FEI Helios G4 PFIB CXe: EBSD using EDAX APEX

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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.

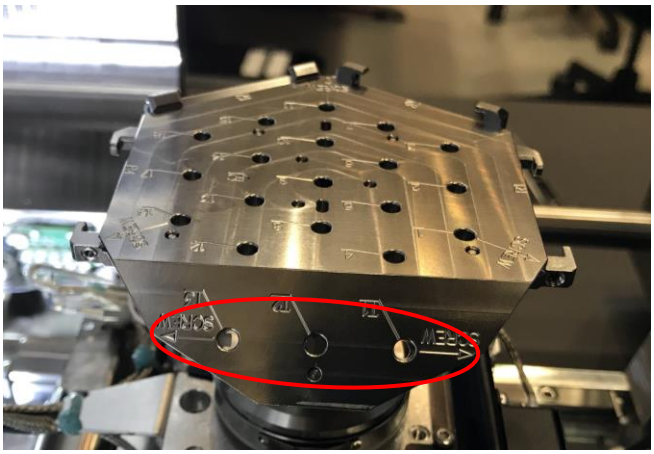
1. Sample mounting, preparation, and constraints

    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 and then using conductive tape or paint to ground the surface.

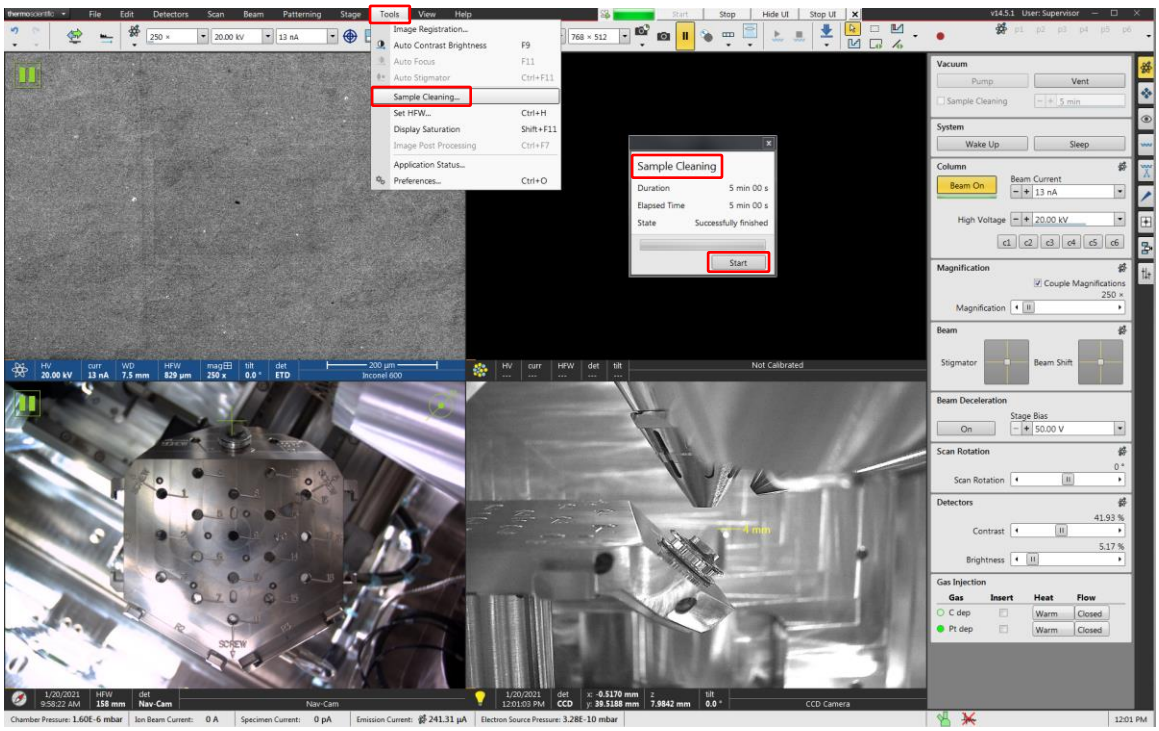
2. Specimen loading

    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. SEM settings

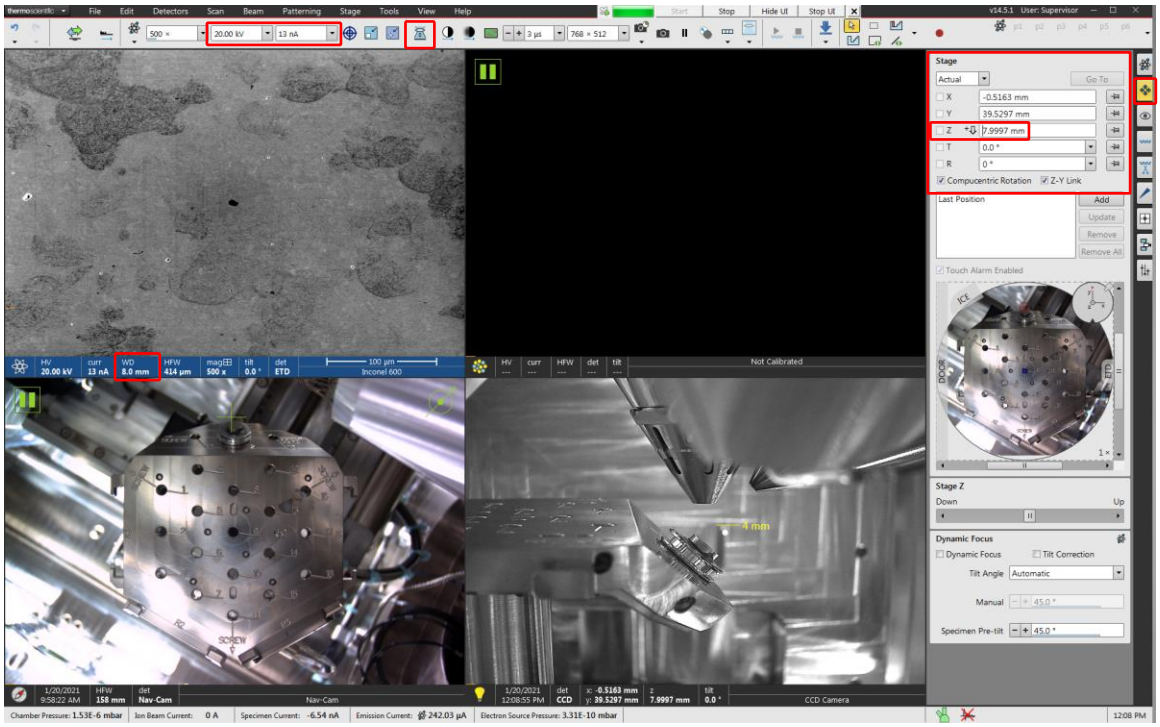
4.1. EBSD may be performed at voltages of 10 – 30 kV with up to 100 nA of current. In principle, the spatial resolution of EBSD improves as the beam voltage decreases, but this also results in noisier EBSD patterns; additionally, decreasing beam current will also improve spatial resolution via reduced probe size, but also at the expense of noisier EBSD patterns (or longer mapping time due to increased exposure time).

4.1.1. Voltage = 20 kV with current = 13 nA are recommend as general all-around settings 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 an area of interest the specimen (ideally, in the middle of the sample away from any edges), bring it to WD = 8 (WD for optimal EBSD camera performance), and select “Link Z = WD”.

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



4.3. Center a recognizable feature in the SEM image, enter the Navigation module, and navigate to the “Stage” panel.

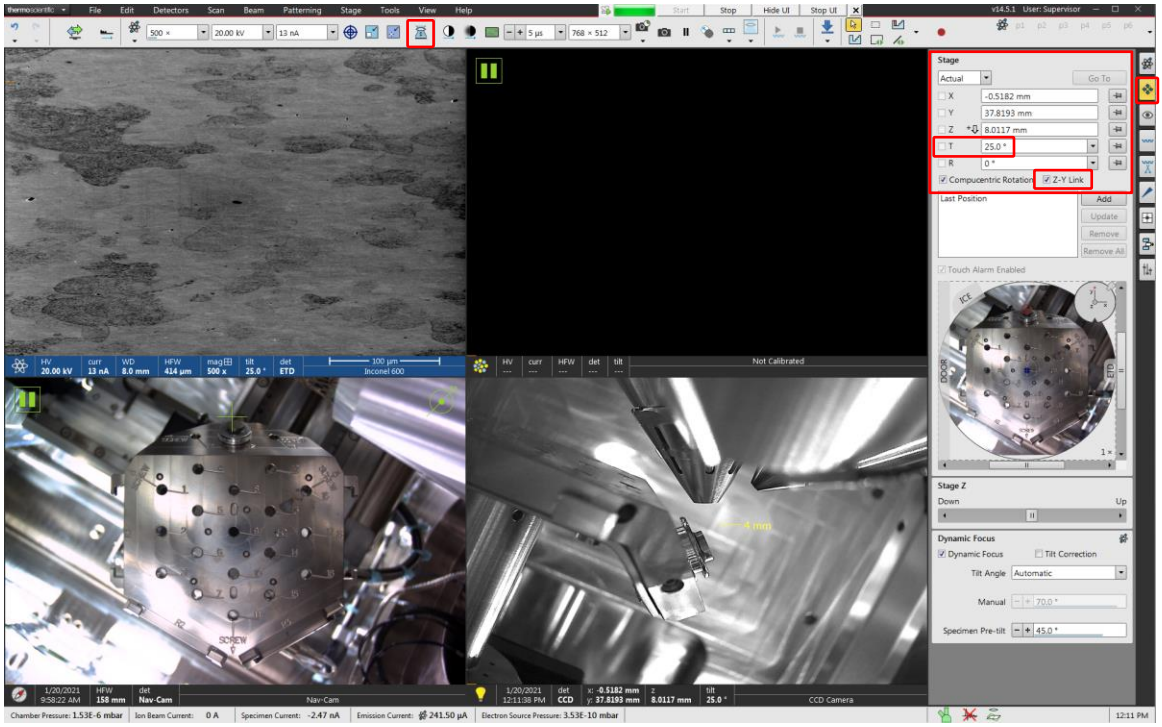
4.3.1. Check “Z-Y Link”; this ensures the area of interest remains centered when tilting at the specified WD.

4.3.2. Tilt the stage in 5° increments until T = 25° is achieved (resulting in actual specimen tilt = 70°); after each increment, center and focus the feature again.

4.3.3. NOTE: <u>if it appears that something will contact the SEM pole piece while tilting, STOP and contact RSC staff immediately</u> for help determining the maximum possible safe specimen tilt.

4.3.4. While not ideal, EBSD may still be effectively performed down to minimum specimen tilt = 60°. <u>However, if this minimum specimen tilt cannot be safely obtained, the sample is not suitable for EBSD</u>.

4.3.5. After the final specimen tilt is obtained, update “Link Z with WD” and set Z = 8 mm; then, center and focus the feature one more time.



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” = “Automatic”.

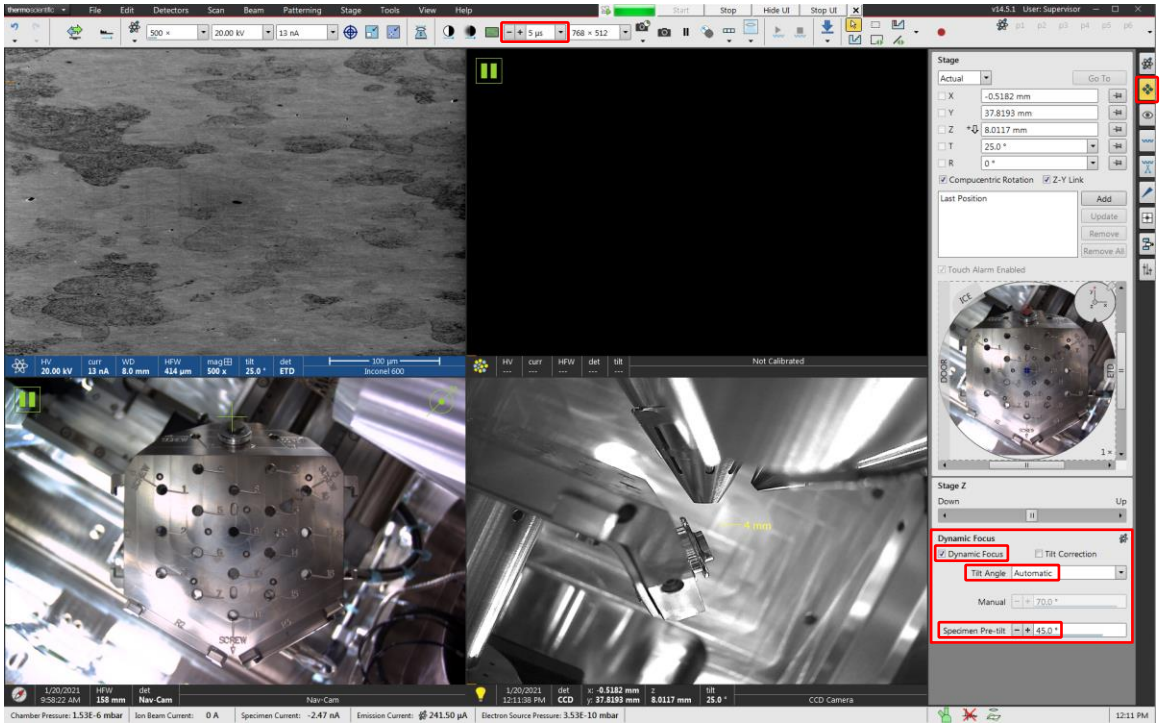
4.4.3. Set “Specimen Pre-tilt” = 45°.

4.4.4. DO NOT activate “Tilt Correction” here; tilt correction will be applied later using APEX.

4.4.5. Focus precisely on the middle of the image (use of the reduced scanning area may help here).

4.4.6. Adjust the magnification as appropriate for EBSD mapping of the sample.

4.4.7. Set the dwell time ≥ 5 µs for dynamic focus to work properly.



5. Starting APEX

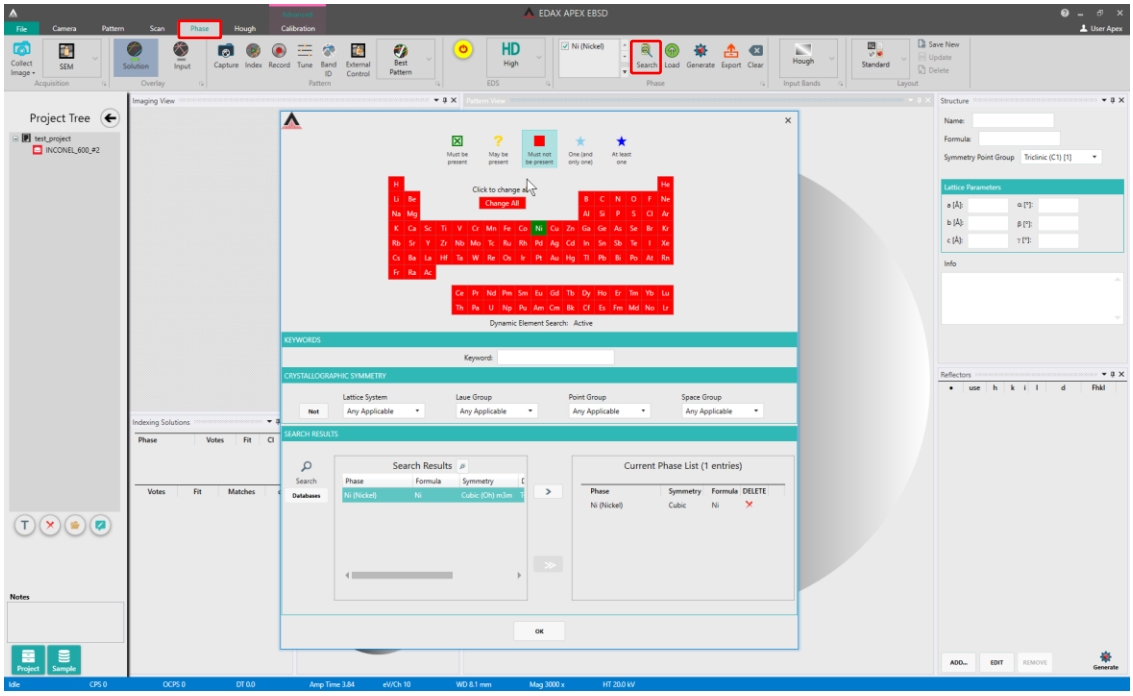
5.1. Open the APEX EBSD software (no login needed); a dialogue box asking to create a project and sample name (or to load a previously created project) will pop up.

5.2. Select the “Phase” tab; in the tool ribbon, select “Search” and use the periodic table to search for, select, and add the structure(s) to be used.

5.2.1. NOTE: if a structure is not found in the database, it may be added manually; please contact RSC staff for assistance.

5.3. At least one candidate structure must be added, but <u>it is best to keep the number of candidate structures as small as possible.</u>

5.3.1. I.e., if the material is known to be only one phase, use <u>only</u> that phase (and no others) as the candidate structure.



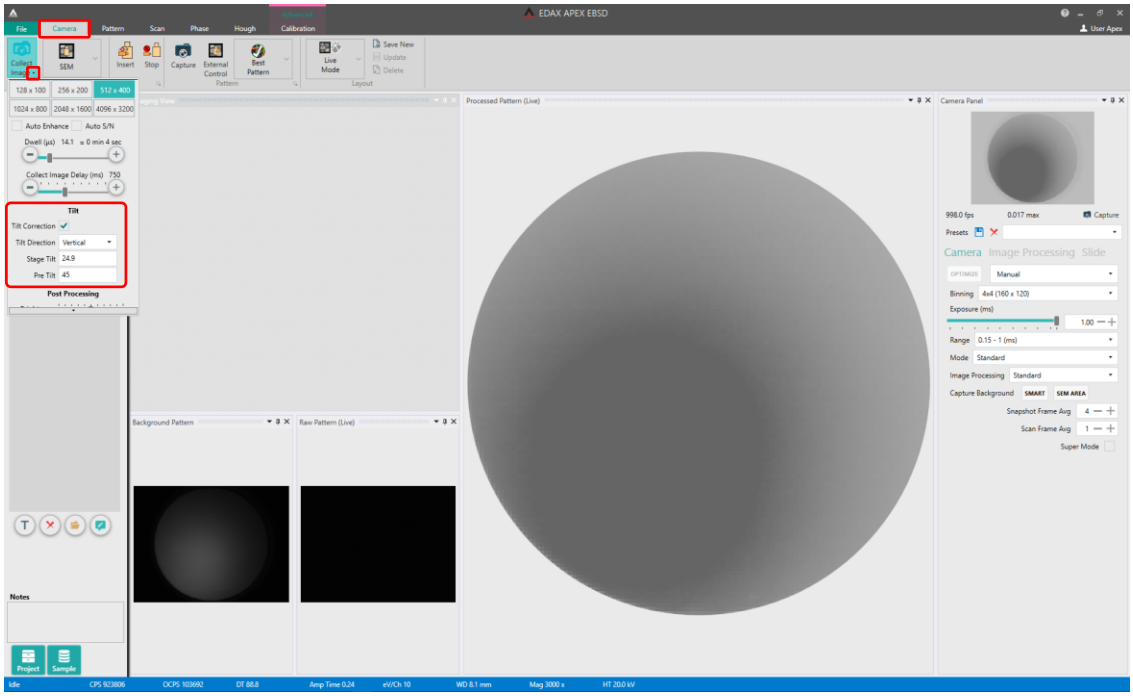
6. Inserting the EBSD camera

6.1. Select the “Camera” tab; in the tool ribbon, select the downward pointing arrow in “Collect Image” to show the image acquisition parameters.

6.1.1. Verify “Tilt Correction” is checked.

6.1.2. Verify “Stage Tilt” = 25° and “Pre-Tilt” = 45° to give a total specimen tilt = 70°.

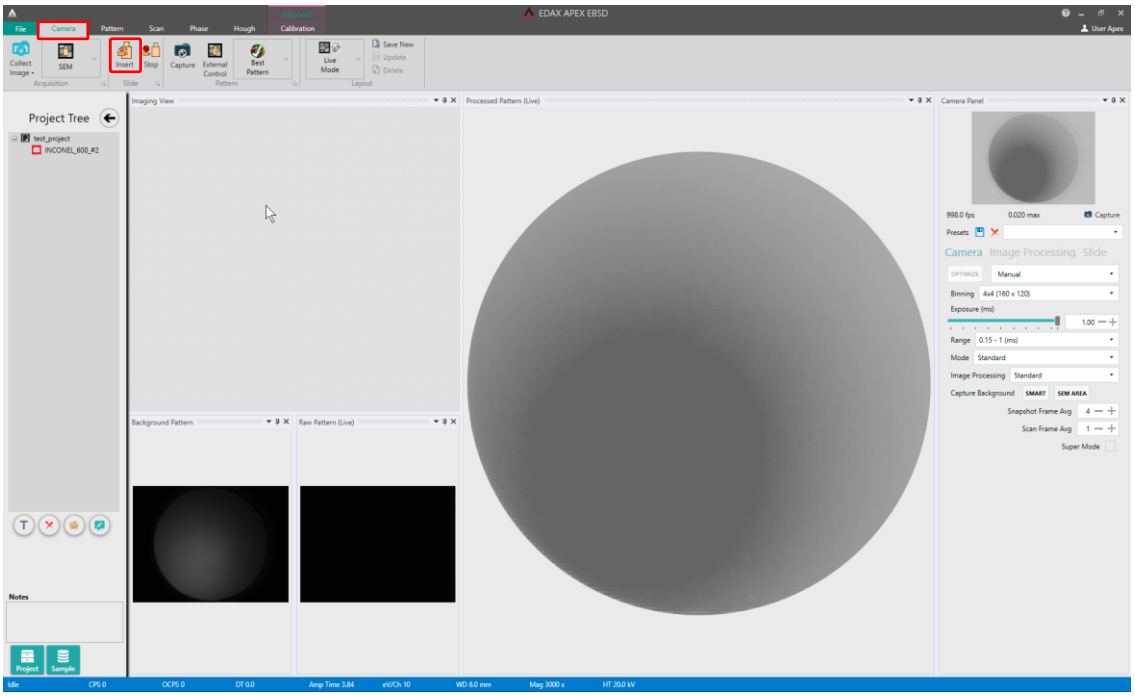
6.1.3. NOTE: specimen tilt = 70° is preferred, but EBSD may still be performed down to a minimum specimen tilt = 60°; the important point is that stage tilt + pre-tilt = actual total specimen tilt.



6.2. Remain in the “Camera” tab; in the tool ribbon, select “Insert” to insert the EBSD camera (listen for the slide motor).

6.2.1. In Microscope Control, monitor the insertion process in the “CCD Camera” quad; in APEX in the tool ribbon, be prepared to select “Stop” if any collision appears imminent.

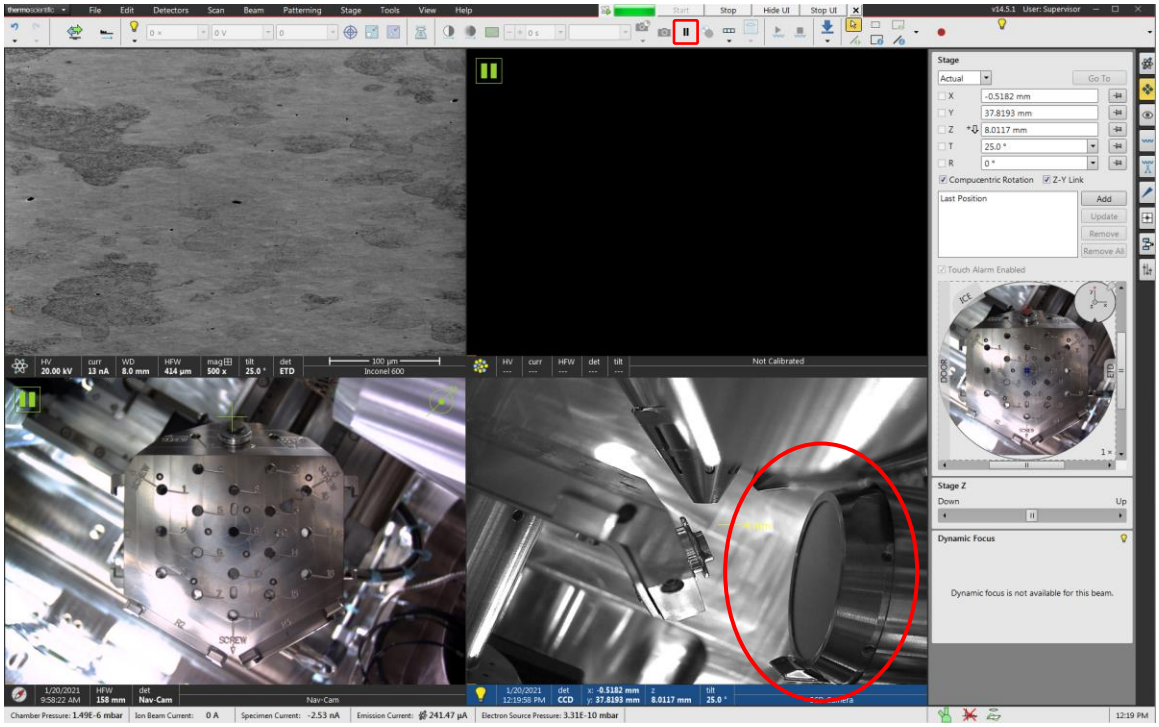
6.2.2. NOTE: APEX will give a clear warning if attempting to insert the camera with the total specimen tilt < 60°.



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

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

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

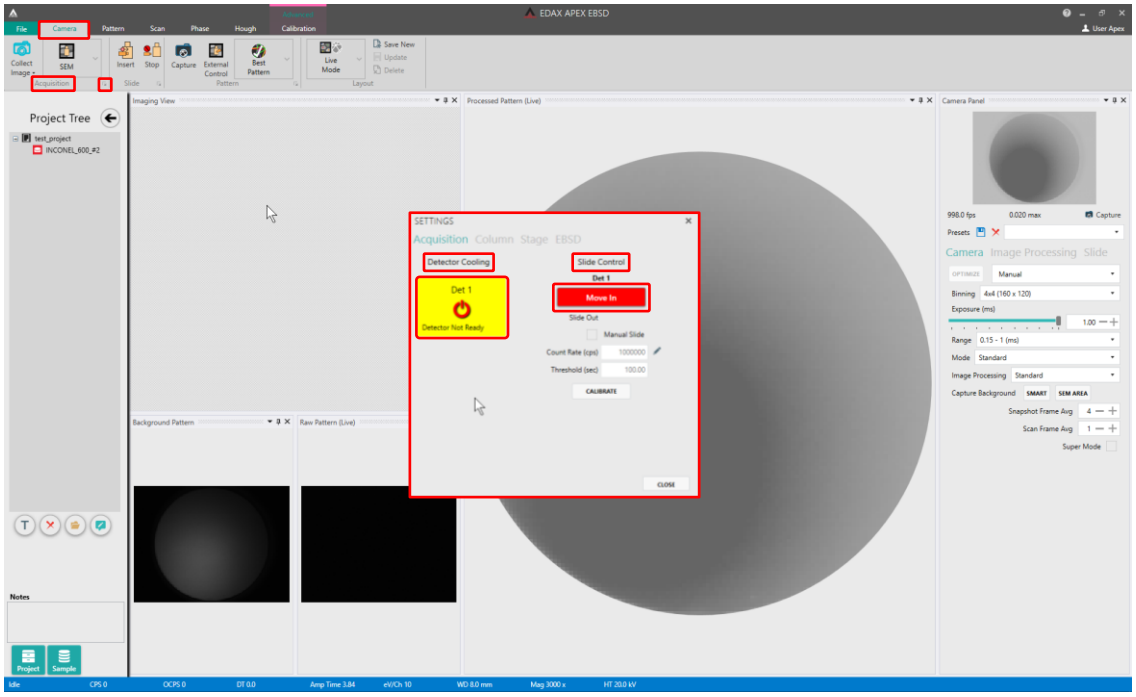


6.4. Simultaneous EDS data collection during EBSD mapping (if desired).

6.4.1. Remain in the “Camera” tab; in the tool ribbon, select the arrow in the lower right corner of the “Acquisition” field to open the “Settings” window.

6.4.2. Under “Detector Cooling”, select “Det 1” to start cooling the EDS detector; cooling will take several minutes to complete.

6.4.3. Under “Slide Control”, select “Move In” to insert the EDS detector (listen for the slide motor).



7. ROI image collection

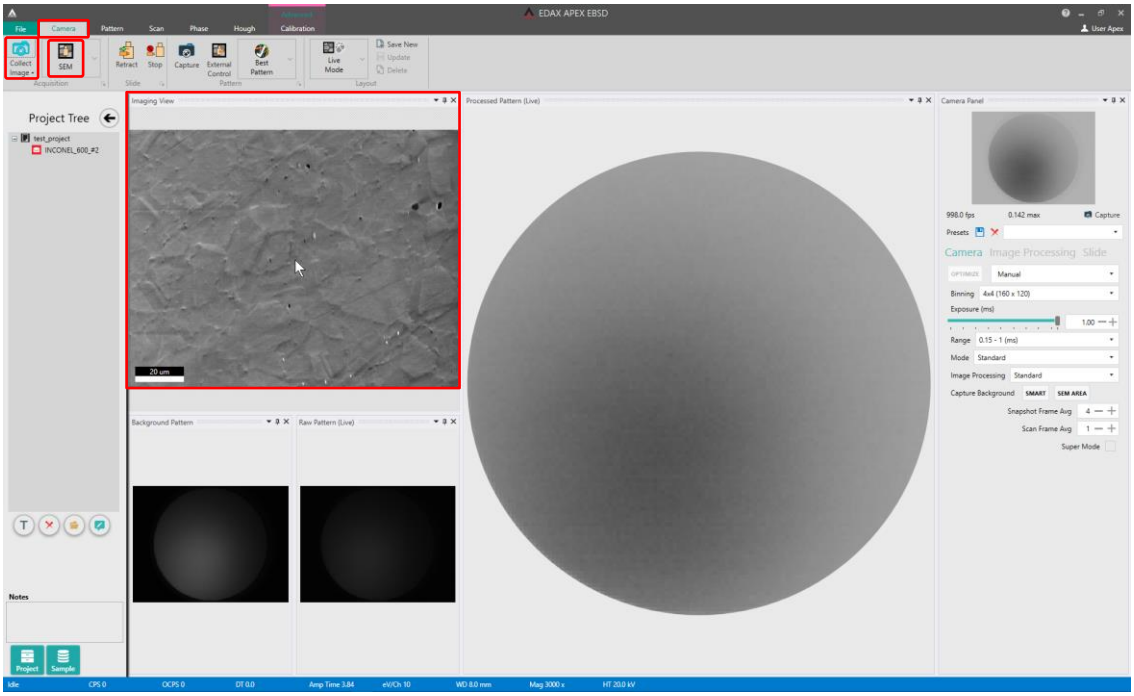
7.1. In Microscope Control, verify the magnification is set as appropriate for performing EBSD mapping and that the image is well-focused.

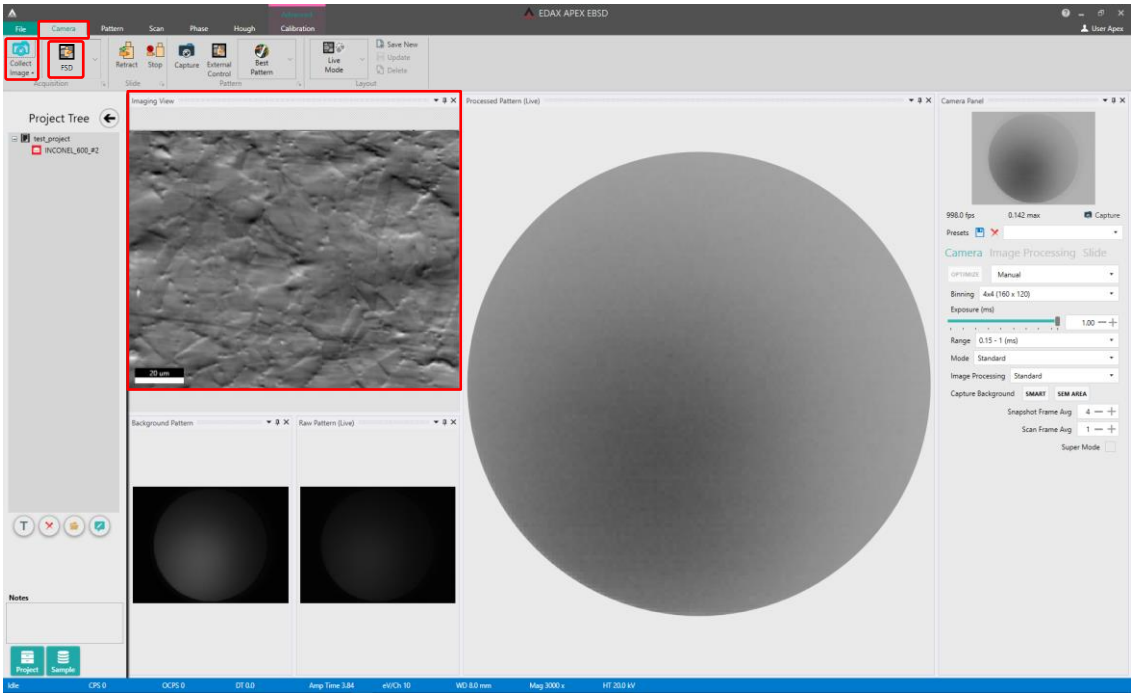
7.2. Remain in the “Camera” tab; in the tool ribbon, select “SEM” to use the same signal used for the SEM image in Microscope Control to collect the ROI image.

7.3. Alternatively, “FSD” may be selected in the tool ribbon to use the forward scattered detector near the bottom of the EBSD camera to collect the signal for the ROI image

7.3.1. NOTE: the choice of signal for the ROI image has no impact on the resulting EBSD map; however, use of the FSD tends to show microstructure more readily.

7.4. Select “Collect Image” to acquire the ROI image in the “Imaging View” panel.




EDAX APEX EBSD
File
Camera
Pattern
Scan
Phase
Hough
Calibration
Collect Image
FSD
Retract
Stop
Capture
External Control
Best Pattern
Live Mode
Save New
Update
Delete
Project Tree
test_project
INCONEL_600_#2
Imaging View
20 um
Processed Pattern (Live)
Camera Panel
Background Pattern
Raw Pattern (Live)
Notes
Project
Sample
Camera
Image Processing
Slide
Binning
Exposure (ms)
Range
Mode
Image Processing
Capture Background
Snapshot Frame Avg
Scan Frame Avg
Super Mode

8. Camera settings; background collection

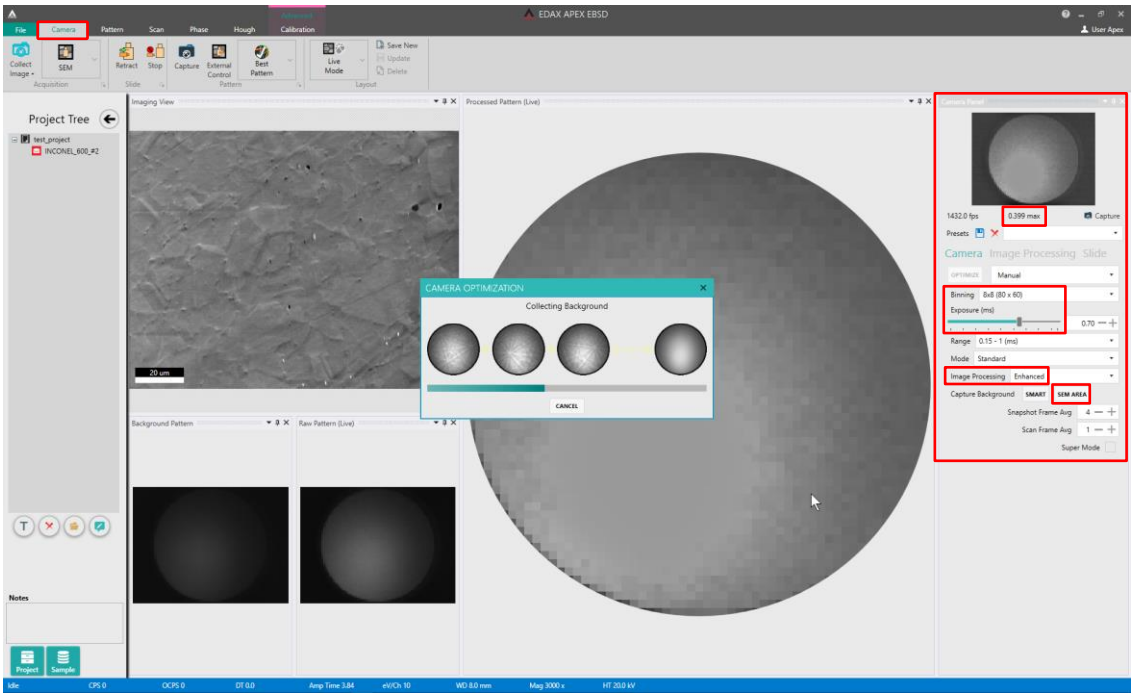
8.1. Remain in the “Camera” tab and navigate to the “Camera” panel; select the desired settings for the EBSD camera (binning, exposure, and image processing).

8.1.1. “Binning” = 8×8 is recommended for EBSD mapping; this results in a good all-around compromise between pattern resolution, camera speed, and pattern intensity; additionally, if the patterns are saved, this will keep the dataset size more manageable.

8.1.2. Set “Image Processing” to “Enhanced” (effectively for most polycrystalline samples).

8.1.3. 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.

8.2. Next to “Capture Background”, select “SEM Area” to use the ROI to collect the background.



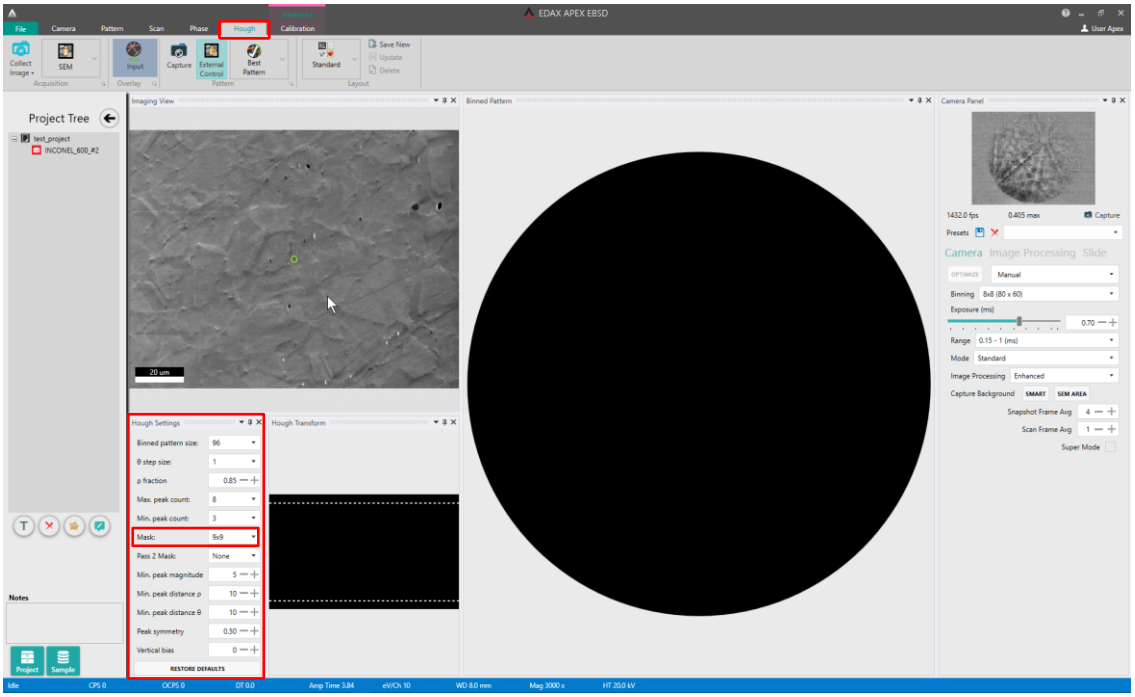
9. Indexing tuning

9.1. Before proceeding, return to Microscope Control and accurately focus the live SEM image; the focus may be noticeably off after background collection.

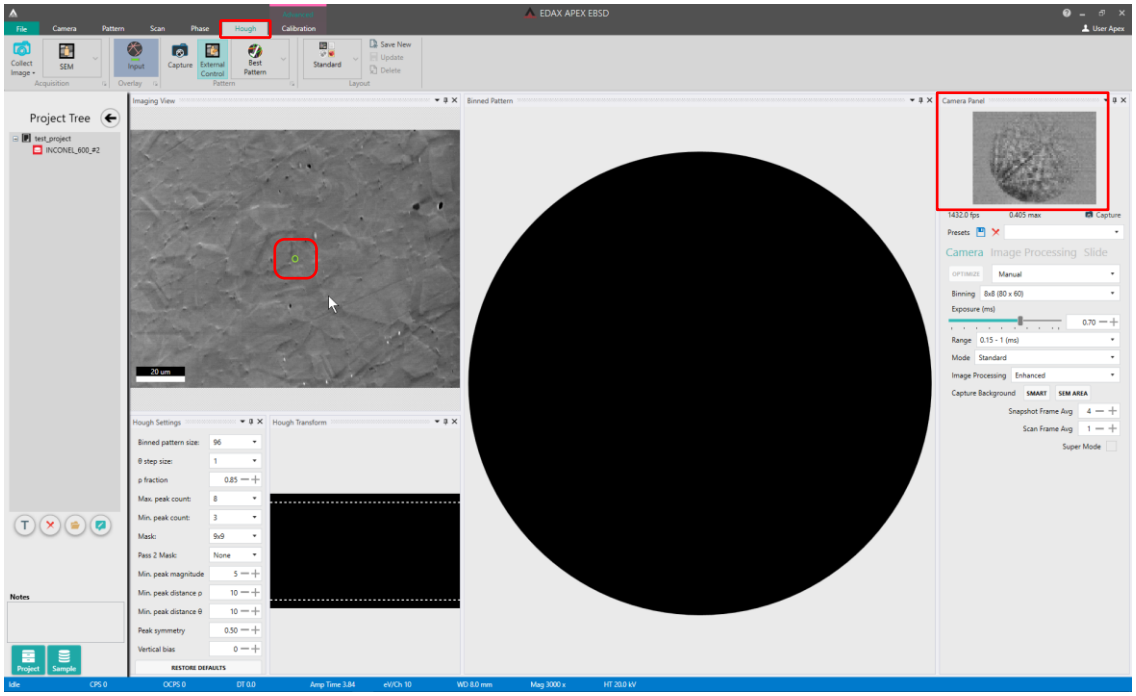
9.2. In APEX, select the “Hough” tab and navigate to the “Hough Settings” panel.

9.2.1. Choose the optimal “Mask” setting based on the beam voltage as indicated by the following table:

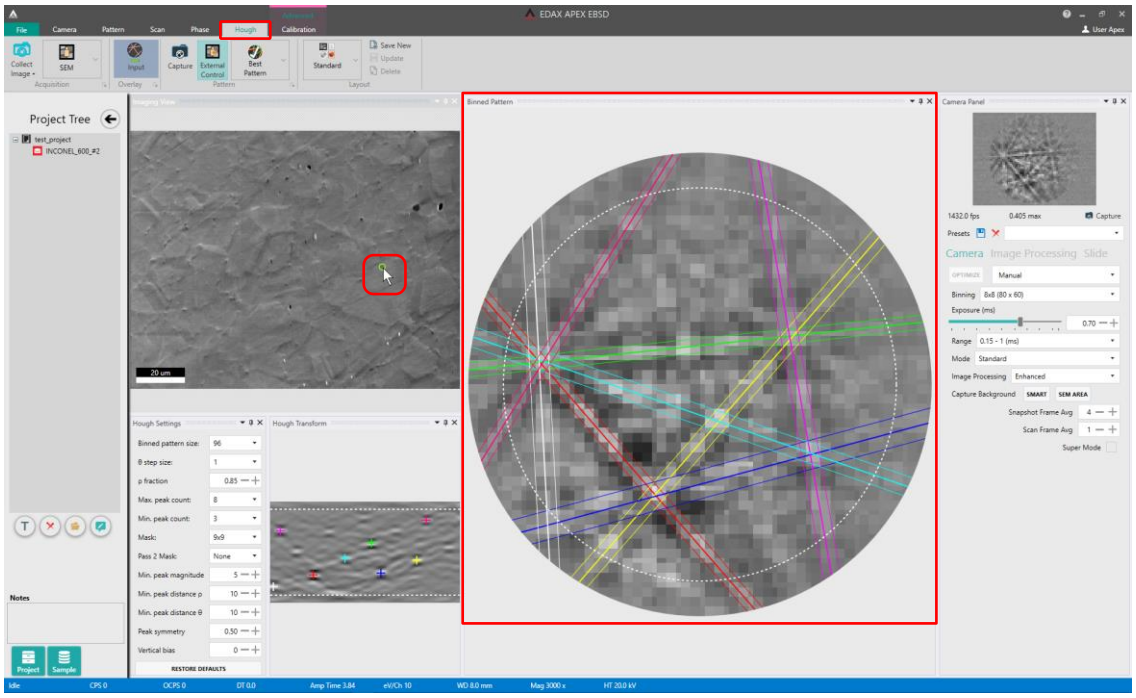
Beam voltage (kV)	Mask
10	MediumLarge_11×11
15	Medium_9×9
20	Medium_9×9
25	SmallMedium_7×7
30	Small_5×5



9.3. A beam position marker will appear in the center of the ROI image. Click, hold, and drag on the marker to move the beam position; watch the EBSD pattern in the “Camera” panel change as the beam is moved.



9.4. Once a suitable EBSD pattern (from only one grain) is found (ideally, near the center of the ROI image), release the mouse to record the pattern in the “Binned Pattern” panel.



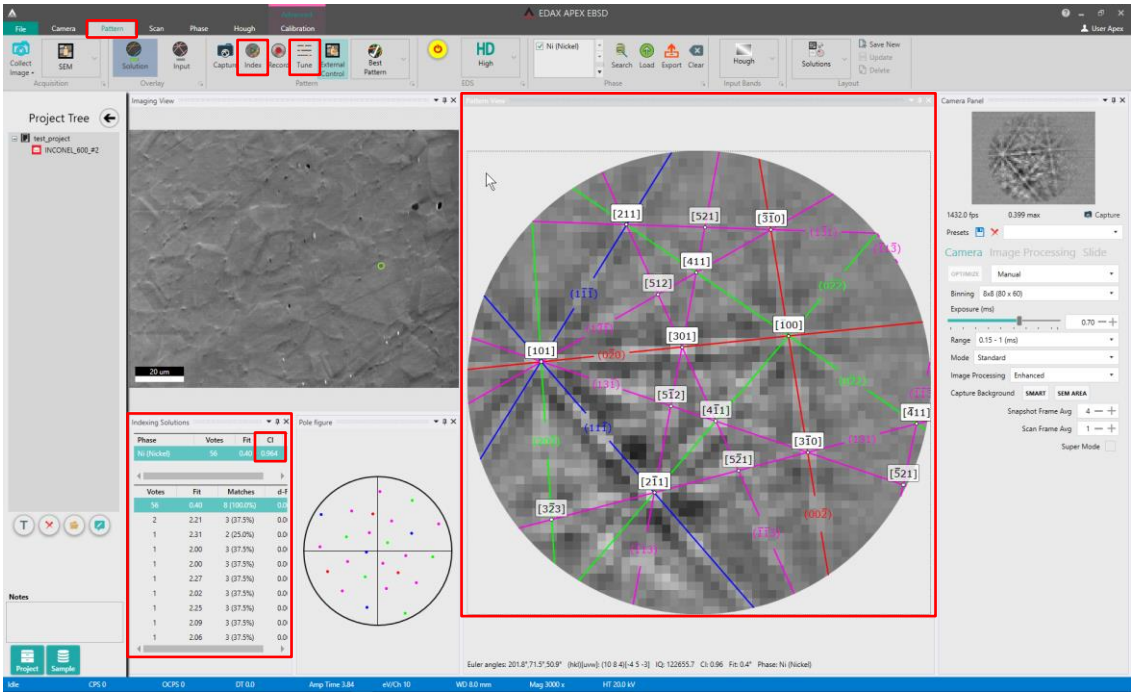
9.5. Select the “Pattern” tab; from the tool ribbon, select “Index” and then “Tune” to index the captured EBSD pattern and tune the indexing.

9.5.1. In the “Pattern View” panel, inspect and evaluate the solution overlayed on the pattern.

9.5.2. In the “Indexing Solutions” panel, verify that the “CI” (confidence index) value is satisfactory; CI may take values $0 \leq CI \leq 1.0$, but if $CI > 0.1$, the probability of the solution being correct is > 90%.

9.5.3. NOTE: if the pattern does not appear to index correctly, this may indicate a problem with the phase file(s) and/or the chosen indexing parameters.

9.5.4. NOTE: maps may be re-indexed during post-processing using OIM; if useful patterns are evident, mapping may still be performed even if the initial indexing does not appear to be accurate.



10. Collecting the EBSD map

10.1. Select the “Scan” tab; the ROI image will appear in the “Imaging View” panel.

10.1.1. If desired, adjust the size and position of the green defining box surrounding the ROI image to define the area to be mapped.

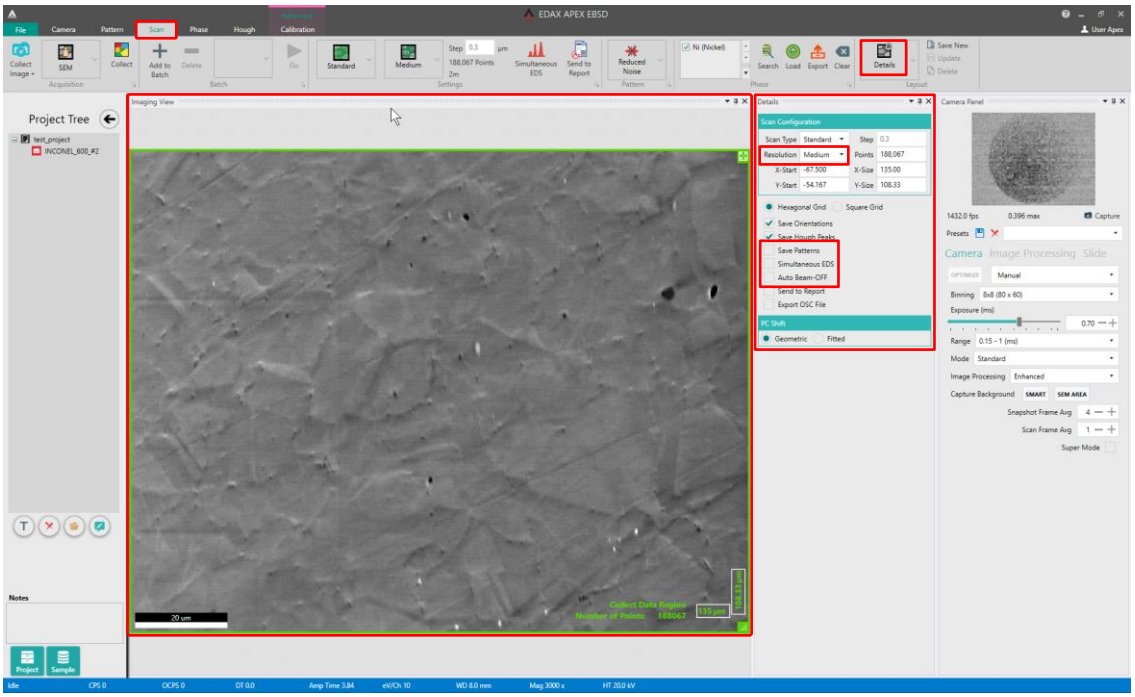
10.2. In the tool ribbon, make sure the layout is set to “Details” and navigate to the “Details” panel.

10.2.1. Set the “Resolution” (mapping step size) as desired.

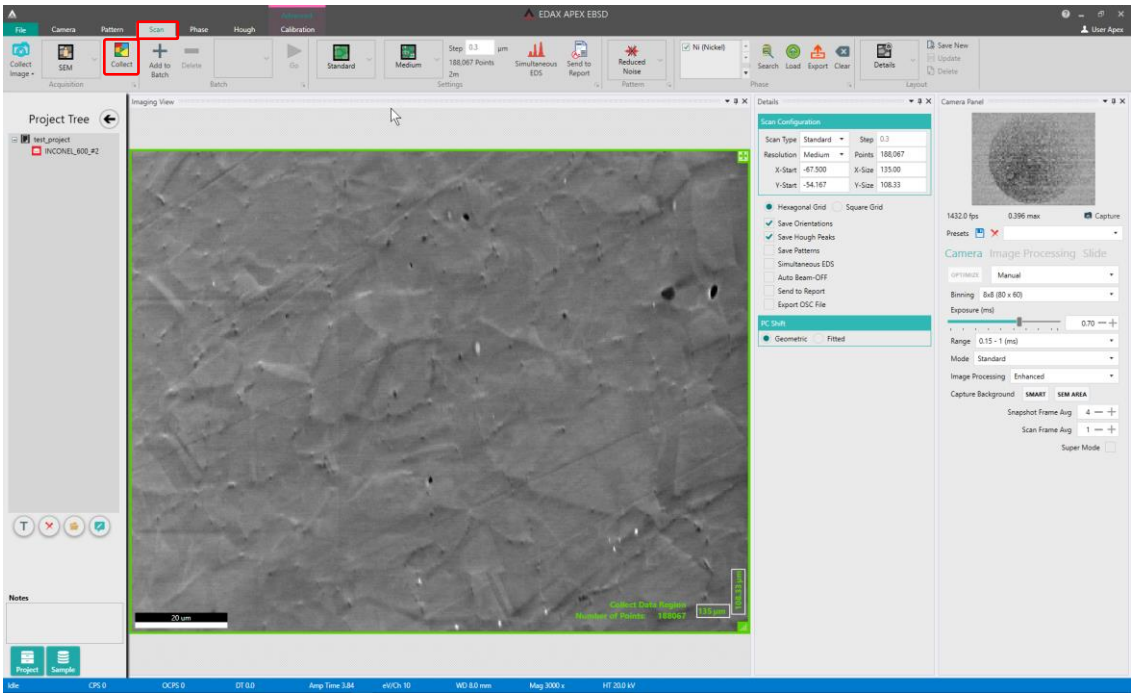
10.2.2. Check “Save Patterns” to save the patterns while mapping; this will dramatically increase the dataset size, so only do this if necessary and for camera binning = 8×8 (or higher).

10.2.3. Check “Simultaneous EDS” if collecting EDS data while also performing EBSD mapping.

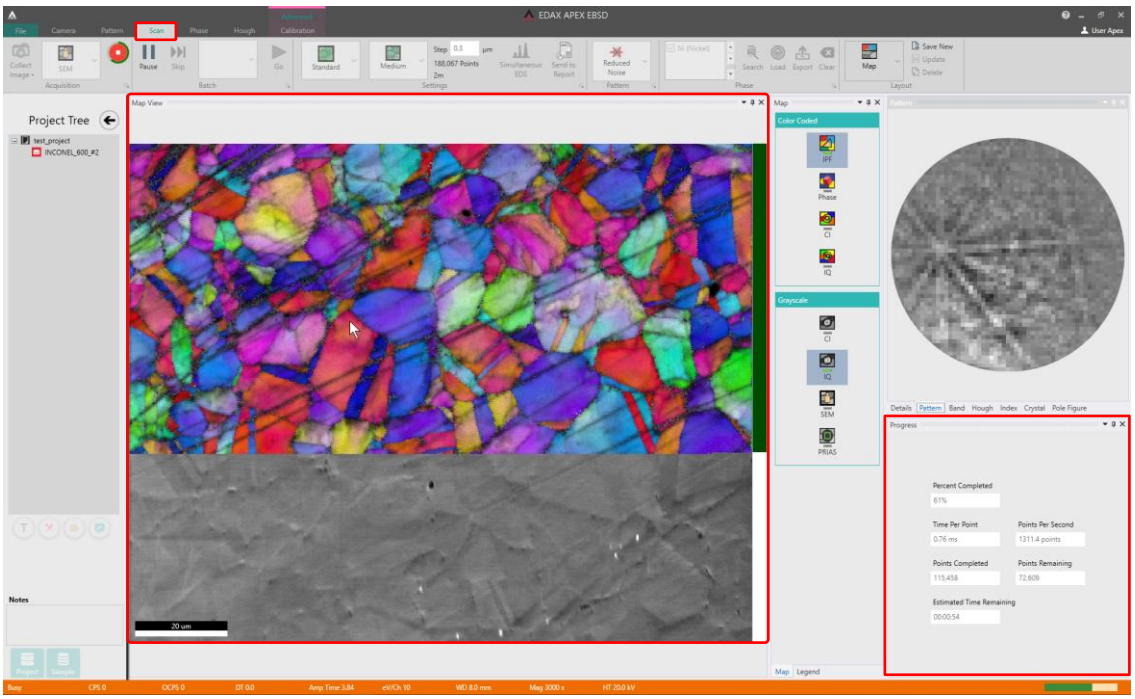
10.2.4. Check “Auto Beam-OFF” to automatically turn the SEM off when finished (recommended for scans taking several hours or longer).



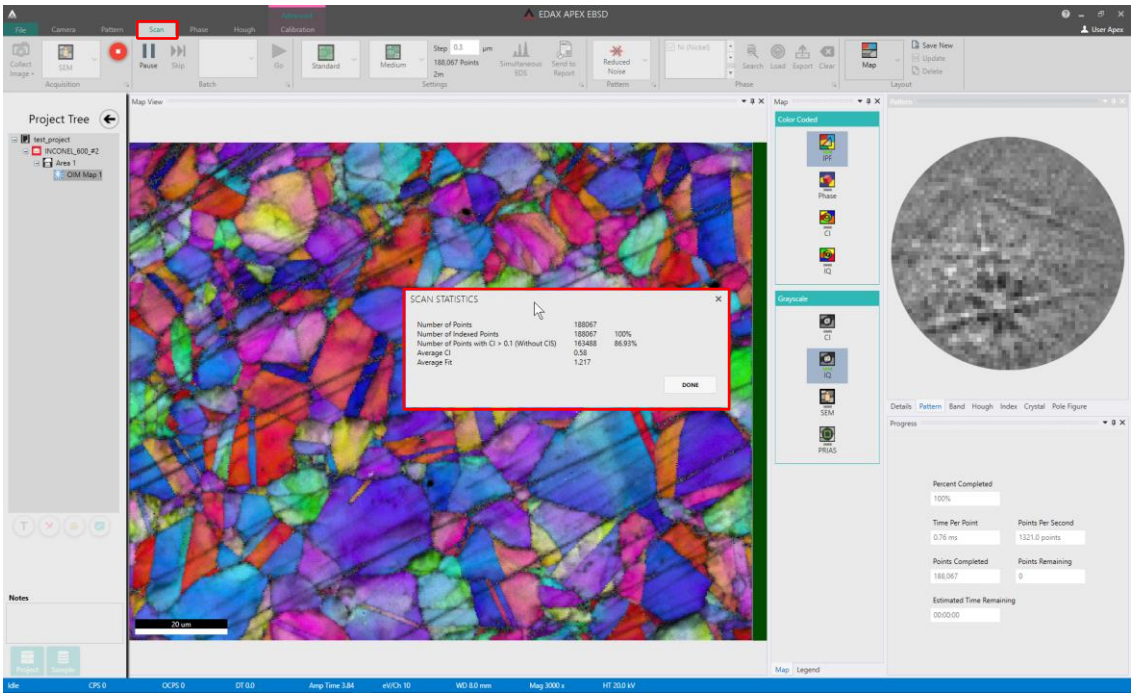
10.3. Remain in the “Scan” tab; in the tool ribbon, select “Collect” to start collecting the map.



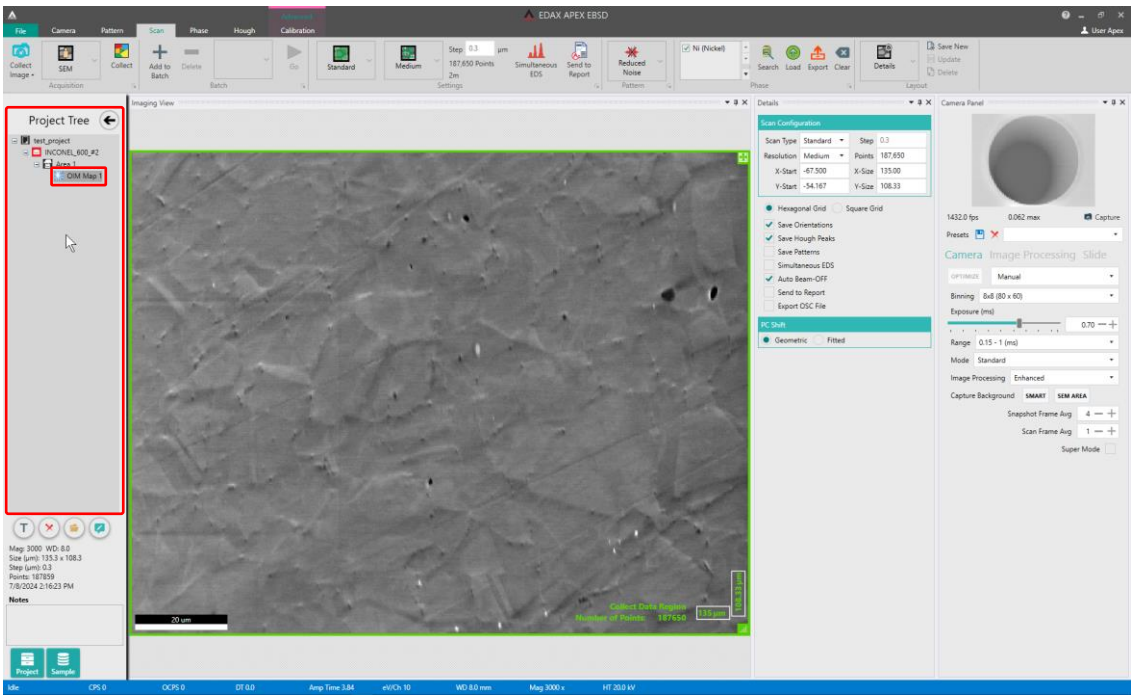
10.4. As the scan proceeds, an IPF map overlayed on an IQ (pattern quality) map will show in the “Map View” panel; additionally, the “Progress” panel shows the progress of the scan.



10.5. When the scan is complete, a “SCAN STATISTICS” window will pop up with the results of the scan.

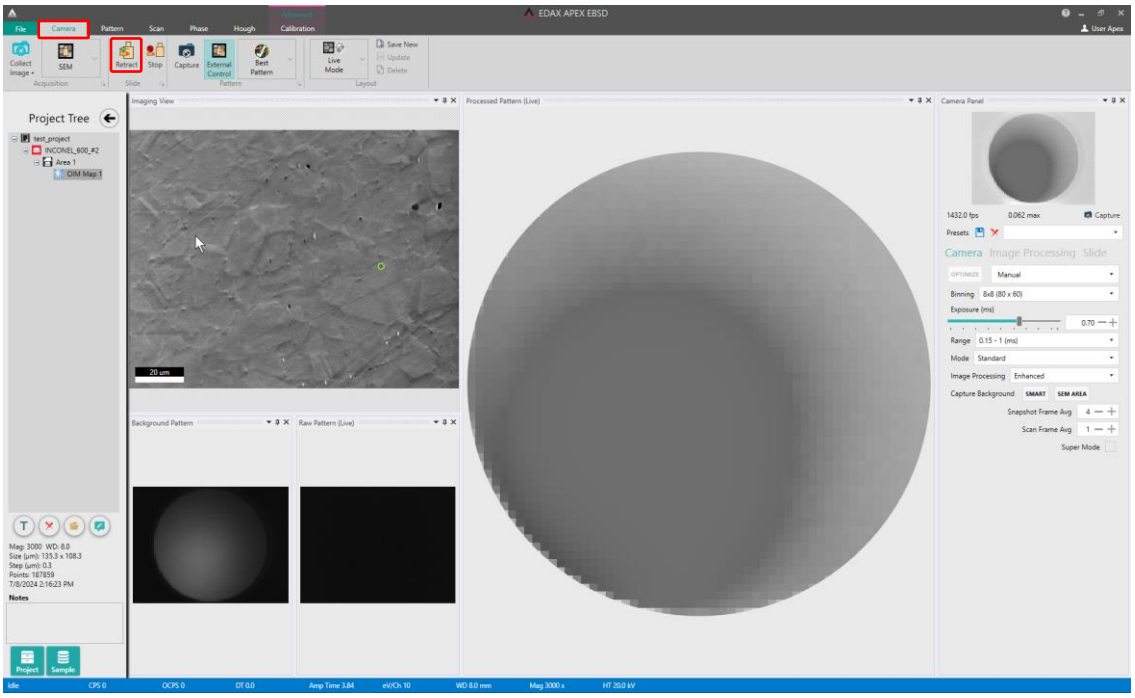


10.6. In the “Project Tree” panel, an “OIM Map” object will be generated for the map. To export the map to OIM for further processing/analysis, simply double left-click on the object.

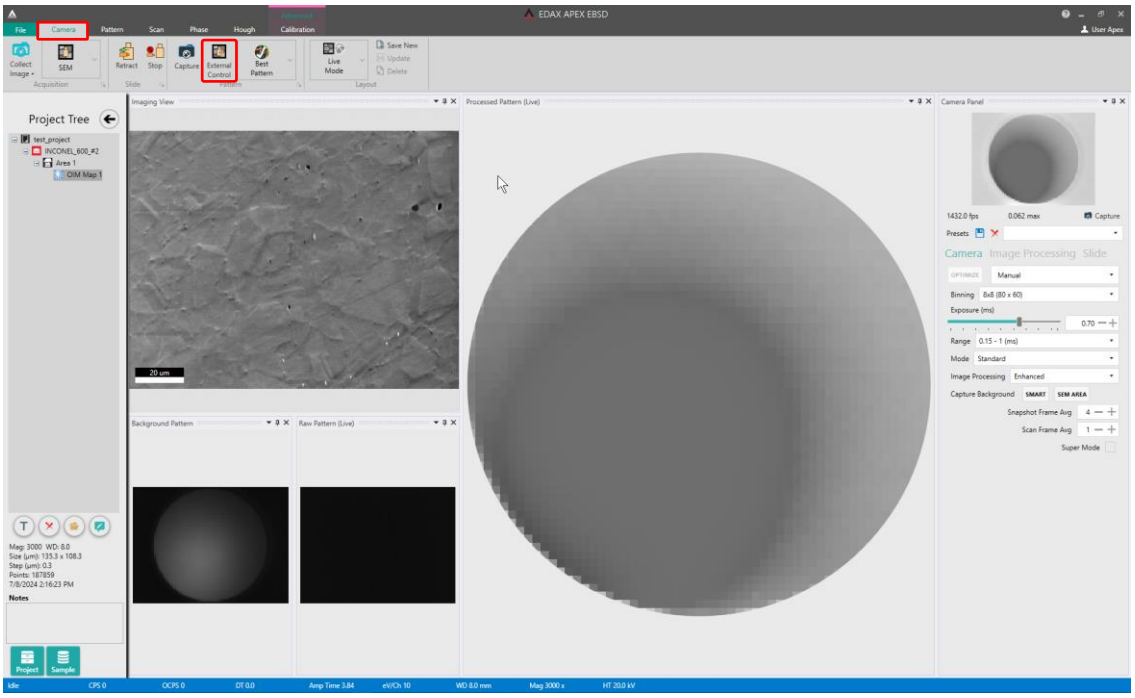


11. Finishing the session

11.1. In APEX, select the “Camera” tab; in the tool ribbon, select “Retract” to retract the camera (listen for the slide motor).



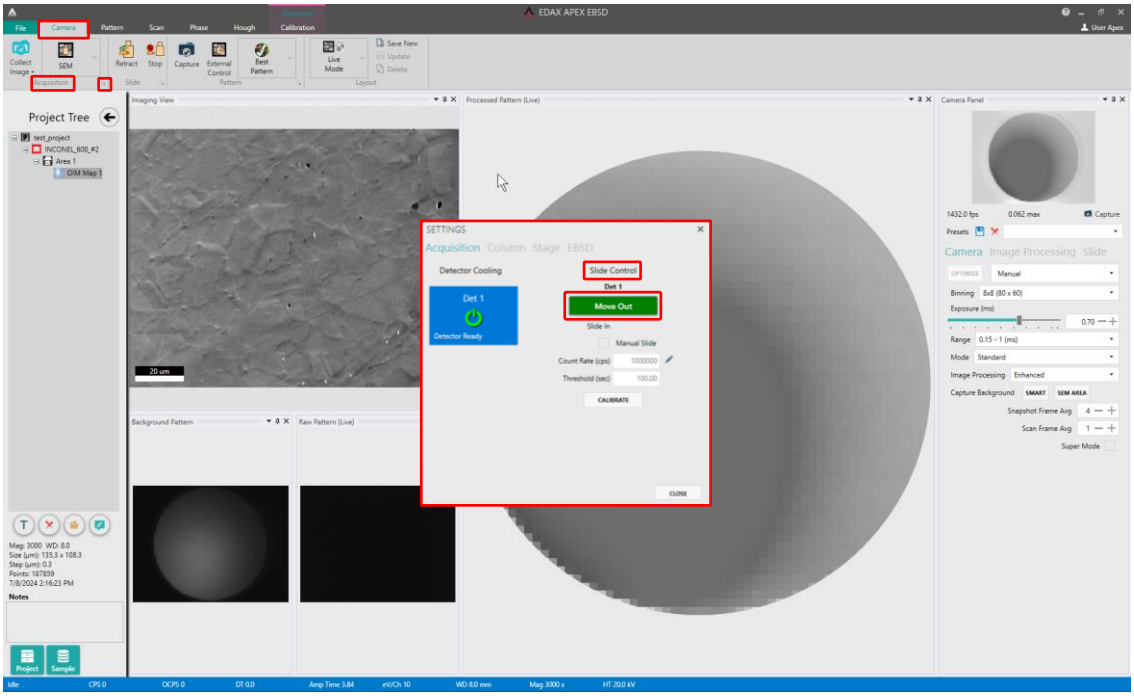
11.2. Remain, in the “Camera” tab; in the tool ribbon, verify “External Control” is deactivated (as shown below); if not, then select it to deactivate it.



11.3. To retract the EDS detector (if necessary), remain in the “Camera” tab; in the tool ribbon, select the arrow in the lower right corner of the “Acquisition” field to open the “Settings” window.

11.3.1. Under “Slide Control”, select “Move Out” to retract the EDS detector (listen for the slide motor).

11.3.2. NOTE: it is not necessary to turn off cooling to the EDS detector when finished using it.



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

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

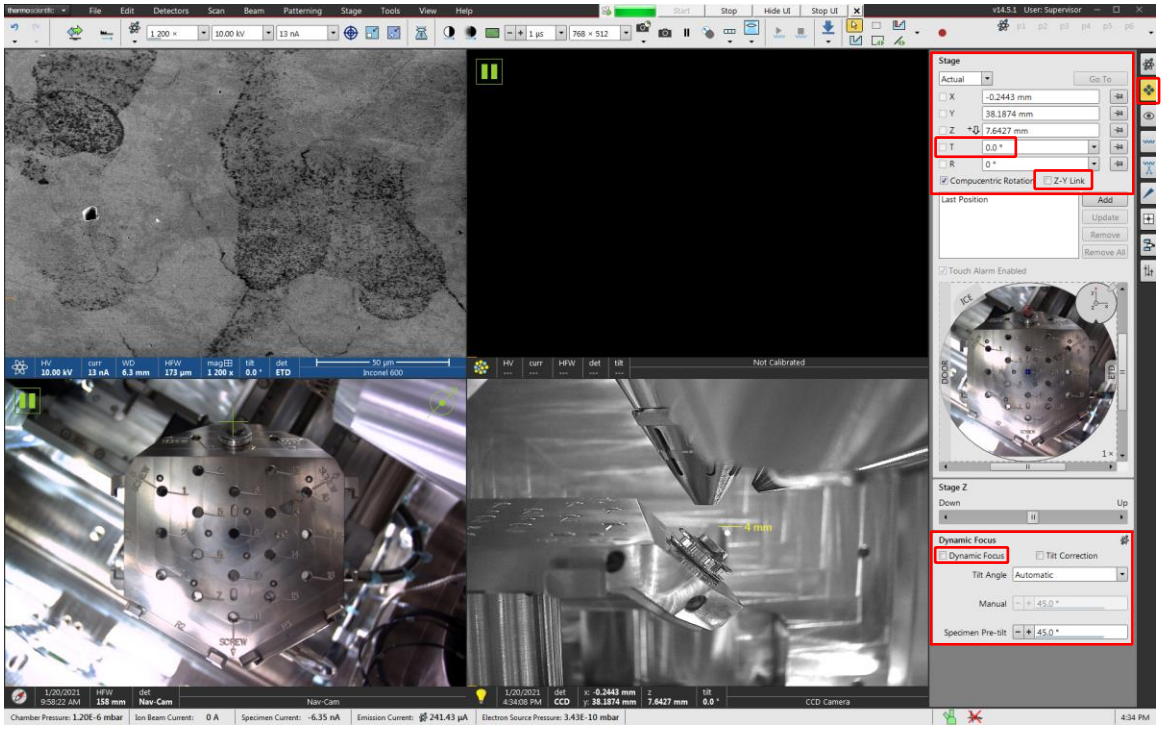
11.5.1. Tilt the stage back to T = 0°; this may be done all at once, it is not necessary to do this incrementally.

11.5.2. Uncheck “Z-Y Link”.

11.5.3. NOTE: it is extremely important that “Z-Y Link" is unchecked when finished; otherwise, this will cause confusion for the next user or possibly lead to a collision with the SEM pole piece.

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

11.6.1. Uncheck “Dynamic Focus”.



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