NANO 703/703L

Lab: Convergent-Beam Electron Diffraction

Due: Following Lab Session

BACKGROUND

Convergent-beam electron diffraction (CBED) provides structural information from small specimen regions. CBED patterns are produced in TEM by focusing the electron beam to crossover in the specimen plane and viewing the diffraction pattern in the back focal plane of the objective lens. Most commonly, CBED zone-axis patterns (ZAPs) are obtained to determine the crystal symmetry. Unlike selected-area diffraction (SAD) ZOLZ patterns, CBED ZAPs contain 3-D information, due to the enhanced HOLZ signatures.

Kikuchi lines are more pronounced in CBED patterns than in selected-area patterns. This occurs both because the pattern arises from a smaller, and therefore more uniform region than selected-area diffraction, and because of the enhancement due to coherent, elastic scattering.

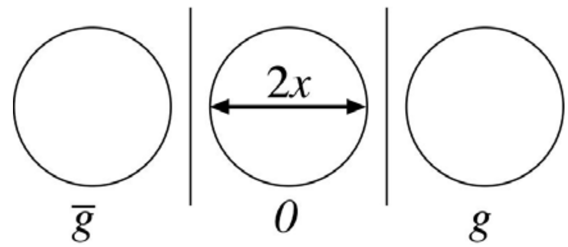

Fig. 1: The CBED disk diameter is proportional to the convergence angle of the incident beam.

Convergence Angle

The range of incidence angles within the convergent probe makes CBED analogous to the x-ray diffraction rocking-curve method, i.e., both represent the diffracted intensity over a range of orientations of the sample with respect to the incident beam. In CBED, the range is limited by the convergence angle α of the incident beam. In a calibrated pattern, α can be related to the radius x of the bright-field disk (Fig. 1) and the wavelength λ, using:

$$\alpha_i = \lambda x_i .$$

Generally, we would like to use the largest convergence angle obtainable to view as much structure as possible within the CBED disks, and to allow a reduction in probe size without loss of beam current.

Rocking-Curve Oscillations

It is also useful to examine the intensity profile with a CBED disk acquired away from a low-index zone axis, and with a particular excitation condition reached within the disk. When the Bragg condition is satisfied in the center of the disk (coinciding with the excess Kikuchi line), the theoretical form for the intensity oscillations (Fig. 2) with excitation error is closely reproduced. Dynamical theory predicts an oscillation as:

$$I_{\mathbf{g}} \propto \mathrm{sinc}^2\left(\pi s_{eff} T\right)$$

where $s_{eff} \equiv \sqrt{s^2 + \left(1/\xi\right)^2}$. The minima occur at $s_{eff} T = n$, where $n = \pm 1,\ \pm 2,\ \pm 3,\ \ldots$ A plot of s_i^2 vs. n_i^2, with $i = 1,\ 2,\ 3,\ \ldots$, gives a straight line, with slope $1/T^2$ and intercept $1/\xi^2$, if $n_i = i + \Delta n$, where Δn is an index offset.

For a fringe separation of $2x_i$, the excitation error is $s_i = \pm \lambda g x_i$.

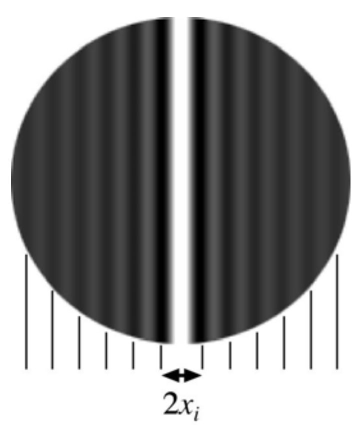

Fig. 2: Simulated CBED disk showing rocking-curve intensity oscillations.

GOAL

We will examine the influences of the CA and the alpha selector feature on α.
We will examine CBED and SA patterns from Si at <100>, <110> and <111> orientations.
We will analyze the intensity profile of a dark-field disk at a two-beam condition.

The double-tilt holder is needed to accurately orient the specimen. Use the dark-tilt alignment to precisely orient the beam.

EXPERIMENT

1) Observe influences of CA diameter and α-selector setting on α:
   a) Move the sample out of the field of view. Select CBD mode.
   b) Switch to the largest CA (#1).
   c) Select a value of α using the knob labeled α SELECTOR. Perform alignments.
   d) Converge the beam. Switch to SA DIFF mode.
   e) Measure the diameter $2x$ of the bright-field disk in nm^{-1}. Increase the camera length if necessary.
   f) Repeat for each CA (#2-4).
   g) Change the α selector setting and repeat b-f.

2) Acquire CBED and SAD patterns at <100>, <110> and <111> orientations.

3) Acquire CBED patterns using a two-beam conditions at two different sample thicknesses.
   a) Identify a strong reflection and tilt to a nominal two-beam condition.
   b) Orient the sample and beam so that $s = 0$ is near the center of the CBED disk. Record the pattern.
   c) Now translate the sample to a different location and repeat.

ANALYSIS

1) Create a table listing α for each of the conditions used in 1).
2) Observe any differences in the 002 diffracted intensity on <100> and <110> zones.
3) Determine ξ and T for each two-beam disk:
   a) Measure $2x_i$ for all visible rocking-curve oscillations in the disk.
   b) Compute s_i for each fringe pair.
   c) Plot s_i^2 vs. n_i^2 and fit the curve.
   d) Adjust Δn for each pattern to find a consistent solution for ξ.

REPORT

Tabulate the values of α measured in 1). Include and describe representative diffraction patterns. Submit calculations of ξ and T, including the graphs.