

JEOL 2100F Daily Operation Guide

Last modified on October 22, 2017

Note: This guide is NOT complete operation manual of JEOL 2100F. It is only for your daily operation quick reference.

Remember:

1. Please acknowledge NUANCE-EPIC facilities in your publication if you have TEM results taken with this microscope.

2. In emergency, close gun valve by pressing the **BEAM button at the top-left corner of left control panel.**

BEFORE YOU START

- Check conditions of JEOL 2100F before login NUcore access control.
 - Left-bottom rack: Vacuum meter $<3 \times 10^{-5}$ Pa with blue scale
 - JEOL PC (right): Vacuum (TEMCON – VAC), V1=OFF, V2=OFF, PIG1<35, PIG4~255, PIG3<45, PIG5<65, PIG6<40.
 - JEOL PC: Acc.= 200kV, Emission ~180uA, TEM Spot=1, Alpha=3, X=Y=Z=0, TX=0 (TY doesn't matter)

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 through NUcore. (<https://nucore.northwestern.edu/facilities/nuance>)
3. Start the computers if they are not running.
 - a. Gatan PC (middle): start FilterControl, then DigitalMicrograph (DM), and Sirius (username: admin, password: sirius)
 - b. JEOL PC (right): start TEMCON
 - c. Oxford PC (left): start EDS Remote Server, and click connect; then start INCA (**Only if you will use EDS**)

Load specimen

1. Vent Fischione Pumping Station (in Room JG20) and take the holder out, then make sure ALL valves are CLOSED.
2. Put the 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. Check O-ring and clean with air blow or clean gloves if necessary (NO NOT USE TWEEZER OR CHARPS).
5. Align holder guide pin with the guide groove on the goniometer, push till it stops (**DO NOT TURN!!**).
6. Keep pushing the holder in position, pull PUMP/AIR switch on goniometer and turn it up to PUMP. For double tilt holder, put the cable across the goniometer cover to prevent rotation of sample holder due to cable weight.
7. **Wait (DO NOT TURN!!) until the **GREEN** LED on goniometer turns on and PIG4 is below 35.**
8. Turn holder clockwise in two steps and insert specimen holder into the goniometer (**follow the chart on TEM column!!**). Try to insert smoothly and gently. Never use force sideways! Connect the cable to goniometer if you use double-tilt holder.
9. Wait till column pressure is below 1.3×10^{-5} Pa (lower-left panel).
10. JEOL PC: TEMCON – upper right corner – select “EM-21010/21020” for single-tilt holder, 31630 for double-tilt holder, or 31640 for low background Be double-tilt holder. **MAKE SURE YOU SELECT THE CORRECT HOLDER!!!**

TEM alignment

1. Press **BEAM** button (left panel) to open the gun valve.
2. Make sure the operation mode is TEM MAG mode (TEMCON or CRT); if not, press **MAG 1** button.

- Decrease magnification knob and move sample around if cannot see the beam. Go to **LOW MAG** mode to find the beam if necessary.
3. Press **STD FOCUS** (right panel) and make sure OL current is 4.31 (CRT at right side of column).
 4. Select **CL-2** (left panel, Aperture Controls). Adjust BRIGHTNESS knob to get the smallest beam, use SHIFT X&Y knobs to center the beam; spread beam, use CL-2 aperture x-y knobs to center the beam again.
 5. Condenser stigmatism: select **COND STIG** and adjust DEF/STIG X&Y knobs to make beam in 3-fold symmetry with brightest spot at the center (>300kx); if you are unable to adjust, load a recent alignment file (JEOL-PC: Maintenance menu – alignment – load alignment file (bottom-right of the window))
 6. Find sample. Smallest beam, lower mag (~20kx), adjust **Z-height** button (right panel) to minimize clouds around beam; or turn on **WOBB X** and minimize the image vibration. 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!
 7. 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.
 8. Turn on **HT WOBB** (right panel), and perform HT centering with CLA **BRIGHT TILT** + DEF/STIG X&Y knobs under 100kx.
 9. Objective stigmatism: select **OBJ STIG** and adjust DEF/STIG X&Y knobs to make the LiveFFT of amorphous round (>10kx)

TEM imaging with upper camera

1. Gatan PC: Start FilterControl and then DigitalMicrograph (DM) software if they are not started.
2. FilterControl – Check primary energy (200kV) and image mode (Imaging instead of Spectroscopy).
3. DM – Change Layouts (top-right corner) to TEM. Find and expand Camera View and Camera Acquire tabs.
4. DM – Camera Menu – Camera – select Upper Camera (Erlangshen).
5. Center area of interest to the center of viewing screen. Adjust BRIGHTNESS and FOCUS if necessary. Insert **HC** aperture with desired size (1–4) to enhance contrast if necessary.
6. DM – Camera View tab – Check “insert camera” checkbox to insert camera, check Auto Exposure and click Start View.
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 “My Documents\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. “My Documents\Users\” folder is also dumped regularly.
9. To save image in other formats, choose Save Display As.

Selected Area Diffraction (SAD)

1. Align microscope under image mode. Make sure GIF mode (**F6**) is turned off.
2. Center area of interest to center of screen. Spread beam with BRIGHTNESSSS knob.
3. Press **SA** and select desired aperture size (left panel) to insert SAD aperture.
4. Press **SA DIFF** button (right panel) and spread beam to have smallest transmitted spot.
5. Use MAG/Cam L 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 & 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 (Erlangshen). 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.

GIF alignment and HRTEM imaging

1. Align microscope under image mode.
2. Start FilterControl and then DigitalMicrograph (DM) software if they are not started.
3. DM – Change Layouts (top-right corner) to TEM. Find and expand Camera View and Camera Acquire tabs.
4. DM – Camera menu – Camera – select GIF camera.
5. Press **F6** (right panel) to switch to GIF mode, then press **F1** to lay down the phosphors screen.
6. Move sample to screen center at mag 10kx, focus beam to 6-10mm, center it to GIF entrance aperture. (8 O'clock direction, 3 mm away from the screen center spot).
7. Press **F1** (right panel) to raise screen.
8. FilterControl – make sure Primary Energy: 200keV, Mode: Imaging. If primary energy is not 200keV, close DM and FilterControl and restart the software set. If Spectroscopy mode is seen, go to DM-AutoFilter panel, click EELS button (one of the top four buttons) and then quickly click TEM button.
9. Expand Camera View tab in DM, click Start View to see live image. Check Auto Exposure time or use manual exposure time.
10. (*optional step*) Click “Align ZLP” (~1 minutes), and then click “Tune GIF” (~5 minutes). If you are asked to adjust beam intensity, adjust accordingly, then you have to press the ENTER key to continue (mouse click won't work!)
11. Find amorphous area on specimen, click Camera View and Start to view CCD image. At high mag (10kx or up), DM – Process – Live – FFT, press **OBJ STIG** and adjust DEF/STIG X & Y to correct objective stigmatism.
12. 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.

STEM alignment

1. Align microscope under image mode and make sure sample is at eucentric height and Mag \geq 250kx.
 2. Start FilterControl and then DigitalMicrograph (DM) software if they are not started.
 3. FilterControl – make sure Primary Energy: 200keV, Mode: Imaging. (Same as step 8 in previous GIF alignment part)
 4. DM – Change Layouts (top-right corner) to STEM. Find and expand DigiScan and HAADF control tabs.
 5. Make sure Upper camera (Erlangshen) is retracted and GIF CCD camera is selected under Camera menu.
 6. Take out any aperture you are previously using. Change CL aperture to #1.
 7. Open Sirius Client software if it is not running. (username: admin, password: sirius)
 8. Click “STEM” button in Detector part of Sirius Client, wait until Sirius Client turns into STEM control interface.
 9. Press **STD FOCUS** button (right panel), Change SMMAG to 300kx.
 10. DM – DigiScan tab – click “control beam”.
 11. In Sirius Client, select camera length = 40cm; select appropriate probe size (1nm for microanalysis, smaller probe for dedicated STEM imaging). Observe ronchigram on TEM screen with binocular (or use upper camera). Adjust sample Z-height to form ronchigram. Adjust **COND STIG** + DEF/STIG X&Y knobs to make ronchigram round (or use upper camera, see next step).
 12. Press **F1** (right panel) to lift up screen. In DM, insert upper camera (exposure time = 0.01s). Adjust **COND STIG** + DEF/STIG X&Y knobs to make ronchigram round and uniform. Insert CL aperture #3 and align it to the center of ronchigram.
 13. In Sirius Client, select camera length = 2cm, detector = EXT, center ronchigram (9 O'Clock, 3 mm away from center) with **PLA** + DEF/STEG X&Y knobs.
 14. 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.
 15. 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.
 16. Click “EM” in Sirius Client to switch back to TEM mode after finishing STEM.
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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. On Oxford PC (left), start 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.

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. DM – AutoFilter – Select EELS. DM – EELS/EDS acquisition tab, set exposure time to 1×10^{-6} s, set energy loss to 0, adjust to 0, aperture size to 1mm. set proper dispersion and start acquisition.
3. View EELS, adjust zero loss peak (ZLP) intensity (maximize) by FOCUS X/Y, SX and SY in FilterControl, and adjust ZLP position by Adjust value.
4. Input proper energy loss value to desired EELS peak position, start acquisition. Increase exposure time and aperture size if EELS is too weak.
5. Use Spectral Acquisition (DM) or INCA software (Oxford PC) to collect EDS spectra.
6. **After you finished using EELS, click EELS and then TEM button in AutoFilter control panel to change back to TEM mode!**

Spectrum Imaging (mapping or linescan)

1. Get an STEM image.
2. Go to DM – Spectrum Imaging (SI) tab, click Assign Image.
3. Draw a line or box ROI for spectrum imaging (collect set of EELS/EDS spectra), click Assign ROI for spectrum imaging. Set dimension of SI collection. You may draw another rectangle for sample drift correction.
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 is inserted during your session except leaving the CL aperture #2 in.
2. Go back to TEM MAG mode, SPOT size 1 and Alpha 3.
3. Turn off **BEAM** valve (left panel).
4. JEOLPC: TEMCON – **Double click** the black **StageNeutral** button to neutralize sample position.
5. Make sure **sample position (X/Y/Z) and tilts (TX/TY) are all ZERO**, then remove specimen holder from the microscope.
6. Load new specimen if you want, or take specimen out of holder and store the holder in Fischione Pumping Station.
 - a. Login NUcore and see if there is any user after you today.
 - b. If you are not the last user of the day, follow this procedure:
 - i. Refill LN2
 - ii. Logoff your session in NUcore.
 - c. If you are the last user of the day, follow this procedure:
 - i. Plug in ACD Heater.
 - ii. JEOL PC: TEMCON – Maintenance Menu – ACD and Bake to open ACD Heat window. Turn ACD Heat On.
 - iii. Logoff your session in NUcore.