Tescan MIRA3 SEM: EBSD using EDAX TEAM

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Last updated: 10/10/19

This procedure assumes the user is already familiar with basic operation of the SEM and the MiraTC interface.

1. Sample mounting, preparation, and constraints

    1.1. *EBSD specimens must be 15 mm or less in thickness with a flat, damage-free, smooth surface and have a footprint that fits basically within the area of standard 3 mm-thick SEM pin stubs* (Ted Pella #16111, diameter ~13 mm or Ted Pella #16144, diameter ~25 mm). Ideally, the thickness to footprint ratio of the specimen should be kept as small as possible (subject to the above constraints) as this will increase specimen stability.

    1.2. It is preferable to mount specimens using *conductive paint* instead of carbon tape to limit specimen drift; *if you use conductive paint, make sure it is well-dried before loading your sample into the chamber.* If your sample is non-conductive, it should be given a light C coat (few nm) *after* being mounted on a stub to ensure a path to ground is produced.

2. Specimen loading

    2.1. Specimens must be loaded in the “7” position of the carousel; additionally, if your specimen is ~3 mm or less in thickness, load the 12 mm stub extender (Ted Pella #15318) and then load your specimen on top of the stub extender (*the stub extender is required for specimens in this thickness range*; otherwise, it will not be possible to attain the necessary stage tilt and working distance for performing EBSD). *Do not load specimens in any other carousel position besides “7” if attempting EBSD.*

3. Beam and detector settings for EBSD

3.1. In principle, the spatial resolution of EBSD improves as the beam voltage and probe size (controlled by beam voltage and intensity) are both lowered. However, the signal to noise ratio in the EBSD patterns will *decrease* at these settings, which makes indexing of the patterns less accurate and/or more time intensive. If you have a rough idea about the average grain size in your specimen, this can be helpful for determining appropriate beam settings for EBSD. If the average grain size in your sample is a few 10s μm, a beam voltage of 30 kV with a beam intensity of 15 – 20 will probably be sufficient. As grain size decreases, a lower beam voltage (and possibly a lower beam intensity) may be needed. If you are unsure as to what beam settings you should use for EBSD, please consult with staff for recommendations.

3.2. The only detector on the SEM that may be used when performing EBSD is the ETD (erroneously called "SE") detector; *do not insert the BSE detector if you intend on performing EBSD or a collision between the sample and BSE detector will probably occur*. There is a forward scattered detector (FSD) on the EBSD camera that can also be used when performing EBSD as will be described later.

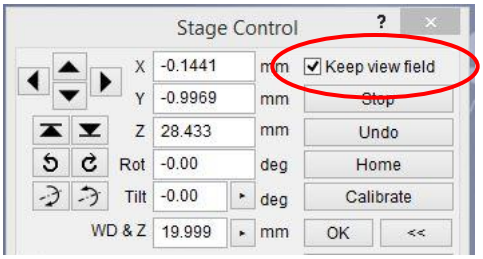
3.3. Once appropriate beam settings have been selected, find a region of interest on the specimen (ideally, away from the specimen edges); then focus the image and set WD = 20 mm in the "Stage Control" panel (this is the optimal WD for performing EBSD).

3.4. Perform the basic SEM alignment: auto gun centering, beam centering, and astigmatism correction.

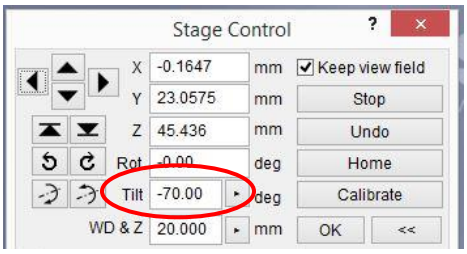
4. Setting specimen tilt and magnification

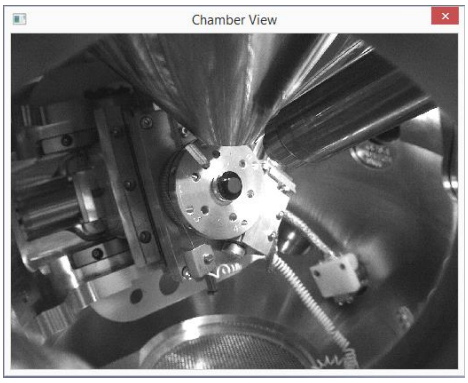
4.1. Verify that the *BSE detector is retracted* using the "Chamber View" panel before attempting any tilting of the stage; otherwise, a collision with the BSE detector could result.

4.2. Center a recognizable feature in the live image. In the "Stage Control" panel, make sure "Keep view field" is checked; this keeps the feature roughly centered in the image and at (close to) the same WD after tilting.

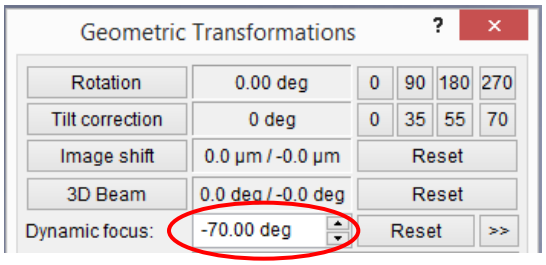


4.3. Using the “Stage Control” panel, tilt the stage to –70° (*negative* 70°) *in 10° increments*; after each increment, perform auto contrast/brightness, re-center the feature in the image, refocus on the feature, and set WD = 20 mm in the “Stage Control” panel. When finished tilting, the “Chamber View” panel should look similar to what is shown below.





4.4. In the “Geometric Transformations” panel, enter –70° in “Dynamic focus” (be sure to hit the “Enter” key) and then finely focus the feature of interest (which again should be at the *center* of the image). Then set the scan speed to 4 or higher (the scan speed must be slow for dynamic focus to work); the entire surface of the specimen will now be in focus.

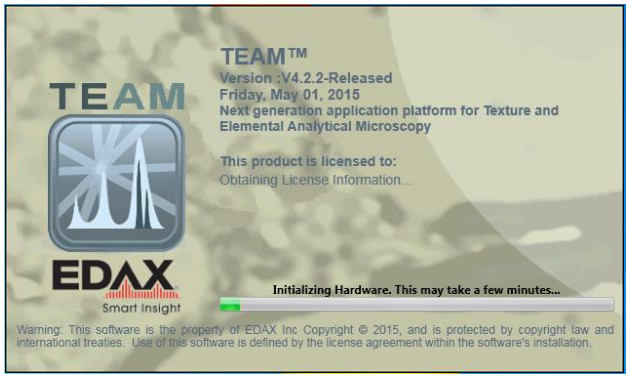


4.5. Set the magnification as needed. Any magnification can be used, but it should be high enough such that if reduced by 50%, no specimen edges will be present in the image. As a general recommendation, a magnification of 500 – 1000× will usually be sufficient for a specimen with an average grain size of a few 10s of µm.

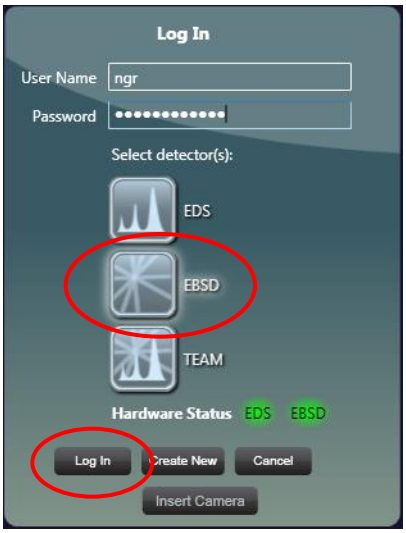
5. Starting TEAM and setting up your project file

5.1. You must be logged on to MiraTC *before* starting TEAM or TEAM will not function properly; *do not* attempt to open TEAM without being logged on to MiraTC.

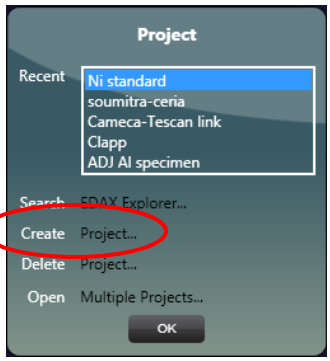
5.2. Open the TEAM software. A splash screen will pop up as the hardware is initialized (listen for the motor on the EBSD camera to turn on).

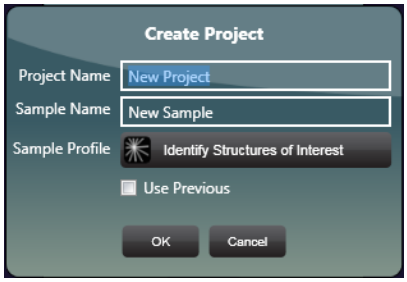


5.3. Enter your username into the “Log In” dialogue box and then select EBSD; then enter in your password and select “Log In”.

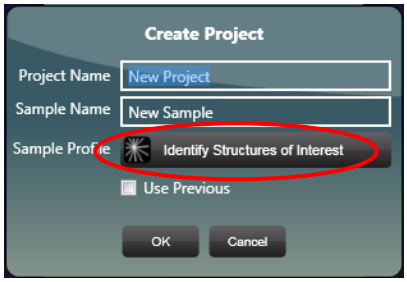


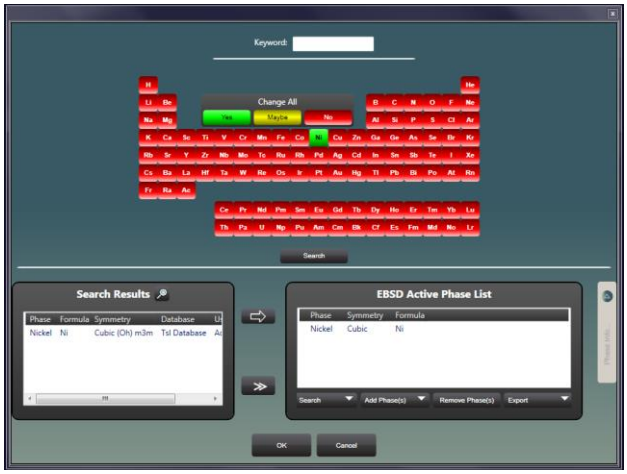
5.4. In the “Project” dialogue box, go to “Create” and select “Project” to create a new blank project; this will bring up the “Create Project” dialogue box, where you can name the project and specimen.





5.5. In the same “Create Project” dialogue box, select “Identify Structures of Interest”; you can search for the phases present in your sample based the elements you expect to be present and then add these to the “EBSD Active Phase List”. You must add at least one phase to the list or the EBSD patterns cannot be indexed. Select “OK” when finished, and then select “OK” again in the “Create Project” dialogue box.

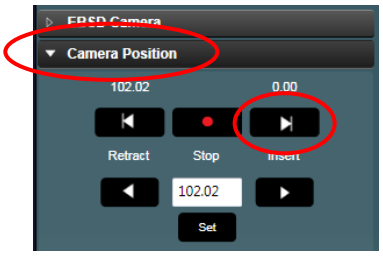


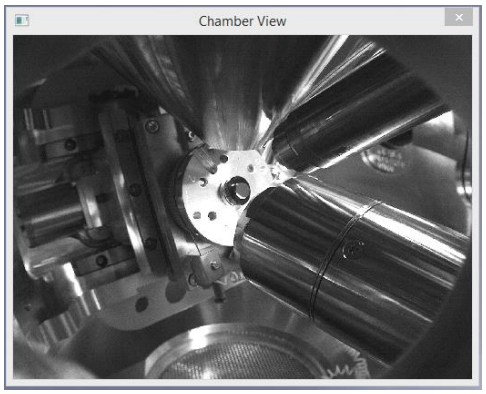


6. Inserting the EBSD camera

6.1. The stage tilt must be set to –70° (*negative* 70°) and the image in focus at WD = 20 mm *prior* to insertion of the EBSD camera. Insertion of the camera under any other conditions may cause a collision of the camera with the specimen or stage. *There is no interlock preventing insertion of the camera under inappropriate conditions.*

6.2. Expand the “Advanced Properties” tab (right side of the window) and select “Camera Position”; select “Insert” to insert the camera (you should hear the motor start). Be sure to monitor the insertion process in the “Chamber View” panel and be prepared to select “Stop” if any collision appears imminent. The “Chamber View” panel should look similar to below after insertion is complete.

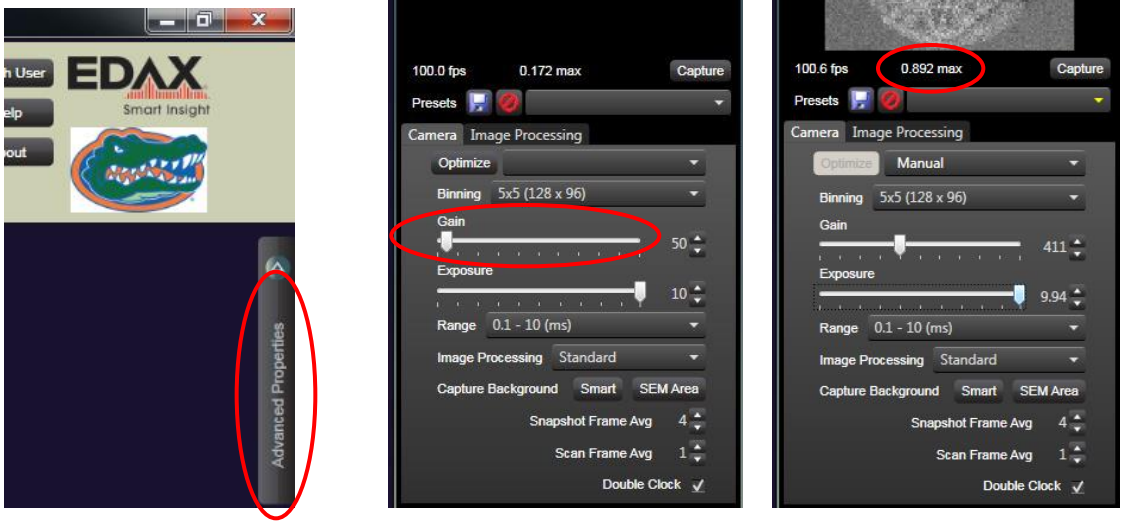




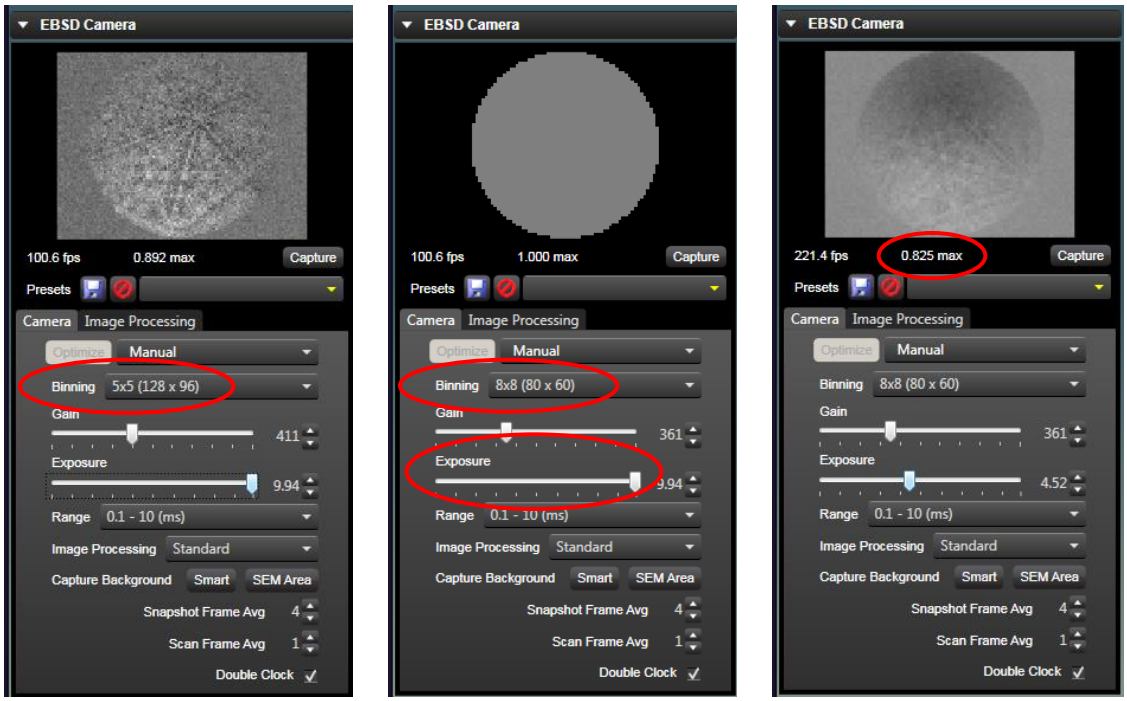
7. EBSD camera settings

7.1. Turn off the CCD camera in MiraTC; this is necessary to ensure the EBSD camera functions properly.

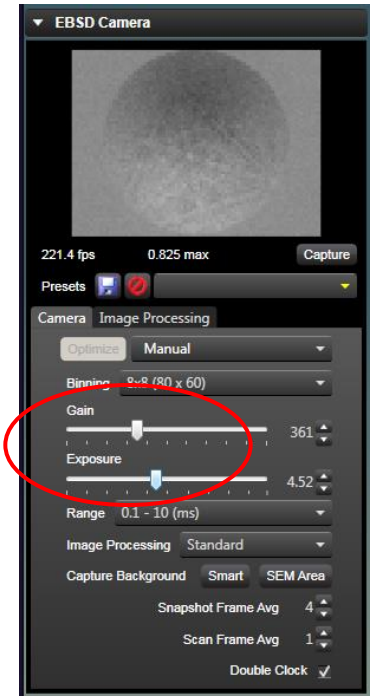
7.2. In TEAM, expand the “Advanced Properties” tab and select “EBSD Camera”; adjust “Gain” until an EBSD pattern is visible in the window; the saturation index (number below the pattern) should be 0.8 – 0.9.

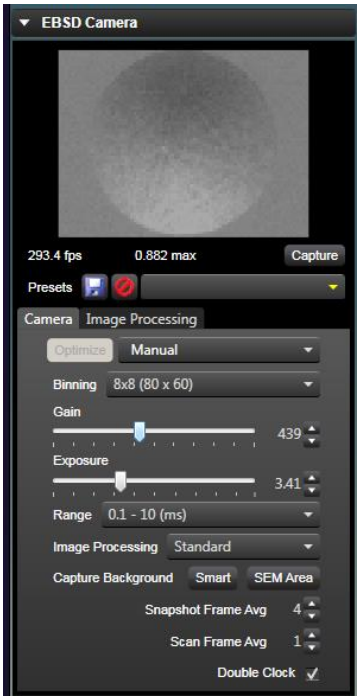


7.3. The default “Binning” (pixel resolution of the pattern) is 5×5. If the binning is *increased*, the “Exposure” of the camera should be reduced to bring the saturation index back within the optimal range. As a result, the camera speed will greatly *increase*, but pattern indexing will tend to become less accurate as this also decreases pattern resolution. Generally, the higher the beam voltage and the better the specimen quality, the higher the binning can be set when performing *mapping* (so it won’t take as long to produce the map). If you will be performing *point* analysis, you can use a low binning setting as only a few patterns will be collected anyways. For mapping purposes 5×5, 6×6, and 8×8 are usually sufficient

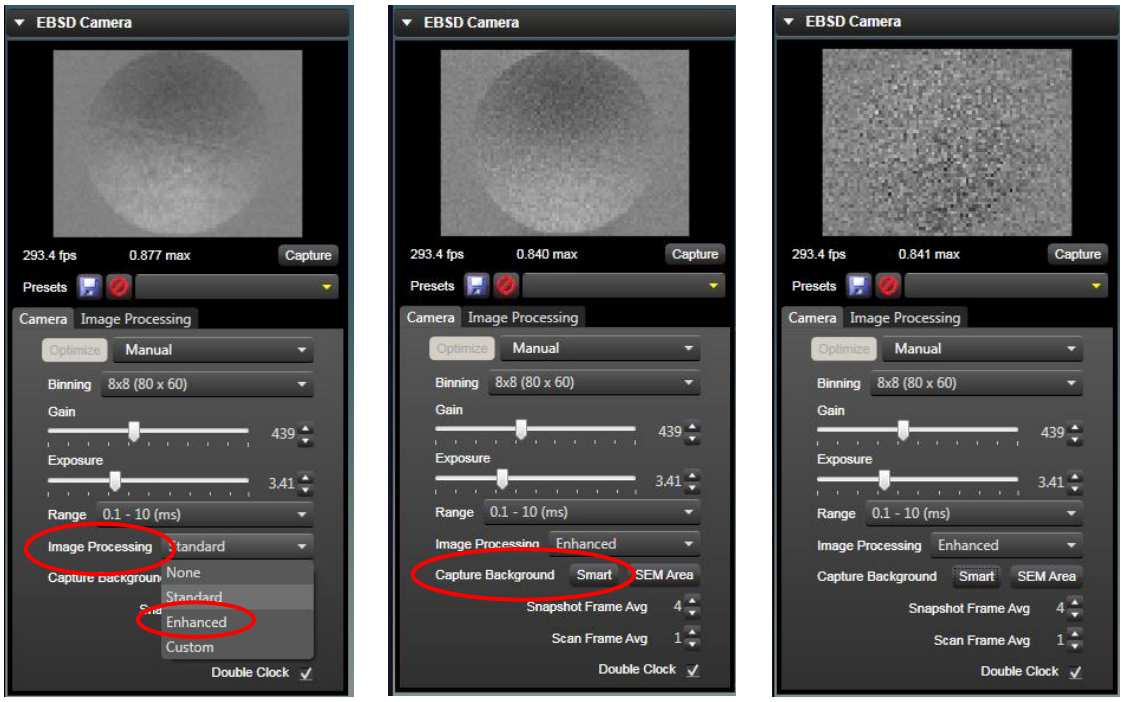


7.4. Once an appropriate binning has been selected, “Gain” and “Exposure” can be further adjusted. Increasing “Gain” will increase the saturation index without decreasing camera speed, but at the expense of a poorer signal-to-noise ratio (and require a concomitant decrease in “Exposure” to maintain an optimal saturation index). Conversely, increasing “Exposure” will increase the signal-to-noise ratio, but at the expense of decreased camera speed (and require a concomitant decrease in “Gain” to maintain an optimal saturation index). Generally, with higher beam voltages and good specimen quality, higher “Gain” and shorter “Exposure” can be used when performing ***mapping*** (again, to reduce the time it takes to produce the map). If you will be performing ***point*** analysis, you should opt for lower “Gain” and longer “Exposure” as only a few patterns will be collected anyways and speed is not a major concern.





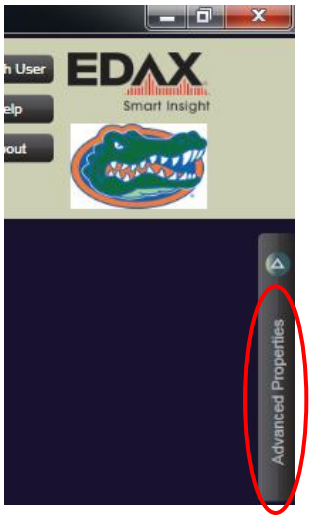
7.5. Increase the scanning speed to maximum value; under “Image Processing”, select “Enhanced”; then, next to “Capture Background”, select “Smart” to capture the background (the EBSD pattern should now look basically featureless).

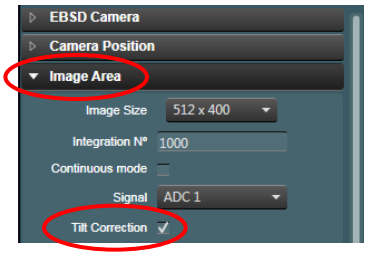


7.6. Decrease the scanning speed back to the previous setting (4 or higher); the camera is now ready to collect and index EBSD patterns.

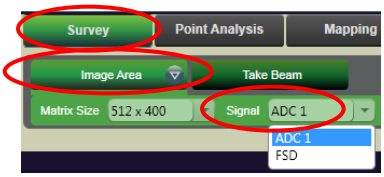
8. Imaging and tuning the indexing

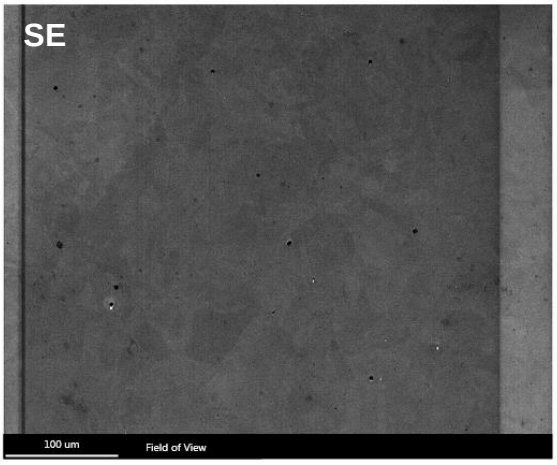
8.1. In TEAM, expand the “Advanced Properties” tab, select “Image Area”; and verify that “Tilt Correction” is enabled.

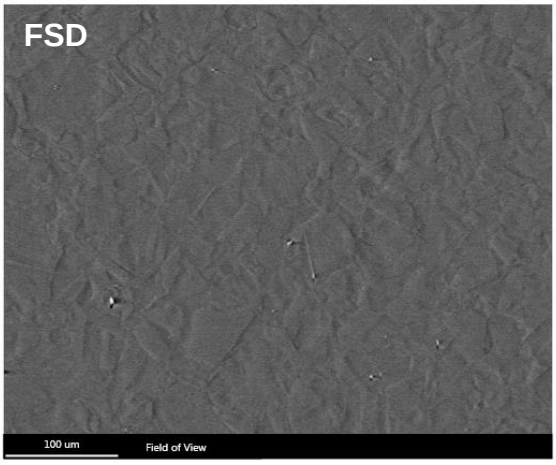




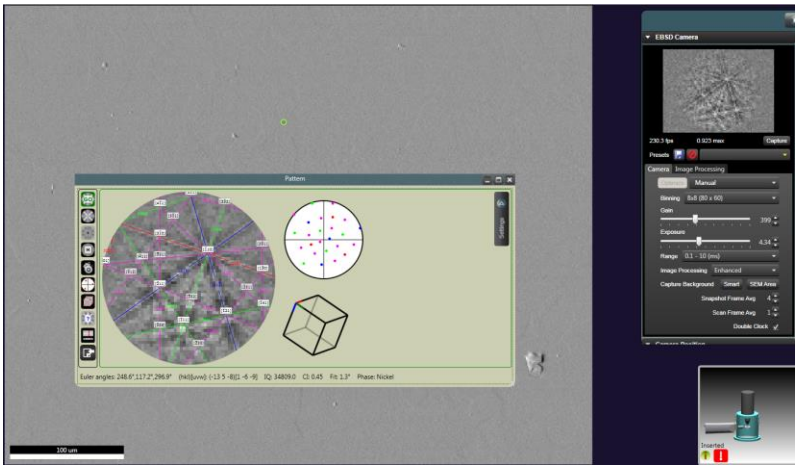
8.2. In TEAM, select “Survey” from the top menu bar to enter survey mode. Then hover over “Image Area” to see the options for “Matrix Size” (image resolution) and “Signal”. Selecting the “ACD 1” signal (default) will use the SE detector on the SEM to form the image while selecting the “FSD” signal will use the FSD on the EBSD camera to form the image. Generally, the FSD signal will be more useful for highlighting the grain structure of the specimen compared to the ACD 1 signal. When ready, select “Image Area” to collect an image.



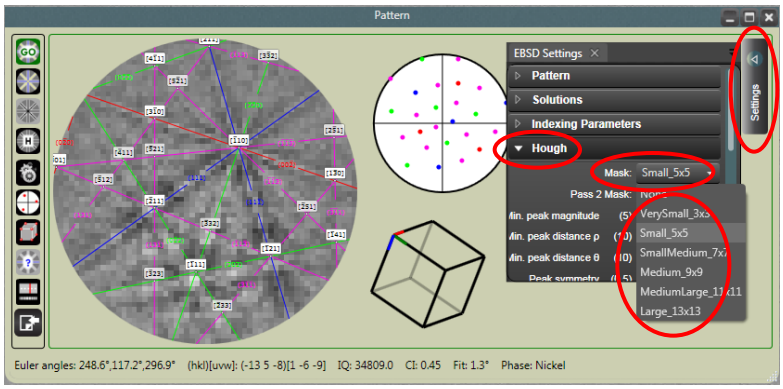




8.3. A small green circle will appear in the center of the acquired image, which corresponds to the position of the beam. If you click, hold, and drag on the image, you can move the beam position (and watch the EBSD pattern change accordingly as the beam is moved). Now click, hold, and drag on the image and you will see the live EBSD pattern under “EBSD Camera” change as you move the cursor. Once you find a position that produces a clear EBSD pattern, release the mouse and a “Pattern” window will pop up showing the indexed solution for the pattern.

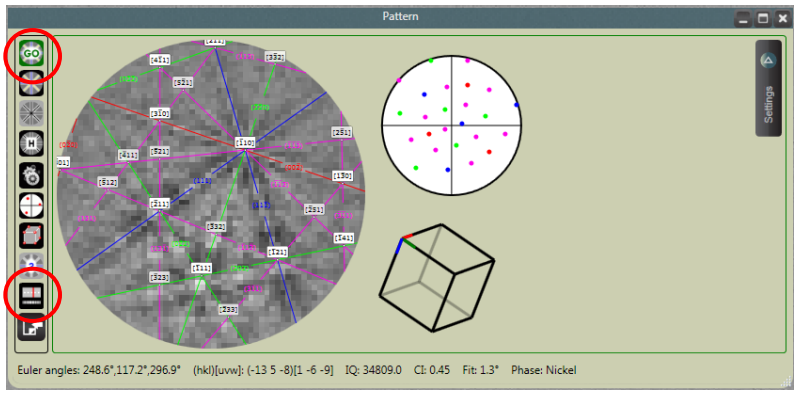


8.4. In the “Pattern” window, expand the “Settings” tab (right side of the window) and then select “Hough”. Select the pull-down menu next to “Mask” to see a list of options for the Hough mask (used to index the patterns); select the appropriate Hough mask for the current beam voltage.

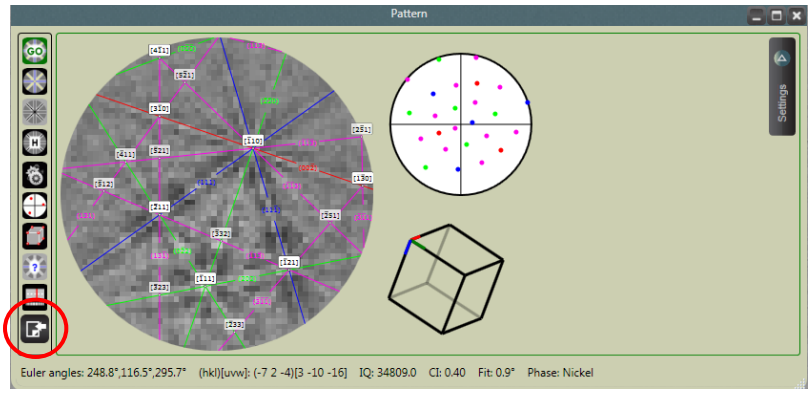


Beam voltage (kV)	Hough Mask
10	MediumLarge_11×11
15	Medium_9×9
20	Medium_9×9
25	SmallMedium_7×7
30	Small_5×5

8.5. If the Hough mask setting was changed, select “Go” to re-index the pattern and then “tune” to tune the solution. The indexed bands in the solution should coincide well with the actual bands in the pattern; if this is not the case, there may be a problem with the phase list and/or the indexing parameters (or the pattern may not be of sufficient quality).

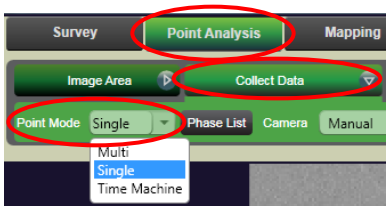


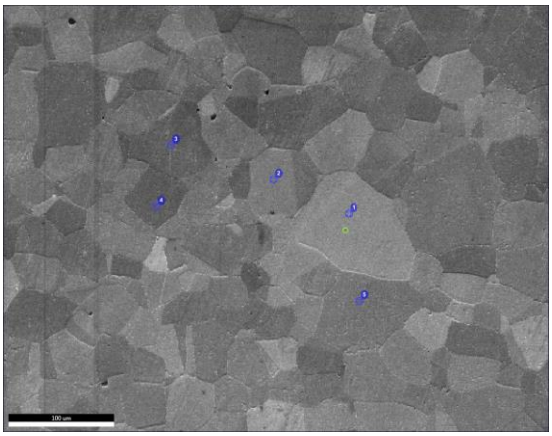
8.6. Once all tuning is complete, select the disk icon to save the pattern (and, more importantly, the tuning) to the project.

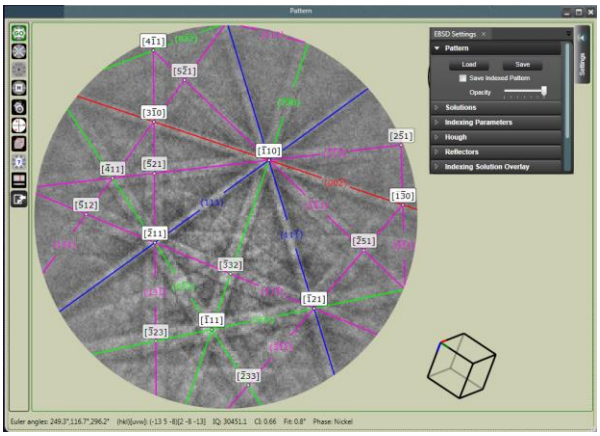


9. Performing EBSD in “Point Analysis” mode

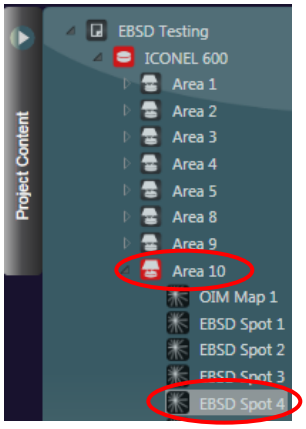
9.1. Select “Point Analysis” from the top menu bar; the image you acquired in survey mode will appear, so you don’t need to acquire another image; hover over the “Collect Data” button; under “Point Mode”, you can select options to analyze single or multiple points. If the “Single” option is selected, just click on the image where you want to analyze to start. If the “Multiple” option is selected, click on all the points on the image you want to analyze and then select the “Collect Data” button to start the analysis. As each point is analyzed, the “Pattern” window will pop up and show the acquired pattern and the indexed solution.

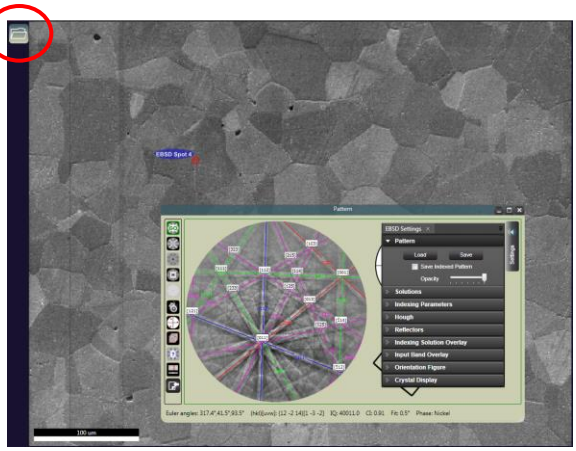




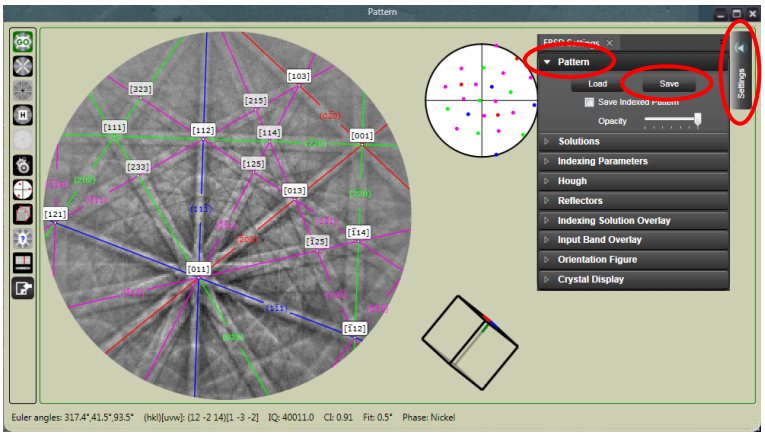


9.2. To recall a saved pattern, expand the “Project Content” tab and select the area of interest; the reference image will appear with all analysis points superimposed. Then select from the list of spots to show only that spot on the SEM image along with the associated EBSD pattern. To save the SEM image with the superimposed spot location to your designated folder, select the “Folder” button at the upper left side of the image. Keep in mind the EBSD pattern will not be saved when doing this (see next step).



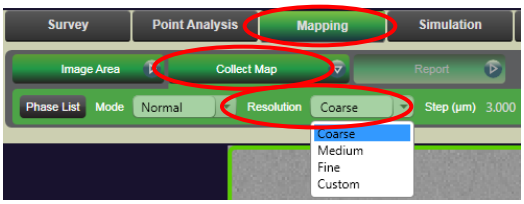


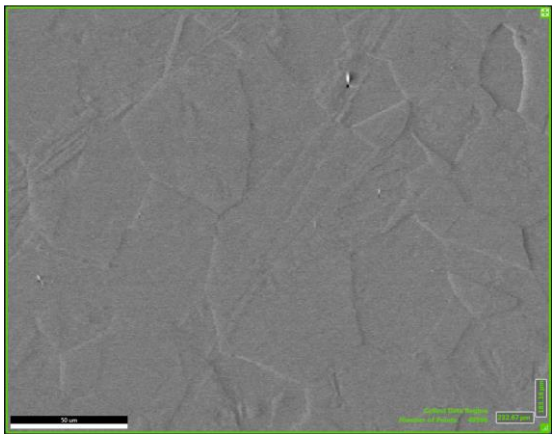
9.3. To save the EBSD pattern, go to the “Pattern” window and expand the “Settings” tab; under “Pattern”, you can elect to have the solution overlay saved by checking “Save Indexed Pattern”. Select “Save” to save the pattern.



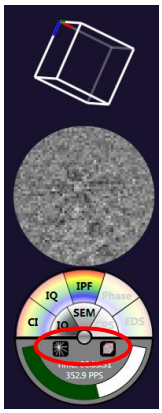
10. Performing EBSD in “Mapping” mode

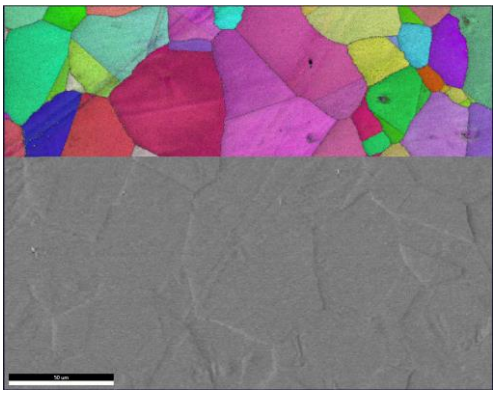
10.1. Select “Mapping: from the top menu bar; the image you acquired in survey mode will appear, so you don’t need to acquire another image. Hover over the “Collect Map” button and then select an option for map resolution. As the resolution increases, more points will be mapped (as indicated in the lower right corner of the green defining box), so the time required to performing mapping will increase. Once a map resolution is selected, select “Collect Map” to start mapping



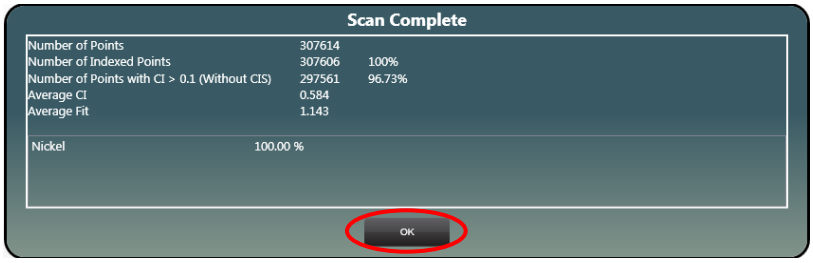


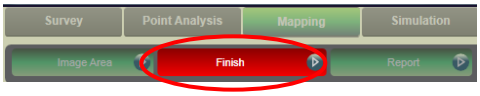
10.2. A live crystal orientation (or inverse pole figure) map showing the specimen normal direction overlaid on a pattern quality map will be generated. To view the live EBSD pattern and indexed unit cell orientation, select the EBSD pattern and unit cell icons from the compass (lower right side of window), respectively.



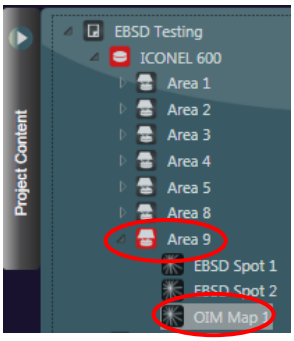


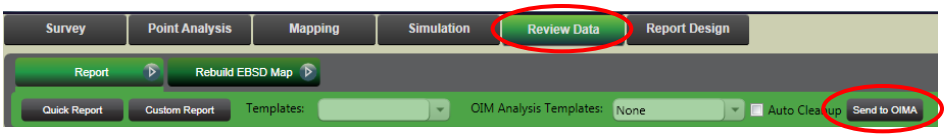
10.3. When the map is finished, the “Scan Complete” window will pop up giving a synopsis of the mapping results. Select “OK” to close the window and then select “Finish” to save the map.





10.4. Further processing, analysis, and saving of map data (IPF maps, grain maps, grain boundary maps, etc.) should be done using OIM Analysis. To export the data to OIM Analysis, select the area of interest and then the map of interest from the project tree; then select “Review Data” from the top menu bar; hover over “Report” and select “Send to OIMA” to export the data to OIM Analysis. OIM Analysis will immediately open with the imported data set.





11. Changing analysis areas on the same specimen

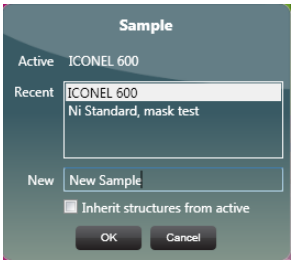
11.1. In the live SEM image, you can move either directly to the left or right and still reasonably maintain WD = 20 mm without any possible risk of collision with the camera. After moving, you should finely refocus the center of the image. *Do not attempt any other types of stage movements with the camera inserted.*

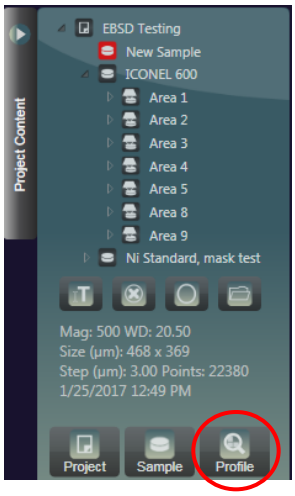
12. Changing samples; manipulating project content

12.1. If you want to change specimens and add another sample to the project, first retract the EBSD camera and unload the current specimen as described in the next step (you do not need to close TEAM or MiraTC). Then, load the new sample, set the correct stage tilt and working distance, and reinsert the EBSD camera.

12.2. Generally speaking, if you keep the beam settings the same between specimens, the SEM alignment will still be reasonably accurate when the new sample is loaded and brought to WD = 20 mm (though it doesn't hurt to check it again anyways). That being said, if the beam voltage and/or intensity are changed for analyzing a new sample, the complete SEM alignment should be repeated.

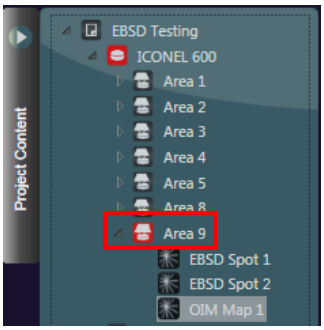
12.3. From the expanded "Project Content" tab, select "Sample" and follow the instructions to create a new sample and follow the instructions to create a new sample. If the material(s) is (are) the same as the previous sample, you can check "Inherit structures from active". Otherwise, from the expanded "Project Content' tab, select "Profile" and follow the instructions to add new phases.



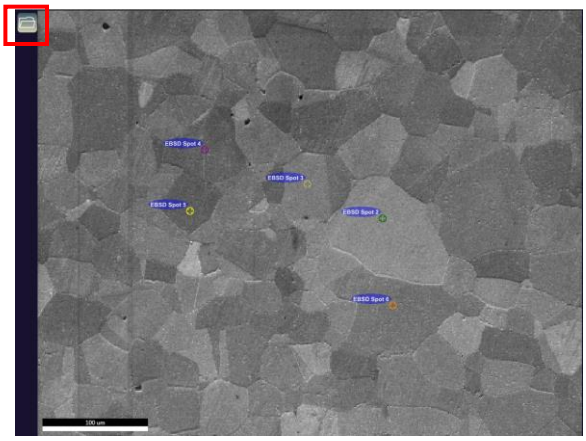


12.4. <u>Every time a new sample is loaded and/or beam conditions are changed, you should reconfigure the EBSD camera (binning, exposure, gain, background capture) and tuning of the EBSD pattern indexing.</u>

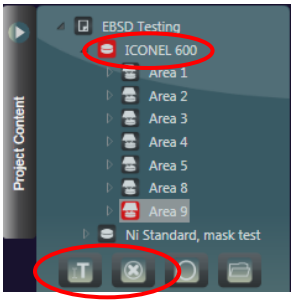
12.5. Expand the “Project Content” tab (upper left side of window); expand the current sample to see a list of all the imaged areas used for analysis (every time “Image Area” is selected, this generates a new area). Then expand each area to see a list of all the data (EBSD spots and OIM maps) associated with that area.



12.6. Double clicking on an area will bring up the SEM image collected for that area. If any point analysis was done on that area, the analysis points will also be shown on the image. To save the image to your designated folder, hover over the image and select the “folder” button (top left side of the image); any analysis points will be overlaid on the saved image.



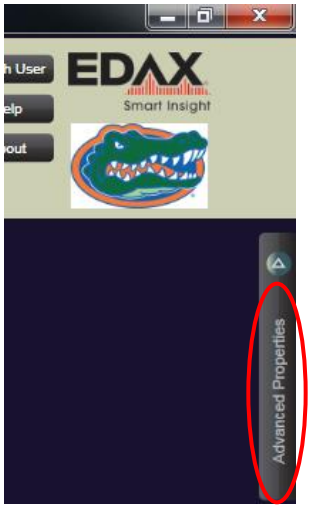
12.7. Any sample, area, EBSD spot, or OIM map can be renamed or deleted from the project by selecting the item and then selecting the “Edit” or “Delete” buttons, respectively.

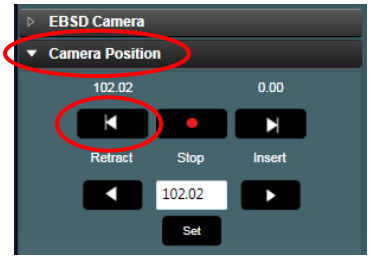


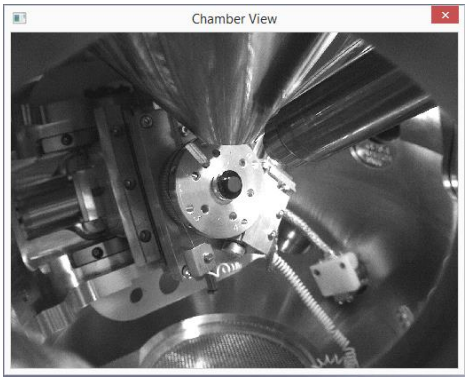
13. Finishing the session

13.1. In MiraTC, turn the CCD camera back on.

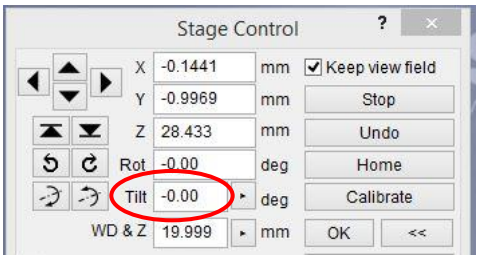
13.2. Expand the “Advanced Properties” tab and then “Camera Position”; select “Retract” to retract the camera (you should hear the motor start). The “Chamber View” panel should look similar to below after retraction is complete (and you should hear the motor stop, too).

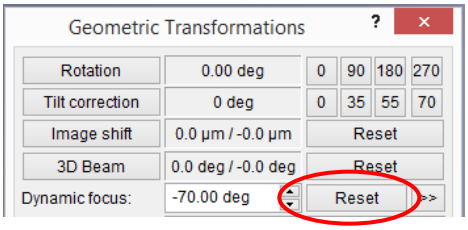






13.3. In the “Stage Control” panel, tilt the stage back to 0° (this does not need to be done incrementally). In the “Geometric Transformations” panel, select “Reset” to turn off dynamic focusing.





13.4. Close the TEAM software (all data is saved automatically).

13.5. Turn off the beam, calibrate the stage, vent the chamber, remove the specimen (and stub extender, if applicable), pump the chamber back down, and log off MiraTC.