NANO 703/703L
Lab: TEM Dark-Field Imaging
Due: Following Lab Session

BACKGROUND

TEM dark-field (DF) imaging is a useful mode of operation for the analysis of crystalline materials. The primary contribution to dark-field images is diffraction contrast, where the image intensity is directly determined by the local scattering strength for a particular Bragg reflection. Different reflections may be sensitive to different microstructural characteristics, such as defects and composition. We will use DF imaging to extract specific material properties for a few different thin film systems. Centered DF imaging should be used whenever possible, to improve image resolution.

Strain Contrast

One appeal of TEM for materials science is the extent of information that can be obtained on crystallographic defects, particularly dislocations. The observation of dislocations by TEM is widely cited as one the early successes of the technique. A set of DF images can be used to infer dislocation character. The Burger's vector **b** can often be deduced from a set of DF images acquired with different active **g** vectors. The invisibility criterion (for a screw dislocation) stipulates that a dislocation is not observed when $\mathbf{g} \cdot \mathbf{b} = 0$. This condition arises because the inhomogeneous strain field associated with the dislocation must have a component parallel to the active imaging reflection to generate contrast. Thus, a single image using reflection *g*, showing no direct contribution from a particular dislocation, limits the possible orientations of **b** to a plane normal to **g**. If this dislocation is observed to interact with other dislocations, relationships among the Burger's vectors for the collection of dislocations can be established.

One region where the presence of dislocations is assured is at the interface between a relatively thick film and a substrate, when the film has a net lattice mismatch with respect to the substrate, i.e., a difference in lattice constant. This leads to the formation of a misfit-dislocation network, which is situated in the interface plane to relieve coherency strain.

Composition Contrast

Dark-field imaging is an excellent means to generate composition contrast among layers with different alloy compositions, particular when the layers have the same crystallographic orientation. This is especially true for alloys and epitaxial multilayers, when the reflections used for imaging have nearly zero structure factor for a random, solid solution of the alloy or multilayer constituents, but increase in magnitude when the components are segregated. Any local variations in composition will give strong contrast in a DF image formed with a reflection of this type.

Grain Structure

For a polycrystalline specimen, a reflection can be used that highlights only grains having nearly the same orientation. A composite of several images gathered from the same region, using different reflections, can provide an excellent representation of grain distribution.

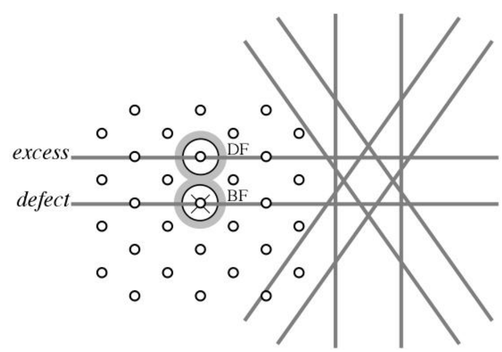

FIG: Bright-field (BF) and dark-field (DF) images are typically formed near a two-beam condition, with the excess and defect Kikuchi lines passing through *g* and *0*, respectively.

EXPERIMENT

Acquire TEM DF images of the following materials:

1) An [001]-oriented film prepared in plan-view (PV) or cross-section (x-sec).
a) Orient the specimen near the (PV) [001] or (x-sec) <110> zone axis;
b) Tilt to a 220 two-beam condition;
c) Generate a 220 centered DF image.

2) A compositional superlattice:
a) Orient the specimen near the (PV) [001] or (x-sec) <110> zone axis;
b) Tilt to an 002 two-beam condition and generate an 002 DF image;

3) A polycrystalline film.
a) Align the microscope for hollow-cone DF imaging;
b) Generate conical-DF images using a small objective aperture.

ANALYSIS

1) For the relaxed film, measure either:
a) (PV) the misfit dislocation density
  i) Measure the distance L spanned by a number N of misfit dislocations;
  ii) Compute the misfit dislocation density $\eta = N / L$. Express η in units of cm^{-1}.
b) (x-sec) the layer thicknesses of the various layers;

2) For the superlattice, measure the period as follows.
a) Measure the distance L spanned by a number N of superlattice periods;
b) Compute the period from $d = L / N$;
c) Calculate the period by measuring $2/d$ from an FFT of an 002 DF image.

3) For the polycrystalline film, measure the average grain size for several representative grains.
For each grain:
a) Estimate the centroid position of the grain.
b) Measure the maximum dimension $L_{\max}$ by drawing the longest possible line spanning the grain that passes through the centroid;
c) Measure the minimum dimension $L_{\min}$ by drawing the shortest possible line spanning the grain that passes through the centroid;
d) Compute the grain area as $A = L_{\max} \cdot L_{\min}$.
e) Find the average grain area $\overline{A}$.
f) Compute the average grain size as $\overline{L} = \sqrt{\overline{A}}$.

REPORT

Submit representative images of each sample and results of the analysis.