

FEI Helios G4 PFIB CXe: EBSD using EDAX TEAM

Nicholas G. Rudawski

ngr@ufl.edu

Office: (352) 392-3077

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ANALYSIS OF RADIOACTIVE SPECIMENS IS STRICTLY PROHIBITED

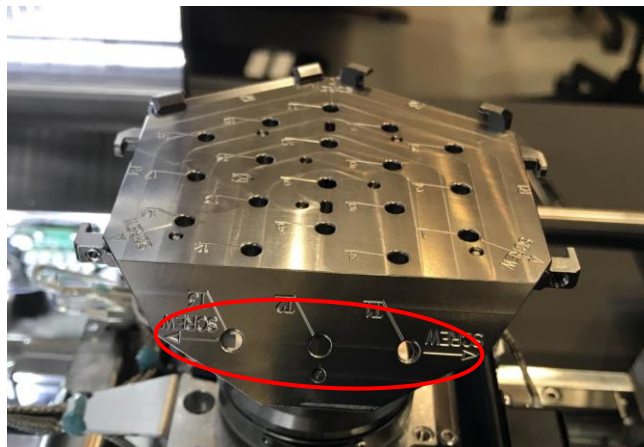
This document assumes the user is already familiar with and proficient in basic operation of the instrument and the Microscope Control user interface; certain details are thus omitted for purposes of clarity.

1. Sample mounting, preparation, and constraints

- 1.1. EBSD specimens should be mounted on ~13 or ~25 mm diameter 3 mm-thick pin stubs; the preferred stubs for this are Ted Pella #16111 (13 mm diameter) or #16144 (25 mm diameter). The specimens should have a flat, damage-free, and smooth surface and have a footprint that fits basically within the surface of the stub, the thickness to footprint ratio of the specimen should be kept as small as possible to 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 mounting on a stub to ensure a path to ground is produced.

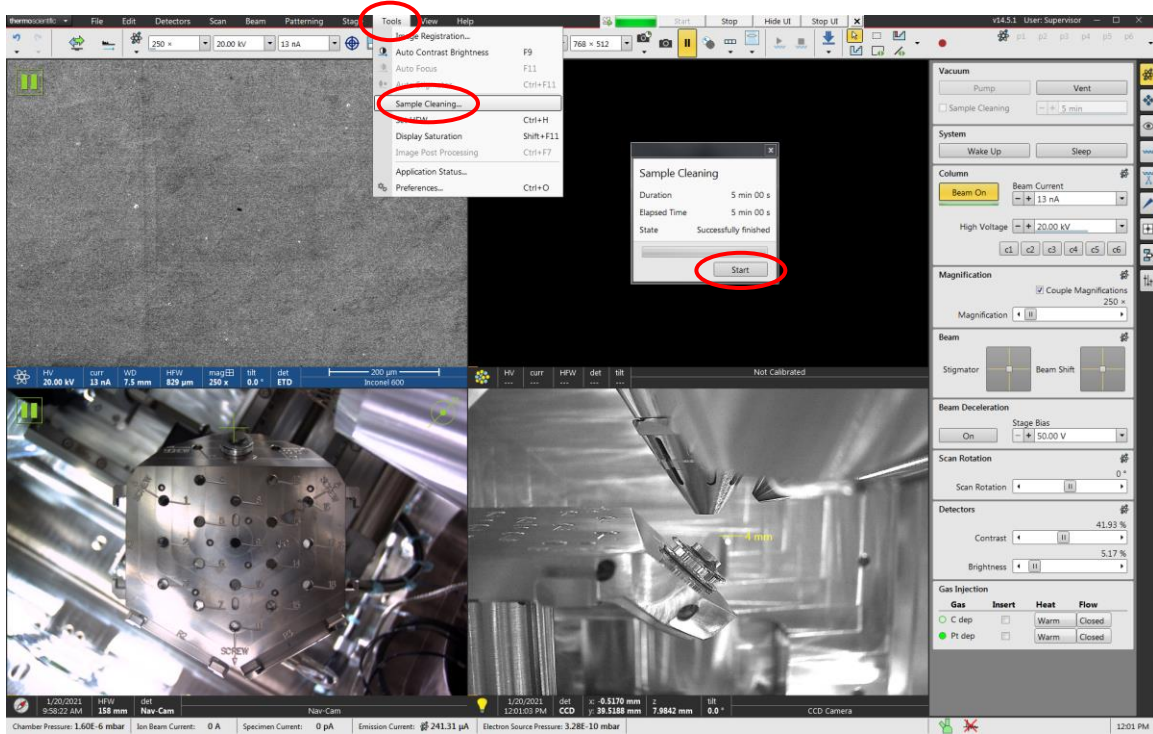
2. Specimen loading

- 2.1. Specimens must be loaded in one of the 3 positions on the 45° pre-tilted section of the multi-purpose holder (note that the outer two positions require securing with screws; rotating the stage – 90° provides easy access to each.



3. Plasma cleaning

3.1. Once all specimens are loaded and the chamber pumped down, it is advisable to plasma clean the specimens for optimal EBSD results. In Microscope Control, navigate to the “Tools” pull-down menu, then select “Sample Cleaning” and then “Start” (about 5 min to complete).



4. Instrument settings

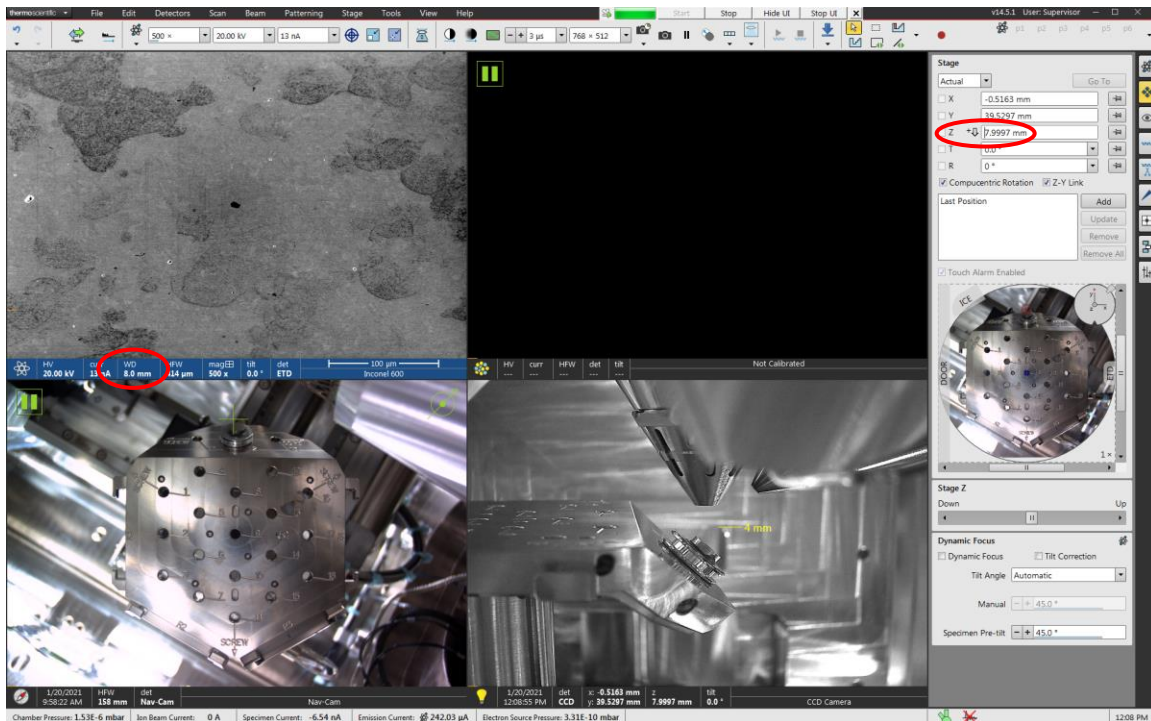
4.1. EBSD is generally performed at voltages of 10 – 30 kV using a few to 10s of nA of current. In principle, the spatial resolution of EBSD improves as the beam voltage is decreased, but this also results in poorer signal to noise in the EBSD patterns; decreasing the current will also improve spatial resolution via reduced probe size, but again also at the expense of poorer signal to noise in the EBSD patterns.

4.1.1. 20 kV with 6.4 or 13 nA of current is recommend as a general all-around setting for performing EBSD.

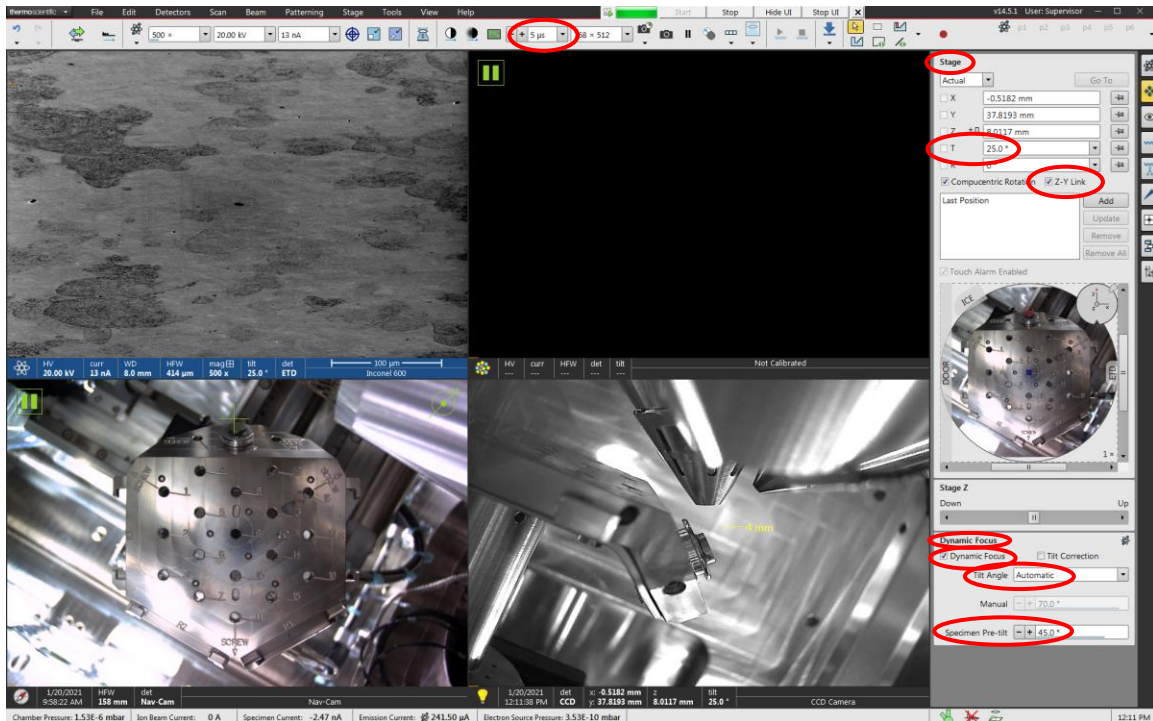
4.1.2. If unsure as to what beam settings to use for EBSD, please consult with staff for recommendations.

4.2. Navigate to the specimen and find an area of interest (ideally, in the middle of the piece away from any edges) and bring it to WD = 8 (this is the WD for optimal EBSD camera performance).

4.2.1. At this point, perform basic E-beam alignment (source tilt, lens alignment, and astigmatism correction).

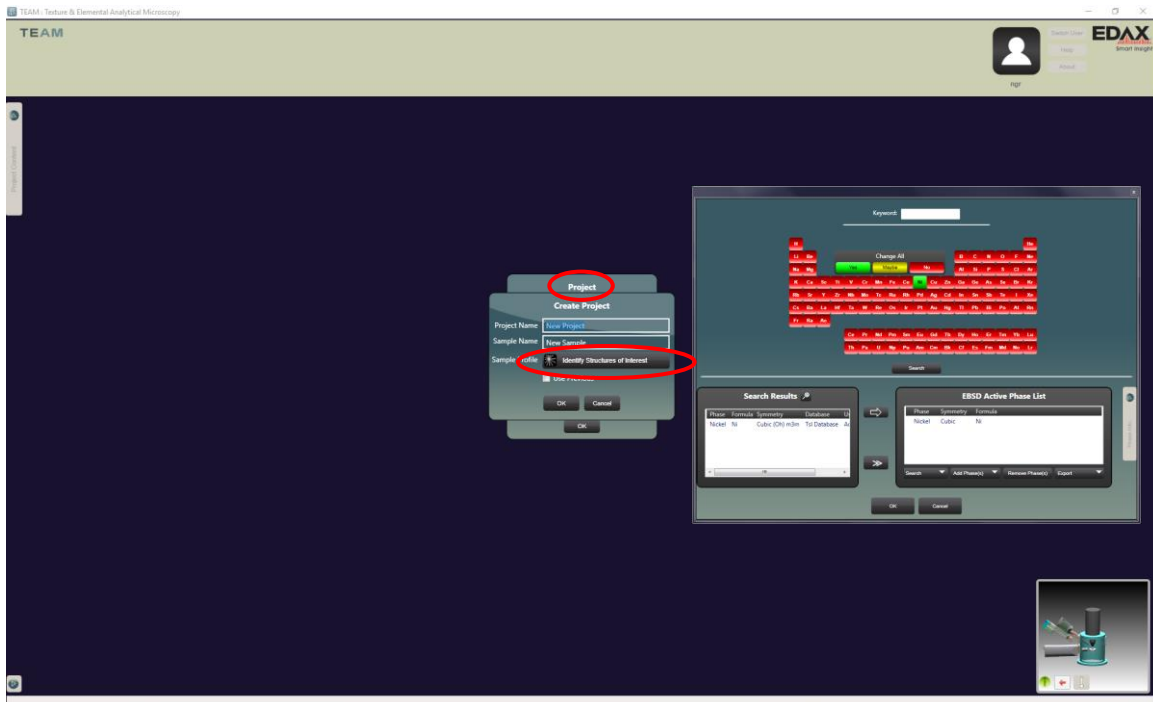


- 4.3. Center a recognizable feature in the live image, enter the Navigation module, and navigate to the “Stage” panel.
 - 4.3.1. Check “Z-Y Link”; this ensures the area of interest remains centered even though the sample is not at eucentric height (WD = 4 mm).
 - 4.3.2. Tilt the stage in 5° increments until T = 25° is achieved (actual specimen tilt is now 70°).
 - 4.3.3. Recenter and refocus the feature; WD should still be ~ 8 mm.
- 4.4. Remain in the Navigation module and navigate to the “Dynamic Focus” panel.
 - 4.4.1. Check “Dynamic Focus”.
 - 4.4.2. Set “Tilt Angle” to “Automatic”.
 - 4.4.3. Set “Specimen Pre-tilt = 45°”.
 - 4.4.4. Focus precisely on the middle of the image (use of the reduced scanning area may help here).
 - 4.4.5. Adjust the magnification as appropriate for EBSD mapping.
 - 4.4.6. Set the dwell time $\geq 5 \mu\text{s}$ for dynamic focus to work properly.



5. Starting TEAM and setting up a project file

- 5.1. Open the TEAM software, enter the log in name, select the “EBSD” option at the log in screen, and log in.
- 5.2. The “Project” dialogue box will pop up. Assuming this work is part of a new project, select the option to create a new project; in the “Create Project” dialogue box, name the project and specimen as desired.
- 5.3. Select “Identify Structures of Interest” and use the period table to add candidate structures to be used for the analysis.
- 5.4. At least one candidate structure must be added to perform EBSD, but it is best to keep the number of candidate structures to as small as possible (i.e., if the material is known to be only one phase, use only that phase as the candidate structure and no others).



6. Inserting the EBSD camera

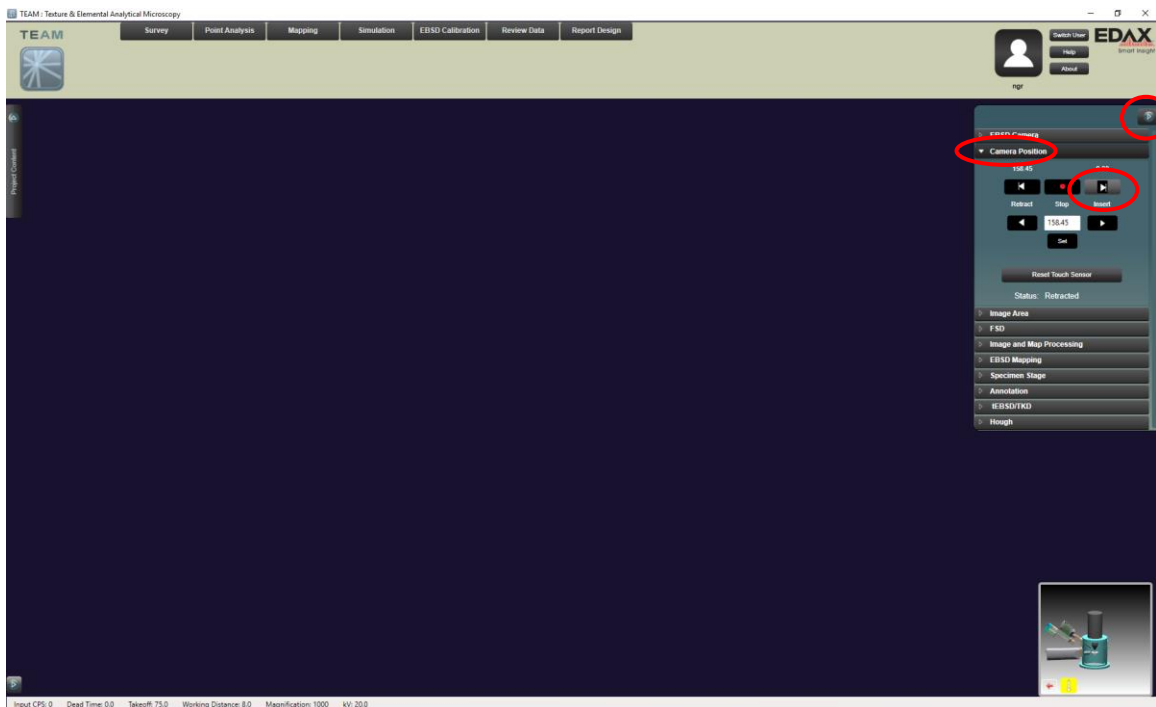
6.1. The stage tilt must be set to 25° and the SEM image in focus at WD ~ 8 mm prior to insertion of the EBSD camera.

6.1.1. 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”.

6.2.1. Select “Insert” to insert the EBSD camera (listen for the motor to start).

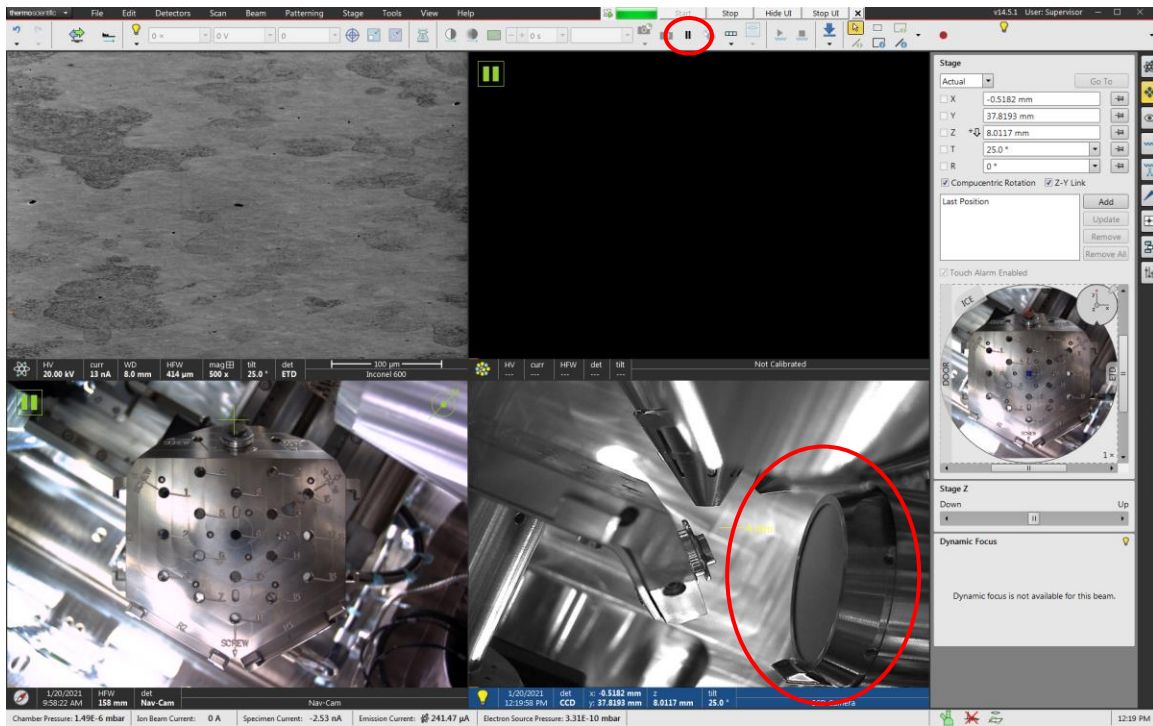
6.2.2. In Microscope Control, monitor the insertion process in the “CCD Camera” quad and be prepared to select “Stop” if any collision (though unlikely) appears imminent.



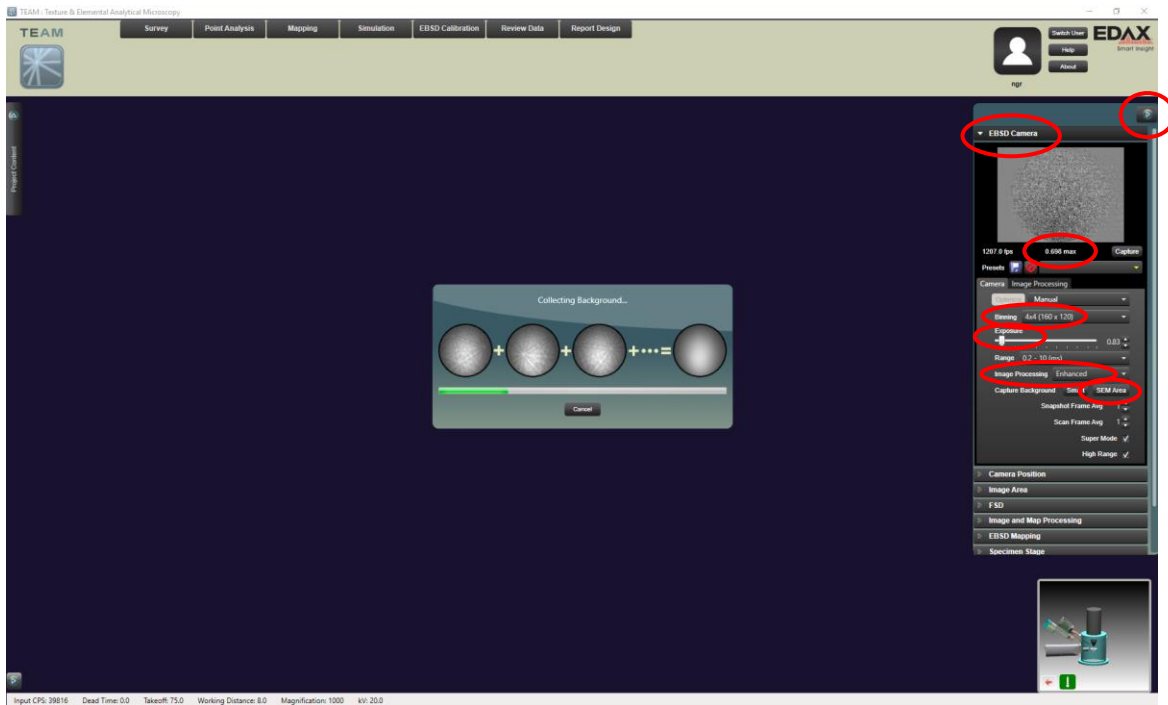
6.3. In Microscope Control, the CCD Camera quad should look similar to as shown below once camera insertion is completed.

6.3.1. Select the CCD Camera quad and then “Pause” from the main toolbar to turn off the CCD Camera.

6.3.2. This is necessary for optimal EBSD camera performance; the signal from the CCD camera will otherwise interfere with the EBSD signal.



7. EBSD camera settings for polycrystalline samples
 - 7.1. In Microscope Control, verify the magnification is set as appropriate for performing EBSD mapping and the image is focused.
 - 7.2. In TEAM, expand the “Advanced Properties” tab and select “EBSD Camera”.
 - 7.2.1. Set “Binning” = 4×4 if the patterns **do not** need to be saved (usually the case when the patterns are high quality).
 - 7.2.2. Set “Binning” = 8×8 if needing to save the patterns (useful for post processing when patterns are low quality and/or very noisy).
 - 7.2.3. Set “Image Processing” to “Enhanced” (again, assuming the sample is polycrystalline).
 - 7.2.4. Adjust “Exposure” to set the pattern intensity (number below pattern); a longer exposure will improve pattern quality, but at the expense of camera speed. Pattern intensity = 0.1 is generally the lowest value that will produce usable EBSD patterns.
 - 7.2.5. Next to “Capture Background”, select “SEM Area” to automatically capture the background for the area being scanned.



8. Image Area settings

8.1. In TEAM, expand the “Advanced Properties” tab and select “Image Area”.

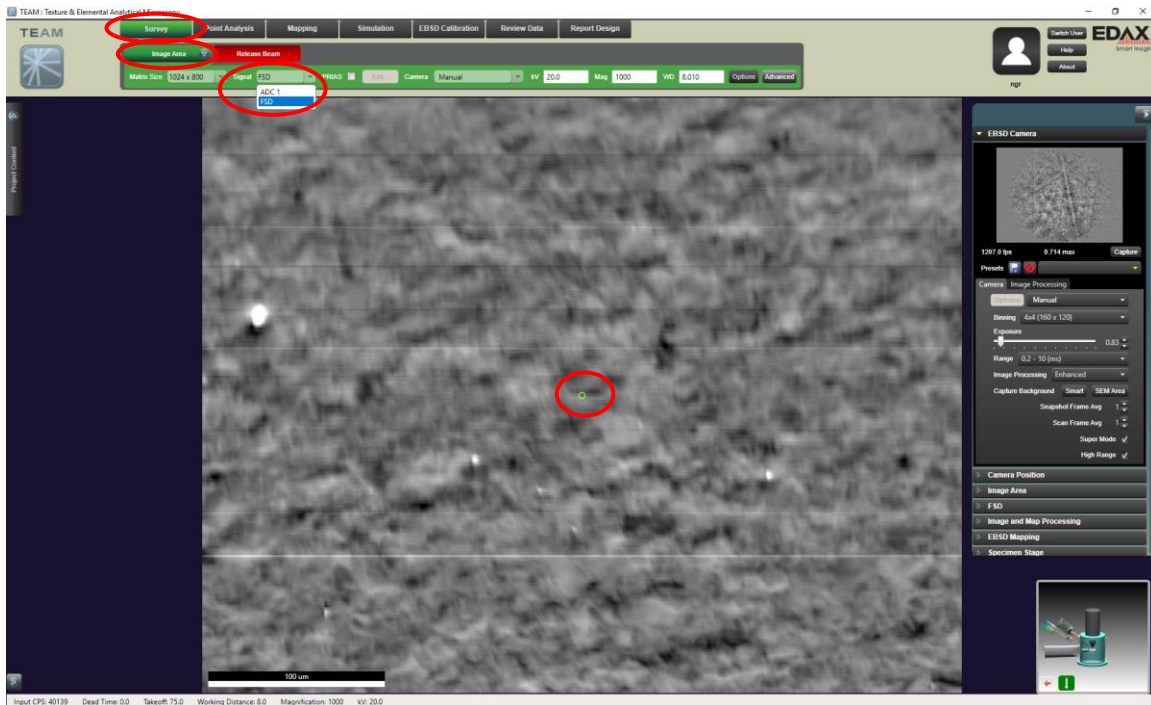
8.2. Verify “Tilt Correction” is checked.

8.3. Verify “Pre-Tilt” is set to 45°.



9. Survey imaging and index tuning

- 9.1. Before proceeding, return to Microscope Control and accurately focus the live SEM image; the focus is often noticeably off after background collection.
- 9.2. In TEAM, select “Survey” from the top menu bar to enter survey mode. Then hover over “Image Area” and select the “Signal” pull-down menu. After selecting a signal, select “Image Area” to collect an image.
 - 9.2.1. Selecting the “ACD 1” signal (default) will use the ETD detector on the SEM to collect the image while selecting the “FSD” signal will use the FSD on the EBSD camera to collect the image.
 - 9.2.2. Generally, the FSD signal will be more useful for highlighting the grain structure of the specimen compared to the ETD signal but collecting the FSD image may take longer.
 - 9.2.3. The signal used for collecting the SEM survey image has no impact on the resulting EBSD map.



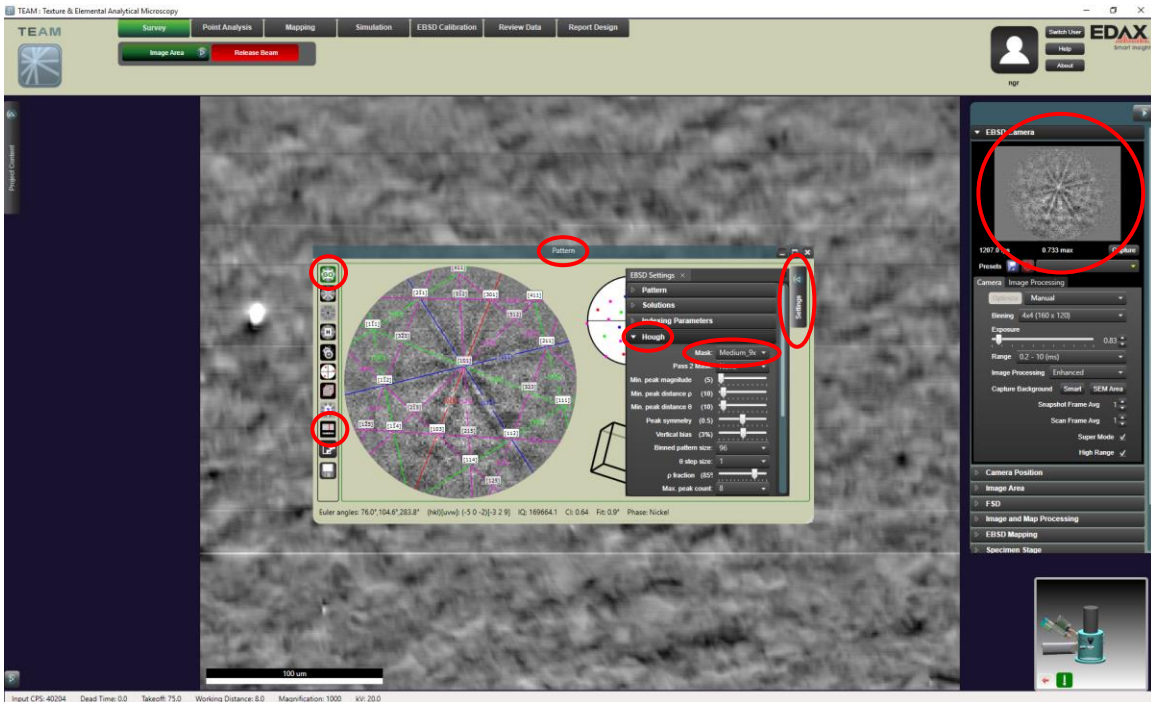
9.3. A small green circle will appear in the center of the acquired image, which corresponds to the position of the beam. If you click, hold, and drag on the image, you can move the beam position (and watch the EBSD pattern change accordingly as the beam is moved). Once you find a position that produces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

9.3.1. In the “Pattern” window, expand the “Settings” tab and select the “Hough” panel.

9.3.2. Select the “Mask” pull-down menu 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_11x11
15	Medium_9x9
20	Medium_9x9
25	SmallMedium_7x7
30	Small_5x5

9.3.3. In the “Pattern” window, select “Go” to re-index the pattern and then “tune” to tune the indexing. Verify that the solution coincides well with the actual bands in the pattern before proceeding.



10. Collecting the EBSD map

- 10.1. In TEAM, select “Mapping” from the top menu bar; the image you acquired in “Survey” mode will appear, so there is no need to acquire another image.
- 10.2. If desired, the individual patterns acquired during EBSD mapping can be collected and saved. Expand the “Advance Properties” tab and select “EBSD Mapping”.
 - 10.2.1. Check “Save Patterns”.
 - 10.2.2. Only camera binning = 8×8 (or higher) should be used when recording patterns.
 - 10.2.3. Pattern recording can improve results during subsequent data processing if the patterns are of lower quality.
 - 10.2.4. Only save the patterns, if necessary, since saving the patterns will dramatically increase the size of the data set.



10.3. Hover over (but do not select yet) the “Collect Map” button and select the “Resolution” pull-down menu to see options for map resolution.

10.3.1. As the resolution increases, more points will be mapped (as indicated in the lower right corner of the green defining box).

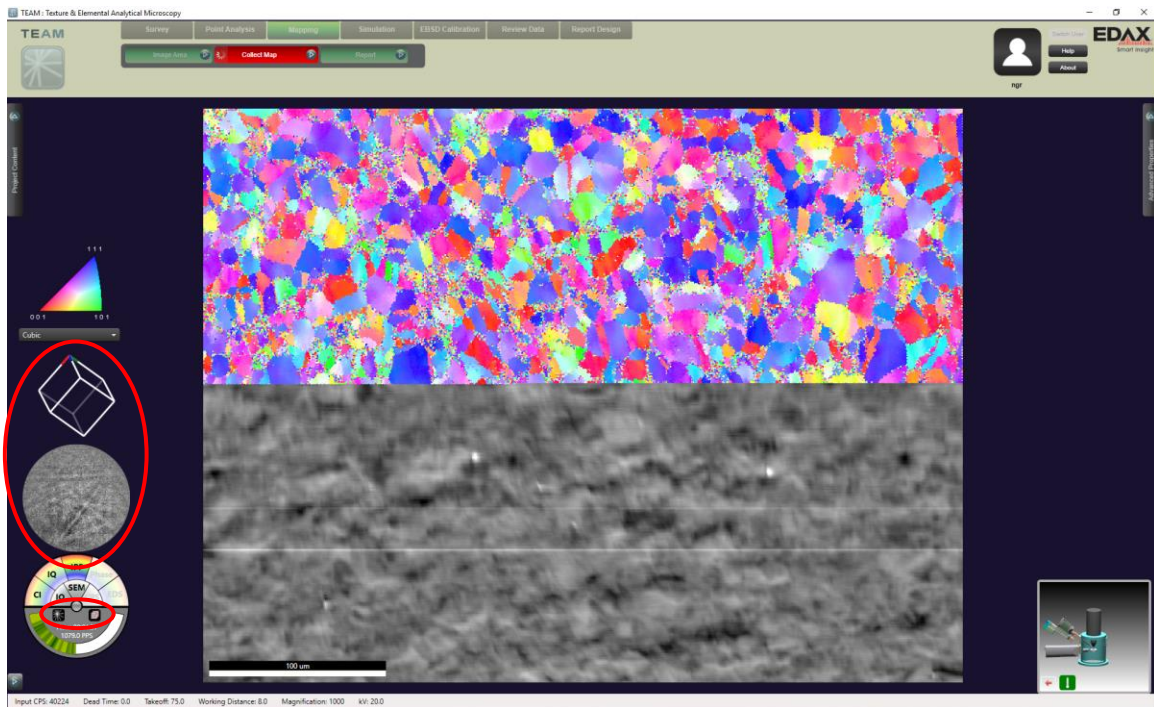
10.4. Once a map resolution is selected, select “Collect Map” to start mapping.

10.4.1. The “Fine” resolution is recommended for the highest quality mapping.



10.5. While mapping, a live crystal orientation (IPF) map showing the set of crystallographic planes running parallel to the specimen surface overlaid on a pattern quality map will be generated.

10.5.1. To view the live EBSD pattern and solved unit cell orientation, select the EBSD pattern and unit cell icons from the compass (lower left side of window), respectively.



10.6. When the map is finished, a “Scan Complete” window will pop up giving a synopsis of the mapping results.

10.6.1. Select “OK” to close the “Scan Complete” window.

10.6.2. Then select “Finish” to save the map.

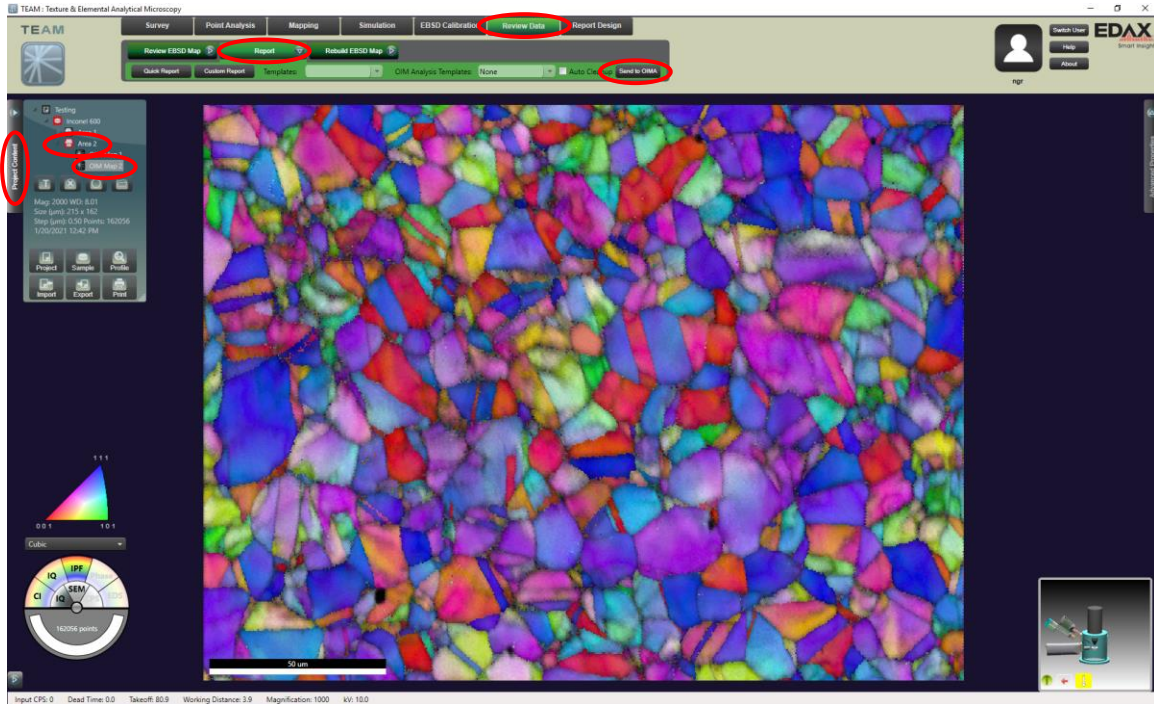


10.7. Further processing, analysis, and saving of map data may be done using OIM Analysis; expand the “Project Content” tab.

10.7.1. Select the area of interest and then map of interest.

10.7.2. Select “Review Data” from the top menu bar

10.7.3. Hover over (but do not select) “Report”, then select “Send to OIMA”; OIM will open automatically with the imported map data.

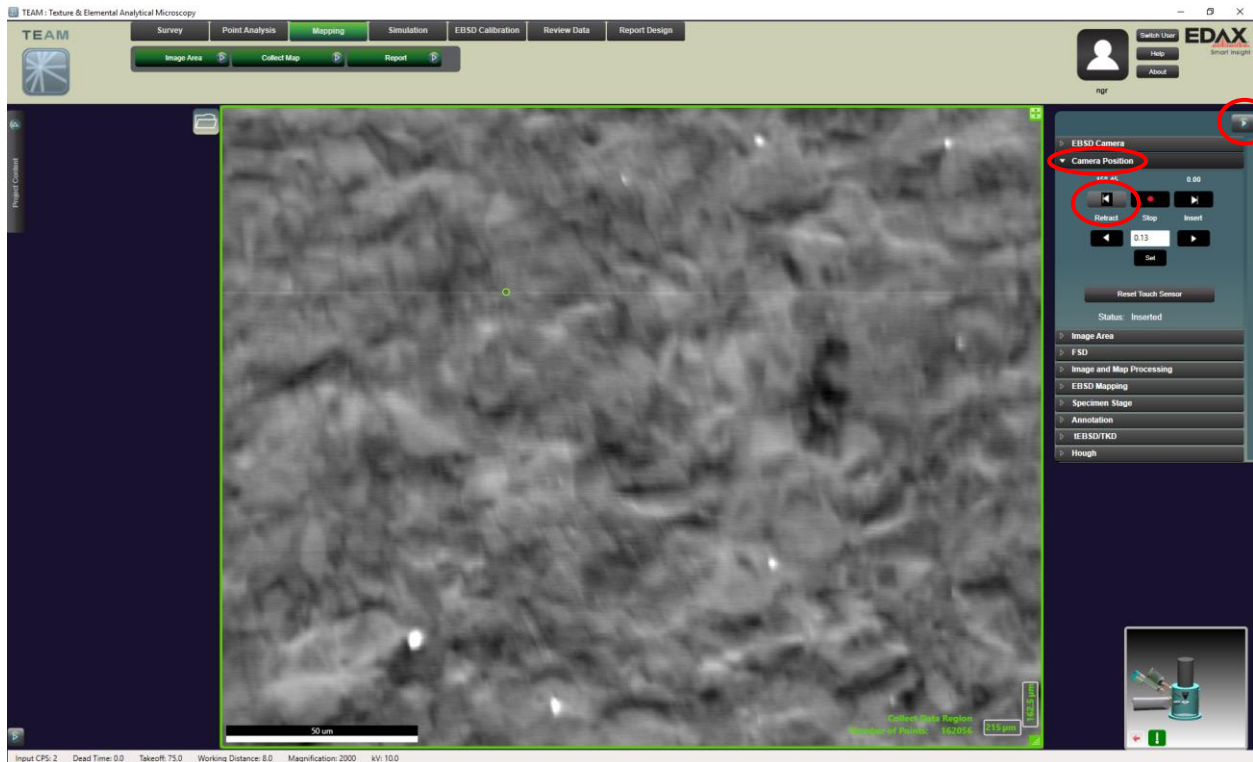


11. Finishing the session

11.1. In Microscope Control, select the CCD camera quad and un-pause the image.

11.2. In TEAM, expand the “Advanced Properties” tab.

11.2.1. Select “Camera Position” and then “Retract” to retract the camera (listen to hear the motor start).



11.3. Wait for the camera motor to stop before proceeding; in Microscope Control, the CCD camera quad should look similar that shown below when the EBSD camera is fully retracted.

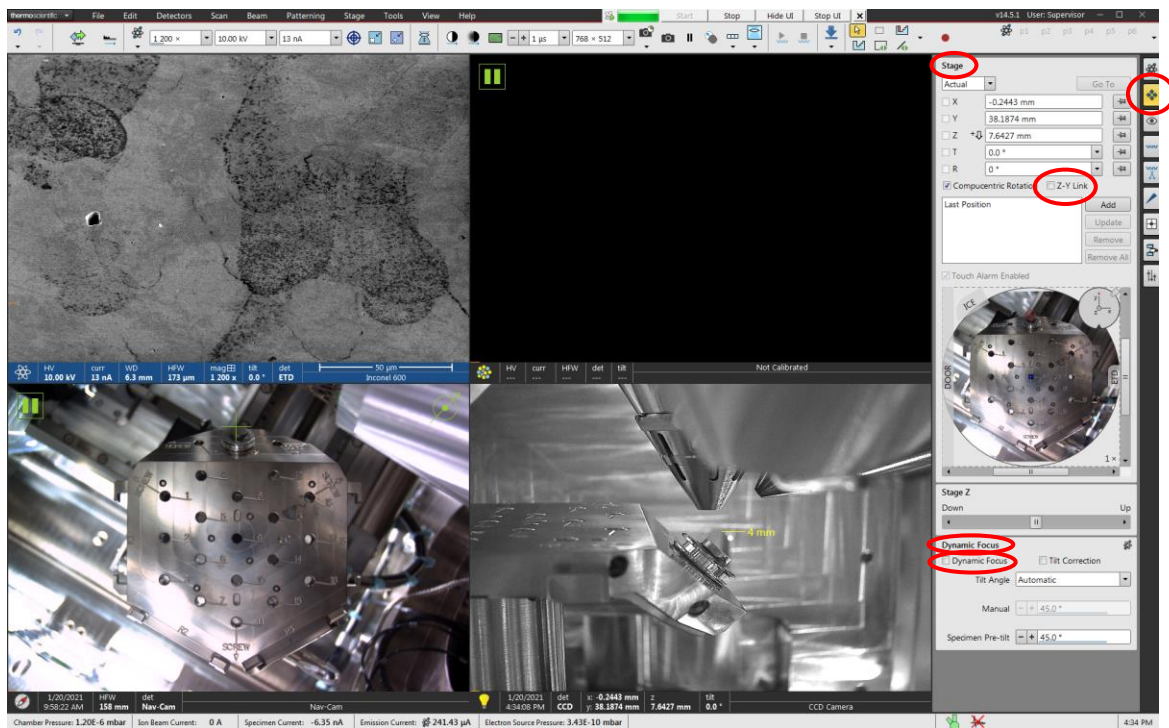
11.4. Enter the Navigation module and navigate to the “Stage” panel.

11.4.1. Uncheck “Z-Y Link”.

11.5. Remain in the Navigation module and navigate to the “Dynamic Focus” panel.

11.5.1. Select the “Tilt Correction” tab.

11.5.2. Uncheck “Dynamic Focus”.



11.6. Finish the session as per usual (turn off beam, home stage, vent chamber, remove specimens, return chamber to vacuum).