MSE 595T
Basic Transmission Electron Microscopy

Laboratory III
TEM Imaging - I

Purpose

The purpose of this lab is to:

1. Make fine adjustments to the microscope alignment
2. Obtain a diffraction pattern
3. Obtain an image

Report Requirements

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

Alignment Procedures

I. Pivot Point Adjustment

In the next step, we will use the BRIGHT TILT controls to set the beam parallel to the optic axis, but it is first necessary to ensure that the tilting operations are done about a pivot point located in the specimen plane.

1. Load a specimen in the specimen holder and complete all of the alignments that you learned in Lab. 2. The adjustments in this Lab will not work unless these are completed first.
2. With the specimen in focus, and at the correct height, select a magnification of about 40kX. [Alternately, with no specimen in the beam path, adjust the objective focus so that the objective lens current reads 7.04 mA.]
3. Focus the illumination to a small spot (BRIGHTNESS) and center it on the screen (SHIFT).
4. Engage the IMAGE X wobbler. This tilts the beam to its maximum deflection in the X-axis, alternating between positive and negative beam tilts. You may see two illumination spots, corresponding to these deflections.
5. Bring the two spots together, using the IMAGE WOBBLER ADJ – X controls. [note – always make certain that you are grasping the correct pair of knobs.]

6. Disengage the IMAGE X wobbler.
7. Engage the IMAGE Y wobbler. This tilts the beam to its maximum deflection in the Y-axis, alternating between positive and negative beam tilts. You may see two illumination spots, corresponding to these deflections.
8. Bring the two spots together, using the IMAGE WOBBLER ADJ – Y controls.
9. Disengage the IMAGE Y wobbler.
10. Recenter the illumination with the SHIFT controls.

II. Current Centering

This procedure makes the electron beam parallel to the optic axis of the objective lens. This is accomplished by changing the focal length of the lens. If the beam is parallel to the optic axis, then an object at the center of the screen should remain at the center. If the beam is tilted, the image at the center of the screen will move.

1. Select a magnification below 50kX.
2. Locate a small, distinct image feature and place it at the center of the screen.
3. Center the illumination and spread it to at least fill the screen, thus providing plane-wave illumination.
4. Select BRIGHT TILT on the DEFLECTOR control.
5. Engage the OBJ WOBBLER. This continuously cycles the objective lens excitation above and below its set point. The image *will* go in and out of focus, and will rotate as the spiraling action of the lens varies.
6. Use the DEFLECTOR controls to make the image stationary at its center, so it rotates about this point. You may wish to use the small screen and binoculars; this allows you to concentrate on the center point of the screen, allowing less distraction.
7. Disengage the BRIGHT TILT and OBJ WOBBLER controls.

III. High Voltage Centering

This technique is used to set the illumination parallel to the objective lens axis at higher magnifications. It is more sensitive than current centering. Instead of varying the objective lens excitation, we now vary the accelerating voltage, in a saw-tooth fashion, causing the electron wavelength to be changed. The fixed lens excitation then provides a varying focal length according to the varying electron wavelengths.

1. Select a magnification above 50kX.
2. Locate a distinct image feature, place it at the center of the screen and focus.
3. Center the illumination and spread it to fill the screen, thus providing plane-wave illumination.
4. Select BRIGHT TILT on the DEFLECTOR control.
5. Engage the HT WOBBLER. This continuously cycles the accelerating voltage. The image *will* go in and out of focus, and will rotate as the spiraling action of the lens varies.

6. Use the DEFLECTOR controls to make the image stationary at its center, so it rotates about this point. Again, you may wish to use the small screen and binoculars.
7. Disengage the BRIGHT TILT and HT WOBBLER controls.

This adjustment must be repeated periodically during any high magnification or high resolution work.

IV. Image Focus.

This is the process of changing the objective lens current, to alter its focal length, and bring the image into focus. The objective lens current value that corresponds to the proper focus position is about 7.04mA. This value, of course, depends upon the specimen height first being set correctly, using the "Z" control. There are two methods of focusing the image:

1. Rely upon your experience in identifying a focused image. You should select an area which is the thinnest, most-curved region near an edge, if possible.
   An overfocused image (lens too strong) produces a dark Fresnel fringe around denser objects in the specimen. An underfocused image (lens too weak) produces a bright Fresnel fringe.
   The ideal focus is obtained when there is no fringe. Contrast is also minimized at the point of focus. Both of these effects will be most noticeable at an edge of a very thin specimen region.
2. Alternatively (though not ideal for high-magnification work) you can use the IMAGE WOBBLER, either X or Y. When the wobbler is engaged, the image appears to oscillate if it is out of focus. Adjust the FOCUS control to eliminate the oscillation. Do not forget to disengage the WOBBLER.

V. Obtaining a Diffraction Pattern

1. Select MAG1 or MAG2.
2. Center the electron beam using the SHIFT knobs.
3. Spread the beam to fill the screen (or more) using the BRIGHTNESS control.
4. Locate a suitable part of the specimen, and place it at the center of the field of view.
5. Insert a Selected Area Diffraction (SAD) aperture, and center it carefully.
6. Press DIFF. A diffraction pattern will appear on the screen.
7. Focus the pattern using the DIFF FOCUS control.
   - Note that you can return to the image by strengthening the diffraction lens.
   - Note the effect of the BRIGHTNESS control on your ability to form a well-focused diffraction pattern.

VI. Obtaining Convergent Beam Diffraction Pattern

1. Select MAG1 or MAG2.

2. Center the electron beam using the SHIFT knobs.
3. Spread the beam to fill the screen (or more) using the BRIGHTNESS control.
4. Locate a suitable part of the specimen, and place it at the center of the field of view.
5. Using the BRIGHTNESS control, focus the illumination to the smallest possible spot (crossover). You may wish to turn off the Bright 16X button when doing this.
6. Press DIFF. A diffraction pattern will appear on the screen. Instead of spots, however, the pattern should consist of discs.
7. Focus the pattern using the DIFF FOCUS control. In this case, the edge of the central disk will be sharpest at good focus, fuzzy when out-of-focus.

Obtaining a Bright Field Image

This is normally done after obtaining a diffraction pattern on the screen.

1. Insert an objective aperture and center it on the transmitted beam.
2. Select MAG1 to return to image mode.
3. Select the desired magnification.
4. Focus the image.

Note: The following sections are provided for your information. Image recording on film is not part of this laboratory exercise, but is something you may wish to do in future.

VII. Image Recording on Film

While the computer is convenient and suitable for recording many images, the best quality, resolution and permanence is obtained by using sheet film.

1. Select the desired magnification.
2. Bring the features to be recorded within the borders of the framed screen.
3. In order to measure the exposure for a small area, place the small screen in the beam path and place the area of interest on the small screen; leave the small screen in place for the remainder of the process. For an exposure time averaged over the whole image, remove the small screen from the beam path.
4. Adjust the image brightness so the EXPOSURE TIME displayed on Page 1 reads less than 5 seconds, if possible. Longer exposures tend to exhibit the effects of specimen drift.
5. If the camera is in MANUAL advance mode, press the PHOTO button once to advance a piece of film into position for exposure. In AUTO film advance mode, this process is handled automatically.
6. Cover the viewing port.
7. Depress PHOTO a second time to expose the film, being careful to avoid any vibration of the microscope. The PHOTO lamp will turn on while the shutter is in place.

VIII. Film Processing

1. Open the camera chamber by turning the handle clockwise by 90 degrees. Open the valve on the nitrogen tank in order to fill the camera to atmospheric pressure. The door will open in about two minutes.
2. Close the valve on the nitrogen tank after the door opens.
3. Wearing gloves, remove the receiving cassette from the camera chamber. Replace it with an empty one from the desiccator.
4. Remove the dispensing cassette and replace it with a full one from the desiccator.
5. Check the O-ring for any lint or deposits.
6. Close the camera door and turn the handle anti-clockwise by 90 degrees. This starts the pumping cycle.
7. Take the camera cassettes to the dark room. Process the used film. Reload the dispensing cassette. Place the filled dispensing cassette and the empty receiving cassette in the desiccator.