

J2100F Daily Operation Guide

Last modified on Jan. 17 2007 by Dr. Shuyou Li

Note: This guide is NOT complete operation manual of J2100F. It is only for your daily operation quick reference. For detailed operation manual, please refer to <http://www.nuance.northwestern.edu/epic/manuals.htm>

Remember:

1. Please send me a copy of your publication (e-format preferred) if you have TEM results taken with this microscope.
- 2. In emergency, close gun valve by pressing the “Beam” button at the upper-left corner of left control panel.**

BEFORE YOU START

- Check conditions of J2100F before login FOM access control.
 - Left-bottom rack: Vacuum meter $<3 \times 10^{-5}$ Pa with blue scale
 - JEOL PC (right): Vacuum System (TEMCON – upper left corner – VAC), V1=OFF, V2=OFF, PIG1<30, PIG4<35, PIG3<45, PIG5<65, PIG6<40. (PIG4 may be over 200 if dummy holder is not in. This is ok.)
 - JEOL PC: Acc.=160kV or 200kV, Emission ~144uA, TEM Spot=1, Alpha=3, X=Y=Z=0, TX=0 (TY doesn't matter)
- Check logbook to see notes by previous user. Write your name / specimen info on logbook.

START-UP

1. Fill LN2, wait one minute till boiling then top off LN2 again. (Take out ACD heater if you are the 1st user of the day)
2. Log in Access Control with FOM.
3. JEOL PC: HV control window (TEMCOIN – upper left corner – HT): turn on to Normal (200kV) if HV=160kV.

Load specimen

1. Vent Dry Pumping Station: Make sure DPS is power off. Close V2, open V1, turn leak valve CCW until chamber is vented.
2. Take holder out of DPS, put holder on sample loading station, with appropriate holder support lying underneath hold tip.
3. For single tilt holder, loosen two screws TWO turns, rotate the cover plate away and mount specimen with film side UP. For double tilt holder, loosen two screws TWO turns, rotate the two plate-clamping fingers and mount sample facing DOWN. NEVER OVERTIGHTEN THE SCREWS!!!
4. Remove dummy specimen holder from goniometer if it is inserted.
5. Check O-ring and clean with duster if necessary.
6. Align holder guide pin with the guide groove on the goniometer, push till it stops.
7. Keep pushing the holder in position, pull PUMP/AIR switch on goniometer and turn it up to PUMP. Do not release your hand until PIG4 goes below 200. For double tilt holder, put the cable across the goniometer cover to prevent rotation of sample holder due to cable weight.
8. Wait two minutes till PIG4 is just below 40.
9. Turn holder clockwise and insert specimen holder into the goniometer (follow the chart on TEM column). Try to insert smoothly and gently. Never use force side ways!
10. Wait till column pressure is below 1.3×10^{-5} Pa and PIG4 is below 37.
11. JEOL PC: TEMCON – upper right corner – select “EM-21010/21020” for single tilt holder or 31630 for double tilt holder.

TEM alignment

1. Press Beam button (left panel) to open the gun valve.
2. Press STD focus (right panel) and make sure OL current is 4.31 (CRT at right side of column).
3. Select CL 2 (left panel, Aperture Controls), center the CL2 aperture with the x-y controls.
4. Change Magnification and Brightness to check if the beam spreads/shrinks uniformly and remains round and approximately centered well. If TEM alignment is too bad, load a recent alignment file (JEOLPC: Maintenance menu – alignment – load alignment file (bottom of the window))
5. Find sample. Align sample eucentric height using Z up/down buttons (right panel).
Note: Move sample SLOWLY when trying to find sample in low-mag mode! You may turn on PIEZO when you want to move sample under very high magnification. Remember to turn it off after use!
6. This step is for advanced user only. Do NOT try if you are not confident doing it!
 - a. Check/Perform Gun tilt alignment. Anode Wobble & gun DEF at 40kx.
 - b. Check/Perform Gun shift alignment. 1G-5C at 40kx.
 - c. Check/Perform tilt balance alignment. Compensator Tilt X and Angle, tilt Y and Angle.
 - d. Check/Perform shift balance alignment. Compensator Shift X and Angle, Shift Y and Angle.
7. Turn on HT wobbler (right panel), and perform HT centering with CLA (Brightness) TILT under 40kx.

TEM imaging with upper camera

Note: Never use OL aperture before talking to me! You may use HC aperture to improve image contrast.

1. Gatan PC: Start INCA, FilterControl and then DigitalMicrograph (DM) software if they are not started.
2. DM – Change Layouts (upright corner) to TEM. Find and expand Camera View and Camera Acquire tabs.
3. DM – Camera menu – Camera – select Upper Camera.
4. Center area of interest to the center of viewing screen.
5. DM – Camera Ciew tab – Check “insert camera” checkbox to insert camera, check Auto Exposure and click Start View.
6. Adjust brightness and focusing if necessary. Insert HC aperture to enhance contrast if necessary.

7. Check Auto exposure in Camera Acquire tab and click Start Acquire. To use manual exposure, uncheck auto exposure.
 8. To save image in DM3 format, go to File menu – Save As. You may temporarily save your file in MyDocuments\Users folder during experiment. After experiment please MOVE your files to TEMServer (Z:\) under folder with your own name. Any file saved in public folder will be deleted right away. MyDocuments\Users\ folder is also dumped regularly.
 9. To save image in other formats, choose Save Display As.
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Selected Area Diffraction (SAD)

1. Align microscope under image mode. Turn off GIF mode (F6) if necessary.
 2. Center area of interest to center of screen. Spread beam with Brightness knob.
 3. Press SA and desired size number (left panel) to insert SAD aperture.
 4. Press SA DIFF button (right panel) and spread beam to have smallest transmitted spot.
 5. Use MAG knob to adjust camera length.
 6. If the transmitted spot is not at center of screen, press PLA button (left panel) and use shift X and Y knobs to center it.
 7. Press MAG1 button (right panel) to return to image mode as soon as all adjustment are done in order to avoid beam damage.
 8. If you want to record SAD pattern with CCD camera, go to DM – Camera menu – Camera – select upper camera. In Camera Acquire panel (DM right side), uncheck Auto Exposure (NEVER use auto exposure for SAD), set exposure time to 0.1 second, click insert camera checkbox to insert camera, and then click acquire. Increase exposure time and re-acquire if necessary. Uncheck insert camera to take camera out and return to image mode as soon as acquisition is complete.
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EDS spot analysis

1. Align microscope under image mode.
 2. If top-hat aperture is to be used, go to TEM 6000x, insert top-hat aperture and center it. Do not adjust aperture knobs too much otherwise it may be damaged!
 3. Focus beam to area of interest, which should be thin.
 4. Go to INCA – Option menu – Detector Control – Shutter and open shutter.
 5. Follow normal INCA procedure to obtain EDS spectra. You may also use Spectral Acquisition in DM to obtain EDS.
 6. Make sure that dead time (<35%), sample thickness, CL aperture and sample orientation are suitable.
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GIF alignment and imaging

1. Align microscope under image mode.
 2. Start INCA, FilterControl and then DigitalMicrograph (DM) software if they are not started.
 3. DM – Change Layouts (upright corner) to TEM. Find and expand Camera View and Camera Acquire tabs.
 4. DM – Camera menu – Camera – select GIF camera.
 5. Move sample hole to screen center, focus beam to ~10mm, center it to GIF entrance aperture.
 6. Press F6 (right panel) to switch to GIF mode.
 7. Press F1 (right panel) to raise screen.
 8. Make sure Technique-TEM is selected in AutoFilter of DM. Expand Camera View tab in DM and click Start View to start image view. Check Auto Exposure time or use manual exposure time and adjust exposure time with up or down arrow keys.
 9. Click AlignZLP (1min), and then click TuneGIF (5min).
 10. Find amorphous area on specimen, click Camera View and Start to view CCD image. Use live reduced FFT (process menu) to correct obj stigmatism.
 11. Expand Camera Acquire tab and click Start Acquire button to record image. Check auto exposure time or uncheck auto exposure and adjust time manually. Save image on your own folder in DM3 format.
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EFTEM (energy filtered imaging)


1. Zero-loss imaging: choose slit width (in FilterControl, ~20eV). Insert slit. If image disappears redo Align ZLP.
 2. Low loss imaging: With slit in, change Energy Offset to a desired value (20-70eV). Refocus image if necessary.
 3. Elemental mapping: Align ZLP again. Hold Alt key, Click Filter menu-Elemental mapping in DM window.
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EELS in imaging mode

1. Click EELS to switch to spectrum mode.
 2. Select desired dispersion and entrance aperture size.
 3. Make sure exposure time is minimum 0.02 second, click Idle button to view EELS on PC.
 4. Adjust Focus X, Focus Y, ACComp to get narrowest ZLP.
 5. Change exposure time with up and down arrow keys to get enough counts and keep spectrum in green. Hit spacebar immediately if the spectrum turns into yellow or red.
 6. Change Energy offset for core-loss spectroscopy.
 7. Alt-click Acquire button to change acquire time. Click Acquire to record spectrum.
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STEM mode Alignment

1. Align microscope under image mode and make sure sample is at eucentric height and Mag >= 250kx.
2. Start INCA, FilterControl and then DigitalMicrograph (DM) software if they are not started.
3. DM – Change Layouts (upright corner) to STEM. Find and expand DigiScan and HAADF control tabs.
4. Make sure CCD camera is selected by choosing GIF camera under Camera menu.
5. Take out any aperture you are previously using except the CL aperture #2.
6. Start ASID PC-TEM software on JEOL PC.
7. Open Sirius Client software if they are not running. (username: basic, password: b)

8. Press STD Focus button (right panel)
9. Click STEM button in Detector part of Sirius Client, wait until Sirius Client turns into STEM control interface.
10. In Sirius Client, select camera length = 40cm, and click RONCHI button. Change SMMAG to 200Kx. Observe ronchigram on TEM screen with binocular. Adjust sample height if image is seen instead of ronchigram. Center ronchigram to center of screen with PLA DEF knobs. Center CL aperture relative to the ronchigram. Adjust CLA STIG to make ronchigram round.
11. In Sirius Client, select appropriate probe size, 1nm for microanalysis, smaller probe size for dedicated STEM imaging, and camera length (2cm typical).
12. Make sure STEM disk is at middle of screen (9 O'Clock, 5 mm away from center). If not, center it using PLA (left panel) and DEF/STIG knobs.
13. If beam rastering motion is observed, go to ASID PC-TEM control panel and turn off Normal Scan Mode (.
14. Press F1 (right panel) to lift up screen.
15. DM – Camera View tab – Start View, you should be able to see portion of the Ronchigram in the image window.
16. Center Ronchigram using PLA + DEF/STIG, correct astigmatism using CLA STIG + DEF/STIG.
17. Insert appropriate CL aperture (#3 typical) and center it.
18. In Sirius Client software, change Detector to EXT (Gatan HAADF detector).
19. DM – DigiScan – Search or Review – Start, you should see STEM image. If necessary, go to DM – HAADF control – Auto PMT Gain to optimize image contrast.
20. Adjust fine focus (right panel) and click DigiScan – Record to record STEM image. If necessary, click DigiScan setup button at lower right corner of the tab to change parameters for search/preview/record.
21. Click EM in Sirius Client to switch back to TEM mode after using STEM.

EELS and EDS in STEM/nanodiffraction mode

1. Stop STEM scanning by clicking Stop button in DigiScan tab. Check Control Beam checkbox in DigiScan. Move beam position with mouse in STEM image.
2. Switch to EELS in AutoFilter control tab (DM). Set Turbo exposure time to 0.001s, set dispersion, aperture size and start acquisition.
3. Increase Camera Length in Sirius Client or use smaller condenser aperture if EELS intensity is too high.
4. Use Spectral Acquisition (DM) or INCA software to collect EDS spectra.

Spectrum Imaging or Spectrum linescan

1. Get an STEM image.
2. Go to DM – Spectrum Imaging tab, click Assign Image.
3. Draw a line or a subarea for spectrum imaging (collect set of EELS/EDS spectra), click Assign ROI.
4. Click SI setup button at lower-right corner of Spectrum Imaging tab, set parameters for Spectrum Imaging. Note the total time needed for full set collection.
5. Click Start button to start SI imaging.

Change specimen and shutdown

1. Remove any aperture that was used during your session except the CL aperture #2.
2. Turn off Beam valve (left panel).
3. JEOLPC: TEMCON – Double click black **StageNeutral** button to neutralize sample position.
4. Make sure sample shifts and tilts are zero, then remove specimen holder from the microscope.
5. Load new specimen if you want, or take specimen out of holder and store the holder in Dry Pumping Station.
6. Login FOM and see if there is user after you today. Log off your session in FOM.
 - a. If you are not the last user of the day, follow this procedure:
 - i. Refill LN2
 - ii. Insert dummy sample holder. Align holder guide pin with the guide groove on the goniometer, push till it stops. Keep pushing it for about one minute. Observe on Vacuum diagram V34 and V36 open and then close.
 - iii. Now pull PUMP/AIR switch on goniometer and turn it up to PUMP.
 - iv. Wait two minutes till PIG4 is just below 40. Then turn the dummy holder 15 degrees clockwise till it stops.
 - v. Make sure that V8, V26 and V21 are lit before you leave.
 - b. If you are the last user of the day, follow this procedure:
 - i. Click Stand-By button to bring HT down to 160kV. (Dialog menu – HV Control if the window is not open).
 - ii. Check column pressure when no sample holder is in the TEM. If the column pressure increases, you must follow steps iii-viii to recover column pressure. If the column pressure does not change, you may do step iii only.
 - iii. 0
 - iv. Wait till ACD HEAT turns to Off after about two hours (DONOT insert dummy holder in before ACD heat finishes!)
 - v. Insert dummy sample holder. Align holder guide pin with the guide groove on the goniometer, push till it stops. Keep pushing it for about one minute. Observe on Vacuum diagram V34 and V36 open and then close.
 - vi. Now pull PUMP/AIR switch on goniometer and turn it up to PUMP.
 - vii. Wait two minutes till PIG4 is just below 40. Then turn the dummy holder 15 degrees clockwise till it stops.
 - viii. Make sure that V8, V26 and V21 are lit before you leave.