TEM Crystallography and Diffraction

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Contents

- Introduction to electron diffraction
- Basics of crystallography and symmetry
- Electron diffraction theory
  - Bragg law and 2-beam electron diffraction
  - Reciprocal lattice and Ewald sphere
  - Multiple beam scattering
  - Shape effects in reciprocal space
  - Deviation from Bragg and the excitation parameter
- Summary
Diffraction pattern formation

- In back focal plane of objective lens parallel rays focused to point
- Diffraction – coherent scattering – creates sets of parallel rays from different crystal planes
- Focusing of these parallel rays in back focal plane creates spots of strong intensity: the diffraction pattern
Why use electron diffraction?

Diffraction: constructive and destructive interference of waves

✔ wavelength of fast moving electrons much smaller than spacing of atomic planes
  => diffraction from atomic planes (e.g. 200 kV e, \( \lambda = 0.0025 \) nm)

✔ electrons interact very strongly with matter => strong diffraction intensity
  (can take patterns in seconds, unlike X-ray diffraction)

✔ spatially-localized information
  (≈ 200 nm for selected-area diffraction; 2 nm possible with convergent-beam electron diffraction)

✔ close relationship to diffraction contrast in imaging
  ✔ orientation information
  ✔ immediate in the TEM!

(✘ diffraction from only selected set of planes in one pattern - e.g. only 2D information)

(✘ limited accuracy of measurement - e.g. 2-3%)

(✘ intensity of reflections difficult to interpret because of dynamical effects)

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Image formation

BaTiO₃ nanocrystals (Psaltis lab)
Image formation

Insert selected area aperture to choose region of interest

BaTiO$_3$ nanocrystals (Psaltis lab)

Press “D” for diffraction on microscope console - alter strength of intermediate lens and focus diffraction pattern on to screen

Find cubic BaTiO$_3$ aligned on [0 0 1] zone axis
Basic crystallography & symmetry

Crystals: translational periodicity & symmetry

Repetition of translated structure to infinity
Crystallography: the unit cell

Unit cell is the smallest repeating unit of the crystal lattice
Has a lattice point on each corner (and perhaps more elsewhere)

Defined by lattice parameters $a, b, c$ along axes $x, y, z$
and angles between crystallographic axes: $\alpha = b c, \beta = a c, \gamma = a b$

Building a crystal structure

Use example of CuZn brass
Choose the unit cell - for CuZn: primitive cubic (lattice point on each corner)
Building a crystal structure

Use example of CuZn brass
Choose the unit cell - for CuZn: primitive cubic (lattice point on each corner)
Choose the motif - Cu: 0, 0, 0; Zn: \( \frac{1}{2}, \frac{1}{2}, \frac{1}{2} \)

Structure = lattice + motif => Start applying motif to each lattice point
Building a crystal structure

Use example of CuZn brass
Choose the unit cell - for CuZn: primitive cubic (lattice point on each corner)
Choose the motif - Cu: 0, 0, 0; Zn: 1/2, 1/2, 1/2
Structure = lattice + motif => Start applying motif to each lattice point
Extend lattice further in to space

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Introduction to symmetry

Flamingos know they are beautiful and strange, and play at symmetry.

Excerpt from Bruno Munari’s “Zoo”, First Chronical Books
Introduction to symmetry

As well as having translational symmetry, nearly all crystals obey other symmetries - i.e. can reflect or rotate crystal and obtain exactly the same structure

Symmetry elements:

Mirror planes:

Rotation axes:

Centre of symmetry or inversion centre:

Inversion axes: combination of rotation axis with centre of symmetry

Example - Tetragonal lattice: \( a = b \neq c; \alpha = \beta = \gamma = 90^\circ \)

Anatase TiO\(_2\) (body-centred lattice) view down [0 0 1] (z-axis):
Introduction to symmetry

Example - Tetragonal lattice: \( a = b \neq c; \alpha = \beta = \gamma = 90^\circ \)

Anatase TiO\(_2\) (body-centred lattice) view down [0 0 1] (z-axis):
Identify mirror planes

Identify rotation axis: 4-fold = defining symmetry of tetragonal lattice!
More defining symmetry elements

**Cubic crystal system:** \(a = b = c; \alpha = \beta = \gamma = 90^\circ\)
View down body diagonal (i.e. \([1 1 1]\) axis)
Choose Primitive cell (lattice point on each corner)
Identify rotation axis: 3-fold (triad)
Defining symmetry of cube: four 3-fold rotation axes (not 4-fold rotation axes!)

![Cubic Crystal System Diagram](image)

More defining symmetry elements

**Hexagonal crystal system:** \(a = b \neq c; \alpha = \beta = 90^\circ, \gamma = 120^\circ\)
Primitive cell, lattice points on each corner; view down \(z\)-axis - i.e.\([1 0 0]\)
Draw 2 x 2 unit cells
Identify rotation axis: 6-fold (hexad) - defining symmetry of hexagonal lattice

![Hexagonal Crystal System Diagram](image)
The seven crystal systems

7 possible unit cell shapes with different symmetries that can be repeated by translation in 3 dimensions
=> 7 crystal systems each defined by symmetry

<table>
<thead>
<tr>
<th>Crystal System</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Triclinic</td>
<td>$\alpha, \beta, \gamma \neq 90^\circ$</td>
</tr>
<tr>
<td>Monoclinic</td>
<td>$\alpha \neq 90^\circ$, $\beta, \gamma = 90^\circ$</td>
</tr>
<tr>
<td>Orthorhombic</td>
<td>$a \neq b \neq c$</td>
</tr>
<tr>
<td>Tetragonal</td>
<td>$a \neq c$</td>
</tr>
<tr>
<td>Rhombohedral</td>
<td>$a, \beta, \gamma \neq 90^\circ$</td>
</tr>
<tr>
<td>Hexagonal</td>
<td>$a = c$</td>
</tr>
<tr>
<td>Cubic</td>
<td>$a = b = c$</td>
</tr>
</tbody>
</table>

Diagrams from www.Wikipedia.org

Four possible lattice centerings

P: Primitive - lattice points on cell corners

I: Body-centred - additional lattice point at cell centre

F: Face-centred - one additional lattice point at centre of each face

A/B/C: Centred on a single face - one additional lattice point centred on A, B or C face

Diagrams from www.Wikipedia.org
14 Bravais lattices

Combinations of crystal systems and lattice point centring that describe all possible crystals

- Equivalent system/centring combinations eliminated \(\Rightarrow\) 14 (not \(7 \times 4 = 28\)) possibilities

Diagrams from www.Wikipedia.org

<table>
<thead>
<tr>
<th>Crystal System</th>
<th>Defining Symmetry (rotation or inversion)</th>
<th>Conventional Unit Cell</th>
<th>Conventional Lattice Types</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cubic</td>
<td>4 triads (a = b = c) (\alpha = \beta = \gamma = 90^\circ)</td>
<td>P, I, F</td>
<td></td>
</tr>
<tr>
<td>Hexagonal</td>
<td>1 hexad (a = b \neq c) (\alpha = \beta = 90^\circ, \gamma = 120^\circ)</td>
<td>P</td>
<td></td>
</tr>
<tr>
<td>Trigonal</td>
<td>1 triad (a = b \neq c) (\alpha = \beta = 90^\circ) (\gamma = 120^\circ)</td>
<td>P, R</td>
<td></td>
</tr>
<tr>
<td>Tetragonal</td>
<td>1 tetrad (\alpha = \beta = \gamma = 90^\circ)</td>
<td>P, I</td>
<td></td>
</tr>
<tr>
<td>Orthorhombic</td>
<td>3 diads (a \neq b \neq c) (\alpha = \beta = \gamma = 90^\circ)</td>
<td>P, C, I, F</td>
<td></td>
</tr>
<tr>
<td>Monoclinic</td>
<td>1 diad (a \neq b \neq c) (\alpha = \gamma = 90^\circ, \beta \geq 90^\circ)</td>
<td>P, C</td>
<td></td>
</tr>
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<td>- (\alpha \neq \beta \neq \gamma) (\alpha \neq \beta \neq \gamma)</td>
<td>P</td>
<td></td>
</tr>
</tbody>
</table>
Crystallography – lattice vectors

A lattice vector is a vector joining any two lattice points. Written as linear combination of unit cell vectors \( \mathbf{a}, \mathbf{b}, \mathbf{c} \):

\[
\mathbf{t} = U \mathbf{a} + V \mathbf{b} + W \mathbf{c}
\]

Also written as: \( \mathbf{t} = [U V W] \)

Examples:

\[
\begin{array}{ccc}
[1 & 0 & 0] & [0 & 3 & 2] & [1 & 2 & 1]
\end{array}
\]

Important in diffraction because we “look” down the lattice vectors (“zone axes”)

Crystallography - lattice planes

Lattice plane is a plane which passes through any 3 lattice points which are not in a straight line. Lattice planes are described using Miller indices \((h \ k \ l)\) where the first plane away from the origin intersects the \(x, y, z\) axes at distances:

\[
\begin{align*}
\alpha/h & \text{ on the } x \text{ axis} \\
\beta/k & \text{ on the } y \text{ axis} \\
\gamma/l & \text{ on the } z \text{ axis}
\end{align*}
\]
Crystallography - lattice planes

Sets of planes intersecting the unit cell - examples:

Lattice planes and symmetry

Lattice planes in a crystal related by the crystal symmetry

For example, in cubic lattices the 3-fold rotation axis on the [1 1 1] body diagonal relates the planes (1 0 0), (0 1 0), (0 0 1):

Set of planes \{1 0 0\} = (1 0 0), (0 1 0), (0 0 1), (-1 0 0), (0 -1 0), (0 0 -1)
Weiss Zone Law

If the lattice vector \([UVW]\) lies in the plane \((h k l)\) then:

\[ hU + kV + lW = 0 \]

Electron diffraction:

Electron beam oriented parallel to lattice vector called the “zone axis”

Diffracting planes must be parallel to electron beam
  - therefore they obey the Weiss Zone law
  (*at least for zero-order Laue zone)
Diffraction theory – Bragg law
Path difference between reflection from planes distance $d_{hkl}$ apart = $2d_{hkl}\sin\theta$

$2d_{hkl}\sin\theta = \lambda/2$ - destructive interference

$2d_{hkl}\sin\theta = \lambda$ - constructive interference
Diffraction theory – Bragg law

Path difference between reflection from planes distance $d_{hkl}$ apart $= 2d_{hkl}\sin\theta$

$\Rightarrow$ Bragg law:
$n\lambda = 2d_{hkl}\sin\theta$

Electron diffraction: $\lambda \sim 2–3$ pm
therefore: $\lambda \ll d_{hkl}$
$\Rightarrow$ small angle approximation: $n\lambda \approx 2d_{hkl}\theta$
Reciprocity: scattering angle $\theta \propto d_{hkl}^{-1}$

Rewrite Bragg law:
$\lambda = 2d_{hkl}\sin\theta$
Diffraction theory – 2-beam condition

2-beam condition: strong scattering from single set of planes
Electron wave function and wave vector

- Position-dependent wave function:
  \[ \psi = \psi_0 \exp(-2\pi i k \cdot r) \]

- Amplitude vs. Position
- \( k \) is the wave vector
- \( |k| = \frac{1}{\lambda} \)

Diffraction theory – 2-beam condition
Diffraction theory – 2-beam condition

- By comparing to Bragg law find diffraction vector \( \mathbf{g}_{\mathbf{hkl}} \):
  \[
  |\mathbf{g}_{\mathbf{hkl}}| = \frac{1}{d_{hkl}}
  \]

Example 2-beam diffraction pattern
**Diffraction theory – 2-beam condition**

- By comparing to Bragg law find diffraction vector $g_{hkl}$:
  \[ |g_{hkl}| = \frac{1}{d_{hkl}} \]

- Small angle approximation:
  \[ 2\theta_B = \frac{\lambda}{d_{hkl}} \]

*Important because scattering angles are essentially linear with reciprocal spacing in TEM, and so plane spacings are easy to measure from diffraction patterns!*

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**The reciprocal lattice**

- Construct a lattice where each node represents a possible diffraction event.
- Reciprocal lattice vector: any possible diffraction vector $g_{hkl}$.
- $g_{hkl}$ is perpendicular to $(hkl)$ lattice plane and has length:
  \[ |g_{hkl}| = \frac{1}{d_{hkl}} \]
The reciprocal lattice

- Add plane $d_3$ with $(h_3 \ k_3 \ l_3)$ which is related to planes $d_1$ with $(h_1 \ k_1 \ l_1)$ and $d_2$ with $(h_2 \ k_2 \ l_2)$ by:

$$(h_3 \ k_3 \ l_3) = (h_1+h_2 \ k_1+k_2 \ l_1+l_2)$$

- In this case find:

$$d_3^* = d_1^* + d_2^*$$

The reciprocal lattice basis vectors

- Now define reciprocal lattice basis vectors $a^*, b^*, c^*$:

$$a^* = d_{100}^* \quad b^* = d_{010}^* \quad c^* = d_{001}^*$$

- Therefore:

$$|a^*| = \frac{1}{d_{100}} \quad |b^*| = \frac{1}{d_{010}} \quad |c^*| = \frac{1}{d_{001}}$$

- For planes $(h00), (0k0), (00l)$:

$$d_{h00}^* = ha^* \quad d_{0k0}^* = kb^* \quad d_{00l}^* = lc^*$$

- Since $d_3^* = d_1^* + d_2^*$:

$$d_{hkl}^* = ha^* + kb^* + lc^*$$
Properties of reciprocal lattice basis vectors

- For plane \((hkl)\): \[ d_{hkl}^* = h a^* + k b^* + l c^* \]
- \(a^*\) is perpendicular to \(b\) and \(c\), hence scalar products:
  - \(a^* \cdot b = 0\)
  - \(a^* \cdot c = 0\)
  - And so on…
- Also obtain scalar products: \(a \cdot a^* = 1\), \(b \cdot b^* = 1\), \(c \cdot c^* = 1\)
- For volume of unit cell \(V_c\):
  - \(a^* = \frac{b \times c}{V_c}\)
  - \(b^* = \frac{a \times c}{V_c}\)
  - \(c^* = \frac{a \times b}{V_c}\)

Visualising the reciprocal lattice

- Instead of drawing vectors, show lattice of points or “nodes”
- Each node represents plane \((hkl)\) in reciprocal space
- Illustrative example: section of reciprocal lattice for BCC metal:
The Ewald sphere

- Diffraction corresponds to elastic scattering (no energy loss)

- Scattering event:
  - $E, \lambda$ constant
  - Momentum changes

- Possible scattering wave vectors $\mathbf{k}_D$ inscribe a sphere: the Ewald sphere

---

The Ewald sphere

- Interact Ewald sphere with reciprocal lattice

- Exact Bragg condition when scattering wave vector $\mathbf{k}_D$ intersects a reciprocal lattice node

- $\mathbf{g}_{hkl} = d_{hkl}$
The Ewald sphere

- Interact Ewald sphere with reciprocal lattice
- Exact Bragg condition when scattering wave vector $k_D$ intersects a reciprocal lattice node
  $$g_{hkl} = d^I_{hkl}$$

Realistic representation of Ewald sphere

- $\lambda \ll d_{hkl}$
  Therefore Ewald sphere almost flat
- Illustration:
  200 keV e$^-$
  BCC $\alpha$-Fe
- Crystal and plane still inclined to e$^-$-beam
Multiple beam scattering

- Crystal aligned on low index lattice vector \([UW]\)
- This is called the "zone axis"
- Diffraction from planes parallel to e-beam
- Multiple beams excited giving diffraction pattern of high symmetry from many planes \((hkl)\)
- Example: diffraction from Si crystal:

![2-beam: \(g_{220}\) excited](image1)

![[001] zone axis pattern](image2)

Diffraction grating analogy

- In zone-axis condition our TEM optics and sample can be analogised to a diffraction grating in Fraunhofer far-field diffraction geometry
- Incident plane wave on diffraction grating
  \[ \Rightarrow \text{periodic array of spots in far-field diffraction plane} \]

\[
\begin{align*}
\text{Incident plane wave} & \quad \Rightarrow \\
\text{Diffraction grating} & \quad \Rightarrow \\
\text{Periodic array of spots} & \quad \Rightarrow \\
\end{align*}
\]

- Note: In "far-field" parallel rays converge to a point, as happens in the back-focal plane of the objective lens. Hence the two are equivalent!
Multiple beam scattering: theoretical example

- Crystal viewed down zone-axis condition is like diffraction grating with crystal planes parallel to e⁻-beam. Obtain diffraction spots perpendicular to plane orientation
- Example: diffraction from primitive tetragonal unit cell with e⁻-beam parallel to $[0 \ 0 \ 1]$ zone axis

$2 \times 2$ unit cells

- Note reciprocal relationship: smaller plane spacing $\Rightarrow$ larger indices $(h \ k \ l)$ & greater scattering angle on diffraction pattern from $(0 \ 0 \ 0)$ direct beam
- Also note Weiss Zone Law obeyed in indexing $(hU + kV + lW = 0)$
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2 x 2 unit cells

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- Also note Weiss Zone Law obeyed in indexing \((hU + kV + lW = 0)\).
In zone-axis condition: one layer of reciprocal lattice ("zero order Laue zone") tangential to Ewald sphere.

Sphere does not intersect reciprocal lattice nodes around (000)  
⇒ Bragg condition not met

However experimentally we have strong scattering from many planes.

Ewald sphere/reciprocal lattice representation

In zone-axis condition: one layer of reciprocal lattice ("zero order Laue zone") tangential to Ewald sphere.

Sphere does not intersect reciprocal lattice nodes around (000)  
⇒ Bragg condition not met

However experimentally we have strong scattering from many planes.

Same even with realistic representation of Ewald sphere/reciprocal lattice.
Fourier transforms in reciprocal space

- Reciprocal lattice is the spatial Fourier transform of real lattice

Each reciprocal lattice node contains frequency information on its corresponding lattice plane

Basic properties of Fourier transform

- Linearity:
  \[ FT(\alpha g(x)) = \alpha FT(g(x)) = \alpha G(u) \]
  \[ FT(g(x) + \beta h(x)) = \alpha FT(g(x)) + \beta FT(h(x)) = \alpha G(u) + \beta H(u) \]

- Shift:
  \[ FT(g(x - a)) = e^{-2\pi i u a} FT(g(x)) \]
  \[ I(u) = (e^{2\pi i u a} FT(g(x))) \cdot (e^{-2\pi i u a} FT(g(x))) = G^*(u) \cdot G(u) \]

- Reciprocity:
  \[ FT(g(ax)) = \frac{1}{|a|} G\left(\frac{u}{a}\right) \]

- Convolution:
  \[ FT(g(x) * h(x)) = FT(g(x)) \otimes FT(h(x)) = G(u) \otimes H(u) \]

- Conservation of angle
Fourier transforms and reciprocal lattice

- Real lattice is not infinite, but is bound disc of material with diameter of selected area aperture and thickness of specimen – i.e. thin disc of material

\[ \mathcal{F}\{\text{lattice}\} \]

\[ \mathcal{F}\{\text{shape}\} \]

= 2 lengths scales in reciprocal space!

Ewald sphere and reciprocal lattice rods

- Elongation of nodes to rods \( \Rightarrow \) “Relaxation” of Bragg condition and hence scattering from many planes at the same time
- Also scattering from higher order Laue zones (HOLZ)
Ewald sphere and reciprocal lattice rods

- Elongation of nodes to rods \(\Rightarrow\) “Relaxation” of Bragg condition and hence scattering from many planes at the same time
- Also scattering from higher order Laue zones (HOLZ)

\[
\begin{align*}
hU + kV + lW &= 0 \\
hU + kV + lW &= 1 \\
hU + kV + lW &= 2 \\
hU + kV + lW &= 3
\end{align*}
\]

Higher order Laue zone (HOLZ) scattering

- Scattering from HOLZ observed at higher angles
- Experimental example from Si on [0 0 1] zone axis

Increase brightness, contrast
**Tilting off zone axis**

- When we tilt the specimen away from a zone axis we see arcs of bright diffraction spots for strongly excited planes.
- These correspond to reciprocal lattice rods intersected by the Ewald sphere.
- While the Ewald sphere/reciprocal lattice is in reality a framework for interpreting diffraction phenomena, it is one that works very well!

**Deviation from Bragg scattering**

- 2-beam scattering at the Bragg condition: consider intensity profile along reciprocal lattice rod.
- Taking simple approximation – “kinematical scattering” – this will be the Fourier transform of the sample thickness.

$$I = I_0 \frac{\sin^2(\pi t s)}{(\pi t s)^2}$$
Deviation from Bragg scattering

- Intensity profile peaks in middle at perfect Bragg condition, then tails with modulations
- Introduce reciprocal lattice distance $s$ along the rod length; $s = 0$ at Bragg
- “Extinction distance” $\xi_g$ can be introduced into expression for intensity $I_g$

$$I_g = \frac{\sin^2(\pi ts)}{(\xi_g s)^2}$$

where:

$$\xi_g = \frac{\pi V_0 \cos \theta_B}{\lambda |F_g|}$$

$V_0$: volume of unit cell
$F_g$: structure factor for reflection

---

Deviation from Bragg scattering

- Exact Bragg condition: Ewald sphere intersects relrod at $s = 0$
  $\implies$ strong intensity in diffraction spot $g$
Deviation from Bragg scattering

- Tilt plane slightly by angle $\Delta \theta$ away from Bragg angle $\theta_B$  
  $\Rightarrow$ much weaker intensity in diffraction spot $g$

Introduce new vector $s_g$ to describe this deviation away from the perfect Bragg condition:

$$k_D = k_1 + g + s_g$$

Known as the “deviation vector”

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Excitation error/deviation parameter

- Define vector $s_g$ parallel to $k_i$; then $s$ is scalar quantity of $s_g$
- $s$ is known both as the “excitation error” or the “deviation parameter”
- $s$ is specified in nm$^{-1}$ (reciprocal space) and follows these sign conventions:
Quiz: graphite → graphene

Diffraction pattern of graphite on [0 0 1] zone axis:
What happens to relrod shape as graphite thinned → graphene?
How will this affect diffraction spot intensity for tilted sample?


Summary on diffraction basics

- Crystals: atomic structures with translational symmetry
- Diffraction: constructive interference of waves from scattering by crystal planes
- Interpret using framework of reciprocal lattice and Ewald sphere
- Electron diffraction has many particular characteristics:
  - Wavelength $\lambda \ll$ plane spacing $d_{hkl}$
  - Scattering angle $2\theta_B = \lambda/d_{hkl}$ \Rightarrow linearity in reciprocal lattice spacing
  - Thin sample leads to elongation of reciprocal lattice node into rod shape (“relrods”) which in turn produces relaxation of Bragg condition and scattering from many different planes at the same time
  - Measure deviation from perfect Bragg condition with parameter $s$
  - Scattering is also typically dynamical (multiple elastic scattering) not kinematical (single elastic scattering); see course on Wednesday
  - Electron diffraction also important because it determines much of the contrast we see in TEM images of crystalline objects: what we know as diffraction contrast imaging