FEI Talos F200i S/TEM: selected area diffraction using the Ceta camera

Nicholas G. Rudawski
ngr@ufl.edu
(805) 252-4916
(352) 392-3077

Last updated: 03/14/23

ANALYSIS OF RADIOACTIVE SPECIMENS IS STRICTLY PROHIBITED

A user may only perform diffraction work with the Ceta camera after completing hands-on training with RSC staff covering this SOP; this SOP alone is NOT a substitute for hands-on training.

This document assumes the user is already familiar with basic operation of the instrument in TEM mode (particularly, collecting SADPs using the FluCam) and use of Microscope Control.

1. Instrument settings

    1.1. Select voltage, apply alignments, and select and apply the appropriate FEG register for TEM operation.

    1.2. Set spot size as per the following guidelines:

        1.2.1. Polycrystalline samples (no SA aperture), set spot size = 6.
        1.2.2. Single crystal samples (10 µm SA aperture), set spot size = 4.
        1.2.3. Single crystal samples (40 µm SA aperture), set spot size = 7 (shown here).

    1.3. Verify instrument in SA mode with indicated magnification = 14000×.

    1.4. Perform basic instrument alignment (maintain 14000× indicated magnification):

        1.4.1. Set specimen at eucentric height
        1.4.2. 70 µm C2 aperture centering
        1.4.3. Condenser astigmatism correction
        1.4.4. Condenser deflector balancing
        1.4.5. Rotation Centering

    1.5. If needed, tilt the stage to crystallographically align the area of interest as desired (using diffraction mode).

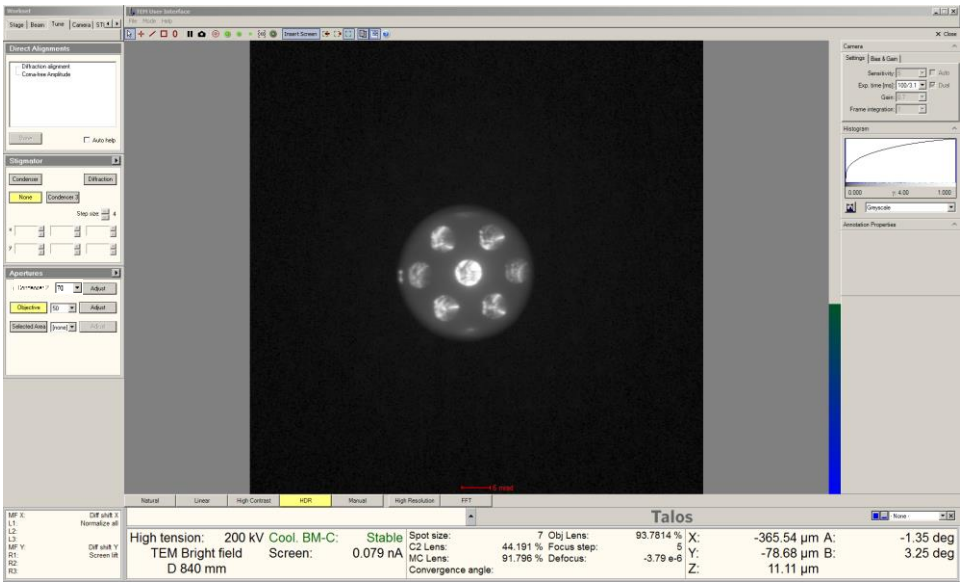
        1.5.1. Remember to re-establish eucentric height after tilting the stage.

    1.6. Verify the objective and SA apertures are both currently retracted.

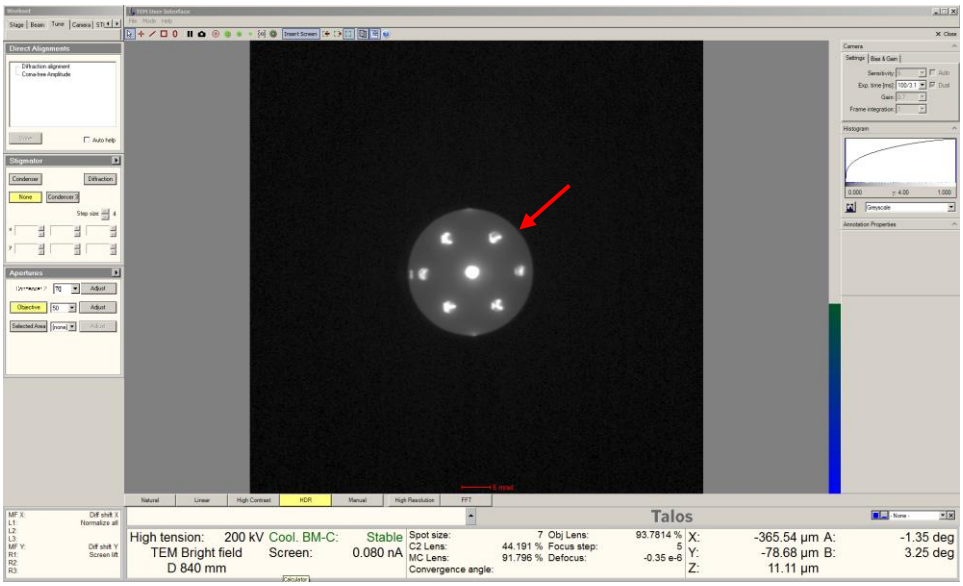
2. Obtaining a parallel beam

2.1. Enter diffraction mode and set the camera length as desired; select and insert the 50, 60, or 70 µm objective aperture.

2.1.1. The objective aperture does not need to be centered; all that matters at this point is that the aperture edges are clearly visible.

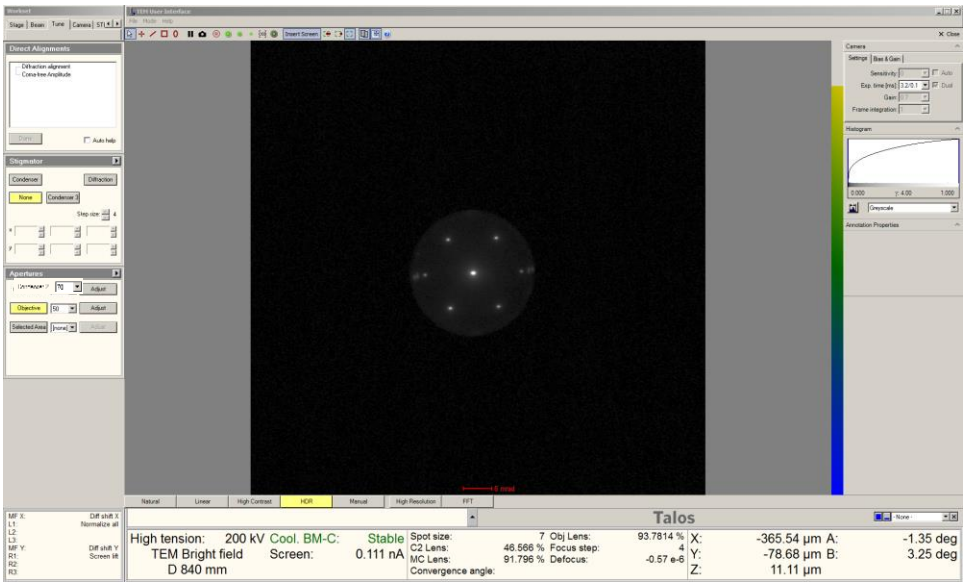


2.2. Use the “Focus” knob to focus the edge of the objective aperture (arrow).

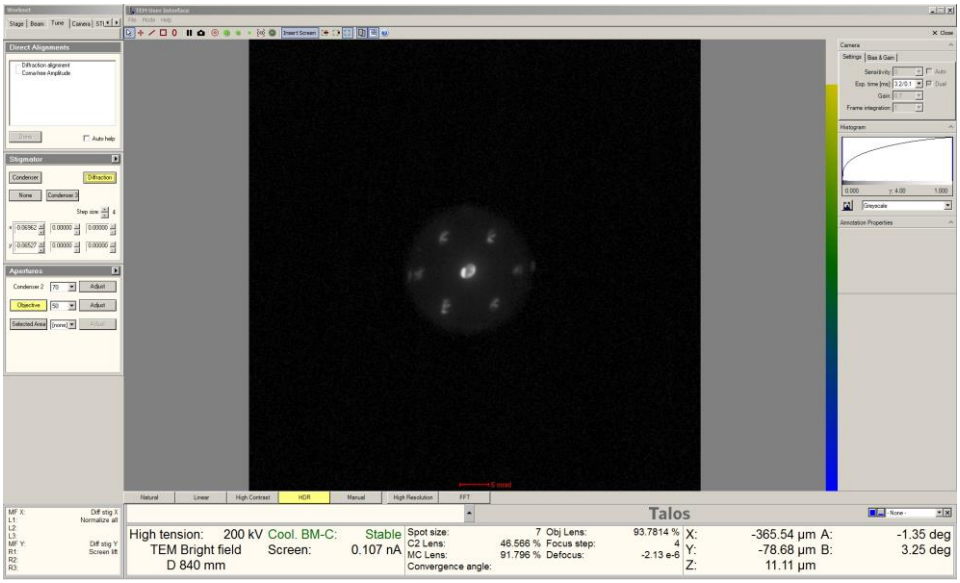


2.3. Use the “Intensity” knob to focus the spots in the diffraction pattern; the incident beam is now parallel.

2.3.1. DO NOT perform any further adjustments to the “Intensity” knob.

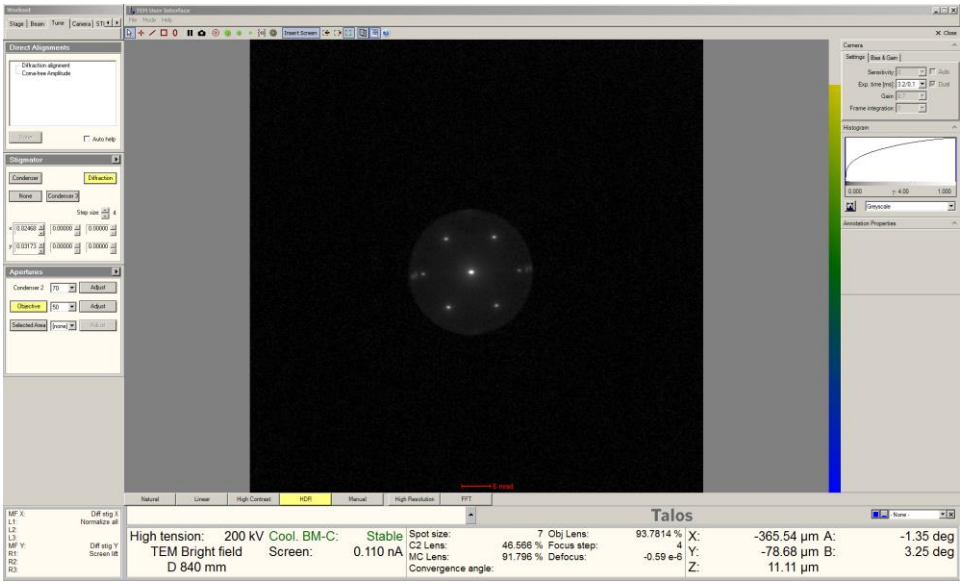


2.4. Use the “Focus” knob to defocus (expand) the spots; if the spots appear astigmatic (shown below), correct using the “Diffraction” stigmators.

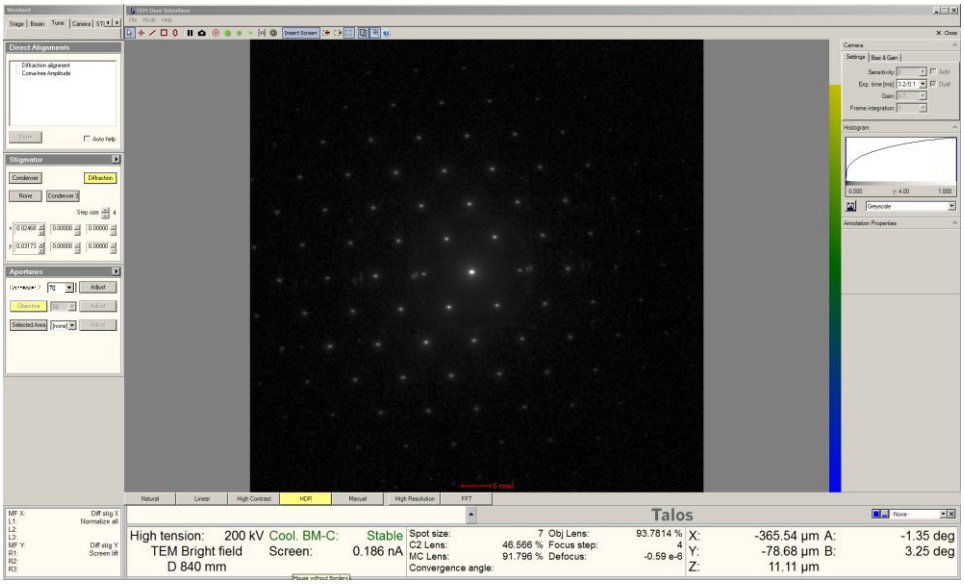


2.5. After correcting astigmatism in the diffraction pattern, refocus the spots with the “Focus” knob.

2.5.1. NOTE: if the objective aperture now appears out of focus, refocus it with the “Focus” knob and then refocus the spots with the “Intensity” knob.



2.6. Retract the objective aperture and then return to SA mode.



3. SA aperture selection/positioning

3.1. Use of the 10 or 40 µm SA aperture is **<u>required</u>** when collecting single crystal DPs to reduce the intensity on the Ceta camera to a safe level.

3.2. An SA aperture is not required when collecting polycrystalline DPs, unless wishing to isolate the DP from a specific region of material.

3.2.1. NOTE: if working with a polycrystalline sample, there is no field-limiting benefit to using an SA aperture larger than 40 µm.

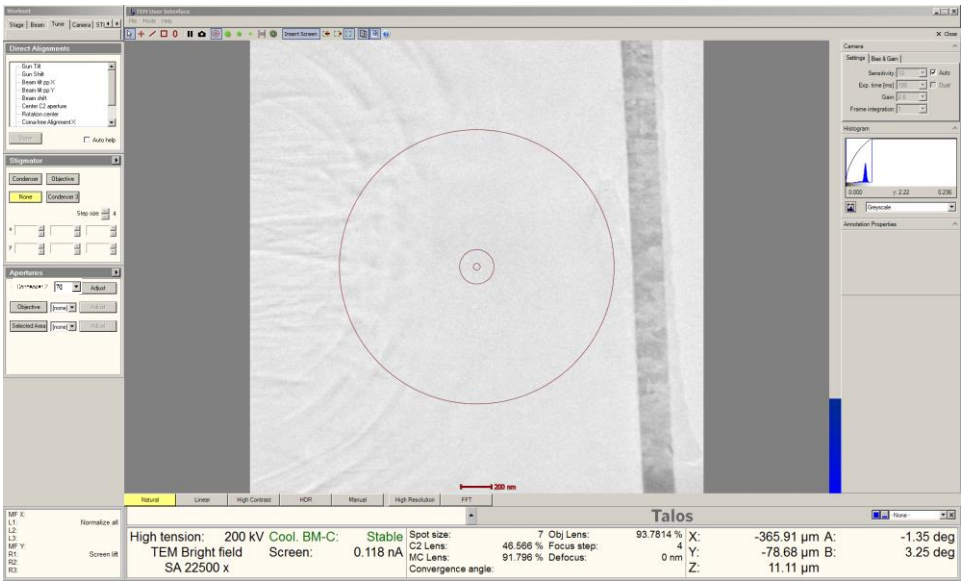
3.2.2. With no SA aperture selected, the size of the incident (parallel) beam defines the “selected” area; at 200 kV using the 70 µm C2 aperture, the beam diameter is ~7 µm.

3.3. Upon returning to SA mode, center the area of interest on the FluCam using the joystick.

3.3.1. If necessary, use the beam shift trackball to re-center the beam, but <u>DO NOT adjust the “Intensity” knob</u> (even if the beam edges are visible inside the FluCam viewable area).

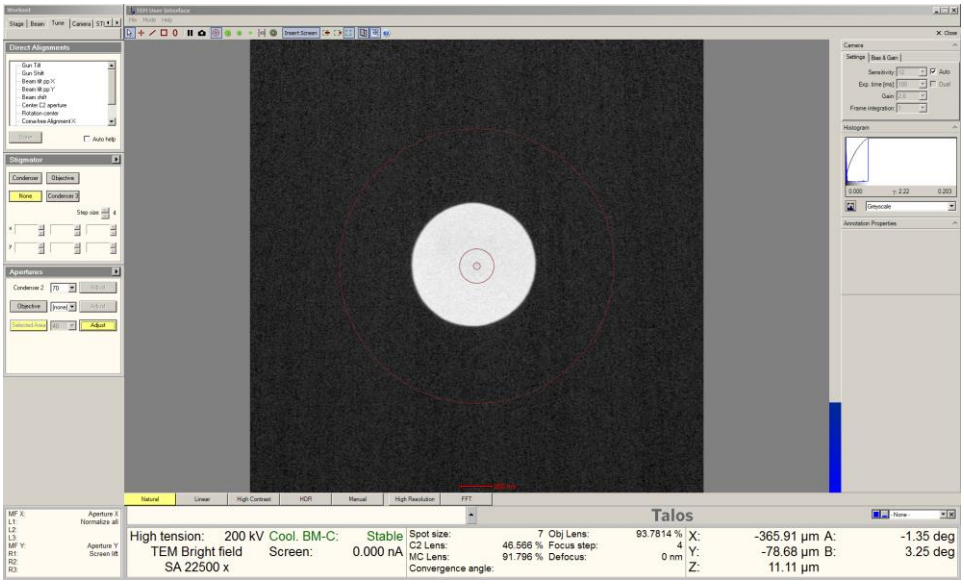
3.3.2. If needed, the indicated magnification may be increased up to 28500×, but <u>DO NOT adjust the “Intensity” knob</u> at any point.

3.3.3. At indicated SA magnifications ≤28500×, the beam will remain well-centered as the indicated magnification is increased provided the beam was well-centered at a lower magnification first.



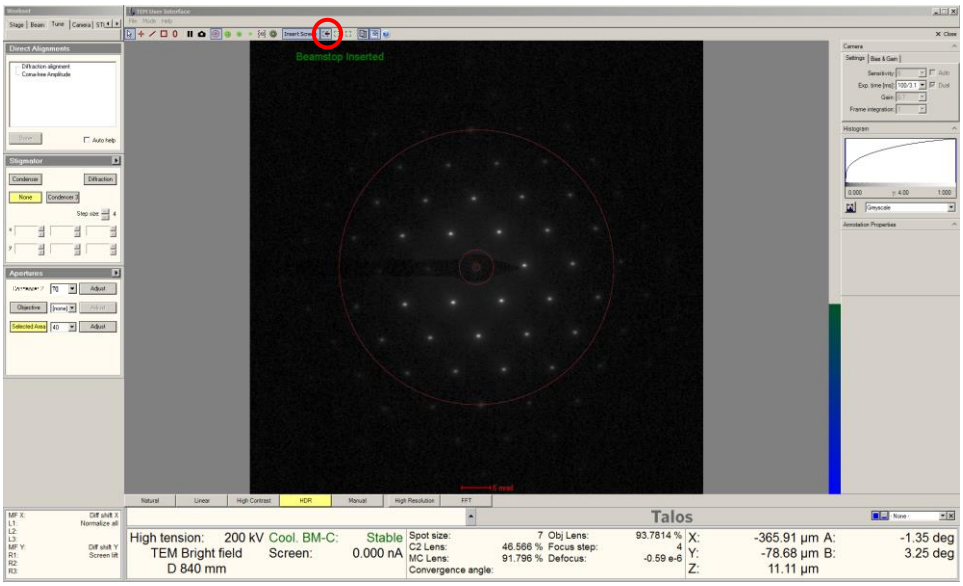
3.4. Select and insert either the 10 or 40 (shown here) µm SA aperture and center it on FluCam; this defines the “selected area” contributing to the DP.

3.4.1. This will result in the SA aperture being centered on the area of interest, since the center of the FluCam was used as the common reference point.



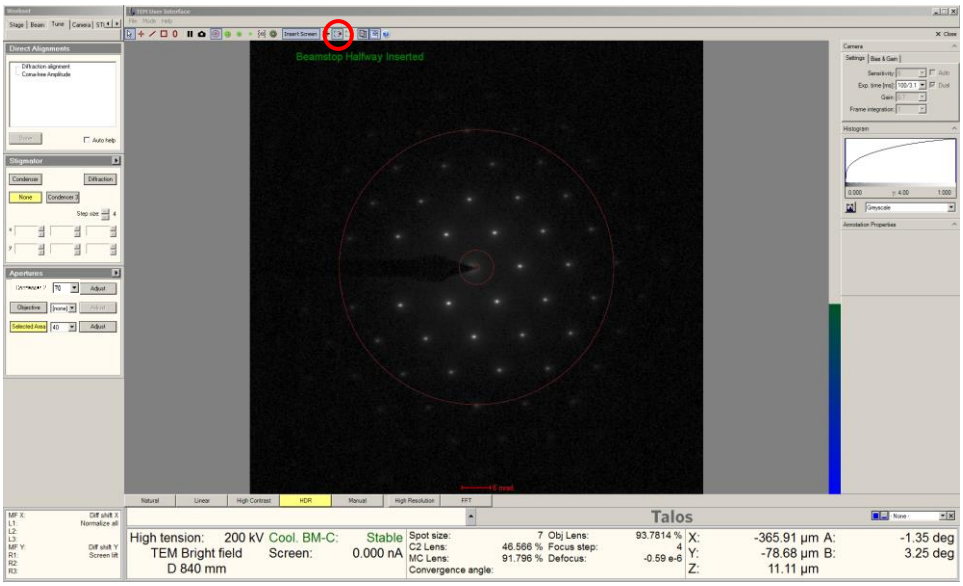
4. Blocking the direct beam

4.1. Enter diffraction mode and set the indicated camera length as needed; then center the direct beam on the FluCam using the “Multifunction” knobs; the beam stop may then be <u>fully inserted</u> to completely block the direct beam.



4.2. Alternatively, the beam stop may be <u>partially inserted</u>, but this will result in the (centered) direct beam still being unblocked.

4.2.1. in this case, the direct beam will need to be shifted slightly to the left using the “Multifunction” knobs, so the direct beam is blocked by the tip of the beam stop (shown here).



5. SADP acquisition with the Ceta camera using Velox

5.1. Verify the SADP as viewed on the FluCam is satisfactory before proceeding; if it is not, adjust the stage tilts (in diffraction mode) and/or the selected area (in SA mode) accordingly.

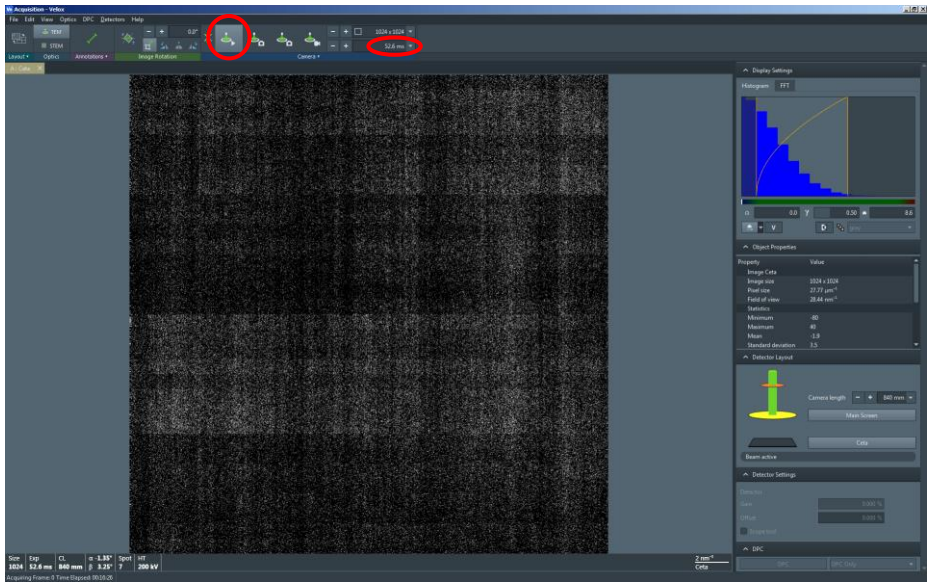
5.1.1. DO NOT perform any of these adjustments while viewing the SADP on the Ceta camera.

5.1.2. Once again, if the stage is tilted, be sure to reestablish eucentric height.

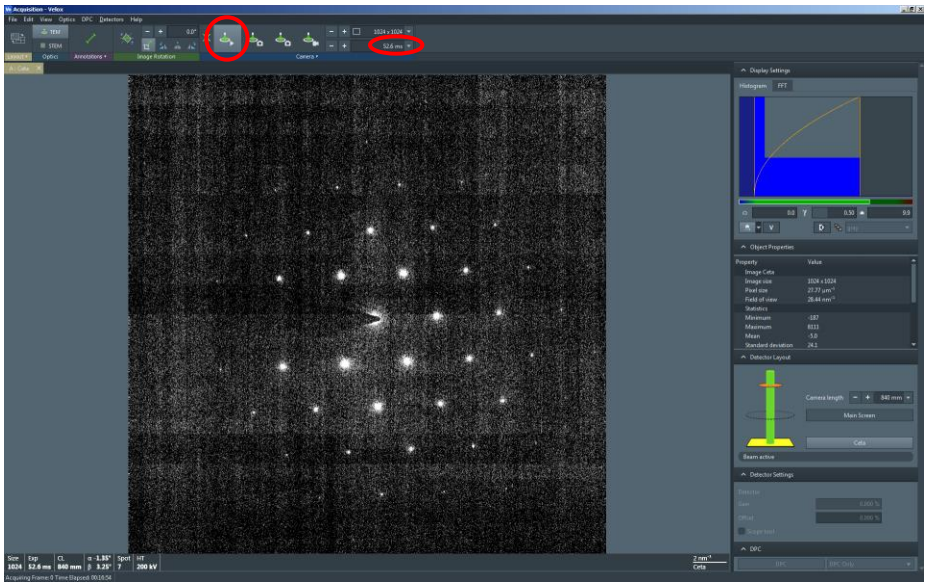
5.1.3. NOTE: if the “Intensity” knob is adjusted (intentionally or accidentally), it will need to be made parallel again before proceeding as per step 2.

5.2. With the viewing screen still inserted, start live imaging in Velox using “Camera View” mode with “**Exposure time” = 52.6 ms**.

5.2.1. This is the shortest possible exposure time and thus the safest for avoiding Ceta camera saturation/damage.



5.3. After starting live imaging with “Exposure time” = 52.6 ms, retract the viewing screen to live image the SADP with the Ceta camera.



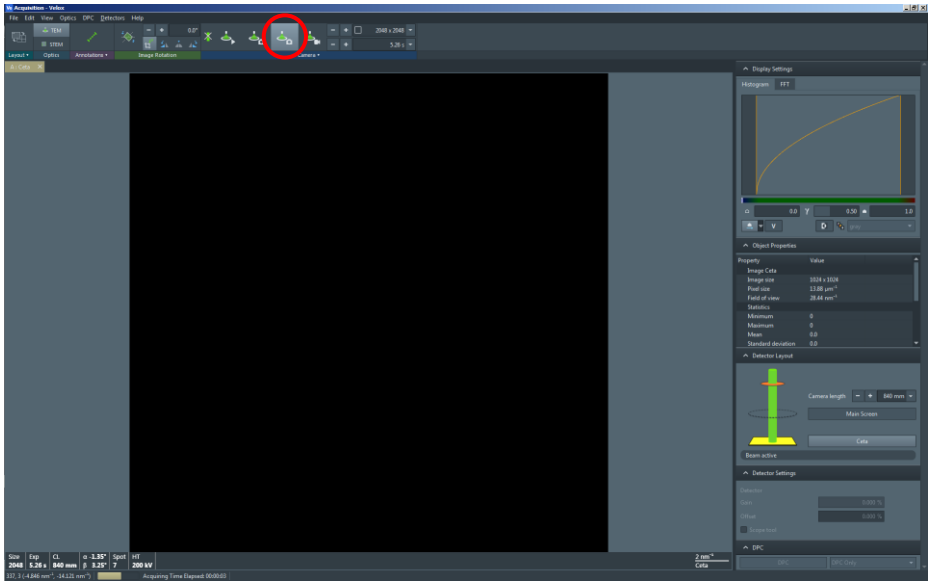
5.4. Select the second “Camera Acquire” option from the toolbar to acquire the final SADP.

5.4.1. DO NOT select the first “Camera Acquire” option from toolbar as this will likely overexpose the Ceta camera.

5.4.2. The settings for the second “Camera Acquire” mode are: “Exposure time” = 5.26 s with “Frame combining” = 100 frames.

5.4.3. NOTE: this is identical to collecting and summing together 100 images each taken with “Exposure Time” = 52.6 ms; this is the longest possible cumulative exposure time using the largest possible number of frames and should produce the most optimal result in the safest possible manner.

5.4.4. DO NOT adjust or deviate from these settings without consulting RSC staff first.



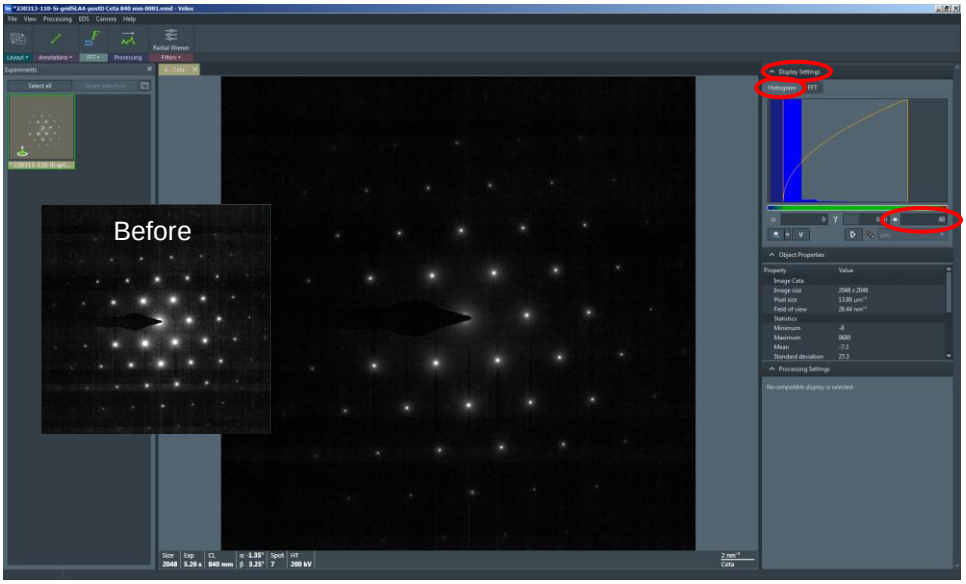
5.4.5. Reinsert the viewing screen **immediately** after SADP acquisition is complete.

5.4.6. When performing diffraction work with the Ceta camera, the goal is always to minimize exposure of the camera.

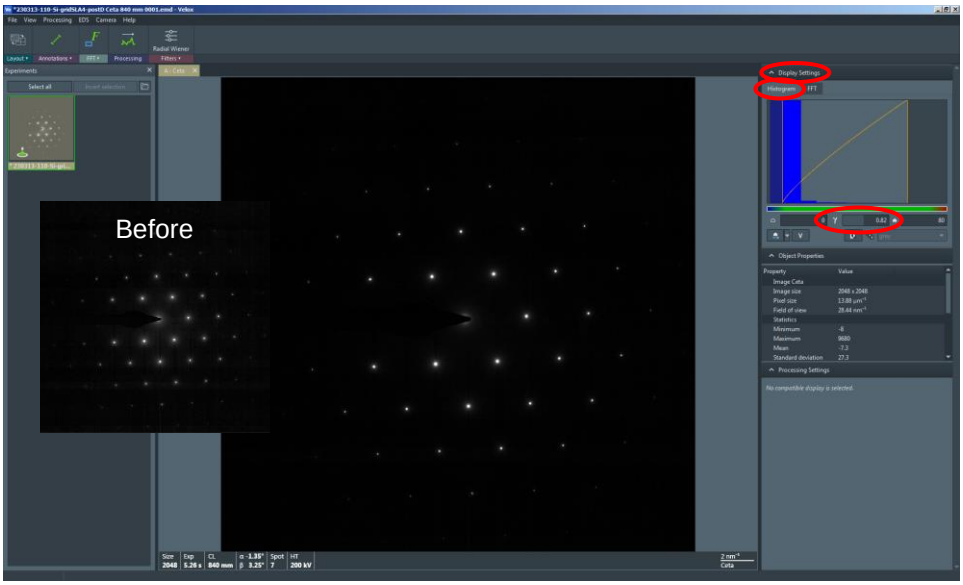
6. SADP digital processing (as needed)

6.1. Navigate to Velox Processing and open the experiment containing the SADP.

6.2. Select the “Display Settings” side panel and then the “Histogram” tab; increase the white level as needed to reduce saturation of the spots.



6.3. Remain in the “Display Settings” side panel and select the “Histogram” tab; increase γ as needed to remove any horizontal/vertical streaking in the pattern.



7. Collecting SADPs at different indicated camera lengths

    7.1. DO NOT change the indicated camera length while observing a live SADP with the Ceta camera in Velox; this should only be done with the viewing screen inserted while using the FluCam.

    7.2. Verify the viewing screen is inserted and the live SADP is displayed on the FluCam.

    7.3. Fully retract the beam stop

    7.4. Set the indicated camera length as needed.

    7.5. No adjustment to the focus of the SADP should be needed; if the SADP was properly focused at the previous indicated camera length, it should still be properly focused at the new indicated camera length.

    7.6. Re-center the direct beam on the FluCam.

    7.7. Reinsert the beam stop as desired; if reinserting partially, again be sure to shift the center of the direct beam under the tip of the beam stop.

    7.8. Retract the viewing screen; the SADP can now be collected using Velox as described previously; the default acquisition settings should be sufficient for SADP collection at any indicated camera length.

8. Rotation calibration (applies equally for 80 or 200 kV operation)

    8.1. If the SADP was acquired with an indicated camera length <98 mm, no rotation is needed to obtain proper alignment with the SA mode image.

    8.2. If the SADP was acquired with an indicated camera length ≥98 mm, rotate the pattern 180° to obtain proper alignment with the SA mode image.

9. Scale bar calibration for (applies equally for 80 or 200 kV operation)

    9.1. For SADPs acquired with the Ceta camera, the scale bars automatically generated by Velox will be correct as indicated and allow d spacing measurements to better than ±0.1 Å precision.

        9.1.1. This assumes the specimen is correctly at eucentric height and the DP is properly focused as specified previously.

        9.1.2. If d spacing measurements more precise than ±0.1 Å are needed, XRD should be used.

10. When finished collecting SADPs

    10.1. Verify the viewing screen is inserted.

    10.2. Retract the Ceta camera.

    10.3. Fully retract the beam stop.

    10.4. Place the instrument in SA mode.

        10.4.1. DO NOT leave in diffraction mode.

    10.5. Retract the SA aperture.