Please follow these instructions for use of the Philips CM100 TEM. Adopted from website below.

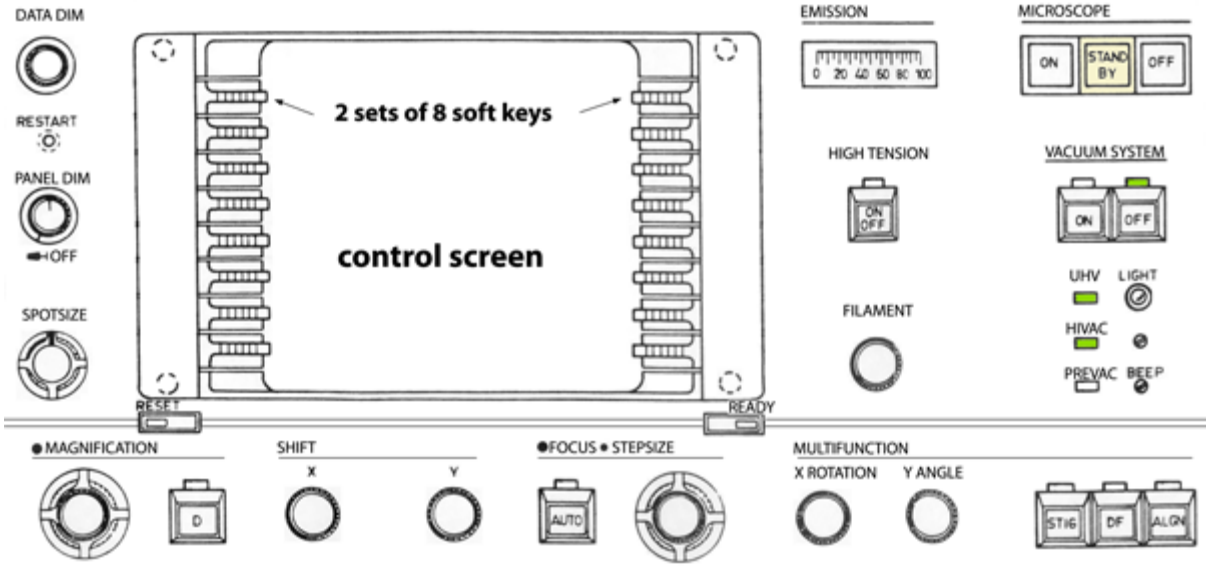
http://staff.washington.edu/wpchan/if/cm100_inst.shtml

Instructions for the Philips CM100 TEM and peripherals

Please refer to the Philips CM100 Operating Manual (PW6021) on the console for additional information.

Check-in

1. enter the In time on the log sheet and check previous entries for unusual remarks
2. initial setup for the right-hand control panel

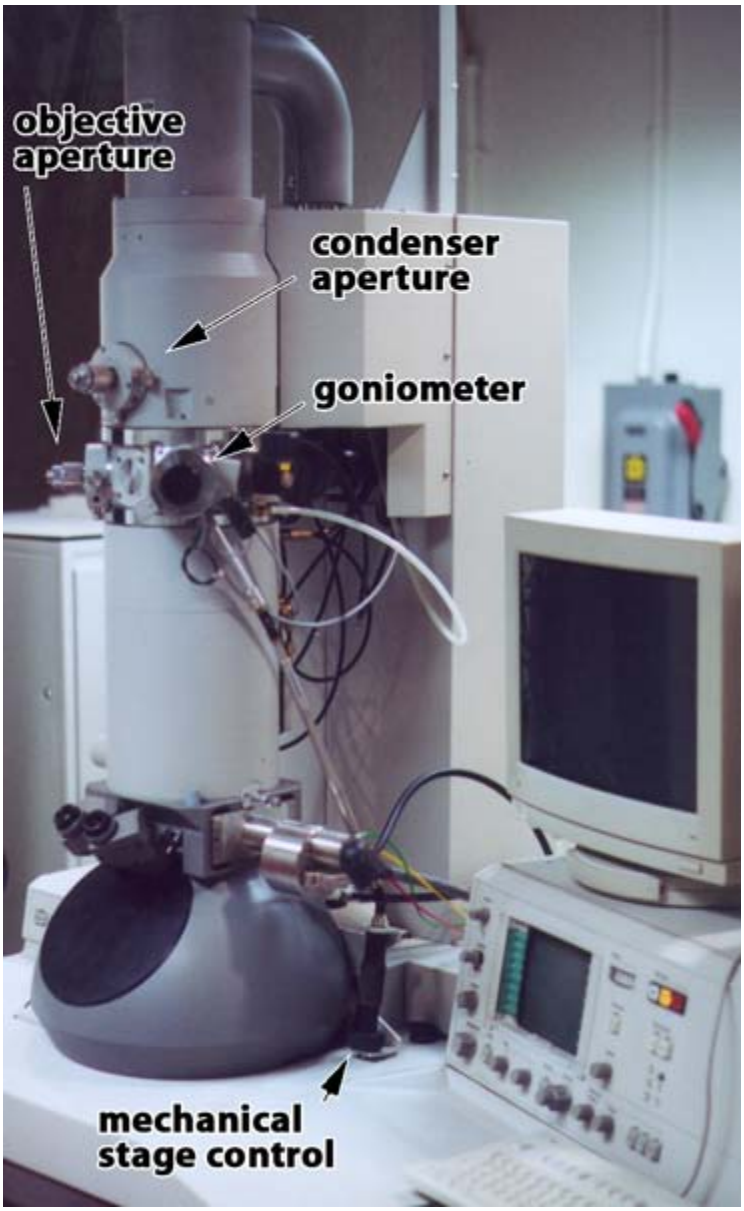


   - push in PANEL DIM knob to turn on the display and turn clockwise to bring up the panel light and indicators
   - UHV and HIVAC should be on, if not **STOP**
   - turn DATA DIM knob clockwise to bring up the Control Screen display menu: should be on either the Startup page showing MICROSCOPE STATUS, or TEM BRIGHT FIELD page (press MODES to go back to the MODE SELECTION page, hit READY for Startup page)

     Note: Whenever the READY button is lit, you can press it to return to an upper level page.

3. VACUUM STATUS should read READY on the Startup page
4. press soft key for CONFIGURATION on either the Startup or MODE SELECTION page
    - TUNGSTEN should be selected for filament, if not **STOP**
    - make sure FIL LIMIT is set as posted and not 0, if not **STOP**
5. hit READY to back out to the previous page
6. press key for MODES to go to the MODE SELECTION page then select TEM for the TEM BRIGHT FIELD page
7. set MAGNIFICATION to 1100× and select SPOT 2
8. press key for PARAMETERS
9. make sure EMISSION and HIGH TENSION are set as posted

    Note: Check the Appendix if you want to use a different accelerating voltage.
10. hit READY to back out to the TEM BRIGHT FIELD page



Specimen loading

1. DO NOT touch any parts of the specimen holder except the black cap
2. grasp the black cap securely and pull the specimen holder out until it stops
   Caution: Place the other hand on the goniometer to catch the holder in case it accidentally slips and is being sucked back in (it is under vacuum!). If that happens, the crystals at the end of the holder and the seat inside the column can be damaged; a rather costly repair and serious down time.

3. rotate the specimen holder clockwise until the marker is at 5 o'clock position and then ease it out gently to break the vacuum

   Caution: It does take a certain amount of force to break the vacuum. If you do it too fast, you are more likely to scrape the holder against the inner surface of the airlock damaging both parts.

   Here are 2 ways to do this safely:

   - if you have longer fingers, you can grasp the black cap with your thumb, ring and small fingers, then use your index and middle fingers to push against the goniometer gently until the vacuum breaks
   - otherwise, you can rest your left hand with the knuckle on the goniometer just adjacent to the z-position thumb screw, then use the thumb, index, and middle finger to push on the cap gently until the vacuum breaks while stabilizing the holder with the right hand

4. place the specimen holder on the support
5. steady the holder and lift open the spring loaded clamping device with the pin tool (very carefully since the clamp can easily snap off)
6. place the grid in the recess and close the clamp
7. check for dust, lint, or other debris on the rod; if present, carefully remove them with Ross lens paper
8. gently re-insert the holder with the marker at 5 o'clock position and make sure that you do not bump the rod against the inside of the chamber, the red airlock indicator light on the specimen chamber should come on to indicate initiation of pre-pumping
9. fully insert the rod and then turn it counterclockwise until marker is at 4 o'clock position

   Note: as soon as a vacuum is formed, the holder will get sucked in further and you should hear a click

10. when the indicator light goes out (~ 10 s), turn the holder counterclockwise until marker is at 12 o'clock position and guide the holder to slide in slowly; put the other hand on the goniometer to catch the holder if it accidentally slips and slams in
11. gently wriggle the holder slightly to make sure that it is all the way in and sits squarely in the chamber

12. wait 5 min or monitor the VACUUM page until IGP is < 27 before proceed to Filament saturation

Beam Control

The filament is pre-saturated by the facility staff and needs only to have the proper high tension set and the filament heated.

Use the soft key labeled parameters to open the high tension control. The microscope is stored in the 100kv position, use the soft keys to reset the high tension to 80kv, the normal working kv for this unit. It a different kv is needed, please see the facility staff as the filament saturation must be changed.

Quick quality check (notify Facility staff for any alignment problems)

- if possible, locate a hole or area devoid of material in your preparation and then set the magnification to 5800×
- bring the beam to crossover with INTENSITY (left-hand control panel), center it with SHIFT XY (right-hand control panel), and perform the following

action	observation	possible problem
change INTENSITY upon crossover	beam shift (see example in appendix)	CONDENSER APERTURE alignment

Specimen exchange

1. do Check-out procedure (see below) steps 1 to 4
2. perform Specimen loading procedure steps 1 to 6
3. replace the grid and complete the procedure, wait 5 min or monitor the VACUUM page until IGP is < 27
4. wait 5 min or monitor the VACUUM page until IGP is < 27, then do Beam Control as stated above.

Check-out

1. select magnification 1100×, SPOT 1, make sure both apertures are selected as posted and engaged
2. turn the FILAMENT knob counterclockwise, pause between each click until the on screen message is clear or wait 0.5 s
3. when the filament setting is down to 0, the system will beep
4. Enter the parameters screen and readjust the high tension back to 80kv
5. remove your sample and replace the specimen holder in the scope according to the sample exchange directions above.
6. return to the Startup page (MICROSCOPE STATUS)
7. turn DATA DIM fully counter-clockwise
8. turn PANEL DIM until the desk light is the dimmest, pull out PANEL DIM to turn off the display
9. enter Out time in the log sheet, check the shutdown procedures listed and complete the log
10. cleanup the work area before you leave

Using the TEM Camera

1. On the TEM plate, there are four hash marks, the center point of these marks represent the four corners of the film. Center your desired image into these marks.
2. Bring up the ocular plate and use the intensity knob to bring the exposure time to 2.00 seconds, any shorter an exposure, the image is not clear.
3. Make sure the room lights are out and the screen is relatively dim and pull up the main plate. At this time, the exposure button is lit. Depress the button and the film will be exposed. The process is complete once the microscope lights come back up.
4. Readjust the intensity and continue to work.

Exchanging the Film Canister

1. Once you have finished your work, you must develop your film. First, open the nitrogen gas tank and set the output pressure to the black line on the gauge, any higher will blow out the viewing windows.
2. On the vacuum map page, use the soft key next to vent film compartment to begin venting. This will take several minutes. At this time, vent the film desiccators in the utility room, remove a fresh film canister and your labs box of film.
3. Once the venting is done, the back cover to the film compartment will be able to be lifted. Switch the film canisters, so that a fresh batch of film is loaded.
4. In the SEM room, use the back sink for developing. Make sure window shade is closed as well as the door. Use the sodium safe lamp (takes several minutes to warm up). Remove all of your exposed film and replace in the canister with fresh film. Develop you film as follows
    a. 4 minutes in developer, followed by a quick rinse in water
    b. 4 minutes in fixer
    c. 2 minutes in water for washing. Let dry over night.
5. Replace extra film in box and refilled canister into film desiccators.

Appendix

Saturating the tungsten filament

Caution: Do NOT perform these steps unless you have been trained by Facility staff on this procedure.

1. load the specimen as usual
2. push HIGH TENSION on
3. go to CONFIGURATION page to monitor FILAMENT HEATING

4. turn the FILAMENT knob clockwise, allowing 0.5 s wait between each click (or until the message is clear) and pause when ACTUAL reaches 19, or 5 below the posted FIL LIMIT, whichever is lower
5. the emission current meter should read less than 10 µA, otherwise turn down FILAMENT (ACTUAL to 0) and **STOP**
6. if there is no beam, check the following:
    - if there is no emission current, **STOP**
    - if emission current registers between 0–10 µA
        i. try adjusting INT, it may be near the limits; press RST
        ii. beam may be off the center, try SHIFT XY
        iii. the specimen holder may be blocking the beam because the mechanical stage controls are too far from the mid positions: left is ~10, right is ~0; center the holder
        iv. grid bars may be blocking the beam, use the mechanical stage controls to move them out of the way
7. **STOP** if there is still no beam, otherwise proceed with a clear area or a hole on your grid
8. push FIL LIMIT to deselect (unlock)

   Note: You can start from here if the filament desaturates right in the middle of your session.
9. push ALGN on right-hand control panel
10. set magnification to 5800×
11. focus beam with INT and center with SHIFT XY
12.  The beam will probably look irregular with some dark areas, continue turning the filament knob clockwise, you will see an "eye" shape image

13. turn FILAMENT further clockwise until there is minimal amount of or no serration (dark areas) with maximum and even brightness
14. if emission current is > 20 µA, turn down FILAMENT and **STOP**; otherwise continue
15. center beam with SHIFT XY
16. optimize gun tilt (optional)
    i. select TILT on right-hand panel (detailed in Gun TILT adjustment)
    ii. use MULTIFUNCTION XY to get a symmetrical image with maximum brightness i.e., shortest exposure time

    push ALGN to exit back to CONFIGURATION

    lock FIL LIMIT

    scope is now ready

    repeat this same procedure if you have more samples

    at the end of the session, set the FIL LIMIT back to 24
    . go to CONFIGURATION page to monitor FILAMENT HEATING
    i. turn FILAMENT down until ACTUAL reaches 25
    ii. deselect FIL LIMIT, turn FILAMENT down one click so ACTUAL is now 24
    iii. select FIL LIMIT to lock it at 24
    iv. continue check-out as usual

Changing HIGH TENSION

Caution: Do NOT perform these steps unless you have been trained by Facility staff on this procedure.

The filament is usually saturated at 100 kV but you should checked the posted operating parameters. Other kV values can be used with an appropriate EMISSION and saturation (FIL LIMIT) of the filament, please check with Facility staff.

1. make sure HIGH TENSION is off, press soft key to change EMISSION first then select desirable HIGH TENSION
2. saturate the filament; currently, 80 and 100 kV use the same FIL LIMIT
   Note: If the HIGH TENSION fails to turn on, the Wehnelt s.w. protection may be tripped and requires a reset.
3. make sure that the emission current does not exceed 20 µA

4. it may be necessary to correct for objective lens astigmatism (see below)
5. return the scope to the posted EMISSION and HIGH TENSION setting after your session unless otherwise instructed by Facility staff

Objective lens stigmation for 80 and 100 kV

Caution: Do NOT perform these steps unless you have been trained by Facility staff on this procedure.

1. locate a roundish object at a magnification higher than the max. that you will use
2. focus beam, adjust for max. and even illumination; overfocus to spread the beam, if needed
3. focus image
4. press STIG on right-hand control panel
5. press soft key for OBJ
6. press soft key for CHANNEL to highlight 1 for the 80 kV preset or 2 for the 100 kV preset
7. record the current reading for both A and B
8. use MULTIFUNCTION XY to correct for any astigmatism, check image focus
9. repeat step 8, if necessary
10. if things go south, return to the preset A B reading, focus image and do step 8 again
11. press STIG to exit this procedure
12. at the end of your session, restore the preset A B reading

Gun TILT adjustment

Caution: Do NOT perform these steps unless you have been trained by Facility staff on this procedure.

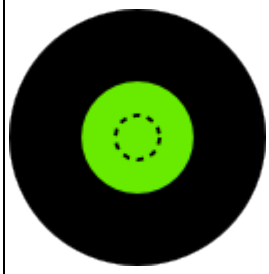
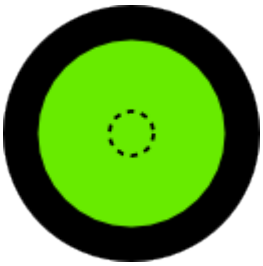
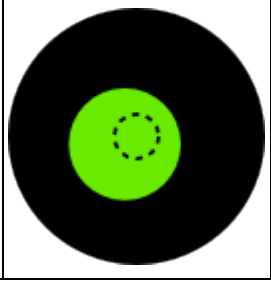
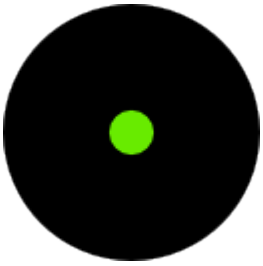
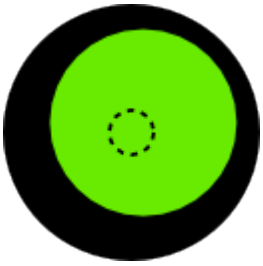
1. locate a hole or an empty area on the grid
2. bring magnification to 5800×
3. focus and center beam
4. check exposure time against posted value

5. press ALGN on right-hand control panel
6. press soft key for gun TILT SHIFT to highlight TILT
7. use MULTIFUNCTION XY to maximize the brightness (minimize exposure time) and use SHIFT XY to keep the beam centered
8. if exposure still differ much from posted value, notify Facility staff
9. press ALGN to exit this procedure

Condenser aperture alignment

Caution: Do NOT perform these steps unless you have been trained by Facility staff on this procedure.

1. move to a less important part of your prep if you want to minimize beam damage
2. select low to medium M range magnification
3. select SPOT 5, center and focus beam
4. underfocus and then overfocus the beam to observe for lateral shift

	underfocus	crossover	overfocus
aligned			
misaligned			

5. use the 2 mechanical adjustment knobs to shift the beam until there is no more lateral displacement going from underfocus to overfocus

Objective aperture selection and alignment

Caution: Do NOT perform these steps unless you have been trained by Facility staff on this procedure.

1. move to a less important part of your prep if you want to minimize beam damage
2. select medium M range magnification
3. center and focus beam, adjust for max. and even illumination; overfocus to spread the beam, if needed
4. focus image
5. press D on right-hand control panel next to MAGNIFICATION for diffraction mode
6. you will see a brighter spot centered more or less in the beam on the fluorescent screen
7. camera length will show on the display instead of magnification
8. use the MAGNIFICATION knob to change the camera length to 640 mm
9. carefully rotate the objective aperture holder to the desirable position

   objective aperture size

position	1	2	3	4
diameter (μm)	30	40	70	100

10. use the 2 mechanical adjustment knobs to center the beam with respect to the bright spot

    Note: The aperture could be way off the center, simply turn the adjustment knob to move the beam until you can see the central bright spot.

11. press D again to go back to bright field mode
12. you should check the stigmation after changing to a different size aperture
13. change back to position 4 and center the aperture after you are done

On Screen Measurements

- quick size estimation: the 2 concentric marks on the large viewing screen are 40 and 5 mm in diameter e.g., at 25 000×, the inner circle indicates 200 nm
- use RSET DEFOC for height

1. set eucentric height
2. focus on structure
3. press soft key for RSET DEFOC
4. focus on substrate
5. defocus displays the height

Note: Do not use the wobbler (WBL) to focus in this procedure, it will set the defocus readout to 0.

- use MEASURING for distance with respect to an external reference e.g., the pointer or any screen markings
  - single measurement
    1. set eucentric height
    2. focus on structure and align one end to the reference
    3. press soft key for MEASURING
    4. press soft key for ENTER
    5. use SHIFT XY to move the structure and align the other end to the reference
    6. d1 displays the distance
    7. press soft key for ENTER twice to clear d1
  - cumulative measurements
    1. do single measurement
    2. use the mechanical specimen translation controls to align the 2nd item to the reference
    3. check the focus
    4. use SHIFT XY to align the other end of the 2nd item to the reference
    5. d1 displays the cumulative distance
    6. repeat for more
  - comparative measurements
    1. do single measurement
    2. press the soft key for ENTER to store d1 and activates d2
    3. measure the 2nd item
    4. display will show d1/d2 and the angle between them

Note: Press READY to exit MEASURING.