

## CBED

### Goal

In this experiment, you will be introduced to (CBED) convergent-beam electron diffraction. The experiment shall teach you how to set up a transmission electron microscope for this powerful technique of electron diffraction and demonstrate some of the most important applications. The instrument we will be using is the Philips CM-20, operating at an accelerating voltage of 200 kV and equipped with a thermal emitter (LaB<sub>6</sub>). The specimen will be a Si single crystal, prepared for a  $\langle 111 \rangle$  viewing direction.

### Experiment

1. Load the specimen, start up the microscope, select a *large* condenser aperture, suitable for CBED, and perform the standard alignment. Most important, you should have the condenser aperture centered and correct for intermediate lens astigmatism. The alignment of the current center and the objective lens stigmators should be checked, but is not critical.
2. Select a region of interest, preferably in a location where thickness fringes indicate that the specimen edge has the shape of a regular, flat wedge.
3. Observe and record CBED patterns under the following three conditions:
  - A. Tilt the specimen to a “random” viewing direction.
    - Choose a sufficiently large camera length.
    - Observe the central disk on the focusing screen with the binoculars.
    - Operate on the “brightness” (condensor focus). Observe demagnified images of the specimen in underfocus and overfocus.
    - Then adjust the “brightness” for “infinite magnification” to obtain the CBED diffraction pattern. Focus pattern with the “diffraction focus.”
    - Study the effect of varying the condenser aperture.
    - Choose a condenser aperture suitable for Kossel-Möllenstedt patterns.
    - Observe HOLZ lines and bright-field contours in the central disk.
    - Study the effect of varying the camera length.
    - Study the effect of defocussing the pattern (overfocus, underfocus).
    - Study the effect of the local specimen thickness.
  - C. Tilt the specimen to a low-indexed zone axis ( $\langle 111 \rangle$ ).
    - Move to a specimen area of suitable thickness (fine detail in the pattern).
    - Choose a large camera length, observe and record HOLZ lines in the 000 disc.
    - Use the dark-field mode for precisely orienting the primary beam.

- Choose a small camera length, observe and record the FOLZ ring.
- C. Tilt the specimen to a two-beam condition.
- Observe fringes in the two disks.
  - Without changing the specimen orientation, move to a region of different thickness and observe how the fringe pattern changes.

## Report

- No more than 5 pages, please!
- Speculate why we used a Si single crystal, and not, for example, a foil of polycrystalline aluminum for this experiment.
- For the CBED patterns obtained in a “random” orientation, describe the effect of
  - changing the condensor aperture,
  - defocusing the pattern
  - varying the specimen thickness.
- For the CBED patterns obtained with the primary beam parallel to a low-indexed zone-axis (“ZAP”),
  - determine the zone-axis from the ZOLZ pattern, applying the rule explained in the lecture,
  - describe the symmetry of the HOLZ-line pattern,
  - calculate the spacing of the lattice planes normal to the viewing direction from the radius of the FOLZ ring and compare the result with the value you would expect given that  $a_{\text{Si}} = 0.543 \text{ nm}$ .
- For the CBED patterns obtained in the two-beam condition,
  - describe the effect of the specimen thickness on the fringe pattern,
  - estimate the local specimen thickness for one location by applying the graphical method explained in the lecture.