Tescan VEGA3 Scanning Electron Microscope

Standard Operating Procedure

Faculty Supervisor: Prof. Robert White, Mechanical Engineering (x72210)
Safety Office: Peter Nowak x73246 (Just dial this directly on any campus phone.)
(617)627-3246 (From off-campus or from a cell phone)
Tufts Emergency Medical Services are at x66911.

Revised: January 2, 2020

Warnings:

The SEM utilizes high voltage components, do not open the chassis.

While scanning your specimen it is possible to drive the stage into the microscope optics. Please exercise **extreme** care as you drive the stage around the chamber. **Crashing the sample into the optics is the most likely way to damage the tool. Keep a close eye on the camera view and be sure not to allow this to happen. Be especially careful when tilting the sample.**

1.0 Material Requirements:

1.1 Equipment: specimen mounted on a 12.5mm stub with carbon tape and coated with conductive material (sputtered Au for example), stub tweezers

1.2 Personal Protective Equipment: nitrile gloves, safety glasses

2.0 Procedure:

Important Note – There are multiple ways to work with the user interface –

- **Control Panel**
- **Trackball**
- **Mouse**

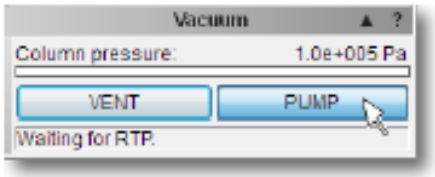
This procedure generally describes how to use the mouse but feel free to use the other input devices. If using the trackball note that the sensitivity is adjustable.

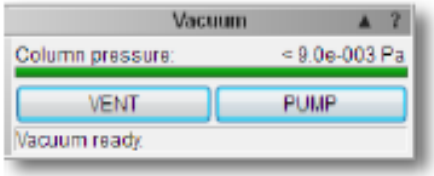
Log in and load configuration:

1. The SEM should be powered, under vacuum with PC running. If not, notify management. If logging into the PC no password is required
2. Start and log into the Vega software. The first time you log in please change your password immediately. Logging into your account will allow the software to save all your preferences. Using the SEM without logging into your account will disqualify you from future use
3. If the filament has been changed since your last login, please load the SEM_USER configuration. This will load the beam parameters (they are unique to each filament) then load your personal configuration file.
4. If you have logged in since the last filament change simply load your personal config file. Configuration files location -
   Options>Configurations>Load>YourFolder>YourFile.cfg.
   Preferences and Settings saved in your config file include the screen panels/locations, the default saved image size, etc.

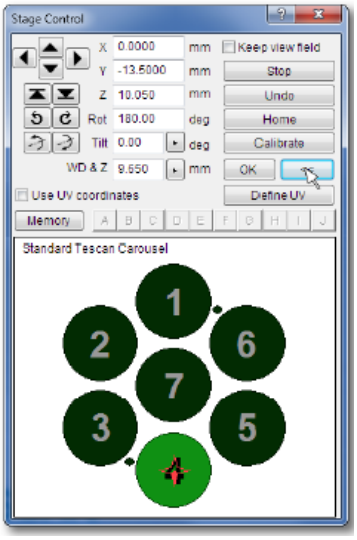
Load/Unload Specimens

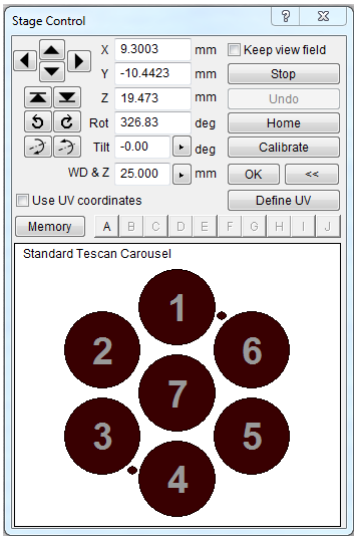
1. Vent the chamber by using the VENT button on the Vacuum panel portion of the screen (image below) and open the N2 cylinder by the instrument. Do adjust N2 pressure





2. Send the stage to the Home position (0 tilt, maximum distance from optics)
3. Once the pressure is at atmosphere open the chamber door with a gentle pull
4. Use the Stage Control panel (image below) to rotate the specimen holder as needed. While at atmosphere the circles will appear red, clicking on one will rotate the stage to the exchange position. NOTE – the specimen holder should not be removed!

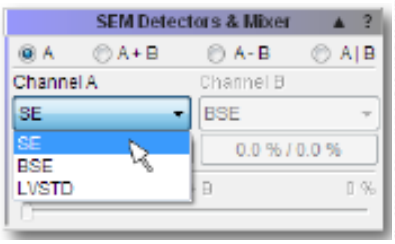


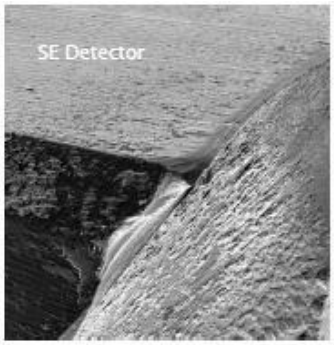


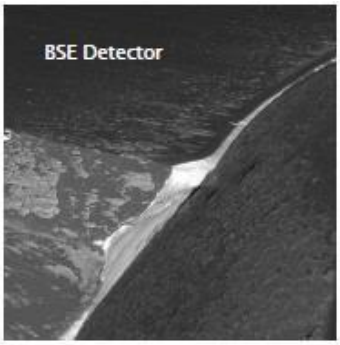
5. Loosen the screw securing the specimen (the appropriate screwdriver should remain near the SEM) and remove using the tweezer.
6. To load new specimens, place into specimen holder and gently tighten the screw. Rotate holder using Stage Control panel as necessary
7. Inspect stage and holder – is everything secure? Will any specimens touch the chamber walls, or other components?
8. Gently close the chamber door
9. Initiate pump down by pressing PUMP on the vacuum panel. Typical specimens should be imaged at high vacuum, low vacuum imaging is available for specialized applications, please discuss with management. Once the Column Pressure indicates green you may proceed.
10. Use the Stage Control panel to center the specimen of interest, while under vacuum the circles will appear green, clicking on one will rotate it to the observation position.

Powering Up Optics:

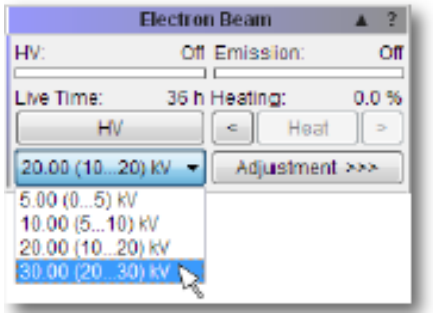
1. In the SEM Detectors & Mixer panel select a detector (images below) –
    a. SE – secondary electrons, basic SEM detector appropriate for almost all specimens
    b. BSE – back scattered electrons, scintillation type. Enhances material contrast, retractable, used for difficult to image samples. We recommend using the SE detector – easier to use, the vast majority of samples will not benefit from the BSE detector. If you wish to use the BSE, contact management first

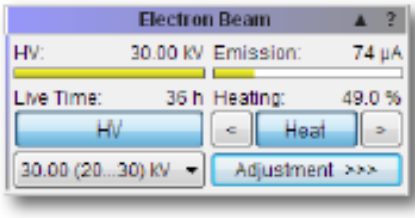






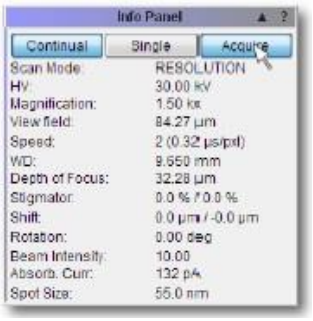
2. Using the Using the Electron Beam panel select an accelerating voltage (there are four factory presets (5kV, 10kV, 20kV, 30kV. It is possible to manually enter different values). This will turn on the high voltage and begin heating the tungsten filament. Higher voltage will achieve ultimate resolution (less than 10nm on our system) but ignore surface features. Lower voltages will achieve slightly less resolution but highlight surface features. You may notice greater "charging" effect or surface damage at higher voltages.





Acquiring an Image (the following assumes you are using the SE detector)

1. Set your default scan window and Image Save parameters – SEM>Image Parameters>Select window size, etc
2. Right click in the SEM scanning window to open a menu and select Minimum Magnification
3. Right click in the SEM scanning window and select Auto Signal to set brightness and contrast
4. If the SEM scanning window remains black go to the Electron Beam panel, click Adjustment and select Auto Gun Heating
5. Click on the Scan mode function on the Info Panel and select Resolution



5. To autofocus – right click in the scanning window and select Auto WD. To focus manually click on the WD icon  and turn the track ball right and left
6. To set Beam Intensity left click on the BI icon, see recommendations
7. To magnify, click on the Magnify icon and turn the trackball left to right

Now you are in the main part of the process – trying to get a high quality image. These are the things to work with:

A. Set the z- height so your sample is approximately 5-15 mm from the optics. Move up slowly, don't do it all in one shot – you don't want to crash.
B. Adjust x and y position to see the part of your sample you want.
C. Zoom in and out as needed.
D. Adjust working distance (WD) to focus your sample
E. Adjust the x and y stigmators as needed to reduce distortion of the image. If these get very far from 0 and you get lost trying to improve the image, set them back to 0 and try again.

F. Adjust beam intensity (BI) as needed. A lower beam intensity will require you to use a slower scan speed (in order to get more averaging with the lower signal level) but may improve resolution at higher magnifications or with samples that are charging. BI of 10 is the default setting.
G. Adjust scan speed to get a high resolution image. This will take a slower scan with more averaging.

- **Note** – if you wish to rotate the image, select rotation as the trackball parameter, and use the trackball to rotate the image angle by fine increments. This is *not* a physical rotation of the sample – it is an image processing rotation of the image only.

- **Note** – if you wish to tilt the stage to get a tilted image, be very careful – this is where it is easy to crash. First, set x=0 y=0 to center the stage, then set the stage z height to be as far from the sample as possible. Now change the tilt – slowly – go in increments … 30^o then 45^o then 60^o up to a max of 70^o. If needed. Once the stage is tilted, now, at low mag, move x and y to get to the region of interest. Finally, change z to bring the sample closer to the optics… ideally about 10-15 mm away… be very careful here and do not crash (!!), pay careful attention to the chamber camera. When adjusting the tilt back to zero – FIRST go to x=y=0 and increase the z-separation to bring the stage far from the optics. Then set tilt back to 0.

8. To acquire an image click on the Acquire button on the Info Panel or the Icon on the toolbar. Fill in the fields, select a folder. To change the image parameters use Image Parameters in the main SEM menu

   Generating good SEM images requires experience, skill and an understanding of electron optics that a simple SOP cannot replicate. If you are having issues getting good images, talk to more experienced users. Optimum parameter settings will vary depending on a number of factors, please keep in mind –
   - Shorter WD will give greater resolution but sacrifices depth of field. Tall, out of plane objects may require longer WD.
   - Greater accelerating voltage will improve resolution at the expense of greater edge effect, possible specimen damage and charging effect. Lower accelerating voltage will reduce resolution but reveal more surface structure detail. Recall that higher energy electrons will penetrate deeper into the specimen when determining the optimum accelerating voltage
   - Specimens with greater atomic weight typically respond well to higher accelerating voltage, low atomic weight specimens typically require lower accelerating voltage for optimum imaging.
   - It can be useful to image the test specimen (Au on carbon) at high resolution (objects down to 50nm or smaller can be resolved) first and then switch to your intended specimen, tweaking parameters as needed.

Back Scattered Electron Detector

1. Select the BSE detector from the SEM Detector panel
2. Drive the specimen stage to the home position (maximum distance between stage and optics)
3. Manually drive the BSE detector into position, there is a hand crank on the backside of the electron column, crank all the way in (it does not require much torque, if you are cranking very hard something is wrong)
4. Follow the motion using the chamber view camera. Once in position you can see the detector occupies space very close to the optics, minimum sample distance is 10mm or greater. Whatever WD you use, approach it incrementally and confirm the move with the camera view beforehand
5. Proceed with your image acquisition
6. Once complete, manually drive the BSE detector back to its home position
7. Follow the When you are Finished instructions below
8. Reconfirm that the BSE detector is fully retracted
9. Confirm that the chamber is tightly closed and pumping down

When you are finished

1. Turn off the high voltage by clicking on the HV button on the Electron Beam panel
2. Remove your specimens, see the Load/Unload portion of the SOP
3. Make sure the chamber is sealed, the SEM is pumping back down and the N2 cylinder is closed
4. If you used the BSE detector confirm that it is fully retracted
5. Log out.
6. Make sure the tools – screwdriver and stub tweezer – are there for the next user
7. **Before walking away, ensure that the chamber vacuum status is GREEN**

If at any time you feel unsure about how to use the tool, please stop work and contact a qualified user or faculty advisor. Please don't forge ahead when you are unsure, you may end up damaging the tool.

Important Notes regarding image acquisition -

Generating good SEM images requires experience, skill and an understanding of electron optics that a simple SOP cannot replicate. If you are having issues getting good images, talk to more experienced users. Optimum parameter settings will vary depending on a number of factors, including specimen composition and structure, magnification, region of interest, etc. Please keep in mind –

- Shorter WD will give greater resolution but sacrifices depth of field. Tall, out of plane objects may require longer WD.
- Greater accelerating voltage will improve resolution at the expense of greater edge effect, less surface detail, possible specimen damage and charging effect. Lower accelerating voltage will reduce resolution but reveal more surface structure. Recall that higher energy electrons will penetrate deeper into the specimen when determining the optimum accelerating voltage
- Specimens with greater atomic weight typically respond well to higher accelerating voltage, low atomic weight specimens typically require lower accelerating voltage for optimum imaging.
- It can be useful to image a test specimen (Au on carbon or In on silicon) at high resolution (objects down to 50nm or smaller can be resolved) first and then switch to your intended specimen, tweaking parameters as needed.

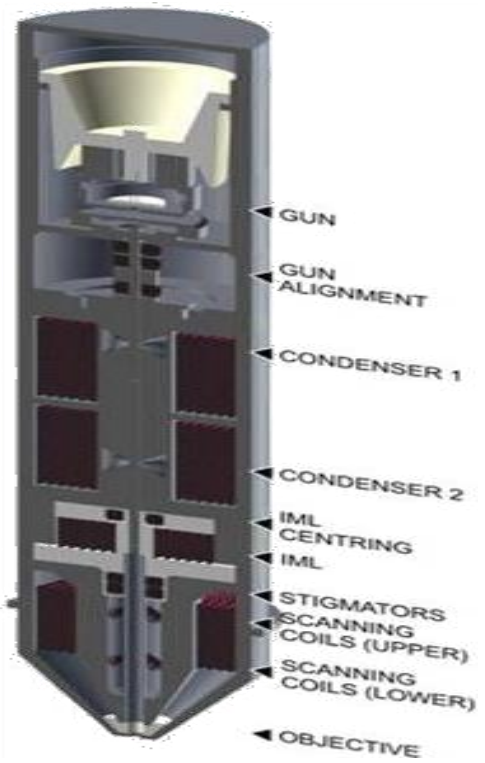
Recommended BI-WD settings

Magnification	Working Distance (WD)	Beam Intensity (BI)
Min – 200x	15-30+	13-18
200-2000x	12-20	8-12
2k-10kx	8-12	7-10
>10kx	5-7	4-7

Column Alignment - This procedure should be performed after filament replacement or if it seems the Gun Tilt parameters have drifted significantly.

1. Load a sample that will give good SE detector images from 100x – >10kx. The Au on C resolution target works well. The specimens of interest may be loaded at the same time.

2. Confirm settings - Resolution Mode, 30kV, BI=10, Auto Gun Heating, SE Detector. Note the initial gun tilt parameters
3. Focus on the specimen and obtain a good image at a magnification of at least 2kx. Adjust WD, brightness, contrast as needed. Image should be mid gray, full tone, not washed out white or black, with lots of edges in various orientations. Histogram function can be helpful to optimize brightness and contrast
4. Work from the top of the column to the bottom, see image
5. Select Auto Gun Centering from the Emission Adjustment control and click thru. Let the software complete the process
6. Select Auto Column Centering, let the software complete the process.
7. To confirm the software has done a good job select Manual Column Centering and proceed with the Wizard. As the image wobbles in and out of focus there should be no image shift. If necessary, adjust manually, sensitivity can be optimized on the Info Panel.
8. Finally, adjust stigmators. Circular objects are the easiest to work with. Open a reduced raster window around a circular object, select Stigmator from the Info Panel – you can now use the knobs on the Control Panel to adjust X and Y. You can use the onscreen Info Panel to adjust the sensitivity of the knobs. Stigmate so that edges in all orientations appear equally sharp.
9. If the resulting images are unsatisfactory you can reset the gun tilt parameters from step 2 and run through the process again
10. For ultimate resolution perform procedure at 30kV, 20kV, 10kV, 5kV in order. This is usually necessary after a filament change. To correct drift, one voltage setting (30 or 20kV) should be sufficient.



Further reading – There are a number of texts and online resources available that provide more background and detail. See, for example, "Scanning and Transmission Electron Microscopy, An Introduction," Flegler, Heckman and Klomparens or http://www.ammrf.org.au/myscope/pdfs/sem.pdf. A better understanding of the physics involved is crucial in obtaining the best quality images.

Report all accidents or tool issues to Jim Vlahakis – james.vlahakis@tufts.edu