JEOL 2010F: dark-field operation for single crystals

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

This application note assumes the user is already familiar with basic operation of the instrument, location of different controls, etc.

1. C2 aperture selection

    1.1. If imaging needs to be performed at magnifications < 10 k× (i.e. a wide field of view is needed), then the 70 μm C2 aperture should be selected (rod flipped to the left, middle white dot selected on aperture selection knob). If imaging can be performed at magnifications > 10 k×, then the default 50 μm C2 aperture (rod flipped to the left, smallest white dot selected on aperture selection knob) should be used.

    1.2. Once the appropriate C2 aperture has been selected, align it as usual.

2. Basic alignment

    2.1. Perform the following basic alignments of the instrument that you would normally: gun tilt alignment, condenser astigmatism correction, and eucentric height adjustment. It is best to set “SPOT SIZE” = 1 if you intend on performing DF imaging.

    2.2. For DF imaging, there is no practical reason to use magnifications > 30 k×, so set “α-SELECTOR” = 3.

    2.3. Since the magnifications used will be low, deflector coil balancing can be skipped.

    2.4. Perform current centering as you would normally; center the beam on the viewing screen when finished.

3. Setting up the base DF setting

    3.1. Activate “DARK TILT” (L1 panel) and use the “SHIFT-X” and “SHIFT-Y” knobs to center the beam on the screen (it will usually be shifted somewhat when you enter DF mode.

    3.2. Perform current centering as you would normally; now the beam tilt is nominally identical between BF and DF modes.

4. Specimen orientation adjustment

4.1. Activate “BRIT TILT” to go back into BF mode; then enter “DIFF” mode and tilt the sample as needed to set up a two-beam condition (direct beam = 0, Bragg beam = G) with s = 0 or *slightly* positive. You may need to periodically return back to “MAG” mode to move the stage so the sample is still under the beam. *Also keep in mind that your sample will probably be bent, so the two-beam condition you set up on one spot on the sample may not be valid on another spot.*

4.2. If a lot of tilting is performed, you should go back and reestablish eucentric height; also note that after you reestablish eucentric height, the two-beam condition may have slightly changed, so you may need to perform some additional fine adjustments to the specimen tilt (and then possibly go back and reestablish eucentric height yet again).

5. Objective aperture centering in BF mode, fine tuning the base DF setting

5.1. “BRIT TILT” should still be activated at this point; enter “DIFF” mode and adjust the camera length as needed (L = 15 – 25 cm is usually sufficient). If the DP needs to be shifted closer to the middle of the viewing screen, select “PROJ” under the “DEFLECTOR” control (R2 panel) and using the “SHIFT” knobs (R2 panel).

5.2. Insert the *largest* objective aperture; if the objective aperture edge does not appear sharp, use “DIFF FOCUS” to focus the *edge of the objective aperture*. Then use “BRIGHTNESS” to focus the direct spot in the DP.

5.3. Insert the third smallest objective aperture and center it on the direct beam; now activate “DARK TILT”; the direct beam should still be centered in the objective aperture, but may be slightly shifted. If it is shifted, use the “DEF-X” and “DEF-Y” knobs to move the beam so it is centered in the objective aperture. Retract the objective aperture when finished.

5.4. The beam tilt is now identical for BF and DF modes.

6. Setting up DF conditions

6.1. Leave “DARK TILT” activated and remain in “DIFF” mode; with the object aperture retracted, make note of where the Bragg beam is in relation to the direct beam.

6.2. Insert the *third smallest* objective aperture and center it on the direct beam.

6.3. Setting up a CDF condition

6.3.1. Use the “DEF-X” and “DEF-Y” knobs to shift the beam that is the opposite (negative) of the Bragg beam (–G) so it is now centered in the objective aperture; –G should now be much *brighter* than it was before tilting.

6.4. Setting up a WBDF condition

6.4.1. Use the “DEF-X” and “DEF-Y” knobs to shift the Bragg beam (G) so it is now centered in the objective aperture; G should now be much *weaker* than it was before tilting (hence, *weak* beam dark-field).

6.5. Leave the object aperture inserted; return to MAG mode when finished.

7. Acquiring images

7.1. After returning to “MAG” mode, activate “BRIT TILT” to observe the BF image. When ready,start acquiring a live image in Digital Micrograph ®.

7.2. Adjust the “FOCUS” control to focus the image (this is usually easier to do in BF rather than DF mode); remember, the “DV” value should be close to +0 after focusing if the sample is at eucentric height.

7.3. Once rough focus is achieved in the BF image, activate “DARK TILT” to produce a DF image. It should be re

7.4. *Be careful when adjusting “Exposure” in Camera View for viewing of the live DF image.* Even though the overall appearance of the image may be somewhat faint, if certain portions of the specimen are strongly diffracting, they can become as intense as spots in a DP. *It is best to view a histogram of the live image to make sure no saturation is occurring.*

7.5. It is usually necessary to use fairly long exposure times (1 – 5 s) when acquiring DF images using “Camera Acquire”. If you need to use exposure times much longer than this, your specimen is probably too thick to obtain a good DF image.

7.6. When finished imaging, retract the digital camera and flip the viewing screen back down as usual.

8. Finishing

8.1. Make sure “BRIT TILT” is activated and then place the instrument in its normal idling state before holder retraction. Also be sure to set the C2 aperture back to the default 50 μm setting if this was changed.