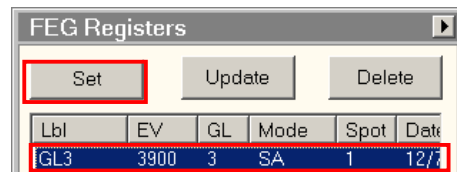


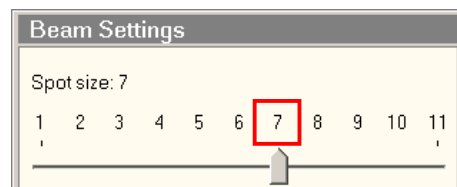
FEI Tecnai F20 S/TEM: acquiring diffraction patterns
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This document assumes the user is already familiar with basic operation of the instrument in TEM mode and use of the digital camera.

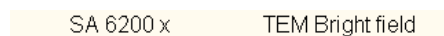
1. Find a region of interest and bring it to eucentric height.
2. Instrument settings
 - 2.1. Select and apply the “GL3” FEG register (unless done previously during the session).



- 2.2. In Microscope Control, select the **Beam** tab; navigate to the “Beam Settings” control panel and select spot size = 7.



- 2.3. Center the C2 aperture and correct the condenser astigmatism; no other alignments need to be performed.
 - 2.4. Set magnification to SA 6200x; you must be in SA mode if you need to use an SA aperture.



- 2.5. Expand the beam clockwise from crossover and center it on viewing screen

3. Setting up the DP

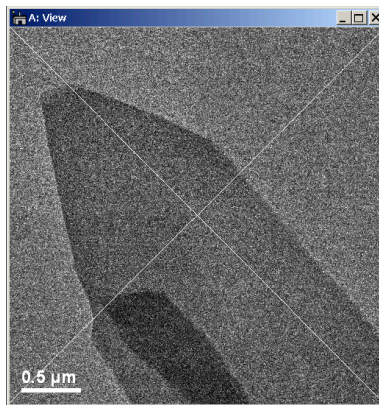
- 3.1. Select “Diffraction” (right-hand control pad) to enter diffraction mode.
- 3.2. Use the “Magnification” knob (right-hand control pad) to adjust the camera length as desired for observing the DP.

D 300 mm TEM Bright field

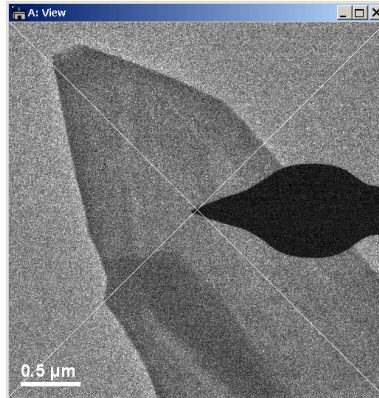
- 3.3. Insert an objective aperture into the DP (which one doesn't matter, as long as you can see it); use the “Focus” knob (right-hand control pad) to focus the objective aperture so the edge is sharp; when finished, retract the objective aperture
- 3.4. Use the “Intensity” knob (left-hand control pad) to focus the direct spot (make as small as possible) in the DP.
- 3.5. Select “Diffraction” to return to TEM mode; *do not* adjust the “Intensity” knob after you return to TEM mode.

4. Centering the beam stop

- 4.1. Start live imaging with the digital camera (insert the digital camera, flip the viewing screen up, etc.) with an integration time = 0.01 s; move the region of the specimen you wish to take the DP from to the center of the image; then draw 2 diagonal lines in the live image to mark the center of the image.

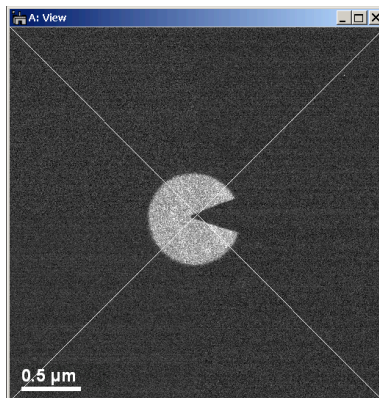


- 4.2. Insert the beam stop into the image and then center it (this will ensure your acquired diffraction patterns are always centered when collected); if you wish, you can acquire an image to have for reference purposes (the lines will not appear in the acquired image).



5. Inserting the SA aperture (if needed)

- 5.1. If your specimen is coarsely grained or single-crystal, then you must use an SA aperture when acquiring your DPs to prevent damage to the camera. However, if your specimen consists of very small particles or is very finely grained, an SA aperture may not be necessary. If you are unsure as to whether you should use an SA aperture or not when acquiring DPs from your specific samples, please consult with RSC staff *before* attempting to collect any DPs.
- 5.2. If an SA aperture is needed, insert and center the #2 or #1 SA aperture over the area of interest (the beam stop, region of interest, and SA aperture should all now be approximately centered); acquire another image (again, for reference purposes, so you know where in the image the DP originated).



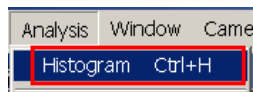
- 5.3. Flip the viewing screen back down when finished, but continue to leave the camera inserted and acquiring a live image.

6. Centering the DP

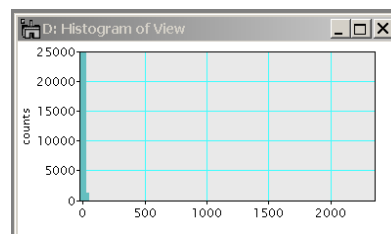
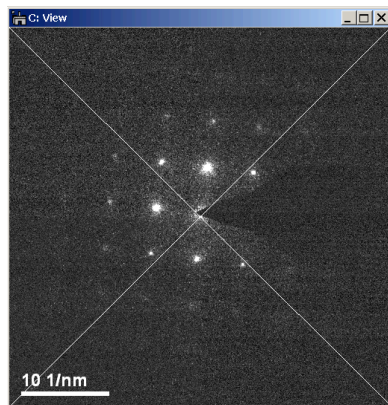
- 6.1. Select “Diffraction” to return to diffraction mode; use the “Focus” knob to focus the direct spot (if necessary); do not adjust the “Intensity” knob.
- 6.2. Use the “Multifunction” knobs to move the direct spot so it is under the tip of the beam stop (the knobs will automatically be set to shift the DP when diffraction mode is entered)

7. Acquiring the DP

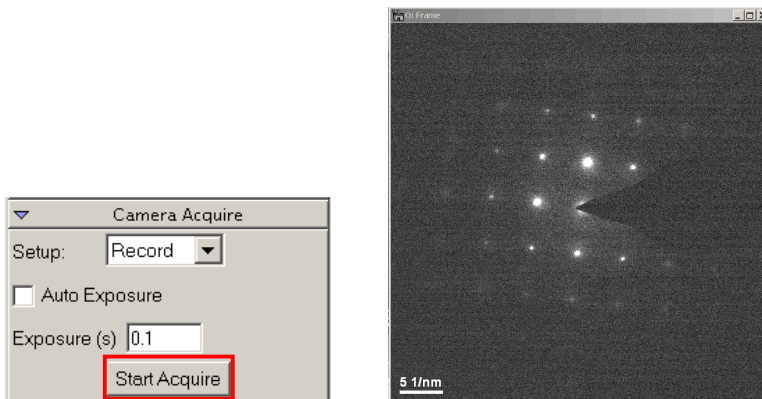
- 7.1. In DigitalMicrograph, select “Analysis” from the pull-down menu and then “Histogram” to view a histogram of the live image.



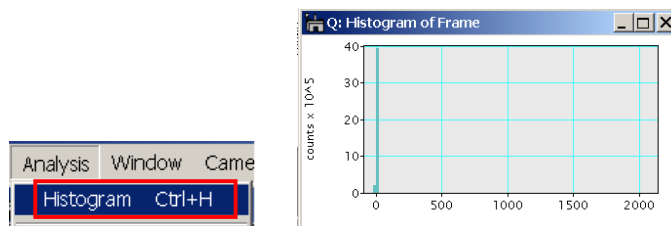
- 7.2. Flip the viewing screen up to start collecting a live image of the DP (still using a 0.01 s exposure time); note the right limit of the x-axis of the histogram, which indicates the maximum counts present in the DP. This number should not exceed 2000 – 3000 counts; do not proceed if the maximum counts in the live DP are higher than this range.



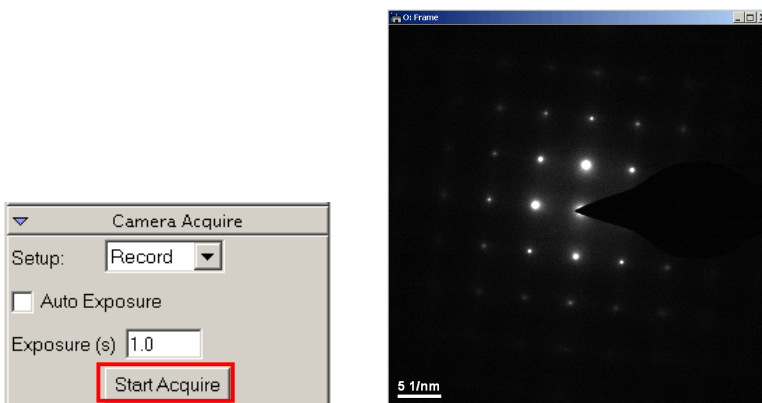
- 7.3. In the “Camera Acquire” tool, input a value for “Exposure (s)” of 0.1 s; select “Start Acquire” to acquire a test exposure of the DP (it should be very noisy).



- 7.4. In DigitalMicrograph, select “Analysis” from the pull-down menu and then “Histogram” to view a histogram of the test exposure; again, noting the right limit of the x-axis, which indicates the maximum counts present in the test exposure.

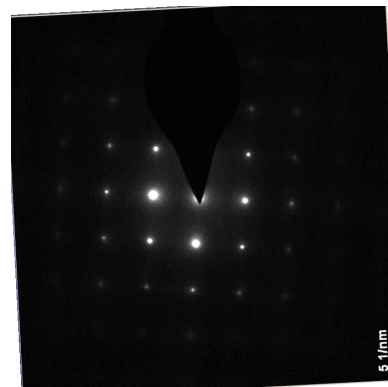
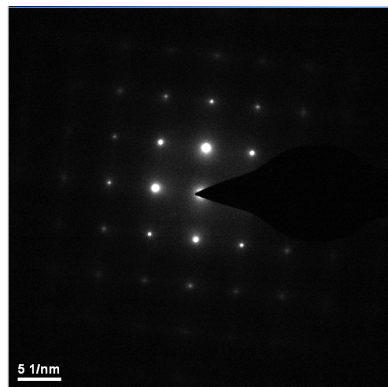


- 7.5. Based on the maximum counts in the test exposure, determine a suitable value for “Exposure (s)” such that the maximum counts in the acquired DP will be in range of 10000 – 20000 counts. For example, if the maximum counts in the test exposure is ~2000 counts, then a value for “Exposure (s)” of 1.0 s will be suitable; select “Start Acquire” to acquire the DP.



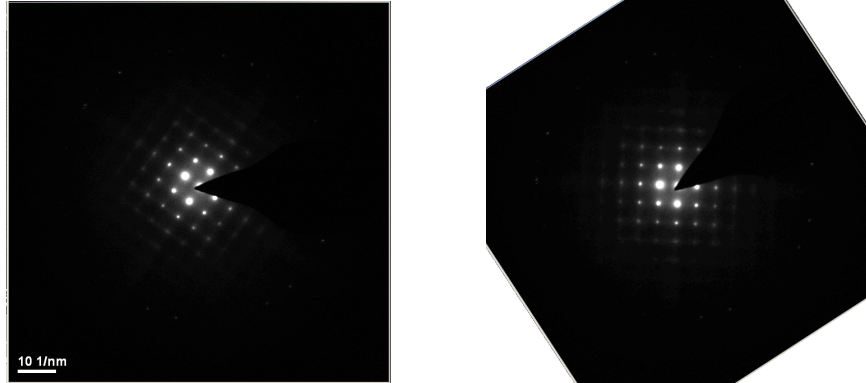
- 7.6. Flip the viewing screen back down after acquisition is finished.

- 7.7. If you wish to acquire the DP at a different camera length, make sure the viewing screen is flipped down, adjust the “Magnification” knob to change the camera length accordingly, then use the focus knob to focus the direct spot and then use the diffraction alignment to move the direct spot under the beam stop.
 - 7.8. If DP acquisition from a different region is needed, select “Diffraction” to return to TEM mode, retract the SA aperture, center the new area of interest on the screen, insert the SA aperture again, and repeat the procedure again.
8. When finished recording DPs
 - 8.1. Make sure the instrument is left in TEM mode, the SA aperture is retracted, the viewing screen is flipped down, and the digital camera is retracted.
 9. Rotation calibration
 - 9.1. If the indicated camera length is >135 mm (with the viewing screen flipped down), then the DP needs to be rotated counterclockwise 93° to establish the proper DP-image orientation (assuming the TEM image was collected in SA mode).



Left: as collected; right: after counterclockwise rotation of 93°

- 9.2. If the indicated camera length is ≤ 135 mm (with the viewing screen flipped *down*), then the DP needs to be rotated counterclockwise 33° to establish the proper DP-image orientation (assuming the TEM image was collected in SA mode).



Left: as collected; right: after counterclockwise rotation of 33°

Appendix: shorthand procedure for acquiring diffraction patterns

1. Set area of interest at eucentric height
2. Set spot size = 7
3. Perform C2 aperture alignment and correct condenser astigmatism
4. Set magnification to SA 6200 \times
5. Expand beam clockwise from crossover
6. Center beam on viewing screen
7. Enter diffraction mode
8. Set camera length as needed
9. Insert an objective aperture into the DP
10. Use "Focus" to focus edge of objective aperture
11. Retract objective aperture
12. Focus direct spot with "Intensity"
13. Return to TEM mode
14. Collect live image with digital camera with 0.01 s exposure time
15. Mark center of image with 2 diagonal lines
16. Move area of interest to center of image
17. Insert beam stop and position on center of image
18. Insert SA aperture and position on center of image
19. Flip viewing screen back down
20. Enter diffraction mode
21. Use "Focus" to focus direct spot (if needed)
22. Use "Multifunction" knobs to center direct spot under beam stop tip
23. Collect histogram of live image
24. Flip viewing screen up, verify max counts in histogram within limits
25. Acquire DP with 0.1 s exposure time (test exposure)
26. Use histogram to check max counts in test exposure
27. Adjust exposure time to generate appropriate counts and acquire DP
28. Flip viewing screen back down after DP acquisition