FEI Talos F200i S/TEM: weak-beam dark-field operation

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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 in TEM mode, use of Microscope Control, and is working with a single-crystal specimen.

1. The double tilt holder must be used if weak-beam dark-field (WBDF) imaging is to be performed. DO NOT attempt to use this holder without being trained to properly load and unload it. Also, note the following two requirements if you intend to image a self-supported specimen (e.g. a 3 mm disc of cored-out material):

    1.1. The thickest part of specimen must not exceed 50 μm; otherwise, it will not be possible to safely secure the specimen in the holder.

    1.2. If the hex ring cannot be properly secured in the basket with both the washer and sample loaded, the washer should be removed. Do not insert the holder into the column without the hex ring being properly secured.

    1.3. The specimen should be loaded into the holder such that the electron transparent portion (or side) is at the lowest possible position in the basket.

2. Instrument settings

2.1. WBDF imaging may be performed at 80 or 200 kV; select the desired voltage and then apply the corresponding alignment file and FEG register; in the case of 200 kV operation, be sure to apply the FEG register for TEM mode operation.

2.2. Set the spot size = 1; this is necessary due to the inherently weak (hence the name) signal in WBDF imaging.

2.3. Find a region of interest and bring it to eucentric height; when performing WBDF imaging, the region of interest should be thick enough to produce Kikuchi diffraction, but not so thick to distort/obscure image details.

2.4. Perform the basic alignment of the instrument: for 80 (200) kV operation, set the indicated magnification to 36000× (14000×) and select/center the C2 aperture, correct the condenser astigmatism, balance the deflector coils, and perform rotation centering.

2.4.1. NOTE: recommended C2 apertures for WBDF imaging at 80 and 200 kV are 150 and 100 µm, respectively.

2.4.2. For 200 kV operation with spot size = 1 using the 100 µm C2 aperture, this will result in ~12 nA of beam current; do not attempt high-resolution and/or bright-field imaging with this much current; it will likely damage your sample as well as the Ceta camera.

2.5. Make sure you are in SA mode; for 80 (200) kV operation, set the indicated magnification to 36000× (14000×); these are the minimum recommended magnifications for performing WBDF imaging.

2.6. Use the “Intensity” knob to bring the beam to crossover and then center it on the FluCam using the beam shift trackball; turn the “Intensity” knob *clockwise* to expand the beam from crossover so it is slightly larger than the field of view (no edges of the beam are viewable on the FluCam).

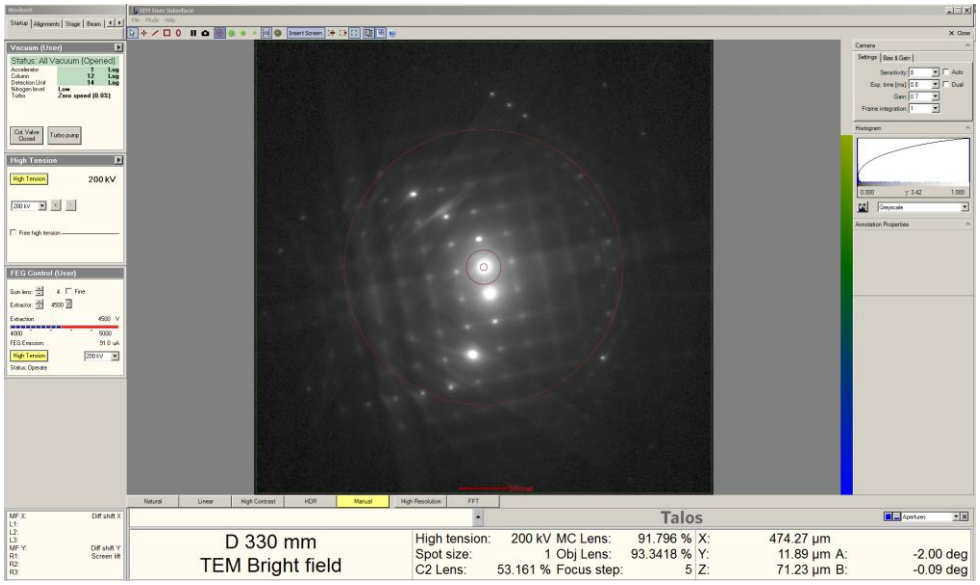
3. Setting up the 2-beam condition

3.1. Enter diffraction mode; use the “Magnification” knob (right-hand control pad) to adjust the camera length as desired for observing the DP.

3.2. Insert the 100 µ objective aperture into the DP; use the “Focus” knob to focus the objective aperture edge so it is sharp; when finished, retract the objective aperture.

3.3. Use the “Intensity” knob to focus the spots in the DP (make as small as possible); the incident beam is now parallel; use the “Multifunction” knobs to center the direct beam on the FluCam.

3.4. Adjust α and β tilt (left-hand control pad) to set up the desired 2-beam condition, with s ~ 0. If the DP suddenly disappears during tilting, the specimen is probably no longer under the beam; return to TEM mode and center the specimen and then return to diffraction mode and resume adjusting the specimen orientation



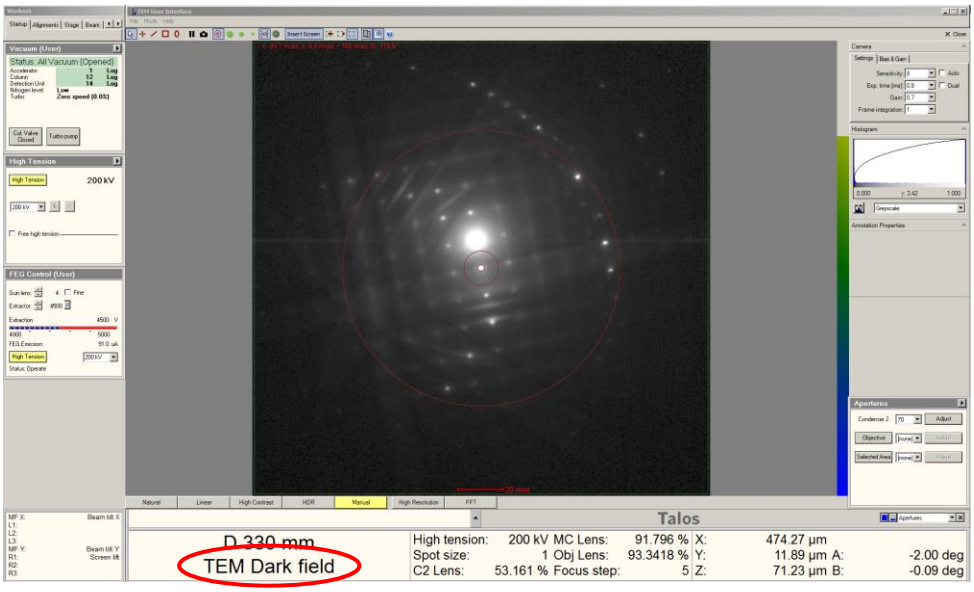
3.5. Return to TEM mode; re-center the area of interest on the FluCam and re-establish eucentric height (this should be done *every* time after the specimen is tilted).

3.6. The area of interest may move when tilted; return to diffraction mode and verify the 2-beam condition is as desired at the area of interest. If the orientation is not as desired, repeat the previous two steps iteratively until the desired orientation is obtained (if the amount of tilting performed is <2°, 1 or 2 iterations will likely be sufficient).

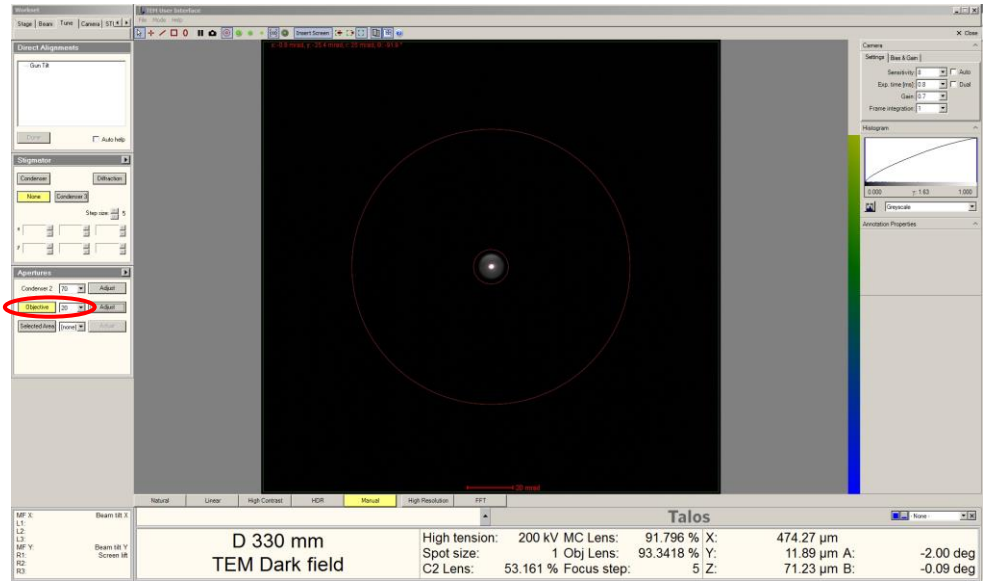
4. Setting up the WBDF condition

4.1. Enter diffraction mode; use the “Multifunction” knobs to center the direct beam on the FluCam.

4.2. Select the “Dark-field” button (right-hand control pad) to activate dark-field mode (note the bottom information panel will now indicate the system is in dark-field mode); use the “Multifunction” knobs to tilt the incident beam to move the Bragg spot to the original position of the direct spot (center of the FluCam).



4.3. Insert the 20 µm objective aperture and center it on the Bragg beam (though any objective aperture that is small enough to allow only the Bragg beam to pass through it will work).



4.4. Return to TEM mode; set the indicated magnification as needed; for 80 (200) kV operation, an indicated magnification range of 36000 – 74000× (14000 – 46000×) is usually sufficient.

4.5. Turn the “Intensity” knob *counterclockwise* until the edge of the beam is visible; then center the beam on the FluCam using the beam shift trackball.

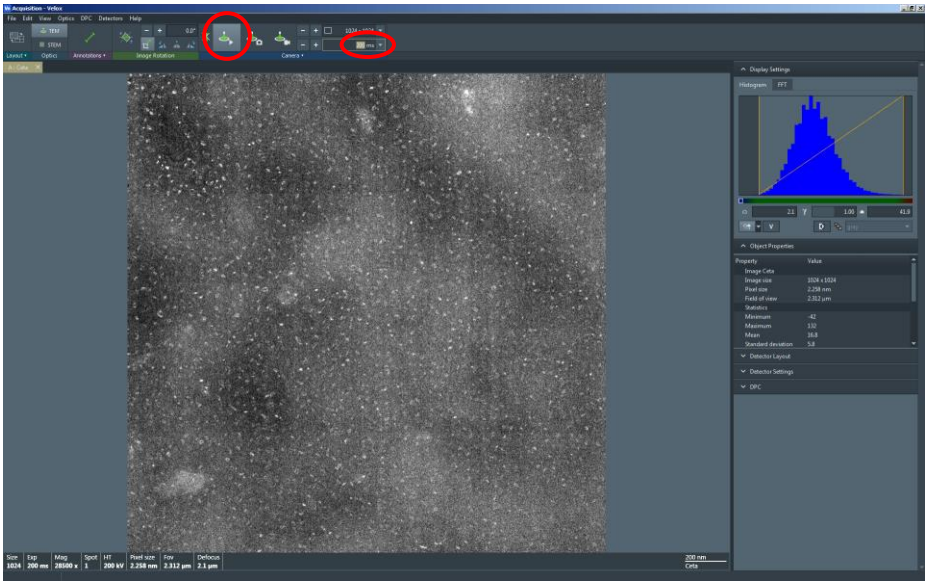
4.6. Turn the “Intensity” knob *clockwise* to expand the beam to slightly beyond the viewable area on the FluCam.

4.7. Precise focusing of the WBDF image is best accomplished using the live Ceta camera image in Velox (described subsequently).

5. WBDF imaging in Velox

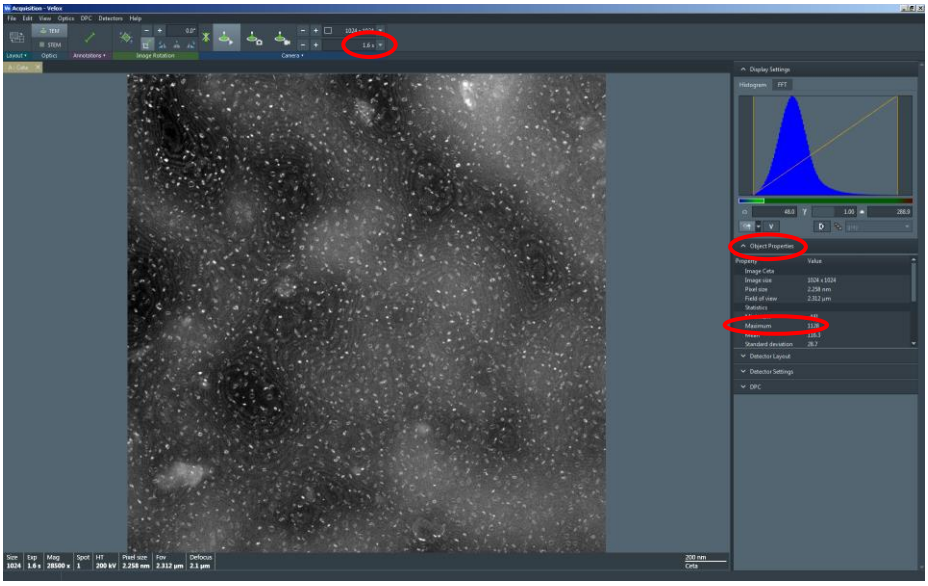
5.1. Retract the viewing screen and start acquiring a live Ceta camera image in Velox using “View” mode.

5.1.1. In the Velox toolbar, set the initial exposure time = 200 ms.



5.2. Adjust exposure time until signal to noise is sufficient; use the “Focus” knob to precisely focus the image (the response time may be slow).

5.3. Select the “Object Properties” side panel and note the “Maximum” counts value; the maximum counts should not exceed ~8000 to avoid damage to the Ceta camera.

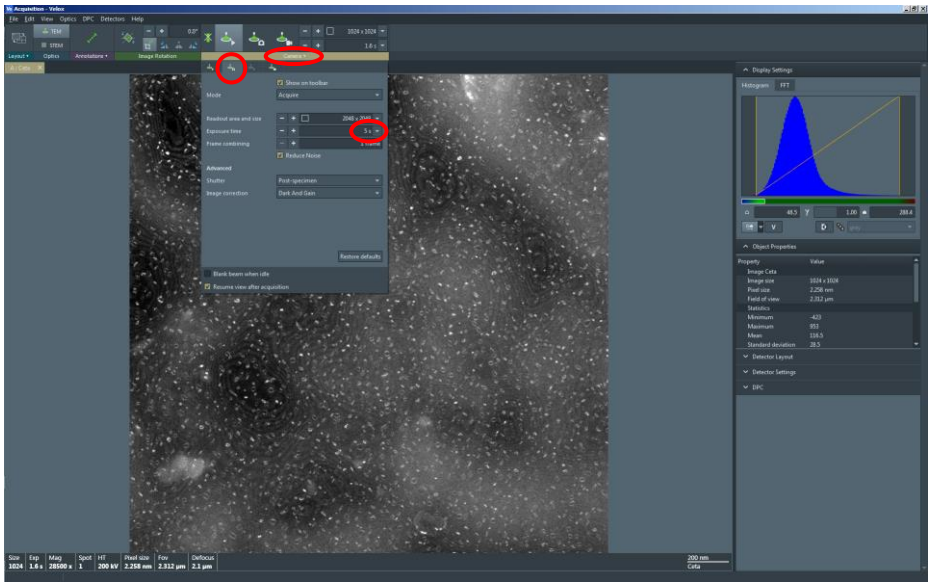


5.4. When ready to acquire the final WBDF image, select “Camera” from the Velox toolbar.

5.4.1. Select the tab for “Acquire” mode and input an appropriate value for “Exposure time”; it is common to use exposure times of several seconds for WBDF images.

5.4.2. NOTE: you can usually safely use up to 4× the “View” mode “Exposure time” for the “Acquire” mode image.

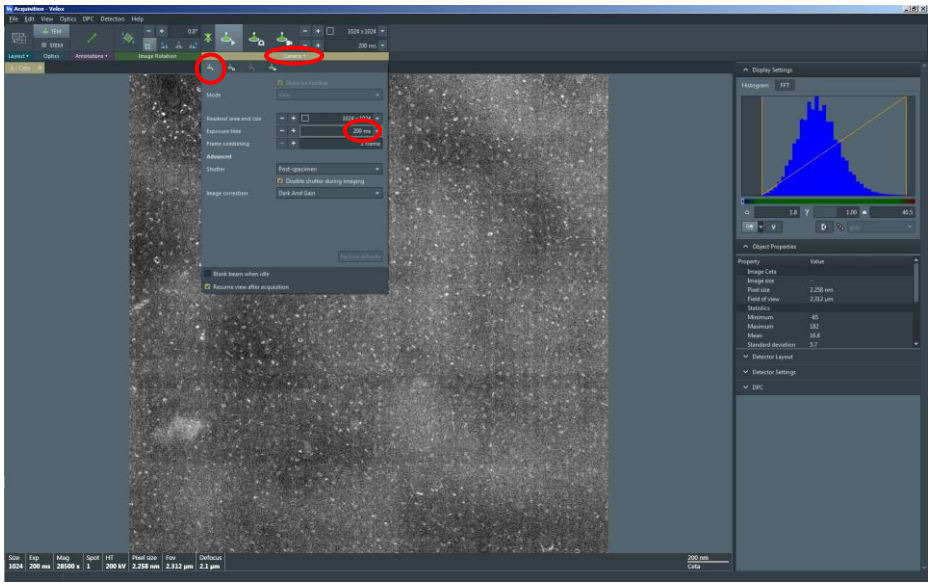
5.4.3. <u>The maximum counts in the final acquired image should not exceed ~8000 to avoid damage to the Ceta camera</u>.



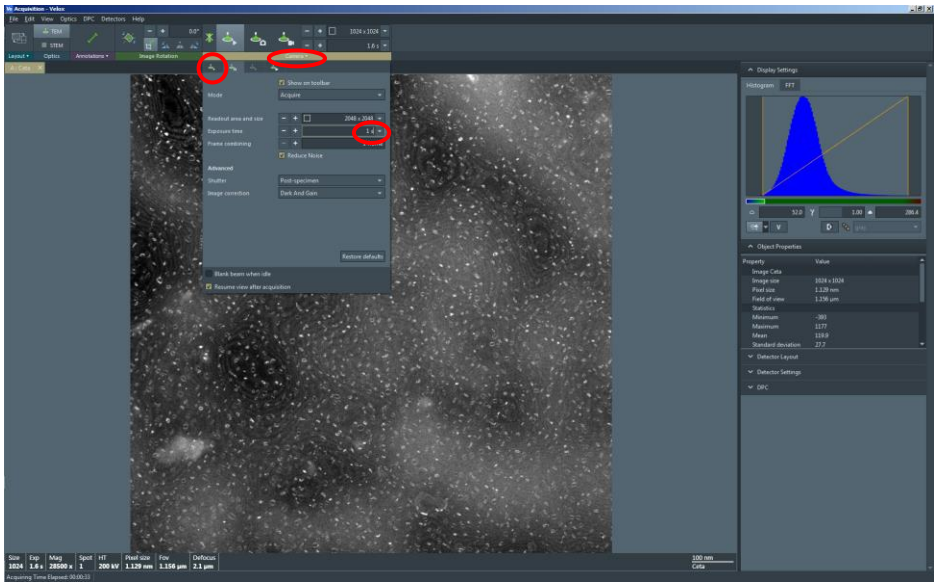
6. When finished WBDF imaging

6.1. In Velox, the “View” and “Acquire” mode exposure times should be returned to the default values.

6.2. Select “Camera” from the toolbar and then the “View” mode tab; set “Exposure time” = 200 ms.

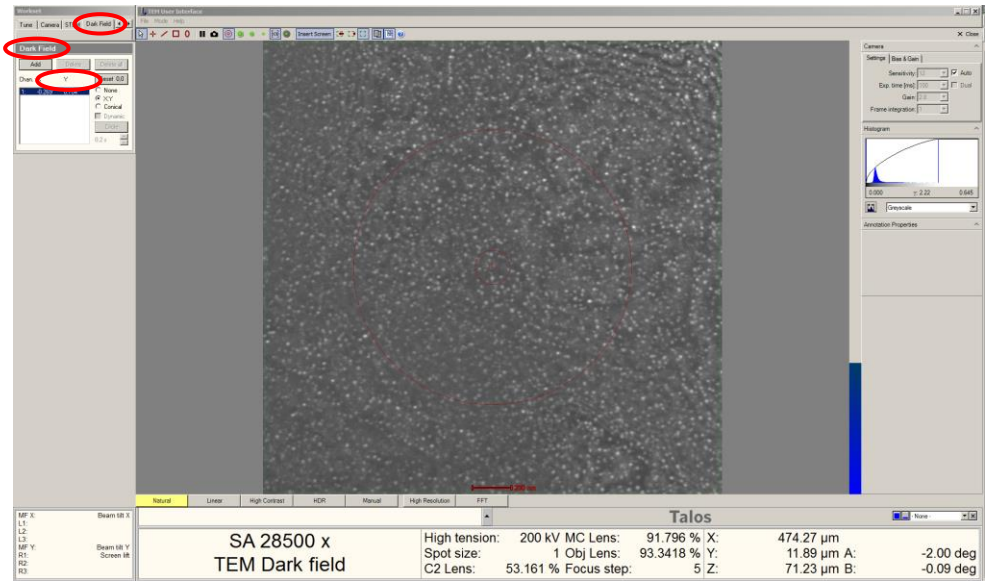


6.3. Select “Camera” from the toolbar and then the “Acquire” mode tab; set “Exposure time” = 1 s.



6.4. In Microscope Control, select the “Dark-Field” tab and navigate to the “Dark-Field” control panel.

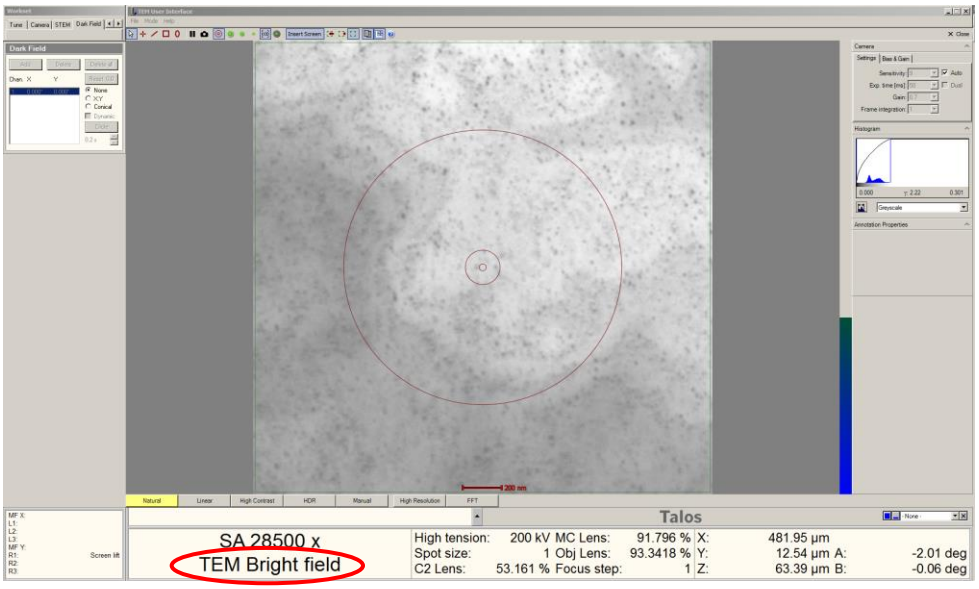
6.4.1. Select “Reset 0,0” to reset the beam tilt.



6.5. Select the “Dark-field” button (right hand control pad) to turn off dark-field operation.

6.5.1. Check the bottom information panel to verify the system is in “Bright-field” mode.

6.5.2. <u>DO NOT leave the instrument in “Dark-field” mode when finishing the session.</u>



6.6. If finished using the microscope, reinsert the 70 µm C2 aperture and then finish the session normally: close column valves, retract objective aperture, reset holder, retract double-tilt holder from column, unload sample from double-tilt holder, and reinsert single tilt holder into column.

6.7. If additional conventional mode (high-resolution, bright-field) imaging is to be performed, reinsert the 70 µm C2 aperture, set spot size = 3 or 4, and perform full instrument alignment.