TEM Imaging and Diffraction Examples

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Objective lens focus

- A full ray diagram schematic of the objective lens will go from object plane to image plane and shows both focusing of the objective lens and diffraction pattern formation in the back focal plane — see first TEM lectures and exercises today.

- The image plane of the objective lens forms the object plane for the next lens in the series (i.e. the first intermediate lens); this is defined as the image plane. That is, the two lenses are coupled.

- At correct focus a point object is focused to a point in this image plane.

- If we decrease objective lens strength the rays come to a point below this plane; this is called “under focus”. At the image plane there is an out of focus image which is then projected onto our detector.

- This “under focus” image is basically equivalent to having correct objective focus but moving the sample down.

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Objective lens focus

- Very important: when the sample is in focus there is minimum contrast (see phase contrast lectures).

- Quiz 1: which of these images is in focus?

- Image 3 is in focus: no Fresnel fringe at edge of hole, no specular (“speckled”) contrast in the carbon film, therefore it has little contrast.
Objective lens astigmatism

- When image is astigmatic different axes in the image plane have different focal points.
- This can be seen as different Fresnel fringes for different image directions.
- Here seen for different objective foci.

Which of these images of GaN nano-wires was taken with an objective aperture?
Image delocalization

TEM image with no objective aperture. Image formed from direct beam and diffracted beams. Dark-field images from diffracted beams delocalize from bright-field image of direct beam. Gives shadow images that move with objective focus (draw ray diagrams for out of focus image).

Image delocalization

Image of same nanowires but with objective aperture to make bright-field image. No diffracted beams => no shadow images. *This is how you should take your TEM data!*
Diffraction contrast on/off zone axis

In bright-field imaging, zone axis condition => more scattering to diffracted beams. Therefore intensity in direct beam goes down and bright-field image has strong contrast.

Example: GaN nanowire

Bend contours

From Williams & Carter: Transmission Electron Microscopy
Bend contours

Example: mechanically polished and ion beam milled silicon

Bend contours

Example: electropolished Ni$_3$Al superalloy

Bright-field images. Same region at different sample tilts.
See bend contours interacting with dislocations.
Bend contours

Example: electropolished Ni$_3$Al superalloy

Zone axis Kikuchi pattern in real space

Sample at lower magnification.

Twinning in diffraction

Example: FCC twins
Stacking of close-packed \{1 1 1\} planes reversed at twin boundary:

\[
\begin{align*}
\rightarrow A & B & C & A & B & C & B & A & C
\end{align*}
\]

View on [1 1 0] zone axis:

\{1 1 1\} planes:
**Twinning in diffraction**

Example: Co-Ni-Al shape memory FCC twins observed on \([1 \ 1 \ 0]\) zone axis

\((1 \ 1 \ 1)\) close-packed twin planes overlap in SADP

Images provided by Barbora Bartová, CIME

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**Epitaxy and orientation relationships**

SADP excellent tool for studying orientation relationships across interfaces

Example: Mn-doped ZnO on sapphire

Zone axes:
\([1 \ -1 \ 0]_{\text{ZnO}} \parallel [0 \ -1 \ 0]_{\text{sapphire}}\)

Planes:
\(c-\text{plane}_{\text{ZnO}} \parallel c-\text{plane}_{\text{sapphire}}\)
Ring diffraction patterns

If selected area aperture selects numerous, randomly-oriented nanocrystals, SADP consists of rings sampling all possible diffracting planes - like powder X-ray diffraction

Example: “needles” of contaminant cubic MnZnO$_3$ - which XRD failed to observe! Note: if scattering sufficiently kinematical, can compare intensities with those of X-ray PDF files

Larger crystals => more “spotty” patterns

Example: ZnO nanocrystals ~20 nm in diameter
Ring diffraction patterns

“Texture” - i.e. preferential orientation - is seen as arcs of greater intensity in the diffraction rings

Example: hydrozincite $\text{Zn}_5(\text{CO}_3)_2(\text{OH})_6$ recrystallised to ZnO crystals 1-2 nm in diameter

Nanocrystalline sample image/diffraction

Bright field image setup - select direct beam with objective aperture

Contrast from different crystals according to diffraction condition
Nanocrystalline sample image/diffraction

Dark field image setup - select some transmitted beams with objective aperture

Diffraction mode

Image mode

Only crystals diffracting strongly into objective aperture give bright contrast in image
Nanocrystalline sample image/diffraction

DF images allow us to pick out individual grains

Amorphous diffraction pattern

Crystals: short-range order and long-range order

Amorphous materials: no long-range order, but do have short-range order (roughly uniform interatomic distances as atoms pack around each other)

Short-range order produces diffuse rings in diffraction pattern

Example:

Vitrified germanium
(M. H. Bhat et al. Nature 448 787 (2007))
Diffraction contrast summary

- Materials science specimen: CVD-grown ZnO thin film. Elastic scattering of the electrons (i.e. diffraction) by crystal is major contribution to contrast.

- Changes in crystal orientation from grain to grain or within grains at defects change the diffraction condition and hence the contrast.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Incident beam</th>
<th>Backscattered electrons (BSE)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-100 nm</td>
<td>direct beam</td>
<td>inelastically scattered electrons</td>
</tr>
<tr>
<td>&quot;absorbed&quot; electrons</td>
<td>inelastically scattered electrons</td>
<td></td>
</tr>
</tbody>
</table>

Amorphous? Or low diffraction contrast?

Bright-field image of Al-Cr-N-O thin film

- Some regions show clear diffraction contrast, so obviously crystalline.
- Other regions did not. Combined with contrast from probable amorphous surface layer, looked amorphous.
- However CBED proved crystalline, just in low diffraction contrast condition.
Phase contrast, grain boundaries, bend contours, dislocations in one image

Summary

When imaging crystalline samples in the TEM, any change in the crystal will change the elastic scattering diffraction condition and hence intensity and contrast in the resulting image. This can be from:

- crystal orientation relative to the electron beam and deviations from bending
- defects in the crystal
- changes in phase (e.g. precipitates)
- changes in thickness (dynamical scattering).

Using an objective aperture to select the direct beam (bright-field image) or diffracted beam (dark-field image) combined with carefully selected diffraction conditions gives many possibilities for analysing and understanding your specimen.

Even without pursuing this you still have to learn to live with diffraction contrast and how it can unhelpfully affect your TEM data (e.g. bend contours). In this way TEM is not like other types of microscopy!

No objective aperture => ill-defined diffraction condition and image delocalization!