2009 spring

Microstructural Characterization of Materials

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Diffraction Pattern

1. Diffraction pattern capability is one of the most important features of the TEM, because we can relate the crystallography to the images obtained.
2. The ability to determine crystallographic orientations locally (down to the nm level) gives the TEM its great advantage over the SEM and visible-light microscopes.
3. The questions that we can address using diffraction patterns obtained in the TEM include the following:
   - Is the specimen crystalline? Crystalline and amorphous materials have very different properties.
   - If it is crystalline, then what are the crystallographic characteristics (lattice parameter, symmetry, etc.) 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?

Diffraction Pattern

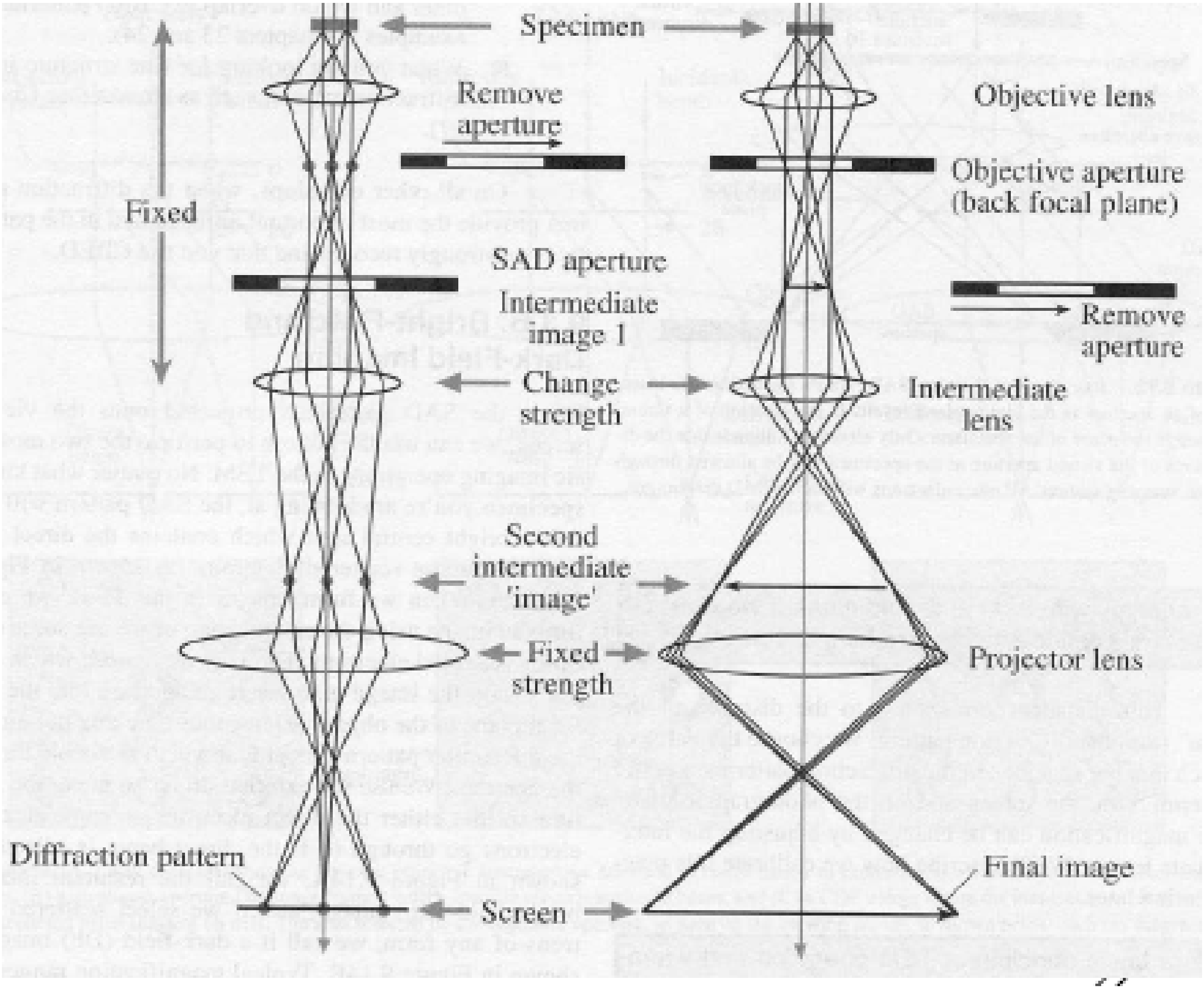
- Since the strength of the TEM is that you can obtain both crystallographic data and an image from the same part of your specimen, a method for interpreting the DP is essential.

- The first step in any interpretation is to index your DP. Using the DP, we can identify the crystal and its orientation. The positions of the allowed hkl reflections are characteristic of the crystal system.

- Indexing associates each spot in the DP with a plane, (hkl), in the crystal. From indexing of the spots, you can deduce the orientation of the crystal in terms of the zone axis [UVW] in which the indexed planes lie. This direction is normal to the plane of the DP and anti-parallel to the electron beam. It is convention to define [UVW] as the beam direction.

Diffraction versus the image

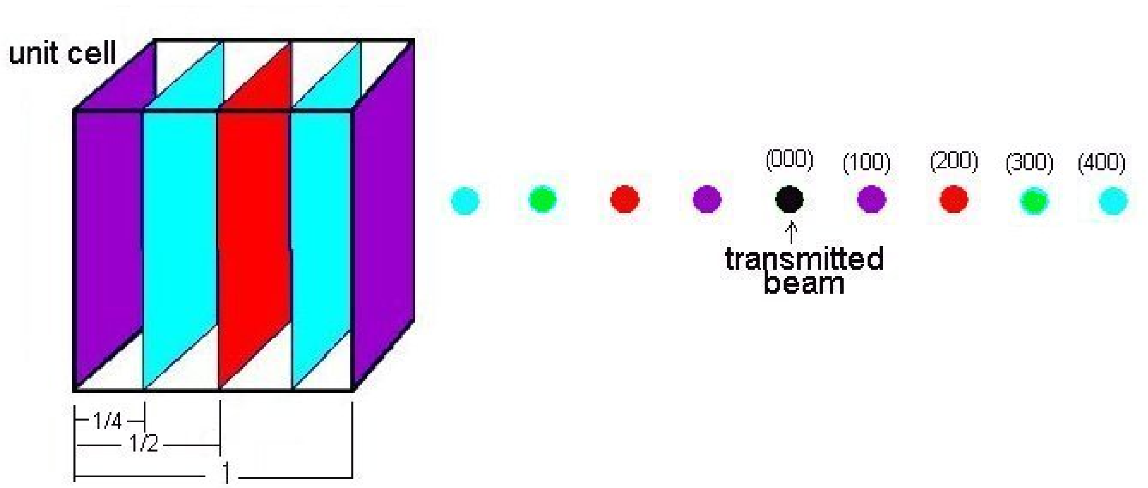
The SAD aperture is placed at the image plane of the objective lens. Standard optics tells us that a diffraction pattern is formed on the back focal plane which then recombines to form the image in the image plane.



Diffraction pattern

✓역격자에서는 차원(dimension)이 실격자의 역수로 나타나며, 실격자에서의 결정면이 역격자에서는(즉, 회절패턴에서) 점으로 나타남

✓실격자에서의 결정면의 방향이 역격자에서는 면에 대한 법선으로 나타남



가로방향을 결정의 a축이라고 하고 격자상수가 1이라고 하면 (100)면(그림에서 보라색 결정면)은 역격자 혹은 회절패턴에서 1/1=1이므로 오른쪽 그림의 보라색 점에 나타나고 이러한 역격자점, 즉 **회절패턴의 점들의 방향은** transmitted beam (000)을 출발점으로 하여 선을 그으면 모두 **실제 결정면에 수직한 법선의 방향이** 된다.

The reciprocal lattice is important because it may be used as a tool in conjunction with the Ewald sphere construction to simplify considerably the Interpretation of electron diffraction patterns.

The reciprocal lattice has the following two properties:

(1) The vector g(hkl) to the point (hkl) of the reciprocal lattice is normal to the plane (hkl) of the crystal lattice.

(2) The magnitude of g(hkl) is 1/d(hkl) where d(hkl) is the interplanar spacing of the family of (hkl) planes.

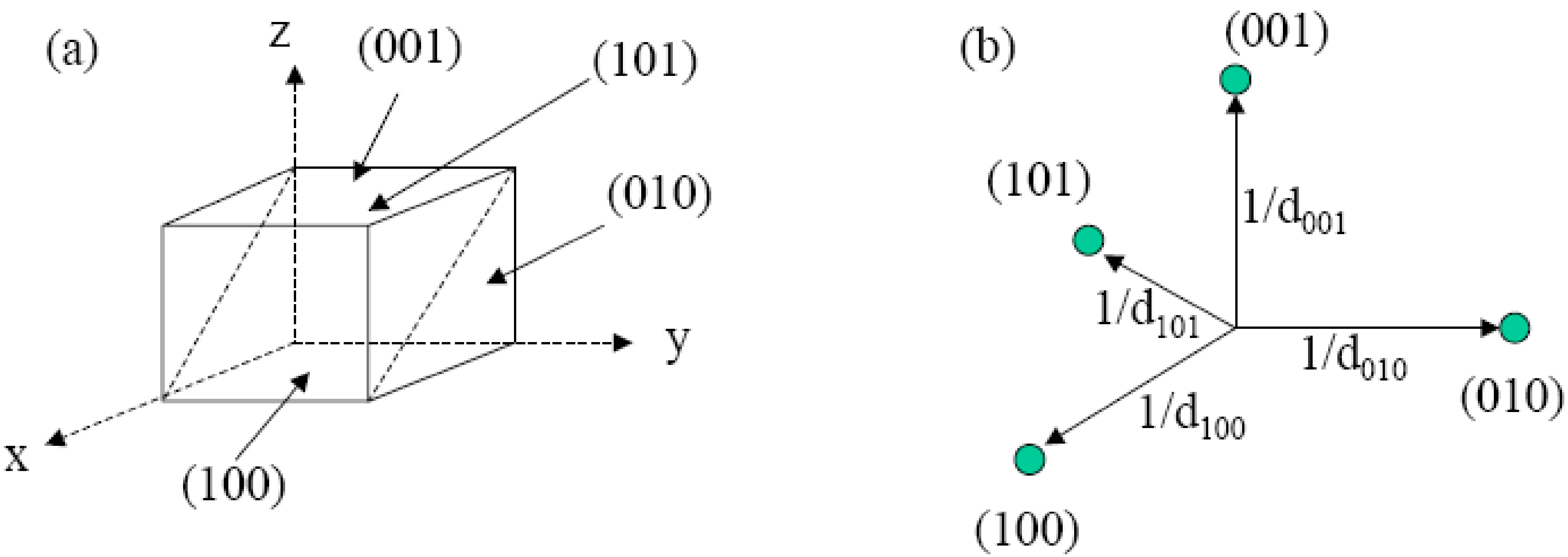
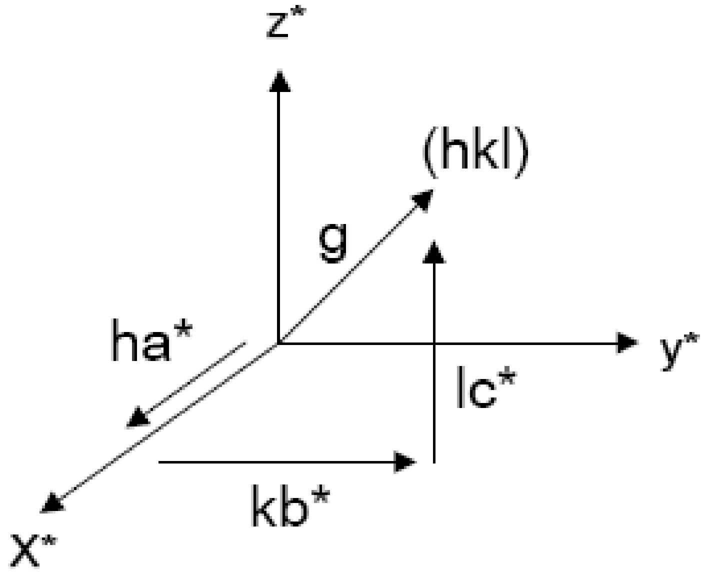


Figure (a) The relationship between crystal planes. (b) The relationship between equivalent reciprocal lattice points

- Thus we have defined the reciprocal lattice as an array of points, each point corresponding to a particular (hkl) plane and defined by a vector g. Figure (a) and (b) shows this relationship between planes in the real lattice and points in the reciprocal lattice for a cubic crystal structure. Each point is labeled with the particular (hkl) indices of the corresponding reflecting plane. Note that a point (hkl) in reciprocal space [Figure (c)] is defined by the steps ha* along the x axis, kb* along the y axis and lc* along the z axis. Thus, as shown in Figure (c):

(c) $$g_{(hkl)} = ha^* + kb^* + lc^*$$



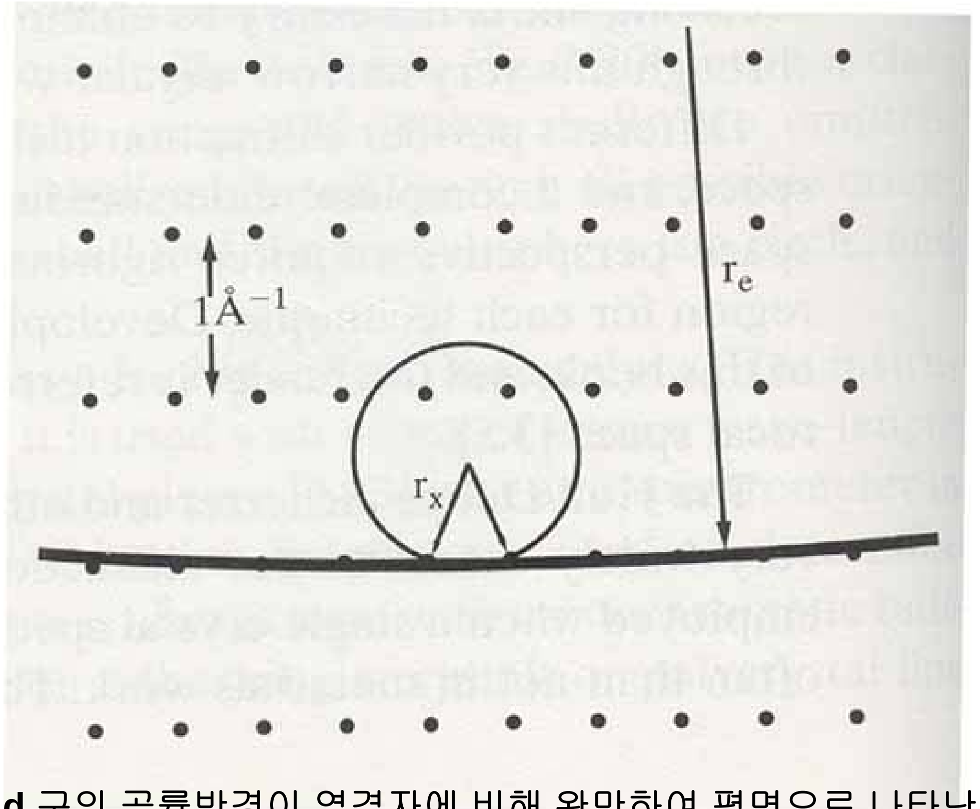
TEM: Experimental visualization of the reciprocal lattice

- **TEM**은 역격자를 직접 영상화

- 가속전압 **100 keV,** 파장 **0.037A ,** 두께 **1000 A** 시료를 투과

- 전자의 파장이 작다는 것 **– Ewald** 구의 반경이 크다

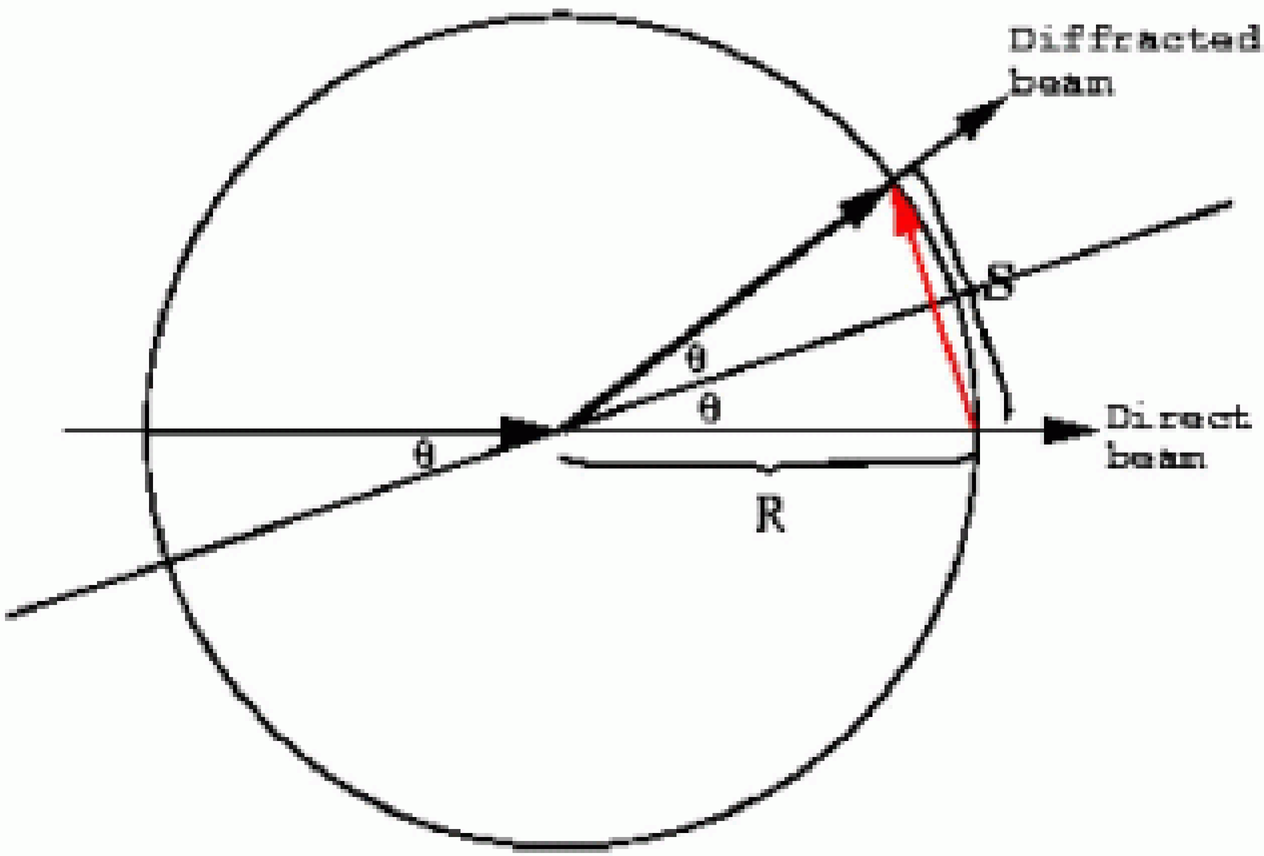
- **0.037 A** 에서 **Ewald** 구의 반경 **: 25A^{-1}**



Ewald 구의 곡률반경이 역격자에 비해 완만하여 평면으로 나타난다**.**

Ewald Sphere: 회절된 빔의 역공간에서의 격자점

The Ewald sphere construction shares the properties of Bragg's law.

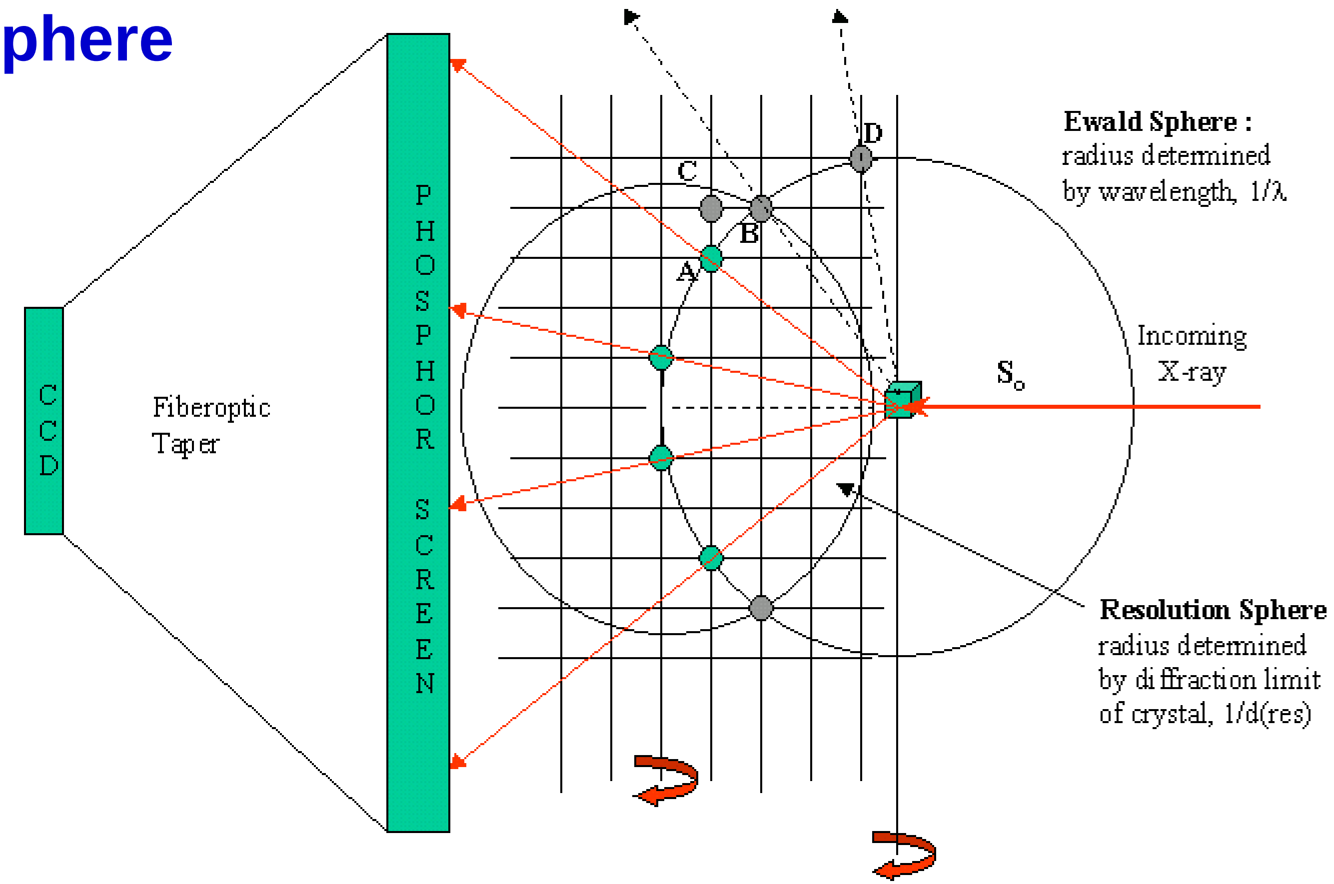


$$S = 2R\sin\theta \quad \text{Choose } R = 1/\lambda \;\&\; S = 1/d$$

$$\frac{1}{d} = \frac{2\sin\theta}{\lambda} \quad \text{(Bragg's law)}$$

회절강도의 최대값(diffraction spots)은 3개의 Laue equations이 만족되거나 혹은 Bragg 조건이 만족될 때 나타난다. 이 조건은 아래의 그림과 같이 역격자점이 정확하게 Ewald sphere에 놓일 때 성립한다.

Ewald Sphere



역격자점들이 회절점으로 기록되기 위해서는 다음과 같은 조건들이 만족되어야 한다.

1. 시편을 세로축(omega axis)주위로 회전시켜서 **역격자점들이 Ewald sphere를 교차하여야**
2. **역격자점들이 결정시편의 resolution sphere (RS)내에 놓여야**
3. 회절된 빔이 detector에 기록되어야 한다.

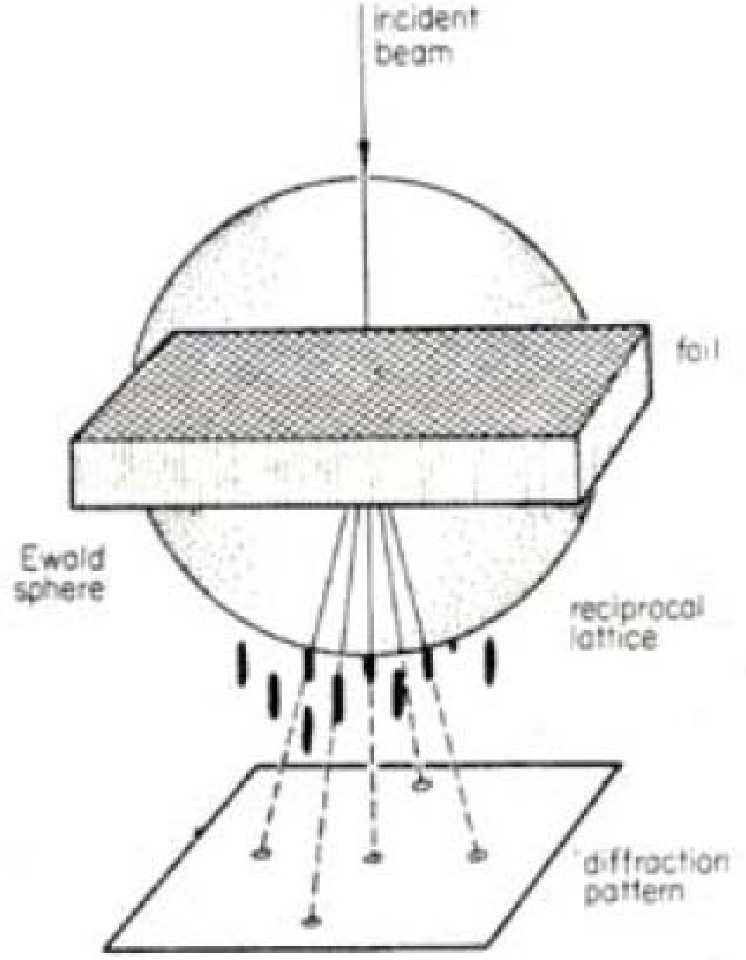
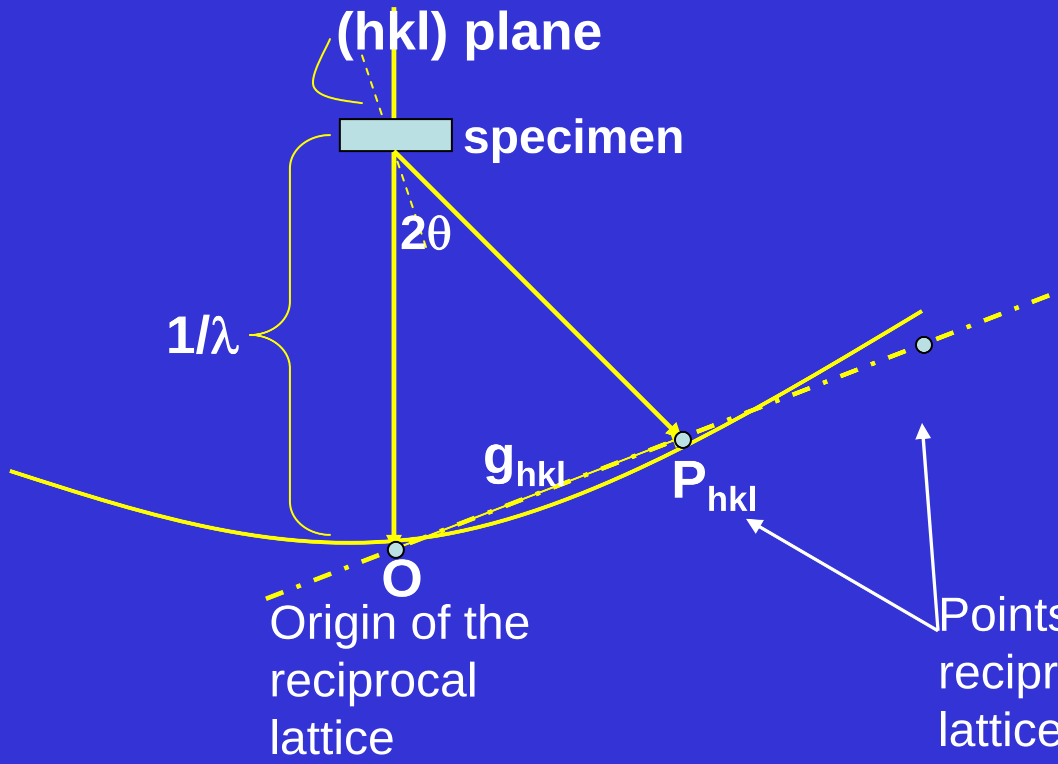


Figure 2.17 The specimen, transmitted and diffracted beams and the diffraction pattern. Superimposed is the Ewald sphere construction in reciprocal space that describes the diffraction pattern

Ewald sphere construction:

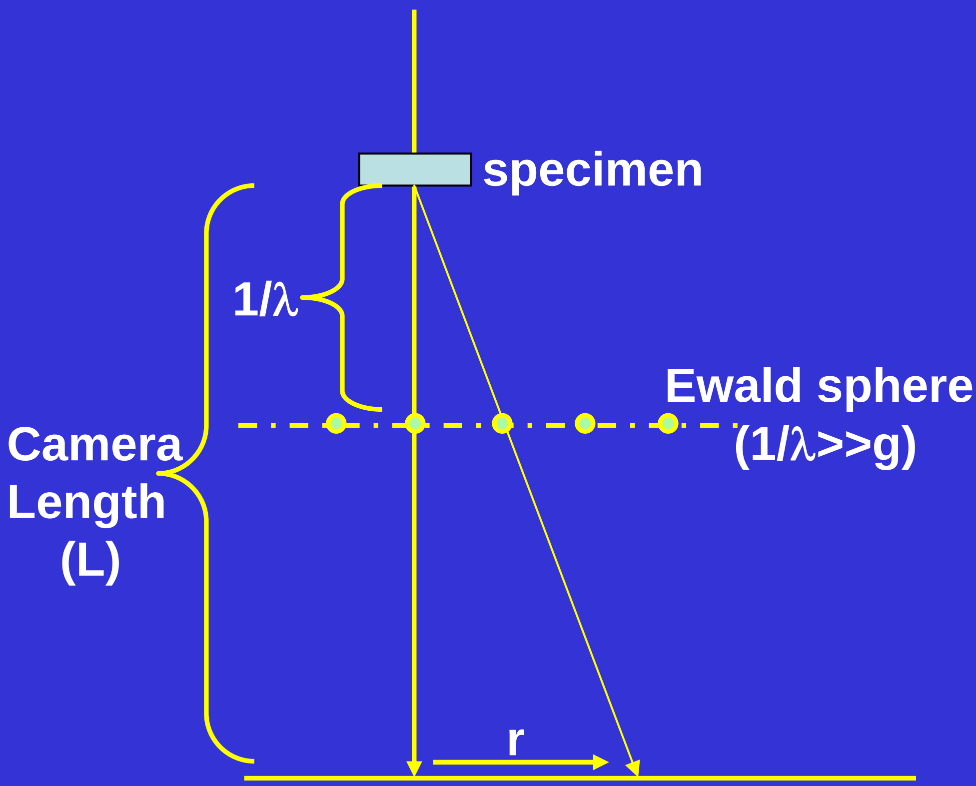


$$\sin\theta = \frac{g/2}{1/\lambda} = \frac{1/d_{hkl} * 1/2}{1/\lambda} = \lambda/2d_{hkl}$$

Bragg's law

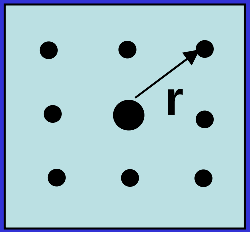
Bragg's conditions are satisfied when the Ewald sphere cuts a reciprocal lattice point specified by the indices of the reflecting plane.

For diffraction in electron microscope:

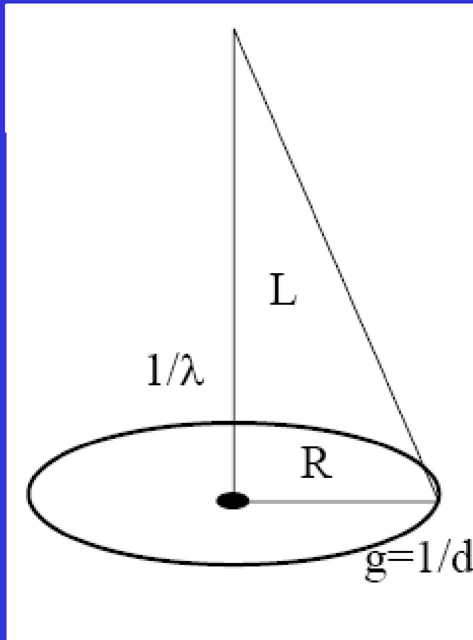


The single crystal electron diffraction pattern is a series of spots equivalent to a magnified view of a planar section through the reciprocal lattice normal to the incident beam.

$$\frac{1\backslash\lambda}{L} = \frac{g}{r};$$

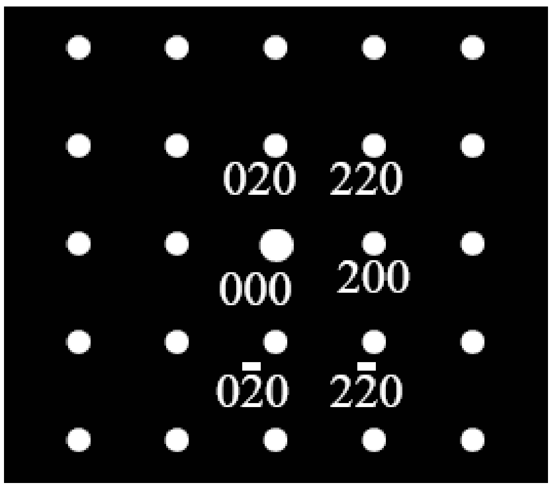


$rd_{hkl}=L\lambda$, $L\lambda$ - camera constant

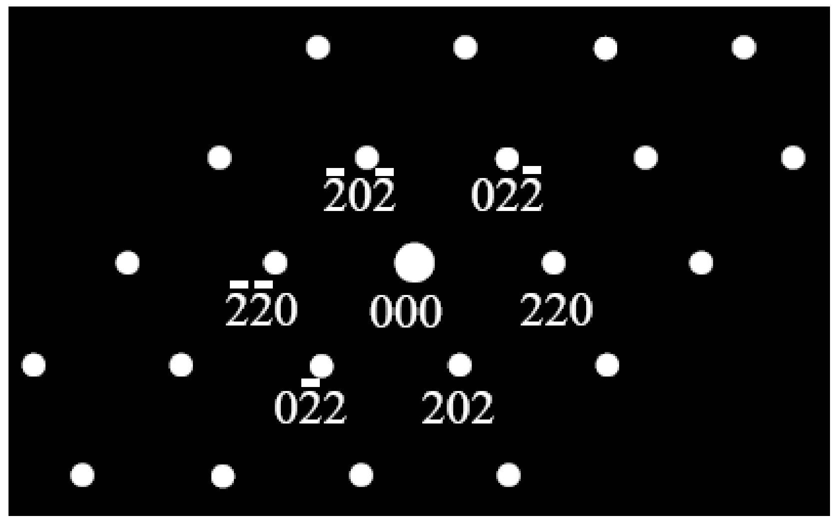


Standard spot pattern

- Example 1: f.c.c



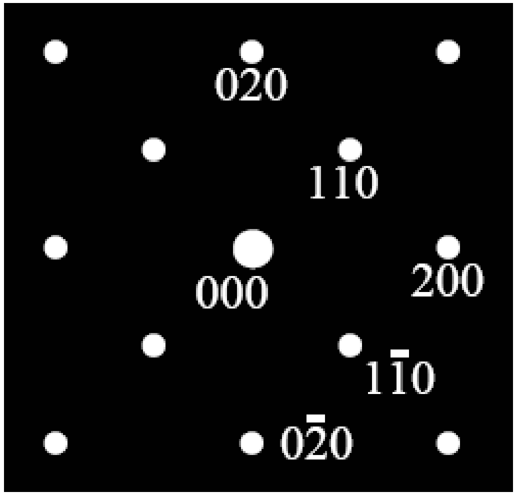
[001]



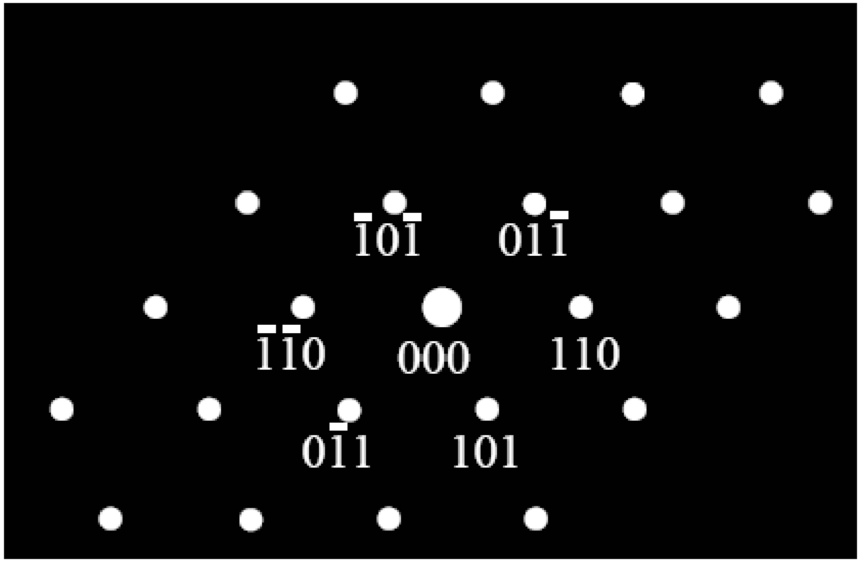
[$\bar{1}$11]

Standard spot pattern

- Example 2: b.c.c

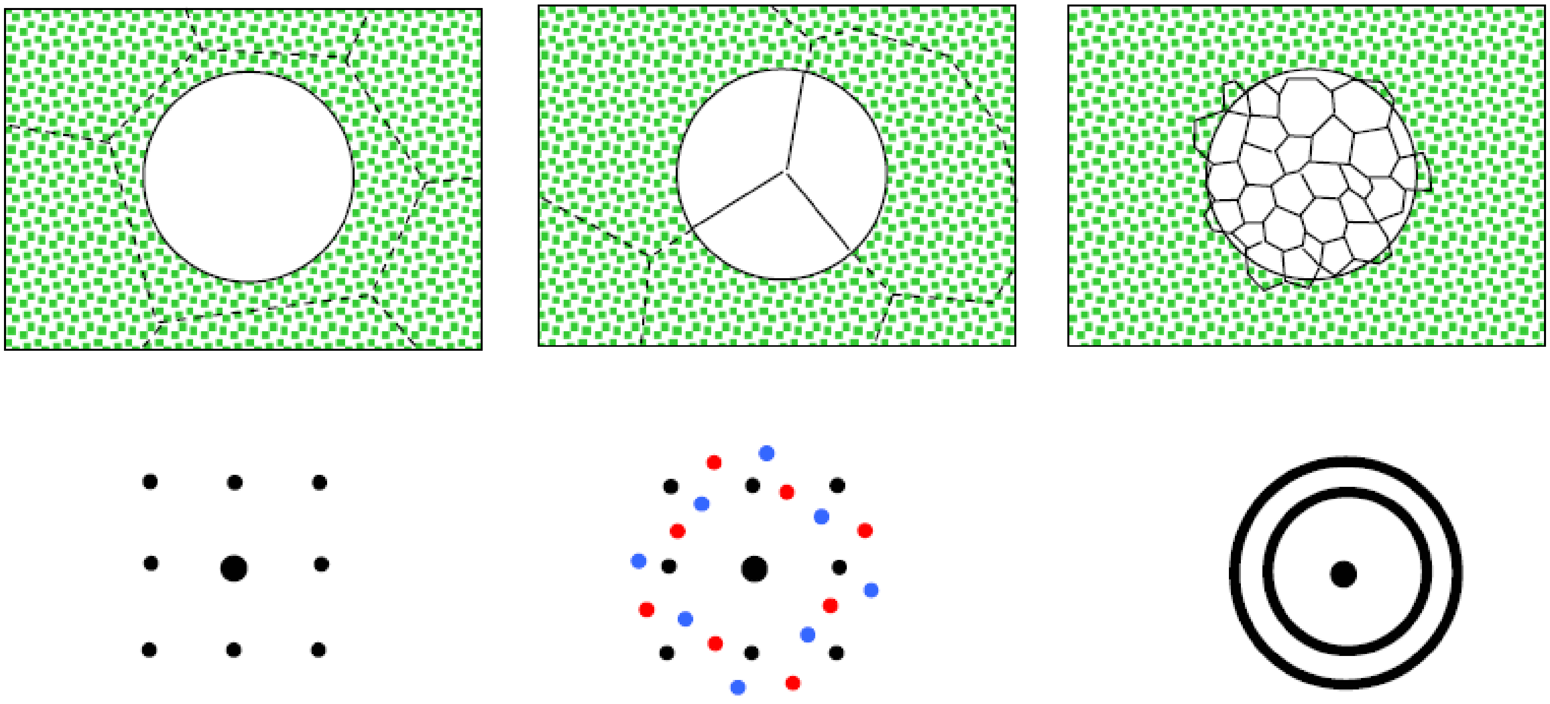


[001]



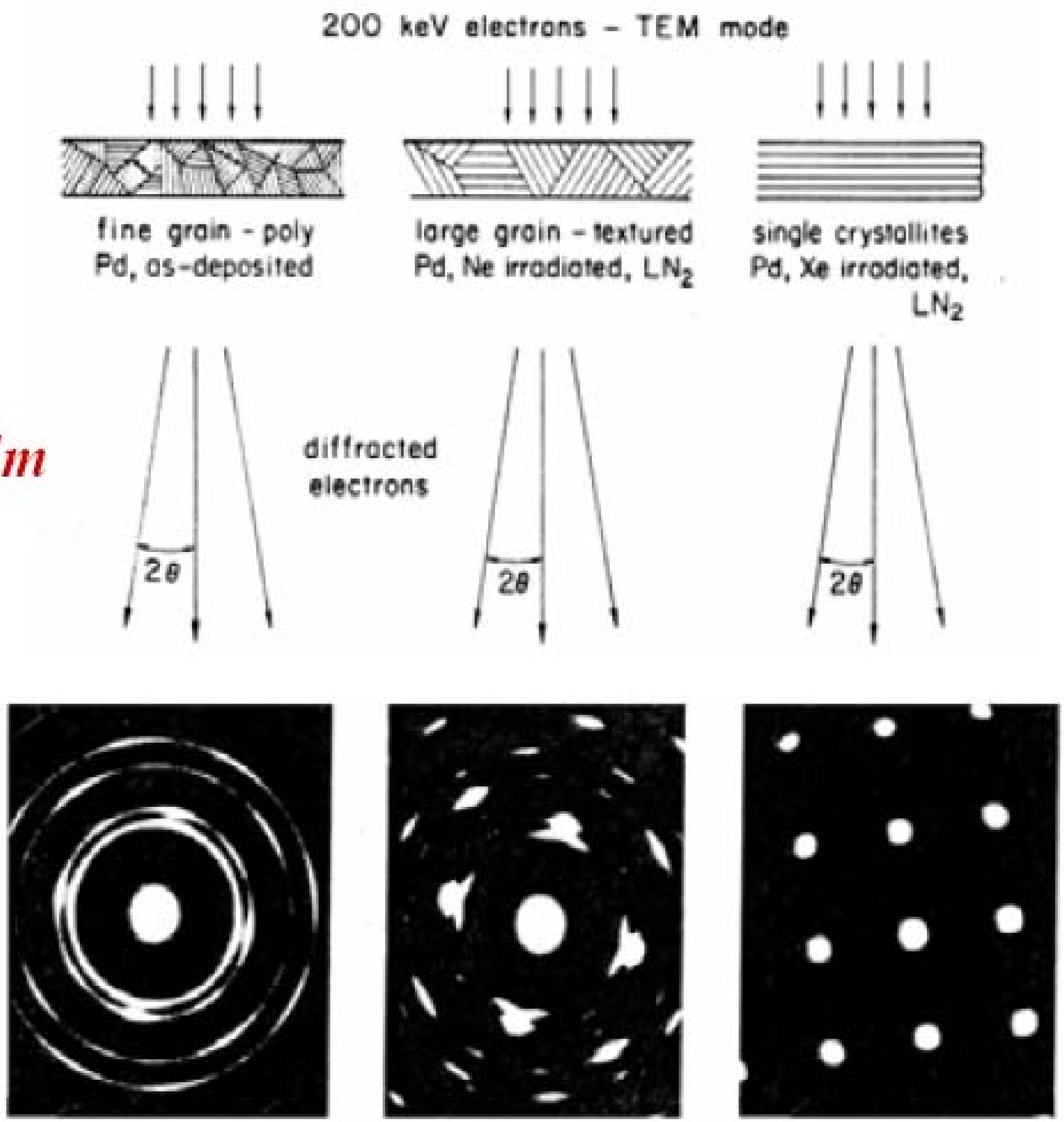
[$\bar{1}11$]

Electron Diffraction Pattern--Spot to Ring



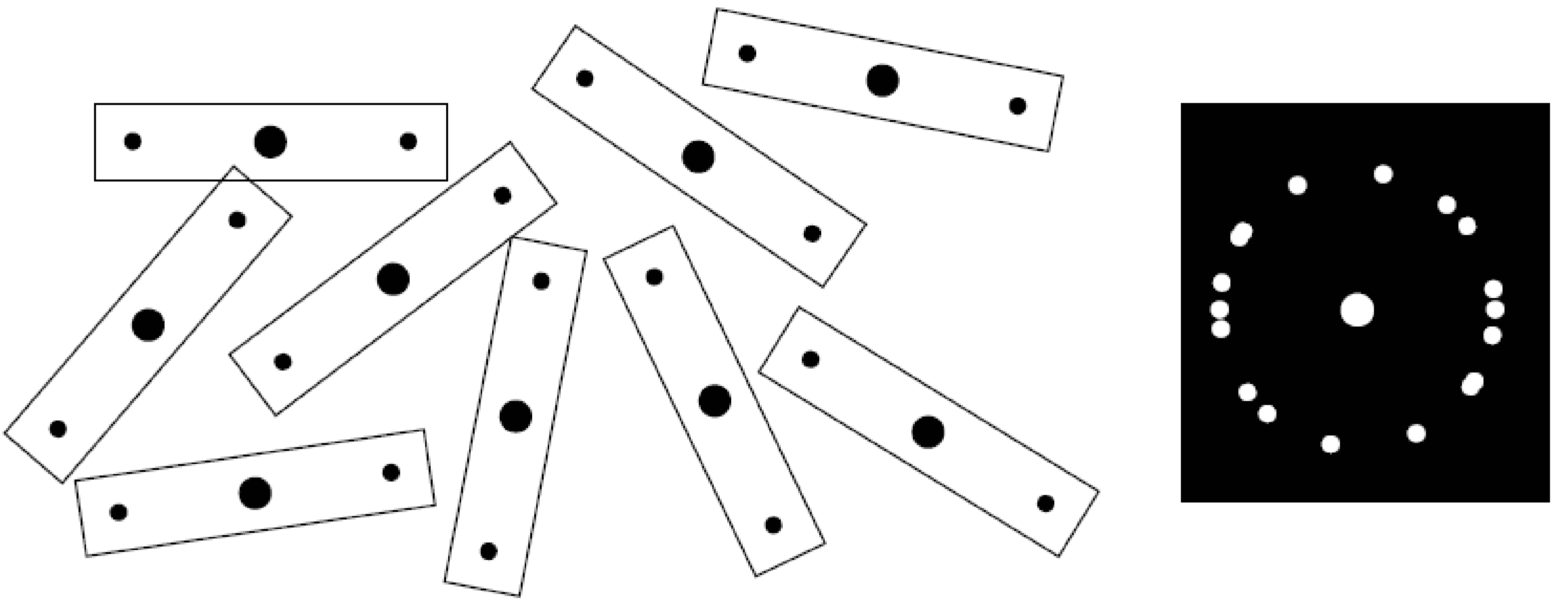
(a) (b) (c)

Electron Beam Diffraction of a Pd film

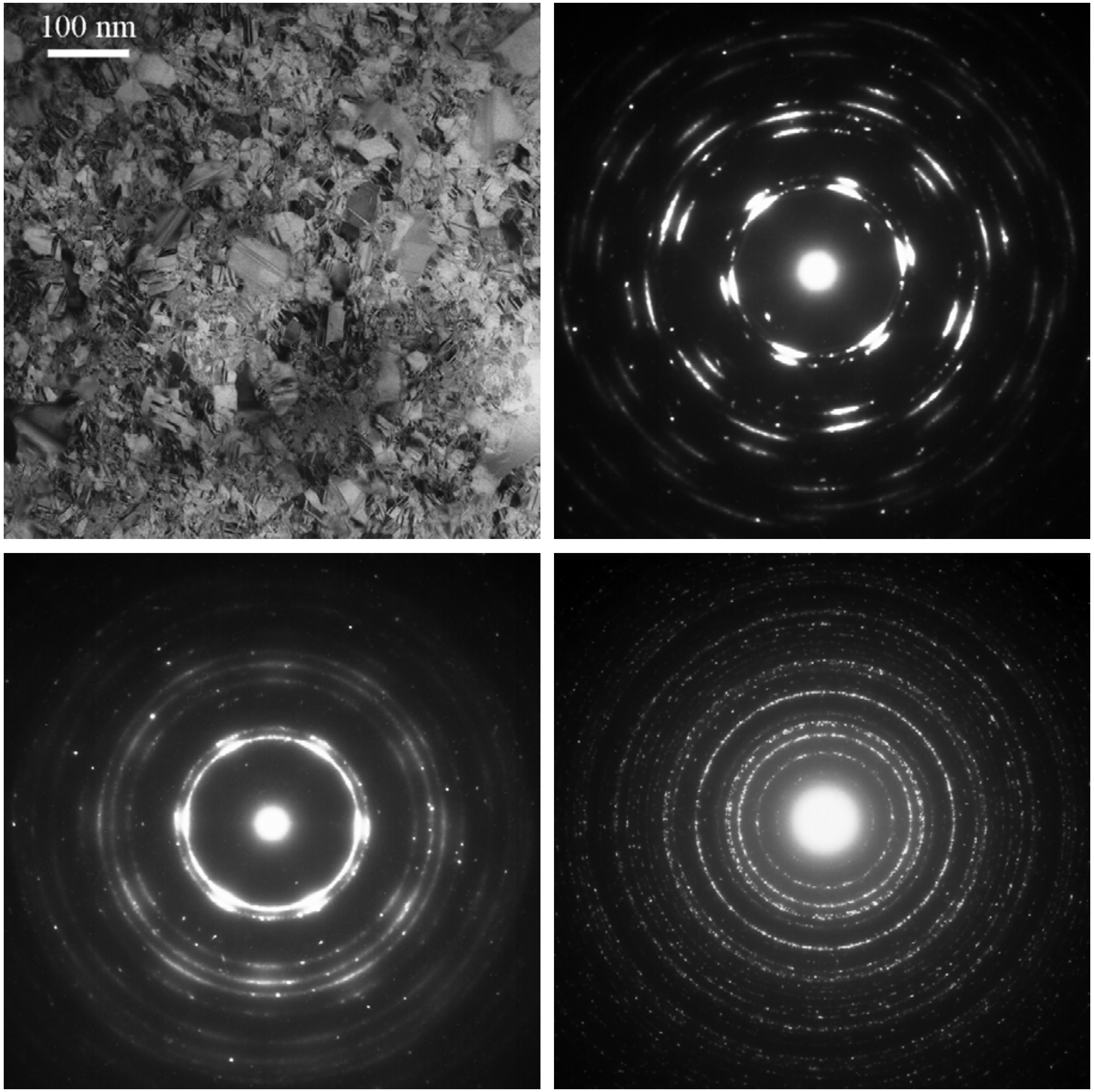


Ring pattern

- Many fine particles in the illumination area, each of them is a single crystal and orientated randomly

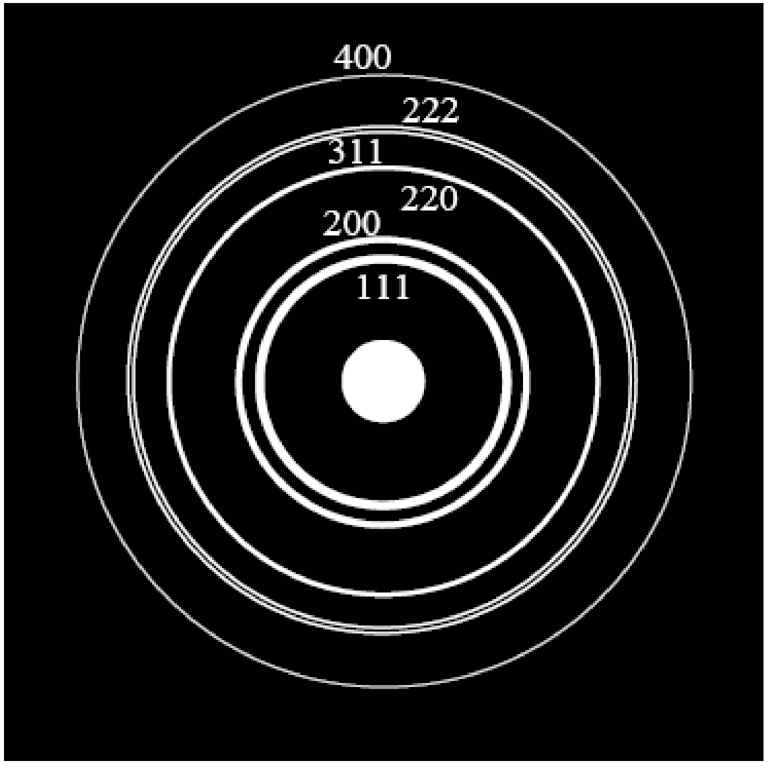


Diffraction from polycrystalline phase



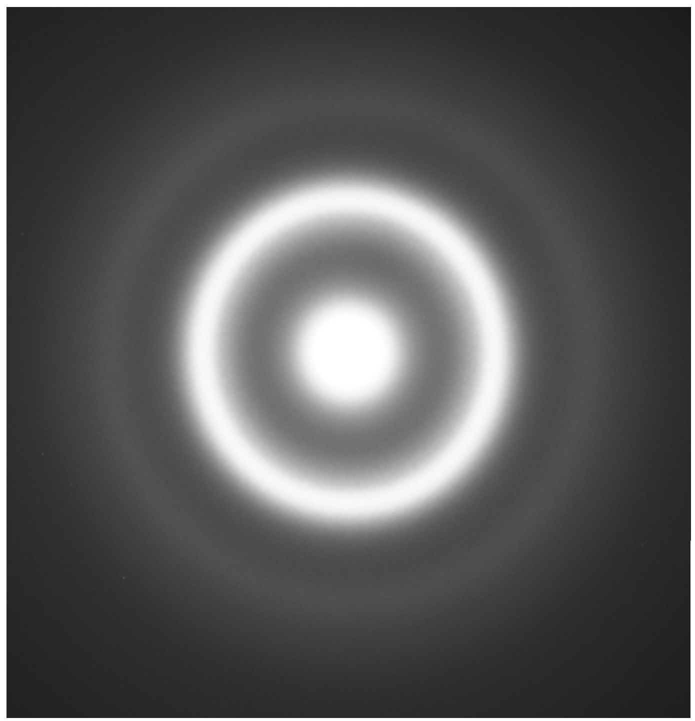
Ring pattern

- Typical polycrystalline Au diffraction pattern



Amorphous materials

- Diffused ring pattern
- Reflecting the short range ordered structure
- Often seen at contamination layer or on carbon support film

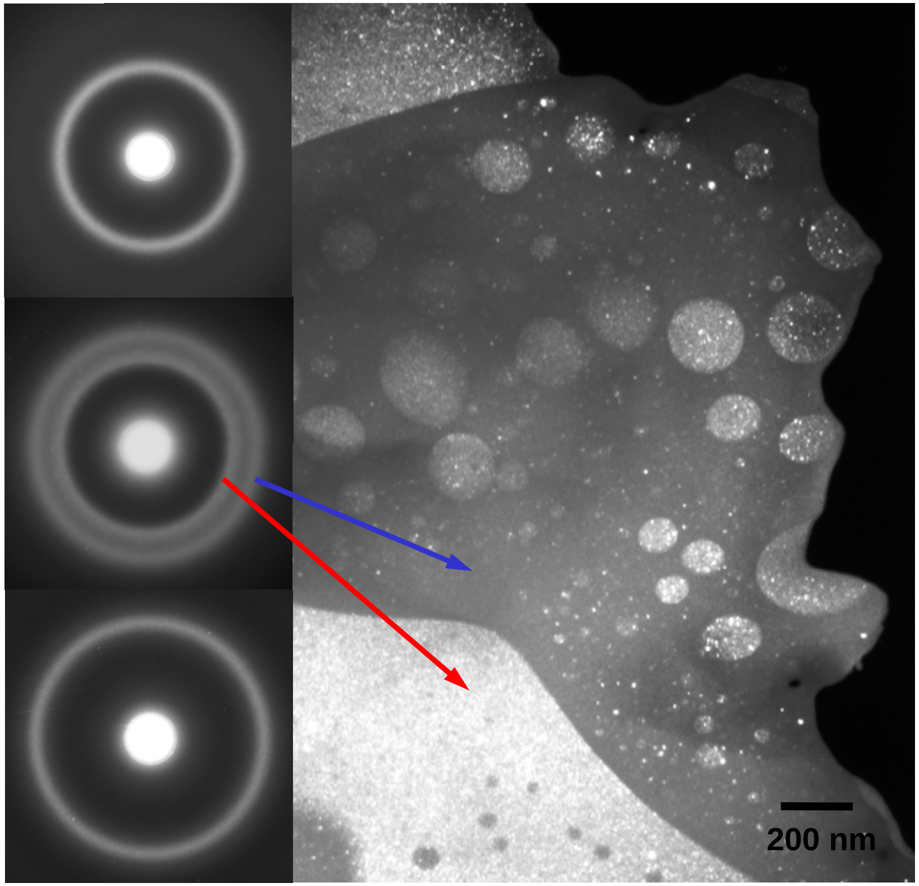


TEM results for $Nd_{30}Zr_{30}Al_{10}Co_{30}$ alloy

$Nd_{60}Al_{10}Co_{30}$
2.91 Å

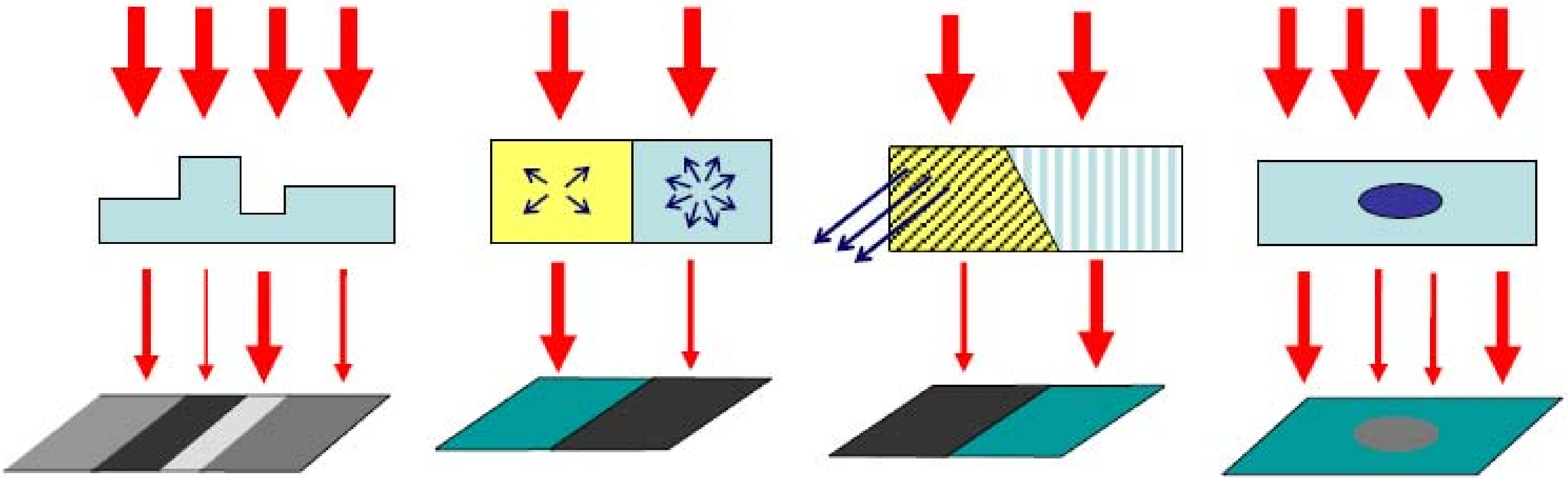
$Nd_{30}Zr_{30}Al_{10}Co_{30}$
2.37 Å, 2.99 Å

$Zr_{60}Al_{10}Co_{30}$
2.40 Å



SADP and Dark-field TEM image

Major Factors affecting TEM Image Contrast



Thickness

Scattering

Atomic number

Scattering

Orientation

Diffraction

Defects

Scattering & diffraction