Transmission Electron Microscopy

- Instrument
- Imaging & Diffraction
- Electron Energy Loss Spectroscopy (EELS)

Transmission Electron Microscopy
David B. Williams and C. Barry Carter
TEM: the power and limitations.

When do we see anything? – Contrast

How do we see fine detail? – Resolution

How can we see better? – The TEM

What is meant by ‘High-Resolution’?

What things are there to be ‘seen’? – Imaging

Electron Diffraction in the TEM.
TEM: High-resolution phase contrast image

High-resolution micrograph from a Lu-Mg doped Si$_3$N$_4$ sample showing the presence of an Intergranular Glassy Film (IGF).
Atomic-resolution scanning transmission electron microscope (STEM) images of an intergranular glassy film (IGF) in La-doped $\beta$-Si$_3$N$_4$:

a) High-angle annular dark-field (HAADF-STEM)

b) Bright-field (BF-STEM)


“Observation of rare-earth segregation in silicon nitride ceramics at subnanometre dimensions”

HAADF-STEM images of the interface between the IGF and the prismatic surface of an $\beta$-Si$_3$N$_4$ grain. The $\beta$-Si$_3$N$_4$ lattice structure is superimposed on the images. 

**a)** La atoms are observed as the bright spots (denoted by red arrows) at the edge of the IGF. The positions of La atoms are shifted from that of Si atoms based on the extension of the $\beta$-Si$_3$N$_4$ lattice structure; these expected positions are shown by open green circles. 

**b)** reconstructed image of a, showing the La segregation sites more clearly. The predicted La segregation sites obtained by the first-principles calculations are shown by the open white circles.

“Observation of rare-earth segregation in silicon nitride ceramics at subnanometre dimensions”
Diffuse Dark Field image from a grain boundary in SrTiO$_3$
**Advantages and disadvantages of TEM**

**Disadvantages**
- Low sampling volume and rather slow process of obtaining information.
- High capital and running cost.
- Special training required for the operation of the equipment.
- Difficult sample preparation. Possibility of electron beam damage.
- Samples which are not stable in vacuum are difficult to study.
- Magnetic samples require special care.
- Non-conducting samples require gold or carbon coating.
- Difficulty in the interpretation of images. In usual mode of operation information is integrated along the beam direction.

**Advantages**
- Real (Image) and reciprocal space (diffraction pattern) information can be obtained from same region of sample.
- Chemical information via EDX and EELS possible (with additional attachments). Energy filtered images possible via EELS filter.
- High resolution imaging possible (via HRLFI & HAADF in STEM).
- Possible to obtain amplitude and phase contrast images. Many different kinds of phase contrast images can be obtained.
Lower resolution/large area techniques should be first performed to get a ‘broad picture’ about the sample.
- This includes XRD and SEM. We can even start with optical microscopy.
- Phase related information should be obtained via XRD.
- Chemical information via EDX in SEM should also be obtained (any chemical inhomogeneity should be noted).

On ‘usual’ samples conventional TEM should be performed before trying out HRTEM.

<table>
<thead>
<tr>
<th></th>
<th>Eye</th>
<th>Optical</th>
<th>SEM</th>
<th>TEM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Magnification</td>
<td>?</td>
<td>1000×</td>
<td>100 kX</td>
<td>500 kX</td>
</tr>
<tr>
<td>Resolution (max)</td>
<td>0.1 mm</td>
<td>0.2 µm</td>
<td>~0.5 nm</td>
<td>1 Å</td>
</tr>
<tr>
<td>Chemical information</td>
<td>-</td>
<td>-</td>
<td>Via EDX</td>
<td>EDX, EELS</td>
</tr>
</tbody>
</table>
- The wavelength of the electrons in a 10 kV SEM is $12.3 \times 10^{-12}$ m ($12.3$ pm).
- In a 200 kV TEM the wavelength is $2.5$ pm.
- Wavelength of X-rays usually used in XRD is in the order of $100$ pm (Cu kα: $\lambda=154$ pm).

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Operating Voltage</th>
<th>$\lambda_{\text{X-ray}}$ or $\lambda_{\text{Electron}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>X-rays</td>
<td>-</td>
<td>$1.54$ Å</td>
</tr>
<tr>
<td>SEM</td>
<td>10 kV</td>
<td>$0.12$ Å</td>
</tr>
<tr>
<td>TEM</td>
<td>200 kV</td>
<td>$0.02$ Å</td>
</tr>
</tbody>
</table>
The TEM sample & the projected image

- The sample is a thin 3mm disc. The central portion of the disc is thinned down further to make it electron transparent (< 1000 Å in thickness). The process of sample preparation usually leaves a hole(s) in the ‘middle’ with electron transparent region next it.
- The thin regions in the sample can bend.
- The standard image seen on the screen or captured in the camera is a *projected image integrated* through the thickness.
In a TEM we can switch from an Image (Real Space) to Diffraction (Reciprocal Space) by the switch of a button.

We can get spectroscopic information and use it for forming images or diffraction patterns.

<table>
<thead>
<tr>
<th>Imaging</th>
<th>Diffraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Real Space $\rightarrow$ Crystal</td>
<td>Reciprocal Space $\rightarrow$ Reciprocal crystal</td>
</tr>
<tr>
<td>$r \rightarrow$ ‘Near field’ information (Local)</td>
<td>$1/r \rightarrow$ ‘far field’ information (Global)</td>
</tr>
<tr>
<td>Defects (Break in symmetry)</td>
<td>‘Periodicity’ (Symmetry)</td>
</tr>
</tbody>
</table>
2D image of 3D specimen and hence ‘information is lost’.

‘Usual’ Bright Field Image (BFI) is diffraction contrast dominated. Absorption contrast is usually small.

Contrast depends on many aspects of the apparatus (brightness of the electron source, apertures, lenses, etc.).

Phase contrast related to specimen and not to topography (or “depth”).

Hence, any feature observed has to be interpreted via an understanding of the mechanism behind the contrast seem (if possible via corroboration with image simulation).
We see something if:
(i) Light (visible part of the spectrum) enter our eyes,
(ii) the light has sufficient intensity,
(iii) There is sufficient contrast in the image.

Contrast is a *dimensionless number* as defined below. It is to be noted that contrast is *not* the difference in intensity between the light ($I_1$) and dark ($I_2$) regions, but the difference divided by the (say) higher of the two intensities ($I_2$).

A contrast value of 5-10% can be picked discerned by our eyes.

We have strong or weak contrast* (but not bright or dark contrast).

\[ C = \frac{I_1 - I_2}{I_2} = \frac{\Delta I}{I_2} \]

> 5-10 % ⇒ we see

We have divided by $I_2$ in the equation.
*But not bright or dark contrast (these terms refer to intensity and not to contrast)*
Contrast

Amplitude Contrast
- Fringes
  - Thickness Fringes
  - Bend Contours
  - Fresnel Fringes
  - Moiré Patterns
  - Lattice Fringes

Phase contrast
- Fringes
  - Bright Field & Dark Field Images

In-coherent elastic scattering
- Absorption contrast
  - Mass-thickness contrast
    - small effect in a thin specimen

Coherent elastic scattering
- Diffraction contrast
  - Beats is the time analogue of Moiré patterns
In most of the situations both type of contrasts contribute to an image—although one will tend to dominate.
In most of the situations both type of contrasts contribute to an image—although one will tend to dominate.
How do we see fine details? - *resolution*

- Resolution of human eyes \( \sim (0.1 - 0.2) \text{ mm} \).
- Highest useful magnification is governed by the resolution.
- Raleigh criterion is used for the definition of resolution.

#### Optical Microscope

\[
\delta = \frac{0.61\lambda}{\mu \sin \beta}
\]

\[
\delta \sim \frac{\lambda}{2}
\]

*Green light*

\( \lambda = 550 \text{ nm} \)

\( \delta \sim 300 \text{ nm} \)

\( \rightarrow 1000 \text{ atomic diameters} \)

#### TEM

\( \lambda \sim \frac{1.22}{E^{1/2}} \)

100 keV electrons \( \rightarrow \lambda = 4 \text{ pm} \)

#### Aspects determining the resolution

- Diffraction limit of the imaging system
- Aberration of the lenses in the imaging system
- Wavelength of the radiation

\( \delta \rightarrow \text{Smallest distance that can be resolved} \)

\( \lambda \rightarrow \text{Wavelength of radiation} \)

\( \beta \rightarrow \text{Semi-angle of collection} \)

\( \mu \rightarrow \text{Refractive index of viewing medium} \)
Magnification without detail in image does not help!
Rayleigh criterion

- Not a fundamental rule but a practical definition.
- Figure of merit in terms of the eyes ability to distinguish separate images of two self-luminous incoherent point sources.
- A single point source will not be imaged as a point even if aberrations are absent.
- Any physical limit in the path of the rays (*outer boundary of the lens / aperture*) will lead to diffraction effects.
- Diffraction → point is imaged as a disc (*Airy disc*).

Fully resolved

Unresolved

Just resolved according to Rayleigh criterion

\[ \frac{P_2}{P_1} = 0.61 \]

20%
What is meant by High-Resolution?

In general

- **High Spatial Resolution**
  - microscope’s ability to process high spatial frequencies

- **High Energy Resolution**
  - spectrometer’s ability to resolve two closely positioned energy peaks

- **High Chemical Resolution**
  - ability to detect small quantities of a given chemical species

Additionally, one would like to differentiate two structures adopted by a material of a given composition

  - This is essentially related to the above-mentioned points
Phase contrast (lattice fringe) image but not ‘high’-resolution

Phase contrast image of as-cast Mg$_{37}$Zn$_{38}$Y$_{25}$ alloy showing a 18 R modulated phase
*Picture taken in a JEOL 2000FX microscope with W filament. Resolution ~ 2.8 Å*

High-resolution but not a lattice fringe image

Fourier Filtered Fresnel Contrast Image from Si$_3$N$_4$ – grain boundary having an IGF
*Picture taken in a JEOL JEM 4000 EX, with LaB$_6$ filament. Resolution ~ 1.8 Å*
How can we see better? – The TEM
## Characteristics of the three principal sources operating at 100kV

<table>
<thead>
<tr>
<th></th>
<th>Units</th>
<th>Tungsten</th>
<th>LaB$_6$</th>
<th>Field emission</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Work function, $\Phi$</strong></td>
<td>eV</td>
<td>4.5</td>
<td>2.4</td>
<td>4.5</td>
</tr>
<tr>
<td><strong>Richardson’s Constant</strong></td>
<td>A/m$^2$K$^2$</td>
<td>$6 \times 10^5$</td>
<td>$4 \times 10^5$</td>
<td></td>
</tr>
<tr>
<td><strong>Operating temperature</strong></td>
<td>K</td>
<td>2700</td>
<td>1700</td>
<td>300</td>
</tr>
<tr>
<td><strong>Current density</strong></td>
<td>A/m$^2$</td>
<td>$5 \times 10^4$</td>
<td>$10^6$</td>
<td>$10^{10}$</td>
</tr>
<tr>
<td><strong>Crossover size</strong></td>
<td>$\mu$m</td>
<td>50</td>
<td>10</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td><strong>Brightness</strong></td>
<td>A/m$^2$/Sr</td>
<td>$10^9$</td>
<td>$5 \times 10^{10}$</td>
<td>$10^{13}$</td>
</tr>
<tr>
<td><strong>Energy spread</strong></td>
<td>eV</td>
<td>3</td>
<td>1.5</td>
<td>0.3</td>
</tr>
<tr>
<td><strong>Emission current stability</strong></td>
<td>%hr</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>5</td>
</tr>
<tr>
<td><strong>Vacuum</strong></td>
<td>Pa</td>
<td>$10^{-2}$</td>
<td>$10^{-4}$</td>
<td>$10^{-8}$</td>
</tr>
<tr>
<td><strong>Lifetime</strong></td>
<td>hr</td>
<td>100</td>
<td>500</td>
<td>&gt;1000</td>
</tr>
</tbody>
</table>
Elastic scattering is usually **coherent** if specimen is thin & crystalline.

In TEM elastic scattering is predominant in $(1 - 10)^\circ$ in the forward direction.

Elastically scattered electrons at large angles $(> 10^\circ)$ can be **incoherent**.

Inelastic scattering $\rightarrow$ almost always incoherent.

Most of scattered electrons $< \pm 5^\circ$ ($<$ about $3^\circ$ they are **coherent**).

**Specimen**

- **Thin** $\rightarrow$ Coherent forward scattering
- ** Thick** $\rightarrow$ Incoherent back scattering
Resolution

Electron Source

Imaging System

Lenses

Resolution

$\lambda = f(\text{Voltage}), \text{High coherency} \rightarrow \text{FEG}$

Lenses

- Lens defects affects the resolution but give us increased ‘Depth of Focus’ & ‘Depth of Field’
- Can suffer from 10 kinds of aberration

Spherical Aberration

Chromatic Aberration

Astigmatism

Apertures

And Slits

Control the quality of:
- Images
- Diffraction patterns
- Analytical signals

Control:
- Beam current and Convergence
- Cutoff paraxial rays which suffer aberration
- Image contrast
- Type of image (BF / DF)
- Select areas from which we want diffraction patterns (SAD)
- Chose an energy range in from spectroscope
“The current best electromagnetic lens is like using a coke bottle for a magnifying lens”

“If the lens of our eyes was as good as the best electromagnetic lens available then we would be legally blind”
Bright Field and Dark Field Images

(a) Bright Field Image

Inset

Dark Field Image

B2 phase particles

(b) Diffraction pattern with indices

\[ [001] \]

Indices:
- 020
- 110
- 200
- 110
- 100
- 010
- 200
- 110
- 020
Aperture Position Bright field & Dark Field Images

Bright Field Image

“Transmitted beam”

Diffracted beams

Objective Aperture

Dark Field Image

“Transmitted beam”

Diffracted beams

Objective Aperture

Centered Dark Field Image

“Transmitted beam”

Diffracted beams

Use a tilted beam

Objective Aperture
Bright Field and Dark Field images are complementary ONLY in TWO BEAM condition.

Note: the positions of the aperture for BFI and DFI have been overlaid in the same figure (actually only one of them will be used in a single imaging condition).

“Absorption” Contrast

Only Mass thickness contrast

Shadowing using Au (or Au-Pd)

Contrast inverted print of shadowed image

Thickness Fringes and Bend Contours
Stacking Fault
Dislocations
Coherent precipitates
Thickness fringes

DF Image from a Grain Boundary

Thickness fringes

Edge

Bottom

Bright Field Image

Dark Fringes

Edge

Intensity

Top 1 0 1 0

Bottom

Direct Beam Diffracted Beam
Bend Contours

Zone axis patterns: two bend contours from each diffracting plane ($\theta_B, -\theta_B$)
Stacking Fault

\[ \vec{g} \cdot \vec{b} \neq 0 \]
Strain Fields
Dislocations

Dislocation tangles in an Fe-35% Ni-20%Cr alloy

Dislocation is invisible if $\mathbf{b} \perp \mathbf{g} \Rightarrow \mathbf{g} \cdot \mathbf{b} = 0$ (using effective invisibility criterion)

$\Rightarrow$ just because a dislocation is not visible it does not mean it is not there

“it could just be out of contrast”
Dislocations in a heterojunction

No visible Grain Boundary

Fourier filtered image

Dislocation structures at the Grain boundary

~8° TILT BOUNDARY IN SrTiO$_3$ POLYCRYSTAL

2.761 Å
When precipitates are small the image may be dominated by strain field contrast.

Coherent precipitates in Cu-Co

Strain fields are being imaged!!

Coherent precipitates in Cu-Co

Line of no contrast if $g \cdot R = 0$.

$g \cdot R = 0 \Rightarrow NO$ contrast

$R$ shows radial symmetry

$\Rightarrow$ the horizontal planes are being imaged

Planes undistorted by the strain field

MOIRÉ FRINGES

Translational Moiré

MISFIT SETS

Rotational Moiré

IDENTICAL SETS
ROTATED

Mixed Moiré

MISFIT SETS
ROTATED

Fringes ⊥ to the average direction of the initial lines

Alignment to reference sets not clear

Dislocation networks in graphite

Interpretation of Moiré fringes: should be extremely cautious!

Dislocated lattice + Perfect lattice

Dislocated lattice + Perfect lattice (rotated)

Dislocated lattice + Perfect lattice (with different spacing)
Low angle grain boundary in NiO: GND (structural dislocations): “focal series”

Fresnel Contrast Images (FCI)

Defocus value of +1.2 μm

Defocus value of −1.2 μm

FCI from Lu-Mg doped Si₃N₄

Electron Diffraction
Information from Diffraction Patterns

- Atomic Structure (*Crystalline, Amorphous...*)
- Atomic Spacings
- Shape (*Specimen, Structures within...*)
- Internal stresses

- \( \text{Cu (111)} \rightarrow d = 2.1 \, \text{Å} \)
- \( 120 \, \text{kV electrons} \rightarrow \lambda = 0.0335 \, \text{Å} \)

Bragg’s Equation

\[ \theta = 0.46 \, ^\circ \ (7.97 \, \text{mrad}) \]

\[ 10 \, \text{mrad} = 0.573 \, ^\circ \]

*Angles in a TEM \sim 1 \, ^\circ*
Diffraction

Selected Area Diffraction  
*SAD*

Convergent Beam Electron Diffraction  
*CBED*

*Place an aperture in the first image plane below the objective lens*
Inserting an aperture in the image plane creates a virtual aperture in the object plane.

- **Selected Area Diffraction (SAD)**

- Error in actual area selected due to spherical aberration
- At 50× a 50μm aperture will select 1 μm on specimen
  - Smallest aperture ~10μm
  - Smallest area selected ~ 0.5μm

Parallel illumination
Example of Diffraction Patterns in a TEM

- Amorphous C
- Al single crystal
- Polycrystalline Au
- CBED from Si (single crystal)
Thin specimens (& Excitation Error)

In a TEM exact Bragg’s condition relaxed due to

- Convergence of beam
  - Illumination is not parallel
- Thin Specimen
  - Has to be electron transparent (~500 Å)
  - For HREM specimen has to be very thin

Many of the diffraction effects can be understood by these two factors.

Real Space

Reciprocal Space

Anything constrained in Real Space expands in Reciprocal Space & vice-versa!
Ewald sphere → X-rays

\[ \lambda (\text{Cu } K_\alpha) = 1.54 \text{ Å}, \quad \frac{1}{\lambda} = 0.65 \text{ Å}^{-1}, \quad a_{\text{Cu}} = 3.61 \text{ Å}, \quad \frac{1}{a_{\text{Cu}}} = 0.28 \text{ Å}^{-1} \]
Ewald sphere construction

Reciprocal Lattice

Reciprocal lattice of a square ‘crystal’ with lattice parameter of 0.4 nm (4 Å) (1/a = 0.25 Å$^{-1}$): Ewald spheres are drawn to scale.

Sample thickness (t) = 100 Å (1/t = 0.01 Å$^{-1}$)

In TEM the Ewald sphere is very flat!!

$V = 200$ keV, $\lambda = 0.002$ nm (0.02 Å), $1/\lambda = 50$ Å$^{-1}$

Sample thickness (t) = 100 Å (1/t = 0.01 Å$^{-1}$)

Reciprocal lattice of a square ‘crystal’ with lattice parameter of 0.4 nm (4 Å) (1/a = 0.25 Å$^{-1}$): Ewald spheres are drawn to scale.
20× Lens

30× Lenses

Rel rod
Perspective representation of the spatial relation between the Ewald sphere and a plane in the reciprocal lattice:

(a) Plane is tangential to the sphere
(b), (c), (d) Reciprocal lattice is tilted at various angles (implying that the crystal is tilted)

Laue circle (intersection of the Ewald sphere with the Reciprocal lattice plane)

Rel rods
<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value (Å)</th>
<th>Reciprocal dimension (Å⁻¹)</th>
<th>Approximate Relative Scale in reciprocal space</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\lambda_{200\text{ kV}}$</td>
<td>0.02</td>
<td>Radius of Ewald Sphere: $1/\lambda = 50$</td>
<td>5000</td>
</tr>
<tr>
<td>Crystal Lattice Parameter</td>
<td>4</td>
<td>0.25</td>
<td>25</td>
</tr>
<tr>
<td>Thickness of specimen</td>
<td>100</td>
<td>0.01</td>
<td>1</td>
</tr>
</tbody>
</table>

- The lengthscales involved span more than 3 orders of magnitude.
- At the scale the Ewald sphere is visible the rel-rods are practically invisible (hence, we had to use a 30× lens).
- The relative scale between the lattice parameter and $\lambda$ is 200 → this implies that many rel-rods can be intersected by the Ewald sphere, even though the height of the rel-rod is small (‘1’ on that scale).
Shapes of Rel Rods

- Cube
- Tapering Rod
- Sphere
- Shells
- Disc
- Tapering Banded Rod
- Rod
- Disc & Rings
Tilting the beam
Tilting the specimen (*crystal(s)*)
GaAs/Al$_x$Ga$_{1-x}$As artificially created superlattice: 4 layers of GaAs alternates with 4 layers of Al$_x$Ga$_{1-x}$As

Ordered domains in Cu₃Au

Ion irradiated crystal → localized disorder of crystals due to cascades

Inclined ‘Antiphase Domain Boundaries’ → Dark

Locally disordered regions (due to irradiation) → Dark

Ordered regions → bright

Dark Field Image using the 110 superlattice reflection

For more details: ordered_structures.ppt
GaAs/Al$_x$Ga$_{1-x}$As quantum well structure

GaAs $\rightarrow$ Dark  
Al$_x$Ga$_{1-x}$As $\rightarrow$ bright (*Due to lighter Al $\rightarrow$ chemically sensitive reflections*)

Superlattice reflection

*Dark Field Image using the 002 superlattice reflection*
Artificially created superlattice of Si and Mo layers (~5nm)
Twin boundary in Fe doped SrTiO$_3$ bicrystals (artificially prepared)


GP Zones

GP zones in an Fe-2.9 at.% Mo alloy

Diffraction from an ordered array of dislocations

Due to dislocation arrays 2 arrays → only 1 is visible

‘s’ is large for one grain and near zero for another

Dislocation Arrays

Structured Grain Boundary

Twin Boundaries

Diffraction from two twin boundaries

Spacing of spots: 15 nm$^{-1}$
Low Angle Grain Boundary

FFT from the GB region

~7°
SAD Patterns from the boundary
Kikuchi patterns

- In ‘thick specimens’ incoherently scattered (not necessarily inelastically scattered) electrons travel in all directions.
- These can further be Bragg diffracted.
- These cones of diffracted intensity are called Kossel Cones.
- The intersection of the Kossel cones with the Ewald sphere gives rise to Kikuchi lines.
- These intersections are paraboloid and can be approximated to straight lines.
- Kikuchi lines come in pairs ($\pm G$, $\pm 2G$ ...) ➔ a bright line and a dark line.

- Energy of the scattered electrons is $\sim E_0$ ($\Rightarrow \lambda \sim \lambda_0$) for specimens which are not too thick.
- If the specimen is oriented such that the diffracting planes are parallel to the beam this simple explanation breaks down.
- In a SEM backscattered electrons (BSE) can give rise to Kikuchi lines.
  ➔ Electron Backscatter patterns (EBSP) → can be used to rapidly map the texture of polycrystalline materials (Orientation Imaging Map). EBSP is also called EBSD (electron back scattered diffraction).
Peaked in the Forward direction

Initially closer to optic axis and hence produces a brighter line.

http://math2.org/math/algebra/conics.htm
The Kossel cones behave as though they are rigidly fixed to the diffracting plane (hkl) and hence are fixed to the crystal.

A line drawn between the two Kikuchi lines is a trace of the plane (hkl) 
- thus they are crystallographic markers.

The position of the Kikuchi lines is very sensitive to small tilts.

When the crystal is tilted the Kikuchi lines will move (but the position and intensities of the diffraction spots change very little).

As the angle between the $+G$ and $-G$ line is $2\theta_B \rightarrow$ the distance between them in reciprocal space is $g$ (and not $2g$).
Reconstruction of the reciprocal space by tilting the specimen

- As we get only a 2D section in a single SAD the specimen is tilted at various angles to obtain various other sections → which can give a picture of the 3D ‘reciprocal crystal’
- Kikuchi lines help us in tilting the crystal from one zone axis to another
- SAD is not an accurate method for determining \( d_{hkl} \) or angles between zone axes
- Measurements of \( d \) made with \( s = 0 \) will be more accurate \( (s \text{ is made zero by tiling the specimen}) \)
The camera length ($L$) gives the magnification in reciprocal space.

To calibrate $L$ the diffraction pattern from a specimen with known d-spacing is used.

The digital readout from the microscope may not be accurate (need for calibration by user).

‘$L$’ typically 100s to 1000s of mm.

\[
\frac{R}{L} = \tan(2\theta) \sim 2\theta
\]

Bragg's Equation \(\frac{\lambda}{d} = 2\sin\theta \sim 2\theta\)

\[
\frac{R}{L} = \frac{\lambda}{d}
\]

\[
Rd = \lambda L
\]

\[
d = \frac{\lambda L}{R}
\]
Note that in BCC there are no forbidden reflections in the [111] pattern.
Forbidden reflections

\[ \frac{L}{M} = \frac{\sqrt{2}}{1} = 1.414 \quad B = [001] \]

\[ \frac{L}{M} = \frac{2}{\sqrt{3}} = 1.155 \quad B = [011] \]

\[ \frac{M}{N} = \frac{\sqrt{8}}{\sqrt{3}} = 1.633 \quad L = \frac{\sqrt{11}}{\sqrt{3}} = 1.915 \quad B = [\bar{1} \bar{1} \bar{2}] \]
HCP

foil plane (2110)

$\frac{N}{L} = 1.09 \quad \frac{M}{L} = 1.139 \quad B = [2110]$

foil plane (0110)

$\frac{N}{L} = 1.587 \quad \frac{M}{L} = 1.876 \quad B = [01\overline{1}0]$

foil plane (0001)

$\frac{N}{L} = 1.520 \quad \frac{M}{L} = 1.820 \quad B = [0\overline{1}2]$

$B = [0001]$
Amorphous Materials

- Ring pattern from glasses is ~ to polycrystals → but without speckle and broader

- Computer plot of diffraction intensity from an amorphous material
  - Note the Multiple Diffuse rings

Bright Field Image

Diffuse Dark Field Image

Amorphous Carbon
Advanced Techniques of TEM

- Convergent Beam Electron Diffraction
- Weak beam dark field imaging
- Diffuse dark field imaging
- Electron holography
- Fresnel contrast imaging
- Through focal series reconstruction (Fresnel and High resolution)
- Tilt series reconstruction
- Interfacial plasmon imaging
- ...

...
End