

## TEM: JEOL 100CX-II: Operation Instructions:

Cold System: this is how you should find the TEM as first user of the day

- DP (Diffusion Pump), HIGH VACUUM and Ready indicator lights are green.
- HT button is off. Filament Emission knob is off
- Imaging Mode: Should be on Mag.
- Specimen selector : Should be at #1
- Objective Aperture: Out
- Condenser Aperture: #2 (default position)
- Spot Size: # 2

### Startup:

Acceleration Voltage: Press HT and then sequentially increase the accelerating voltage buttons (20, 40, 60, 80, 100). Make sure the dark current stabilizes before you increase the accelerating voltage to the next step. We usually use 100kV for most material samples and 80kV for biological or organics.

### Loading Sample:

- TEM holder will be in the black case.
- Load your 3mm TEM grids. The sample closest to the tip of the sample holder is position #1. Use toothpick to open the grid hold downs. Load first grid in position # 1 (nearest the tip).  
After loading the sample, tap the holder gently to make sure the grid is secure.
- Insert sample holder in the Goniometer with the pin of the holder at 9 o'clock position.  
Apply slight pressure which initiates the vacuum process indicated by the red light (Right Side of the Goniometer). Wait until the red light turns off and you hear two valve sounds. With a firm grasp, turn the holder clockwise and guide it gently into the chamber.

### Filament Emission:

At this stage, the accelerating voltage is on and should be stable. Turn the filament emission knob gently with constant speed, while watching the emission current gauge (should increase about 15 mA), until it reaches saturation (metal stop). You should see the beam on the fluorescent screen. If not, you are imaging on the metallic part of the grid or off the edge of the grid. Select Low Mag or Scan mode to find the electron beam. This is also a good time to search for a good specimen area.

Selecting grids: To change from grid #1 to grid #2, lower the magnification and turn the thumbwheel on the column from 1 to 2. The new grid should be visible on the view screen.

Routine Alignment (Mag mode):

1 - **Condenser Alignment** (general): Using the condenser knobs spread and converge the beam through crossover (smallest illumination spot) several times. The beam should move in and out concentrically. If there is beam movement, do adjustments below, Repeat this step after alignment.

2 - **Gun Alignment**: Magnification 19 kX, Spot Size #2, bring Condenser to crossover. Undersaturate the Filament (emission current) until the image of the filament tip appears. Focus with the Condenser. Center the filament image (dark halo) with the Gun Align Tilt knobs. Focus striations with Condenser Stigmators x and y. Resaturate Filament.

3 - **Condenser Alignment**: Magnification 19 kX,

-Spot Size at 3, bring Condenser to cross-over. Center beam with Beam Alignment Trans knobs.

-Switch Spot Size to 1, bring Condenser to cross-over, center beam with Gun Align Trans knobs. Repeat until spots 1 and 3 coincide.

4 - **Condenser Aperture**: (only if beam seems to sweep when performing the step #1 above) Magnification 19 kX, Condenser Aperture at #2.

Bring Condenser to cross-over and center beam with Alignment Trans knobs. With Condenser, spread beam clockwise and center beam with Condenser Aperture x and y knobs.

Repeat until beam spreads and converges evenly with no sweep.

5 - **Condenser Stigmation**: If spot is not the same shape on both sides of crossover, adjust roundness with Condenser Stigmator x and y. Make small adjustments to spot one side then move to other side to compare until shape is similar.

6 - **Z Axis (eucentric height) Correction**: (Usually do this when you need to have specimen at Eucentric height)

Magnification  $\sim 10$  kX, focus a recognizable specimen object.

Unlock the Goniometer and tilt it  $20^\circ$  towards you. The object should remain in its position, if not, bring it back to its position using the z-adjust knob on the bottom of the Goniometer. Return to  $0^\circ$  tilt, recenter object and refocus. Repeat until the object remains in center.

7 - **Current Center Alignment**: Magnification 20-72kX, Tip: Image should not move when focusing (medium knob).

Focus an object at the center of the screen Turn medium focus knob 2-4 clicks to the left (underfocus), if the object has moved, bring it back to its position using the Alignment: Tilts knobs. Focus and re-center the object using the stage drives. Repeat the procedure until the object does not move during medium focusing.

8 - **Objective Lens Alignment** (Voltage Centering): Magnification 58 kX, center an object. Turn HV Wobbler on. Minimize image movement with the Align Tilt knobs (left and right).

9 - **Deflector Compensation Alignment** (Left side, lower panel): Magnification 19 kX, Bring Condenser to cross-over.

Switch Wobbler up to X. Converge the 2 spots with X Corrector and X Compensator knobs. Repeat procedure for Y position, using the Y Corrector and Y Compensator knobs. Bring the Wobbler position back to the center. (Caution: Y Corrector Knob is very sensitive)

10 - **Image Wobbler Alignment**: Magnification 19 kX, Focus Image, bring Condenser to cross-over. Turn the Image Wobbler on. Converge the 2 spots with the Image Wobbler A and B knobs. Switch off Im Wobbler. Center beam.

Alignment should be completed at this stage.

#### Obtaining Images Using Gatan DigitalMicrograph:

Open the Gatan DigitalMicrograph Software. Under the Video menu, select Start Diffraction. Focus on the grid. Spread the beam uniformly to a very low light level on the fluorescent screen. Insert the Camera with the toggle on the column. The image will be displayed on the TV monitor. Proceed with imaging your sample. If you prefer retract camera to view the sample on the fluorescent screen. Make use of the binoculars and the pull up view screen (green button) to locate and focus your sample. Always recenter beam with align: trans knobs after any magnification change. Adjust brightness contrast of the field of view before viewing on DigitalMicrograph. To capture images, click Start Acquire. The software prompts you to enter the magnification. Use File:Save Display As to save images to your designated folder or on your flash drive. Please do not store images on the hard drive.

#### Viewing image in the DigitalMicrograph window.

Select Start View on the DigitalMicrograph interface. A live image will appear. This mode is used for the focusing and astigmatism correction to obtain the highest resolution and to use the FFT function.

Objective Lens Stigmatism: Use Start View to obtain a live image. Select an area with a part of the field showing the carbon film on the grid. Select the dotted RectangleROI from the toolbar. Press ALT and click and drag to form a square on the clear area of the image. Open Process:Live FFT (Fast Fourier Transform) from the toolbar. Another display window opens up, converting the image into a Power spectrum. On an amorphous substrate i.e. the carbon film of the grid, the FFT should be a nice circle. If the FFT looks oblique use the objective Stigmatism fine knobs. Use one axis at a time (either X or Y knobs). Capture image. The FFT can be turned off by deleting the box on the image and closing the small FFT spectrum image.

#### Unloading Samples:

Bring specimen position to #1. Slowly turn the Filament Current knob down but do not turn off the HV. Remove the specimen holder by gently pulling the rod out to a stop (about 3 inches), then rotate the rod counter clockwise to a stop. Pull the holder completely out of the column. Remove your grids. If finished put the sample holder back in its case.

#### Shut Down Procedure:

1. Slowly turn the filament emission back to zero.
2. Press any kV button half way till you see that no light is lit on the kV.
3. Press HT button to turn the kV off.
4. Make sure the camera is retracted.
5. Login and log time usage. It is better to login at beginning of session.
6. Turn off the TV monitor and Computer monitor.