SEM QUICK GUIDE/NOTES

Zeiss EVO MA15 15.40

Questions or concerns? Please contact Jeff Rahl or Emily Flowers

-ALWAYS use the log book to document hours used, any errors, filament changes, etc.

-When not in use, the SEM should be kept in the yellow button mode. This keeps the vacuum on, but the majority of the power off. Red button mode shuts off all power. It will take a really long time to pump down if left in red button mode. Green button is for power on.

1. To start, push the green button. This will automatically turn on the computer. Get into the SmartSEM software. A login box will appear, enter your user name and password.

2. You should see 6 tabs to the right of the screen. Click on the 'Vacuum' tab and then click 'Vent' to release the vacuum and open the chamber door. This may take a few minutes.

-Your sample should be mounted to a stub. If HV mode is preferred, then your sample should be coated with either gold or carbon. If VP mode is preferred, coating is not necessary. HV is recommended for high resolution imaging.

3. Using lint free gloves, remove the carousel, or sample holder, from the stage. Place your sample stub into one of the numbered holes. Tighten with the Allen key. Multiple samples can be arranged on the carousel. Please be careful when using different heights of samples in the chamber.

-Always wear lint-free gloves when handling the specimen, the sample holder, or stage. If not, fingerprints can lead to vacuum deterioration or prolonged pumping times.

4. Mount the carousel to the stage. Be sure that the flat side (dovetail fitting) of the bottom of the carousel is flush with the flat side of the stage mount.

5. Click on the 'Pump' button under the vacuum tab. This will take a couple of minutes. The longer the chamber door is kept open, the longer it takes for the vacuum to pump down. If working in VP mode, it will not take as long as HV.

-When working in VP mode, the VP aperture has to be in place in the bottom of the column inside the chamber, the outside column aperture has to be set at 100µm opening, and the software has to be in VP mode. HV can be used while in VP mode, but the 20µm and 30µm apertures cannot be used. To switch modes in the software, the vacuum has to be vented.

6. While the vacuuming is pumping, the TV detector, or Chamber Scope, may be used to help find your specimen.

7. Under the 'Stage' tab at the top toolbar, the 'Stage Navigation' should be opened. In this window, the safe navigation can be set to the specimen's height and diameter to prevent collision of the specimen and the column. In the diagram, by double clicking on the stub, the stage automatically navigates to the one clicked.

8. Use the joystick in the 'Z' direction to bring the sample closer to the objective lens. To start, it should be less than 10mm.

9. When the vacuum is ready, the gun/beam may be turned on. Select which type of detector. It is usually easiest to find the image with the magnification at a minimum, 20kv, the probe current around 150, the reduction box, and a slower scan speed.

-If using HV mode: use the focus, stignators, and then 'focus wobble'. 'Focus wobble' is not needed in VP mode.

10. After the gun is on, pick an even spot on sample or background of holder. Check the 'Emission' under the 'Apertures' tab. Adjust the 'shift' and 'tilt' as needed. The most centered, brightest blob is desired. 'Auto Align' can be used, but be sure to check it after completed. 'Shift'= outer circle, 'tilt'= white blob

-The TAB key will toggle between coarse and fine focus.

-To save an image, first freeze the image and then wheel click and send to TIFF file. Choose the appropriate directory

-The reduced raster box is helpful when trying to obtain an image and focusing. To remove the box, push the reduced button again

-When shutting down the instrument, turn the beam off, remove the sample from the chamber and be sure to pump it back down afterwards. Get out of the software and shutdown the computer. Once the screen is black, put the instrument in Yellow button mode.

TO CHANGE TO VP

1. Move to lowest aperture (100μm opening) using the 'y' knob on the outside of the column.

2. Try to center the image

3. Turn off gun.

4. Vent the chamber

5. Under the 'Apertures' tab, click 'Select Aperture', choose 'VP'

6. Open the chamber door.

7. BE SURE TO REMOVE THE BACKSCATTER DETECTOR!!!

8. Insert VP aperture into the bottom of the column using the Ap tool.

9. Put the backscatter detector back in place and make sure wire is out of the way.

10. 'Vacuum' → 'Pump' → 'Go to VP'

11. When switching, 40 Pascal is good to start with

12. Choose the VP detector (VPSE G3) when the gun is on

NEVER GO TO VP MODE WITHOUT THE VP AP IN!!!

SEM Random Notes

-For the chamber/stage, North faces to the left

-Make sure 'Go to HV @ Shutdown' is checked.

-When looking in the chamber, the SE detector is to the left and the VP detector is to the right.

-Under 'View', 'SEM Status' different parameters can be set to be displayed to keep track of hours used, filament life, etc.

-To send an image to a TIFF file: 'Freeze' image, Click mouse wheel, send to TIFF, and then can choose name and directory.

-The less probe current, the more noise.

-To create a new user: 'Tools' → 'Administrator' → 'Users' → 'New'. Be sure to change name, password, and directories for saved images.

-Hysteresis- for major measurements- demagnetizes the lens- so measurements are more accurate.

-Focus and Z = Working distance

-Occasionally wipe the chamber door connection with alcohol

-If out of focus and at the desired working distance, adjust 'Z' (height of stage)

-For better brightness and contrast: under scanning, check 'line scan', use the brightness and contrast knobs to get the red line in the center of the box. Want the line near the bottom with the maximum stretch within the box.

-Lens → EVO 80, NOT 1400:110

-'Resume' button sets everything back to before the chamber door was opened. Automatically turns the beam on.

-If right click on background of stage panel, can get box to calibrate

-Good book for theory: "Scanning Electron Microscopy and X-Ray Analysis" by Goldstein

-To obtain the smallest spot, use highest voltage and lowest probe current

-Can use 'Image Dual Channel' to get two screens with two different detectors

-Stig is for correcting for beam shape

-For the external camera and smart stitch:

-Put carousel on camera mount

-Use the EOS software

-Then image navigation in the side panel of the SmartSEM software

-Easier if sample holder is oriented the same direction as the picture is taken

-Turn on gun etc

-Register the image under image navigation, setup, then find 3 points and match them

-Use the silver spacer about every three months to check the air pressure for the chamber. The spacer is used to check the space between the table surface and the chamber. The empty space should not be lower than the small side of the spacer and not greater than the larger side of the spacer. Check all sides of the chamber. If low, use the foot pump and the valves in the back of the SEM to raise the chamber.

-To edit the data zone, the 'Select Annotation Object' button must be clicked, then it may be edited when the blue handles are visible. To upload a non-SEM image, it must be saved as a bit map (BMP) file.

-In HV mode:

--~9 x 10^{-5} makes the pump ready

-If the kV is changed, be sure to adjust the 'focus wobble'

-From HV to VP, the gun stays on

-The higher the kV, the better the image

-30μm aperture is better for resolution

-20μm aperture is better for depth

-The higher the kV, but lower probe current, the better the resolution, but more noise

-Samples should be coated either with gold or carbon.

-If using thin sections, put a piece of carbon tape from top of stub (bottom of thin section) to top edge of thin section

-In VP mode:

-'Bias' works with vacuum

-If streaking, usually adjust 'Bias'

-VP works better under poor vacuum

-Can go to HV in VP, turns gun off and automatically switches

-Collector bias should be at the highest collector for maximum collection with use of a slightly lower scan speed

-In a thin section, the darker the grain, the lower the atomic number

-The VPSE detector can show photoluminescence with the collector bias at zero

-shades because of topography

-If you think you might have a pit, do a 180° scan rotation, it might actually be a ridge

-In Backscatter mode:

-BSE can be tricky

-likes slow scan speed, 5 or 6

-can adjust 'line scan' once the image is found

-better in medium gain

-Do not use additional chamber camera while in backscatter mode, the light effects it

-With BSE, usually in high gain, except when looking at specimens with high atomic numbers

-Can use ++ -- for better topography

-All + is for composition, not topo

-Add fifth + for general imaging

-Vacuum between 15-30

CHANGING THE FILAMENT

-ALWAYS WEAR GLOVES!

-ALWAYS USE LINT FREE CLOTH!

1. Vent the chamber. Once the chamber door can be opened, then the white cover can be removed from the top of the column.

2. Turn the instrument off.

3. Wait for the firing pin to cool, about 15 minutes.

4. Open the silver lid. There is a hinge to the right side. Pull the lid straight up and then rest to the right. It can be a little snug to remove.

5. Remove the firing unit by unscrewing the 4 screws with the Allen key. Please do not fully remove the screws. Be careful not to let the firing unit drop when removing. Place the firing unit on a lint free cloth.

-Check the plate on the inside of the column. If dirty, the two screws can be removed to release the plate for cleaning. Be careful not to let anything drop down the column.

6. Place a lint free cloth over the column or replace the silver lid (if column plate was not removed) to keep dust and particles out of the column.

7. Use the filament tool to remove the brass retaining washer from the firing unit and then place on the lint free cloth.

8. Turn the firing unit upside down to remove the filament holder and spring.

9. Remove the old filament. Please be careful not to fully remove any of the screws.

10. Clean all of the dirty pieces with metal polish every three times the filament is changed or when needed. Wipe clean with a lint free cloth. Place in a beaker with alcohol. Place beaker in the ultrasonic cleaner. Make sure water level in cleaner is lower than the beaker. Ultrasonic clean for 10 to 15 minutes.

11. Remove beaker. Remove the firing unit and parts from the beaker with tweezers. Make sure the screws are still in place of the parts before dumping the used alcohol.

12. Use canned compressed air to FULLY dry the parts.

13. Insert the new filament being sure to not touch the tungsten filament.

14. Center all the parts back together.

15. After centering the filament, bring the filament to the top of the firing unit hole using the filament tool and the brass retaining washer. Use the magnifying lens to make sure filament is perfectly centered. Then turn the brass retaining washer 1 ¼ turns back lower the filament into place.

16. Remove lint free cloth from on top of column or open silver lid back up. If the column plate was removed and cleaned, put back, being careful not to drop the screws into the column.

17. Align the firing unit back onto its holder. Tighten Allen screws to hold it in place.

18. Check the column O-ring. Use canned compressed air to remove any particles.

19. Put the silver lid back over the column. Make sure that it is aligned properly with no spacing, otherwise the vacuum will not be sealed.

20. Replace the white cover.

21. Turn power back on the instrument.

22. Under the 'Gun' control tab, check the 'New Filament' check box. If using a different type of filament, please select it from the drop box.

23. Pump the vacuum.

24. Lower the kV to around 10. Adjust the probe current to around 150pA. Adjust the filament target to around 2.75A.

25. Turn on the gun. With the 'New Filament' box checked, this will take a bit longer than usual.

26. Check the filament alignment: 'Apertures' then 'Emission'. Use 'Shift' and 'Tilt' to center it. Or 'Auto Align' can be used. It is usually easier if the scan speed is 6 in 'Pixel Average'.

27. Make sure the filament is saturated. Bring the 'Filament target' up to see the maximum brightness and then back down. Adjust to have it 2 clicks below the max brightness.

28. After the new filament is warmed up, about20 minutes, then the kV and probe currents may be adjusted.

QUICK KEYS

<F1>: Help key

<F5>: Toggles between low magnification and high mag

<F6>: Stage stop (aborts stage movement)

<F9>: list of special keys

<Ctrl+B> Showing or hiding toolbars

<Ctrl+D> Data Zone: to toggle between the data zone being displayed at the bottom of the image

<Ctrl+G> Control Panel

<Ctrl+I> Gives SEM status

<Shift+F2> Hysteresis

<Shift+F3> Showing and hiding a full screen image

<Tab> Toggles between coarse and fine focus

EDS/EBSD Random Notes

Oxford Serial #35836

-The room temperature should be 76°F or less

-Use INCA Tidy Up as often as needed

    -little broom and dustpan icon

    -reboots hardware/detector

-Certain sequence for using:

    -Start Smart SEM software

    -Start RemCon32

        -Comms

        -Open Port

    -Start AZTEC

-Routine backup

    -'All Programs' → Maintenance → Backup and Restore

    -Set for every Tuesday at 12:00

    -When hooked up to the network, change to 'Save on Network'

-Maintenance

    -CCleaner- if internet is used- run frequently

        -Don't change options

    -Defraggler- 1 time a week

        -Analyze

            -Do before system backup

-Anode inside column needs to be cleaned every six months depending on usage

-3 hardware boxes

-X-Stream-2 → for detector

-MicsF+ → with computer

-HKL → EBSD

-If any have red lights, failed power on self test

-Allow detector 15 minutes for cool down

-EDS

-EDS needs shorter working distance for resolution, 8.5 is preferred

-The detector should be removed and covered if vacuum is gone for more than 72 hours

-Detector-Blue light- cools at -60°C

-Detector-Red light- Bad!- Power down, then back up. Call Oxford.

-Does not like the additional camera on!

-Needs at least 10,000 cps for the EDS

1. After Aztec software is started, choose the 'Operator' mode.

2. Can save in preferred directory if new project

3. Choose either EDS or EBSD from left tab

4. Scan Image → Start

    -Can adjust the contrast/brightness at the bottom right

5. Acquire Spectra

    -Settings, number of channels, auto, start

-After the scan is complete, in the Acquire Spectra tab, the tools on the left side of the screen can be used to analyze other points on the sample (points, squares, circles, etc)

-Optimize tab

    -Beam measurement (30 seconds)

        -read box

-Energy calibration (5 minutes)

-already calibrated on Cu

-done once in several months

-get a piece of pure copper → Select Copper → Start

-Should do whenever the filament is replaced!

-If not seeing anything on EDS scan, check the working distance

-When switching areas to scan, be sure to click 'New Site' before 'Start'

-Live time + Dead time = Real time

-Can adjust time in acquisition mode

-If you right click in spectra box, (EMSA) saves as text file

-Settings- can adjust 'Autolock' for image drift caused by charging

-For a better, more precise EDS scan:

-Remove VP aperture from column

-Install the Upper 100 aperture assay (red tool)

-Install the 1000 EDS using the black tool into the upper 100.

-While vacuum is vented, change aperture settings in the software

-EBSD

-EBSD needs longer working distance

-Requires sample to be angled at 70°

-If using for a longer period of time 6+ hours, the filament needs to be resaturated before shutting the gun off. Or the 'long filament life' box needs to be checked.

The Carbon Coater

Leica CED 030

Serial #590586

1. Insert specimens to be coated
    -Be sure to put the protective shield over the chamber

2. Prepare carbon thread evaporator flange and put it on top of working chamber

3. Connect high current cables.
    -Cables should go on either side of thread to be used

4. Switch power on from mains switch

5. Evacuate working chamber
    -Turn on pump
    -The better the vacuum, the better the coat
    -Desired vacuum around 10^{-2}

6. Close shutter

7. Degas carbon thread using UP and DOWN buttons
    -Press the UP button until carbon thread glows orange/red then press DOWN until it is black again.
    -Repeat at least one more time

8. Press RESET button to switch current off

9. Open shutter

10. Press HIGH CURRENT button to evaporate carbon thread
    -BE SURE NOT TO LOOK AT CHAMBER!! Unless you are wearing protective goggles
    -It is easier to tell if the lights are off and you look at the wall
    -It will only take a second to coat

11. Switch power off from mains switch and pump

12. Turn on the nitrogen tank to release the vacuumed chamber