Electron Diffraction II

Goal

At this point of the course, you should have understood that the contrast of TEM images of crystalline material and structural defects therein sensitivity depends on the crystal orientation. Therefore, it is important to be able to tilt the specimen into an appropriate crystallographic orientation with respect to the primary beam. A very sensitive measure for the crystallographic orientation is provided by Kikuchi lines. In this experiment, you will observe Kikuchi lines in single crystalline and polycrystalline specimens and learn how to tilt the area of interest into particular crystallographic orientations.

Experiment

1. Load a single-crystalline specimen, start up the microscope, and align it.
2. Record a few images of characteristic areas at an appropriate magnification.
3. Focus the beam near the specimen edge, switch to diffraction, and observe the CBED pattern. *Tilt the specimen away from any highly symmetric viewing direction.* Choose an appropriate camera length, focus the pattern, and record it.
4. Switch back to image and position the beam in a significantly thicker specimen area. Return to diffraction and observe *pairs* of Kikuchi lines. Make sure the pattern exhibits at least three non-collinear pairs of Kikuchi lines – if not, tilt the specimen until this condition is fulfilled. Record the pattern.
5. Tilt the specimen by a small amount ($\leq 3°$) and observe the change of the Kikuchi line pattern versus the change of the spot pattern. Record the pattern for comparison with the previous pattern.
6. Investigate the effect of beam convergence and condensor aperture size on the appearance of Kikuchi lines. Record a few patterns under different conditions.
7. Tilt the specimen and observe how the Kikuchi line pattern moves. By following prominent Kikuchi lines, tilt the specimen to viewing directions of high symmetry (low-indexed zone axes, including $\langle 100 \rangle$, $\langle 110 \rangle$, and $\langle 111 \rangle$). Record the corresponding Kikuchi line patterns.
8. Load the polycrystalline specimen and verify it resides in the eucentric height.
9. Practice tilting of individual grains to highly symmetric viewing directions without losing the region of interest. Repeat after intentional misalignment of the specimen height.

Report

- Compile a table of the most important low-indexed reflections of the material under study in which you list the spacings between the corresponding Kikuchi line pairs for the nom-

inal camera length you employed. Using this table, index the spots and the Kikuchi lines in the first CBED pattern you obtained from the thick specimens area.

- Identify three non-collinear pairs of Kikuchi lines in your indexed pattern and use them to calculate the exact crystallographic orientation of the optic axis.
- Comparing the pattern you obtained after slightly tilting the specimen to the pattern you had before (step 5), comment on the sensitivity of Kikuchi lines versus the current sensitivity of the spot pattern against small specimen tilts.
- Discuss the appearance and symmetry of the Kikuchi line patterns you have obtained after tilting the specimen to high-symmetry viewing directions.
- Comment on the challenge of tilting a small grain of a polycrystalline specimen to a particular crystallographic viewing direction.