JEOL 2200FS OPERATION MANUAL

Center for Advanced Microscopy, MSU

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Note: Each button/knob position is indicated in [XX], [LP]=left panel, [RP]=right panel. [SC]=specimen control, [M1]= monitor 1, [M2]= monitor 2, [M3]=monitor 3

I. Pre-Use Check-list:(If any of the following is not right, stop and seek help)

1. Check no or only the dummy holder is inside the microscope. Check "BEAM VALVE" [LP] is OFF.
2. Check new message in file "TEM update" on desktop[M1]. Check log book for machine status.
3. Check program "Controller for JEM2200FS" and "Screen Monitor" is on [M1]. If they are not running, restart "TEMCOM" and "ScreenMonitor" icon on desktop[M1]. Once "Screen Monitor" is opened, click "Tool" button to open its control panel.
4. On "Controller"[M1], check Acceleration voltage (Acc:) is at 200kv and the emission is non-zero.
5. Check the ACD heater device is removed. *If it is still plugged in,*
   - *In "Controller" menu, open "Maintenance -> ACD & Bake" window. Select "ACD Heat" tab. If "ACD heat" is still "ON", click "OFF" or seek help.*
   - Carefully remove the ACD heating device(it is fragile and may be very hot or cold).
6. Check the all three ion gauge green light is ON(at far right-back side). *If not, go to **E**.*
7. Turn "BEAM VALVE" [LP] ON, check beam is on screen. Turn "BEAM VALVE" OFF.
8. Log in name, department, account number or class name, and filament meter starting time.

II. Routine Operation:

A. Initial Operation

1. Fill cold traps with liquid nitrogen(LN_2). Make sure to refill liquid nitrogen every 2-3 hours.
2. Remove dummy holder. Load sample and insert the sample holder. (see Sample Exchange section).
3. Wait for vacuum pressure reduce to < 2.5×10^{-5} Pa (on the lower ion gauge).
4. Turn BEAM VALVE [LP] on.
5. In "APERTURE CONT" area[LP], depress "CLA" button[LP], then depress the desired condenser aperture button (#2 is recommended).
6. In "Controller" menu, open "Dialogue -> Filter Control" window, press "Degauss" and wait for finish.

B. Image Observation

7. Find electron beam and sample.
   If no electron beam on "Screen Monitor", or partially blocked, proceed the following:
   a. Check "Screen monitor" is in "play" mode, "MON SCRN" button[RP] is on. Check "TEM" [LP] and "MAG-1" [RP] are on. Check "Slit"[LP], "ENGY SHIFT"[LP] and "SPCTR"[RP] are off. Set "MAG/CAM L" [RP] to X30K. Set to SPOT SIZE[LP] to 2.
   b. On "Screen Monitor", in "Camera Control" tab, set "binning" to 2x2, adjust exposure to the left of the 60ms mark. In "Brightness/Contrast", click "Adjust", enable "auto-adjust". In "Gamma"(G:) click "Reset", disable "auto-adjust". [Note: this may need re-check later].
   c. Low magnification mode sample search:

- Turn on "LOW MAG" [RP]. Set "MAG/CAM L" to ~X500.
- When beam is found, center beam with SHIFT-X[LP] and SHIFT-Y [RP].
- Search sample use stage SHIFT[SC] or track ball and center it.
- Increase magnification to X1500. Center area of interest.
- Turn on "MAG 1"[RP].

8. Correct sample Z height:
   - Set image magnification to ~X30K. Press "STD FOCUS" [RP] once.
   - Turn on either "IMAGE WOBB X" or "Y" [RP], image will split in two.
   - Turn on CRS[SC], adjust the Z [SC] to make split image merge into one(image in focus).
   - Turn off "IMAGE WOBB X" or "Y".
9. Align the microscope (See Basic Alignment)
10. To save picture and diffraction pattern, use ScreenMonitor(see Digital Camera A)
11. For taking high resolution images, use Gatan Multiscan Cammera(see Digital Camera B)
12. Take pictures with negative film when needed (see Film Exposure)

C. Return to Standby

13. When ready to stop, stop MSC camera and turn off "MSC" button[RP].
14. Set Magnification to around 30K.
15. Spread beam to cover the entire screen.
16. In "Controller", double click "Stage Neutral" (at lower right corner). Wait for all settings go to nearly zero(<1μm). If stage does not go to zero, double click "Stage Neutral" again.
17. Turn off BEAM VALVE [LP] – (light off).
18. Remove ALL apertures by depress OPEN for each aperture.
19. Remove sample holder (see Sample Exchange). Then insert the dummy holder.
20. Logout with filament time and number of negatives taken.

D. Overnight Standby

If you are the last person of the day before 5pm, perform the following:

a. Make sure the dummy holder is inserted and the green light is on.
b. Insert the ACD heating device.
c. In "Controller" menu, select "Maintenance -> ACD & Bake". In "Bake Out/ACD Heat" window, select ACD Heat tab, press ON button. Confirm "Yes" or "Okay" in prompted window.

Note: Always perform "Overnight Standby" if you are an after hour user.

E. Ion Gauge Reset

If the lower ion gauge green light is off, Open "Maintenance -> ACD & Bake" window, check either:

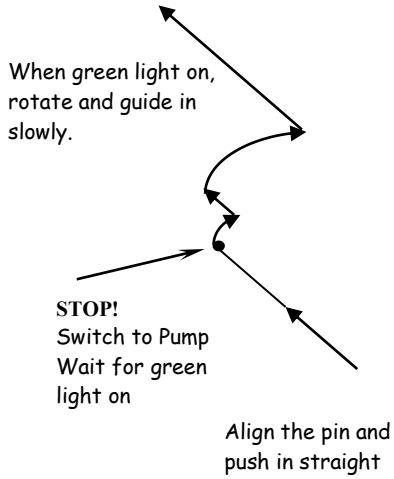
a. If the "ACD heat" is "ON", turn it "OFF", wait the ion gauge light to come back on. *(Depend on the stage of the ACD heat, this may takes from a few second to up to 2 hours.)*
b. If the "ACD heat" is already "OFF" and usually accompanied by a valve click sound, you may reset the ion gauge by turning the switch off and on again. If doesn't, seek help.
c. Wait for vacuum pressure reduce to < 2.5×10^{-5} Pa .

F. HT Standby:

If the voltage is at 160kv, then in "Controller" menu, open "Dialogue -> High Voltage Control" window, check "Stand by" is depressed. Press "Normal" and then click "Yes" in prompted window to confirm to raise voltage automatically to 200kv(about 13-20min).

III. Sample Exchange

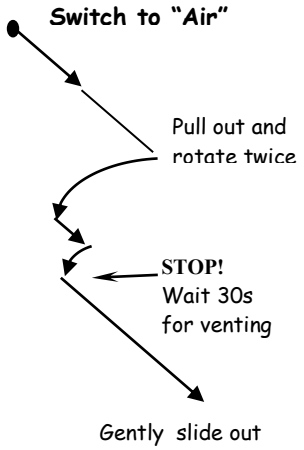
1. **Sample Holder Insertion**
   a. Load specimen on holder and secure sample properly. Make sure there is no dust on o-ring.
   b. Align the holder guide pin to the notch at 9 o'clock position, without any rotation, insert sample rod straight in to the end and **STOP!**. DO NOT TURN!
   c. **Set goniometer PUMP/AIR switch to "PUMP"**. If the orange lamp does not light, slightly turn the handle **counter-clockwise** and hold for ~15s and wait for the pump to start.
   d. **Once pump started, release hands, wait ~3min for the GREEN lamp lights up.**
   Note: if the green lamp does not lights up after 3min, open "Monitor->vacuum status" window and check specimen vacuum.



Insertion

   e. When green light lit, turn specimen rod clockwise fully (about 30°) and it will automatically being inserted about 5mm into the column.
   Note: The column vacuum tends to strongly suck the holder in the following steps. Therefore it is recommended to hold the holder with the both hand to prevent losing control.
   f. Slowly turn clockwise another 60° to stop. With control, slowly let the holder rod slide into the column while pay special attention at the last one inch. Make sure the plastic tab on the right side is aligned with the slot.
   g. In "Controller", select the appropriate holder type(e.g JEOL single tilt holder).

2. **Sample Holder Removal:**
   a. Check the air-lock between gun and column is closed (BEAM VALVE [LP] is dimmed)
   b. In "Controller" , double click "Stage Neutral". Wait for all settings go to nearly zero. If stage does not go to zero(<1μm), double click "Stage Neutral" again.
   c. Turn goniometer PUMP/AIR switch to AIR. --- Very Important!!!
   d. Pull specimen rod straight out slowly and firmly to the end.
   Note: If the microscope beeps and/or feel unusual resistance during pulling, return sample holder back to the column immediately. Check for errors.
   e. Turn specimen rod about 60° anti-clockwise to the stop(do not over turning!).
   ***Note:* While Pulling, Do not Turn! While Turning, Do not Pull!**



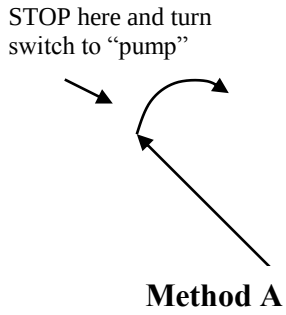
Removal

   f. Pull specimen rod out to the end(about 5mm) and then anti-clockwise about 30° and **STOP**!
   g. Check goniometer PUMP/AIR switched to AIR, if not, turn it to AIR.
   h. Wait for venting the sample chamber. Pull specimen rod straight out carefully.
   i. Place the holder on the stand and remove the sample.

3. **Dummy Holder Insertion/Removal:**

Insertion :

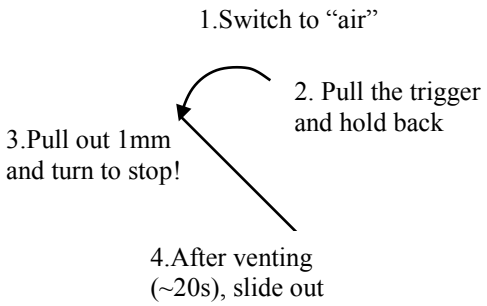
a. Align the holder guide pin to the notch at the 9 o'clock position. Push straight to the end.
b. Hold the handle by slightly turning it counter-clockwise. Turn the goniometer PUMP/AIR switch to "PUMP". Wait for the pump to start.
c. After the green light lit, turn holder clockwise slowly (about 30°) until the tab clicks in.



Method A

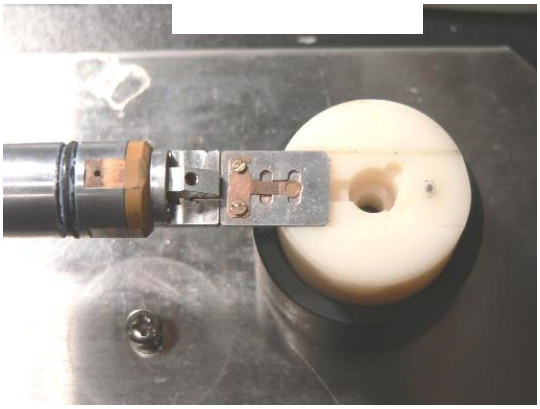
Removal:

a. Turn the goniometer PUMP/AIR switch to “Air”.
b. Pull and hold back the pin on the right hand side.
c. Turn the holder counter-clockwiseto the stop(about 30°) .
d. Wait for 20s for the sample for the air lock to vent.
e. Carefully pull the dummy holder straight out.
f. Rest the dummy holder vertically on the table.



4. Sample Loading (JEOL single tilt holder) :

a. Put sample holder on support.
b. Unscrew two screws half way, do not remove them completely.
c. Lift the holding copper T-plate and swing anticlockwise to expose grid area
d. Load TEM grid, use washer if needed.
e. Swing back the copper holding T-plate, **make sure it sits down into the groove**.
f. Retighten two screw snuggly, **do not over tightening**.



IV. Cameras

A. Digital imaging with Screen Monitor

1. On "Screen Monitor"[M1], click "camera" button to save the display, or go to menu -> Operation->Snapshot.
2. In new pop-up image window, click "save" to save file to desired folder, then close this window. Note: the normal folder is "User(Gatan)" on desktop, and 8-bit bitmap format is recommend.
3. Remove background gain reference defect(optional)
   Take a gain reference picture: Find an empty area without any sample, take a picture with "camera" button.

   [Optional] Remove background gain reference defect on "Screen monitor"
   - Take normal pictures as above but use the green "Save original" button (e.g. as "Img01.bmp").
   - Find an empty area without any sample, take a picture.
   - Save this image with the *green* "Save original" button and save as "Ref.bmp
   - Open both "Ref.bmp" and "Img01.bmp" in Gatan Digital Micrograph.
   - Select menu "Process -> Simple Math". In the "Simple Math" window, at "operation" section, select "a/b". Select "a" to be "Img01.bmp" and "b" to be "Ref.bmp", click OK.

B. Gatan Digital Multi-Scan Camera(MSC)

1. **Do NOT use this digital camera in SA-DIFF mode!**
2. Make sure it is on MAG1 mode and beam is dispersed to the size of the screen [M1].
3. Start DigitalMicrograph[M3] software if not already open.
4. In DigitalMicrograph, check the CAMERA VIEW "Camera Acquire" windows are at at the righthand side. If you can not find it, do the following:
   - In "Window" drop-down menu, choose "Layout manager->1. Basic TEM".
5. Press MSC button [RP].
6. In DigitalMicrograph CAMERA VIEW window, press "Start View" box. Click "Yes" if prompt for "inserting camera?".
7. A live image should appear. While changing magnification, check scale bar should change correspondently. If not, restart DigitalMicrograph.
8. Press "Stop View" to stop live view.
9. When ready to take a picture. Press "Start Acquire" in "Camera Acquire" window.
10. Click "Stop Acquire" if it is in continuous acquiring mode.
11. Save image in the desired folder or disk.
12. When finish, make sure to "Stop View".
13. Close all image windows.
14. Turn off MSC[RP] to return to view with "Screen Monitor" [M1].

    [Optional] **One-click "saving" procedure:**
    - Click the toolbox, then select "Save numbered", type or choose your subdirectory, give the name of the sample, reset counter to 1.
    - For any image that is need to be quick saved, click "save numbered", then the image will be saved as "filename-000counter" in your preset directory. The counter will auto advance.

V. Basic Alignment

1. Correct the condenser lens astigmatism(recommended at ~200K).
   a. Using BRIGHTNESS [LP], converge the electron beam to half of the screen size. (If it is too bright, reduce exposure time if necessary).
   b. Center beam with SHIFT-X[LP] and SHIFT-Y [RP].
   c. If the beam is *elliptical*, correct the condenser astigmatism as follow
   i. turn on the COND STIG switch[LP];
   ii. **make the beam round with DEF/STIG-X[LP] and DEF/STIG-Y [RP];**
   iii. **turn off the COND STIG switch[LP].**
2. Center condenser aperture (recommended at ~200K)
   a. *Try to complete the following steps (i-iii) within 30sec*
      i. Using BRIGHTNESS [LP], converge the electron beam to minimum size.
      ii. Using the SHIFT-X [LP] and SHIFT-Y [RP], move the beam to the center of the screen.
      iii. Turn BRIGHTNESS clockwise to expand the beam to a larger circle.
   b. Use the aperture control X/YX/Y[LP] to move this large circle to center.
   c. Repeat from step 1(d) if beam is elliptical again.
3. Carry out current center alignment.
   a. Set the magnification to X30K with MAG/CAM L[RP]
   b. Expand the beam with the BRIGHTNESS [LP] to cover the entire screen.
   c. Find a sample area with distinctive feature.
   d. Turn on BRIGHT TILT [LP].
   e. Turn on OBJ-WOBB [RP]. (The image will oscillate)
   f. Adjust the DEF/STIG-X [LP] and DEF/STIG-Y [RP] so that the center part of the image stay stationary(or expand concentrically).
   g. Turn off OBJ-WOBB [RP]. Turn off BRIGHT TILT [LP]
4. Carry out the voltage center alignment (for high magnification imaging).
   a. Set the magnification to X200K using MAG/CAM L[RP].
   b. Adjust BRIGHTNESS so that the electron beam covers the entire screen.
   c. Turn on BRIGHT TILT [LP].
   d. Turn on HT-WOBB [RP]. (The image will oscillate)
   d. Adjust the DEF/STIG-X [LP] and DEF/STIG-Y [RP] so that the center part of the image stay stationary(or expand concentrically).
   e. Turn off the HT-WOBB [RP]. Turn off BRIGHT TILT [LP].

VII. X-ray Energy Dispersive Spectroscopy

Pre-Use Check:

Make sure both EDS control unit lights are GREEN. If not, do the following:

a) On desktop, double click icon "INCATidy-up". 
Alternatively click: Start->Program->Oxford Instrument->Tools->INCA Tidy-up.

b) In the opened window, click "OK", wait internal reboot and the two light turn to green.

A. EDS setups:

1. Double click INCA icon  [M2] . It may take a couple of minute to start.
2. Find an interesting area with a distinctive feature (e.g. a particle).
3. Try to converge the beam to one particle only. If camera gets saturated, increase the magnification, or retract the camera.
4. Select "Project" Box, enter project name and comments(optional).
5. Select "Sample" Box, enter sample name and comments(optional). Select element to be de-convoluted in the spectrum, e.g. select Cu if you are using Cu-grid.
6. In "Option" menu, select "Detector control". Once the detector control window opened, click "Open" in "shutter" page. This will open the detector shutter(you may need to click twice).
Note: When detector is overloaded, the shutter will be closed automatically. Reduce the X-ray intensity by either reducing the electron bean intensity(spot size) or changing the sample position. Then click "Open" in "shutter" page. Detector overload happens when electron beam strike thick sample, edge of grid or go to LOW MAG. Therefore close shutter before move the sample.
7. Select "Analyzer" in INCA window(*lower left corner*).

B. EDS spectrum acquisition:

8. Select "Microscope Setup" Box, click on "cyclic" button to check "live" spectrum. Change beam intensity or sample position to make the dead time to the optimal value < 50%. Change beam intensity and sample position when necessary. Click "stop" button to finish.
9. In Acquire Box, click the acquire button (green circle) to acquire spectrum. Click the stop button (red square) to stop the acquisition if early finishing is desired.

C. EDS spectrum analysis:

10. Go to "Confirm Element" Box, verify element in spectrum.
11. Record the corresponding images using either "Screen Monitor" or Gatan MSC camera.
12. Quantify elements in the sample. Determine elemental composition in sample.
13. Put results together in "Report". Use template: Spectrum/Compare Spectrum"
14. Export the report in Word format. Insert the image in the empty area. Indicate each spectrum collection area on the image.
15. Put results together in "Report". Use template: Spectrum/Quantify Spectrum."
16. Export the report in Word format. Insert the image in the empty area.
17. Save the project in your own directory.
When finish EDS,
18. In INCA "option->detector control" menu, click "Close" in "shutter" page. Quit INCA
19. Depress "TEM"[LP] to return to "TEM" mode.
20. Return TEM to standby. Remove sample.

VII. Emergency Shut Down

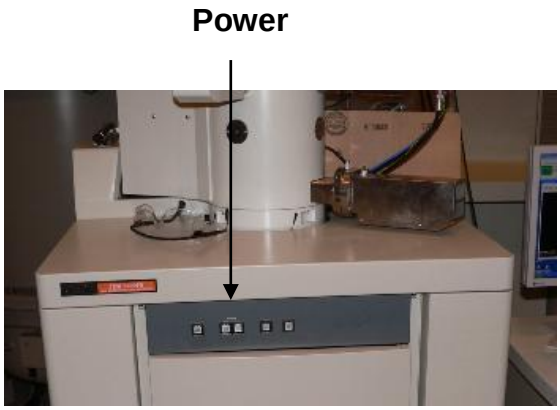
If you have any doubt about the emergency procedure, LEAVE IT ALONE!

In any of these emergency situation, first close the "BEAM VALVE" immediately!

A. In case of emergency such as cooling water interruption or power failure:

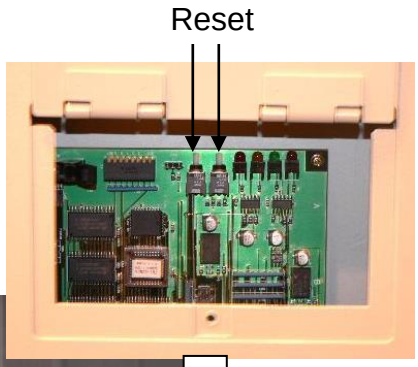
Note: this microscope is equipped with an uninterrupted power supply(UPS) system, therefore there should be enough time to shutdown regularly in case of normal power failure.

1. Notify CAM staff immediately if you can, otherwise proceed as follow:
2. In "Controller" menu, open "Dialogue -> High Voltage Control" window, click "more >> " button, the lower part of window appears, then click "Turn Off".
3. **Wait until high voltage and emission drop to 0.**
4. Turn POWER switch key to O (off position).
5. Make notes in the logbook.



B. In case of microscope auto-shutdown due to mishandling or malfunction:

1. Notify CAM staff immediately if you can, otherwise proceed as follow:
2. Wait for the auto-shutdown to complete.
3. Depress POWER switch "O".
4. If you can make sure that power and water supply is okay, then depress POWER switch "I".
5. Make notes in the logbook and notify CAM staff member.



C. In case of stage lock up due to exceeding limit:

1. A "stage limit" window should pop up.
2. Turn off "BEAM VALVE" if you can.
3. Wait for 2 minutes try to click "OK" to reset.
4. If it does not reset, perform a "hard reset":
   - At the back panel, find the window.
   - Press reset buttons inside
5. Wait for 2 minutes for panel light to go off and come back again.
6. Re-open/close BEAM VALVE twice.

D. If sample holder would not reset to zero before removal after repeatedly click "Holder exchange", DO NOT PROCEED. Just close the BEAM VALVE and leave the holder inside. Make note and notify CAM staff member.