

Imaging II

Goal

In this experiment, you will learn how to analyze the crystallographic line direction and the Burgers vector of crystal dislocations by TEM. This technique, which exploits variations in dislocation contrast observed under different imaging conditions, has been very important for gaining insight into crystal plasticity over the past decades. The specimen for this experiment is a single-crystalline Ge-rich $\text{Ge}_x\text{Si}_{(1-x)}$ layer epitaxially grown on a single crystalline Si {001} substrate and prepared for plan-view TEM by ion-beam milling. Owing to the lattice mismatch between $\text{Ge}_x\text{Si}_{(1-x)}$ and Si, dislocations exist in the $\text{Ge}_x\text{Si}_{(1-x)}/\text{Si}$ interface. At the ends of each such “misfit dislocation” segment, a (relatively short) “threading dislocation arm” extends from the interface to the surface of the $\text{Ge}_x\text{Si}_{(1-x)}$ layer. In this experiment, we are exclusively concerned with the extended misfit dislocations segments. In addition to the experiments to determine the crystallography of the dislocations, you will learn how to setup weak-beam dark-field conditions and how to optimize the excitation error for imaging dislocations under two-beam conditions.

Remember that you should document the conditions under which each image was recorded by acquiring corresponding diffraction patterns.

Experiment

1. Load the specimen, start up the microscope, and align it. In order to obtain high-quality images also at high magnification, carefully align the current center and the objective lens stigmators.
2. Tilt the specimen to align the $\langle 001 \rangle$ substrate normal parallel to the primary electron beam. Insert an adequate objective aperture and observe the defect structure - you will see crystal dislocations in the $\text{Ge}_x\text{Si}_{(1-x)}/\text{Si}$ interface appear as extended straight dark lines. Locate a suitable region of interest, re-adjust the orientation of the specimen and record and image at a suitable magnification.
3. Focus the beam and switch to diffraction. Defocus the diffraction pattern and observe a de-magnified image in the central disk (you will need to use the binoculars and the small viewing screen). From the orientation of the dislocation lines with respect to the spot pattern determine the crystallographic directions of the dislocation lines.
4. Without losing the area of interest, tilt the specimen to obtain “two-beam” conditions with $\{200\}$ and $\{220\}$ reflections. Record the corresponding bright-field images and observe how the contrast of individual dislocations changes with changing imaging conditions.
5. Set up the microscope and for on-axis dark-field imaging with a $\{220\}$ reflection (tilt the primary beam to bring to a $g = 220$ reflection on the optic axis). Record the dark-field image and the corresponding bright-field image.

6. Next, set up the microscope for weak-beam dark-field imaging with the same $\{220\}$ reflection: From the condition you had for the $g = 220$ dark-field image in step 5, tilt the specimen further until it reaches a two-beam condition with $3g = 660$ as the strongest reflection. Re-insert the objective aperture and center it on $g = 220$. Return to image and record with approximately half of the indicated automatic exposure time.
7. Focus the beam and find a specimen area sufficiently thick for observing Kikuchi lines. Tilt the specimen to obtain an *exact* two-beam condition with $g = 220$. This corresponds to the situation where the excess Kikuchi line intersects with the $\{220\}$ reflection. Record the corresponding bright field image.
8. Now slightly tilt the specimen such that the excess Kikuchi line is moved (i) towards the primary beam and (ii) farther away from the primary beam than the $\{220\}$ reflection. Record bright-field images for each case.

Report

- What are the crystallographic directions of the misfit dislocations?
- How does tilting the specimen object the contrast by which dislocations appear in bright-field images?
- What are the crystallographic directions of the Burgers vectors?
- Compare the dark-field to the bright-field images of the dislocations.
- What are the typical features of the weak-beam dark-field images, and how do these arise? What is the advantage of the weak-beam dark-field technique over conventional bright-field or dark-field imaging?
- Which excitation error s gives the best (sharpest) contrast of crystal dislocations under bright-field conditions - $s = 0$, $s < 0$, or $s > 0$?