

Instructions for JEM-2100 LaB₆ Transmission Electron Microscope

General Information

Location: EP 112

Serial Number: EM17420082

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If service is needed, contact Dr. Ahrenkiel.

If unavailable, call JEOL Service at 877-414-5365 or 303-421-9106.

Instrument Overview

The TEM can be operated at up to 200 KV.

The TEM is configured with a high-resolution pole piece (HRP).

The instrument has scanning capability (ASID).

A JEOL STEM bright-field detector is mounted above the viewing screen.

A Gatan 805 STEM bright/dark-field detector system is mounted below the viewing screen.

A JEOL secondary/backscattered electron detector is mounted above the sample holder.

A Gatan Orius SC1000 CCD camera is mounted below the viewing screen.

An Oxford EDX system is attached.

Holders - There are four sample holders available, which allow different types of analysis:

JEOL single-tilt holder. A high-tilt sample retainer can be used with this holder.

JEOL double-tilt holder (Model EM-31630)

JEOL low-background double-tilt holder (Model EM-31640).

Gatan single-tilt heating holder (Model 628.TA).

Computers - There are three PCs that control different features of the TEM:

JEOL: Main user control interface to the TEM (TEMCON).

Gatan: CCD camera and Gatan DigiScan STEM control.

Oxford: Oxford EDX detector and acquisition control.

Default Settings - The TEM is normally left by the previous user with the following settings:

Spot size: 2; Alpha: 2; Condenser aperture: 2; Magnification: ~10K ×

Procedures

1. Starting a session:

- a. *JEOL* is normally on, with TEMCON running. If not in this status, contact Dr. Ahrenkiel.
- b. The TEM is normally left on, with the vacuum system running, the high tension at 200 KV, and the beam off. In TEMCON, select High Voltage Control from the Dialogue menu, or press HT on the toolbar. The message "HT status: ON", indicates that the high tension is on. The Beam Current indicator should show approx 100 μA (called the dark current). Further below are the HT software buttons; ON should be depressed. If another message appears, contact Dr. Ahrenkiel.
- c. Logon to *Gatan*. Run Gatan Digital Micrograph (DM).

- d. Record in the log book the starting time indicated on "Filament" on the power supply behind the TEM column. Provide a valid project name or account.

2. Loading the sample:

If a holder is already in the TEM, see 7. Removing the holder.

All holders: With the holder on its base, set the base in a comfortable and accessible position. The holder should be shifted fully to the right so that the sample retainer is suspended above the black cup.

Do not touch with fingers the o-rings or any parts of the holder to the right of the o-rings (opposite end from the handle.). Specimens should be picked up with tweezers *from the edge only*. The direction of the holder pin will be facing upwards when the specimen is in the TEM column.

JEOL Single-Tilt Holder:

- a. A cartridge tool is kept on the back of the base for releasing the sample cartridge. Place the cartridge tool in the hole of the cartridge clamp at the end of the holder. Secure the holder in place with one finger (thumb), while pulling up the cartridge tool with another finger (index). The retainer should drop onto the black loading cup.
- b. Pick up the cartridge with tweezers and place on the white loading stage, with the pin passing through the retainer guide hole.
- c. Two cartridges are available for this holder:

Standard Cartridge:

- i. Loosen the retainer screw(s) and rotate the retainer to one side. Place the specimen in the cartridge with the side of interest facing upward. Rotate the retainer back to hold the specimen down and tighten the screws (finger tighten only.)
- ii. Lift the cartridge with tweezers. Open the cartridge clamp as before and attach the cartridge by connecting the guide hole and pin.

High-Tilt Cartridge: It is best to mount this cartridge on the holder clamp when loading samples.

- i. Lift the cartridge with tweezers. Open the cartridge clamp as before and attach the cartridge by connecting the guide hole and pin.
- ii. Loosen the screw and rotate the retainer to one side. Place the specimen in the cartridge with the side of interest facing upward. Rotate the retainer back to hold the specimen down and tighten the screws (finger tighten only.)

JEOL Double-Tilt Holder: The cartridge is mechanically fixed to the holder rod.

- a. Loosen the retainer screw and rotate to one side.
- b. Place the sample in the cartridge. Rotate the retainer to secure the sample and tighten the screw (finger tighten only.)

JEOL Double-Tilt, Low-Background Holder: The cartridge is mechanically fixed to the holder rod. A Be bracket is used to hold the sample in place. Do not touch the bracket with fingers.

- a. Loosen the two screws and rotate the clips out of the way. Lift the holder with two hands. Suspending the holder above a clean cloth, rotate the holder to allow the bracket to fall. Return the holder to its base.
- b. Place the sample in the recess of the cartridge. Set the bracket on top of the sample. Rotate the clips back above the bracket and tighten the screws (finger tighten only.)

Gatan Single-Tilt Heating Holder: Contact Dr. Ahrenkiel before using this holder.

All holders: Lift the holder with two hands, touching only the handle, or the portion of the rod between the handle and o-rings. Holding the rod horizontally, rotate both ways about its axis and jiggle the holder slightly to verify that the sample is secure.

3. Inserting the holder:

- a. Lift the holder off the holder stage. Maintain a firm grip on the handle with one hand and use the other hand to lightly support the holder between the handle and the o-ring.
- b. Align the holder with the circular port in the specimen chamber. With the pin aligned with the notch, slide the holder into the port until it reaches the stop. Release your grip on the handle, but maintain a slight force on the end of the handle.

The holder is now in position A.

- c. Pull the specimen chamber evacuation switch outward and toggle the switch up to PUMP. Several valves will open and close within the first 10 sec. Completely release the holder and wait for the green light below the switch to turn green (about 2 min.).
- d. From position A, rotate the holder 10° CW, maintaining a firm grip on the handle. Slowly allow the holder to slide about 0.5" into position B.

The holder is now in position B.

- e. From position B, rotate the holder about 80° CW, maintaining a firm grip on the handle. Slowly allow the holder to slide about 3" into position C.

The holder is now in position C.

- f. If using a double-tilt holder, connect its cable to the electronic port below the specimen chamber.
- g. Select the appropriate holder model in TEMCON using the pull-down menu on the upper right.

4. Turning on the beam:

A holder must be inserted into the sample evacuation chamber to observe the beam. If no holder is in the column, go to 2) Loading the sample.

- a. On High Voltage Control, press Filament: ON, or select **BEAM**. The filament current will slowly increase to the set limit (typically ~64% of maximum). The Beam Current indicator should show approximately 108 μA . If the reading differs substantially from this value, turn off the filament [see 10. Turning off the TEM] and contact Dr. Ahrenkiel.
- b. Remove the screen cover to observe the electron illumination on the phosphorescent screen. Move to a hole or thin region of the specimen using the trackball.
- c. Increase the magnification using **MAG/CAMLEN** to approx. 40 K \times . Focus the image of the filament (crossover) using **BRIGHTNESS**. Then turn **BRIGHTNESS** CW to overfocus the C3 condenser lens and spread the illumination to fill the screen.

5. Aligning:

Note that pressing **BRIGHT TILT** is equivalent to pressing CLA in Alignment.

- a. Center Condenser Aperture: The goal is to align the C3 aperture (CA) with the C3 lens.
 - i. Position a hole or thin region at the screen center.
 - ii. Go to a magnification of approx. 80 K \times . Turn **BRIGHTNESS** to form a crossover.
 - iii. Press **BRIGHT TILT**. Use **SHIFT X** and **SHIFT Y** to translate the beam to the screen center.
 - iv. Turn **BRIGHTNESS** CW to expand the beam until the illuminated area is smaller than the screen.
 - v. Adjust the CA (the uppermost manual aperture) using the two sets of mechanical controls (end and right side), so that the beam is centered on the screen.

You may wish to repeat steps ii-v to further improve the CA position.

b. Adjust Gun Tilt: The goal is to align the electron gun with the CA.

- i. Position a hole or thin region at the screen center.
- ii. In the Alignment panel, select GUN.
- iii. Adjust DEF/STIG X/Y to maximize the brightness. You may wish to monitor the current density indicator in TEMCON.
- iv. In the Alignment panel, deselect GUN.

c. Adjust Sample Height: This procedure should be done when a sample is initially loaded and repeated when the region of interest changes significantly.

- i. Position a thin region or feature of interest at the screen center.
- ii. Press STD FOCUS.
- iii. Press Z (up) and Z (down) to bring the feature into focus (minimum contrast). If necessary, change the sensitivity of the controls by clicking the arrows next to Z in TEMCON.

d. Adjust Pivot Points: Adjust the condenser deflection coils to bring the tilt axis into the sample plane.

This procedure should be done at the beginning of a TEM session.

- i. Position a hole or thin region at the screen center.
- ii. Press STD FOCUS.
- iii. In Alignment, select COMPENSATOR: TILT.
- iv. In Alignment, select WOBBLER: TILT X.
- v. Adjust DEF/STIG X to zero the beam oscillation. Deselect WOBBLER: TILT X.
- vi. In Alignment, select WOBBLER: TILT Y.
- vii. Adjust DEF/STIG Y to zero the beam oscillation. Deselect WOBBLER: TILT Y.
- viii. In Alignment, deselect COMPENSATOR: TILT.

e. Adjust Beam Shift: Adjust the condenser deflection coils to eliminate tilting during beam shifting.

This procedure should be done at the beginning of a TEM session.

- i. Position a hole or thin region at the screen center.
- ii. Press STD FOCUS.
- iii. Turn BRIGHTNESS fully CW to spread the illumination (beep sounds when at limit of range.)
- iv. Select SA DIFF. Turn DIFF FOCUS to obtain a bright sharp image of the caustic (spot).
- v. In Alignment, select COMPENSATOR: SHIFT.
- vii. In Alignment, select WOBBLER: SHIFT X.
- viii. Adjust DEF/STIG X to zero the oscillation of the caustic spot. Deselect WOBBLER: SHIFT X.
- ix. In Alignment, select WOBBLER: SHIFT Y.
- x. Adjust DEF/STIG Y to zero the oscillation of the caustic spot. Deselect WOBBLER: SHIFT Y.
- xi. In Alignment, deselect COMPENSATOR: SHIFT.

f. Correct Beam Tilt: This procedure may be needed repeatedly in a single TEM session.

- i. Position a feature of interest at the screen center.
- ii. Press STD FOCUS. Verify that the feature is at the eucentric height.
- iii. In Alignment, select CLA, or select BRIGHT TILT.

- iv. There are now two possible procedures to follow:
 - A. In Alignment, select WOBBLER: HT, or press **HT WOBBLER**, or
 - B. In Alignment, select WOBBLER: OBJ.
- v. Adjust **DEF/STIG X** and **DEF/STIG Y** to eliminate motion of the feature. (The focus condition should oscillate without lateral motion.)
 - A. Deselect WOBBLER: HT, or deselect **HT WOBBLER**, or
 - B. Deselect **WOBBLER: OBJ**.
- g. Coarse Focusing: This procedure aids in focusing at low magnification, when high resolution and precise calibration are not required.
 - i. Select **IMAGE WOBBLER X** or **IMAGE WOBBLER Y**.
 - ii. Adjust **FOCUS** so that the region of interest is stationary.
 - iii. Deselect **IMAGE WOBBLER X** or **IMAGE WOBBLER Y**.

6. Acquiring data:

The CCD camera can be inserted/retracted using controls in DM.

Reference images for the CCD must be acquired periodically. See Dr. Ahrenkiel for details.

In DM, select *Series/Set_User*. Select your user name. Then select *Series/Set_Info*. Set a sample name, folder, starting letter and index (append to file names) and file format.

On the "Camera View" panel, set "Exposure Time" to around 0.3 s. On the "Camera Acquire" panel, set "Exposure Time" to around 0.7 s. (These are typical values.)

Press *View* so that the camera is continually refreshing the image.

To enter image notes into the log file, select *Image Display/Image/Info* and enter the information in the text box under "Description".

To save an exposure, select *Window/Series/Log_and_Save*.

a. Selected-Area Diffraction:

- i. Select **MAG1**. Translate region of interest to the center. Focus the image (**FOCUS**).
- ii. Insert a diffraction aperture (DA) and center on the viewing screen.
- iii. Rotate the beam stop forwards and look for its shadow on the screen. Rotate and slide to adjust the beam to position its tip precisely in the middle of the screen (black dot).
- iv. Turn **BRIGHTNESS** CW substantially to reduce intensity so the beam is spread to an area much larger than the viewing screen.
- v. Press **SA DIFF** to switch to diffraction mode.
- vi. Focus the diffraction pattern using **DIFF FOCUS**.
- vii. Press PLA. Center the diffraction pattern using **DEF/STIG X/Y**.
- viii. Select **F1** to raise the viewing screen. Continue to focus the image on the CCD. Press "Acquire".

Rule of thumb: It is always better in diffraction to decrease the intensity and increase the exposure time.

b. Bright-Field Imaging:

Follow *Selected-Area Diffraction* steps i-vi above to obtain a diffraction pattern. Then:

- i. Insert an objective aperture (OA) and position, as needed.
- ii. Press **MAG1**.
- iii. Remove the DA.
- iv. Select **F1** to raise the viewing screen.

7. Removing the holder:

- a. Double-click on Stage Neutral.
- b. Press Filament: OFF or deselect BEAM. Wait until the filament current reads zero.

The holder is now in position C.

- b. From position C, with a firm grip on the handle, pull the holder straight out about 3". Rotate the holder about 80° CCW to position B.

The holder is now in position B.

- c. From position B, with a firm grip on the handle, pull the holder straight out about 1". Rotate the holder about 10° CCW to position A.

The holder is now in position A.

- e. Release your grip on the holder. Apply a slight inward force on the end of the holder. Toggle the specimen chamber evacuation switch to VENT. Wait a few seconds until you hear N₂ gas purging the evacuation chamber.
- f. Firmly grip the holder handle with one hand (right) and position your other hand below the entry port to prevent accidentally dropping the holder. Pull the holder straight out, clear of the microscope. Maintain a firm grip on the handle with one hand and use the other hand to lightly support the holder between the handle and the o-ring. Return the holder to its base on the counter.

8. Unloading the sample:

The procedure for unloading samples from the holder is essentially the reverse of that for loading. After loosening the retainer, it is best to pick up the holder (or just the sample cartridge), hold it over a soft cloth, and rotate, allowing the sample to fall onto the cloth.

When done with a holder, place the cover back on the holder base and close the clamp on either end.

9. Ending a session:

- i. Leave the sample holder area and the TEM console clean and organized.
- ii. Record in the log book the ending time indicated on "Filament" on the power supply behind the TEM column.

Notes:

You do not need to leave a holder in the TEM.

The TEM high voltage is normally left on. However, if the L-N₂ anti-contamination device (ADC) is used, the high voltage should be shut off and the ADC warmed. See P. 5-10 of the JEM-2100 manual.

Log off of *Gatan*. Do not log off of *JEOL*.