Tescan MIRA3 SEM

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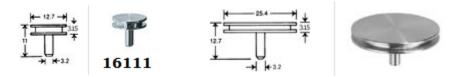
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This procedure covers basic operation of the instrument and use of the MiraTC software; see separate procedures for performing EDS and EBSD.

1. Sample preparation/mounting

1.1. Specimens should be mounted on standard pin stubs (Ted Pella #16111 or #16144, for example) using <u>conductive tape</u>; <u>the use of carbon paint is also permissible</u>, <u>as long as it is well-dried prior to sample loading</u>. If your sample is non-conductive, it may be given a light C coat (few nm) <u>after</u> being mounted on a stub to ensure a path to ground is produced or a path to ground can also be produced using conductive tape.



- 1.2. Assuming a <u>flat</u> sample with thickness t mounted on a standard pin stub, the shortest possible working distance that may be attained is WD = 10 t mm. If a WD shorter than this is needed, it will be necessary to use a stub extender. That being said, unless you need to perform high-resolution imaging, it will not be necessary to use WD < ~10 mm anyways.</p>
- 1.3. If you will be performing EBSD, there are additional constraints regarding specimen preparation that must be addressed (as described in the procedure for performing EBSD).

2. Specimen loading

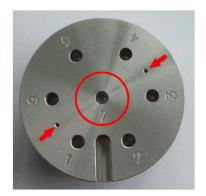
2.1. Log in to the MiraTC control software.



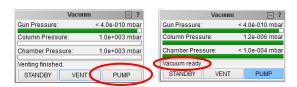
2.2. In the Stage Control, select Calibrate. Then in the "Vacuum" panel, select "VENT" to vent the chamber; the panel will indicate when venting is finished.



2.3. When venting is finished, open the chamber door and load your specimen in the "7" position of the carousel. <u>Do not load specimens in any other carousel position other than "7"</u>.



2.4. Close the chamber door; in the "Vacuum" panel, select "PUMP" to evacuate the chamber; apply light pressure during the initial stage of evacuation. The vacuum panel will indicate "Vacuum ready" when the vacuum level is satisfactory.



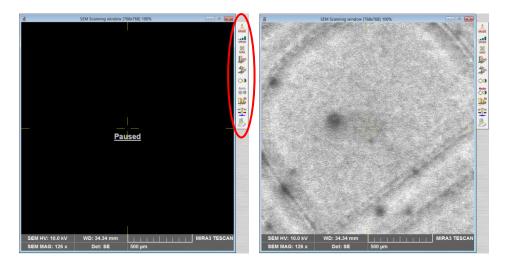
- 3. Turning on the beam, focusing, and navigating
 - 3.1. The beam voltage is set in the "Pad" panel by selecting "High Voltage" from the pull-down menu and then entering in the desired value; the beam voltage will gradually ramp up/down to the input value from the current value.



3.2. In the "Electron Beam" panel, select "BEAM ON" to turn on the beam (the button will turn blue).



3.3. A live image will appear in the "SEM Scanning window"; along the right side of the window will be a column of buttons used to adjust different parameters. If you loaded your sample properly, you should be positioned approximately over the middle of your specimen (and the image will probably be out of focus).



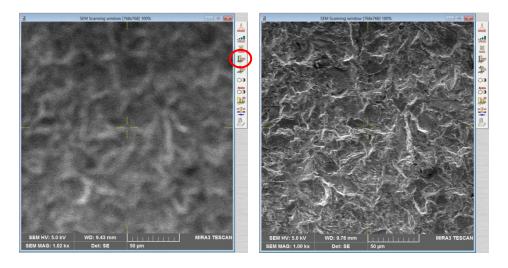
3.4. If the live image is difficult to see due to excessive or insufficient signal, select from the column of buttons to automatically adjust the detector contrast and brightness (this can be done as needed).



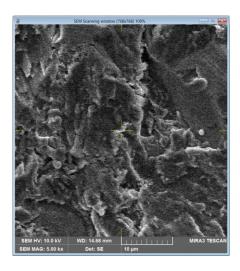
3.5. Next, set the view field to ~200 µm (1 kX Magnification) by turning Mag on the control pad (upper left knob). Look at the magnification values on the bottom of the SEM imaging window.

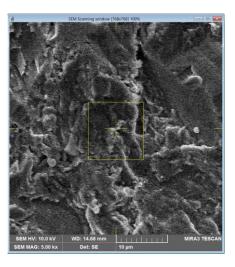


3.6. The specimen now needs to be roughly focused; use the bottom left knob WD on the control panel to focus the image. (<u>note: this adjusts the focus of the objective lens, NOT the actual WD. The stage will not move on the Z direction</u>)



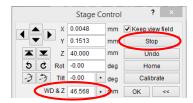
3.7. Sometimes, it can be helpful to use a reduced scanning area when focusing (as well as went performing some of the alignments as will be discussed later). To reduce the scan area, simply double left click on the image; a yellow box will appear and the scanning will be reduced to that area on the sample. To move the box, left click and hold in the box and then drag it to a new area. To change the size of the box, right click and hold in the box and then drag to change the size. To resume full area scanning, double left click anywhere on the image.

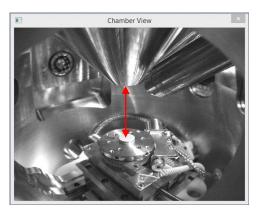


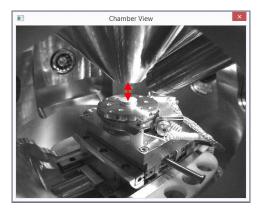


3.8. To move around the specimen, middle mouse click on a spot in the live image (you can mag in or out as needed) and the stage will move this spot to the center of the image; in order for this to be accurate, <u>the sample must be</u> reasonably focused.

- 4. Setting the WD and beam intensity
 - 4.1. Once the sample is roughly focused and a region of interest centered in the live image, incrementally adjust the WD to the desired value in the "Stage Control" panel via work adjust the each incremental stage movement, adjust the focus again WD knob on the control panel. Observe the stage movement via the "Chamber View" panel and be prepared to stop the stage using "Stop" if any collision appears imminent. Do not attempt to adjust the WD unless the image is focused first.



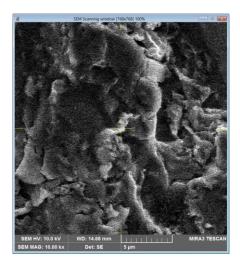


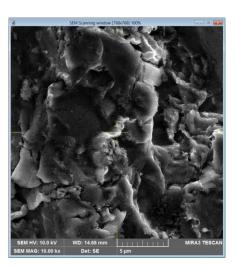


4.2. The probe current can be adjusted using the "Pad" panel by selecting "Beam Intensity" from the pull-down menu and then entering in the desired value (integers of 1 – 20). For a given beam voltage and WD, probe size will usually be minimized at beam intensity = 7 – 9; as the beam intensity is increased above this value (which is usually necessary if performing EDS or EBSD), the probe current will increase, but at the expense of a larger probe.



- 5. Live imaging and alignment
 - 5.1. When performing alignments (particularly manual centering and stigmation), it can be useful to adjust the scan speed of the live image as needed. To do this, simply left click on the live image and then scroll up (down) with the middle mouse button to increase (decrease) the scan speed.

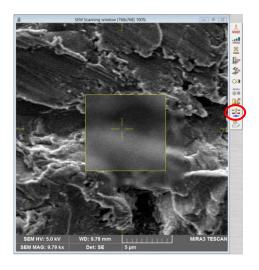




5.2. In the "Electron Beam" panel, select "Adjustment" and then "Auto Gun Centering" to align the gun (will take a couple minutes). When the procedure is finished, immediately select and focus the image to prevent any further modifications to the gun alignment.



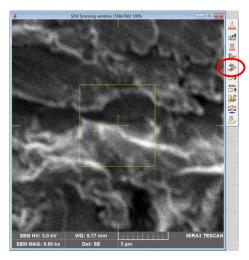
5.3. Find an area of interest on the specimen surface and center it in the live image; then select and set the view field so it is smaller than what you intended to use for collecting your final images. Select from the column of buttons to bring up the "Manual Centering Wizard"; select (the objective lens will start wobbling) and use the X and Y knobs in the control panel to minimize shifting of the image during wobbling. When the wobbling is minimized, select save the alignment and turn off the wobbling.

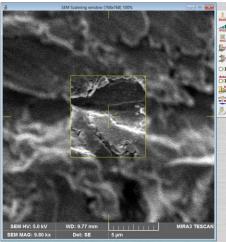






5.4. Select and decrease the view field so it is substantially smaller than what you intended to use for collecting your final images. Select from the column of icons and then use control panel X and Y to stigmate the image. It also helps to do this using a reduced scan area with a slower scan speed.





- 5.5. Note that alignment depends strongly on beam voltage, beam intensity, and WD; if any of these parameters are changed, the alignment should be performed again (*gun alignment only needs to be performed again if the beam voltage is changed*). Additionally, if imaging at view fields smaller than you originally intended are needed, manual centering and stigmation should be performed again using the smaller view fields (preferably, even smaller).
- 5.6. If for some reason you need to leave the SEM for an extended period of time, the beam should be blanked off. Select "SEM" from the pull-down menu and then "Beam Blanker". In the "Beam Blanker" panel, select "Gun" to blank the beam (the button will turn blue). To unblank the beam, select "Gun" again and the live image will appear.



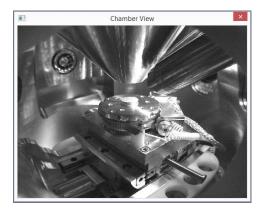


6. Detector selection

6.1. In the "SEM Detectors & Mixer" panel, ETD (incorrectly named as "SE") and dedicated BSE detectors can be selected from the "Channel A" pull-down menu. The default setting is to use the SE detector mounted on the inside of the chamber. If the BSE detector is selected, it must be manually inserted as shown below in the "Chamber View" panel. <u>Do not attempt insertion of the BSE detector if the WD < 8.5 mm OR if you will be performing EBSD</u>.

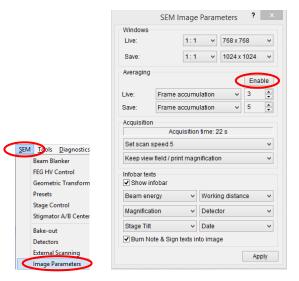




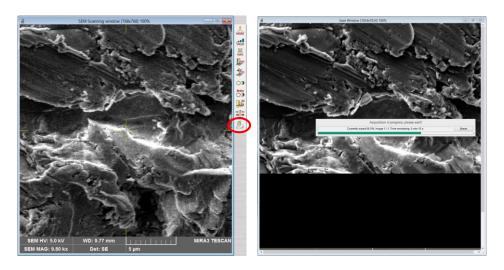




- 7. Adjusting imaging parameters and acquiring final images
 - 7.1. To adjust the various image parameters, select "SEM" from the pull-down menu and then "Image Parameters". In the "SEM Imaging Parameters" dialogue box that pops up, you can select different options for the "Live" and "Save" imaging modes. The settings indicated below are usually sufficient, but make sure under "Averaging" that "Enable" is left gray to turn off averaging. These settings will be saved for your account the next time you log in.



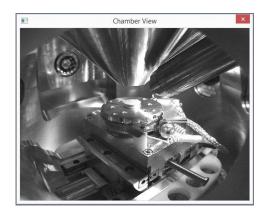
7.2. To acquire a high quality image for saving, first decrease the view field so it is smaller than what you intended to use for your final image, then finely focus the image (use a reduced scan area to help with this, if desired) and then select from the column of buttons.



- 8. Finishing the session
 - 8.1. In the "Electron Beam" panel, select "BEAM ON" to turn off the beam (the button will turn gray). Note that the beam settings you leave the instrument at when you finish will be the beam settings that will be loaded the next time you log on to MiraTC.



8.2. If necessary, retract the BSE detector, and in the "SEM Detectors & Mixer" panel, make sure "Channel A" is set back to "SE".





8.3. In the "Stage Control" panel, select "Calibrate" to calibrate the stage and return it to the home position; "Chamber View" should look similar to shown below after calibration.

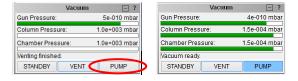




8.4. Once calibration is complete, the chamber can be vented. In the "Vacuum" panel, select "VENT" to start venting the chamber



8.5. When venting is complete, remove your specimen, then close the chamber door, and select "PUMP" to return the chamber to vacuum; again, apply light pressure to the chamber door during the start of evacuation.



8.6. Select "File" from the pull-down menu and then "Log Off" to log off MiraTC. In the "Log off" dialogue box that pops up, select "Yes". In the dialogue box that subsequently pops up asking if you want to go to "STANDBY" mode, select "No" (this makes sure the chamber remains actively pumped).





