Standard Operating Procedure for the JEOL 2010 HRTEM

Nebraska Center for Materials and Nanoscience
Central Facility for Electron Microscopy

12B / 12C Walter Scott Engineering Center
University of Nebraska at Lincoln

This work is intended as a guide to the operation of the **JEOL 2010 HRTEM** by the average user. It details:

- Sample loading
- Bringing the microscope up to operating conditions
- Alignment of the microscope
- Sample removal
- Returning the microscope to a powered down state
- Relevant safety procedures

A more detailed and thorough account of these procedures as well as a more advanced ones can be found in the JEOL Operating Manual provided by JEOL.

This work is under constant revision and all users are encouraged to contribute any material for review by facility staff.

Emergency Operating Procedure

If a user ever has a problem that he/she is even slightly unsure about, **ASK** someone who knows and can help. There are no penalties for asking for help but there may be for not reporting damage to equipment that may delay or prevent others from working.

If, at any time, the user perceives a problem that he/she is unable to remedy, the user is to call or find the following individuals:

Lab Specialist	12C Walter Scott Engineering Center	2-8762	
Faculty Supervisor	318 Walter Scott Engineering Center	2-8762	2-8308

If neither of these individuals are available then the user should do the following:

- Turn the Filament knob fully counterclockwise
- Press the Accelerating Voltage button to switch it off (unlit)

Then, if possible, the user should stay with the microscope while trying to contact the above individuals. If it becomes necessary to leave the microscope then the user should leave a large, legible note on both the microscope and at least one of the above individuals desks stating:

- The problem
- When it occurred
- User name and phone number

If a dire emergency is evident (smoke, fire, sparks.....), if it is safe to do so the user should pull the main electrical box switch (behind the microscope) to turn off power to entire microscope and notify the proper emergency personnel. In any case the user should leave the Facility and contact emergency personnel as soon as possible from a safe place.

Qualification Procedures

To become eligible to use the **JEOL 2010 HRTEM** it is necessary for the user to do the following:

- •Register desire to become a user with the Laboratory Specialist or Faculty Supervisor
- •Read and understand the
  - •Emergency Operating Procedure
  - •Standard Operating Procedure
  - •Scheduling Policy
  - •Charging Structure
  - •Facility Use Policy
- •Pass a written examination on the use of the microscope
- •Pass a "Driving Test" on the microscope with either the Laboratory Specialist or Faculty Supervisor present

No persons shall use the microscope without first becoming a qualified user by completing the above steps. **Any person using the microscope who is not a qualified user will be banned from the facility for an indefinite period of time, as determined by the Laboratory Specialist or Faculty Supervisor.** This policy, although harsh, is intended to prevent damage to equipment and the subsequent loss in productivity by other users and expense to the center and university.

Standard Procedure

This Standard Operating Procedure will refer to the condensed version of the Operating Manual (OM). Please refer to section 4 of the for a complete description of all controls, apertures, etc. and their location.

I Sign in the User-Log with the following information

•Read any previous comments made by the last few users
•Username
•Date
•Time on
•Material being examined including number of specimens
•Type of work to be done (BF, DF, SAED, CBED....)
•Vacuum at start

II Load the Sample

•Use gloved hands when handling samples and holders
•Ensure that samples, tweezers and other tools are absolutely clean and free of possible contamination by contact with fingers, any surface which might be dirty or might have had any contact with fingers or uncleaned surfaces. This applies also to gloves and the sample loading jigs, as well as to the o-ring and specimen locating portion of the sample rod. If you need to clean a surface, use an untouched lens tissue, cotton bud, or cotton wiper and a minute quantity of isopropanol. Be aware of the greater possibility of transfer of organic contaminants in the isopropanol solvent. Be sure that no fibers are inadvertently transferred to the specimen rod and o-rings.
- *Avoid letting the specimen holder touch non-clean surfaces*
- *Never "blow on" or exhale on samples to dry them, use a clean, dry gas source.*
- *Make sure all screws are firmly secured and that you always have a sure grip*
- ***Always** ask if you have a question*

If you are planning to do high resolution microscopyor to work with small electron probes, fill up the anticontamination device cold trap with LN2.

Loading the Sample into the Specimen Holder

- •Select the single tilt or double tilt holder (see the last section in condensed version of Operating Manual)
- •Secure the sample referring to OM Figure 5.2-8 on page 5-7

Insertion of the Specimen Holder into the Microscope

- •Refer to OM Figure 5.2-11 on page 5-9

  - •Make sure that there is no dust or lint on the O-rings and blow any with an Easy Duster if necessary.
  - •Insert the specimen holder (insuring that the specimen holder guide pin is aligned with the guide groove) until firm contact is made. Be very careful with the tab which extends from the handle, parallel to the rod axis; it is essential, but fragile!
  - •**Slowly** turn approximately 5° the specimen holder clockwise until the red LED turns on.
  - •**WAIT** until the green LED comes on.
  - •Turn the specimen holder clockwise until the stop is reached, slide holder in, turn clockwise again, then slide holder all the way in. The black tab on the handle of the specimen holder should line up with the alignment hole to the right of the hole where the specimen holder is inserted.

III Bring the Microscope to Operating Condition

- *•Please refer to OM Section 5.2.6 page 5-10*
- *•Never turn the filament knob past its limit and always turn it slowly (at least 2 minutes for full range)*
- *•Never operate the microscope if the pressure rises to a level greater than $2.5x10^{-5}$ Pa as read on the gauge on the Sublimation Ion Pump power supply behind the microscope OM (Blue Scale)*
- *•**Always** ask if you have a question*

Set the accelerating voltage.

- •Select PAGE-1 on the CRT with the PAGE key located on the lower right of the keyboard.
- •Select the accelerating voltage using the ACCEL VOLTAGE switch (**L1-4** OM Figure 4.2-1 page 4-4).
  Note: Do not change the accelerating voltage if the voltage is already on. Generally, the HT voltage is left on. This is indicated by the HT switch (**L1-5** OM Figure 4.2-1 page 4-4). If it is lit, the voltage is on.

Generate an Electron Beam.

•Depress MAG1 (**R1-3** OM Figure 4.2-2 page 4-7) if MAG is not displayed in the upper left hand corner of the CRT.
•Set the spot size value to TEM1-3 as indicated on the CRT using the SPOT SIZE switch (**L1-12** OM Figure 4.2-1 page 4-4) and the a-SELECTOR (**L1-23** OM Figure 4.2-1 page 4-4).
•Set the magnification to 20,000 times using the SELECTOR switch (**R1-7** OM Figure 4.2-2 page 4-7). The magnification is indicated on the CRT (PAGE-1).
•Depress the HT switch (**L1-5** OM Figure 4.2-1 page 4-4). It will light up. Confirm that the BEAM CURRENT meter (**L1-6** OM Figure 4.2-1 page 4-4) rises. The current will stabilize after several minutes. Determine the current value from Table 5.1 OM page 5-11.
•Insure that the FILAMENT READY lamp (**L1-7** OM Figure 4.2-1 page 4-4) is lit. Turn the FILAMENT knob (**L1-8** OM Figure 4.2-1 page 4-4) slowly (30 seconds per half graduation) until the stopper is reached. Do not change the stop position. Increasing the filament heating above the set point will drastically reduce the filament life! (Both the filament and replacement procedure are very expensive!)

No beam? Make sure that there are no objective or selective area apertures in the path of the beam. If there is still no beam, move the specimen stage to see if the specimen is blocking the beam. Then try pulling the specimen rod out of the chamber and turn to the first stop. Do not pull out further! **Ask for help if you're not sure!**

IV Alignment

The following alignment procedure is a combination of Method A (OM Section 5.2 page 5-2) and Method B (OM Section 5.3 page 5-42).

Alignment of the Condenser Aperture

•Select the condenser aperture to be used. Refer to OM section 4.1 page 4-1.
•Turn the BRIGHTNESS knob (**L1-14** OM Figure 4.2-1 page 4-4) "over and under" as in, clockwise and counterclockwise from the minimum spot size.
•Please refer to OM Figure 5.2-15 page 5-15. If the spot center deviates from the center of the screen, correct the misalignment with the condenser aperture assembly knobs 2 and 3 or 2 and 4 as per OM section 4.1 page 4-1.
•If the beam is elliptical, depress the COND STIG button (**L1-13** OM Figure 4.2-1 page 4-4) and use the DEF X and DEF Y knobs (**L1-18** OM Figure 4.2-1 page 4-4 and **R1-2** OM Figure 4.2-2 page 4-7) to make the beam round.

Alignment of the Condenser Lens Alignment

- •Turn the BRIGHTNESS knob (**L1-14** OM Figure 4.2-1 page 4-4) so that the beam fills the screen. The beam may be centered, if necessary, using the SHIFT X knob (**L1-17** OM Figure 4.2-1 page 4-4) and the SHIFT Y knob (**R1-1** OM Figure 4.2-2 page 4-7).
- •Depress the DEFLECTOR GUN switch (**R2-1** OM Figure 4.2-4 page 4-12) if not lit.
- •Use the DEF knobs (**R2-3** OM Figure 4.2-4 page 4-12), one at a time, to obtain the brightest illumination possible.
- •Turn the BRIGHTNESS knob (**L1-14** OM Figure 4.2-1 page 4-4) and obtain the smallest spot size possible.
- •Use the following method to center the condenser lens:
    a) Set the spot size to TEM**1-3** as indicated on the CRT using the SPOT SIZE switch (**L1-12** OM Figure 4.2-1 page 4-4).
    b) Center the beam, if necessary, using the **DEFLECTOR** SHIFT knobs (**R2-2** OM Figure 4.2-4 page 4-12). Note that these are the knobs in the drawer.
    c) Set the spot size to TEM**4-3** as indicated on the CRT using the SPOT SIZE switch (**L1-12** OM Figure 4.2-1 page 4-4).
    d) Now, center the beam using the **SHIFT X** knob (**L1-17** OM Figure 4.2-1 page 4-4) and the **SHIFT Y** knob (**R1-1** OM Figure 4.2-2 page 4-7).
    e) Reiterate steps a) through d) until the spot remains centered when switching spot sizes.
- •Set the spot size value to TEM1-3 as indicated on the CRT using the SPOT SIZE switch (**L1-12** OM Figure 4.2-1 page 4-4).
- •Turn the BRIGHTNESS knob (**L1-14** OM Figure 4.2-1 page 4-4) so that the beam fills the screen.

Adjustment of the Objective Lens Current and Specimen Height

- •Use the Trackball (**SC-4** OM Figure 4.2-6 page 4-15) to bring an object into the center of the viewing screen.
- •Select PAGE-4 on the CRT with the PAGE key located on the lower right of the keyboard.
- •Set the Objective Lens current to 6.806 by using the OBJ FOCUS switch and knob (**R1-4** OM Figure 4.2-2 page 4-7). Note that the last digit is not displayed. Set the OBJ FOCUS switch to 2, turn theOBJ FOCUS coarse knob counterclockwise until the current reads 6.79, and then turn the knob clockwise 7 steps. Users should check that 10/11 steps will just change from the last 6.79 step to the first 6.81 step.
- •Depress WOBBLER-IMAGE X or Y switch (**R1-5** OM Figure 4.2-2 page 4-7).
- •Focus on the object using the Z CONT switch (**SC-5** OM Figure 4.2-6 Page 4-15). When the object is in focus, the oscillation should be minimal.
- •Turn off the WOBBLER-IMAGE X or Y switch (**R1-5** OM Figure 4.2-2 page 4-7).

Alignment of the Current Center

- •Turn the BRIGHTNESS knob (**L1-14** OM Figure 4.2-1 page 4-4) so that the beam almost fills the screen. Center it with the SHIFT X knob (**L1-17** OM Figure 4.2-1 page 4-4) and the SHIFT Y knob (**R1-1** OM Figure 4.2-2 page 4-7).
- •Turn on the WOBBLER-OBJ switch (**R2-9** OM Figure 4.2-4 page 4-12). The image will expand and contract.
- •Depress DEFLECTOR BRIGHT switch (**L1-13** OM Figure 4.2-1 page 4-4). Note that it is labeled BRIT TILT.
- •Bring the center of the expansion and contraction to the center of the view screen with the DEF X and DEF Y knobs (**L1-18** OM Figure 4.2-1 page 4-4 and **R1-2** OM Figure 4.2-2 page 4-7)
- •Turn off the WOBBLER-OBJ switch (**R2-9** OM Figure 4.2-4 page 4-12).

Alignment of the Voltage Center

- •Set the magnification to 100,000 times using the SELECTOR switch (**R1-7** OM Figure 4.2-2 page 4-7) and use the BRIGHTNESS knob (**L1-14** OM Figure 4.2-1 page 4-4) so that the beam fills the screen. The beam may be centered, if necessary, using the SHIFT X knob (**L1-17** OM Figure 4.2-1 page 4-4) and the SHIFT Y knob (**R1-1** OM Figure 4.2-2 page 4-7).
- •Use the Trackball (**SC-4** OM Figure 4.2-6 page 4-15) to bring an object or the edge of an object into the center of the viewing screen.
- •You may need to check the specimen height again with the WOBBLER-IMAGE X or Y switch (**R1-5** OM Figure 4.2-2 page 4-7) and the Z CONT switch (**SC-5** OM Figure 4.2-6 Page 4-15).
- •Turn on the WOBBLER-HT switch (**R1-5** OM Figure 4.2-2 page 4-7).
- •Turn on the DEFLECTOR BRIGHT switch (**L1-13** OM Figure 4.2-1 page 4-4). Note that it is labeled BRIT TILT.
- •With the WOBBLER-HT on, the focal plane will cycle through under focus, in focus, over focus, in focus, etc. There should be no movement in the x or y direction. Correct any motion with the DEF X and DEF Y knobs (**L1-18** OM Figure 4.2-1 page 4-4 and **R1-2** OM Figure 4.2-2 page 4-7)
- •Turn off the WOBBLER-HT switch (**R1-5** OM Figure 4.2-2 page 4-7).

Alignment of the Micro-Area Illumination Modes (CBD, NBD)

- •Set the magnification to 50,000 times using the SELECTOR switch (**R1-7** OM Figure 4.2-2 page 4-7).
- •Turn on the NBD switch (**L1-21** OM Figure 4.2-1 page 4-4).
- •Set the spot size value to NBD10nm -1 as indicated on the CRT using the SPOT SIZE switch (**L1-12** OM Figure 4.2-1 page 4-4).
- •If the beam is elliptical, depress the COND STIG button (**L1-13** OM Figure 4.2-1 page 4-4) and use the DEF X and DEF Y knobs (**L1-18** OM Figure 4.2-1 page 4-4 and **R1-2** OM Figure 4.2-2 page 4-7) to make the beam round.
- •Turn on the WOBBLER-HT switch (**R1-5** OM Figure 4.2-2 page 4-7).
- •Confirm that the DEFLECTOR BRIGHT switch (**L1-13** OM Figure 4.2-1 page 4-4) is on. Note that it is labeled BRIT TILT.
- •The image will expand and contract similarly to that in the current center alignment. Bring the center of expansion and contraction to the center of the viewing screen with the DEF X and DEF Y knobs (**L1-18** OM Figure 4.2-1 page 4-4 and **R1-2** OM Figure 4.2-2 page 4-7).
- •Turn off the WOBBLER-HT switch (**R1-5** OM Figure 4.2-2 page 4-7).
- •Turn on the TEM switch (**L1-22** OM Figure 4.2-1 page 4-4).

Objective Astigmation Correction

Correcting stigmation is tricky to do well, hard, and takes a lot of practice. Please do not try to correct any stigmation unless a more experienced user is present.

V Removing the Sample

Powering Down the Microscope

- •Never remove a sample with the filament on
- •Return the microscope to suitable condition for the next user. Remove both objective apertures and the selected area aperture if inserted. Set the magnification to 20,000 times using the SELECTOR switch (**R1-7** OM Figure 4.2-2 page 4-7) and set the spot size value to TEM1-3 as indicated on the CRT using the SPOT SIZE switch (**L1-12** OM Figure 4.2-1 page 4-4). Turn the BRIGHTNESS knob (**L1-14** OM Figure 4.2-1 page 4-4) so that the beam is spread to fill the screen.
- •Always restore the stage to an untilted, unrotated and centered position before removal of specimen by using the N button (**SC-2** OM Figure 4.2-6 page 4-15).
- •Slowly turn down the FILAMENT knob (**L1-8** OM Figure 4.2-1 page 4-4) counterclockwise.

Removing the Sample Holder from Microscope

Refer to OM Figure 5.2-11 on page 5-9

- Turn on the dry N_2 supply toggle valve located on the wall to the right of the microscope.
- Reverse the loading process. Pull the specimen holder straight out until the a stop is reached, turn counterclockwise, then slide holder out again, turn counterclockwise, wait until you hear gas purging the airlock (about 30seconds), and pull the holder from the airlock.
- Turn off the dry LN_2 supply toggle valve.

VI Shutdown Procedure

- Be sure that all samples are removed from the microscope
- If the anticontamination device was used and unless you know that another user will load a sample within half an hour, insert the heater into the LN_2 trap (OM Figure 3.3-1 page 3-4 labeled Refrigerant tank), connect its cable, and depress ACD HEAT (**L2-6** OM Figure 4.2-3 page 4-10)
- Remove all samples and possessions from the HRTEM room and clean up used film...etc.
- Turn off room lights and shut door on way out
- Fill out NCMN Charge Sheet and turn in to the Lab Specialist or his/her mailbox (located across from the 2 computers in the main room of 12C)