

## Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

### JEOL 2100 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.*

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#### **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 1.0 and 3.0. If higher, STOP AND CONTACT A CRF STAFF MEMBER.
2. Specimen rod should be in microscope.
3. Open TEMCON software on TEM computer.

#### **High Voltage Start Up**

1. Open TEMCON High Voltage Control.
2. Set HT to 160 kV.
3. Press HT ON button. Beam Current should rise to  $\sim 81 \mu\text{A}$  and stabilize.
4. Use Auto HT to increase HT to 200 kV in 0.1kV/3s steps. Beam current should stabilize at  $\sim 102 \mu\text{A}$ . This step takes 20 minutes.

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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#### Specimen Loading

1. Ensure that binocular caps and viewing screen cover are in place. Partially fill anti-contamination device (ACD) dewar with liquid nitrogen using funnel and footstool. Once the ACD cools (~10 min), top off the dewar. You will need to refill ACD after about 4 to 5 hours of microscope use.
2. 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 counter clockwise until you reach stop. Pull rod gently until you reach stop. Turn counter clockwise (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.
3. Place holder on stand.
4. 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.
5. Inspect the blade and sample under the microscope to be sure they are seated correctly.
6. 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.
7. 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 10° 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

4. Open HT control window on TEM computer. HT should be 200 keV. Filament Ready should be on. Emission current should be ~102 uA (dark current).
5. Open Option>Free Lens Control.
6. In Free Lens Control dialogue, click “LOAD” button (bottom left)
7. Choose file named “degauss.flc”
8. Count to 10.
9. Click “ALL Off” button (upper left)
10. Click Filament On and wait for filament to ramp up. Filament indicator should be green. Emission should rise to ~107 uA (operating current).
11. Check screen for illumination.
12. Check Spicer Field Cancellation System. All lights should be green. If not, press reset button and wait for green Field OK light.

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#### Gun and Beam Alignment

1. Press STD Focus to reset objective lens current.
2. Insert desired Condenser Aperture. Sweep Brightness from underfocus to overfocus. Adjust aperture position until the beam stays at the center of the screen. Use boundaries of viewing screen and focus screen for reference.
3. Gun Shift: Turn GunAlign ON (F4 light on). Set Spot Size to 1. Mag to 60Kx. 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. Click Filament Image in HT control window. Filament current should decrease to ~50%. If not, slow decrease the current to about 50% using the HT control. Look for filament image (halo image). Adjust DEF/STIG (X+Y) to center the intensity and make the filament image symmetrical. Turn GunAlign OFF.
5. Press CONDSTIG. Adjust DEF/STIG (X+Y) to focus the filament image. Click Normal Filament in HT control window.
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 Defocus should be 0).
2. (Voltage Center) Increase MAG to 200Kx. Press HT WOBBLER and BRIGHT TILT. Adjust DEF/STIG (X+Y) so that image does not move as HT wobbles. Increase MAG to 500Kx 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). Make a “power of 2” selection. Select Process>Live>LiveFFT. Adjust Z-height so that the sample is under-focused (white Fresnel fringe). Press OLSTIG and adjust DEF/STIG (X+Y) to get circular rings in FFT.

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#### **Shutdown Procedure (Underlined steps are for partial shutdown between users)**

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. Turn filament off using HT control on TEM computer.
4. Replace binocular caps and viewing window cover.
5. Use Auto HT to bring the voltage from 200 to 160 kV; 0.5 kV steps and 2 seconds time/step.
6. Turn HT off using HT control.
7. Set HT back to 200 kV.
8. Remove specimen rod (see Specimen Loading instructions above).
9. Re-insert specimen rod without specimen blade.
10. Remove all apertures.
11. Switch vacuum gauge on power supply cabinet to kV.
12. Insert heating element into ACD (use footstool). BE CAREFUL TO DIRECT THE PLASTIC TUBING AWAY FROM YOUR FACE AND BODY.
13. Open Maintenance>ACD/Bake Out menu on TEMCON and start ACD heat cycle.
14. Log your session information on the Log Sheet and Log Book. Include the date, your name, specimen description, any comments, filament usage (hours) from power supply at end of session.

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#### Operation of GATAN CCD Camera

1. The camera software DigitalMicrograph (DM) is on the GATAN-PC computer. Open the software. This starts communication between the CCD and computer and starts the Peltier Cooler on the camera. The camera MUST NOT be exposed to the electron beam at room temperature. It is good practice to open DM as soon as you arrive at the microscope, ensuring that the camera will be ready before you need it. You will know the camera has cooled when you see the right-most LED changes from orange to green.
2. To view the image on the computer, lift the viewing screen (F1) and click start view. Adjust exposure time and brightness to get about 0.5 seconds exposure. BE CAREFUL TO NOT FOCUS THE BEAM ON THE CAMERA.
3. To acquire an image, click: acquire. Usually 1 to 2 seconds is good for images.
4. To see the FFT choose FFT or LiveFFT from the 'Analyze' menu.
5. To acquire a diffraction pattern, adjust brightness (fully CW) and diffraction focus to get very dim spots on the viewing screen of the microscope. Position the beam stop (left side of the column) to cover the transmitted beam (brightest spot in pattern). Lift viewing screen. If you see streaking of the spots, close the viewing screen immediately and decrease the intensity of the beams. Counts at the brightest spot should not exceed ~14,000. Acquire diffraction pattern as quickly as possible to minimize exposure of the camera to bright spots.
6. Save images in DM3 format for later use.
7. To convert data, use File>Batch Convert; choose folder and image type.
8. All data left on the local computer will be erased within one week. Please transfer data with a USB memory stick, external hard drive or using your Electronic Research Notebook (internal users only).

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### **JEOL JEM2100 specifications**

Electron Source: LaB6

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

GATAN Orius SC1000 CCD Camera: 4008 x 2672 pixels @ 9 μm/pixel

## JEOL 2100 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 using desaturated filament image
  - a. F4 + DEF/STIG X + Y
5. Correct condenser lens astigmatism using desaturated filament image
  - 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
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. Focus Diffraction Lens using edge of objective aperture and DIFF FOCUS
11. In image mode, 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. Select Power of 2 area (ALT + selection tool) in image on GATAN PC and Process>Live>FFT and adjust OBJ STIG + DEF/STIG X + Y