

## **JEOL 100CX**

### **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:

READY light is lit.

ACCELERATING VOLTAGE is set to 100 kV (100kV push button is depressed and lit).

FILAMENT EMISSION knob is set to OFF.

CAMERA AIRLOCK OPEN light is lit (otherwise you can't take pictures, however you can start operating the microscope).

BRIGHT FIELD button is depressed.

FILM ADVANCE is set to SINGLE.

EXPOSURE SENSITIVITY knob is turned fully clockwise.

Goniometer is at 0 and locked.

Objective and SAD apertures are out.

#### **Sample insertion**

FILAMENT EMISSION should be off, goniometer at 0 and locked.

With the pin at 9 o'clock position, insert the holder until it stops and push in slightly. This will engage the pump that will evacuate the specimen chamber. The goniometer red light will lit. Wait until the light goes off, then turn the holder away from you (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 READY light 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 (2; 3; 4; 5) that do not require the presence of the sample with an empty holder. Other alignments can be done far away from the area of interest or skipped entirely. You might sacrifice the microscope performance but will be able to take images of your sample before it is destroyed by the electron beam.*

Condenser aperture is usually set in the position 2. That is suitable for most work that is usually done on this microscope. If you need to change it, please see Lou or Evgenia.

Turn on the FILAMENT EMISSION slowly.

Switch to LOW MAG mode.

Expand the beam with CONDENSER knob (if necessary) until you see the sample/grid.

Choose an area for focusing and alignment (like an edge of the sample) and bring it to the center.

Switch to MAG mode.

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

\*\*If at crossover (converged beam) the beam is not in the center of the screen, to bring it to the center use GUN ALIGNMENT: TRANS knob if you are in spot size 1 and ALIGNMENT: TRANS knob when you are in spot size 2, 3, 4 (any other than 1).

### **All alignment procedures are done in MAG mode.**

#### 1. Goniometer alignment (eucentric height)

Move some recognizable feature (like an edge of the sample) towards the center of the screen. Set MAGNIFICATION to about 20-30K. Focus the image. 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. When finished, tilt the sample back to 0 degree. Focus the image.

The following alignments are done outside the specimen area (find a hole in your sample).

#### 2. Gun alignment (tilt) and condenser lens astigmatism correction

Set MAGNIFICATION to about 10K (for spot size 1) or 20K (for spot size 2). Converge the beam using the CONDENSER knob to obtain the smallest possible illumination spot (crossover point). Undersaturate the filament slightly until you see a dark halo (filament image). Focus the image of the filament with the fine CONDENSER control.

USE CONDENSER STIGMATOR X and Y controls to make the image of the filament as sharp as possible. Expand the beam with the fine CONDENSER control clockwise and counterclockwise to check if it expands uniformly. Repeat the condenser stigmatism procedure if needed.

Center the dark halo (not the central beam itself) if needed with GUN ALIGNMENT TILT controls (gently!).

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, you will have to adjust condenser astigmatism. You can do it either by observing the shape of the beam as you go through the crossover point and then expand the beam using fine CONDENSER knob or focusing the undersaturated

image of the filament (as described above). The beam should be circular and should expand and converge uniformly around the center.

### 3. Condenser lens system alignment

Set MAGNIFICATION to about 10 - 20 K. Switch to spot size 1. Converge the beam with CONDENSER to obtain the smallest possible illumination spot (crossover). Center the beam with GUN ALIGNMENT: TRANS controls. Switch to spot size 3. Converge the beam with CONDENSER. Center the beam with ALIGNMENT TRANS controls (inside knob). Repeat this sequence until the beam doesn't shift from the center when you switch between spot size 1 and 3.

### 4. Beam deflector coil alignment

Set MAGNIFICATION to about 6-10K. Using CONDENSER control find crossover point and center the beam (see \*\*). Switch COND ALIGNMENT: WOBBLER to position X. The beam will split into two. (If you don't see the two beams, decrease MAGNIFICATION until you see the two beams; if you still can't see them, then turn the WOBBLER OFF and get help or skip this step and go to the next). Operate COMPENSATOR: X and CORRECTOR X knobs until the two beams merge.

Switch COND ALIGNMENT WOBBLER to Y position. The beam will split into two. Operate COMPENSATOR: Y and CORRECTOR Y knobs until the beam until the two spots merge. Switch COND ALIGNMENT WOBBLER to OFF position. Center the beam (see \*\*).

### 5. Image wobbler alignment

Set MAGNIFICATION to about 10K. Using CONDENSER control find crossover point and center the beam (see \*\*). Turn IMAGE WOBBLER to ON. The beam will split into two. Operate IMAGE WOBBLER: A and B knobs to merge the two beams. Turn the IMAGE WOBBLER to OFF.

The following alignment requires presence of the sample.

### 6. Current center alignment

Expand the beam to cover the entire screen using CONDENSER. Bring some recognizable feature of the sample to the center of the screen. Set Magnification to about 30-50K and focus the image roughly. Turn the outside FOCUS: MEDIUM knob 5 clicks counterclockwise. The feature will probably move away from the center of the screen. Re-center it with the left and right ALIGNMENT: TILT knobs. Turn the outside FOCUS: MEDIUM knob back to the in-focus position and then turn it 5 clicks clockwise. If the feature in the image deviates from the center, recenter it with the left and right specimen translation controls. Repeat under- and over-focusing and alignment procedure until the feature doesn't move from the center of the screen as you change focus.

\*\*If at crossover (converged beam) the beam is not in the center of the screen, to bring it to the center use GUN ALIGNMENT: TRANS knob if you are in spot size 1 and ALIGNMENT: TRANS knob when you are in spot size 2, 3, 4 (any other than 1).

### 7. Voltage center alignment

Bring some recognizable feature of the sample to the center of the screen. Increase MAGNIFICATION to about 80 - 100K. Focus the image roughly. Expand the beam, to cover the entire viewing screen. Turn HV WOBBLER ON. The image will oscillate/move around the center. Using ALIGNMENT: TILT controls minimize the movement of the image. Note that the beam will still oscillate. Turn HV WOBBLER OFF.

### 8. Objective lens astigmatism correction

**If you are unsure about objective astigmatism correction, do not adjust it. It is generally pretty good.**

First method: Bring the thin edge of the sample to the center of the screen. Increase MAGNIFICATION TO ABOUT 80 - 160K. Insert and center objective aperture (see below "Bright field image formation"). 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. Use OBJ STIGMATOR: X and Y COARSE (if astigmatism is strong) or X and Y FINE controls until you minimize the unidirectional features in the image as you go in and out of focus. Repeat the focusing and astigmatism correction steps as needed.

Second method: Find a circular hole in your sample or in the support film and bring it to the center of the screen. Adjust MAGNIFICATION to about 20-60K. Focus the sample. Obtain over-, in and under-focus conditions using MEDIUM FOCUS control. Observe a Fresnel fringe around the hole in the under- and over-focused conditions. If the fringe width is not uniform around the hole, you need to adjust objective astigmatism. Use OBJ STIGMATOR: X and Y COARSE (if astigmatism is strong) or X and Y FINE controls to make Fresnel fringe width uniform around the hole.

### Microscope operation

Turn on the FILAMENT EMISSION slowly.

Switch to LOW MAG mode.

Expand the beam with CONDENSER knob (if necessary) until you see the sample/grid.

Check what spot size you are using (smaller spot size is needed to minimize beam damage).

Choose an area of interest or an area for focusing away from the area of interest (for beam sensitive materials) and bring it to the center.

Switch to MAG mode.

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

Focus the image roughly using MEDIUM FOCUS knob (obtain minimum contrast condition). See also \*\*.

\*\*If at crossover (converged beam) the beam is not in the center of the screen, to bring it to the center use GUN ALIGNMENT: TRANS knob if you are in spot size 1 and ALIGNMENT: TRANS knob when you are in spot size 2, 3, 4 (any other than 1).

**Bright field image formation**

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

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

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

Switch to SA 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 **inside** knob of the SA/HD DIFFRACTION: CAMERA LENGTH.

Center the objective aperture around the central beam.

Switch to MAG 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, 2, 3 or 4. You can also undersaturate the filament, use smaller condenser aperture and use STEM mode (talk to Lou or Evgenia).*

**Selected area diffraction (SAD)**

Spread the beam using the CONDENSER knob.

Insert SAD aperture and position it as needed.

Switch to SA DIFF mode.

To focus diffraction pattern properly, insert an objective aperture and focus the outline of the objective aperture using the **inside** knob of the SA/HD DIFFRACTION: CAMERA LENGTH.

Remove the objective aperture.

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

**Using IMAGE WOBBLER for focusing**

Find the area of interest. Expand the beam to cover the entire screen. Turn on IMAGE WOBBLER. The image will split into two. Using FOCUS controls merge the two images together. Turn IMAGE WOBBLER OFF.

In order to obtain so-called optimum underfocus condition (for materials with low contrast), turn on OUF switch prior to using IMAGE WOBBLER. Turn it off after focusing.

### **Taking pictures**

Set shutter speed to the value between 0.5 and 8 s. Adjust CONDENSER until the green EXPOSURE light appears. Cover the screen and turn off lights. Press FILM ADVANCE button, wait for the light to come on. Lift the screen and observe the red EXP light to come on and off. Lower the screen.

**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 mode at around 10-20 K. Expand the beam to cover the entire screen. Take out objective and SAD apertures. Turn FILAMENT EMISSION to OFF slowly. Position goniometer at 0. Make sure it is locked.

### **Sample removal**

Make sure the goniometer is at 0. To take your sample out: pull the sample holder until it stops. Turn towards you (counterclockwise) until it stops. Pull it out, pressing a finger against the goniometer.

**Fill out the log book.**

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

**See separate instructions for camera change.**

**If you are lost during your session and can't see anything. Take out objective and SAD apertures. Decrease MAG to about 5 -10K or go to LOW MAG mode. Spread the beam using CONDENSER. Move around the sample. If you still can't see anything, get help.**