

Acquiring SAED patterns of single crystals

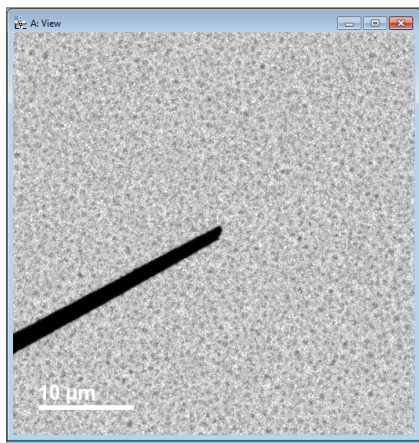
Nicholas G. Rudawski

ngr@ufl.edu

(805) 252-4916

Last updated: 09/16/15

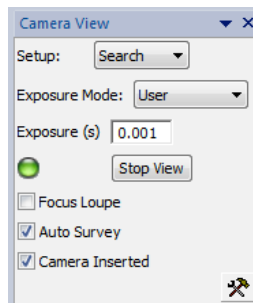
1. Perform normal alignment of the instrument in “MAG1” mode using the usual method with “ α -SELECTOR” = 3. If needed, use “DIFF” mode to orient the specimen as desired (like along a particular zone axis, if the specimen is a single crystal). As always, make sure the specimen is returned to eucentric height after tilting.
2. Set the magnification to 10 k \times (DO NOT go lower in magnification or the objective mini lens turns on).
3. Turn on the FLC (R1 panel) and set the step size to 100; flick the COND1 switch in the FLC to the right until the maximum excitation of the C1 lens is reached. DO NOT TURN OFF THE FLC.
4. Perform gun tilt alignment: activate “ANODE WOBB” (L1 panel), select “GUN” under “DEFLECTOR” (R2 panel), use “DEF” (R2 panel) so the beam expands and contracts evenly.
5. Correct condenser lens astigmatism: activate “COND STIG” (L1 panel) and use “DEF X” and “DEF Y” (L1 and R1 panels) to stigmatize the beam so it is circular.
6. Return magnification back to 10 k \times (if necessary), expand beam clockwise from crossover so it is approximately the size of the small screen and center on the screen, and perform voltage or current centering.
7. While still in “MAG1” mode, start viewing a live image of the specimen in Digital Micrograph; insert the beam stop into the image so the tip is centered in the live image; flip the viewing screen back down when finished.



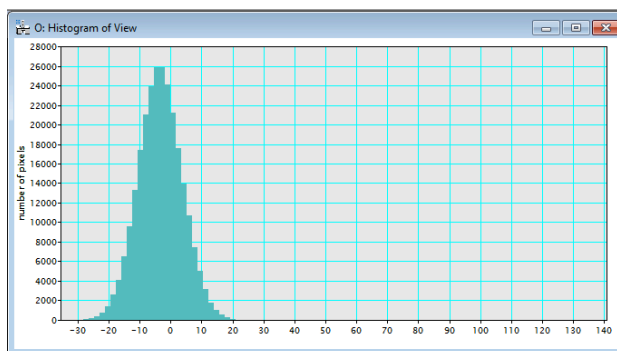
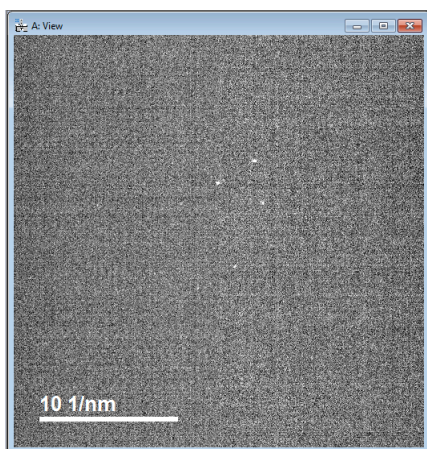
8. Enter “DIFF” mode and adjust the camera length to the desired setting (typically, L = 15 – 25 cm will be sufficient for most applications).
9. Insert the objective aperture into the DP and focus the aperture edge using “DIFF FOCUS”; then focus the spots (and/or rings) in the DP using “BRIGHTNESS”.
10. Return to “MAG1” mode, center the beam on the viewing screen (DO NOT adjust “BRIGHTNESS”), and move the area of interest for DP collection to the center of the screen.
11. Insert SA aperture 3 or 4 (the two smallest) and center it on the viewing screen.
12. Return to “DIFF” mode and focus the DP using “DIFF FOCUS”; under “DEFLECTOR” (R2 panel), activate “PROJ” and use “SHIFT” (R2 panel) to shift the DP so the direct beam is under the tip of the beam stop.

BEFORE PROCEEDING, MAKE SURE THE DIRECT BEAM IS COVERED BY THE TIP OF THE BEAM STOP.

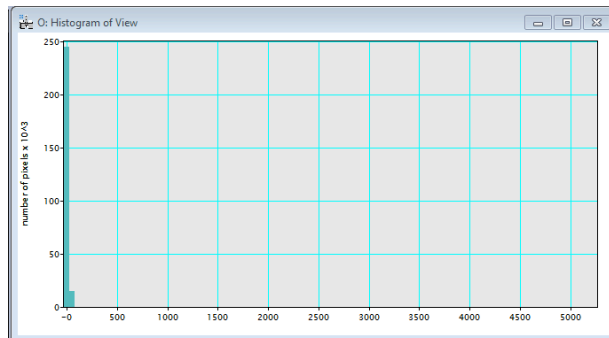
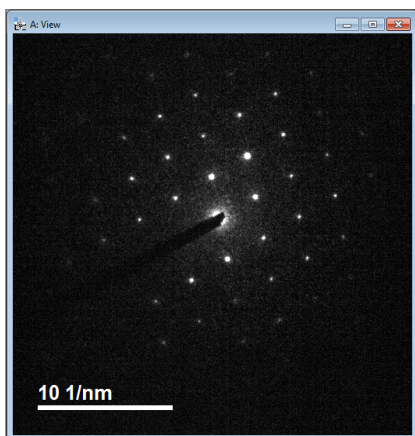
13. Flip the viewing screen up, set the exposure time in “Camera View” to 0.001 s, and start acquiring a live DP (it should be quite grainy and noisy)



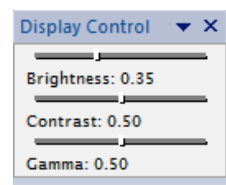
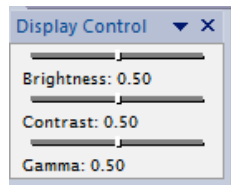
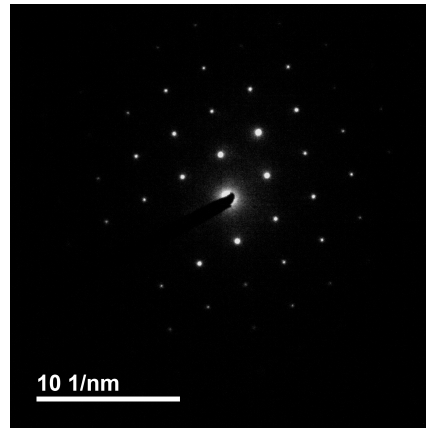
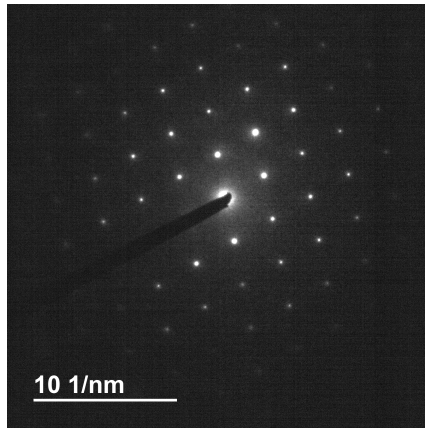
14. Along the top menu bar, select “Analysis” and “Histogram” to view a histogram of the live DP (the DP should look quite grainy and noisy). The rightmost value along the x-axis displays the maximum counts present in the DP.



15. Adjust the exposure time in “Camera View” until the rightmost value in the histogram is a few thousand counts (ideally, ~7000 counts); the live DP should look better after doing this, too. DO NOT EXCEED ~10000 counts.



16. In “Camera Acquire” input an exposure time ~10 times the value used in “Camera View” to acquire a high resolution DP with count distribution similar to the live DP.
17. If the acquired DP appears grainy, reduce brightness under “Display Control” in Digital Micrograph as needed.



18. If DP acquisition from a different region is needed, flip the viewing screen back down, return to "MAG1" mode, retract the SA aperture, center the new area of interest on the screen, insert the SA aperture again, and repeat the procedure starting from step 12.
19. When finished acquiring DPs, return to "MAG1" mode, retract the SA aperture, and turn off the FLC; if regular TEM imaging is needed, the full alignment procedure will need to be performed again.