Chapter 4  Imaging

Lecture 17
Imaging

- Imaging in the TEM
- Diffraction Contrast in TEM Image
- HRTEM (High Resolution Transmission Electron Microscopy) Imaging
- STEM imaging
Imaging in the TEM

• What is the contrast?

In microscopy, contrast is the difference in intensity between a feature of interest ($I_s$) and its background ($I_0$). Contrast is usually described as a fraction such as:

$$C = \frac{I_s - I_0}{I_0} = \frac{\Delta I}{I_0}$$

You won’t see anything on the screen or on the photograph, unless the contrast from your specimen exceeds 5-10%.
Contrast
The difference between foreground and background colors determines text legibility.
In optical micrograph, contrast indicates the grain boundary and grain orientation.

In TEM micrograph, contrast indicates many information about the microstructure of specimen.
Contrast indicates precipitates, dislocations, and thickness variation in Ni-Cr-Ti-Al alloy, Hirsch, Howie, Nicholson, Pashley, Whelan, 1977
Intensity and Contrast

• Intensity and contrast are two different conception.

• We can have strong or weak contrast but not bright or dark contrast

• The term “bright” and “dark” refer to the intensity, i.e. the density of electrons hitting the screen/detector (number of electrons/unit area)

• We usually get the strongest contrast under illumination conditions that lower the overall intensity. By C2 condenser lens, we can adjust the intensity.
Schematic intensity profiles across an image showing (a) different intensity levels ($I_s$ and $I_0$), and the difference ($\Delta I$) between them, which define the contrast. Generally, in a TEM, if the overall intensity is increased (as shown in figure b) the contrast decrease.

\[ C = \frac{I_s - I_0}{I_0} = \frac{\Delta I}{I_0} \]
Images and Diffraction Patterns (DP)

- The uniform electron intensity in incident beam is transformed into a non-uniform intensity after scattering by the specimen as seen in figure.
- We also know DP shows this non-uniformity.
- A basic procedure to image in the TEM is: first view the DP, since the pattern tells you how your sample is scattering.
- The relationship between the image and the DP is the most critical for crystalline specimen showing diffraction contrast.
- You need to view the DP first, whatever contrast mechanism you want to use, and whatever specimen you are studying.

Monte Carol simulation of the electron scattering when an incident electron beam passes the specimen

- The procedure to interpret TEM image usually follows: qualitatively → semi-quantitatively → quantitatively
How to obtain TEM images

- Using the objective aperture to form BF or DF images
- Using the STEM detector to form BF and DF image
- HRTEM: usually without aperture

To obtain meaningful TEM image

- Careful preparation of sample. Garbage in, and garbage out
- Proper setting-up the optics condition
- Study your materials before TEM session
- Correct operation of instrument
- What’s your question?
- Design the lab plan
- More than one session is necessary
- Please add more tips from your practice, share and discuss your experience.
Incorrect results come from incorrect usage
Using the objective aperture to form BF or DF images

When we form images in TEM, we either form an image using the central spot, or we use some or all of the scattered electrons. The way we choose which electrons form the image is to insert an aperture into the BFP (back focal plane) of the objective lens, thus blocking out most of the diffraction pattern except that which is visible through the aperture. We use the external drives to move the aperture so that either the direct electrons or some scattered electrons go through it. If the direct beam is selected we call the resultant image a **bright-field image (BF image)**, and if we select scattered electrons of any form, we call it a **dark-field image (DF image)**.
Parallel beam condition

Center dark field (CDF) image

Off-line dark field (ODF) image or “dirty” dark field image
Using the STEM detector to form BF and DF image: Convergent beam condition

STEM image formation:
A Bright Field (BF) detector is placed in a conjugate plane to the back focal plane to intercept the direct beam while a concentric Annular Dark Field (ADF) detector intercepts the diffracted electrons.

The signals from either detector are amplified and modulate the STEM CRT. The specimen (Au islands on a C film) gives complementary ADF and BF images as can be seen by clicking the button opposite.
In summary, BF and DF images (for TEM and STEM) are formed by using the transmitted and diffracted beam respectively. In order to understand and control the contrast in these images, we need to know what features of a specimen cause scattering and what aspects of TEM operation affect the contrast.

**Mechanism of Contrast for TEM/STEM image**

1. **Mass-thickness contrast**: primary contrast source of TEM/STEM image for non-crystalline materials such as polymers and biological materials.
2. **Z-contrast**: High resolution STEM image
3. **Diffraction contrast**: primary contrast source of BF/DF image especially for crystalline materials such as metals. Amplitude of e-wave contributes to the contrast.
4. **Phase contrast**: High/low resolution TEM image (atomic lattice image).
5. **Both the amplitude and the phase contrast**: are primary contrast source of electron holography image for magnetic materials
6. In some situations, image contrast may arise from more than one mechanism and one may dominate.
7. Crystalline sample may generate mass-thickness and diffraction contrast when aperture is removed.
Mass-thickness Contrast for TEM imaging

- Mass-thickness contrast arises from incoherent elastic scatter of electron (Ruthford scattering), which is a strong function of the atomic number Z, i.e. the mass or the density, as well as the thickness of specimen, t.

- Mass-thickness contrast dominates if the specimen is amorphous. If the specimen is a crystalline, the mass-thickness contrast will compete with the diffraction contrast mechanism.

- Mechanism is shown in figure

- Example: stained bacteria sample with constant thickness by microtome prep.

- The TEM variables affecting the mass-thickness contrast for a given specimen are the objective aperture and the acceleration voltage. Increasing aperture lowers the contrast, and lowering acceleration voltage increases the contrast but reduce the intensity.
Darker contrast

Brighter contrast

The reverse contrast will be formed in DF imaging mode

Less scattering electrons in low mass region

More scattering electrons in high mass region

Mechanism of mass-thickness contrast in a BF image. Thicker or higher-Z areas of the specimen (darker) will scatter more electrons off axis than thinner or lower-mass (lighter) areas. Thus fewer electrons from the darker region fall on the equivalent area of the image plane (and subsequently the screen), which therefore appears darker in BF images.
Impact of Microorganism (e.g. Leptothrix discophora) on the Precipitation and Migration of Metals and Actinides. Samples were sliced into 70 nm by microtome, and then stained using Os, U, and Z etc heavy elements. The contrast are primarily from mass-contrast, since the thickness is constant, 70 nm (Lisa Mullen, former PhD student, HRC).
A smaller aperture enhances the mass-thickness contrast in a similar manner to lowering the kV.
Mass-thickness Contrast for STEM imaging

- In a STEM mode, by varying the camera length, L, we change the collection angle of detector.
- STEM image has more noise and poorer resolution than TEM image, but higher contrast than TEM image.
- In contrast is more important than resolution, STEM imaging is more useful.
- STEM imaging is also useful if the specimen is beam sensitive such as some polymers.
- Examples
STEM showing bright contrast of stained bacteria

TEM showing dark contrast of stained bacteria
Quantitative Mass-thickness Contrast

1. Atom number, $Z$, is constant, and thickness, $t$, is varied

$$C = \frac{\Delta I}{I} = 1 - e^{-Q\Delta t} \approx Q\Delta t$$

for $\Delta t < 1$, $Q$ is the total elastic scattering cross section.

Since the minimum contrast we can see is $\sim 5\%$, then the minimum $\Delta t$ that we can see is

$$\Delta t \approx \frac{5}{100Q} = \frac{5A}{100N_0\sigma \rho}$$

where $A$ is the atomic weight, $N_0$ is Avogadro's number, $\sigma$ is the single-atom scattering cross section, and $\rho$ is the density.
Quantitative Mass-thickness Contrast

2. Atom number, Z, is not constant

The atomic scattering factor, \( f(\theta) \),

\[
f(\theta) = \left( 1 + \frac{E}{E_0} \right) \left( \frac{\lambda}{\sin \frac{\theta}{2}} \right)^2 \left( Z - f_x \right)
\]

The scattering cross section from an angle \( \beta \)

\[
\sigma(\beta) = 2\pi \int_0^\infty \left| f(\theta) \right|^2 \theta d\theta
\]

\[
\sigma(\beta) = \frac{Z\lambda \left( \frac{a_0}{Z^{0.33}} \right) \left( 1 + \frac{E}{E_0} \right)^2}{\pi (a_0)^2 \left( 1 + \left( \frac{\beta}{\theta_0} \right)^2 \right)}
\]

E: Beam Energy after passing the specimen
E0: the beam energy
Z: atomic number
a0: Bohr radius
fx: scattering factor for X-ray
\( \omega \): scattering angle
\( \lambda \): wavelength

A0: the Bohr radius,
\( \Theta_0 \): the characteristic screen angle.
B: the semi-angle of collection of the objective aperture
Assume that \( n \) electrons are incident on the specimen and \( dn \) electron are scattered through an angle > \( \beta \). Then we can simplify the above equations, ignoring any inelastic scattering. Therefore the reduction in the number of electron going through the objective aperture to form the BF image is given by

\[
\frac{dn}{n} = -N\sigma(\beta)dx \quad (a)
\]

where \( N = N_0 / A \), \( N_0 \) is Avogadro's number, and \( x = \rho t \).

Integrate equation (a)

\[
\ln n = N\sigma x + \ln n_0
\]

or \( n = n_0 e^{-N\sigma x} \)

So as the specimen mass-thickness \( (x = \rho t) \) increases, the number of scattered electrons decreases exponentially.

In principal, the contrast arising from \( Z \) and \( t \) can be calculated by the above equations. In practice, however, image contrast calculation are not carried out for simple mass-thickness contrast in materials specimens.
Z contrast

• Z contrast usually refers to STEM high resolution image
• The image is formed by HAADF (high annular angle dark field) detector as shown in figure
• Imaging beam condition is always away two-beam condition and close to zone-axis orientation
• STEM Z-contrast is not equivalent to the TEM Z-contrast image (mass-thickness contrast). STEM image always contains some diffraction contrast
• High resolution Z-contrast will be discussed later.

Schematic of the HADDF detector set-up for Z-contrast imaging in a STEM. The conventional BF detector is also shown along with the range of electron scattering angle gathered by the detectors.
Heavy-Ion-Irradiated interface of ZrN

Low magnification TEM BF image showing the dislocation loop and interface. The gap of interface with substrate is not very clear due to diffraction contrast.

In Z-contrast condition, the irradiated interface is very clear.
Diffraction contrast

- Diffraction contrast imaging uses the coherent elastic scattering beam to form image.
- Diffraction contrast imaging is controlled by the Bragg diffraction through variation of crystal structure and orientation of specimen.
- Diffraction contrast is simply a special form of amplitude contrast because the scattering occurs at Bragg angle.
- BF and DF image is diffraction contrast image by selecting the direct or diffracted beam as seen in the figure.
- Beam condition of diffraction contrast imaging: the incident beam must be parallel in order to give the sharp diffraction spots and thereby strong diffraction contrast. So we need to underfocus C2 to spread the beam.

Parallel beam condition
Two-beam conditions for diffraction contrast imaging for defects study, especially for metallic materials

• To get good strong diffraction contrast in both BF and DF image, we usually tilt the specimen to two-beam conditions, in which only one diffracted beam is strong. The direct beam is the other strong spot in the pattern.

• The electrons in the strongly excited \{hkl\} beam has been diffracted by a specific set of \{hkl\} plane. So the area that appears bright in the DF image is the area where \{hkl\} plane at the Bragg condition.

• The DF image contains specific orientation information, not just general scattering information as is the case for mass-thickness contrast.

• Two-beam conditions are not only necessary for good contrast, they also greatly simplify interpretation of image.
**Diffraction contrast** can be primarily used for imaging the crystalline materials including defects. The DP with defects is different than the DP without defects. It is analogous to the 2-D wall structure.

2-dimensional arrangements of bricks

Brick patterns are represented by commas as motifs showing the difference
Two-beam conditions for diffraction contrast imaging

BF/DF image pairs along with two-beam diffraction of a series of dislocation and a stacking fault in Cu-15at%Al.
Setting up of the two-beam conditions

• While looking at the DP, tilt around until only one diffracted beam is strong as shown in figure.

• The other diffracted beams don’t disappear because of the relaxation of the Bragg conditions, but they are relatively faint.

• In this condition, the contrast might still not be the best (why?). To get the best contrast from defects, the specimen shouldn’t be exactly at the Bragg condition (s=0). The specimen should be tilted close to the Bragg condition, and s is small and positive (the excess \{hkl\} kikuchi line is just outside the \{hkl\} spot).

• Never form strong-beam image with s negative; the defects will be difficult to see.
(a) The [001] zone-axis diffraction pattern showing many planes diffracting with equal strength. In the smaller patterns, the specimen is tilted so there are only two strong beams, the direct [000] on-axis beam and a different one of the \{hkl\} off-axis diffracted beam. (b) and (c) showing the complementary BF and DF image under two-beam conditions. In (b), the precipitates is diffracted strongly and appears dark. In (c), it appears bright.