FEI Tecnai F20 S/TEM: weak-beam dark-field operation Nicholas G. Rudawski ngr@ufl.edu (805) 252-4916 (352) 392-3077

This document assumes the user is already familiar with basic operation of the instrument in TEM mode and use of the digital camera 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. Please do not attempt to use this holder without being trained to properly load and unload it. Also, please note the following two requirements if you intend to image a self-supported specimen (e.g. a 3 mm disc of cored-out Si):
 - 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. The specimen should be loaded into the holder such that the electron transparent portion (or side) is at the <u>lowest possible position in the basket</u>.



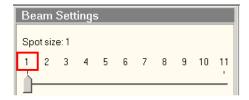
- 2. Find a region of interest and bring it to eucentric height.
- 3. Instrument settings

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3.1. Select and apply the "GL3" FEG register.



3.2. In Microscope Control, select the Beam Settings" control panel and select spot size = 1.



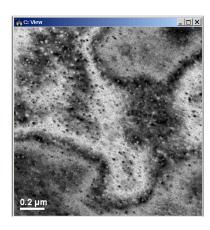
- 3.3. Perform the basic alignment of the instrument: center the C2 aperture, correct the condenser astigmatism, balance the deflector coils, and perform rotation centering.
- 3.4. Make sure you are in SA mode and set the magnification as needed; an indicated magnification of 6200 26000× (with the viewing screen down) is usually more than sufficient for WBDF imaging.
- 3.5. Expand the beam clockwise from crossover using the "Intensity" knob (left-hand control pad) and center it on the viewing screen

4. Setting up the 2-beam condition

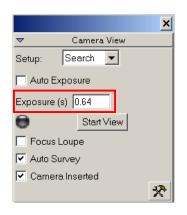
- 4.1. Select the "Diffraction" button (right-hand control pad) to enter diffraction mode.
- 4.2. Use the "Magnification" knob (right-hand control pad) to adjust the camera length as desired for observing the DP.
- 4.3. Insert an objective aperture into the DP (which one doesn't matter, as long as you 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
- 4.4. Use the "Intensity" knob (left-hand control pad) to focus the direct spot (make as small as possible) in the DP; the incident beam is now parallel.
- 4.5. 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; select the "Diffraction" button to return to TEM mode and center the specimen and then select the "Diffraction" button again to return to diffraction mode and resume adjusting the specimen orientation
- 4.6. Select the "Diffraction" button to return to TEM mode and center the beam on the viewing screen; <u>do not</u> adjust the "Intensity" knob after you return to TEM mode (yet); re-establish eucentric height as described previously (this should be done <u>every</u> time after the specimen is tilted).

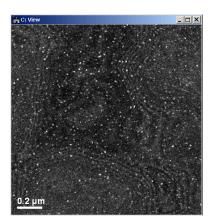
- 5. Setting up the WBDF condition
 - 5.1. Select the "Diffraction" button to enter diffraction mode; insert objective aperture 4 and carefully center it on the direct beam.
 - 5.2. Select the "Dark-field" button (right-hand control pad) to activate dark-field mode. Use the "Multifunction" knobs to tilt the beam so the Bragg spot moves to the original position of the direct spot; the Bragg spot should now be centered in the objective aperture and should be weaker in intensity.
 - 5.3. Select the "Diffraction" button to return to TEM mode; select the "Dark-field" button to switch back to bright-field (BF) imaging (the BF image should be evident on the viewing screen). Adjust the "Intensity" knob so the beam is slightly larger than the 40 mm circle on the viewing screen (at the suggested magnification range, this will not deviate the illumination from parallel to the point of being detrimental).
- 6. Imaging with the digital camera
 - 6.1. Start acquiring a live BF image with the digital camera (insert the digital camera, flip the viewing screen up, etc.); in "Camera View", adjust "Exposure (s)" as necessary and use the "Focus" knob to focus the BF image image.

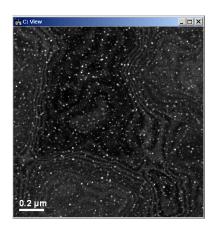




6.2. Select "Dark-field" to switch to WBDF imaging. Adjust "Exposure (s)" as necessary to improve the signal to noise ratio of the image (for live WBDF imaging, an exposure time of 0.5 – 0.6 s is usually sufficient).

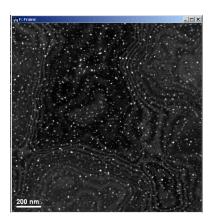






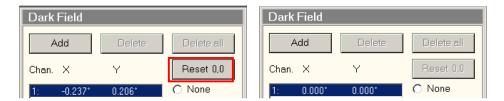
- 6.3. Use the "Focus" knob to finely focus the WBDF image, if needed; the optimal focus for WBDF imaging should not be very different compared to the BF image.
- 6.4. When ready, acquire a WBDF image using "Camera Acquire"; "Exposure (s)" values of 5 6 s are commonly needed for acquiring WBDF images.





6.5. If you wish to acquire a WBDF image at a different magnification, first flip the viewing screen down. Select the "Dark-field" button to switch back to BF imaging, adjust the "Magnification" knob accordingly, and re-center/resize the beam; select the "Dark-field" button to return to WBDF imaging and flip the viewing screen back up; acquire the image as described previously.

- 7. When finished WBDF imaging
 - 7.1. In Microscope Control, navigate to the Dark-Field tab; in the "Dark-Field" control panel, select "Reset 0,0" to reset the beam tilt.



7.2. Select the "Dark-field" button to turn off dark-field mode; all the buttons in the "Dark-Field" control panel should no longer be selectable.



Appendix: shorthand procedure for WBDF operation

- 1. Load sample in double tilt holder
- 2. Find region of interest, set at eucentric height
- 3. Apply GL3 FEG register
- 4. Set spot size = 1
- 5. Perform basic alignment
- 6. Set magnification to SA 6200 26000×
- 7. Expand beam clockwise from crossover
- 8. Center beam on viewing screen
- 9. Enter diffraction mode
- 10. Focus diffraction pattern
 - a. Insert objective aperture
 - b. Use "Focus" to focus objective aperture edge
 - c. Retract objective aperture
 - d. Focus direct spot with "Intensity"
- 11. Set up desired 2-beam condition
- 12. Return to TEM mode
- 13. Reset eucentric height
- 14. Return to diffraction mode
- 15. Insert objective aperture #4 and center around the direct beam
- 16. Select "Dark-field" button
- 17. Use multifunction knobs to tilt beam so Bragg spot centered inside objective aperture
- 18. Return to TEM mode
- 19. Select "Dark-field" button to return to BF
- 20. Adjust area of illumination to be slightly larger than 40 mm circle
- 21. Start acquiring live BF image in DigitalMicrograph
 - a. Focus image
- 22. Select "Dark-field" button to switch to WBDF image
 - a. Adjust exposure time as needed
 - b. Focus image
- 23. Acquiring final WBDF image
- 24. When finished, reset beam tilt
- 25. Select "Dark-field" button to turn off dark-field mode