FEI Helios Nanolab 600i: EBSD using EDAX APEX

Nicholas G. Rudawski
ngr@ufl.edu
Office: (352) 392-3077
Last updated: 01/03/25

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

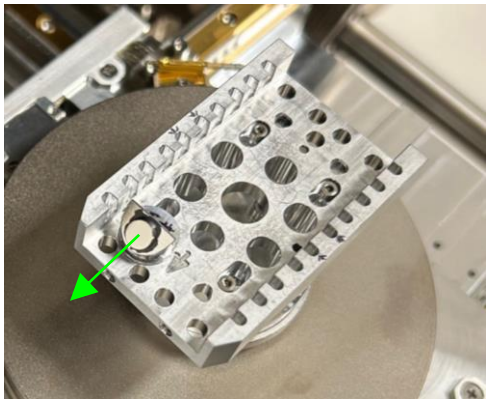
    1.1. EBSD specimens must be mounted on ~13 mm diameter 45° pre-tilt pin stubs; the preferred stubs for this are Ted Pella #16104. The specimens should have a flat, damage-free, and smooth surface and have a footprint that fits basically within the tilted surface of the stub, the thickness to footprint ratio of the specimen should be kept as small as possible to increase specimen stability.

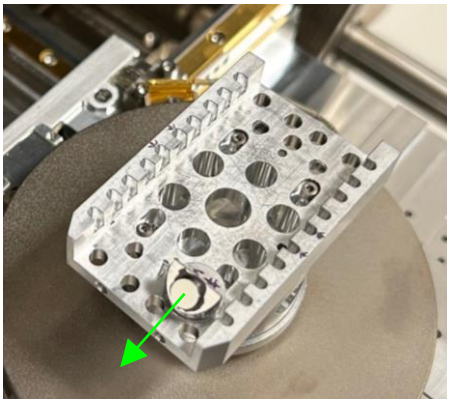
    1.2. It is preferable to mount specimens using *conductive paint* instead of carbon tape to limit specimen drift; *if you use conductive paint, make sure it is well-dried before loading your sample into the chamber.* If your sample is non-conductive, it should be given a light C coat (few nm) *after*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

2. UMB positions for EBSD

    2.1. Stubs must be loaded in two positions shown below: “EBSD_1” (farther from door) and “EBSD_2” (closer to door), at the end of the UMB facing the EBSD camera.

    2.2. With the stage in the stored “load/unload” position, load the stubs to face the specimen as precisely as possible towards the EBSD camera (indicated by arrows).





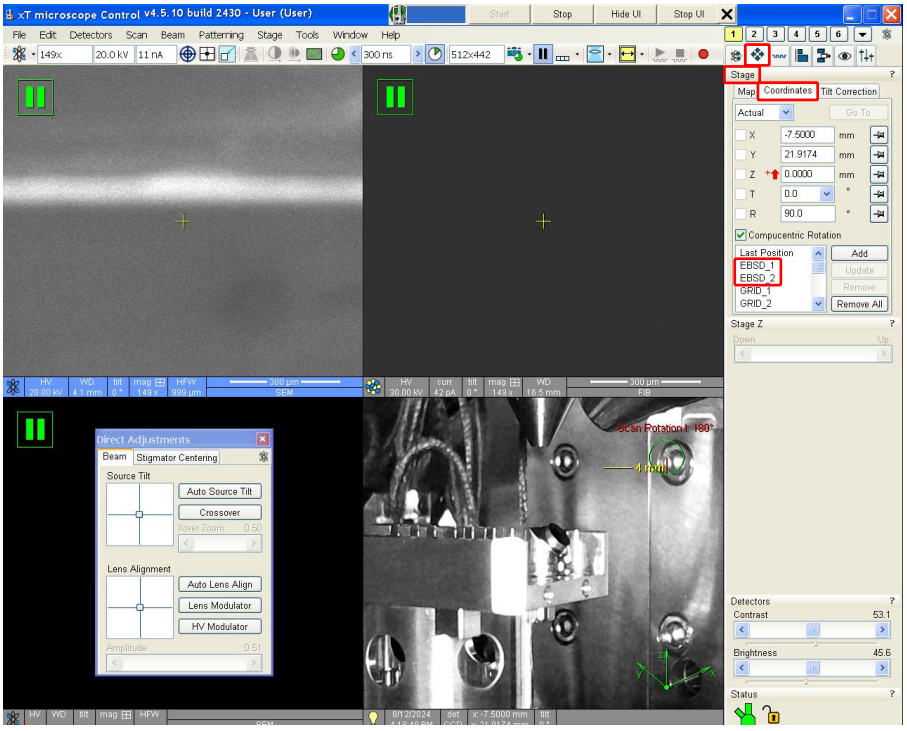
3. Stored UMB EBSD stage positions

3.1. Close the chamber door and pump down the chamber.

3.2. In Microscope Control, enter the “Navigation” module and navigate to the “Stage” panel.

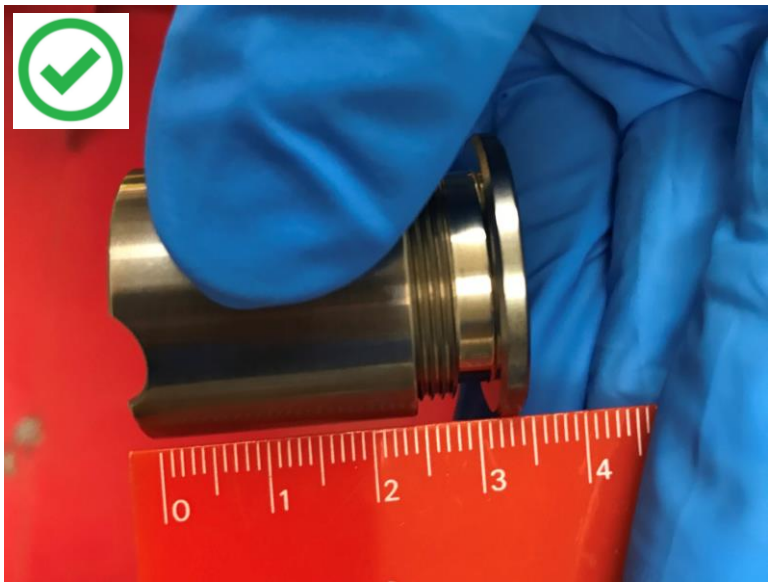
3.3. Select the “Coordinates” tab and then go to the list of stored stage positions.

3.4. Select either “EBSD_1” or “EBSD_2” to move the corresponding sample directly under the electron beam and directly facing the EBSD camera.

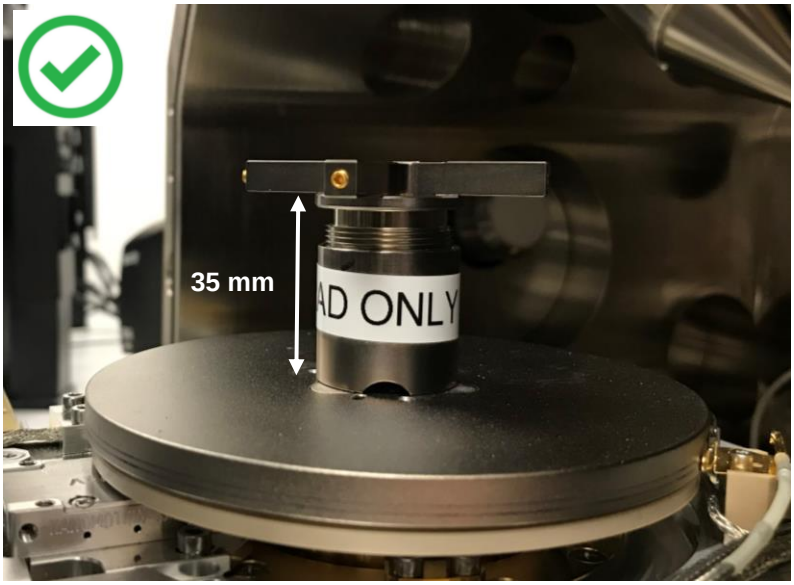


4. Using the 4-post cross stage attachment

4.1. If using this stage attachment (instead of the UMB), the height of the locking cylinder should be adjusted to ~35 mm; this ensures that the specimens can be set to the optimal WD for EBSD.



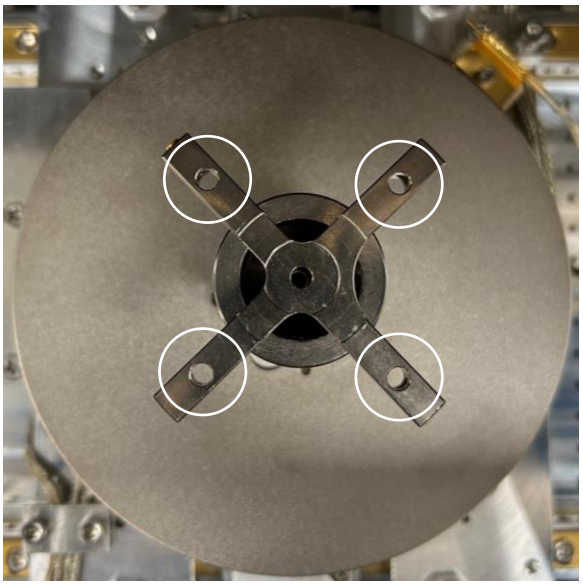
4.2. When installed on the stage, the attachment should appear as shown below.



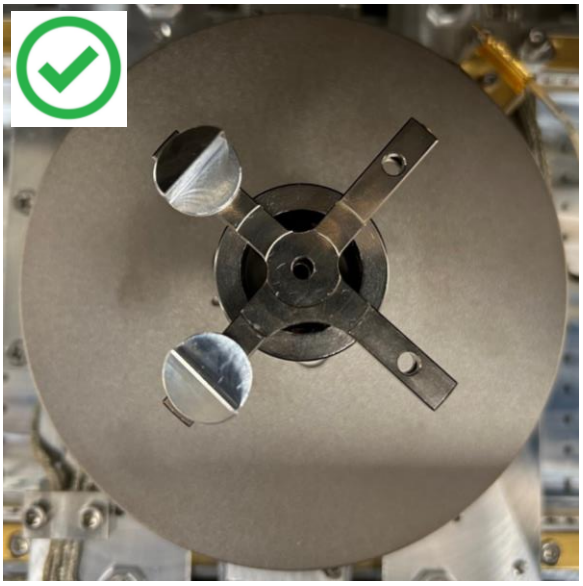
4.3. For EBSD work, stubs must be installed on the four outer post positions (not in the middle position of the cross).

4.3.1. NOTE: these are the only positions that will allow safe insertion of the EBSD camera when using this stage attachment.

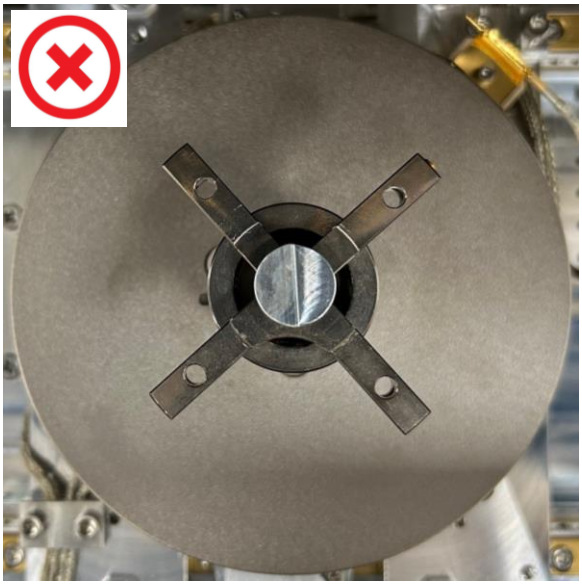
4.4. NOTE: unlike the UMB, there are no stored positions for this stage attachment; thus, the stubs will need to be manually located during live SEM imaging.



The four correct positions for loading stubs



Correct loading using post positions

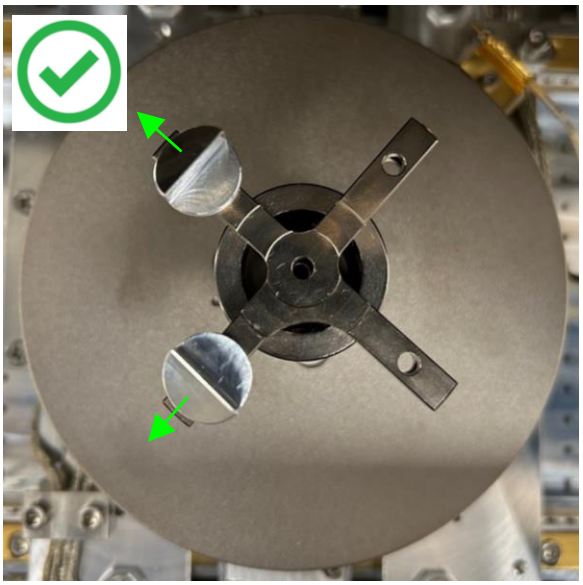


Incorrect loading in middle position

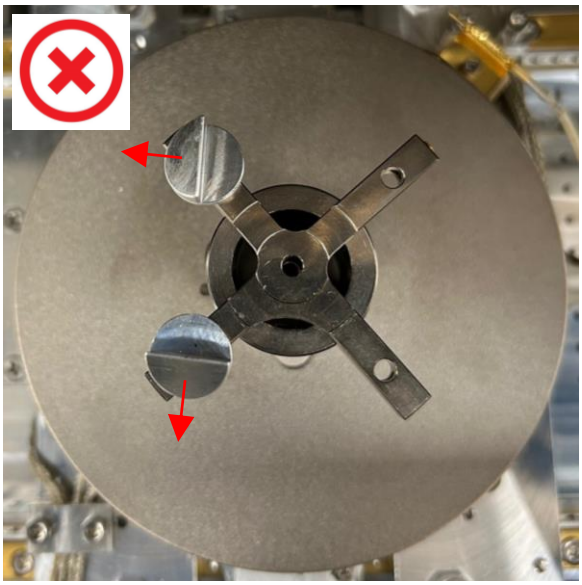
4.5. Each stub must be installed to face the specimen outward from the center position and as closely along the post direction as possible.

4.5.1. NOTE: this is to ensure the EBSD camera can be safely inserted with the sample properly oriented towards the EBSD camera.

4.5.2. For each specimen, the stage will need to be rotated to properly orient it towards the EBSD camera. This will be performed during live SEM imaging as will be described subsequently.



Correct stub orientation on posts



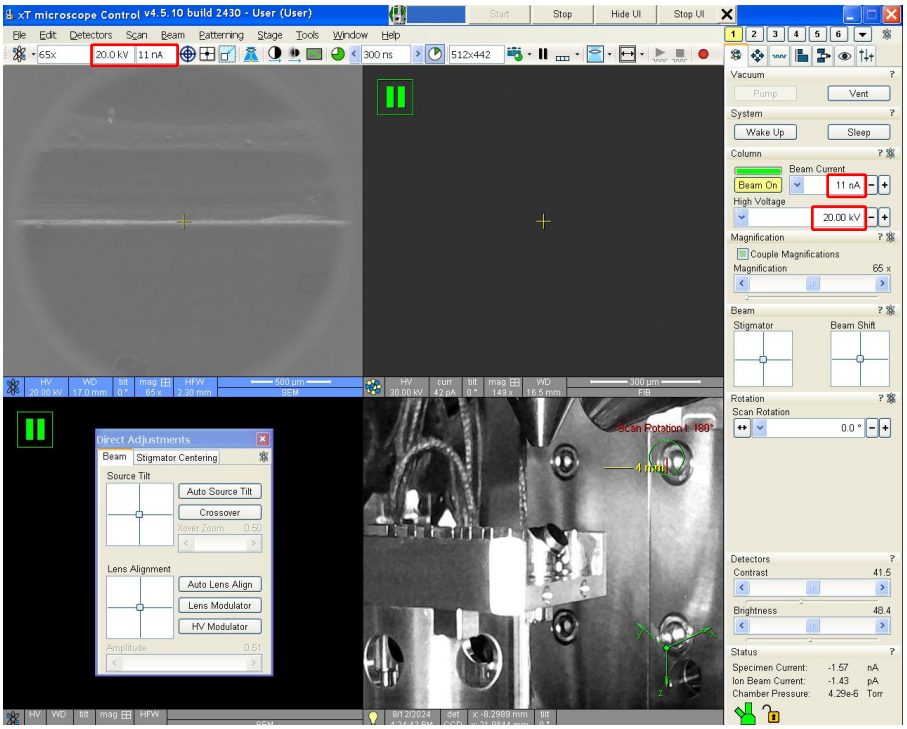
Incorrect stub orientation on posts

5. Instrument settings

5.1. 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.

5.1.1. 20 kV with 11 nA of current is recommend as a general all-around SEM setting for performing EBSD.

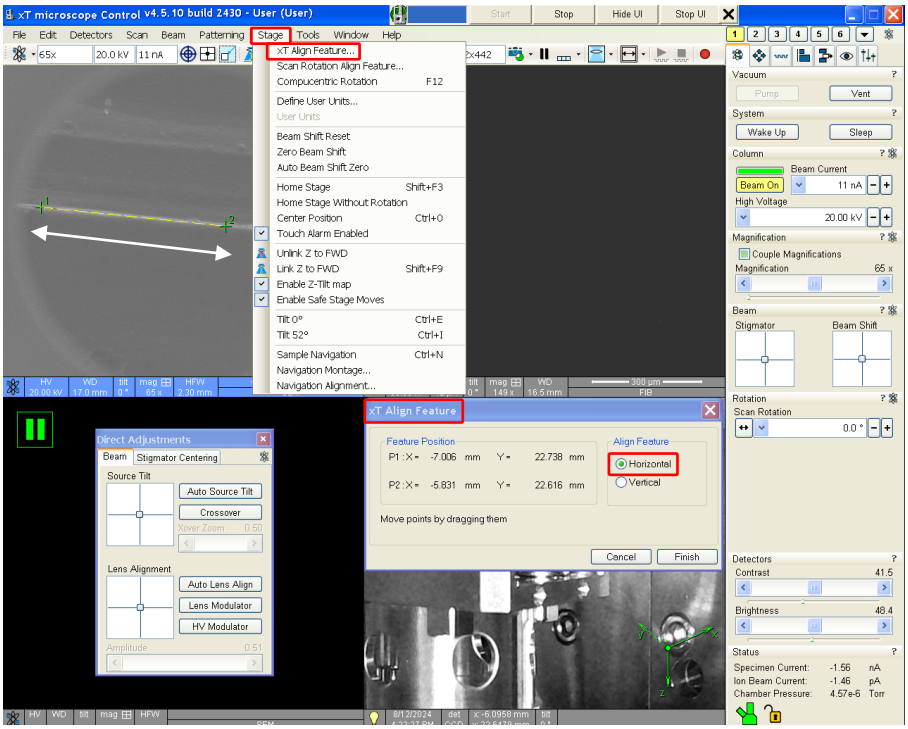
5.1.2. If unsure as to what beam settings to use for EBSD, please consult with staff for recommendations.



5.2. Once the sample is located, the stage must be rotated to face the sample directly towards the EBSD camera as precisely as possible. This can be accomplished using the straight edge of the face of a 45° pre-tilted stub.

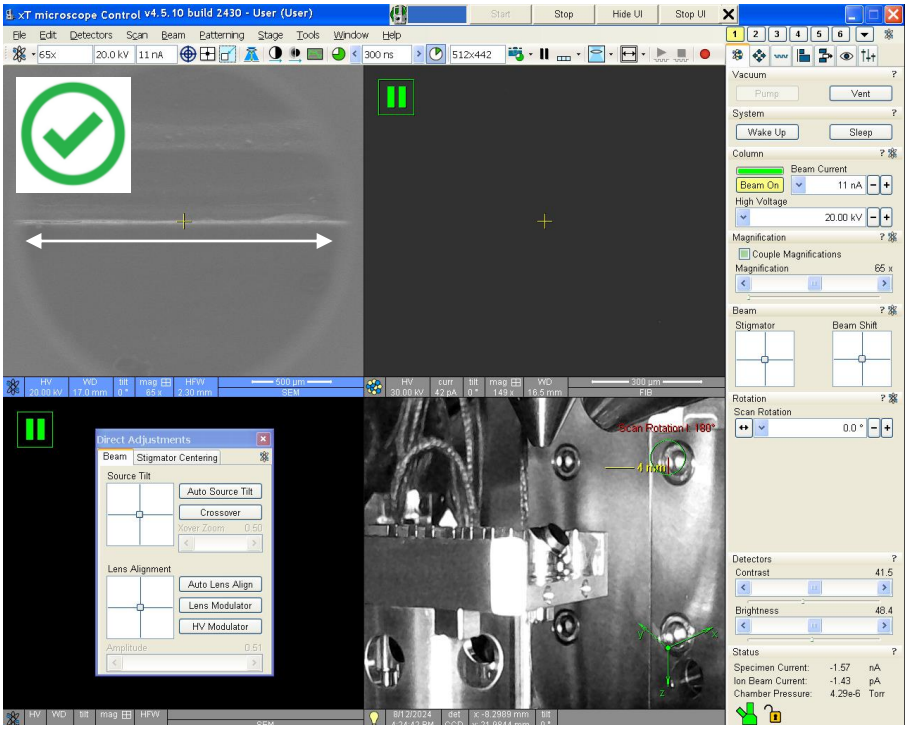
5.2.1. Move the straight edge of the stub face into the live SEM image; select the “Stage” pull-down menu and then “xT Align Feature”.

5.2.2. Click and drag along the straight edge and use the “xT Align Feature” dialogue box to align it with the horizontal direction.



5.2.3. If aligned properly, the SEM image of the straight edge should appear as shown below.

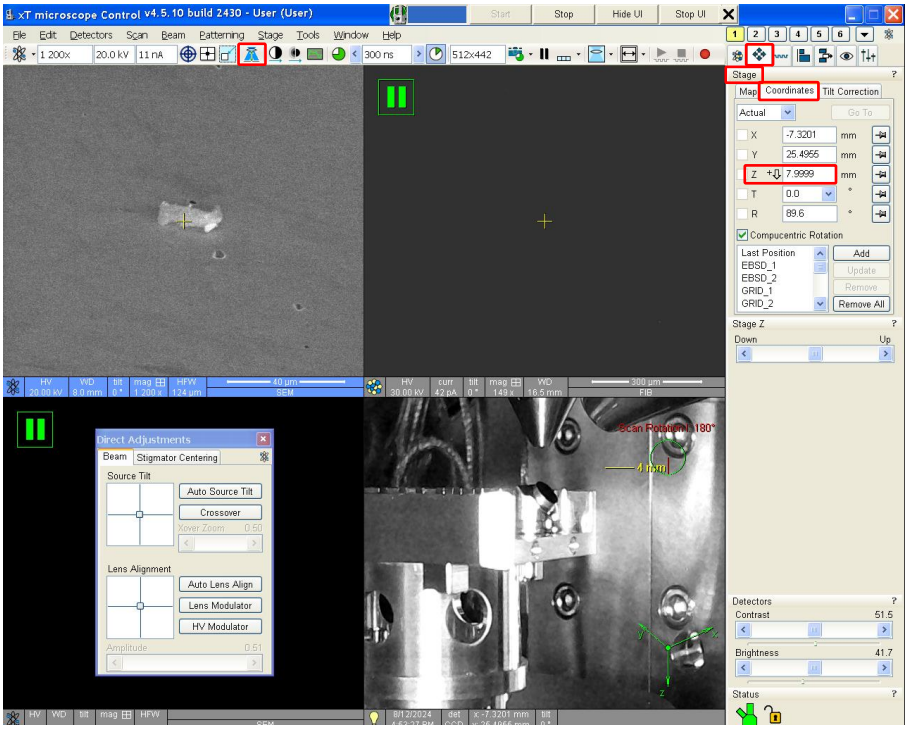
5.2.4. NOTE: the sample surface must face upwards in the SEM image to "see" the EBSD camera; if it is pointing downwards, (relative) rotate the stage by 180° to properly orient it.



5.3. Navigate to the specimen and find a region of interest (ROI); ideally, the ROI should be in the middle of the sample surface away from any edges.

5.3.1. Focus on the ROI, link Z to WD, and bring the ROI to WD = 8; this working distance is optimal for EBSD camera performance.

5.3.2. Perform basic SEM alignment: source tilt, lens alignment, and astigmatism correction (not shown).

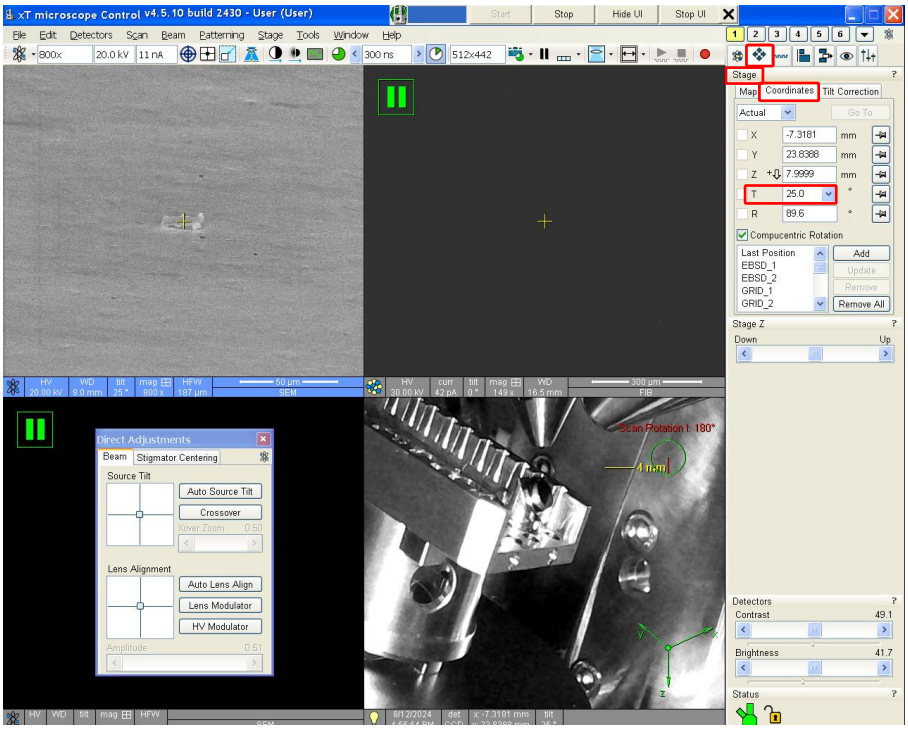


5.4. Near the ROI, center a recognizable feature in the live image; then enter the Navigation module, and navigate to the “Stage” panel.

5.4.1. Select the “Coordinates” tab.

5.4.2. Tilt the stage to T= 25° (actual specimen tilt will now be T = 70°).

5.4.3. Find and recenter the feature, refocus, link Z to WD (again), and set WD = 8 mm (again).



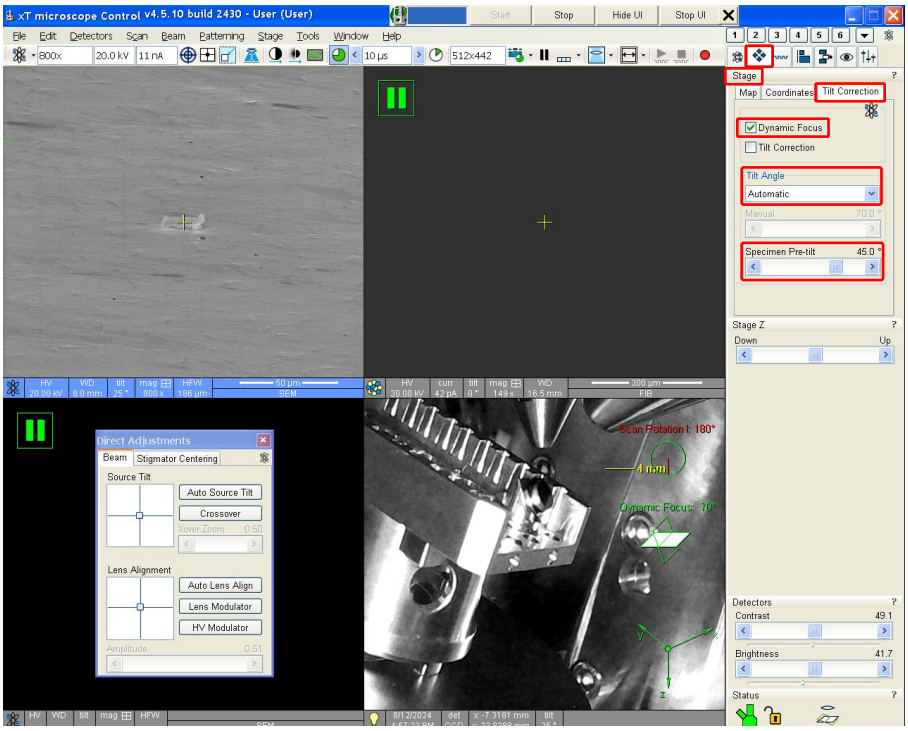
5.5. Remain in the Navigation module and navigate to the “Stage” panel.

5.5.1. Select the “Tilt Correction” tab.

5.5.2. Check “Dynamic Focus”.

5.5.3. Set “Tilt Angle” to “Automatic”.

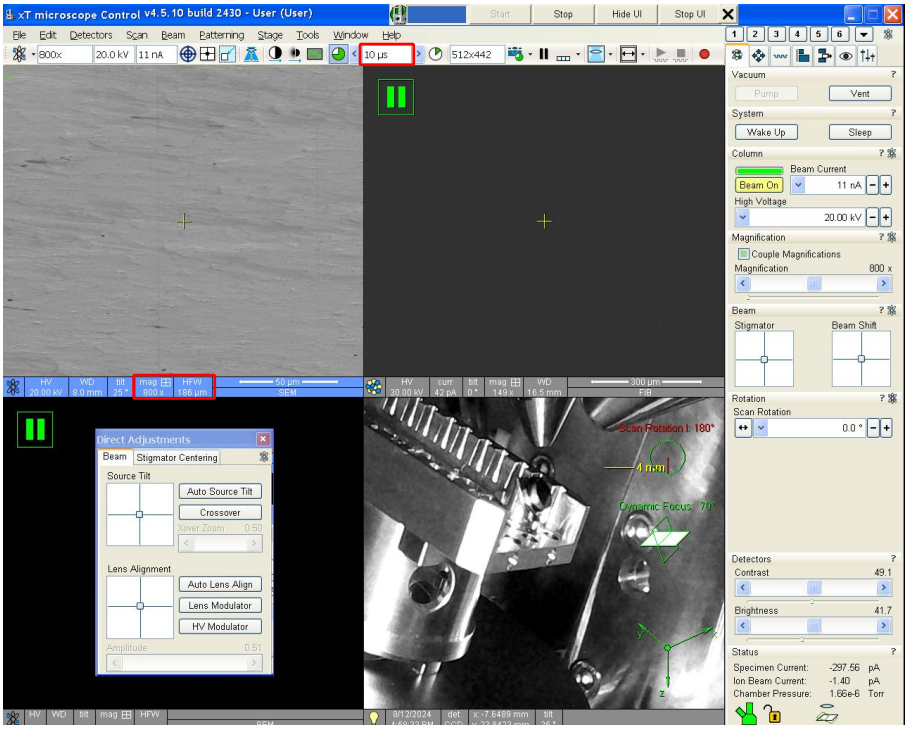
5.5.4. Set “Specimen Pre-tilt = 45°.



5.5.5. Focus precisely on the middle of the image.

5.5.6. Adjust the horizontal field width (magnification) as appropriate for EBSD mapping.

5.5.7. Set the dwell time = 10 µs (for dynamic focus to work properly).



6. Starting APEX

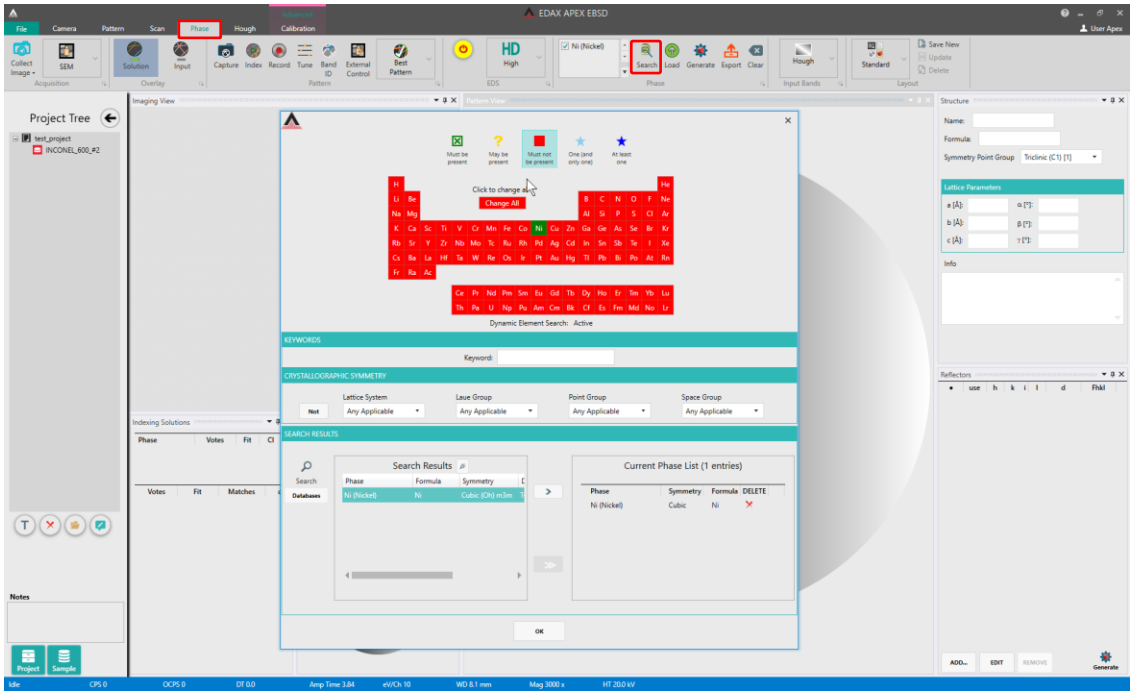
6.1. Open the APEX EBSD software (no login needed); a dialogue box asking to create a project and sample name (or to load a previously created project) will pop up.

6.2. 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.

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

6.3. 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>

6.3.1. 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.



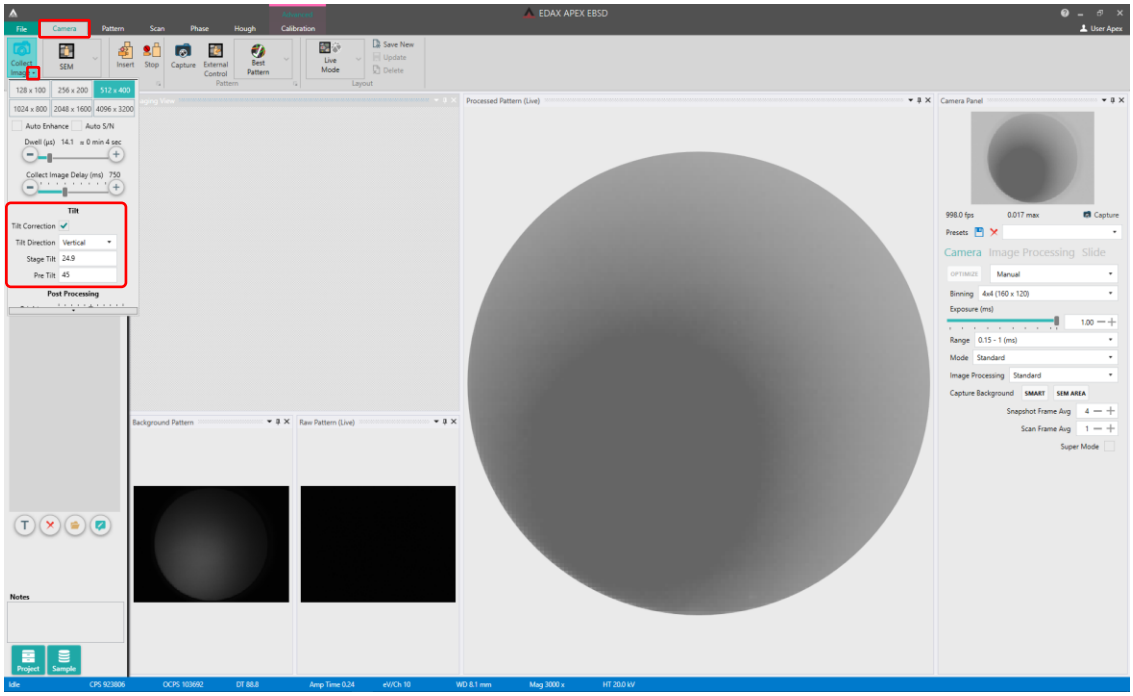
7. Inserting the EBSD camera

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

7.1.1. Verify “Tilt Correction” is checked.

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

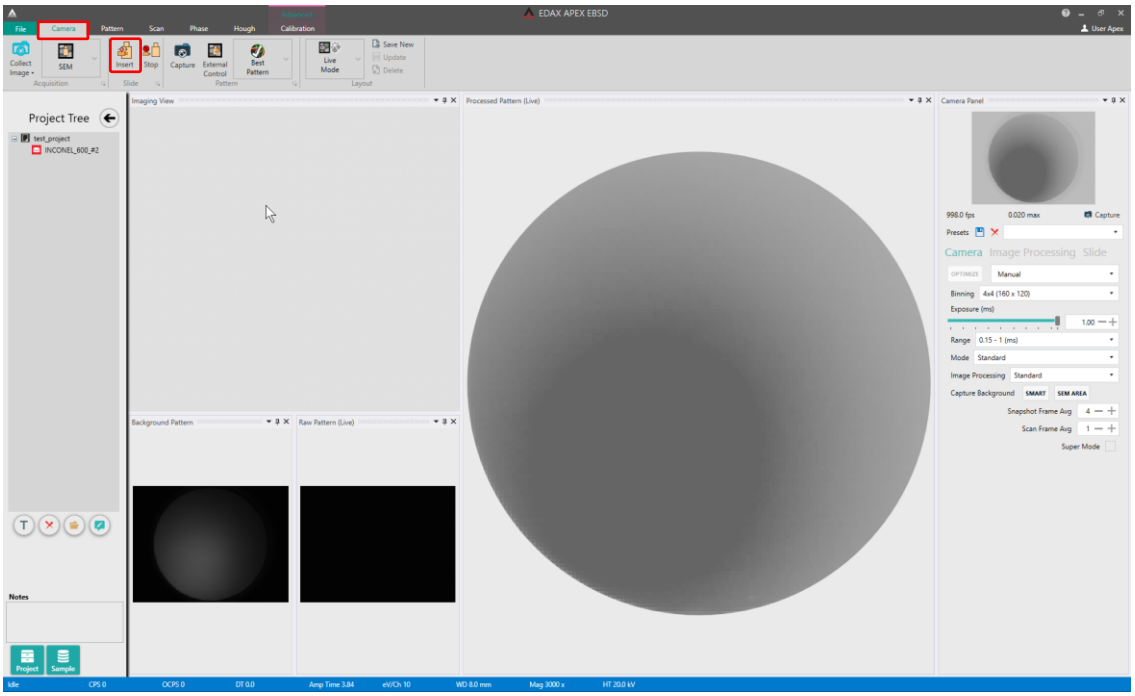
7.1.3. NOTE: specimen tilt = 70° is preferred, but EBSD may still be performed down to a minimum specimen tilt = 60°; the important point is that <u>stage tilt + pre-tilt = actual total specimen tilt</u>.



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

7.2.1. 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.

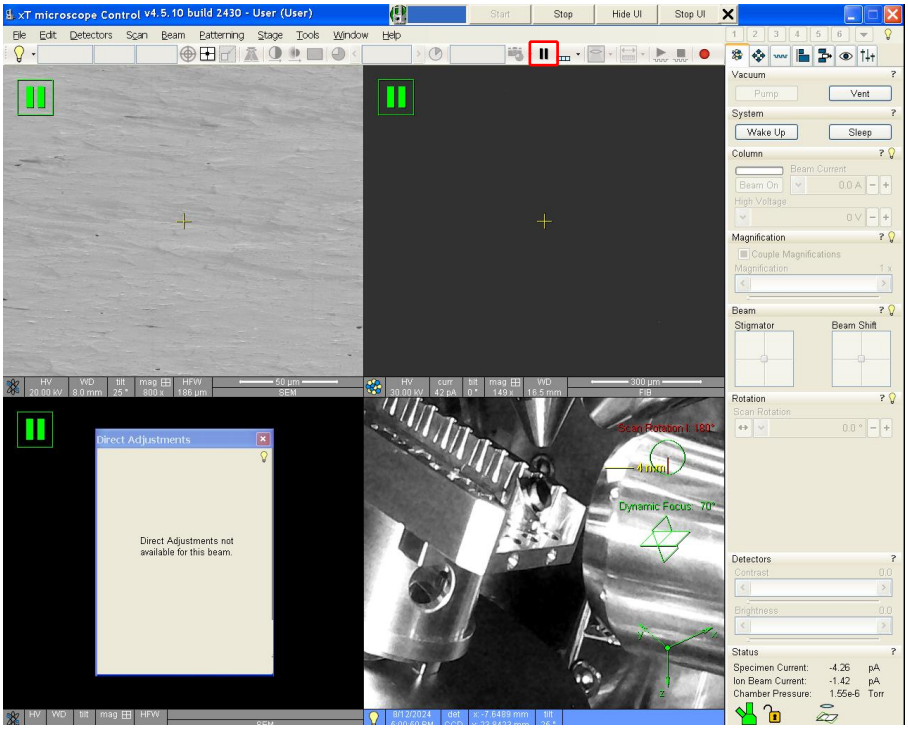
7.2.2. NOTE: APEX will give a clear warning if attempting to insert the camera with the total specimen tilt < 60°.



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

7.3.1. Select the CCD Camera quad and then “Pause” from the main toolbar to turn <u>off</u> the CCD Camera.

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

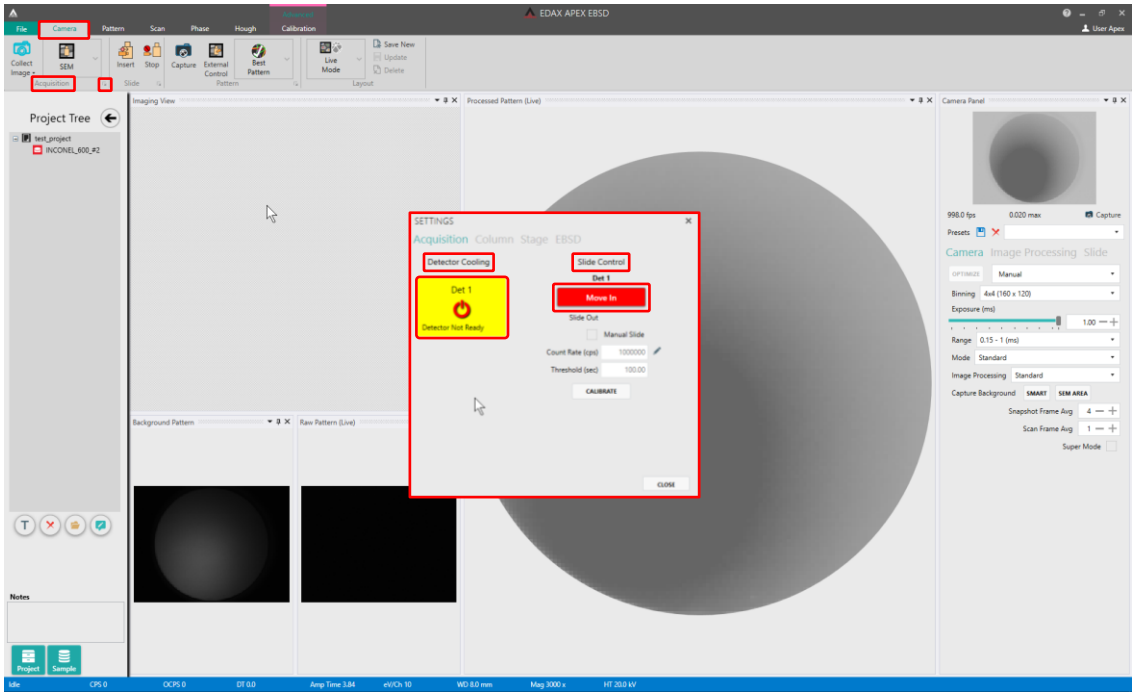


7.4. Simultaneous EDS data collection during EBSD mapping (if desired).

7.4.1. 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.

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

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



8. ROI image collection

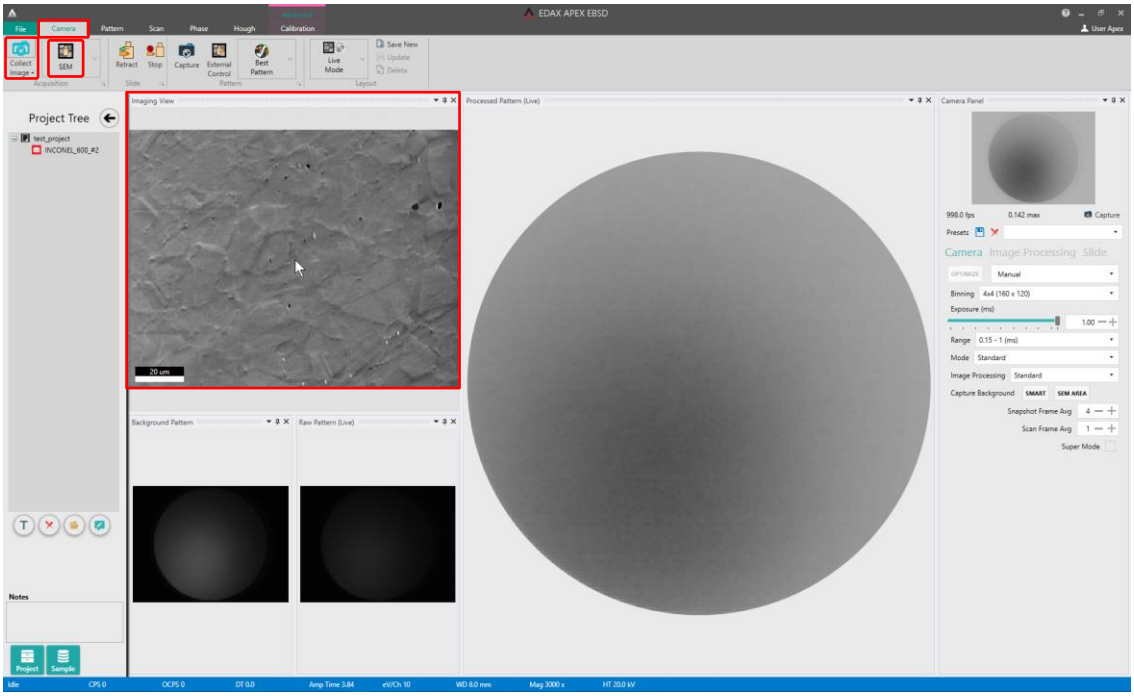
8.1. In Microscope Control, verify the magnification is set as appropriate for performing EBSD mapping and that the image is well-focused.

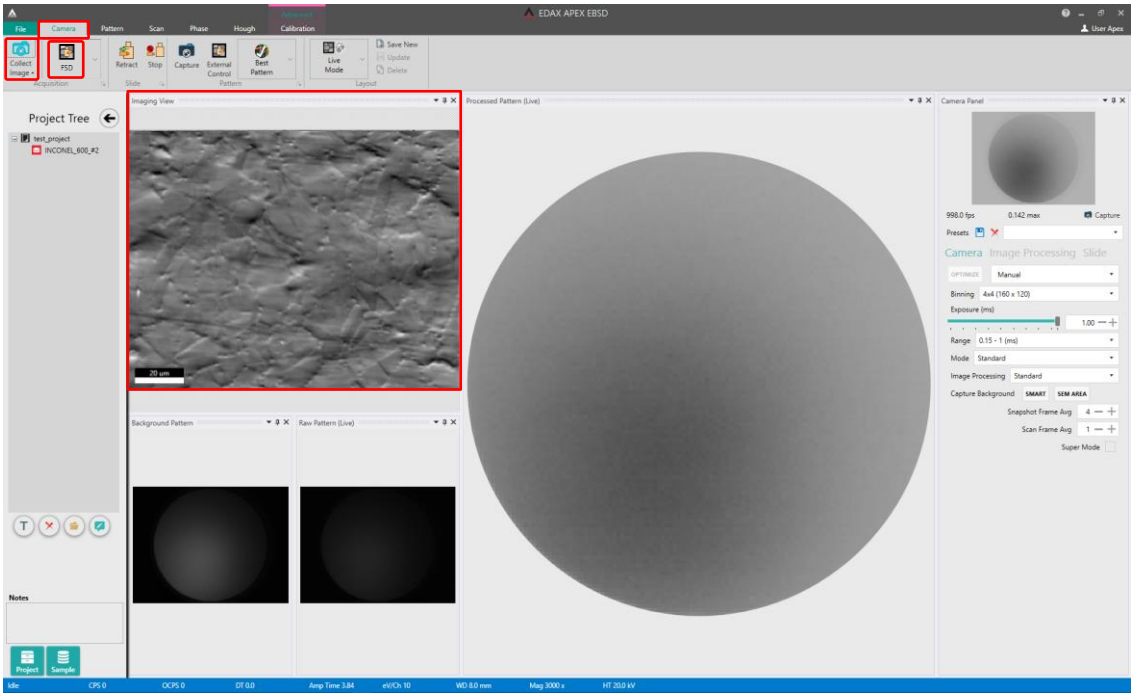
8.2. Remain in the “Camera” tab; in the tool ribbon, select “SEM” to use the same signal used for the SEM image in Microscope Control to collect the ROI image.

8.3. 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

8.3.1. 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.

8.4. 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
Manual
Binning
Exposure (ms)
Range
Mode
Standard
Image Processing
Capture Background
SMART
SEM AREA
Snapshot Frame Avg
Scan Frame Avg
Super Mode
Idle

9. Camera settings; background collection

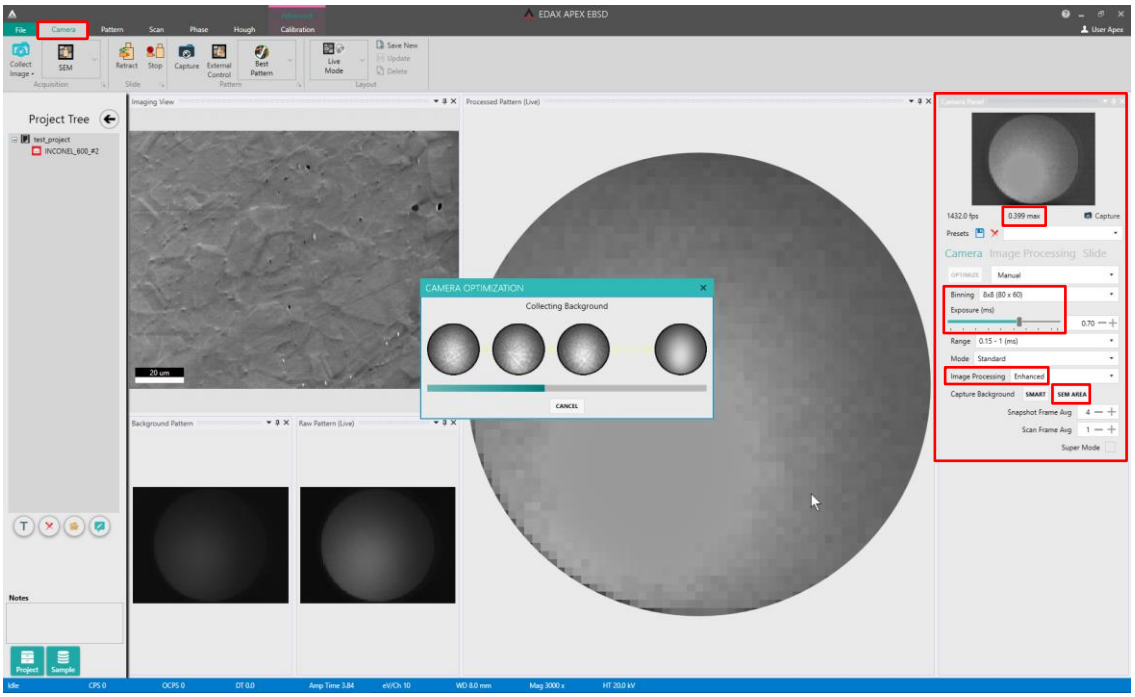
9.1. Remain in the “Camera” tab and navigate to the “Camera” panel; select the desired settings for the EBSD camera (binning, exposure, and image processing).

9.1.1. “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.

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

9.1.3. 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.

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



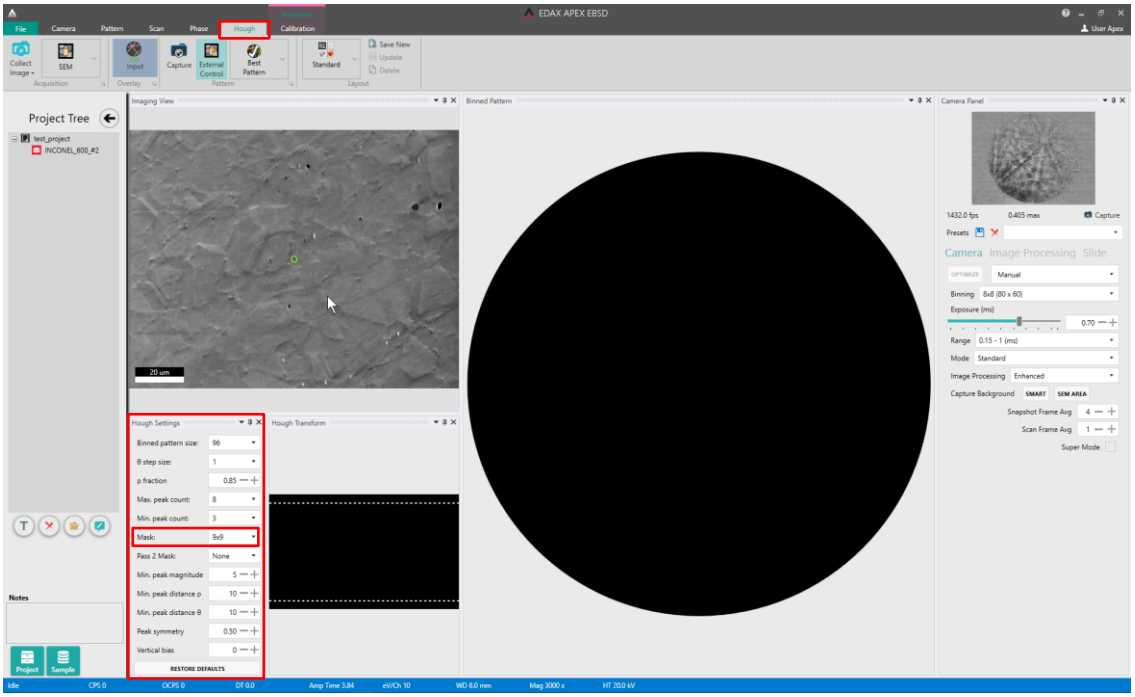
10. Indexing tuning

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

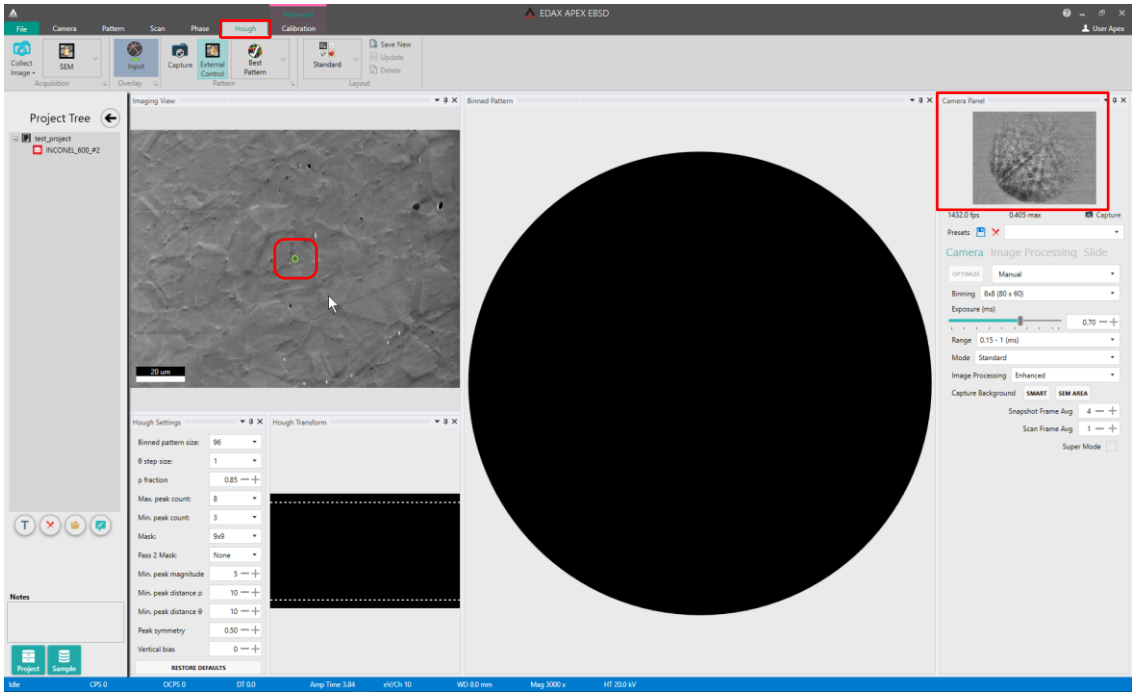
10.2. In APEX, select the “Hough” tab and navigate to the “Hough Settings” panel.

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

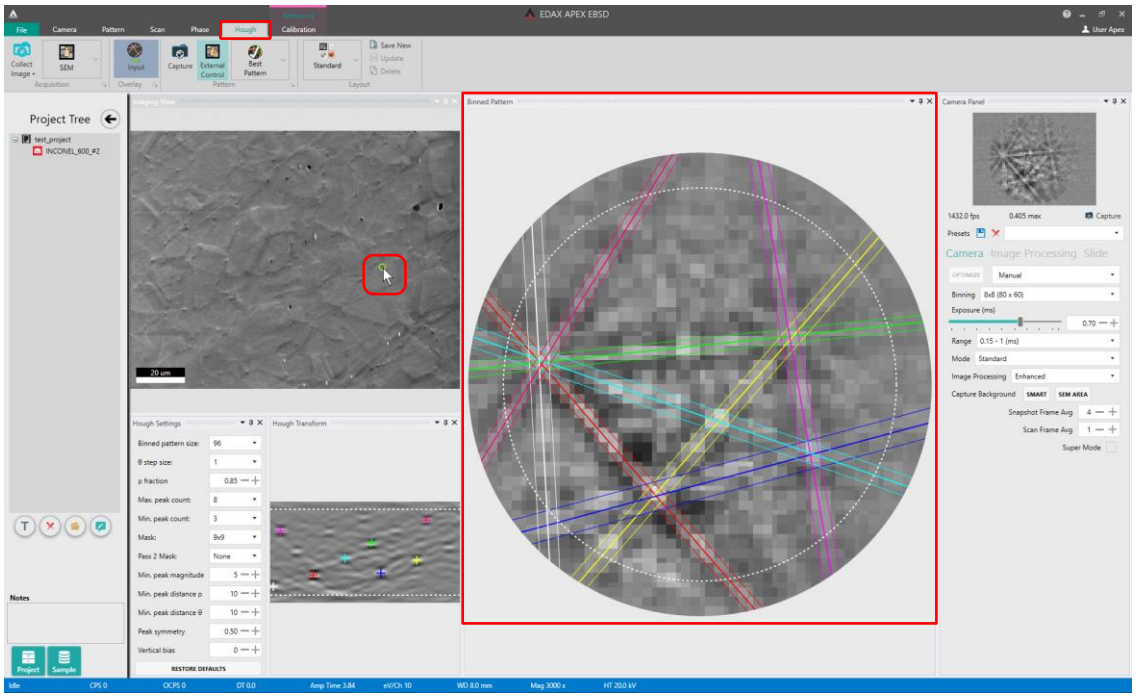
Beam voltage (kV)	Mask
10	MediumLarge_11×11
15	Medium_9×9
20	Medium_9×9
25	SmallMedium_7×7
30	Small_5×5



10.3. 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.



10.4. 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.



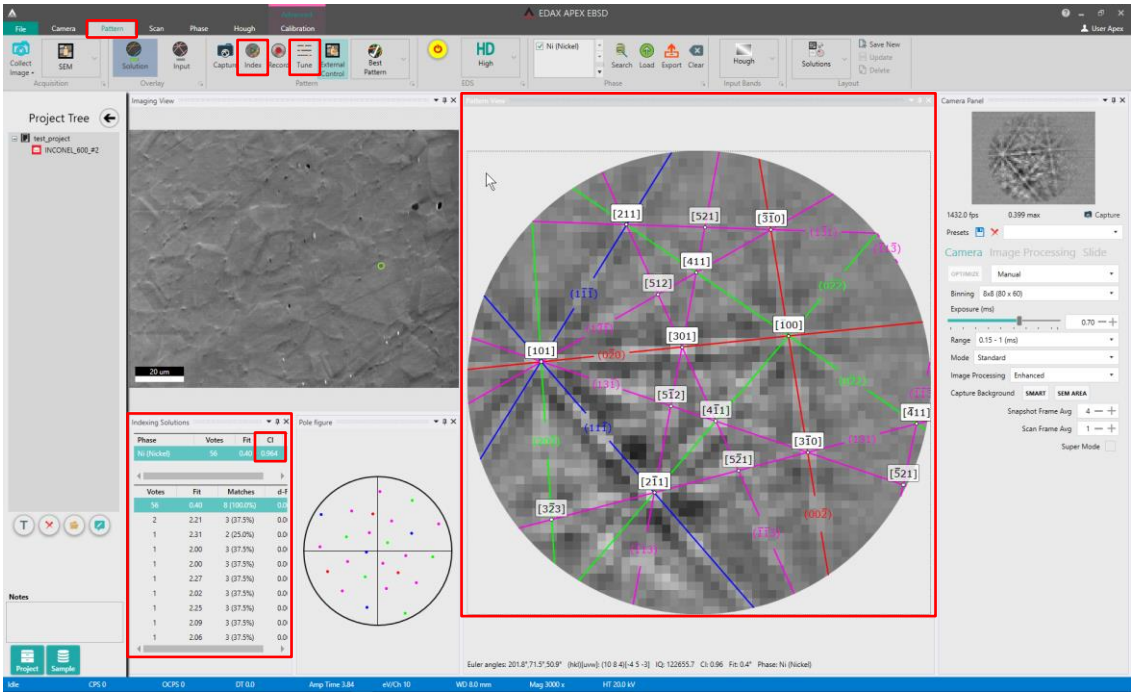
10.5. Select the “Pattern” tab; from the tool ribbon, select “Index” and then “Tune” to index the captured EBSD pattern and tune the indexing.

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

10.5.2. 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%.

10.5.3. 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.

10.5.4. 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.



11. Collecting the EBSD map

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

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

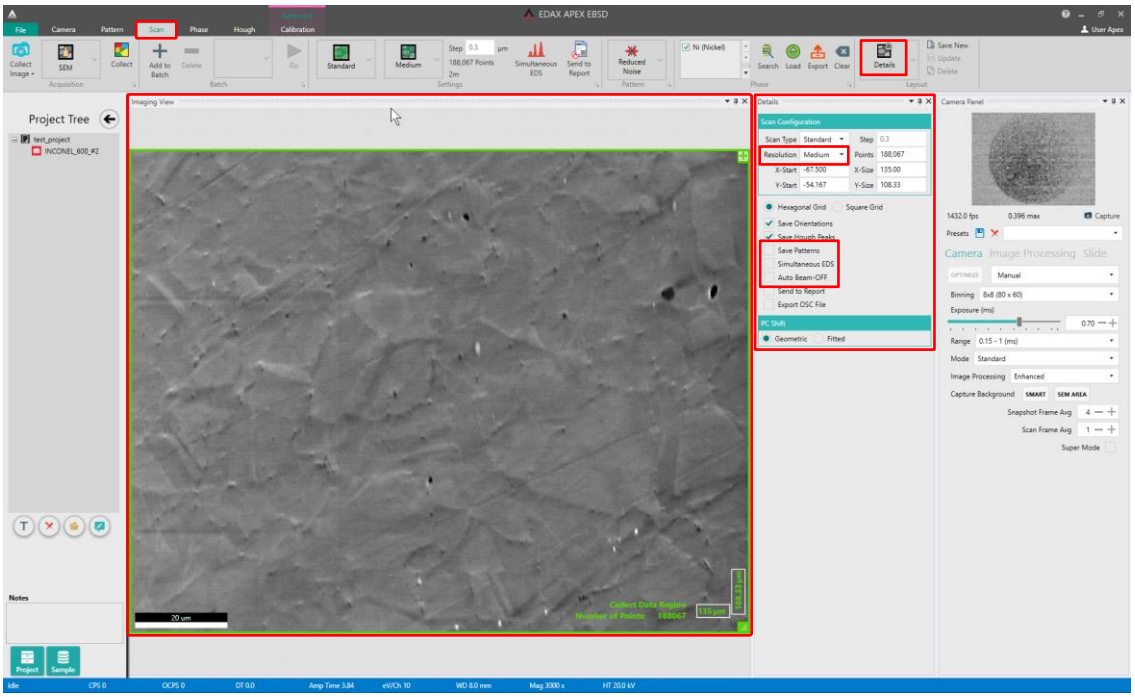
11.2. In the tool ribbon, make sure the layout is set to “Details” and navigate to the “Details” panel.

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

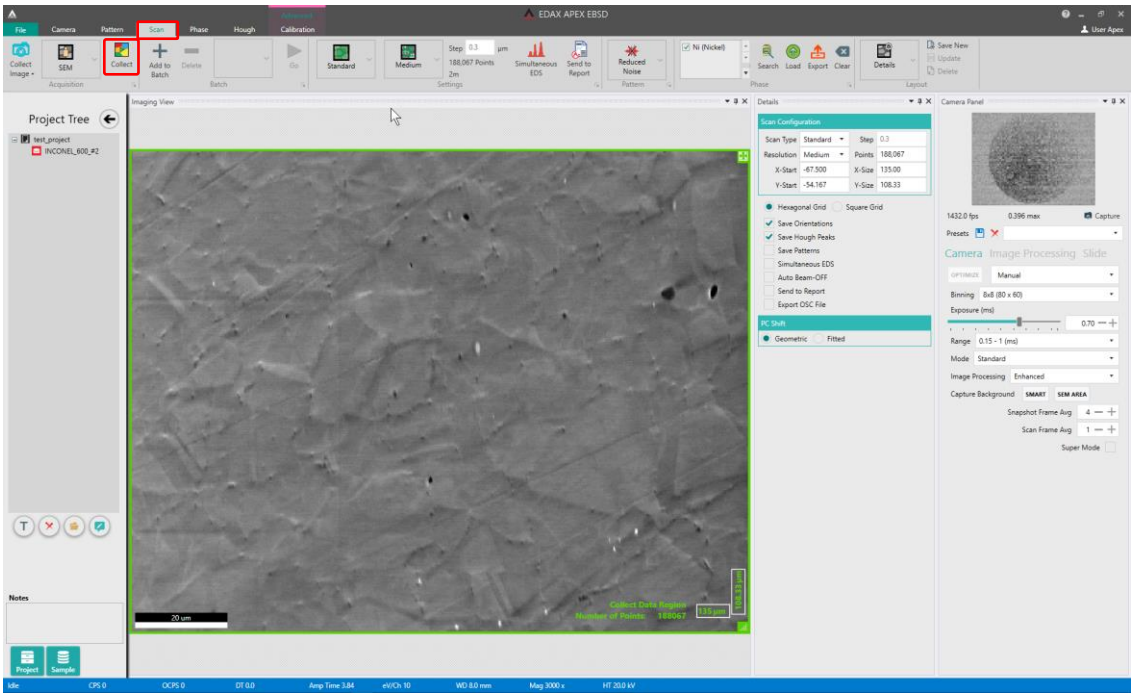
11.2.2. Check “Save Patterns” to save the patterns while mapping; this will dramatically increase the dataset size, so only do this if necessary and for camera binning = 8×8 (or higher).

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

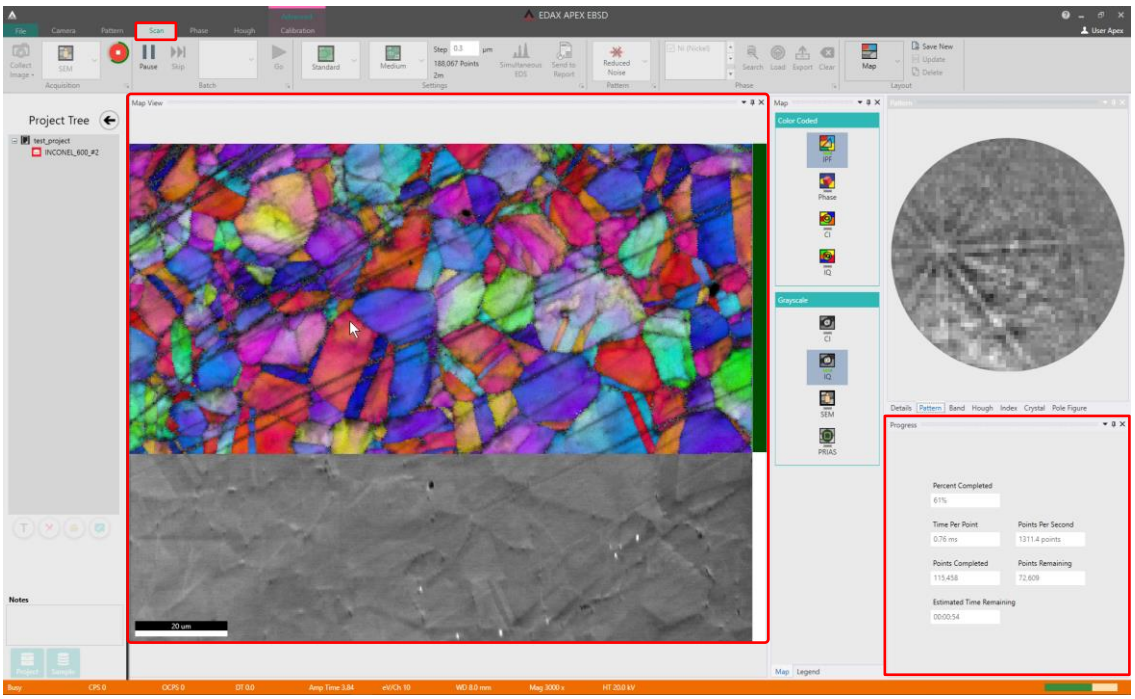
11.2.4. Check “Auto Beam-OFF” to automatically turn the SEM off when finished (recommended for scans taking several hours or longer).



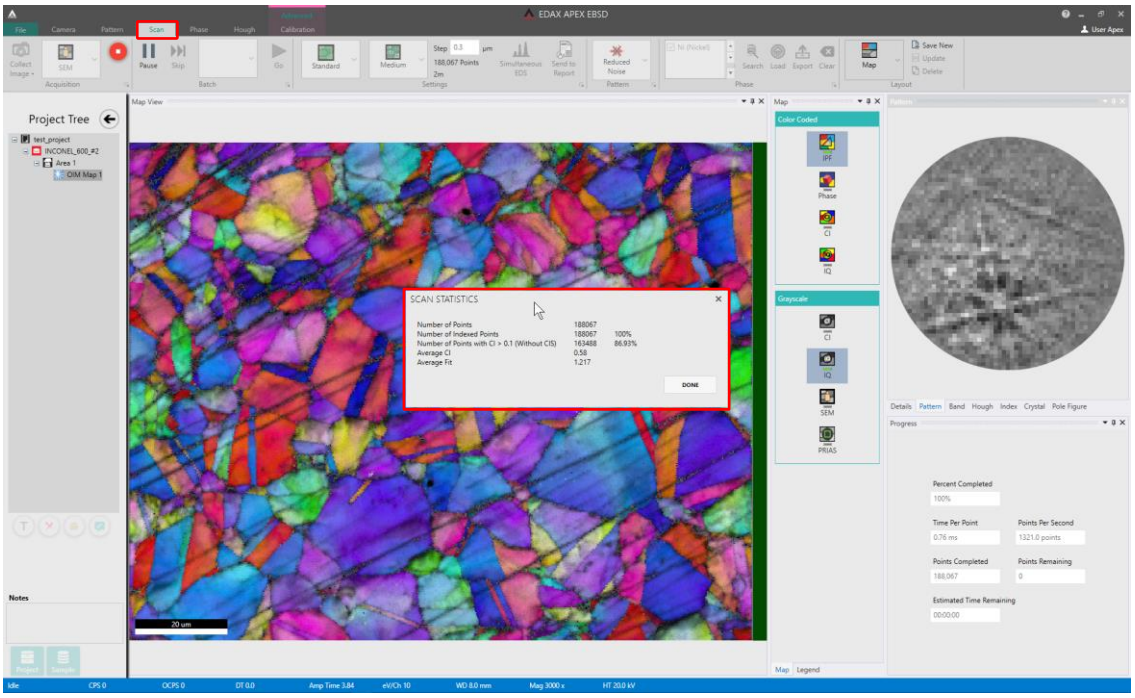
11.3. Remain in the “Scan” tab; in the tool ribbon, select “Collect” to start collecting the map.



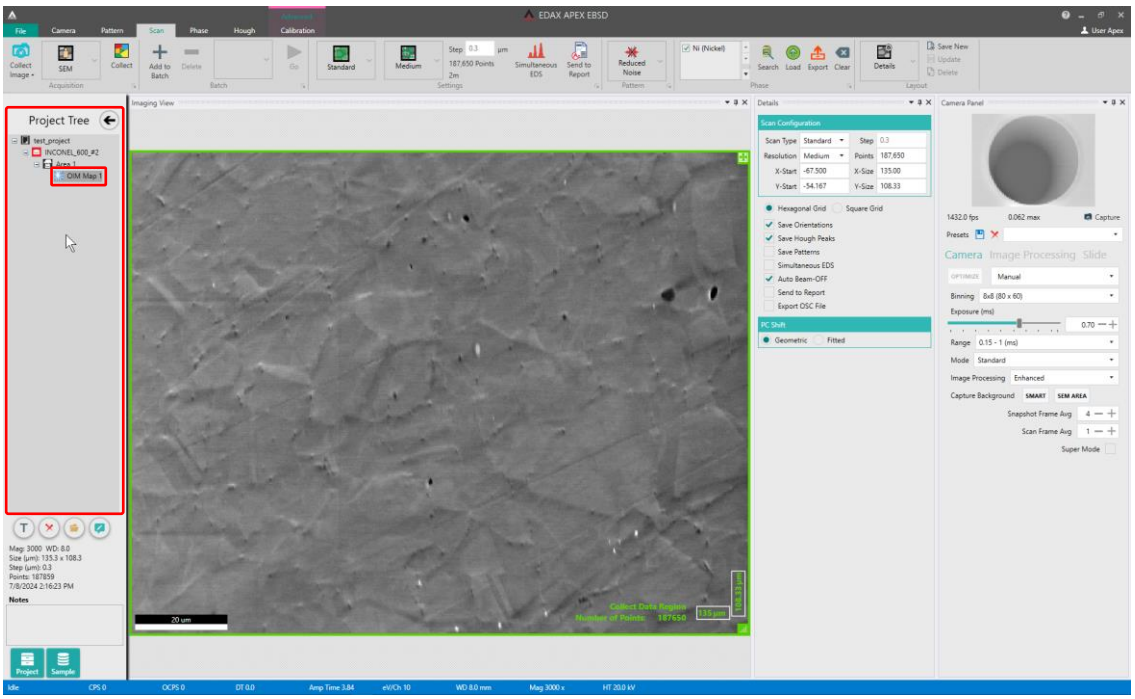
11.4. As the scan proceeds, an IPF map overlayed on an IQ (pattern quality) map will show in the “Map View” panel; additionally, the “Progress” panel shows the progress of the scan.



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

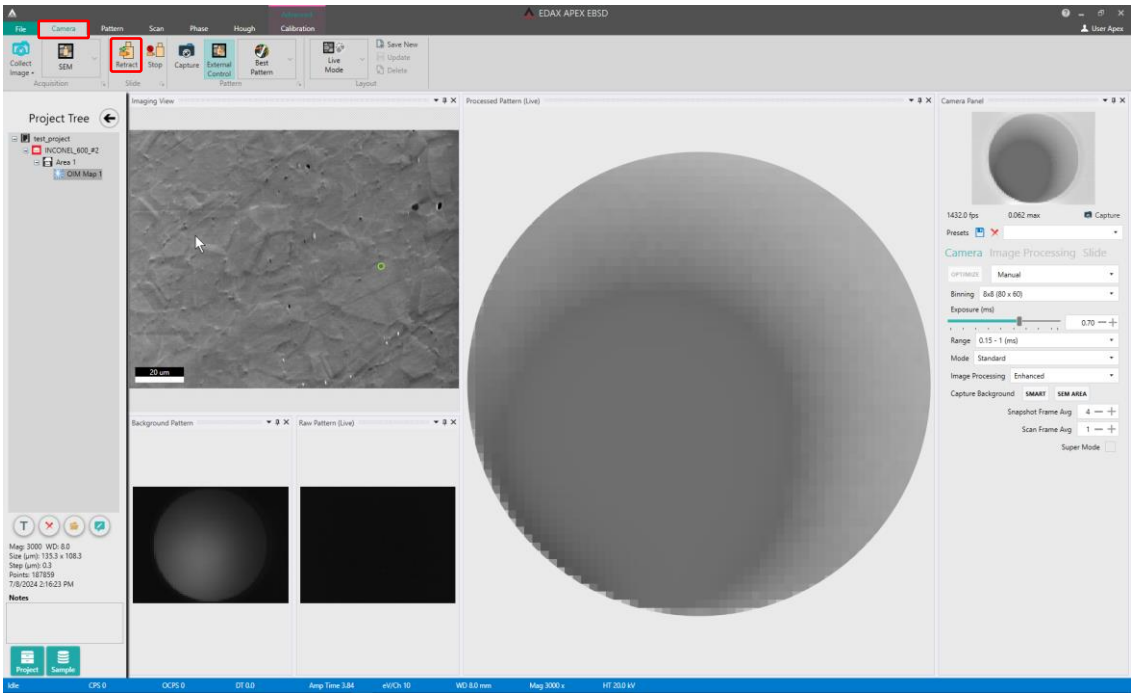


11.6. In the “Project Tree” panel, an “OIM Map” object will be generated for the map. To export the map to OIM for further processing/analysis, simply double left-click on the object.

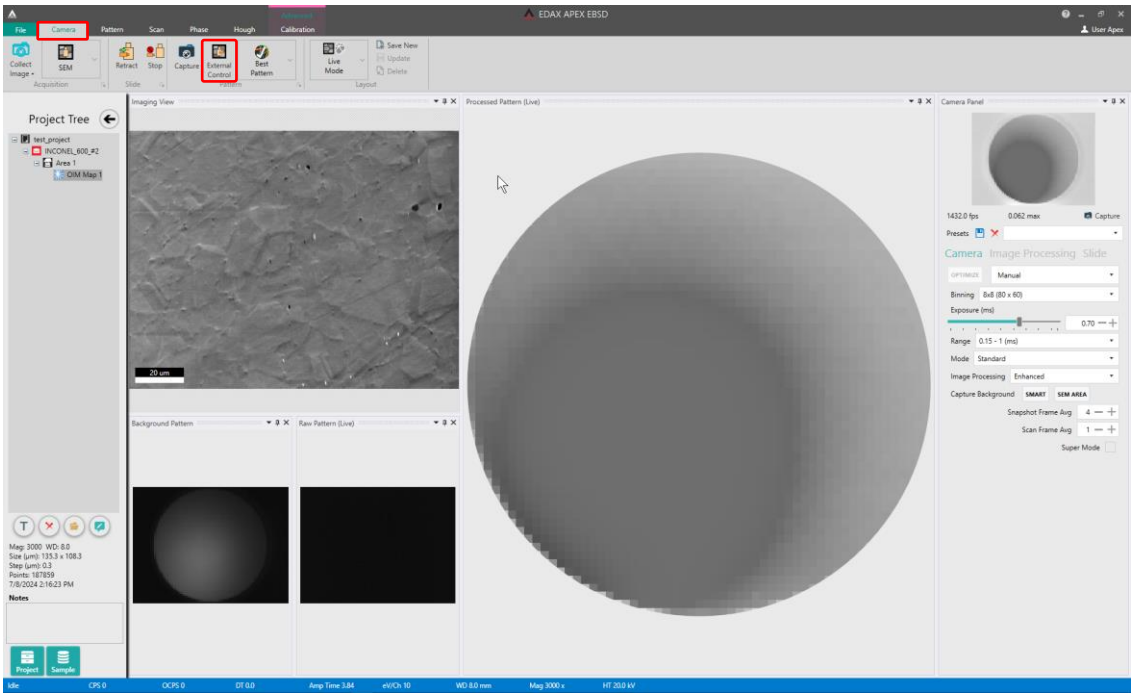


12. Finishing the session

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



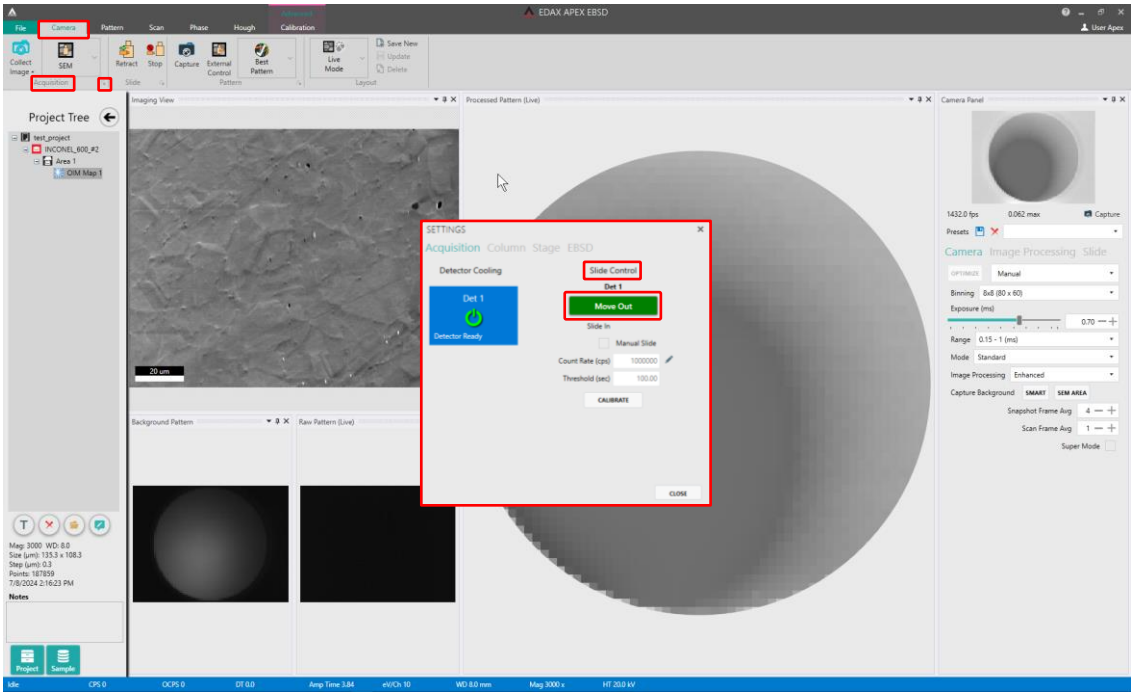
12.2. Remain, in the “Camera” tab; in the tool ribbon, verify “External Control” is deactivated (as shown below); if not, then select it to deactivate it.



12.3. 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.

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

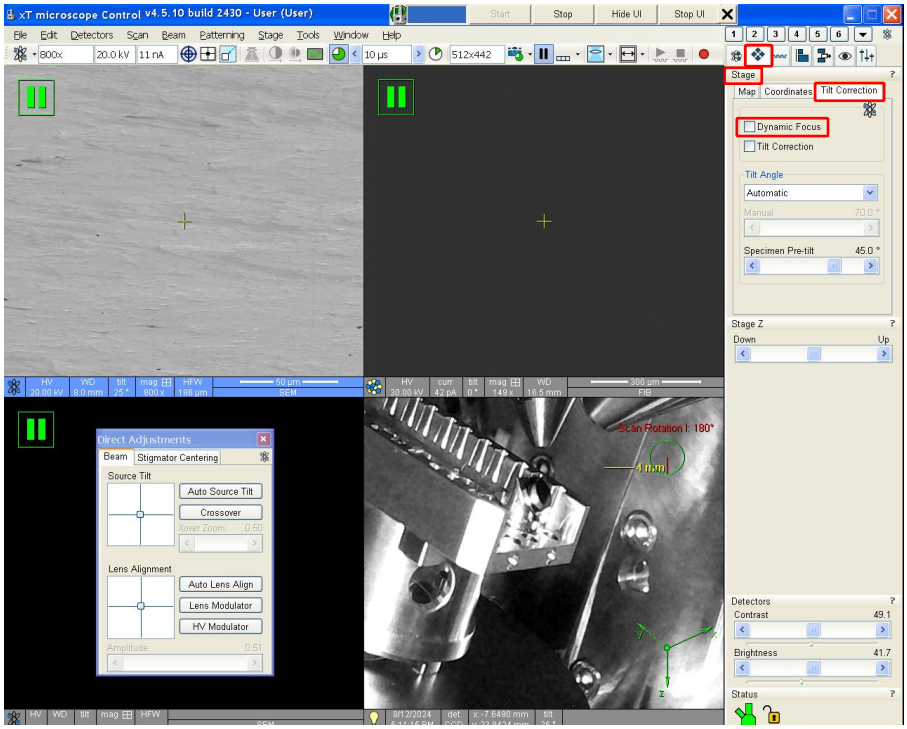
12.3.2. NOTE: it is not necessary to turn off cooling to the EDS detector when finished using it.



12.4. Wait for the slide motor to stop before proceeding; in Microscope Control, enter the Navigation module and navigate to the “Stage” panel.

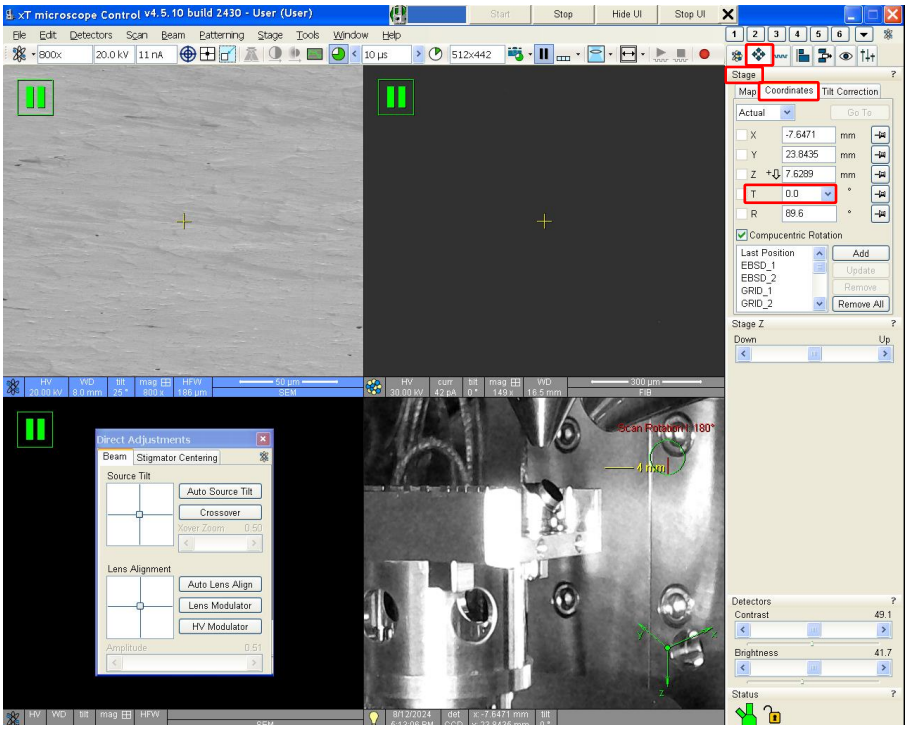
12.4.1. Select the “Tilt Correction” tab.

12.4.2. Uncheck “Dynamic Focus”.



12.4.3. Select the “Coordinates” tab.

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



12.5. Finish the session as per usual: turn off beam, home stage, move stage to load/unload position, vent chamber, remove specimens, return chamber to vacuum.