MSE 595T
Basic Transmission Electron Microscopy

Laboratory II
Basic TEM Alignment

Purpose

The purpose of this lab is to learn the basic operation and alignment of the JEOL 2000FX transmission electron microscope.

Report Requirements

No report is required for this lab. You should, however, make extensive notes for your own use.

Procedure

Follow the safety checks given in Lab. # 1.

Before inserting the specimen holder:

- Set the x-tilt angle to 0º.
- If using the double-tilt holder, make sure that the y-tilt angle is 0º.
- Make sure the specimen translation controls are centered.
- Make sure that the objective aperture is removed.
- Make sure that the filament is off.
- Make sure that the EDS detector is out.
- Make sure that the HT is on.

I. Sample Holder Insertion

1. Load a specimen in the specimen holder. The loading procedure is different from holder to holder and depends on the type of holder you are using.
2. Set the x-tilt angle to zero. If the x-tilt is set at too extreme an angle, damage may be done either to the pole piece or the sample holder. If necessary, disengage the motor and rotate to 0º then re-engage the motor. You may also use the foot pedals to adjust the angles.

3. If using the double-tilt holder, make sure that the y-tilt is at the 0º position. You can do this by plugging in the motor drive with the holder out of the column, then using the foot pedals to center the y-tilt.
4. Make sure that the specimen shift knobs are adjusted such that the specimen is centered. (Use the CRT screen to determine the x and y settings.)
5. Make sure that the objective aperture is out, and the EDS detector is out.
6. Insert the sample holder to the first stop. To do this, press on the holder until you hear the air lock valve open. When the holder is inserted into the air lock valve chamber, the red light on the goniometer will turn on for about 45 seconds. While the specimen holder is in the chamber, the vacuum system will go through cycles of pumping on the chamber for about 45 seconds, and not pumping for 5 seconds. The red light is on during the pumping cycle and off during the rest cycle Allow the system to pump on the chamber **at least 4 times** before inserting the holder.
7. Complete the insertion of the holder. To do this, use both hands: your left fingers will be between the holder and goniometer, and you right hand will grasp the very end of the holder. With your right hand, rotate the sample holder 90° clockwise and slowly let the sample holder enter the vacuum chamber. The sample holder may be damaged if it is allowed to enter the chamber too quickly, and the ruby tip hits the internal bearing with too much force.
8. Set the specimen selector. For sample holders with two specimen positions, set the device to position 1 or 2. For single specimen holders, set the device half way between positions 1 and 2.

Note: For high-resolution work, you may have to wait 30 minutes or more for the O-ring seal to stop drifting. It will typically take you this long to condition the TEM in any case.

II. Removal of Specimen Holder

1. Turn off the filament knob.
2. Return the x and y-tilt controls to 0º.
3. Center the specimen translation controls.
4. Remove the objective aperture.
5. Retract the EDS detector.
6. Pull the specimen holder out until it stops.
7. Rotate the holder by 90º, counterclockwise.
8. Gently *push* the specimen holder out of the airlock. (This is much safer than *pulling* it out.)

III. High Voltage Generation.

Note that the high voltage (HT) will normally be on. The procedure here is what you would follow if it were not on, for example after the machine has automatically shut off because of a safety interlock.

1. Check that the READY lamp is on. If it is not, the microscope will not operate.

2. Check that the FILAMENT is off. If it is not, turning on the high voltage will bring it up to its operating current instantly, causing a thermal shock, and serious damage.
3. Turn on the accelerating voltage, by depressing the HT button.
4. Allow the beam current to stabilize.
5. The accelerating voltage may be adjusted using the HT command to set the step size, then using the panel control. (Not usually adjusted: see manual.)
6. The accelerating voltage should gradually increase to 200kV. At 200kV the dark current should be around 104μA.

IV. Turn on the Filament

1. Increase the filament current ***slowly*** to the saturation point. The process should take no less than 5 minutes, or about 30 seconds per half-division.
2. Select BRIGHT TILT using the DEFLECTOR control, for bright field imaging alignment; DARK TILT for dark field alignment and imaging.
3. Deselect the deflector control you have chosen so that it does not accidentally become misadjusted.
4. Select the LOW MAG function. Check that you have a beam by adjusting the BRIGHTNESS lens current.
5. Center the beam with the SHIFT knobs.

V. Gun and Condenser Alignment

This procedure adjusts the gun deflector coils so the beam is centered on the optic axis of the condenser lens.

1. Select MAG1
2. Set the magnification to 12kX (or slightly higher).
3. Select spot size 3 with the SPOT SIZE switch. (The SPOT SIZE control adjusts the excitation of Condenser Lens 1.)
4. Converge the beam with the BRIGHTNESS knob. (This adjusts the excitation of Condenser Lens 2.)
5. Center the beam with the SHIFT knobs.
6. Select spot size 1.
7. Converge the beam with the BRIGHTNESS knob.
8. Center the beam with the GUN SHIFT controls. (This adjusts the gun deflector coils.)
9. Repeat steps 3 through 8 until the beam remains centered on switching from spot size 1 to 3.

VI. Gun Tilt Adjustment

1. Select a magnification of 25 – 50kX.
2. Select SPOT SIZE 3.
3. Converge the beam with the BRIGHTNESS knob.
4. Center the beam with the SHIFT controls.
5. Select a hole in your specimen, so the beam is not passing through your specimen.

6. Slightly desaturate the filament with the FILAMENT control, until you can first see the image of the filament in the beam spot.
7. Center the filament image in the beam spot using the GUN TILT controls.
8. Turn the FILAMENT control back to the saturation position.
9. Re-check the gun and condenser alignment and repeat if necessary.

VII. Condenser Aperture Alignment

Please be very careful to use the appropriate condenser shifting knobs for the selected aperture.

1. Select MAG1
2. Set the magnification to 10kX (or slightly higher).
3. Converge the beam with the BRIGHTNESS control.
4. Center the beam with the SHIFT knobs.
5. Spread the beam to almost full screen (leave only a few mm at perimeter) using the BRIGHTNESS control.
6. Center the beam with the condenser shifting knobs.
7. Repeat steps 5 at the opposite side of convergence (opposite direction used in step 5).
8. Repeat step 6, but only correct one-half of the difference from actual to exact center.

VIII. Condenser Stigmator Adjustment

1. Select MAG1.
2. Set the magnification to 25 – 50 kX.
3. Center the beam with the SHIFT knobs.
4. Spread the beam slightly with the BRIGHTNESS knob. [Note: it is often most useful to cycle through crossover, alternately spreading the beam back-and-forth slightly onto either side.]
5. Select COND STIG.
6. Make the beam spot circular by adjusting the DEFLECTOR knobs. [Note: using the technique of cycling back-and-forth, the astigmatic condition will be recognized by looking for asymmetric expansion of the beam as it passes through crossover.]
7. Deselect COND STIG.

IX. Adjust the Specimen Height.

This procedure sets the specimen at the object plane of the objective lens. This adjustment is necessary each time a new specimen is inserted.

1. Set the magnification to 12kX (or slightly higher).
2. Set the objective lens current to about 7.04 mA.
3. Converge the beam with the BRIGHTNESS knob.
4. Center the beam with the SHIFT controls.
5. Spread the beam to full screen with the BRIGHTNESS knob.
6. Make sure that the y-axis is at 0^{o}.
7. Find a distinctive object feature and center it on the screen using the specimen translation controls.

8. Adjust the “Z” control knob on the goniometer to bring the feature into coarse focus.

X. Focus the Image

This procedure sets the object plane of the objective lens at the specimen. Provided that coarse focus has been achieved using the “Z” control (objective lens current is about 7.04 mA), this is the process that will be used most often to improve the focus on a region of the specimen.

1. Select MAG1
2. Place a distinctive object feature at the screen center.
3. Make sure the OBJ 16X button is off, so that you are using the two finer controls.
4. Adjust the objective lens current to give minimum contrast, using the FOCUS control.