FEI Helios G4 PFIB CXe: EBSD using EDAX TEAM

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ANALYSIS OF RADIOACTIVE SPECIMENS IS STRICTLY PROHIBITED

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

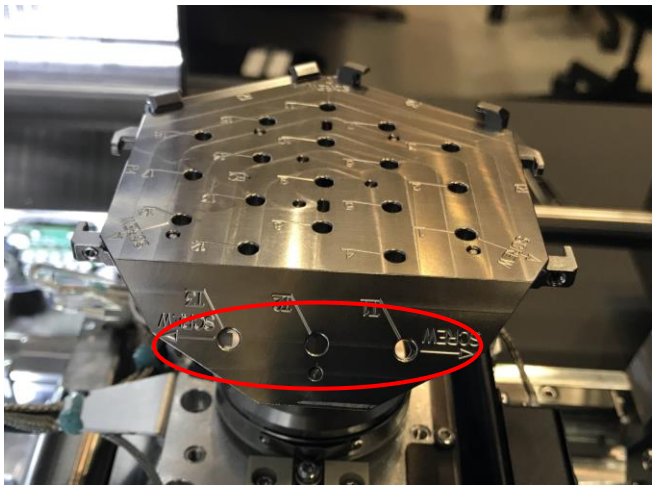
1. Sample mounting, preparation, and constraints

    1.1. EBSD specimens must have 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 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* being mounted on a stub to ensure a path to ground is produced.

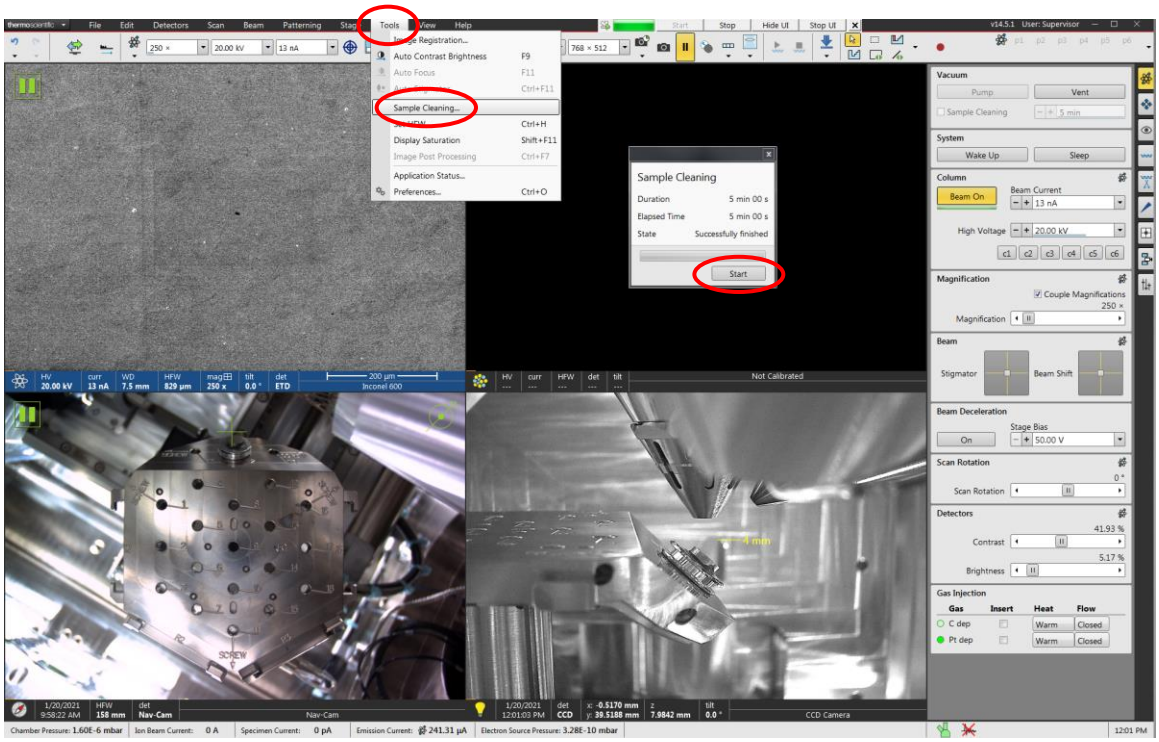
2. Specimen loading

    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).



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).

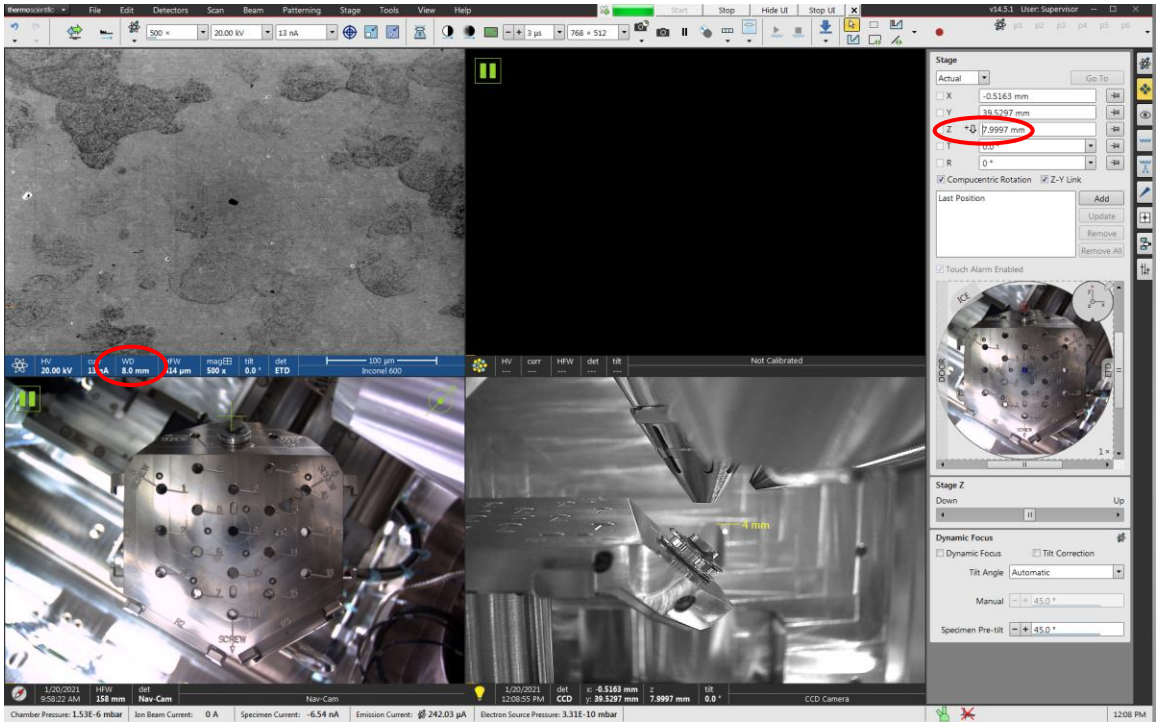


4. Instrument settings

4.1. EBSD is generally performed at voltages of 10 – 30 kV using several 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; If you are unsure as to what beam settings you should use for EBSD, please consult with staff for recommendations.

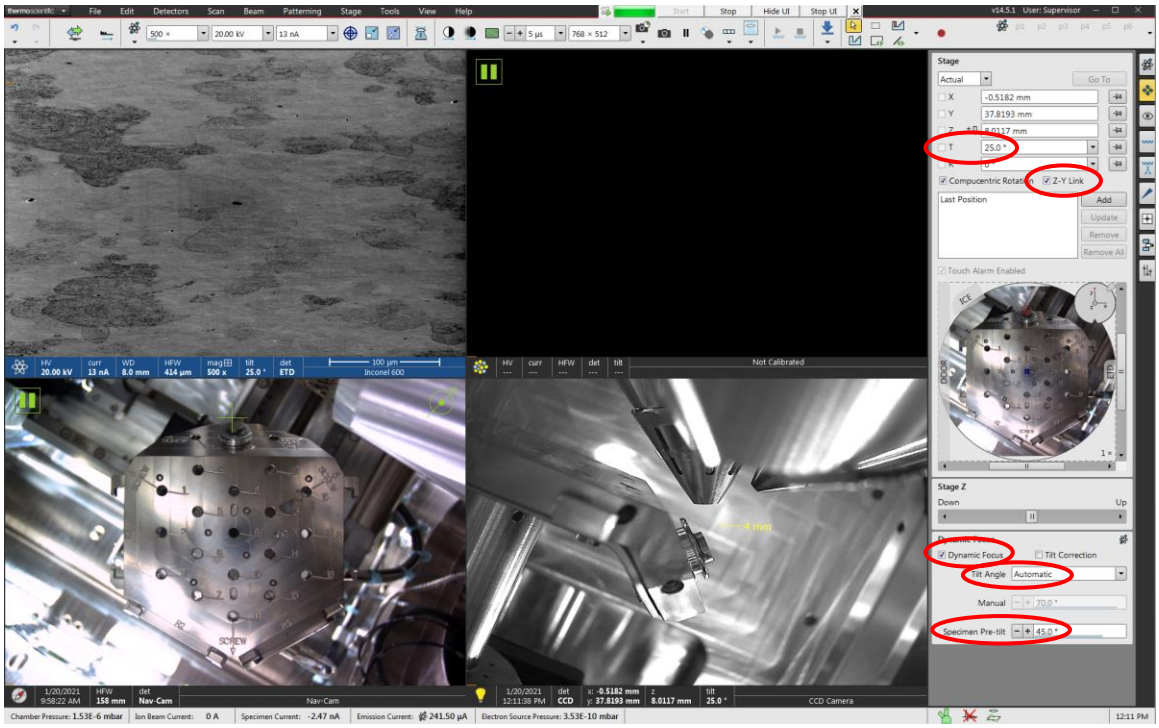
4.2. Navigate to the specimen and find an area of interest (ideally, in the middle of the piece away from any edges) and bring it to WD = 8 (this is the WD for optimal EBSD camera performance).

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 and enter the Navigation module; check “Z-Y Link” and then tilt the stage in 5° increments until T = 25° is achieved (actual specimen tilt is now 70°); recenter and refocus the feature.

4.4. Check “Dynamic Focus” and set “Tilt Angle” to “Automatic”; set “Specimen Pre-tilt = 45°; set the dwell time to ≥5 µs to allow dynamic focus to work properly.

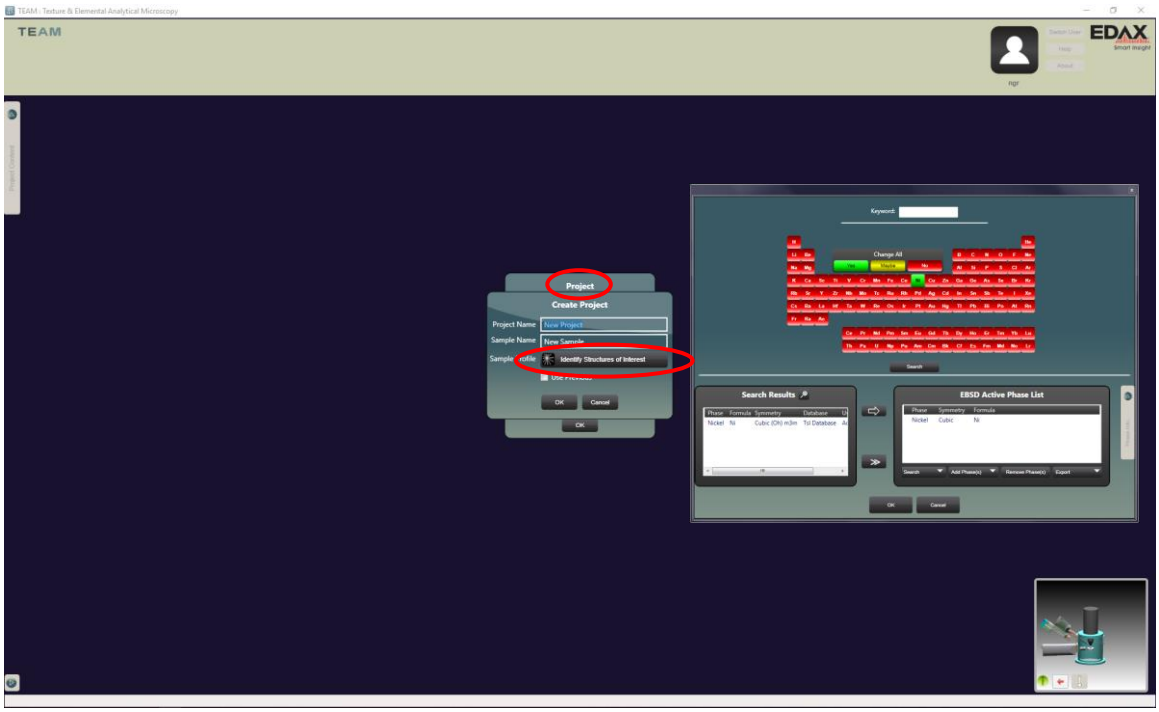


5. Starting TEAM and setting up your project file

5.1. Open the TEAM software, select the “EBSD” option at the log in screen, and log in to your account.

5.2. In the “Project” dialogue box, select the option to create a new project; in the “Create Project” dialogue box, you can then name the project and specimen.

5.3. Select “Identify Structures of Interest” and use the period table to add candidate structures to be used for the analysis; you must add at least one candidate structure to perform EBSD and it is best to keep the number of candidate structures to as small as possible (i.e., if you know your material is only one phase, use only that phase for a 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.

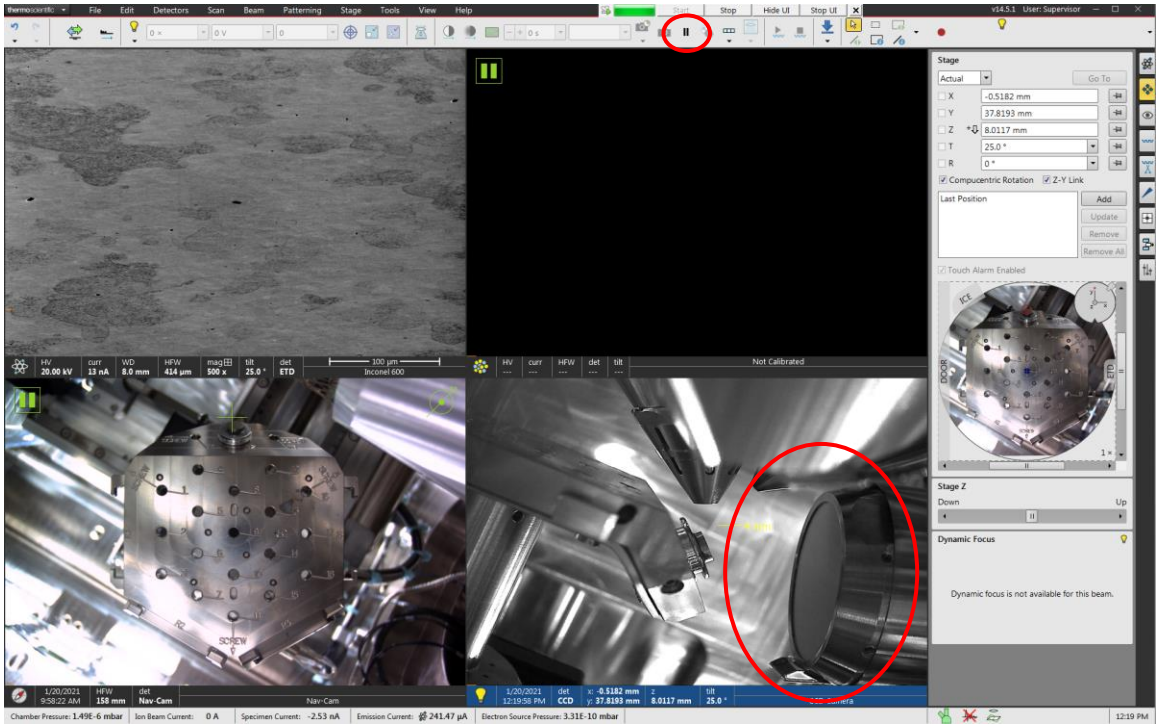
6.1.1. Insertion of the camera under any other conditions may cause a collision of the camera with the specimen or stage. *There is no interlock preventing insertion of the camera under inappropriate conditions.*

6.2. Expand the “Advanced Properties” tab (right side of the window) and select “Camera Position”; select “Insert” to insert the camera (you should hear the motor start).

6.2.1. Monitor the insertion process in the “CCD Camera” quad (in Microscope Control) and be prepared to select “Stop” if any collision appears imminent.

6.3. In Microscope Control, the CCD Camera quad should look similar to as shown below once camera insertion is completed; select the CCD Camera quad and then “Pause” from the main toolbar to turn off the CCD Camera.

6.3.1. This is necessary for optimal EBSD camera performance; the signal from the CCD camera will otherwise obscure the EBSD signal.



7. EBSD camera settings

7.1. In Microscope Control, set the magnification as appropriate for performing EBSD.

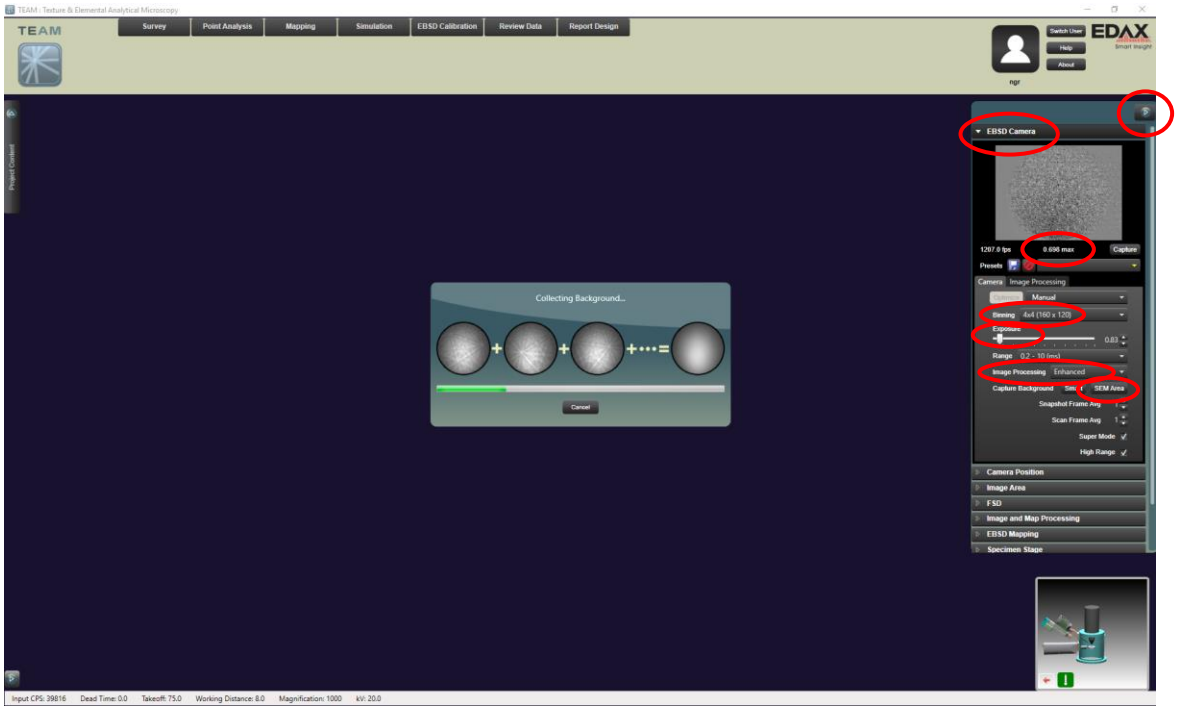
7.2. In TEAM, expand the “Advanced Properties” tab and select “EBSD Camera”.

7.2.1. Set “Binning” to 4×4 (appropriate for most mapping applications).

7.2.2. Set “Image Processing” to “Enhanced”.

7.2.3. Adjust “Exposure” so the maximum pattern intensity is ~0.5 (optimal value); shorter exposures will allow for faster camera speed, but potentially at the expense of poorer signal to noise.

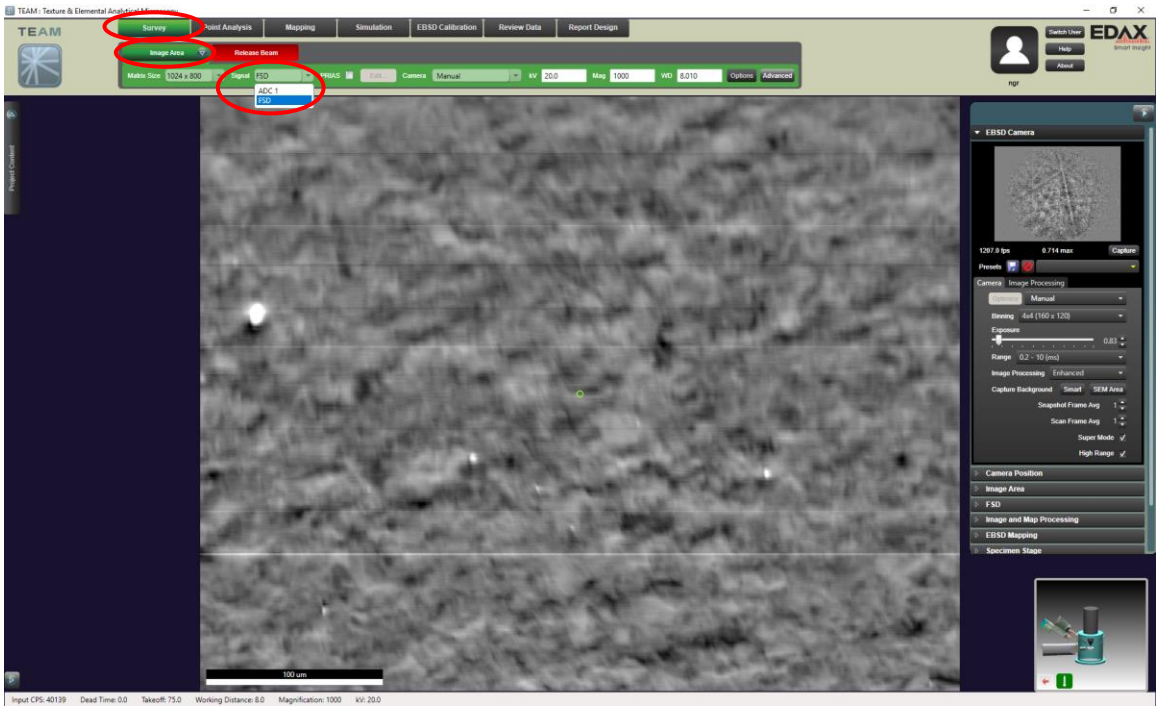
7.2.4. Next to “Capture Background”, select “SEM Area” to automatically capture the background.



8. Imaging and tuning the indexing

8.1. 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. Selecting the “ACD 1” signal (default) will use the ETD 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. When ready, select “Image Area” to collect an image.

8.1.1. 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.



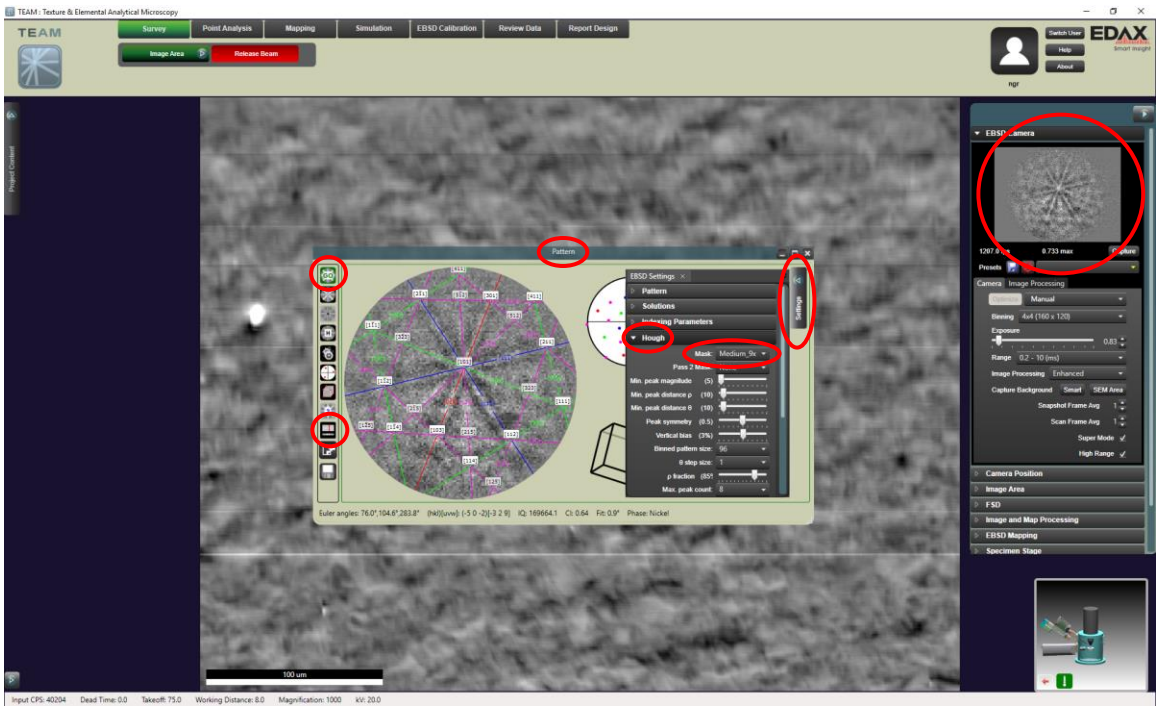
8.2. A small green circle will appear in the center of the acquired image, which corresponds to the position of the beam. If you click, hold, and drag on the image, you can move the beam position (and watch the EBSD pattern change accordingly as the beam is moved). Once you find a position that produces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

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

8.2.2. 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_11×11
15	Medium_9×9
20	Medium_9×9
25	SmallMedium_7×7
30	Small_5×5

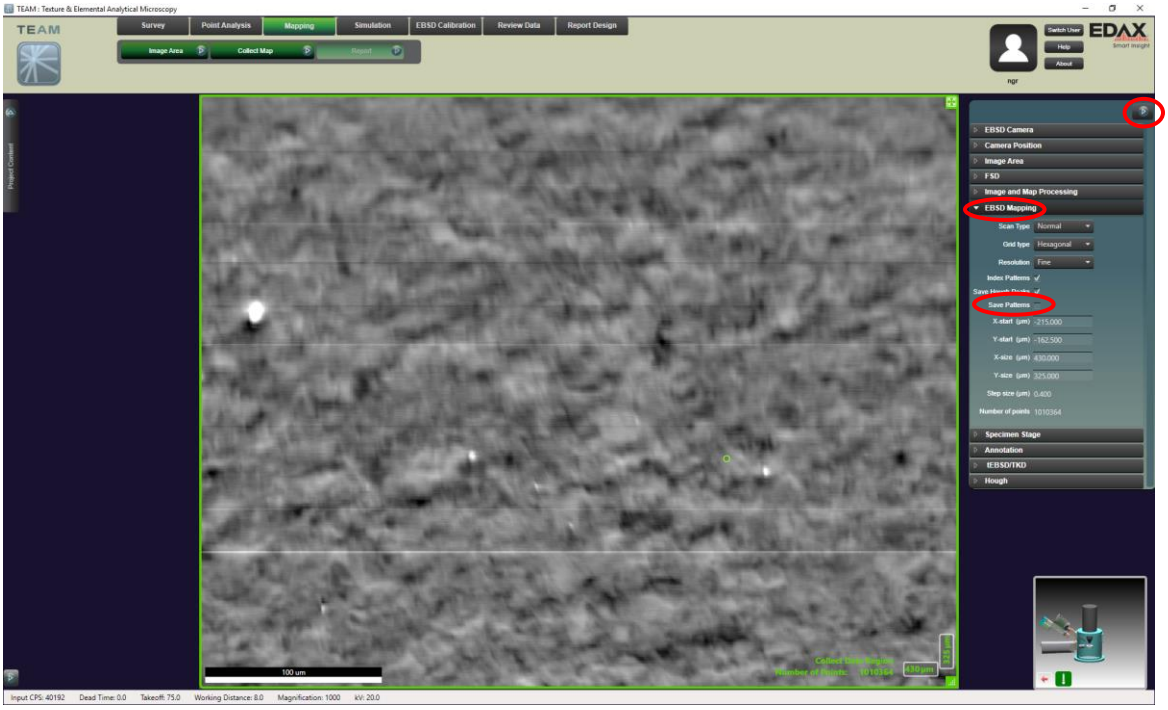
8.2.3. In the “Pattern” window, select “Go” to re-index the pattern and then “tune” to tune the solution. If the solution does not coincide well with the actual bands in the pattern, there may be a problem with the phase list, indexing parameters, or pattern quality.



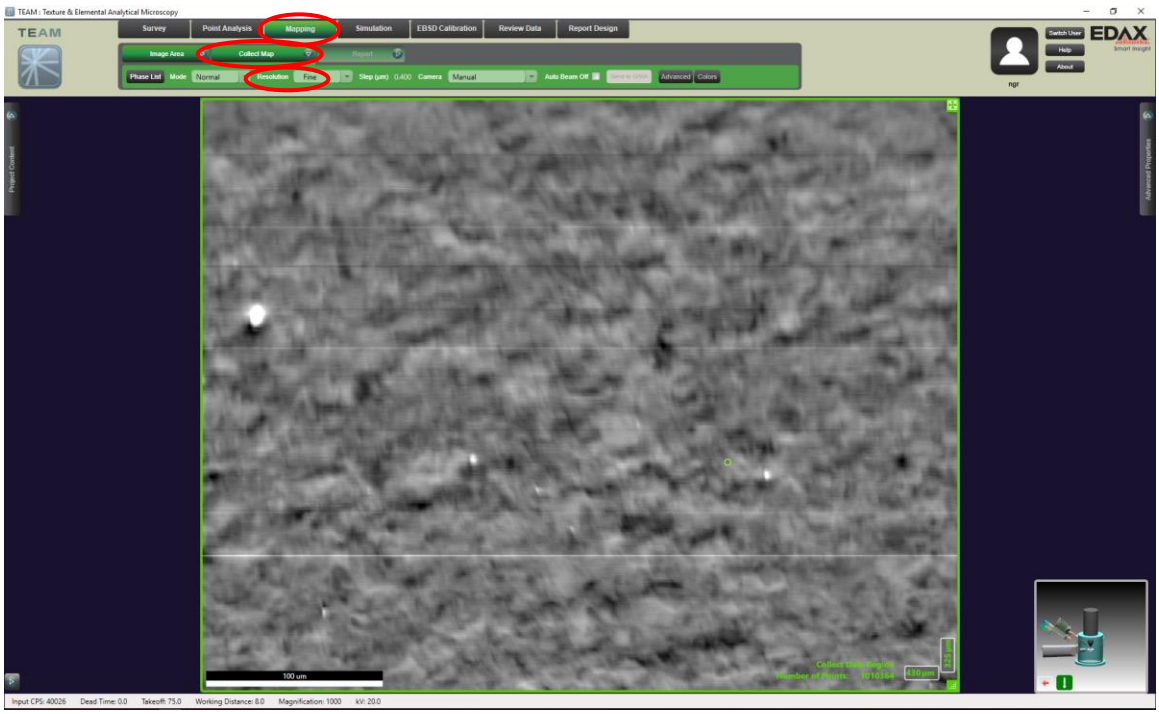
9. EBSD Mapping

9.1. 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.

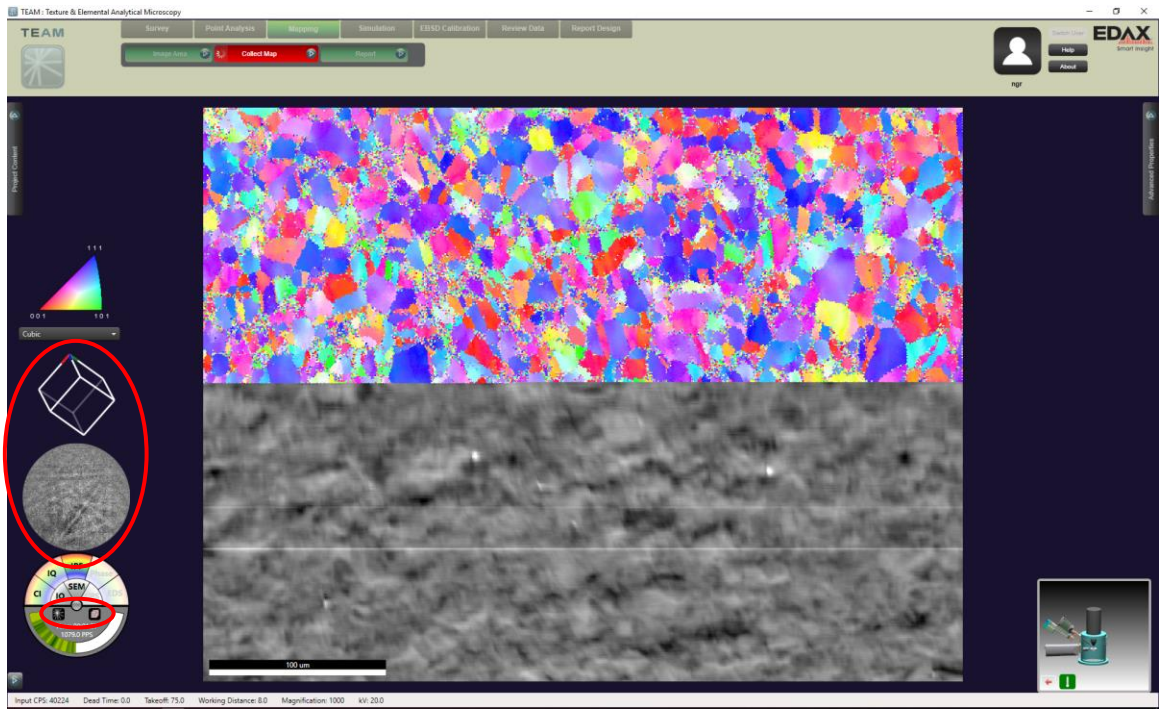
9.2. If desired, the individual patterns acquired during EBSD mapping can be collected and saved; this can be useful to improve data quality during subsequent data processing. Expand the “Advance Properties” tab, select “EBSD Mapping”, and check “Save Patterns”.



9.3. Hover over the “Collect Map” button and select the “Resolution” pull-down menu to see options for map resolution. As the resolution increases, more points will be mapped (as indicated in the lower right corner of the green defining box). Once a map resolution is selected, select “Collect Map” to start mapping.

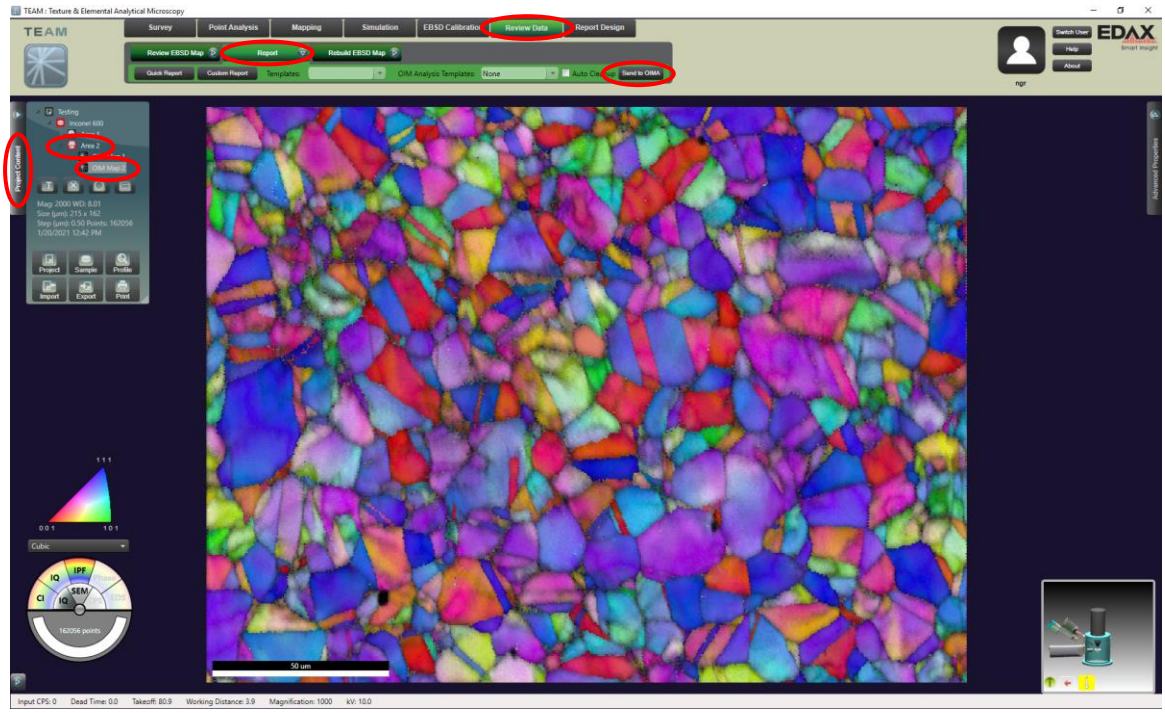


9.4. A live crystal orientation (or inverse pole figure) map showing the specimen normal direction overlaid on a pattern quality map will be generated. To view the live EBSD pattern and indexed unit cell orientation, select the EBSD pattern and unit cell icons from the compass (lower left side of window),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



9.5. When the map is finished, a “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.

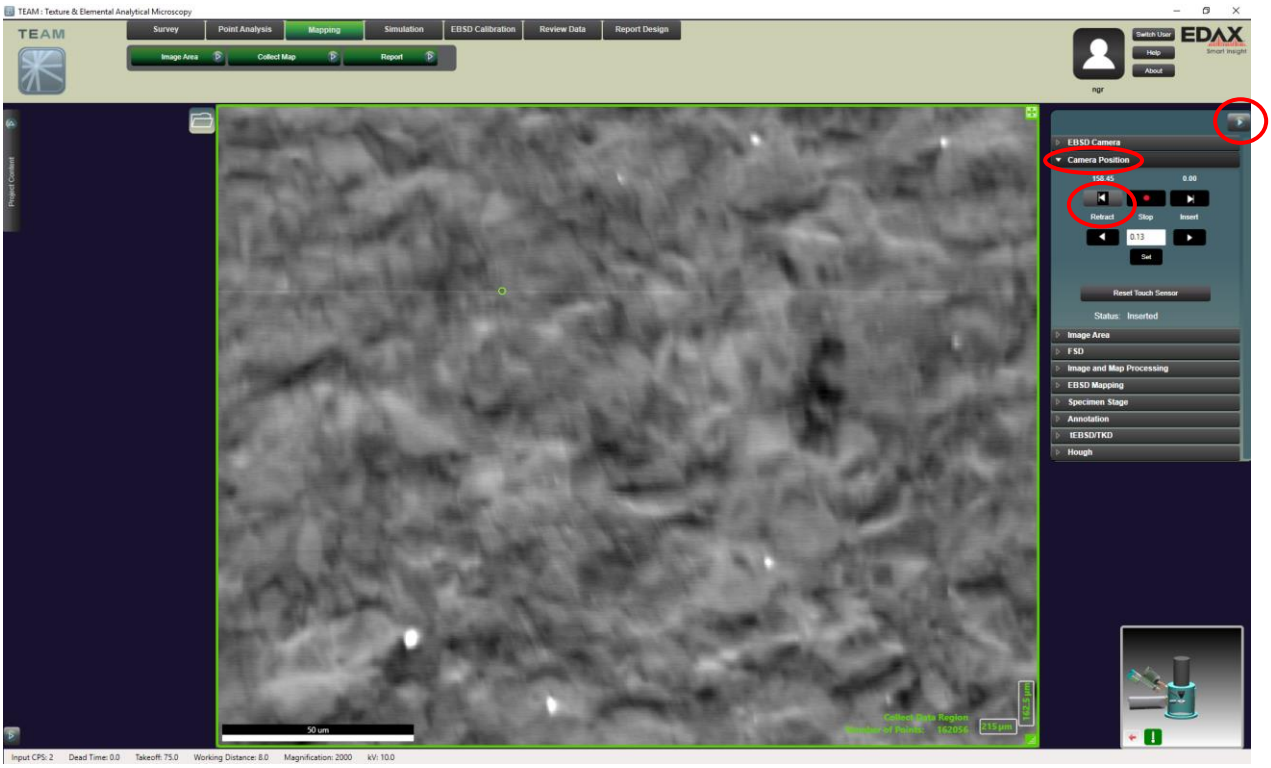
9.6. Further processing, analysis, and saving of map data should be done using OIM Analysis. Expand the “Project Content” tab, then select the area of interest and the map of interest; then select “Review Data” from the top menu bar; hover over “Report” and select “Send to OIMA”.



10. Finishing the session

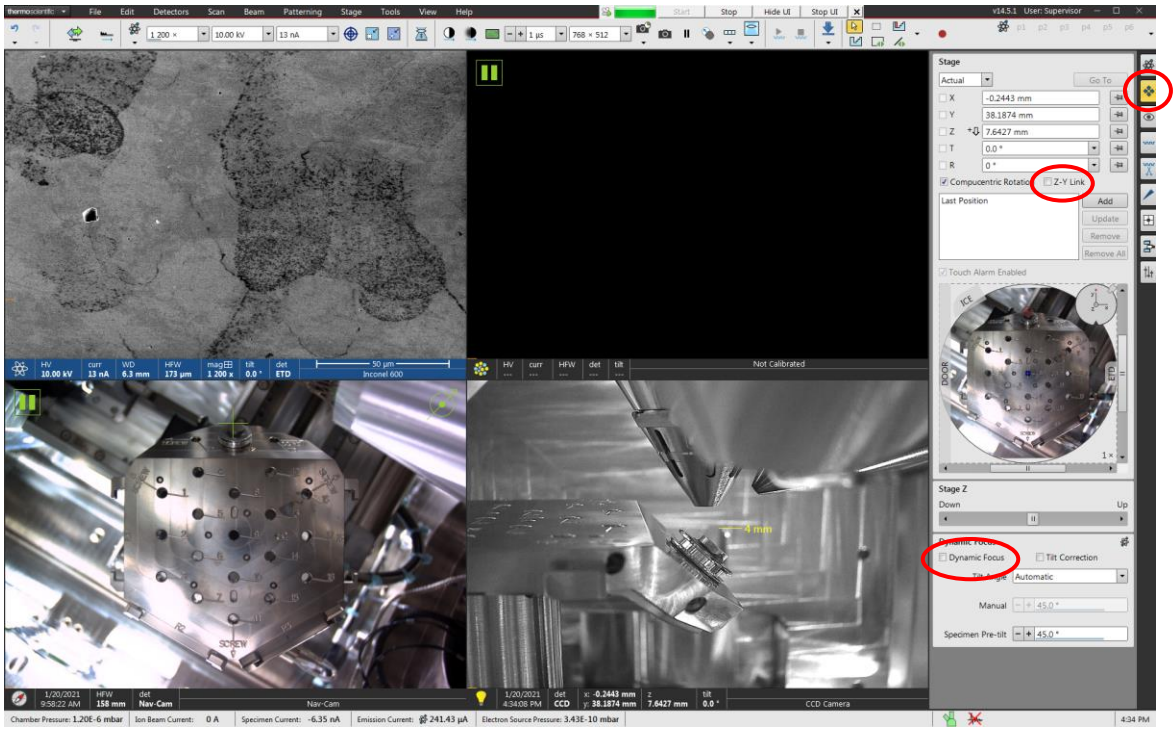
10.1. In Microscope Control, select the CCD camera quad and un-pause the image.

10.2. In TEAM, expand the “Advanced Properties” tab, select “Camera Position” and then “Retract” to retract the camera (you should hear the motor start).



10.3. 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 retracted.

10.4. Enter the Navigation module and uncheck “Z-Y Link” and “Dynamic Focus”.



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