

JEOL 3010 TEM

Basic operating instructions

You have to be trained on the instrument prior to operating it on your own!

Always check the instrument log-book to determine the status of the microscope.
Check the following microscope settings:

ACCEL VOLTAGE READY light is lit.

FILAMENT READY light is lit if the sample holder is inserted.

BEAM CURRENT reader displays "072" at 200kV and "109" at 300kV.

FILAMENT is set to OFF.

Objective and SAD apertures are out.

Adjust the brightness and contrast knobs to view the CRT.

To bring the voltage to 300 kV: type RUN on the keyboard, press return. Type 200, press return. Type 300, press return. Type 30, press return. The microscope will increase the voltage gradually to 300kV. The ACCEL VOLTAGE READY light will flicker. It will take 30 min. BEAM CURRENT reader displays 109 at 300kV

Sample insertion

First time that you are working on the microscope on your own, make sure that either Lou or Evgenia is present during specimen insertion and removal procedures.

In addition to these instructions, see drawing on the last page of this manual.

Fill the liquid nitrogen trap. Make sure that the viewing screen is covered!

FILAMENT EMISSION should be off. ACCEL VOLTAGE READY light is lit.

With pin at 9 o'clock, insert the holder and push it in slightly.

Pull and lift the switch to PUMP position. Wait until the green light comes on. Wait another 7-10 min (you don't need to hold the specimen holder).

Turn the holder clockwise a little bit and let it go in. Wait for two valve sounds.

Turn the holder clockwise until it stops and guide it in (it will be pulled inside the microscope by the vacuum, don't just let it go!).

Check that the ACCEL VOLTAGE - READY lights remains on.

Microscope alignment and astigmatism correction

DO NOT ATTEMPT ANY OF THE ALIGNMENT PROCEDURES IF YOU HAVE NOT BEEN TRAINED FOR IT!!!

For beam sensitive materials, perform alignments far away from the area of interest.

All alignment procedures are done in MAG 1 or MAG 2 modes.

Press the corresponding CRS switch to change from fine to coarse controls.

Turn on the FILAMENT slowly to the position 3. **Wait for 20 min.** Turn it up slowly until you start seeing the beam. Do not saturate the beam at this stage, but perform gun alignment and condenser lens stigmatism.

1. Gun alignment (tilt) and condenser lens astigmatism correction

Converge the beam using BRIGHTNESS knob to obtain the smallest possible illumination spot (crossover point). Set MAGNIFICATION TO 80-100K. Focus the image of the filament with the BRIGHTNESS control.

Turn on the COND STIG switch (the build-in lamp brightens). Using the left and right DEF: X and Y controls, make the image of the filament as sharp as possible. Expand the beam with the BRIGHTNESS control clockwise and counterclockwise to check if it expands uniformly. Repeat the condenser stigmatism procedure if needed. Turn off the COND STIG switch when finished.

If the filament image is not symmetrical, turn on the DEFLECTOR: GUN switch on (in the drawer, the build-in lamp should light up), use DEF: X and Y knobs (in the drawer) to center the filament image (not the beam itself). Turn off the DEFLECTOR: GUN switch. If the beam has shifted from the center, use SHIFT: X and Y knobs to bring the beam to the center.

Slowly increase FILAMENT EMISSION only until the point when the filament image disappears (**DO NOT OVERSATURATE THE FILAMENT!**).

Every time you change the spot size (using the SPOT SIZE switch) during your session, you will have to adjust condenser astigmatism. You can do it by observing the shape of the beam as you go through the crossover point and then expand the beam using the BRIGHTNESS knob. The beam should be circular and should expand and converge uniformly around the center.

Switch to LOW MAG mode.

Expand the beam with BRIGHTNESS knob (if necessary) until you see the sample/grid. Choose an area for alignment (like an edge of the sample with sufficiently large hole nearby) and bring it to the center.

Switch to MAG1 or MAG2 modes. Adjust MAGNIFICATION to about 20K.

Expand the beam with BRIGHTNESS knob if necessary. (If you don't see anything, move the sample a little bit, you might be right on the grid).

Focus the image roughly using OBJ FOCUS knob (obtain minimum contrast condition).

Use OBJ FOCUS STEP switch to change the current variation per notch, 1 is the smallest increment.

If at crossover (converged beam) the beam is not in the center of the screen, to bring it to the center use the SHIFT: X and Y knobs.

2. Condenser aperture alignment

Converge the beam using the BRIGHTNESS knob and center it with the SHIFT: X and Y knobs. Expand the beam clockwise to almost cover the screen. If it expands to one side adjust the condenser aperture translations controls to center the beam. Center the beam with the SHIFT: X and Y knobs. Converge the beam and expand it counterclockwise. If it expands to one side adjust the condenser aperture translations controls to center the beam. Repeat until the beam expands symmetrically around the center of the screen.

3. Condenser lens system alignment

Set MAG to about 40 K. Switch to spot size 1. Converge the beam with BRIGHTNESS to obtain the smallest possible illumination spot (crossover). Depress the DEFLECTOR: GUN button in the drawer (the built-in lamp will light up). Center the beam with the SHIFT: X and Y knobs (in the drawer). Switch to spot size 5. Converge the beam with BRIGHTNESS. Depress the DEFLECTOR: COND button (the built-in lamp will light up). Center the beam with the SHIFT: X and Y knobs (in the drawer). Repeat this sequence until the beam doesn't shift from the center when you switch between spot size 1 and 5. Turn OFF DEFLECTOR: GUN and COND switches.

The following alignment requires presence of the sample.

4. Goniometer alignment (eucentric height)

Set MAGNIFICATION to about 30K. Move some recognizable feature of the sample towards the center of the screen. Adjust the OBJ FOCUS knob so to the value of 6.6 (OBJ on page 4 on the CRT screen). Using Z control lower or raise the sample until it is in focus. This will be very close to the eucentric height. You can check it by tilting the sample using foot pedals by about 5 degrees in both directions, the sample should remain in the center of the screen. Make sure to tilt the sample back to 0 degree when you are done. Tilt angle is displayed on the CRT screen.

Note: If you find it difficult to focus the sample using Z-control, set eucentric height in the following way: set magnification to 10-20K. Tilt the sample using foot pedals by about 5-10 degrees in both directions. The sample will move from the center. Tilt the sample back to 0. Using Z control lower or raise the sample. Repeat tilting procedure and Z adjustments until no specimen movement is observed during tilting. Repeat this procedure at higher magnification (e.g. 40-50K). When finished, tilt the sample back to 0 degree. Focus the image. Check page 4 of the CRT screen, OBJ should have a value close to 6.6.

The following alignments are done outside the specimen area.

5. Image wobbler alignment

Set MAGNIFICATION to about 60K. Using BRIGHTNESS control converge the beam and center it with the SHIFT: X and Y knobs. Depress the COND DEF ADJ - **TILT** button in the drawer. Set the COND DEF ADJ - **TILT** switch to X. The beam will split into two. Merge the two beams together and minimize wobbling using SHIFT: X and DEF: X knobs. Set the COND DEF ADJ - **TILT** switch to Y. Merge the two beams

together and minimize wobbling using SHIFT: Y and DEF: Y knobs. Set the switch to neutral position and turn OFF the COND DEF ADJ - **TILT** button. Center the beam with the SHIFT: X and Y knobs.

The following alignment requires presence of the sample.

6. Current center alignment

Bring some recognizable feature of the sample to the center of the screen. Set MAG to about 100K and expand the beam with the BRIGHTNESS control to cover the entire screen. Focus the image roughly. Turn on the WOBBLER - OBJ switch in the drawer. The image now rotates clockwise and counterclockwise periodically. Depress the DEFLECTOR-BRIGHT TILT switch on the left panel (the built-in lamp will light up). Operate the left and right DEF: X and Y knobs to minimize image movement. Turn OFF the WOBBLER - OBJ switch. Turn OFF the DEFLECTOR-BRIGHT TILT switch.

7. Voltage center alignment

Bring some recognizable feature of the sample to the center of the screen. Increase MAGNIFICATION to about 100K. Focus the image roughly. Expand the beam, to cover the entire viewing screen. Turn on the WOBBLER - HT switch (on the right panel). The image will enlarge and contract periodically. Depress the DEFLECTOR-BRIGHT TILT switch (the built-in lamp will light up). Operate the left and right DEF: X and Y knobs to b to minimize the image movement. Turn OFF the WOBBLER - HT switch and the DEFLECTOR-BRIGHT TILT switch.

8. Objective lens astigmatism correction

Before adjusting the objective astigmatism, write down the values of OBJ STIG on the page 6 of the CRT screen. If you the image worsens after your adjustments, adjust astigmatism to the original values.

First method: Bring the thin edge of the sample to the center of the screen. Increase MAG to >200K. Insert objective aperture. Focus the sample. Use binoculars as you obtain over-, in- and under-focused image. If you observe streaking in the image (unidirectional focusing), then objective astigmatism needs to be corrected. Obtain in-focus condition. Depress the DEFLECTOR - OBJ STIG switch on the left panel (the built-in lamp will light up). Use DEF: X and Y knobs to minimize the unidirectional features in the image. Check it by changing OBJ - FOCUS. Repeat the focusing and astigmatism correction steps as needed. Turn OFF the DEFLECTOR - OBJ STIG switch.

Second method: Find a circular hole in your sample or in the carbon film and bring it to the center of the screen. Adjust MAG to about 20-80K. Focus the sample. Obtain over-, in and under-focus conditions using the OBJ-FOCUS control. Observe a Fresnel fringe around the hole in the under- (bright fringe) and over-focused (dark fringe) conditions. If the fringe width is not uniform around the hole, you need to adjust objective astigmatism. Depress the DEFLECTOR - OBJ STIG switch on the left panel (the built-in lamp will light up). Use DEF: X and Y knobs to make Fresnel fringe width uniform around the hole. Turn OFF the DEFLECTOR - OBJ STIG switch.

Microscope operation

Turn on the FILAMENT slowly to the position 3. **Wait for 20 min.** Turn it up slowly.

Switch to LOW MAG mode.

Expand the beam with BRIGHTNESS knob (if necessary) until you see the sample/grid. Choose an area of interest or an area for focusing far from the area of interest (for beam sensitive materials) and bring it to the center.

Switch to MAG1 or MAG2 mode.

Expand the beam with BRIGHTNESS knob if necessary. (If you don't see anything, move the sample a little bit, you might be right on the grid). Check MAG on the CRT display. Adjust it to the value around 10-20 K for initial observation using the SELECTOR switch.

Check what spot size you are using, the spot size is displayed on the CRT screen (smaller spot size is needed to minimize beam damage). To change the spot size, use the SPOT SIZE switch.

Focus the image roughly using OBJ FOCUS knob (obtain minimum contrast condition).

Use OBJ FOCUS STEP switch to change the current variation per notch, 1 is the smallest increment.

If at crossover (converged beam) the beam is not in the center of the screen, to bring it to the center use the SHIFT: X and Y knobs.

To change the spot size, use the SPOT SIZE switch (the spot size is displayed on the CRT screen).

If at anytime you during your session are lost and can't see anything, do the following. Take out objective and SAD apertures. Decrease MAG to about 5 -10K or go to LOW MAG mode. Spread the beam using BRIGHTNESS. Move the sample to the center: X and Y reading 0 on page 2 of the CRT screen. If you still can't see anything, get help.

Bright field image formation

Spread the beam with the BRIGHTNESS knob to cover the entire screen.

Focus the sample roughly (obtain minimum contrast condition) using the OBJ FOCUS knob.

Put in SAD aperture (this step is not necessary if you are planning to use a large objective aperture).

Switch to DIFF mode.

Put in objective aperture. For conventional bright field images, use one of the small apertures. The outline of the aperture should be sharp (do not confuse it with the outline of the central beam that is bright, the outline of the aperture is dark). Focus the outline of the aperture if needed with the DIFF FOCUS knob.

Center the objective aperture around the central beam.

Switch to MAG 1 or MAG 2 mode.

Take out SAD aperture (if you put it in).

For beam sensitive materials, put in the objective aperture directly in MAG mode to avoid prolong exposures. However, in this case only small adjustments of the aperture position should be done! If you don't see anything when you insert the objective aperture in the image mode, do not adjust its position, but follow the instructions above. For beam sensitive materials, focusing should also be done away from the area of interest. Use a smaller spot size, 3-5. To change the spot size (displayed on the CRT screen) use the SPOT SIZE switch. You can also undersaturate the filament, use a smaller condenser aperture, STEM mode or MDS mode.

Selected area diffraction (SAD)

Spread the beam using the BRIGHTNESS knob.

Insert SAD aperture and position it as needed.

Switch to DIFF mode.

To focus diffraction pattern properly, insert an objective aperture and focus the outline of the objective aperture using the DIFF FOCUS knob.

Remove the objective aperture.

To center diffraction pattern: depress the DEFLECTOR: PROJ button in the drawer (the built-in lamp will light up). Use SHIFT: X and Y knobs (in the drawer) to center diffraction pattern. Turn off the DEFLECTOR: PROJ button.

Spread the beam further using the BRIGHTNESS knob, so that the central beam becomes a very sharp spot.

Using IMAGE WOBBLER for focusing

Note that image wobbler alignment needs to be performed before using this feature.

Find the area of interest. Expand the beam to cover the entire screen. Turn on the WOBBLER - IMAGE: X or/and Y switches. The image will split into two. Using OBJ-FOCUS controls merge the two images together. Turn OFF the WOBBLER - IMAGE: X and Y switches.

Taking pictures

Select automatic or manual exposure time on Page 1 of the CRT screen by pressing the SHUTTER button (A or M is displayed). The full screen or the small screen can be used to estimate the exposure time. For automatic film advance press FILM ADVANCE AUTO button (the built-in lamp will light up). Cover the screen and turn off lights.

For automatic film advance: press PHOTO button once, wait for the green exposure light to come on and off.

For manual plate advance: press PHOTO button, wait until it lights up, then press it again and observe the green exposure light to come on and off.

If the camera is jammed, check both magazines. Avoid putting your hands inside the camera!

Shut down procedure

Either switch to LOW MAG mode or leave it in MAG 1 or 2 mode at around 10-20 K. Expand the beam to cover the entire screen. Take out objective and SAD apertures. Turn FILAMENT to OFF slowly. Position goniometer at 0.

Take out your sample: set the switch to "AIR" position. Pull the sample holder until it stops. Turn towards you (counterclockwise) until it stops. Pull it out just a bit more and turn towards you (counterclockwise) until it stops. Wait for 30s and remove the holder (it should come out easily, do not force it!).

If you are the last user for the day, please bring the voltage down to 200kV using the ACCEL VOLTAGE toggle switch.

Fill out the log book.

Change the camera and **reset the number of negatives to 50.**

See separate instructions for camera change.

Please note that currently we only have 1 magazine for 3010. So, after your session, replace the cassettes that you have used during your session with new film, put the magazine in the dessicator for 4-5 hours, then put in back the microscope. While it is in the dessicator, leave a note for other users on the bench of the 3010 indicating time and date that you put the magazine in the dessicator.

Usage of the double tilt holder

After inserting the double tilt holder, type "selwt 1".
Before removing the double tilt holder, type "selwt 0".

Sample insertion and removal

