Weak-Beam Dark-Field Technique

Weak-Beam Dark-Field Imaging – Basic Idea

- recall bright-field contrast of dislocations:
  - specimen close to Bragg condition, $s \neq 0$
  - near the dislocation core, some planes curved to $s = 0$
  $\Rightarrow$ strong Bragg reflection near dislocation core
  $\Rightarrow$ bright-field image shows dark line on bright background
- drawbacks:
  - contrast not great
  - line of minimum intensity:
    - true position of dislocation core
  - image appears "broad" and "diffuse"

Weak-Beam Dark-Field Imaging of Dislocations – Basic Idea

- tilt farther away from Bragg condition
  $\Rightarrow$ only regions directly at the dislocation core are still curved enough to fulfill $s = 0$
  $\Rightarrow$ line of strongest Bragg reflection $\approx$ true position of dislocation core
- but: Bragg reflection becomes weak
Weak-Beam Dark-Field Imaging of Dislocations – Basic Idea

- drawback: Bragg reflection becomes weak
- but:
  - appears weak only because diffraction pattern averages over entire region
  - locally, near the dislocation core, the reflection is strong
- employ Bragg reflection for dark-field imaging
- dislocations appear with sharp, well-localized, and strong contrast

This is the concept of "Weak-Beam Dark-Field Imaging."

Example: Dislocations in Cu

Adjustment of Diffraction Conditions

- goal: dark-field imaging with "weak" beam
- need to adjust considerably large and well-defined excitation error $s$
- consider systematic row of reflections: $O, g, 2g, 3g, \ldots$
- a large excitation error $s$ of $g$ can be precisely and reproducibly adjusted by setting up exact Bragg condition ($s_n = 0$) for a higher order reflection $n\mathbf{g}$ of the systematic row
- precise adjustment of $s_n = 0$ can be achieved with the help of Kikuchi lines

This setup is denoted as $g - n\mathbf{g}$ diffraction condition.
WBDF: Observation of Dislocation Dissociation

- Burgers vector $\mathbf{b}$ of a dislocation: displacement between region above and below the cut plane
- complete crystal dislocation: $\mathbf{b} = h^i \mathbf{a}_i$, $h^i \in \mathbb{Z}$
- line energy of dislocations:

$$E_L = \frac{G b^2}{4\pi(1-\nu)} \left(1 - \nu \cos^2(\vartheta)\right) \ln \left[ \frac{c_r}{b} \right]$$

$G$: shear modulus; $\nu$: Poisson ratio; $b$: Burgers length; $\vartheta$: dislocation character (angle between $\mathbf{b}$) and local line direction $s$, $c_r$: parameter characterizing the core radius.

- $E_L \propto b^2$

WBDF: Observation of Dislocation Dissociation

- $E_L \propto b^2 \Rightarrow$ energy can be released by dissociation

$$\mathbf{b} \rightarrow \mathbf{b}_1 + \mathbf{b}_2$$

if

$$b_1^2 + b_2^2 < b^2$$

- $\mathbf{b}_1, \mathbf{b}_2$: Burgers vectors of partial dislocations
- not lattice translations $\Rightarrow$ stacking fault inbetween

WBDF: Observation of Dislocation Dissociation

- repulsive interaction between partial dislocations: $\propto x_f^{-1}$
- attractive interaction due to stacking fault energy $\gamma_f$: $\propto x_f$
- result: equilibrium (minimum total energy) corresponds to finite stacking fault width $x_f$

$$x_f = x_f[\gamma_f]$$

- $x_f$: parameter of key importance for mechanical behavior!
- weak-beam dark-field imaging can determine stacking fault energy $\gamma_f$ (temperature dependence of plasticity)

$\xrightarrow{\gamma_f}$ parameter of key importance for mechanical behavior!
**WBDF: Observation of Dislocation Dissociation**

*Example: Cu*

**STEM:**

*Scanning TEM*

- conventional ("still") of TEM: whole specimen area is illuminated simultaneously
- STEM: specimen is scanned point-by-point with focused electron probe, similar to scanning electron microscopy
  - but here: transmission, ⇒ much better resolution!
- realization:
  - pre-field of objective lens as additional condenser lens
  - small electron probe at the specimen, $\varnothing \approx 1 \text{ nm}$
  - no further lenses are needed below the objective lens!
  - TEM/STEM instruments: further lenses may be excited (e.g. to image diffraction pattern through small bores)
STEM Principle

Ray Path in STEM Mode

STEM – Scanning TEM

- saw-tooth voltage generator:
  → acts on deflection coils to scan the beam
  - simultaneously deflects, in synchronism, electron beam of a cathode-ray tube
  - intensity of the CRT beam can be modulated by any signal from the electron-specimen interactions

- bright-field and dark-field mode
  (select by placing large circular or sector diaphragms in front of detector)

- detectors: semiconductor or scintillator-photomultiplier
- detectors “see” stationary Fraunhofer diffraction pattern!

STEM versus TEM

- TEM: small illumination aperture \( \alpha_i \)
- STEM: small electron probe requires large illumination aperture, \( \alpha_i \approx 10 \text{ mrad} \)
- detector aperture \( \alpha_d \) has to be matched
- bright field mode: \( \alpha_d \approx \alpha_i \)
- otherwise: noise
- important options
  1. annular detector
     → record forward-scattered electrons with \( \theta \approx 10^\circ \)
     → “Z contrast” (thermal-diffuse scattering)
**STEM – Annular Darkfield Detector**

2. backscattered electron (BSE) detector
3. secondary electrons (SE) detector
   - exit energies of secondary electrons only \(5\text{..}50\text{ eV}\)
   - spiral trajectories owing to strong axial magnetic field
   - detection by scintillator-photomultiplier combination
   - image the surface structure of the specimen

- important advantages of STEM over TEM:
  - avoids chromatic aberration by inelastic scattering in thick specimens
  - STEM with field-emission gun: very high beam currents – great for analytical work!

**Diffraction Patterns in STEM Mode**

**The Reciprocity Principle**
The Reciprocity Theorem

- scanning unit between source and objective lens deflects the electron probe in a raster across the specimen
- projected backwards, the rays scan over a virtual source, which corresponds to the image plane of a TEM
  - in STEM, the CRT is needed to image this virtual plane by modulating the CRT with the detector signal

STEM versus TEM – Examples

am SiO₂ + Cl bubbles

TEM

STEM

STEM (processed)

stem versus TEM – Examples

two-phase polymer

TEM BF

STEM BF

TEM DF

STEM ADF

STEM versus TEM – Examples

two-phase polymer

TEM DF

STEM ADF