Theory and Practice of Electron Diffraction

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Covered Topics

• Electron diffraction
• Crystal diffraction patterns
• Kikuchi Lines
• Convergent beam electron diffraction
• Electron nanobeam diffraction and examples
Why diffraction:

- Use diffraction patterns to orient crystals for high resolution electron imaging
- Use diffraction patterns to setup diffraction contrast imaging
- Use diffraction patterns for phase identification, crystal orientation determination and structural analysis
I. Electron imaging and diffraction
Formation of Diffraction Patterns
-The Objective Lens Action

Objective Lens

Sample

Diffracted beam

Direct beam

Back focal plane

Illumination  Diffraction  Lens
Figure 9.12. The two basic operations of the TEM imaging system involve (A) projecting the diffraction pattern onto the viewing screen and (B) projecting the image onto the screen. In each case the intermediate lens selects either the back focal plane or the image plane of the objective lens as its object.
II. Choose the electron illumination -Diffraction Techniques

a) Selected Area Electron Diffraction

b) Nanobeam electron diffraction

c) Convergent Beam Electron Diffraction (CBED)
Example of nanobeam illumination

Au nanocrystal

Direct beam
III. Crystal Diffraction

The diffraction condition: diffracted waves from different planes must be in phase

\[ SQ + QT = n\lambda \]

\[ n\lambda = d_{hkl} \sin \theta + d_{hkl} \sin \theta = 2d_{hkl} \sin \theta \quad \text{(Bragg’s law)} \]
Inside TEM

Rewrite the Bragg’s law in 3-D (Laue equation)

\[ 2 \frac{1}{\lambda} \sin \theta = \frac{n}{d_{hkl}} \]

\[ 2 |\vec{K}| \sin \theta = nG \]

\[ |\vec{K}_o| = |\vec{K}| \]

\[ \vec{K} - \vec{K}_o = \vec{g} \]

(Actual diffraction angle is much smaller)
Crystal lattice

- Crystals: with repeated, ordered, atomic arrangements
- Unit cell: (repeating unit)
  - Unit cell vectors, \(a\), \(b\), \(c\)
- Lattice
Lattice planes

Any plane that pass through 3 lattice points (nonsystematic) repeats indefinitely in the crystal lattice (family of planes)

The smallest planar spacing is $d_{hkl}$, hkl Miller indicies
Miller indices

- Take the reciprocal of the intercepts
- Find the smallest multiplication factor
- Enclose three integers in () with no comma in between

<table>
<thead>
<tr>
<th>Intercepts</th>
<th>x</th>
<th>y</th>
<th>z</th>
</tr>
</thead>
<tbody>
<tr>
<td>Intercepts (in terms of lattice parameters)</td>
<td>∞</td>
<td>−1</td>
<td>1/2</td>
</tr>
<tr>
<td>Reciprocals</td>
<td>0</td>
<td>−1</td>
<td>2</td>
</tr>
<tr>
<td>Reductions (unnecessary)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Enclosure</td>
<td></td>
<td></td>
<td>(012)</td>
</tr>
</tbody>
</table>
The reciprocal lattice

- Draw lines from O perpendicular to lattice planes; in general \((hkl)\) is different from \([hkl]\), except in cubic crystals.

- The reciprocal lattice is defined such the distance from \(O\) to a point is the inverse of the planar distance

\[
d^* (hkl) = \frac{1}{d(hkl)}
\]

- Refer \(a^* = d^*(100),\) \(b^* = d^*(010)\) and \(c^* = d^*(001)\)

\[
\vec{d}^* (hkl) = h\vec{a}^* + k\vec{b}^* + l\vec{c}^*
\]

\[
\vec{a} \cdot \vec{a}^* = \vec{b} \cdot \vec{b}^* = \vec{c} \cdot \vec{c}^* = 1 \quad \text{(otherwise zero)}
\]
Reciprocal Lattice Vectors

Reciprocal space lattice vectors

\[ \tilde{a}^* = \frac{\vec{b} \times \vec{c}}{V_{Cell}}, \tilde{b}^* = \frac{\vec{c} \times \vec{a}}{V_{Cell}}, \tilde{c}^* = \frac{\vec{a} \times \vec{b}}{V_{Cell}} \]

\[ V_{Cell} = \left(\tilde{a} \times \tilde{b}\right) \cdot \tilde{c} \]

Useful formula

\[ \left| \tilde{a} \times \tilde{b} \right| = ab \sin \gamma \quad \tilde{a} \cdot \tilde{b} = ab \cos \gamma \]

Relationship between real and reciprocal space

\[ \tilde{a}^* \cdot \tilde{a} = 1, \tilde{a}^* \cdot \tilde{b} = 0, \tilde{a}^* \cdot \tilde{c} = 0 \]

\[ \tilde{b}^* \cdot \tilde{a} = 0, \tilde{b}^* \cdot \tilde{b} = 1, \tilde{b}^* \cdot \tilde{c} = 0 \]

\[ \tilde{c}^* \cdot \tilde{a} = 0, \tilde{c}^* \cdot \tilde{b} = 0, \tilde{c}^* \cdot \tilde{c} = 1 \]
### Table 2.4. Expressions for $d^*(hkl)$ and $d(hkl)$ in the Seven Crystal Systems

<table>
<thead>
<tr>
<th>System</th>
<th>$d^*(hkl)$</th>
<th>$d^2(hkl)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Triclinic</td>
<td>$h^2a^{<em>2} + k^2b^{<em>2} + l^2c^{<em>2} + 2hka^{</em>}c^{</em>} \cos \alpha^{</em>}$</td>
<td>$K^2/d^*(hkl)$</td>
</tr>
<tr>
<td></td>
<td>$+ 2lhe^{<em>}a^{</em>} \cos \beta^{<em>} + 2hka^{</em>}b^{<em>} \cos \gamma^{</em>}$</td>
<td></td>
</tr>
<tr>
<td>Monoclinic</td>
<td>$h^2a^{<em>2} + k^2b^{<em>2} + l^2c^{<em>2} + 2hla^{</em>}c^{</em>} \cos \beta^{</em>}$</td>
<td>$\left{ \frac{1}{\sin^2 \beta} \left[ \frac{h^2}{a^2} + \frac{l^2}{c^2} - \frac{2hl \cos \beta}{ac} \right] + \frac{k^2}{b^2} \right}^{-1}$</td>
</tr>
<tr>
<td>Orthorhombic</td>
<td>$h^2a^{*2} + k^2b^{*2} + l^2c^{*2}$</td>
<td>$\left{ \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \right}^{-1}$</td>
</tr>
<tr>
<td>Tetragonal</td>
<td>$(h^2 + k^2)a^{*2} + l^2c^{*2}$</td>
<td>$\left{ \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2} \right}^{-1}$</td>
</tr>
<tr>
<td>Hexagonal and trigonal (P)</td>
<td>$(h^2 + k^2 + hk)a^{*2} + l^2c^{*2}$</td>
<td>$\left{ \frac{4(h^2 + k^2 + hk)}{3a^2} + \frac{l^2}{c^2} \right}^{-1}$</td>
</tr>
<tr>
<td>Trigonal (R)</td>
<td>$(h^2 + k^2 + l^2 + 2(hk + kl + hl)(\cos \alpha^{*}))a^{*2}$</td>
<td>$a^2(TR)^{-1}$, where $T = h^2 + k^2 + l^2 + 2(hk + kl + hl)[(\cos^2 \alpha - \cos \alpha)/\sin^2 \alpha]$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>and $R = (\sin^2 \alpha)/(1 - 3 \cos^2 \alpha + 2 \cos^3 \alpha)$</td>
</tr>
<tr>
<td>Cubic</td>
<td>$(h^2 + k^2 + l^2)a^{*2}$</td>
<td>$\left{ \frac{h^2 + k^2 + l^2}{a^2} \right}^{-1} = \frac{a^2}{h^2 + k^2 + l^2}$</td>
</tr>
</tbody>
</table>

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$a$ In the monoclinic system, $d(100) = a \sin \beta$, $d(001) = c \sin \beta$, and hence $a = K/(a^{*} \sin \beta^{*})$ and $c = K/(c^{*} \sin \beta^{*})$.

$b$ In the hexagonal system (and trigonal $P$), $a = b = K/(a^{*} \sin \gamma^{*}) = K/(a^{*} \sqrt{3}/2)$, since $\gamma^{*} = 60^\circ$.

(In general, the expressions for $d^*^2$ are simpler in form than the corresponding expressions for $d^2$.)

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**Notes:**
- **DOE BES/DMS:** Materials Science and Engineering/Frederick Seitz Materials Research Laboratory.
Crystal Diffraction Pattern and The Reciprocal Lattice

Bragg’s Law

\[ \sin \theta_B = \frac{g \lambda}{2} \]

\[ g \sim 1/\AA, \quad \lambda \sim 0.025\AA, \quad \theta \sim 12.5\text{mrad} \]
Indexing Diffraction Patterns

The basic equations:

\[ \frac{g}{(1/\lambda)} = \frac{D}{L} \]

\[ g = \frac{D}{L\lambda} \]

For cubic crystals:

\[ g = \sqrt{h^2 + k^2 + l^2} / a \]

h,k,l integers

\[ uh + vk + lw = n \]

n = 0 for zero order

Laue zone

\[ \cos \phi = \frac{h_1 h_2 + k_1 k_2 + l_1 l_2}{\sqrt{h_1^2 + k_1^2 + l_1^2} \sqrt{h_2^2 + k_2^2 + l_2^2}} \]
Find the Crystal Orientation

- The zone axis

- Find two shortest reflections, measure and index to obtain $(h_1,k_1,l_1)$ and $(h_2,k_2,l_2)$
  
- Check the angle between these two and make sure hkl indexing is consistent with the lattice

- Calculate the zone axis using

\[
\vec{Z} = \vec{g}_1 \times \vec{g}_2 = \begin{vmatrix} \vec{a} & \vec{b} & \vec{c} \\ h_1 & k_1 & l_1 \\ h_2 & k_2 & l_2 \end{vmatrix} = \begin{bmatrix} U \\ V \\ W \end{bmatrix} = \begin{bmatrix} k_1l_2 - l_1k_2, l_1h_2 - h_1l_2, h_1k_2 - k_1h_2 \end{bmatrix}
\]
Figure 4.7. Twelve low-index zone-axis diffraction patterns for face-centered cubic (112) + (002).

(111) = + [111] -

(110) = + [110] -

(020) = + [110] -

(111) = + [100] -

Appendix 4
IV. Kikuchi Lines

We see Kikuchi lines because:
1. The specimen is thick enough (~50 nm or above, 200 kV)
2. Electrons are scattered off Bragg angles in all directions (because of inelastic scattering)
3. These electrons are then Bragg diffracted by lattice planes

Some facts:
1. The electron energy loss is small (most less a few tens of eV) and electrons have similar wavelength as the incident beam
2. Most electrons travels in directions close to the incident beam (forward direction)
3. Kikuchi described this in 1928 before invention of TEM

SrTiO₃, 200kV
Construct Kikuchi Maps

- Select a zone axis (e.g. [001], cubic)
- Draw the zone axis diffraction pattern
- For each allowed reflection, draw a line perpendicular to g and at half of g
- Select a second zone axis (e.g. [011] cubic) (use the stereo-projection)
- Draw the Kikuchi lines using the same procedure
- Orient the second map to line up the common pair of Kikuchi lines
The effect of sample rotation

Incident beam || optical axis

Bragg cone fixed to crystal

Kikuchi lines fixed to crystal

Diffracted beam fixed to incident beam
Why CBED?

- Crystal diffraction
- Accurate thickness, unit cell and orientation measurement
- Small probe
- Used in STEM
Resolution

Real Space: 1) Probe size
2) Probe propagation

Reciprocal Space: Beam divergence

Channeling

De-channeling
Probe Size

Not important

Important

Thickness changes across CBED disk

Small probe -> ~constant thickness
Excitation Error Changes Across CBED Disk

\[ S_g \propto g\theta \]
Dynamic Diffraction: Two Beam Case

\[ w = S_g \xi \]

\[ t / \xi_G = 0.1 \]
\[ t / \xi_G = 0.25 \]
\[ t / \xi_G = 0.5 \]
\[ t / \xi_G = 0.75 \]
\[ t / \xi_G = 1 \]
\[ t / \xi_G = 1.5 \]
\[ t / \xi_G = 2 \]
\[ t / \xi_G = 2.5 \]
\[ t / \xi_G = 3 \]
Electron Nanobeam Diffraction

Field Emission Electron Source

10 micron aperture

Magnetic Lens

Condenser Lens III

Upper Objective

Lower Objective

Back Focal Plane

Semi-angle ~ 0.05 mrad

C2 Aperture 10µm

40 nm

J~10^5 e/nm^2/s

Exposure time ~10 s
Where is the strain? Epitaxial Relationship

\[ \frac{a_{Si}}{a_{Ag}} \approx \frac{4}{3} \]

Ag(001)||Si(001)
Ag[220]||Si[220]
Or
Ag[2-20]||Si[2-20]

At RT: \( \Delta a/a = -0.3\% \)
At 450°C: \( \Delta a/a = -1\% \)

\(~13\) degree off [001]
Determination of Individual CNT Structure

Ag/H-Si(111) 2 ML

As deposited

Ag(2-20)
Si(2-20)
Si(4-2-2)/3

Azimuthal Angle

Coverage (monolayer)

(111)
Nano-crystalline or amorphous diffraction

AgCu film
As deposited

AgCu film
Annealed at 260 ºC
References


