

HRTEM – Part 1: Experiment

Goal

This experiment will introduce the basic technique of HRTEM (high-resolution transmission electron microscopy). The main goals is to show how to set up a transmission electron microscope for high-resolution work, how to choose imaging parameters for obtaining meaningful, interpretable images, and to demonstrate that the alignment of the microscope is particularly critical for good high-resolution work. The experiment will be carried out on the Tecnai F30. The specimen will be a Si single crystal, prepared for a $\langle 110 \rangle$ viewing direction. In part 2 of this laboratory, image (and diffraction!) simulations will be carried out for the imaging conditions applied in part 1.

Experiment

1. Load the specimen, start up the microscope, switch on the TV system, select a *small* condenser aperture, suitable for HRTEM, and perform the standard alignment. Most important, you should have the specimen exactly in the eucentric height (to minimize spherical aberration), the condenser aperture perfectly centered, and the gun shift perfectly aligned.
2. Select spot size 1.
3. Remember that any beam tilts with respect to the optics axis of the objective lens will introduce undesired phase shifts! Therefore, perfectly align the direction of the incident electron beam parallel to the optic axis of the objective lens in two steps:
 - A. Perform a current center alignment.
 - B. Perform a coma-free alignment.
4. Obtain a high-resolution image of the amorphous specimen edge on the TV monitor and correct the objective lens astigmatism by adjusting the stigmators and the objective lens focus for minimum contrast.
5. Obtain a diffraction pattern with a focused beam and tilt the specimen exactly to a $\langle 110 \rangle$ viewing direction. Find a region suitable for lattice imaging. This should be some distance away from the amorphous edge, however not beyond the first (dark) extinction contour.
6. Insert one of the larger objective apertures, such that the aperture does not cut off spatial frequencies before the information resolution limit of 0.14 nm.
7. Observe the image on the TV monitor or at a suitable magnification (e. g. 600,000 \times).
8. Carefully tune the objective lens defocus until you observe a lattice image.
9. From now on, exclusively use the electric image shift to shift the image, not the mechanical object translation.

10. Shift back to the amorphous image and check the alignment of the objective lens stigmators.
11. Using the electrical image shift, try to find regions in which the lattice image looks perfectly symmetric (thus, regions where the foil is perfectly oriented parallel to $\langle 110 \rangle$).
12. Record images at different focus settings and keep track of the focus settings you have chosen to be able to compare them to the image simulations you will carry out in part two of the experiment. To calibrate the defocus, move to the amorphous edge and adjust the objective lens focus for minimum contrast. Minimum contrast corresponds to $\approx 1/2\sqrt{C_s\lambda}$.
13. Employing the same procedure, record images at different foil thicknesses.
14. Record images after *intentionally* misaligning the objective lens stigmators.
15. Record images after *intentionally* tilting the specimen away from the $\langle 110 \rangle$ zone axis.
16. Record a low-magnification bright-field image of the area you studied at high-resolution.

Report

- No more than 5 pages, please!
- Draw a model of the atom positions of the diamond structure (the structure of silicon and germanium) in $\langle 110 \rangle$ projection.
- Calculate the theoretical point resolution of the Tecnai F30.
- Compare the features of your experimental images with a projected structure you have drawn and discuss any discrepancies.
- Discussed the effect of increasing under focus and increasing foil thickness.
- Discussed the effect of misaligned objective lens stigmators and deviation of the viewing direction from the exact $\langle 110 \rangle$ zone axis.
- Inspect the low-magnification bright-field image you have recorded for any unusual features. Can you detect surface roughness (thickness fluctuations), irradiation-induced structural damage, or contamination?