MSE 595T
Basic Transmission Electron Microscopy

Laboratory IV
TEM Imaging - II

Purpose

The purpose of this lab is to:
1. Practice the overall alignment procedure for the microscope
2. Obtain bright field (BF) and dark field (DF) images.
3. Record images

Report Requirements

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

Procedure

Preliminaries
1. Go over the safety checks.
2. Insert a specimen in the specimen holder. (Use a polycrystalline gold specimen.)
3. Check that the stage is *centered*, objective aperture is *out*, EDS detector is *out*, tilts are *zeroed*, then insert the specimen holder in the goniometer.
4. Turn on the beam and perform the basic microscope alignments.
5. Choose an interesting field of view, and focus the image. Check that the objective lens voltage is approximately 7.04.

I. Obtaining a Bright Field Image

In this section, we use an objective aperture to select the "transmitted beam" to form the image. As a general rule, apertures should only be moved when you can see them, so this operation is done in DIFFRACTION mode.

I.i Conventional
1. In MAG1 or MAG2 mode, insert and center a selector aperture. Center and spread out the illumination. Ensure that the BRIGHT TILT setting is selected.

2. Select DIFF mode. Focus the diffraction pattern with the DIFF FOCUS knob.
   a. Note the effect of the BRIGHTNESS knob on your ability to focus the diffraction pattern: if the intensity is set very high, the electron beam is convergent rather than parallel. This makes the diffraction spots broad.
   b. Note that you can use the DIFF FOCUS knob to return to the image. This knob controls the first intermediate lens, which can be strengthened by turning the knob clockwise, in order to focus on the image plane of the objective lens.
3. Insert an objective aperture and center it carefully around the transmitted beam. Normally you will want to use #2, the middle-sized aperture.
4. Select MAG1 or MAG2.
5. Remove the selector aperture.
6. Adjust the illuminations, as needed, using the BRIGHTNESS.
7. Select the desired magnification, and focus the image.
8. Record an image as described in the section on image recording.

I.ii Using CBD

1. In MAG1 or MAG2 mode, converge the illumination to the smallest spot (crossover). Ensure that the BRIGHT TILT setting is selected.
2. Select DIFF mode. Focus the diffraction pattern, if necessary, with the DIFF FOCUS knob.
3. Insert an objective aperture and center it carefully on the central disk. Normally you will want to use #2, the middle-sized aperture.
4. Select MAG1 or MAG2.
5. Spread out the illuminations, as needed, using the BRIGHTNESS.
6. Select the desired magnification, and focus the image.

II. Recording Images

II.i Computer Image Capture

Digitally captured images have a finite pixel size, which limits the extent to which they can be enlarged. They are, however, easy to print, easy to manipulate, measure and transmit. They are easy to store and you can erase them to make room for more on a hard disk or zip disk. (This can be a problem, too.)
The final image you will turn in to pass the course (in the next laboratory) will be taken as a digital image.

1. Turn on the computer and start the Digital Micrograph program. (You will need to sign on to ECN before you can start this.)
2. Insert the screen for the digital camera.
3. Click on the “Hare” icon to collect continuous images with short collection times. Adjust and focus the image in this mode. Ensure that the image brightness produces a gray level close to 1,000 for a typical mid-gray pixel.
4. Click on the “Tortoise” or the “Camera” icon to collect a high-quality image at a slow scan rate. In continuous collection mode, press the spacebar to stop collection.
5. Save your image to a zip disk or a directory of your own on the hard disk.

II.ii Film

Photographic film provides the best image resolution and contrast, if properly done. The images are, however, more time-consuming to print, and less easily transmitted than computer images.

1. Bring the feature to be photographed within the borders of the small screen.
2. Select the AUTO exposure mode.
3. Adjust the image brightness to obtain an exposure time of less than 5 seconds, to avoid effects from specimen motion.
4. Depress PHOTO to advance and expose a plate.

III. Recording a Diffraction Pattern

As a general rule, diffuse the electron beam as much as possible, to provide plane-wave illumination, unless you are using a convergent beam technique.

III.i Computer

1. Insert the screen for the digital camera
2. Collect a diffraction pattern using the Digital Micrograph software.
3. Save the pattern.

III.ii Film

1. Obtain a diffraction pattern on the screen. Select an appropriate camera length, and focus carefully.
2. Switch to MANUAL exposure mode, on Page 1 of the CRT screens.
3. Select an appropriate exposure time. (It is often useful to take a few shots at different exposure times, including approximately 2, 8, and 20 seconds. The exposure meter is not a useful guide, but eventually your experience will help.)
4. Optionally, use the beam stop to reduce the intensity of the central spot, making the overall brightness more uniform.
5. Expose a film plate.

IV. Obtaining a Dark Field Image.

IV.i Off-Axis Dark Field ***<u>(Not Recommended)</u>***

The use of off-axis beams results in significant image degradation, because of spherical aberration effects.

1. Obtain a diffraction pattern as described above.
2. Tilt the specimen to obtain a suitable 2-beam condition or until a desired reflection or set of spots becomes strong.

3. Focus the diffraction pattern using the DIFF FOCUS knob.
4. Leaving BRIGHT TILT selected, insert the objective aperture and place it around the desired diffraction spot, corresponding to a diffracted beam.
5. Select MAG1 or MAG2, and select the desired magnification.
6. Focus the image, adjust the illumination and record the image on the computer.

IV.ii Centered Dark Field Imaging ***<u>(Preferred)</u>***

1. Return to the diffraction pattern that you obtained in IV.i.1
2. Place the point of the beam stop at the transmitted beam spot, near the center of the screen.
3. Select DARK TILT. It is a good idea to start by using the DEF x and y knobs to return the transmitted beam to the center of the screen – the deflectors are usually not properly centered, since every dark field orientation is at least slightly different.
4. Use the DEF x and y knobs to bring the *negative* of your chosen diffraction spot to the screen center. Although it might have been dim to start with, it will be bright when it arrives at the screen center. [Note: you may try to use the DEF x and y knobs to move your selected diffraction spot (hkl) to the screen center. Notice that the spot becomes dim when it is at the center.]
5. Switch back to BRIGHT TILT. The transmitted beam should be at the screen center.
6. Insert a suitable objective aperture and center it precisely around the transmitted beam. (This centers the aperture precisely on the optic axis).
7. Select DARK TILT. Use the DEF x and y knobs to center the diffracted beam within the objective aperture.
8. You may wish to go back-and-forth between BRIGHT TILT and DARK TILT, to ensure that the reflected beam (in DARK) is as nearly as possible at the same location as the transmitted beam spot (in BRIGHT).
9. Select MAG1 or MAG2, and select the desired magnification. Note that you can now switch from BF to DF directly, by selecting the appropriate beam tilt setting.
10. Focus the image and adjust the illumination.
11. Record both the BF and DF images on the computer.
12. Compare the centered and off-axis DF images.

Process any film you have exposed, and reload the camera.