Transmission Electron Microscopy
11. Diffraction Patterns

EMA 6518
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Why Use Diffraction in The TEM

Figure 11.1. An experimentally observed diffraction pattern showing the central, intense, direct beam and an array of diffraction spots from different atomic planes. Such a pattern, with sharply focused spots, is best obtained by underfocusing the beam.

Questions:

• Is the specimen crystalline?
• If it is crystalline, what are the crystallographic characteristics of the specimen?
• Is the specimen monocrystalline? If not, what is the grain morphology, how large are the grains, what is the grain-size distribution, etc.?
• What is the orientation of the specimen or of individual grains with respect to the electron beam?
• Is more than one phase present in the specimen?

The TEM, Diffraction Cameras, and the TV

- 1930s, using diffraction cameras

- Electrons vs. XRD
  - Electrons have a much shorter wavelength than the X-rays
  - Electrons are scattered more strongly because they interact with both the nucleus and the electrons of the scattering atoms through Coulomb forces
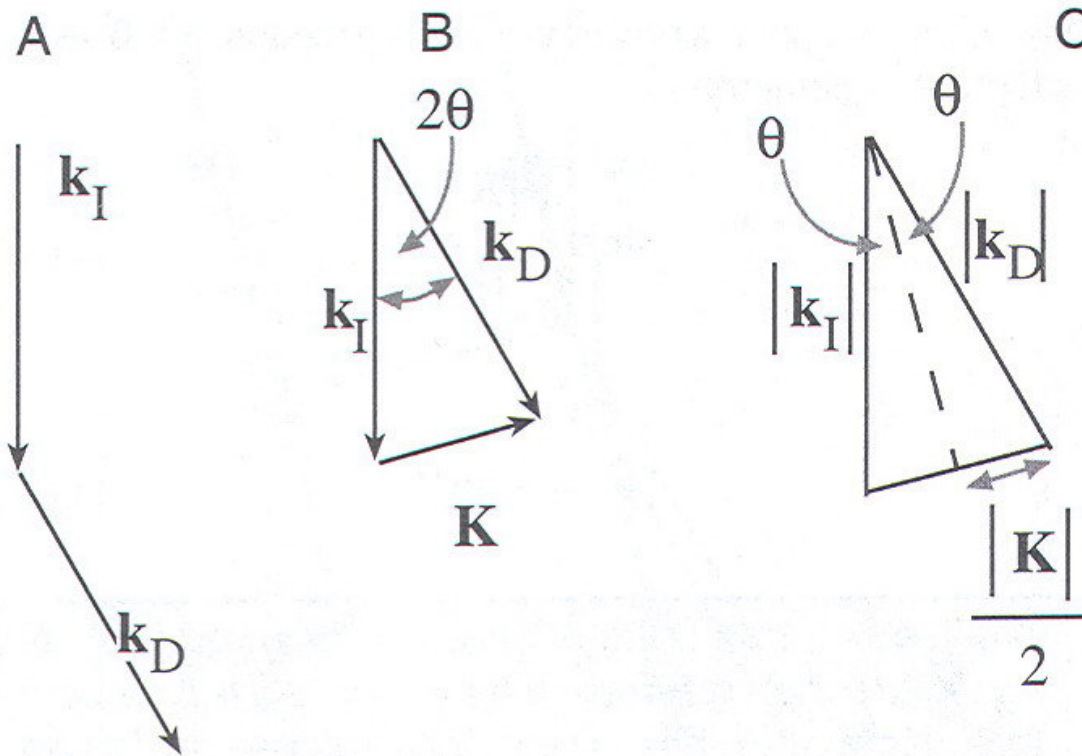 - Electron beams are easily directed because electrons are charged particles.

Scattering from a Plane of Atoms
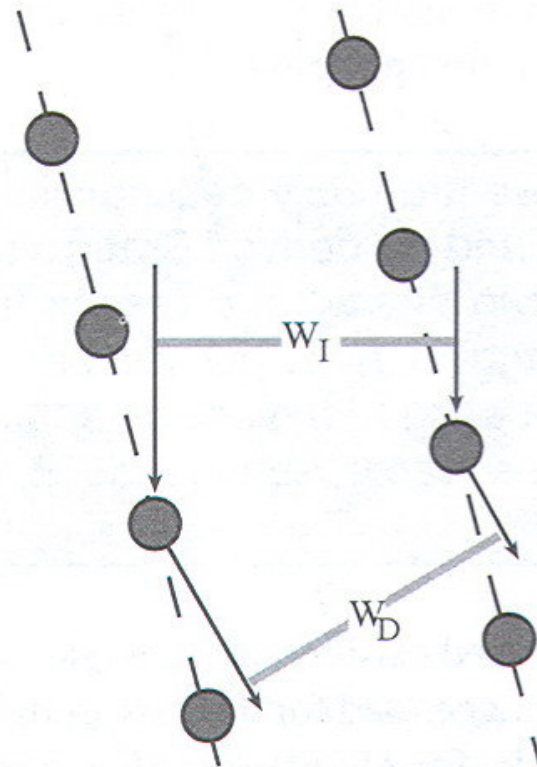
We only consider plane wavefronts, i.e., the wavefront is flat and **k** is normal to this wavefront.

Figure 11.2. Scattering from two planes of atoms. W_I and W_D are the incident and diffracted wavefronts, respectively.

Scattering from a Plane of Atoms

Figure 11.3. Definition of the scattering vectors: (a) the incident wavefront normal is k_I, the diffracted wave normal is k_D; (b) K is the difference vector ($= k_D - k_I$); (c) $\sin \theta$ is defined as $K/2k_I$.

$K = k_D - k_I$

Where k_D and k_I are the **k** vectors of the incident and diffracted waves, respectively. The vector **K** is the change in **k** due to diffraction.

Scattering from a Plane of Atoms

$$\left|k_I\right| = \left|k_D\right| = \frac{1}{\lambda} = \left|k\right|$$

Providing the energy of the electron is unchanged during diffraction, i.e., the scattering process is elastic.
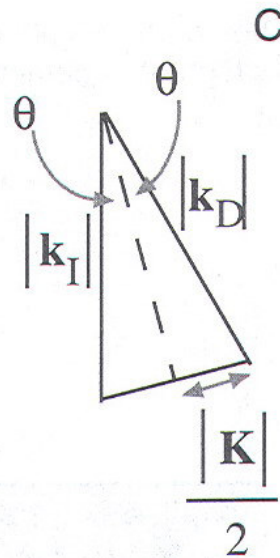
$$\sin\theta = \frac{\left|K\right|/2}{\left|k_I\right|}$$

or

$$\left|K\right| = \frac{2\sin\theta}{\lambda}$$

Scattering from a Plane of Atoms

$$|K| = \frac{2\sin\theta}{\lambda}$$

Unit: Å^{-1} , if λ is measured in Å.

•K and k$_I$ are referred to as *reciprocal lattice vectors.*

•This scattering process is taking place inside the crystal and the **k**-vectors are all appropriate to the electrons inside the crystal (rather than in the vacuum).

•Whenever you see the term (sinθ)/λ remember that it is just **K**/2 and is thus related to a change in wave vector.

Scattering from a Plane of Atoms

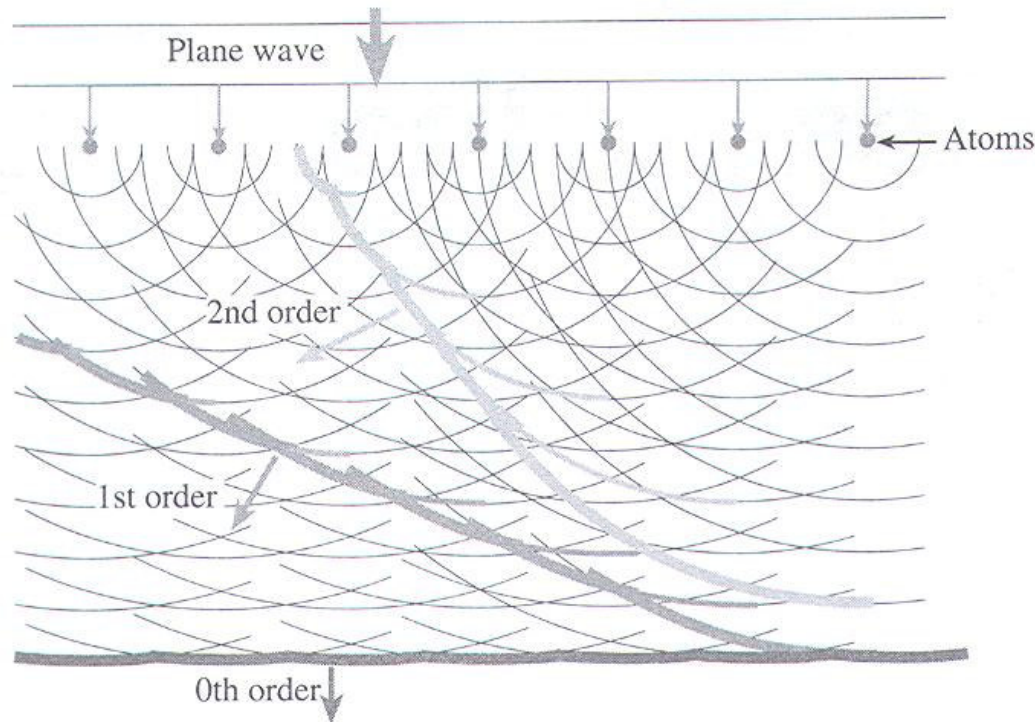Now we extend this argument to consider the interference between waves scattered from two points.
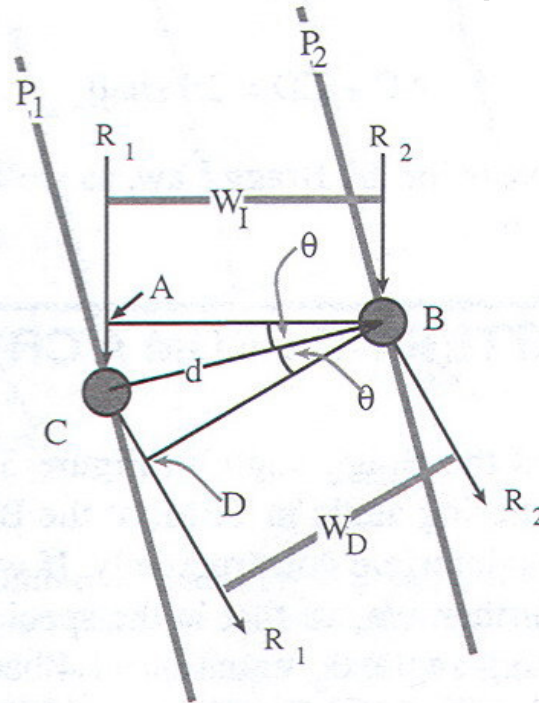
Figure 11.4. Two beams are scattered from two points, C and B, which lie on different planes, P_1 and P_2. The rays travel different distances, giving a path difference of AC + CD.
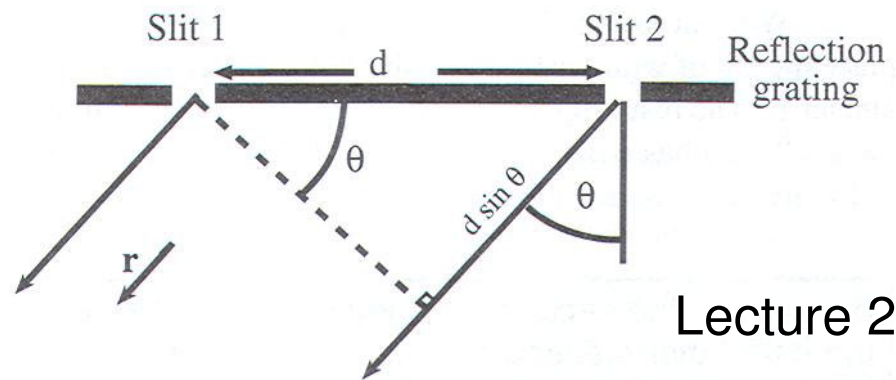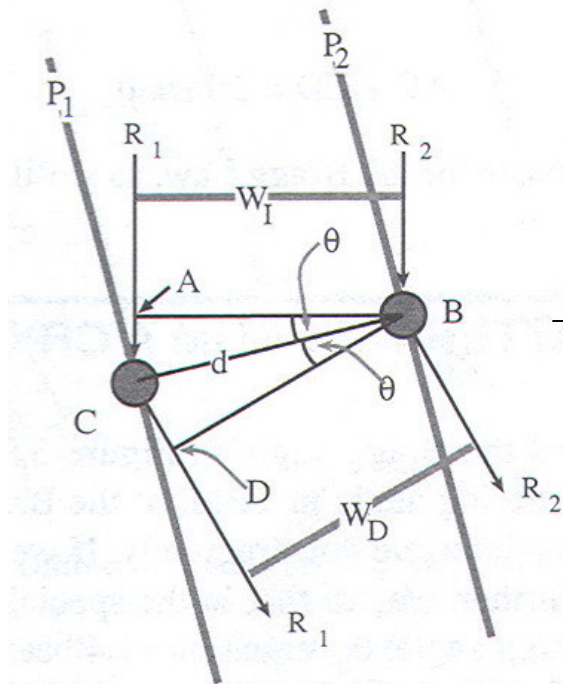
Scattering from a Plane of Atoms

Constructive and destructive interference (lecture 3)
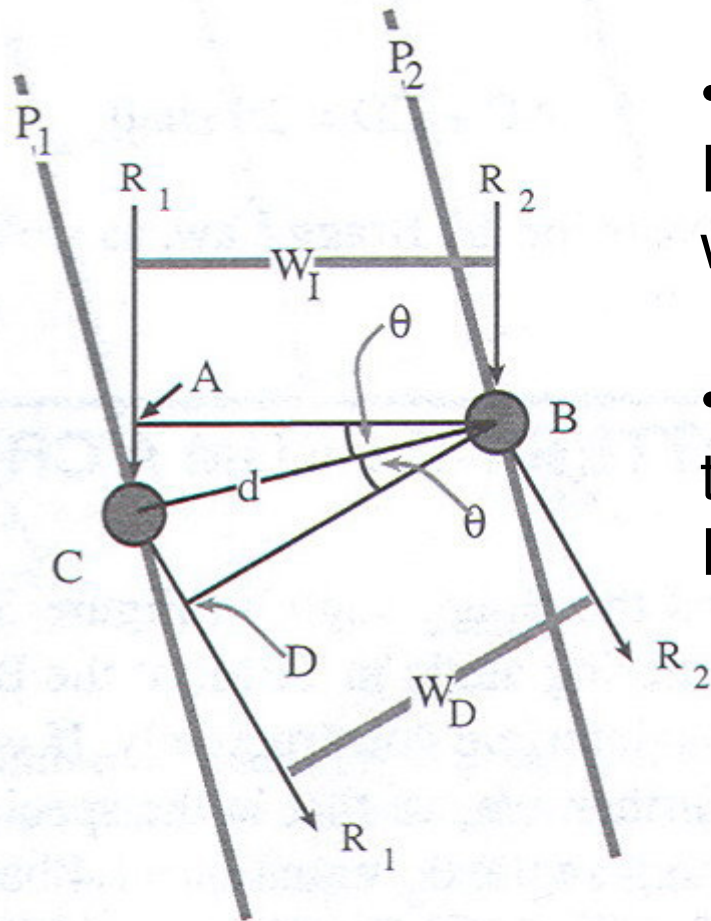
Scattering from a Plane of Atoms

Figure 2.5. An incident plane wave is scattered by two slits, distance d apart. The scattered waves are in phase when the path difference $d \sin \theta$ is $n\lambda$.

Lecture 2

cross section of the two slits used by Young to demonstrate the wave nature of light
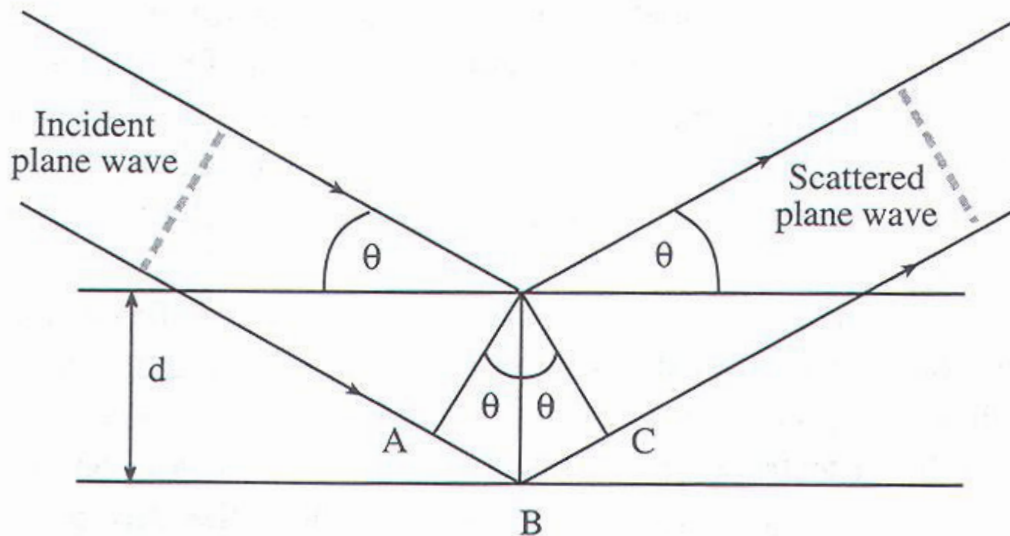
Scattering from a Plane of Atoms

- We can define two planes, P_1 and P_2, to be normal to the vector **CB**, which has length d.

- the distance traveled by ray R_1 is then larger than that traveled by ray R_2 by the path difference AC+CD.

$$AC+CD=2d\sin\theta$$

(the basis for the Bragg Law)

Scattering from a Crystal

Figure 3.9. The Bragg description of diffraction in terms of the reflection of a plane wave (wavelength λ) incident at an angle θ to atomic planes of spacing d. The path difference between reflected waves is $AB + BC$.

$$n\lambda = 2d\sin\theta$$

At the Bragg angle the electron waves interfere constructively.

Scattering from a Crystal

$$\left|K\right| = \frac{2\sin\theta}{\lambda}$$

When $\theta=\theta_B$

$$\left|K\right| = \frac{2\sin\theta_B}{\lambda} \longrightarrow$$

$$2\sin\theta_B = \lambda\left|K\right|$$

$$AC+CD=2d\sin\theta$$

When $\theta=\theta_B$

$$n\lambda=2d\sin\theta_B$$

n=1

$$2\sin\theta_B = \frac{\lambda}{d}$$

So when we are at the Bragg angle, the magnitude of the vector **K** has a special value, K_B

$$\left|K_B\right| = \frac{1}{d}$$

We define this vector, **K$_B$**, to be **g**

$$K_B=g$$

(**g**: diffraction vector)
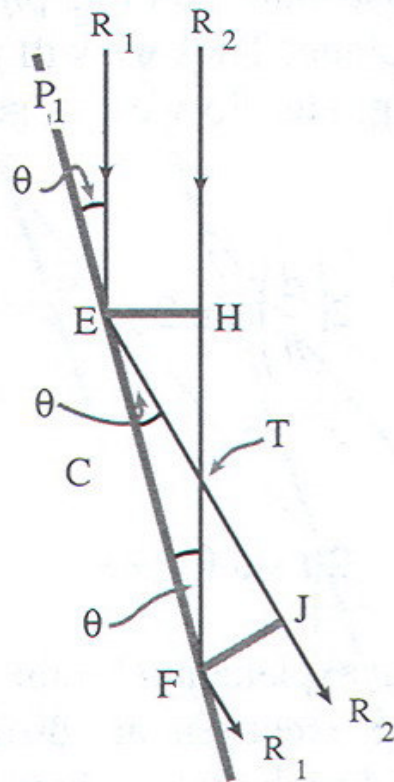
Scattering from a Crystal

- Bragg's Law give us a very useful physical picture of the diffraction process because the diffracting planes appear to behave as mirrors for the incident electron beam. Therefore, the diffracted beams, or the spots in the DP, are often called "reflections" and we sometimes refer to the vector **g** as the diffraction vector.

- Don't forget: we are really dealing with diffraction, not reflection, and we derived Bragg's Law by considering just two atoms. The reason that this derivation of Bragg's Law is not valid is that it really applies to scattering at a glancing angle where the beam exits the same surface as it enters, not transmission.

Scattering from a Crystal


Consider scattering from a single plane:

- Ray R_1 travels a distance EJ, ray R_2 travels a distance HF

- EJ=HF

- There is no path difference for scattering from atoms located anywhere on a particular plane

Figure 11.5. Two beams are scattered from two points, E and F, which lie on the same plane P_1. This simple diagram shows that the two beams travel the same distance since triangles EHF and FJE are congruent.
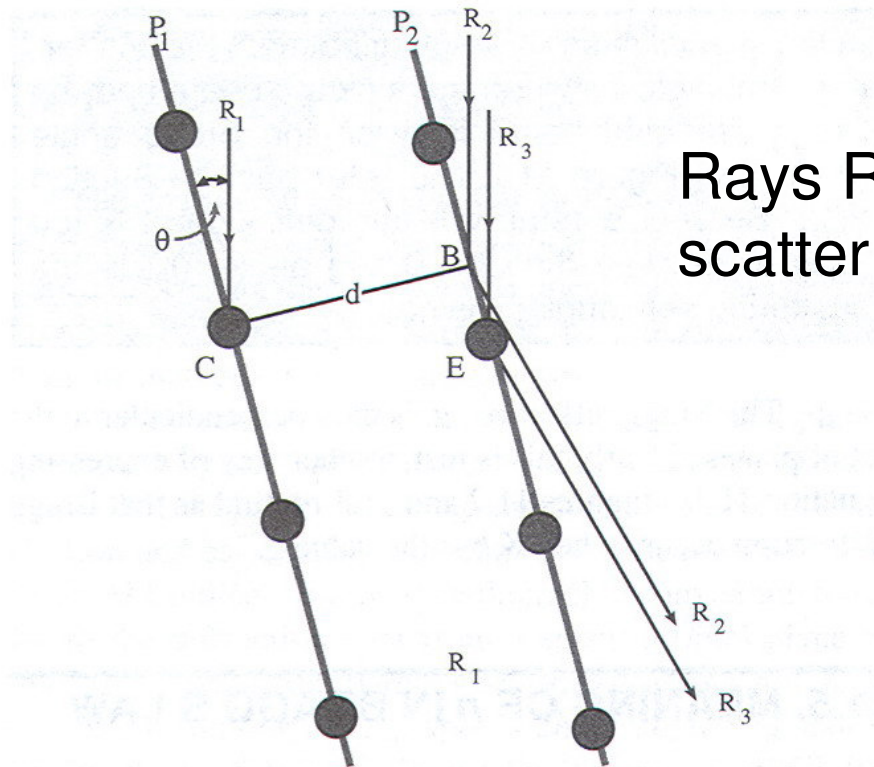
Scattering from a Crystal
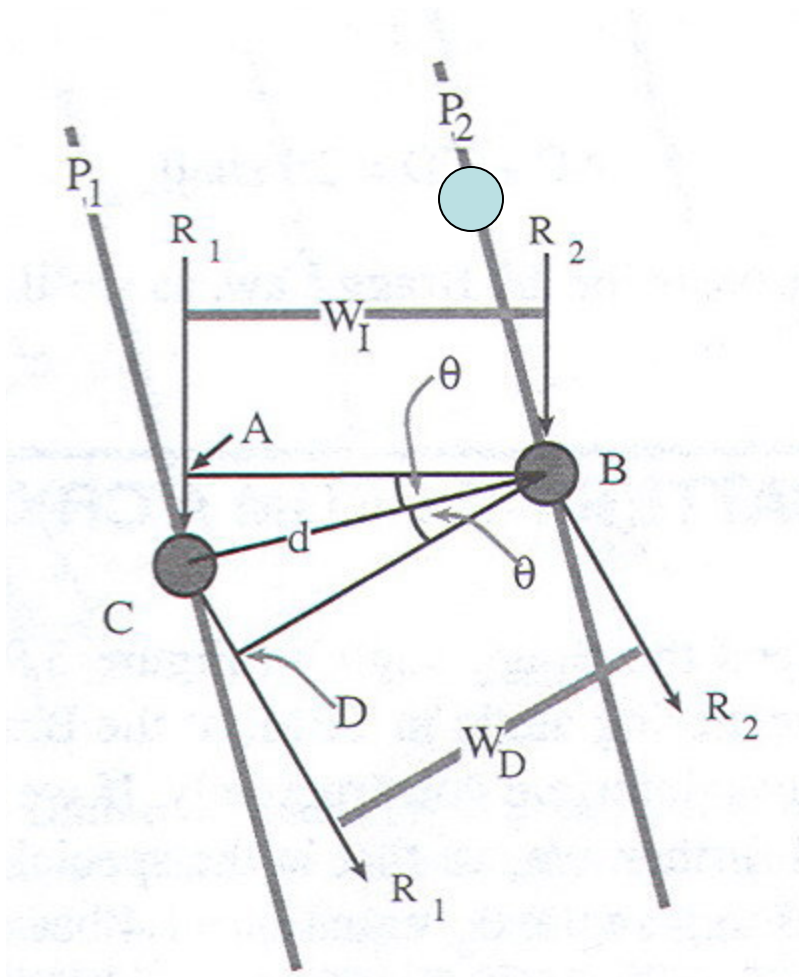
How is the "in-phase" nature changed if we move atom B but keep it on plane P_2?

It does not matter how the atoms (scattering centers) are distributed on these two planes; the scattering from any two points on planes P_1 and P_2 will produce the same path difference $2d\sin\theta$

Scattering from a Crystal

Rays R1, R2, and R3 all scatter in phase, if $\theta = \theta_B$

Figure 11.6. Scattering from three points on two planes. The path difference for scattering from points B and C is $2d \sin \theta$, so the path difference for scattering from points C and E is also $2d \sin \theta$. Hence scattering in the direction of the diffracted beam from all points shown will be in phase if $2d \sin \theta = n\lambda$.

Scattering from a Crystal
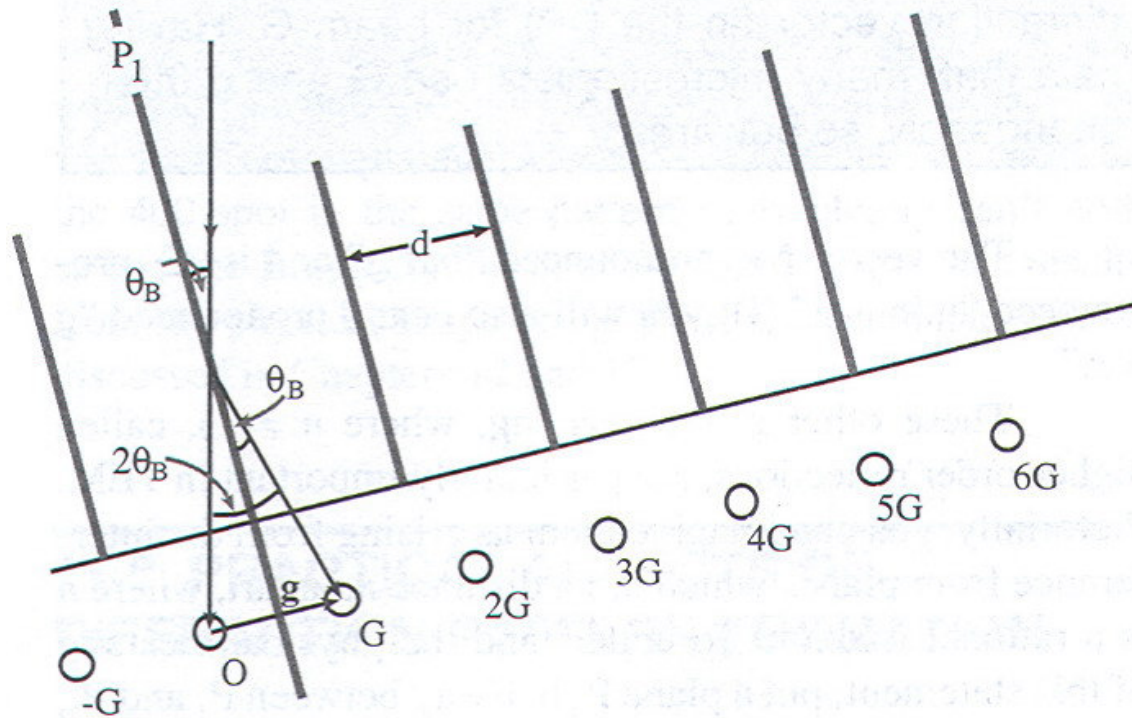
Figure 11.7. Diffraction from a set of planes a distance *d* apart. The planes have been oriented to be in the Bragg diffracting condition (θ_B is the incident angle). Note that the planes are not parallel to the incident beam. The resultant diffraction spots (reciprocal lattice points) are labeled G, 2G, etc. The vector **g** from the origin (O) to the first diffraction spot G is normal to the diffracting plane.

A series of reflections which are periodically spaced along a line, these are known as a *systematic row of reflections*, O, G, 2G, 3G, etc., with corresponding diffraction vectors, 0, **g**, 2**g**, 3**g**, etc.

Meaning of n in Bragg's Law

- Notation: when discussing beams in diffraction patterns, the letter O will refer to the "direct" beam which is present even when there is no specimen, the letter G (not bold- it's not a vector) will refer to any single diffracted beam; the number **0** will refer to the diffraction vector for beam O (it is a vector of zero length), and the letter **g** (always bold to remind us that it is a vector) will denote the diffraction vector (in the DP) for beam G. Having said that, many microscopists use G and **g** interchangeably, so beware.

Meaning of n in Bragg's Law

Incident Beam 1   Incident Beam 3   Incident Beam 2

P_1 P_3 P_2
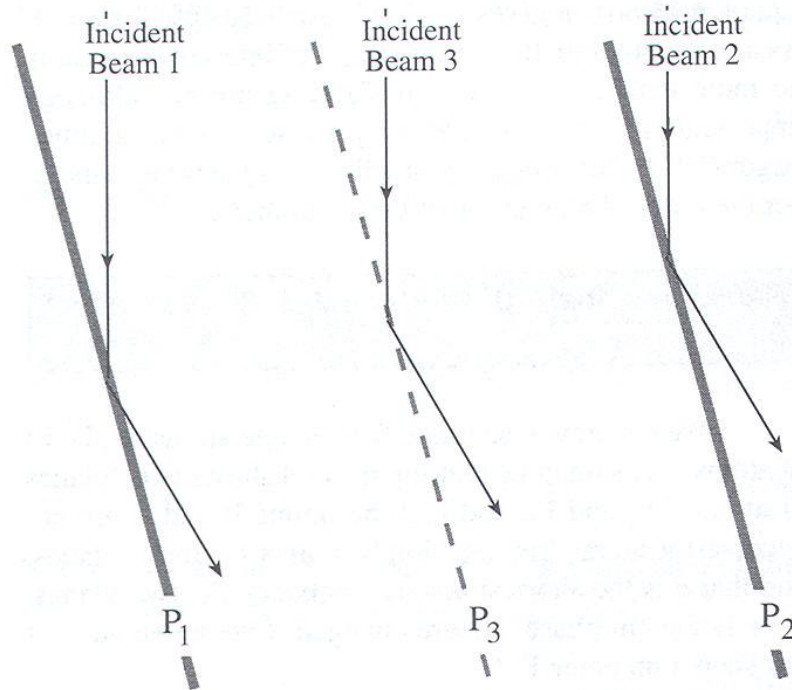
Figure 11.8. Scattering from three planes with plane P_3 positioned exactly halfway between planes P_1 and P_2.

- The other reflections (n**g**, where $n \neq 1$), called higher-order reflections, are particularly important in TEM. You can image them as arising from the interference from planes which are a distance *nd* apart, where n is a rational fraction.

$$2 \left(\frac{d}{2} \right) \sin \theta = \lambda \longrightarrow |g_2| = \frac{2}{d} \longrightarrow |g_2| = 2|g|$$

Meaning of n in Bragg's Law

- $g_2 = 2g$ and similarly $g_3 = 3g$
- We can generalize equation:

$$2(\frac{d}{n})\sin\theta = \lambda \qquad \text{or} \qquad 2d\sin\theta = n\lambda$$

• Electrons are diffracting from a set of planes of spacing *d* such that we have both constructive and destructive interference.

• we can consider *n* in equation as indicating that electrons are diffracting from a set of planes with spacing *d/n* rather than *d.*

A Pictorial introduction to Dynamical Effects
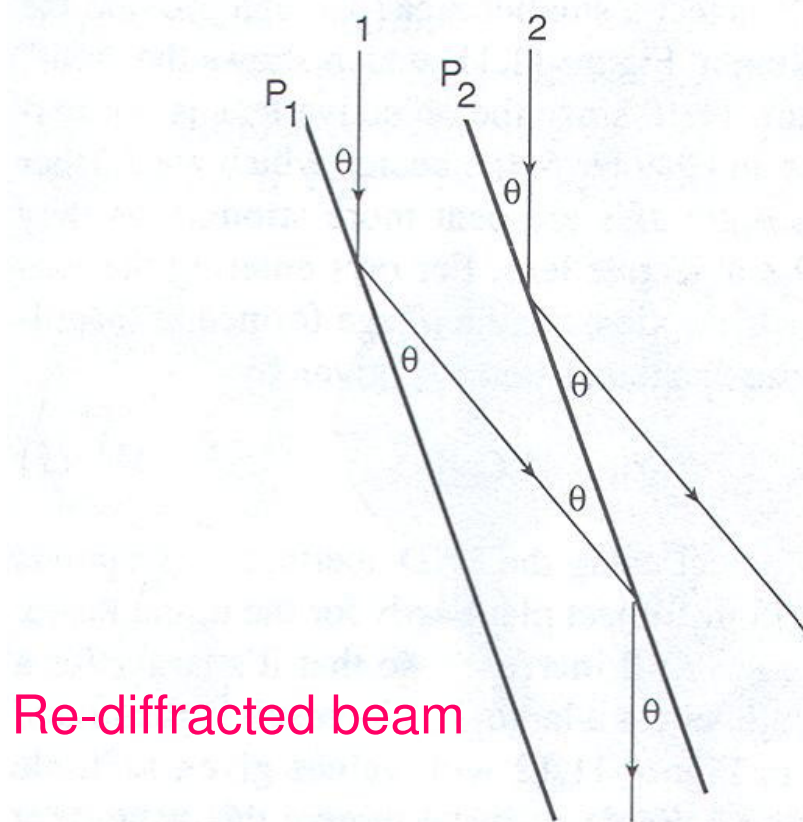
Re-diffracted beam

Figure 11.9. The beam can be scattered more than once. Any beam which is oriented so as to be Bragg-diffracted once is automatically in the ideal orientation to be rediffracted. This gives rise to the phenomenon of dynamical scattering.

• In TEM most practical imaging situations involve dynamical scattering.

• The reason it is very important in electron diffraction is that the electron beam interacts so strongly with the atoms in the crystal.

•The likelihood of this process occurring will increase as the thickness of the specimen increases.

Use of Indices in Diffraction Patterns

- A set of parallel crystal planes is defined by the Miller indices (*hkl*) and a set of such planes is {*hkl*}.

- We define the direct beam as the 000 reflection and each diffracted beam as a reflection with different *hkl* indices.

- It is a crystallogrphic convention to refer to the diffraction spot from a specific (*hkl*) planes as *hkl*.

- If we assign *hkl* to **g**, then the second –order (2**g**) spot is *2h 2k 2l,* the 3**g** spot is *3h 3k 3l,* etc.

- The zone axis, [*UVW*], is a direction which is common to all the planes of the zone. So [UVW] is perpendicular to the normal to the plane *(hkl)* if the plane is in the [*UVW*] zone.

- We will see that [*UVW*] is defined as the incident beam direction. This result applies to all crystal systems and gives the Weiss zone law: *hU+kV+lW=0*

- If there are many planes close to the Bragg orientation, then we will see spots from many different planes.

Practical Aspects of Diffraction-Pattern Formation

- We can form diffraction patterns in the TEM in two complementary ways, SAD and CBED patterns
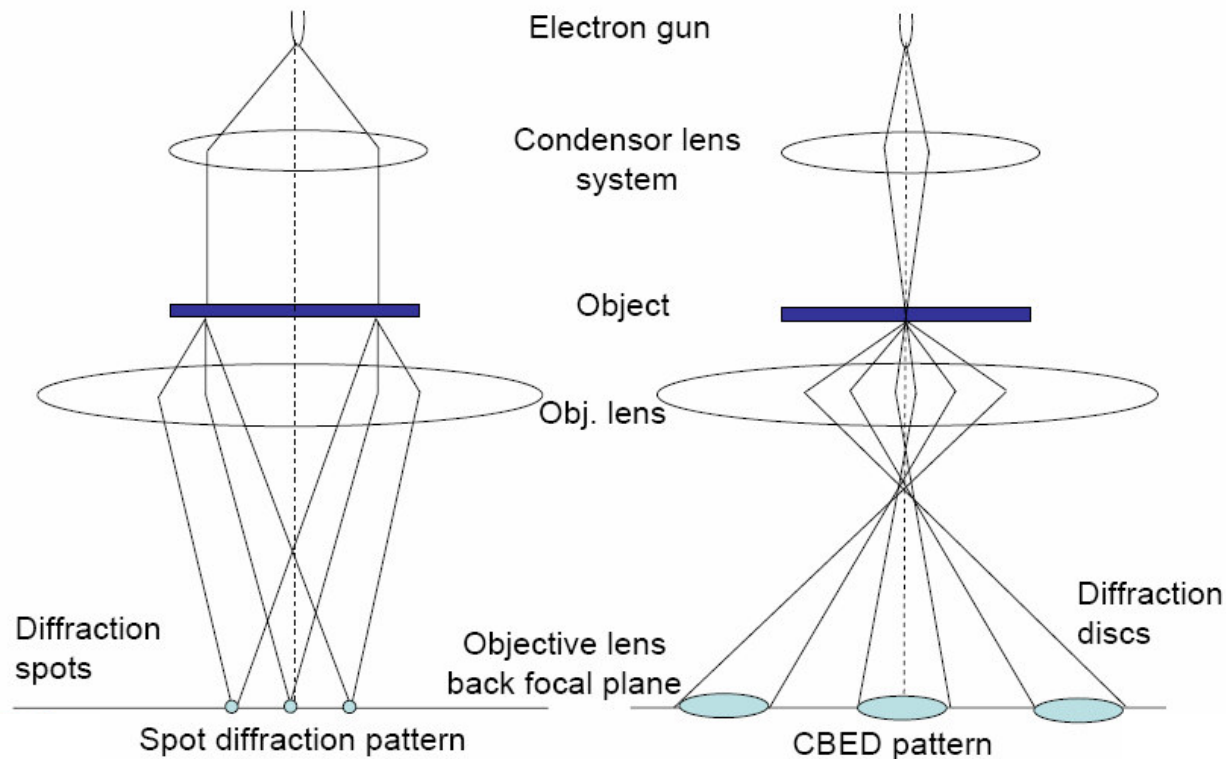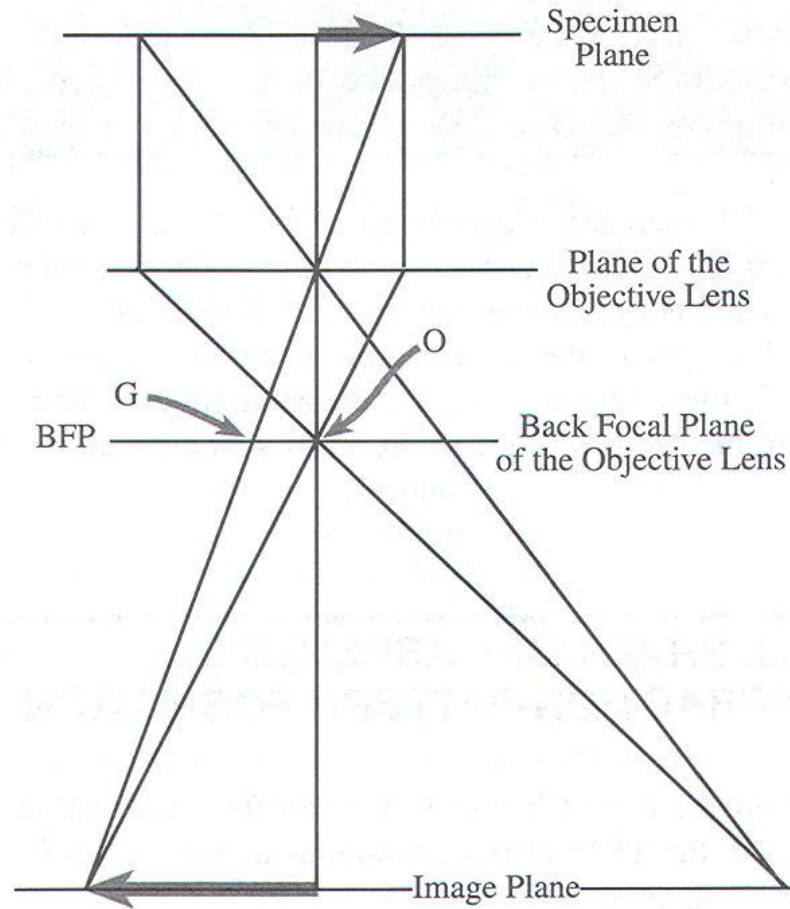
More on Selected-Area Diffraction Patterns

Why do we want to select a specific area to contribute to the DP?

•All foils are distorted to some extent so that diffraction conditions change as we cross the specimen, so we need to select areas of constant orientation.

•Also we may need to determine the orientation relationship between two different crystals, which we can do by selecting the interfacial region.

•We may want to study the DP from a small particle within the foil.

More on Selected-Area Diffraction Patterns

Figure 11.10. The diffraction pattern is formed at or close to the back focal plane of the objective lens. O is the direct beam and G is a diffracted beam.

More on Selected-Area Diffraction Patterns

The key practical steps in forming an SAD pattern are:

❑ Be sure that you are at the eucentric focus position, with an image of the area of interest focused on the screen.

❑ Insert the SAD aperture.

❑ Remove the objective aperture.

❑ Focus the SAD aperture.

❑ Switch to diffraction mode.

❑ Spread the beam using C2, within the limits imposed by your specimen.

❑ Focus the DP with the intermediate lens (diffraction focus).

More on Selected-Area Diffraction Patterns

Why can't we just use a smaller SAD aperture to select a smaller area?

• The objective lens is not perfect. The beams which are further away from the optica axis are bent more strongly as they pass through the objective lens.

•For rays entering the lens at an angle β to the optic axis, the image formed at magnification M, is translated a distance r_M given by

$$r_M = MC_s\beta^3$$
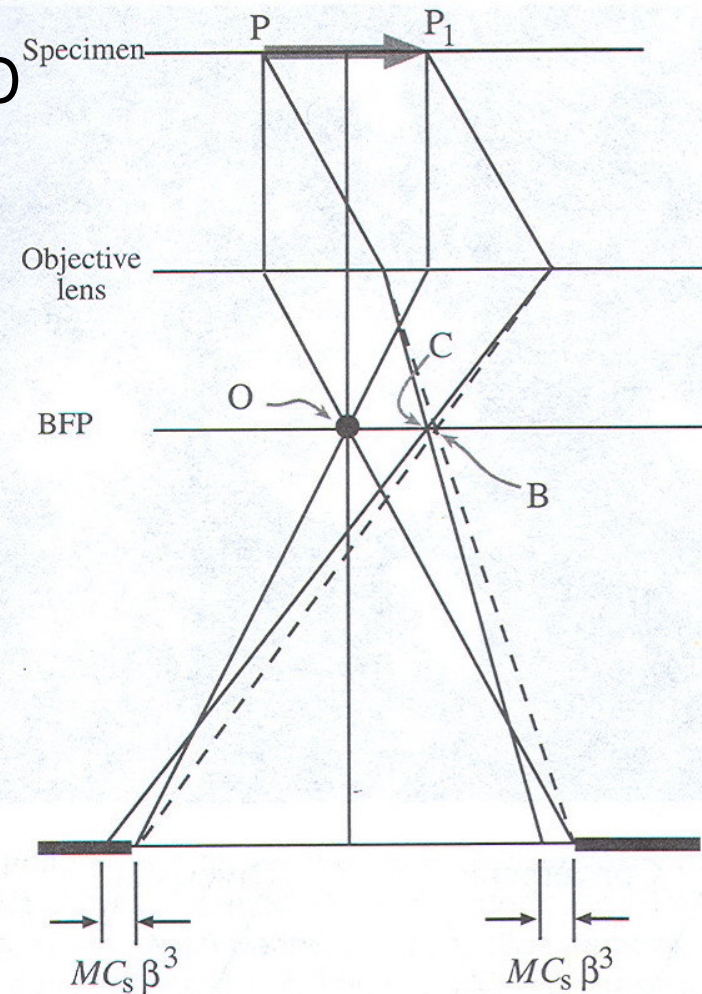
Figure 11.11. Formation of an SAD pattern showing that there is an error in selecting the area if the beams do not travel at the same angle to the optic axis. This difference is due to spherical aberration in the objective lens. B is the diffraction spot position for a perfect lens and C is the spot position with spherical aberration.

More on Selected-Area Diffraction Patterns
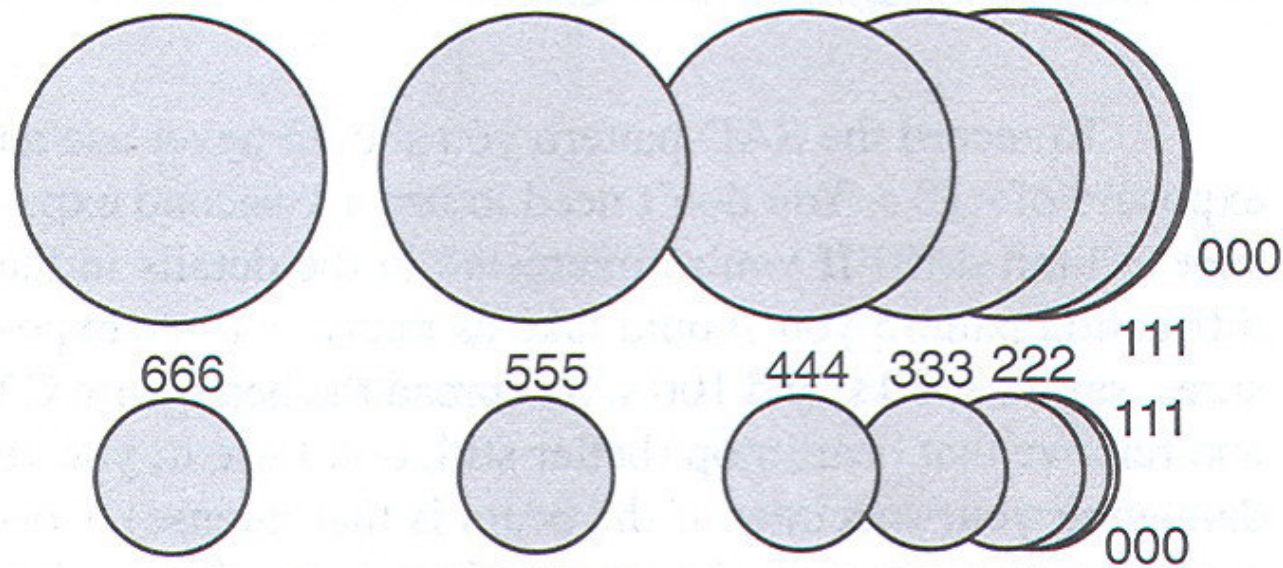
Figure 11.12. Schematic diagram showing the effective error in area selection, due to spherical aberration, for different reflections in the 111 systematic row for Al ($a_0 = 4.04$ Å) assuming 100-keV electrons and $C_s = 3$ mm. The 000 and 111 disks almost exactly overlap (the translation is 13 nm). The diameter of each disk in the top row is 1 µm, and the diameter of each disk in the bottom row is 0.5 µm.
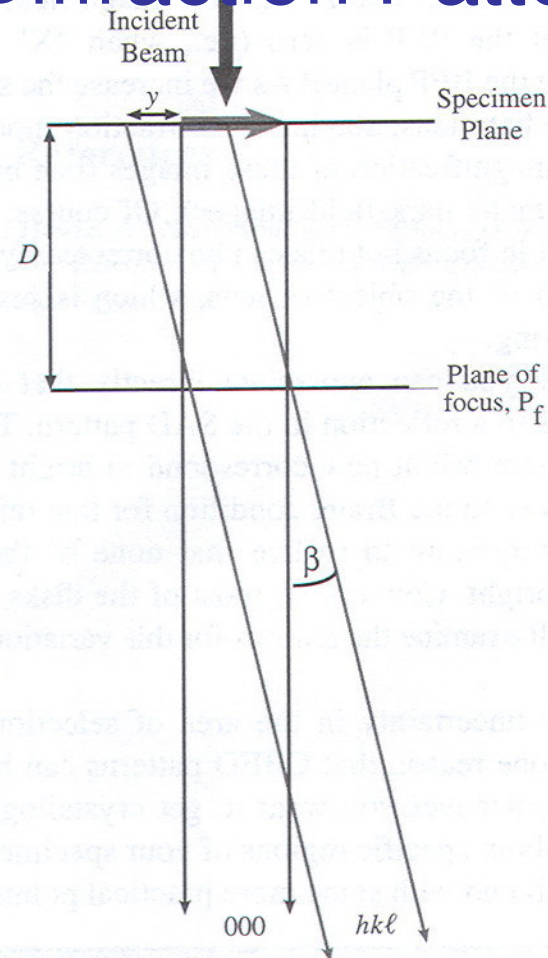
More on Selected-Area Diffraction Patterns

Table 11.1. The Displacement Distance of the Image Formed by "reflection G" Due to Spherical Aberration

| Reflection in SAD pattern	$C_s\beta^3$ (nm) old TEM	$C_s\beta^3$ (nm) modern IVEM
111	13	1.2
222	100	9.1
333	350	31.9
444	760	69.3
555	1620	150
666	2800	250

More on Selected-Area Diffraction Patterns

We will produce another selection error if the aperture is not located at the image plane.

$$Y=D\beta$$

Figure 11.13. If the lens is not focused on the SAD plane, images associated with the different g vectors will be shifted with respect to one another. D is the defocus. The shift in the selected area is given by $y = D\beta$.

More on Selected-Area Diffraction Patterns
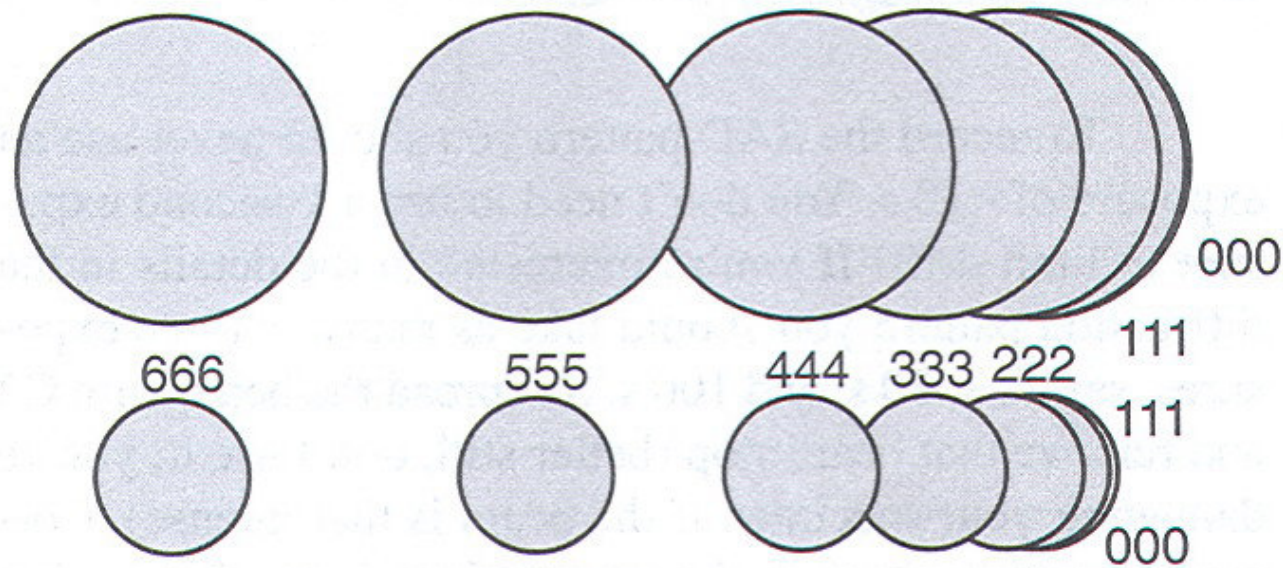
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Homework

1. For 111 planes in Cu, d is 0.21nm, for 120-kV electrons, and n=1, how much is θ_B?