

Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

JEOL 2100F Field Emission Transmission Electron Microscope

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These notes are meant to serve as an aid to assist users who have been trained and certified by MCC Staff. If ever you are unsure about the correct operation of the instrument or any of its components, please consult a MCC staff member before continuing. Never use equipment that you are not trained and approved to use.

Before using the MCC, please review the MCC User Handbook available through our website.

Preliminary Checks

1. Check the analogue vacuum gauge on power supply cabinet. Switch selector to vacuum 10^{-5} Pa range. Normal vacuum without liquid nitrogen in the cold trap should be between <2.5 (blue scale in center).
2. Dummy plug should be in microscope.
3. Open TEMCON software on TEM computer.
4. Check High Voltage Control window to make sure voltage is on (200 kV and ~ 150 - $160\mu\text{A}$). If voltage is off (no emission current) then contact Staff Member immediately.

Specimen Loading

1. Ensure that binocular caps and viewing screen cover are in place. Partially fill anti-contamination device (ACD) dewer with liquid nitrogen using funnel and footstool. Once the ACD cools (~ 5 min), top off the dewer. You will need to refill dewer after about 4 to 5 hours of microscope use.
2. Remove Dummy Plug: Pull out on small metal pin and wait for valve clicks. Rotate plug counterclockwise a few degrees until it stops. Wait for venting process (30 seconds). Pull out to remove plug.

For more MCC SOPs please visit our Training Library at <https://www.drexel.edu/core-facilities/facilities/material-characterization/resources-for-users/training-library/>

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3. Remove specimen rod: Zero goniometer stage with Stage Neutralize button on TEMCON main window. Switch toggle on goniometer to air. Pull rod gently until you reach the first stop. Turn rod counterclockwise until you reach stop. Pull rod gently until you reach stop. Turn counterclockwise (DO NOT PULL ON THE ROD) until you reach the stop. Wait for the goniometer to vent. Listen for nitrogen and the valve clicks. After the venting, the rod will come out easily. BE CAREFUL TO NOT HIT THE ROD ON THE ENTRANCE TO THE GONIOMETER. USE TWO HANDS AND WORK SLOWLY.
4. Place holder on stand.
5. Loading TEM sample onto specimen blade: Loosen screws on the blade with screwdriver. With clean tweezers, gently lift and nudge the clamp out of the way. Use vacuum tweezers to place sample on the blade. Replace the clamp. Tighten the screws. Turn just to the point where they stop, DO NOT USE FORCE WHEN TIGHTENING. Place the blade on the end of the holder using tool and tweezers.
6. Inspect the blade and sample under the microscope to be sure they are seated correctly.
7. Inspect the two O-rings on the holder to make sure there is no lint on them. Remove any lint or dust using a wooden toothpick.
8. Insert specimen rod: Switch toggle on goniometer to PUMP. Line up peg on rod with notch on goniometer entry. Insert the rod until you reach the stop. Keep pressure on the rod and listen for valves and vacuum pump. Wait for the light on the goniometer to turn green. Turn the holder clockwise 10deg and allow the vacuum to slowly pull the holder in. Use two hands to prevent the holder from jerking into the microscope. Turn the holder clockwise until the stop and allow the holder to slowly enter the microscope column.

Start-up Procedure

1. Voltage and emission should be on.
2. Press beam button on left panel to open gun valve.
3. Check screen for illumination.

Gun and Beam Alignment

1. Press STD Focus to reset objective lens current.
2. Insert desired Condenser Aperture (second aperture works best for alignments). Converge beam and center crossover on screen. Spread beam out and adjust aperture position to center disk using edge of viewing screen for reference.
3. Gun Shift: Turn GunAlign ON (F4 light on). Set Spot Size to 1. Mag to 200Kx. Use Brightness to bring beam to crossover in vacuum. Use SHIFT (X+Y) to center the beam on the screen. Switch to Spot Size 5. Turn GunAlign OFF (F4). Use SHIFT (X+Y) to center the beam. Reiterate until beam remains at center at both spot sizes. Set Spot Size to 1. Turn GunAlign OFF and center the beam using SHIFT (X+Y).
4. Gun Tilt: Turn GunAlign ON (F4). Converge beam to crossover. Press Anode Wobbler (F3) and adjust DEF/STIG (X+Y) to make the beam converge concentrically. Turn GunAlign OFF.
5. Press CONDSTIG. Adjust DEF/STIG (X+Y) to minimize the crossover size.

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6. Focus beam to crossover. Open Maintenance>Alignment menu in TEMCON. Click Tilt Compensator and Tilt X Wobbler. Adjust DEF/STIG X to superimpose the spots. Switch to Tilt Y Wobbler. Adjust DEF/STIG Y to superimpose the spots. Turn off compensator and wobbler.
7. Turn Brightness fully clockwise until you hear a beep. Press SA DIFF. In diffraction mode, use DIFF FOCUS to get caustic spot. Click Shift Compensator and Shift X Wobbler. Adjust DEF/STIG X to superimpose the spots. Switch to Shift Y Wobbler. Adjust DEF/STIG Y to superimpose the spots. Turn off compensator and wobbler.

Diffraction Astigmatism

1. Turn ON ILSTIG (F5).
2. Obtain Caustic spot (see Step 6 above).
3. Adjust DEF/STIG (X+Y) to get three-pointed star.
4. Insert an objective aperture and focus the diffraction pattern so that edge of aperture is sharp.
5. Press MAG1 and focus the brightness on the screen and center beam if necessary.

Image Alignments

1. Move sample so that an edge of your specimen is in the field of view. Adjust Z-height until you reach minimum contrast position (Objective Lens should be at standard focus).
2. (Voltage Center) Increase MAG to 400Kx. Find an object that is small and 1D (particle or point). Spread brightness until object is faint on camera. Press HT WOBBLER and BRIGHT TILT. Adjust DEF/STIG (X+Y) so that image does not move as HT wobbles. Increase MAG to 600Kx and adjust again. Turn off wobbler. Center beam and focus to minimum contrast using Z-height.
3. Find amorphous area or amorphous carbon support. Set MAG to >200kx. Insert CCD camera (see camera instructions). Select Process>Live>LiveFFT. Adjust Z-height so that the sample is under-focused (white Fresnel fringe). Press OLSSTIG and adjust DEF/STIG (X+Y) to get circular rings in FFT.

Shutdown Procedure

1. Zero stage position using Stage Neutralize button on TEMCON.
2. Set Mag to 300Kx and center the beam and spread to fill the viewing screen.
3. Close gun valve using “BEAM” button on left panel.
4. Replace binocular caps and viewing window cover.
5. Remove specimen rod (see Specimen Loading instructions above).
6. Insert dummy plug (unless next user is ready).
7. Remove all apertures.
8. Switch vacuum gauge on power supply cabinet to kV.
9. Insert heating element into ACD (use footstool). BE CAREFUL TO DIRECT THE PLASTIC TUBING AWAY FROM YOUR FACE AND BODY.
10. Open Maintenance>ACD/Bake Out menu on TEMCON and start ACD heat cycle.
11. Log your session information on the Log Sheet. And finish session in iLab.

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JEOL JEM2100F specifications

Electron Source: Schottky Field Emitter

Operating Voltages: 200.0 keV and 120.0 keV

Objective Lens Polepiece: High Resolution (HR)

Resolution @ 200 keV: point = 0.23 nm and lattice = 0.14 nm

Tilt-ranges (Double Tilt Holder): X axis = +/-35° and Y axis = +/- 30°

Condenser Apertures: 150 μm – 70 μm – 50 μm – 10 μm

Objective Apertures: 60 μm – 40 μm – 30 μm – 5 μm

High-Contrast Objective Apertures: 120 μm – 60 μm – 20 μm – 5 μm

Selected-area Apertures: 100 μm – 50 μm – 20 μm – 10 μm

JEOL 2100F Alignment Checklist

1. Press STD FOCUS to set objective lens to standard value
2. Insert and align condenser aperture – center x-over with SHIFT X + Y, spread beam and center aperture with aperture drive knobs.
3. Align Gun Shift
 - a. Spot Size 1 – Center beam X-over using F4 + SHIFT X + Y
 - b. Spot Size 5 – Center beam X-over using BRIGHT TILT + SHIFT X + Y
 - c. Repeat until spots stay at the center of the screen
4. Align Gun Tilt by centering convergence of beam with Anode Wobbler
 - a. F3 + F4 + DEF/STIG X + Y
5. Correct condenser lens astigmatism to focus the x-over (round beam).
 - a. COND STIG + DEF/STIG X + Y
6. Adjust Tilt Compensator X and Y – superimpose beam X-overs
 - a. Tilt + TiltX in TEMCON>Maintenance>Alignment + DEF/STIG X
 - b. Tilt + TiltY in TEMCON>Maintenance >Alignment + DEF/STIG Y
7. Overfocus brightness to limit, press SA DIFF, set MAG/CAM L to 150cm and adjust DIFF FOCUS to get caustic spot. Center with PLA and DEF/STIG X & Y
8. Adjust Shift Compensator X and Y – superimpose caustic spots
 - a. Shift + ShiftX in TEMCON>Maintenance>Alignment + DEF/STIG X
 - b. Shift + ShiftY in TEMCON>Maintenance >Alignment + DEF/STIG Y
9. Correct Intermediate Lens Astigmatism using caustic spot – 3-point star
 - a. F5 + DEF/STIG X + Y
10. With beam on sample, focus Diffraction Lens using edge of objective (#3) aperture and DIFF FOCUS
11. In image mode (MAG 1), focus on the sample with Z-height to get minimum contrast (rough focus). Increase mag and adjust z-height to get fine focus
12. Align voltage center (beam tilt) at high magnification
 - a. HT WOBB + BRIGHT TILT + DEF/STIG X + Y
13. Correct Objective lens astigmatism at high magnification using amorphous material and Live FFT to get circular symmetry in diffractogram.
 - a. Use Z-height to slightly underfocus specimen (bright Fresnel fringe)
 - b. Process>Live>FFT and adjust OBJ STIG + DEF/STIG X + Y