OPERATION OF THE PHILIPS CM-200 FEG-TEM

When not in use, the CM-200 should be in the MICROSCOPE ON configuration with the HIGH TENSION ON (illuminates green when the high tension is on).

. The microscope is normally never turned off.

Preliminary

- Add liquid Nitrogen to the Dewar next to the sample holder. (Anticontamination device:) Place the metal-clad glass vacuum dewar into its holder to the right of the column so that the soft copper wire "beard" hangs inside it. The glass viewing windows should be covered for safety. Fill the dewar nearly full with liquid nitrogen and place the styrofoam cap on top.
- Log in (in the hallway)
- Go to vacuum page by pressing button Ready then button Vacuum. Make sure pressure reading of IGP is below 10, preferably at 5 before proceeding to the next step.

1. **Specimen Holder Removal, Loading, and Insertion**

Never remove or insert the specimen holder when the red indicator light (on the front of the compustage housing) is on. Do not touch the leading edge of the specimen holder (from the o-ring to the tip) with ungloved hands. This portion resides inside the vacuum and must be kept clean.

There are several types of sample holders. The first type is single tilt (tilt in X or Alpha direction). The second type is double tilt (tilt in both X and Y directions). Cold holder consists of a sample holder and a chamber to contain liquid nitrogen. Thermometer and heating systems are attached. Hearing holder can be used to hold samples that needed to be heated (up to 700°C) during microstructure exploring.

1.1 REMOVAL: To remove the specimen holder from the column, carefully pull the round black handle straight out until it stops, and hold it firmly so that it does not get pulled back into the 'scope by the vacuum. Now rotate it **clockwise** until it stops again; it may now be pulled straight out (carefully), free of the column. The specimen holder should be set down only on its Lucite stand.

1.2 SPECIMEN LOADING:

(i) Use the pin tool (located in the Lucite stand, under the tip) to lift the grid clamping device at the tip of the specimen holder).
(ii) Transfer your grid to the specimen holder using forceps. To make scanning the grid easier, you may wish to orient one set of grid bars parallel to the long axis of the specimen holder.
(iii) Use the pin tool to carefully lower the clamping device onto the grid and lock it in place.

1.3 INSERTING THE SPECIMEN HOLDER INTO THE COMPUSTAGE:

(i) Touch the sample holder tip to one of the aperture handle to discharge the sample holder.
(ii) With the small pin in the holder tip at the 11:00 o'clock position, carefully insert the specimen holder into the airlock entryway at the center of the compustage. Insertion of the holder will initiate the pre-pumping sequence, and the red indicator light on the front of the compustage will come on. Slide the holder in until it stops; at this point it will not go all the way in. (The data

monitor screen will automatically open to the HOLDER SELECTION PAGE. Press the appropriate key (in most cases you'll choose NO COMPUSTAGE B TILT) and then press the READY button (below the data monitor screen). The data monitor screen will return to the previously selected page (normally TEM BRIGHTFIELD).)

(iii) After the red indicator light goes out, grip the specimen holder firmly by its black plastic handle and rotate it counterclockwise to 6:00 o'clock position. When it reaches the limit of its rotation, the vacuum will begin to pull the holder deeper into the column. Maintain a firm grip on the handle as the holder enters the column so that it does not get drawn in too quickly. Once the holder appears to be all the way in, jiggle it very gently to ensure that it is indeed inserted completely into the column.

2. Initiating Operation

2.1 When the CM-200 is ON, the white STAND BY and red MICROSCOPE OFF buttons will be illuminated; the ON button will be dark. The lit buttons indicate their availability as emergency functions while the microscope is running.

2.2 Depress the PANEL DIM knob to illuminate the data monitor screen and emission gauge. Clockwise rotation of this knob increases the intensity of the panel lighting and the light on the flexible stalk. Clockwise rotation of the DATA DIM knob increases the brightness of the data monitor screen.

2.3 Ensure that the UHV and HIVAC indicator lights are lit (green). If they are not it may be necessary to press the VACUUM SYSTEM ON button and wait (20 to 30 minutes) until the two lights come on, indicating that operational vacuum status has been attained.

2.4 The data monitor screen will display the CM-200 PHILIPS MICROSCOPE STATUS startup page or, more likely, one of the MODES or MODE SELECTION pages. If the READY light (on the pushbutton directly below the data monitor screen; to the right) is lit, pressing it will move the screen selection back in the hierarchy of pages. Press the MODES key to obtain the MODE SELECTION page.

2.5 For transmission electron microscopy, press the HR-TEM key on the MODE SELECTION page. If the letters HR-TEM are highlighted it will be necessary to press only once; otherwise press the HR-TEM key twice. The screen should now display the HR-TEM BRIGHTFIELD page.

2.6 Press the VACUUM key to check the vacuum status of the instrument. It should read READY at the top center of the data monitor screen. If it reads START-UP the operator must wait for the vacuum to improve before the 'scope can be used. Do not uses the microscope unless the ion getter pump (IGP) reading is lower than 10.

2.7 To return to the TEM BRIGHTFIELD page press the READY button.

2.8 To select the operating voltage, press the PARAMETERS key to open the PARAMETERS pages. On the first of these pages the kV may be modified by pressing the left (lower) or right (higher) key adjacent to the HIGH TENSION kV notation on the screen. Most TEM users in this laboratory work at 200 kV.

2.9 Press Ready, Mode, choose configuration page. Slowly increase the filament voltage to 3.8. As a rule of thumb, rotate the knob until you hear three clicks; wait 20 seconds; rotate again for three clicks; wait another 20 seconds; repeat until reaching 3.8.

2.10 Open the valve (from Close to Open, unti-clockwise) to the right of the microscope. You should see the beam. Move sample feature to the middle of the viewing area.

2.11 Also on the first PARAMETERS page is the EMISSION setting, which should be left at 3. Press the READY button to return to the TEM BRIGHTFIELD page.

2.12 With the high tension on, an operator may load his/her own previously stored or default alignment/stigmator settings for the kV at which the 'scope is operating. This requires logging into the computer (rodin) on the table to the right of the microscope, obtaining a CRT terminal screen, and typing, e.g., 's_toscope default' to load the default settings for the currently selected kV. A separate laminated sheet of instructions in the TEM room fully describes these functions.

2.13 If the operator intends to expose TEM negatives using the built-in camera, it is best to ensure that the CCD camera is retracted and shut off so that it cannot interfere with the shutter mechanism. Using the Mac computer, whose CRT and keyboard rest on top on the CM-200 operating console, the operator may open Digital Micrograph (if it is not already open), select MSC from the top toolbar, and from that pull-down menu click to retract the CCD camera. The CCD camera control unit, which rests to the left of the Mac CRT, may also be turned off using the red switch on the front. Left.

3. Beam Alignment

These functions are performed without a specimen in the holder and may be performed without inserting a specimen holder at all. If a holder is not in place, a radiation safety interlock prevents the spot size from being changed to a number lower than 5 (the largest and brightest spot size is no. 1). This will produce a much dimmer beam image than that seen with the holder in place.

3.1 Using the MAGNIFICATION knob, select 17,500x.
3.2 Defocus the electron beam by turning the INTENSITY knob clockwise to strongly overfocus the C2 lens. Bring the beam back to crossover (the smallest image of the beam) and center it using the SHIFT X/Y knobs.
3.3 Choose a C2 aperture (usually position 3, which is a 100-micron aperture) by rotating the largest knurled knob on the topmost aperture control on the column to the desired position.
3.4 Using the INTENSITY knob, slowly overfocus (turn clockwise) the beam to coincide with the diameter of the intermediate-sized black circle on the phosphorescent viewing screen. If the defocused beam is not coincident with the circle, adjust it using the C2 aperture centering controls. These are both the intermediate-sized knurled knob on the aperture rod and the knurled knob on the side of the assembly (to the right). DO NOT TOUCH THE SMALLEST, INNERMOST KNOB BECAUSE IT UNSCREWS THE APERTURE ROD. Repeat these steps (3 and 5) until the beam spreads uniformly around the reference circle while being over focused (INTENSITY knob).
3.5 Using the INTENSITY knob, adjust the beam to crossover. Depress the FINE button (to the left of the INTENSITY knob; the indicator light turns green when it is on) and use the INTENSITY knob to sharpen the de-saturated filament image. Center the image using the SHIFT X/Y knobs.
3.6 Press the STIG button, on the right-hand instrument panel, to open the STIGMATOR CONTROL page.
3.7 Press the COND key on the data monitor screen to select the C2 lens for astigmatism correction. Use the MULTIFUNCTION X/Y knobs, while rotating the beam through crossover using the INTENSITY knob, to obtain the cleanest, roundest de-saturated filament image possible.
3.8 Press the STIG button to return to the CONFIGURATION page.
3.9 Turn the FILAMENT knob clockwise to fully saturate the filament. When the saturation point (as defined by the FIL LIMIT) has been reached, the microscope will emit a 'beep.'
3.10 Press the READY button and then the TEM key to return to TEM BRIGHTFIELD.
3.11 Using the lever to the left of the viewing chamber, lower the small phosphorescent screen into place. Insert the beam stop (at the right-hand side top of the viewing chamber) so that it will be visible across the small screen. Use the binoculars to observe the beam stop. To adjust the binoculars for your eyes, first adjust the interpupillary distance so that you can see through them with both eyes; then adjust each eyepiece so that the rough edges of the beam stop are in focus for each eye. When you are done, retract the beam stop and lift the small screen back out of view.

4. Eucentric Height Adjustment

4.1 Set the MAGNIFICATION to 5,800x and use the X/Y JOYSTICK to center a small notable feature of your specimen.
4.2 Focus your chosen feature using the knobs marked STEP SIZE. The outer knob changes the focus; the inner knob (the step size adjustment) modifies the amount of focus change per 'click' of the outer knob.

4.3 From the TEM BRIGHTFIELD page, press COMPUSTAGE once; the COMPUSTAGE REGISTER CONTROL page will appear.
4.4 Press A-WOBBLER; this will initiate back-and-forth tilting of the goniometer.
4.5 Use the Z control lever on the JOYSTICK to move the specimen up or down and thus minimize apparent movement of the centered feature.
4.6 When the feature moves only minimally or not at all, press the A-WOBBLER key to inactivate tilting; then press the READY button to return to TEM BRIGHTFIELD.

5. Pivot Point Alignment

Ensure that the specimen is eucentric before performing this procedure. For this procedure you may work with the OBJECTIVE APERTURE in place to protect your specimen.

5.1 Center (X/Y JOYSTICK) and focus (concentric knobs under STEP SIZE) an image feature at 24,500x (MAG knob).
5.2 Press the ALGN button to access the ALIGNMENT SELECTION page. (Note that the alignments are divided into PROCEDURES (on the left side of the page) and DIRECT alignments (most of which are on the right side of the page). Most users will not want to access the PROCEDURES, which are long and complicated. For this set of instructions we will be using only the DIRECT alignments.)
5.3 Using the INTENSITY knob, adjust the beam to crossover.
5.4 Press the beamcoils PIVOT POINT X key, on the right side of the page, so that it is highlighted.
5.5 Using the MULTIFUNCTION X/Y knobs, bring the two beam spots (the pivot points, on the fluorescent viewing screen) together so that they overlap.
5.6 Center the coinciding spots using the SHIFT X/Y knobs.
5.7 Press the beamcoils PIVOT POINT Y key.
5.8 Using the MULTIFUNCTION X/Y knobs, bring the two beam spots together so that they overlap.
5.9 Center the coinciding spots using the SHIFT X/Y knobs.
5.10 Press the ALGN button to exit the ALIGNMENT SELECTION page.

6. Rotation Center Alignment

This procedure may be performed with the OBJECTIVE APERTURE in place to protect the specimen and add contrast to the image.

6.1 Focus and center a feature of the specimen at 100,000x.
6.2 Press ALGN to open the ALIGNMENT SELECTION page. On the upper right-hand side of the page, press ROT CENTER so that it becomes highlighted. Now either voltage or current centering may be performed.
6.3 VOLTAGE CENTERING

(i) On the lower right-hand side of the page, select VOLT (under rot center VOLT CURR) so that it becomes highlighted. This will cause the high tension to modulate (the inner STEP SIZE knob adjusts the amplitude of modulation). If the chosen feature shifts off center laterally, the beam is not aligned along the optical axis of the microscope and must be corrected.
(ii) Use the MULTIFUNCTION X/Y knobs to stabilize the feature at the center of the screen, eliminating all lateral movement. The feature should appear to be pulsating.
(iii) Press the ALGN button to return to the TEM BRIGHTFIELD page.

6.3.1 Current centering:

(i) On the lower right-hand side of the page, select CURR (under rot center VOLT CURR) so that it becomes highlighted. This will cause the objective lens current to modulate (the inner STEP SIZE knob adjusts the amplitude of modulation). If the chosen feature shifts off center laterally, the beam is not aligned along the optical axis of the microscope and must be corrected.

(ii) Use the MULTIFUNCTION X/Y knobs to stabilize the feature at the center of the screen, eliminating all lateral movement. The feature should appear to be pulsating.

(iii) Press the ALGN button to return to the TEM BRIGHTFIELD page.

7. Centering the Objective Lens Aperture

For this procedure a specimen must be in place. Choose an area for which it is acceptable to sustain beam damage. With the OBJECTIVE APERTURE out, set the magnification to 5800x and overfocus the beam by turning the INTENSITY knob clockwise from crossover.

7.1 Set the focus step size (inner STEP SIZE knob) to 5 and press the Zoom button to put the 'scope in diffraction mode.

7.2 If necessary, adjust the CAMERA LENGTH to 620 mm using the MAG knob.

7.3 Center the diffraction spot using the MULTIFUNCTION X/Y knobs.

7.4 Using the focus (outer STEP SIZE) knob, refocus the beam to the smallest, brightest spot. It may also be necessary to adjust the INTENSITY knob.

7.5 Insert the OBJECTIVE APERTURE by rotating the aperture displacement lever below it to the left.

7.6 Apertures may be selected by rotating the largest knurled knob on the objective aperture assembly to any one of four numbered positions. (The APERTURE MEMO lists the diameters and positions of the apertures currently installed in the 'scope. It may be accessed from the TEM BRIGHTFIELD page by clicking MODES and then CONFIGURATION.)

7.6.1 Once the aperture has been selected, center it using both the smaller knurled knob in the series on the aperture assembly and the small knurled knob to the right. Remember not to manipulate the smallest, innermost knob, which is not knurled and will unscrew the aperture rod.

7.6.2 Press Zoom to exit diffraction mode.

8. Objective Lens Astigmatism Correction

8.1 Select an area that may be imaged at high magnification without harming any desirable portions of the specimen. Increase the magnification to 175,000x or higher and adjust the illlumination so that the substructure or background grain of the specimen may be observed easily. The INTENSITY will have to be modified and the beam will have to be recentered using the SHIFT X/Y knobs (Deflectors) as the magnification is increased. This latter function may alternatively be controlled using the RST button, on the panel to the left of the column.

8.2 Set the focus step size to 2 and obtain a slightly underfocused image for maximum contrast.

8.3 Press the STIG button to open the STIGMATOR CONTROL page. If it is not already highlighted, press the OBJ key on this page.

8.4 Use the MULTIFUNCTION X/Y knobs, one at a time, to obtain the sharpest possible image of the grain substructure.

8.5 Confirm that any astigmatism has been corrected by varying the focus (back and forth through focus, from underfocus to overfocus) and watching to see if a "streaking" pattern emerges and changes direction between under- and overfocus. If the astigmatism has been corrected, the specimen will vary only in focus, with no pattern evident. Repeat steps 4 and 5 until no pattern is apparent.

8.6 Press the STIG button again to return to the TEM BRIGHTFIELD page.

9. Condense Lens Astigmatism Correction

This procedure is necessary if the beam spot is not a symmetric circle when the intensity knob is rotated.

9.1 Press STIG button to open the STIGMATOR CONTROL page.
9.2 Select Condense Aperture.
9.3 Use the MULTIFUNCTION X/Y knobs, one at a time, such that the beam spot circle has a fixed center when the beam intensity is changed.

10. **High Resolution Images**

10.1 Bring in the small screen into viewing chamber.
10.2 TV on.
10.3 Amplifier on.

11. **Load Films**

11.1 Press CAMERA AIR.
11.2 Put film in.
11.3 PUMP till to IGP less than 10.
11.4 Note films, along with film box should be put into a vacuum for more than 24 hours to degas.

Note: reset the negative numbers