNREF in this case. Reference the stage using the “reference” command in the “stage” menu of the Stage Control window.
Loadlock reference error on SEM display	Re-reference the load lock, either by pushing the REF button on the load lock or using the “Reference Airlock” command in the “Vac” menu of the SEM Control window.
Can’t type user annotations onto the SEM display	Click the mouse on the PC monitor. For some reason, the PC monitor has to be focused for user annotations to work.
“Scan Rot” button won’t turn off (other buttons might do this too)	Push it twice quickly. You may have to do it a few times. I’ll get around to cleaning the contact on it eventually.

Typing on the PC just causes annotation letters to appear on the SEM display.	Go into the “alpha” menu in the “SEM Control” window and turn off “user annotation.”
Can’t get rid of unwanted annotations on SEM display	On the PC monitor, click the “SEM Control” window and hit ESC then 0 . Once you restart scanning, that should clear all annotations.
Can’t save images (TIFFERROR message when you try)	Due to a weird software bug, the system can’t save images if thumbnail images are displayed in an open directory window. If you have a directory window open, either close it or switch to list or detail view.
Software appears to be locked up	Reset it using the “Stop SEM” and “Start SEM” desktop icons. IMMEDIATELY run a stage reference (“reference” command in the “stage” menu of the Stage Control window) after doing this or you risk crashing the stage!
No image visible (black screen at all B/C levels)	If the column chamber valve is open (check that first), the beam is probably mis-aligned. Scan the “beam alignment” knobs (near the focus control) around until you find the setting where the image is brightest (at 5kV, this should be around 12 o’clock, but it could be very different at other voltages). Note that you’ll probably have to do quite a bit of aperture realignment after changing this setting.
Image goes black during aperture alignment	If the beam is partially mis-aligned, certain aperture positions may block it completely. When it goes black, use the beam alignment controls to find a position where the image is restored. You may have to adjust the beam and aperture alignments iteratively to get everything lined up just right.

Can't find features you're attempting to image	Check to make sure the video is working, first of all (see "No image visible" above). If that seems to be OK, try finding a large feature (scratch, speck of dust, nanoparticles) and get the column aligned and focused before you go looking for your features. If you're imaging very thin features, going to a lower beam voltage may be useful; try 2500V and work downward in 500V steps. You'll have to realign the column and aperture at each voltage, unfortunately. Set the high voltage back to 5kV when you're done! A useful trick for locating sparsely-placed features on a sample is to diamond-scribe a circle around the area of interest (assuming you can see it optically). Alternately, using the syringe in the toolbox to put a drop of gold nanoparticles on or near the area of interest can make locating your features much easier too. Make sure you let the drop dry before pumping down the airlock!
Can't image features smaller than 20-25 nm	This is likely a vibration issue. The source of the vibrations is currently unknown, but in the meantime imaging at night (when there's no construction and much less activity in the building) may give you better resolution. Alternately, use the Charfac SEMs for your ultra-high-res needs.
Can't image thin features or surface detail	Turn down the accelerating voltage. The system has a LEAP lens capable of going to voltages as low as 500V without distortion, so you have some headroom here. Lower voltage means more interaction between the beam and the surface, so you'll see more details and may be able to resolve thin features that were invisible at 5000V. Note that the tradeoff here is often increased charging, due to increased beam-surface interaction. No good way around that one.

Sample is charging	There are a few solutions to this. The most obvious is to make sure the sample is solidly grounded (carbon tape holding it on a metal stub will usually work for conductive samples). If possible, coat the sample with a thin layer of metal (preferably Au or Au-Pd) to aid charge dissipation. If you're still having problems, turn down the beam current by setting C1 to a lower negative value in the SEM Control window. -12 is the default; you can go all the way to -4 if needed. You'll need to refocus/realign after each change, so go down in steps of 2-4 for your own sanity. If that still isn't helping, you can sometimes work around charging by getting your focus/stigmation settings optimized at another site, then moving to your imaging site and quickly taking a single slow scan before charging has affected it much.
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