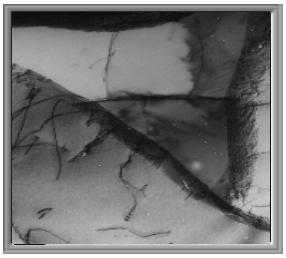
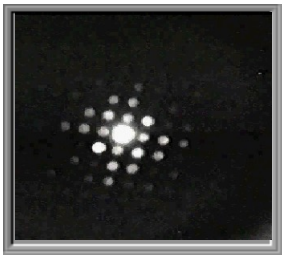
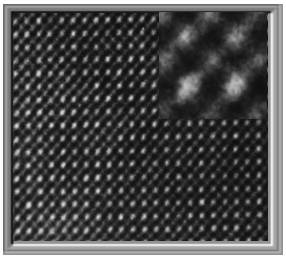
Transmission Electron Microscopy

Conventional Imaging





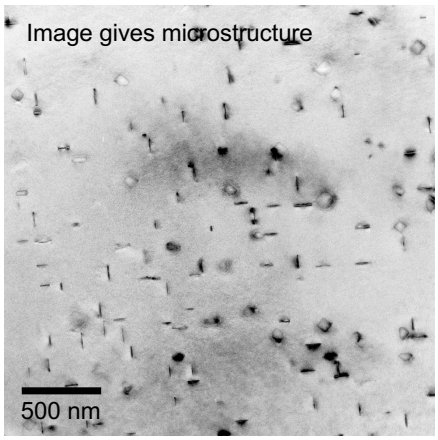
High Resolution Imaging



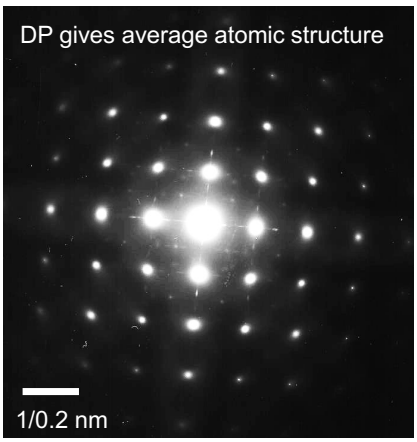
Diffraction

1

Imaging and Diffraction – an example



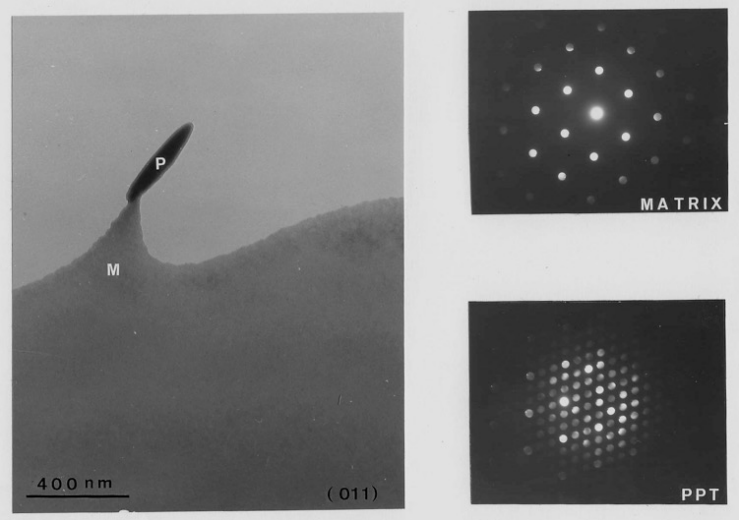
Al-Cu-Sn Alloy – BF TEM image



Al-Cu-Sn Alloy – Diffraction Pattern

2

Electron Diffraction



3

Roles of the Lenses

Gun Lens

Helps form probe

Condenser Lens

Mainly controls:
Spot Size
hence total beam current

Objective Lens

Mainly controls
Focus, 1st Magnification

Diffraction/Intermediate Lens

Controls Mode

Projector Lens

Magnification

Most TEM/STEM have 7-9 Lenses

1 Gun Lens
2-3 Condensers
1 Objective
1-2 Intermediate
1-2 Projectors

Most instruments Have Electromagnetic Round Lenses

Exception: Aberration Corrected Systems

Note the locations of the various Apertures.

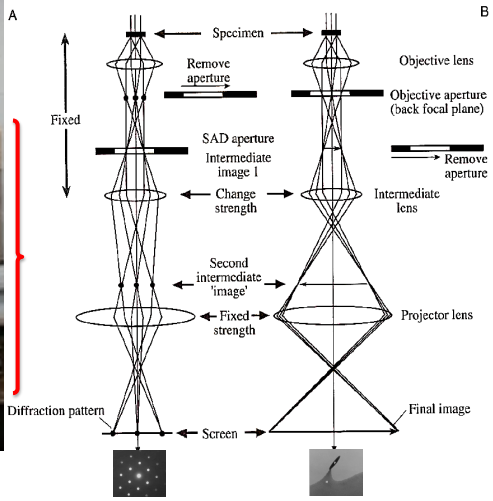
Optimum aperture sizes are needed for various imaging functions.

4

Projection/Imaging System

Projects/Magnifies the Diffraction Pattern (Back Focal Plane)

Projects/Magnifies the Image (Image Plane)



- The TEM in unique in that we can carry easily carry out **imaging** and **diffraction** in the *same instrument*

This is carried out by adjusting the strength of the Intermediate lenses in the **projection system**

Other instruments can do this but it is much more difficult!

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Electron Diffraction

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?

6

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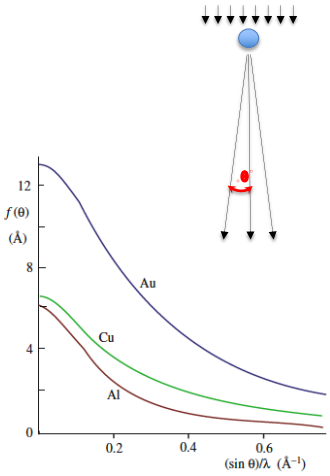
Elastic Scattering from Single Atoms

$$I = |f(\theta)|^2$$

$$f(\theta) = \left(\frac{1+\frac{E_0}{m_0c^2}}{8\pi^2 a_0}\right)\left(\frac{\lambda^2}{\sin^2\left(\frac{\theta}{2}\right)+\left(\frac{\theta_0}{2}\right)^2}\right)(z - f_x)$$

$$\theta_0 = \frac{0.117Z^{1/3}}{E_0^{1/2}}$$



a_o = Bohr Radius ~ 0.52917 Å
E_o = Accelerating Voltage (kV)
z = Atomic Number
f_x = x-ray scattering factor

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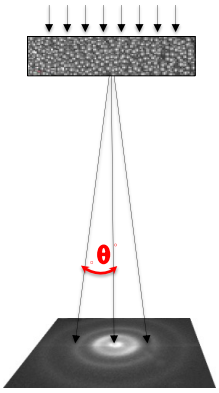
Diffraction Principles : Real Specimen
Scattering from a collection of an amorphous collection of atoms
- neighboring atoms give rise to interference

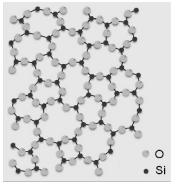


Amorphous Solids:

$$I(\theta) \sim |f(\theta)|^2 \cdot \left|1 + \frac{\sin(kR)}{kR}\right|$$

$$k = \frac{4\pi}{\lambda}\sin(\theta)$$

R = Average Interatomic Spacing



Amorphous Silica (SiO_2)

8

8

Scattering from an collection of an amorphous collection of atoms
- neighboring atoms give rise to interference

Amorphous Solids:

$$I(\theta) \sim |f(\theta)|^2 \bullet \left|1 + \frac{\sin(kR)}{kR}\right|$$

$$k = \frac{4\pi}{\lambda}\sin(\theta)$$

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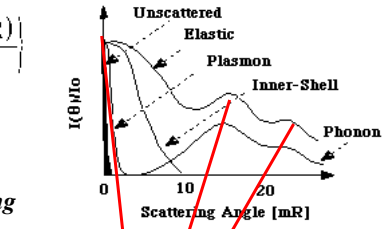
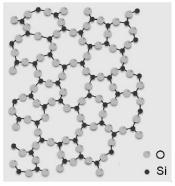
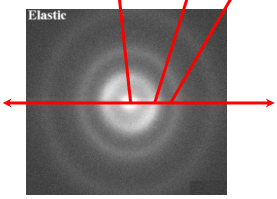


Amorphous Silica (SiO_2)





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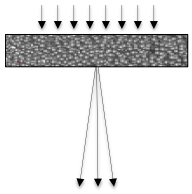
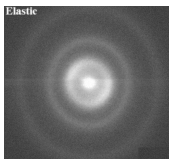
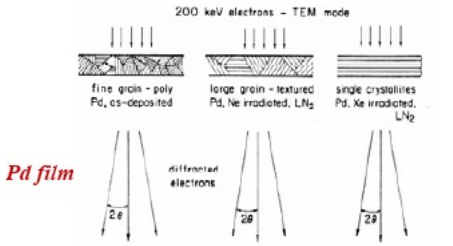
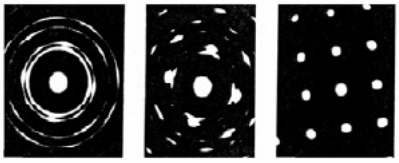
Disordered
Amorphous
Non-Crystalline





Ordered
Crystalline





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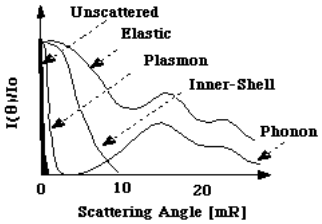
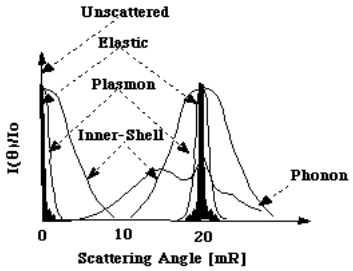
Type of Electron Scattering	Angular Range	Energy Loss
Unscattered	0	0
Elastic	10's-100's mR	~0
Phonon	10's-100's mR	<.025 eV
Inelastic	10's mR	everything else



Amorphous Solids



Crystalline Solids

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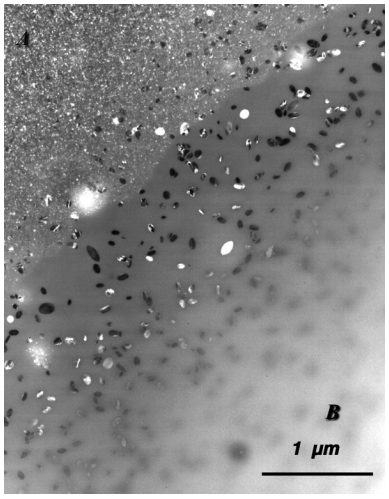
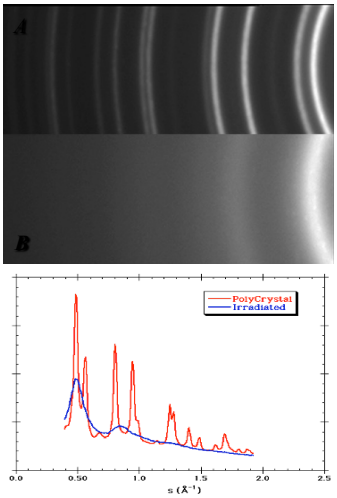
Solid State Order/DisOrder/Amorphization/ Studies





P.C. Liu, P. R. Okamoto,

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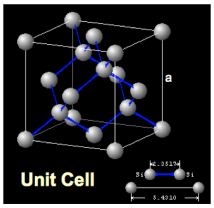
Crystal Structures

- The periodic arrangement of the atoms is called the Lattice.
- Unit Cell:
  Representative of the entire lattice and is regularly repeated throughout the crystal.
- Primitive Cell:
  Smallest unit cell which can be repeated to form the lattices.



Primitive Cell

Each crystal built up of a repetitive stacking of unit cells each identical in size, shape, and orientation with every other one.

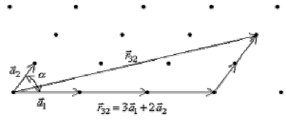
Crystal Structures

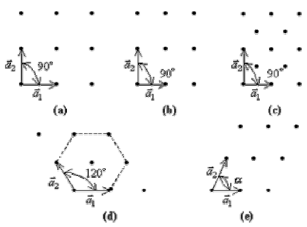
- Coordinates of position in the unit cell
  - x, y, z expressed in terms of the unit cell edges.

$$\vec{r}_{xyz} = x\vec{a} + y\vec{b} + z\vec{c}$$

Example

$\vec{r}_{321}$ reached by moving along the axis a distance of 3x the length of the vector $\vec{a}$, the parallel to $\vec{b}$, a distance $2\times\vec{b}$, and finally parallel to $\vec{c}$, a distance equal to the length of $\vec{c}$.

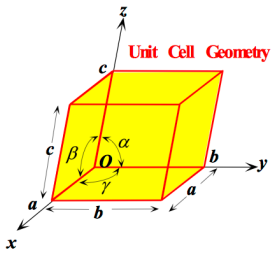




Crystal Lattice Group

- Bravais lattices

Length and Angle



Triclinic	a≠b≠c	α≠β≠γ≠90°	K_2CrO_7
Monoclinic	a≠b≠c	α=γ=90°≠β	β-S, $CaSO_4 \cdot 2H_2O$
Orthorhombic	a≠b≠c	α=β=γ=90°	α-S, Ga, Fe_3C
Tetragonal	a=b≠c	α=β=γ=90°	β-Sn, TiO_2
Cubic	a=b=c	α=β=γ=90°	Cu, Ag, Zn, NaCl
Hexagonal	$a_1=a_2=a_3\neq c$	α=β=90°, γ=120°	Zn, Cd
Rhombohedral	a=b=c	α=β=γ≠90°	As, Sb, Bi

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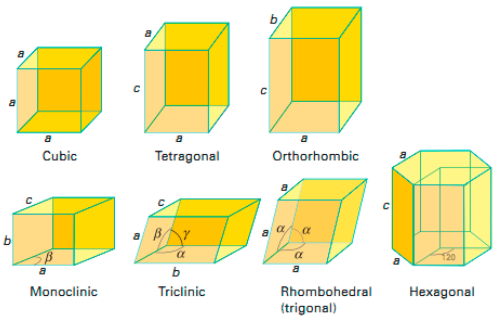

7 Crystal Systems 14 Bravais Lattices

Sr. No.	Crystal System	Axial length of Unit Cell	Inter axial angles	Number of Lattice in the system
1	Cubic	$a=b=c$	$\alpha=\beta=\gamma=90^0$	3
2	Tetragonal	$a=b\neq c$	$\alpha=\beta=\gamma=90^0$	2
3	Orthorhombic	$a\neq b\neq c$	$\alpha=\beta=\gamma=90^0$	4
4	Monoclinic	$a\neq b\neq c$	$\alpha=\beta=90^0\neq\gamma$	2
5	Triclinic	$a\neq b\neq c$	$\alpha\neq\beta\neq\gamma\neq90^0$	1
6	Trigonal	$a=b=c$	$\alpha=\beta=\gamma<120^0,\neq90^0$	1
7	Hexagonal	$a=b\neq c$	$\alpha=\beta=90^0$, and $\gamma=120^0$	1

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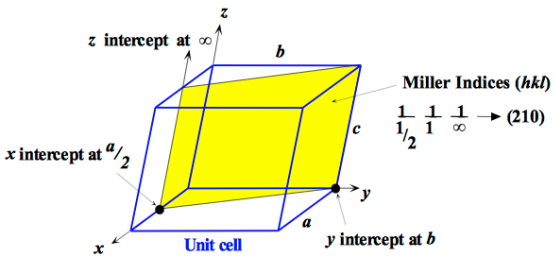
Crystal Plans

- Identification of a plan in a crystal



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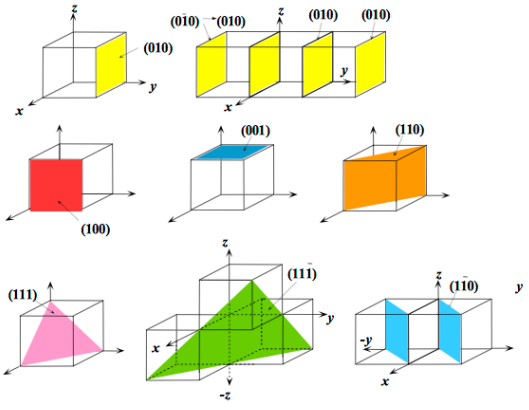
Crystal Planes in the Cubic Lattice

- Various plans in cubic lattice



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Miller Convention Summary

Convention	*Interpretation*
(hkl)	Crystal Plane
{hkl}	Equivalent Planes
[hkl]	Crystal Direction
<hkl>	Equivalent Directions

❑ *Examples*

- plane {111}: (111) (-111) (1-11) (11-1)
- direction <111>: [111] [-111] [1-11] [11-1]

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Periodic Structures
Create Constructive/Destructive Interference

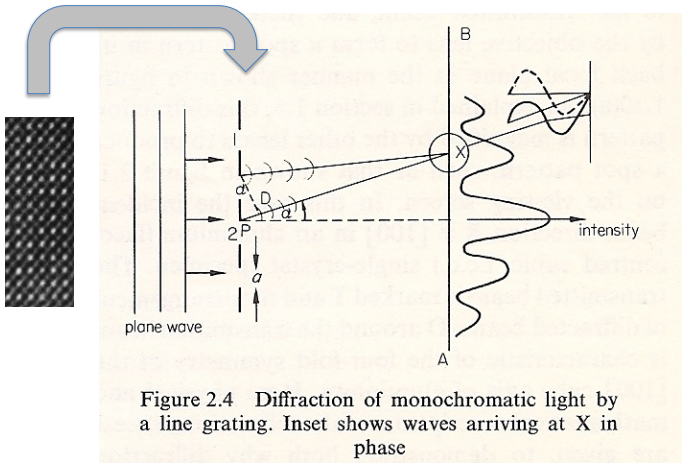
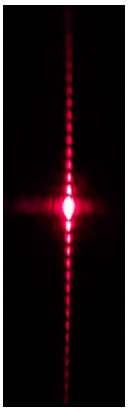


Figure 2.4 Diffraction of monochromatic light by a line grating. Inset shows waves arriving at X in phase



Young's Slit interference Experiment

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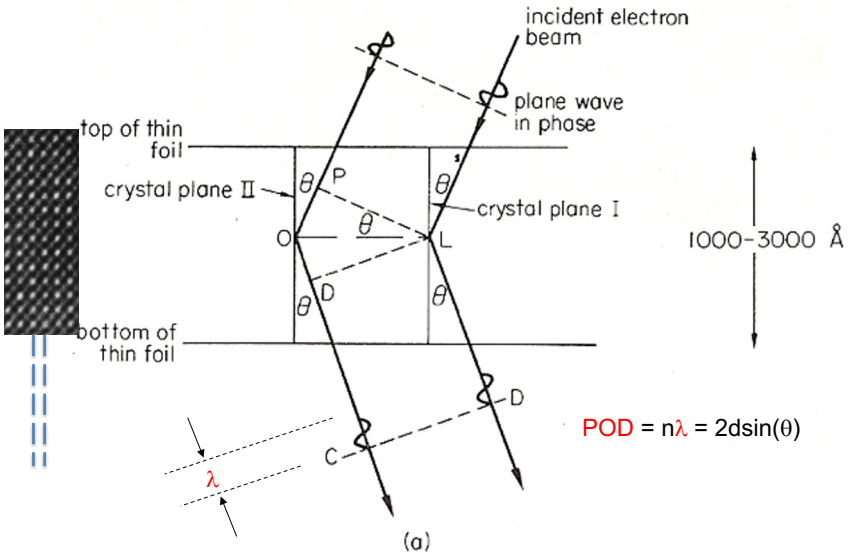
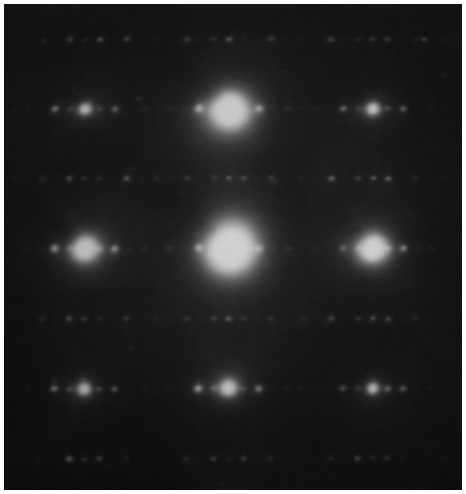
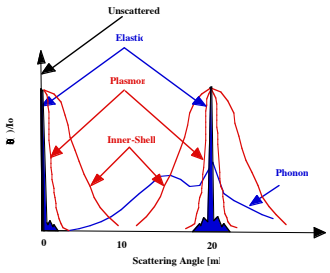
Simple Geometrical Description of Bragg's Law



POD = $n\lambda = 2d\sin(\theta)$





Elastic Scattering : Crystalline Solids

$$I(\theta) \sim |F(hkl)|^2$$

$$F(hkl) = \sum_j f_j(\theta) \exp(-2\pi i \vec{g} \cdot \vec{r})$$

$$V(r) = -\sum_g^? V_g \exp(2\pi i \vec{g}.\vec{r}) \quad ; \quad V_g = \frac{h^2}{2\pi m V_e} f(\theta)$$

Type of Diffraction Patterns

- Electron diffraction patterns produced in transmission in the electron microscope can be of three different types.
  - (a) Ring pattern — Polycrystalline, and amorphous specimen
  - (b) Spot pattern — Single-crystal region of the specimen
  - (c) Kikuchi line pattern
- Type (b) and (c) often occur on the same diffraction pattern. In general, spot and Kikuchi patterns will be taken from a specific area of the specimen and are known as 'selected area' diffraction patterns (SADPs).

Ring Pattern:

- The major use of ring patterns is in the identification of phases using extraction replicas. These diffraction patterns also arise from very fine grain size polycrystalline material such as physically or chemically vapor deposited or electrodeposited thin foils.

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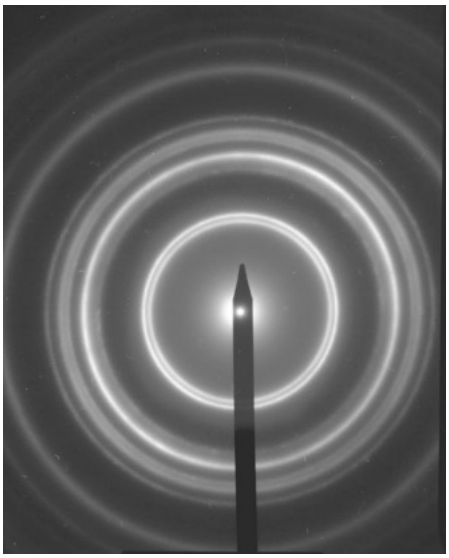
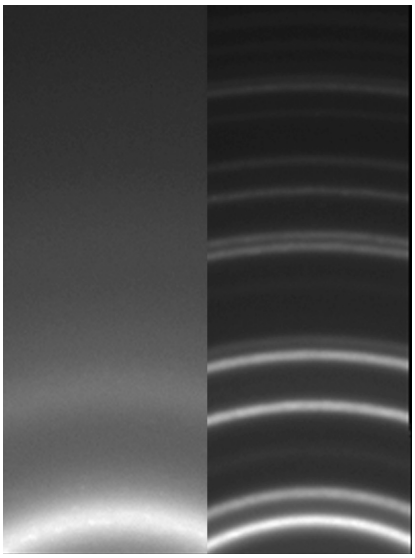
Polycrystalline vs Disordered/Amorphous





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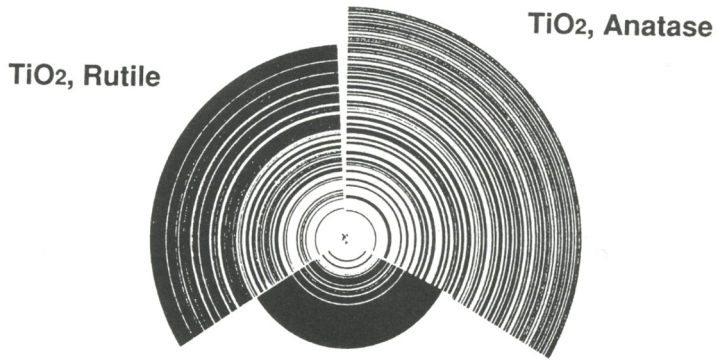

Figure 5.10. Sections of ring patterns of the three phases of TiO_2 (rutile, brookite, and anatase) showing that the largest d-spacings (smallest r-spacings) are the most diagnostic for identifying these compounds.

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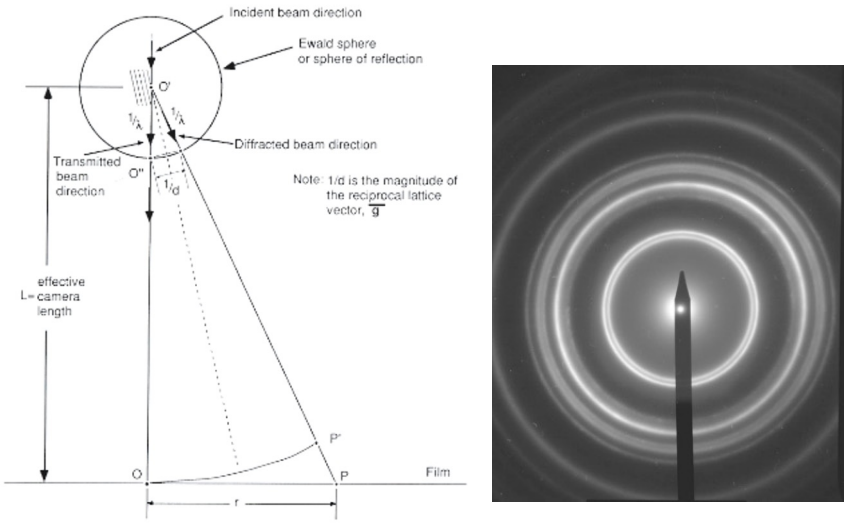

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Ring pattern: what can we obtain

- d-spacing

$$Rd_{hkl} = L\lambda$$

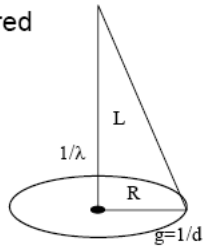
R: the measured ring radius

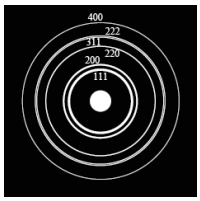
d_{hkl}: the d-spacing being measured

L: camera length

λ: wave length of electron beam

- Camera length calibration
- Crystalline / particle fineness





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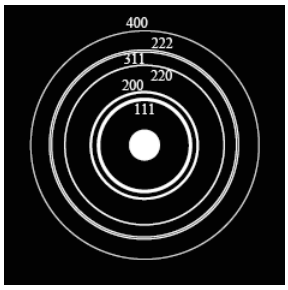
Example

Camera Length Calibration
Aluminum Polycrystalline Specimen

$$Rd_{hkl} = \lambda L$$

a_o= 0.40497 nm

$$d_{hkl} = \frac{a_o}{\sqrt{h^2 + k^2 + l^2}}$$



← R →

R_{hkl} (mm)	hkl	d_{hkl} - spacing (nm)	λL (nm-mm)
10.3	111	0.2338	2.409
11.9	200	0.2025	2.409
16.8	220	0.1432	2.406
19.7	311	0.1221	2.405
20.6	222	0.1169	2.408
		Average=	2.407 nm-mm

28

Au PolyCrystal
200 kV

R_{hkl} (pixels)	hkl	d_{hkl} - spacing (nm)	λL (nm-pixels)
130.5	111	0.2355	30.73
151	200	0.2039	30.78
213	220	0.1442	30.72
250	311	0.1230	30.75
263	222	0.1177	30.77
		Average=	30.75

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Indexing Diffraction patterns

The Ring Patterns

The radius of each ring is characteristic of the spacing of the reflecting planes in the crystal and the magnification settings of the microscope lenses.

Procedure for indexing ring patterns is as follows:

1. When the identity of the material is known, we have the following:
   - Measure the ring diameters.
   - Determine the ratios of the squares of the diameters of the outer rings to that of the first or second (low-index) ring.
   - Check the ratios against a table of ratios of the interplanar spacings for the crystal structure of interest, see table 2.2.
2. When the identity of the substance is unknown, we have the following:
   - Measure the diameter of the rings.
   - Convert the distances into interplanar spacings using the camera constant defined as Rd = λL.
   - Use ASTM (American Society for Testing Materials) index to identify the phase, starting with the most likely on the basis of the known constituents of the alloy.

Diffraction patterns from polycrystalline specimens are most commonly used either to calibrate the camera length or to identify precipitates.

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Table 2.2 Proportionalities of the ratios of the radii of ring patterns for different crystal structures

Crystal structure	Formula for interplanar spacing	Possible values of h, k, l for reflection (up to 20)	Criterion
simple cubic	$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} = \frac{N}{a^2}$	N an integer except 7 or 15	ratios of squares of radii $\propto N$
f.c.c.	$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} = \frac{N}{a^2}$	$N = 3, 4, 8, 11, 12, 16, 19, 20$	ratios $\propto N$
b.c.c.	$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} = \frac{N}{a^2}$	$N = 2, 4, 6, 8, 10, 12, 14, 16, 18, 20$	ratios $\propto N$
diamond structure	$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} = \frac{N}{a^2}$	$N = 2, 8, 11, 16, 19$	ratios $\propto N$
tetragonal	$\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2}$	$h^2 + k^2 = 1, 2, 4, 5, 8, 9, 10, 13, 16, 17, 18, 20$	ratios frequently proportional to 2; use Bunn chart, see Henry *et al.* (1951)
hexagonal	$\frac{1}{d^2} = \frac{4}{3}\frac{h^2 + hk + k^2}{a^2} + \frac{l^2}{c^2}$	$h^2 + hk + l^2 = 1, 3, 4, 7, 9, 12, 13, 16, 19$	ratios frequently proportional to 3; use Bunn chart, see Henry *et al.* (1951)

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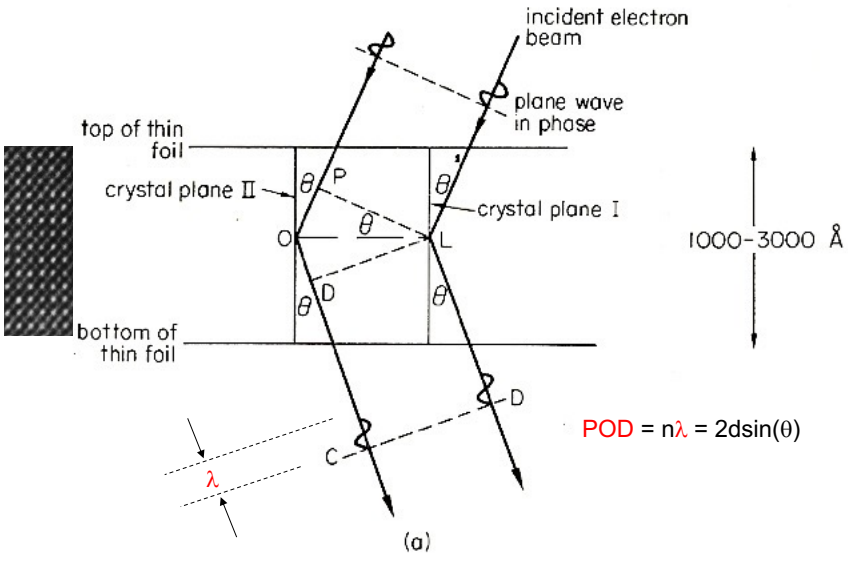
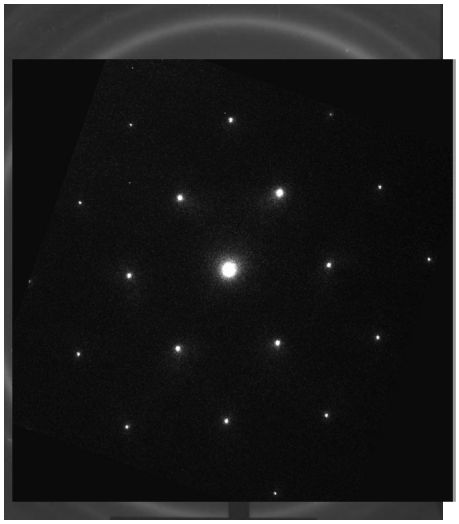
Simple Geometrical Description of Bragg's Law

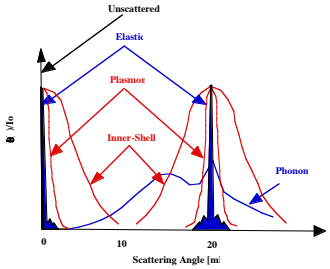


POD = $n\lambda = 2d\sin(\theta)$

32

32





Elastic Scattering : Crystalline Solids

$$I(\theta) \sim |F(hkl)|^2$$

$$F(hkl) = \sum_j f_j(\theta) \exp(-2\pi i \bar{g} \cdot \bar{r})$$

$$V(r) = -\sum_g^{?} V_g \exp(2\pi i \bar{g} \cdot \bar{r}) \quad ; \quad V_g = \frac{h^2}{2\pi m V_c} f(\theta)$$

Index Simple Patterns – spots are produced by planes in one zone

- Frequently these patterns may be easily recognized from their simplicity and their six-, four-, three- and two-fold symmetry.

(a) *Indexing the pattern*. The indexing procedure involves one of two procedures:

(i) Camera constant method –Camera constant known, materials known. Measure the distance R of the spot from the center spot, figure 2.20 (a) (most accurately determined by measuring the total distance between several spots in this direction and dividing as necessary). Divide the camera constant λL (Rd = λL) by R and check the result against a list of d spacings.

(ii) Method of ratios. Camera constant unknown. Material λL known. Measure distances of spots from center spot relative to that between the nearest spot and the center. Check against tables of ratios of d spacings for low-index planes. Account must be taken of the occurrence of planes with the same d spacing, see table A 4.1.

Silicon Diffraction Patterns
CL = 470 mm

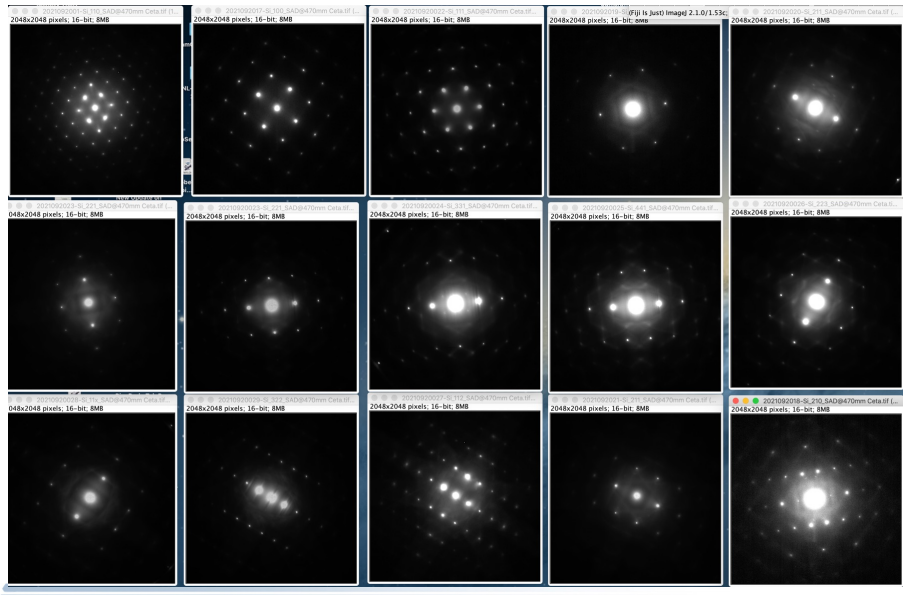


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Table A4.1 Occurrence of reflections for the cubic crystal structures

Line no. $N = h^2+k^2+l^2$	hkl indices	$N^{1/2} = (h^2+k^2+l^2)^{1/2}$	f.c.c.	diamond		Line no. $N = h^2+k^2+l^2$	hkl indices	$N^{1/2} = (h^2+k^2+l^2)^{1/2}$	b.c.c.	f.c.c.	diamond
1	100	1.00				33	522, 441	5.745			
2	110	1.414	×			34	530, 433	5.831	×		
3	111	1.732		×	×	35	531	5.916		×	×
4	200	2.00	×	×	×	36	600, 442	6.00	×	×	×
5	210	2.236				37	610	6.083			
6	211	2.450	×			38	611, 532	6.164	×		
7	—	—				39	—	—			
8	220	2.828	×	×	×	40	620	6.325	×	×	×
9	300, 221	3.00				41	621, 540, 443	6.403			
10	310	3.162	×			42	541	6.481	×		
11	311	3.317		×	×	43	533	6.557		×	×
12	222	3.464	×	×		44	622	6.633	×	×	×
13	320	3.606				45	630, 542	6.708			
14	321	3.742	×			46	631	6.782	×		
15	—	—				47	—	—			
16	400	4.00	×	×	×	48	444	6.928	×	×	
17	410, 322	4.123				49	700, 632	7.00			
18	411, 330	4.243	×			50	710, 550, 543	7.071	×		
19	331	4.359		×	×	51	711, 551	7.141		×	×
20	420	4.472	×	×	×	52	640	7.211	×	×	
21	421	4.583				53	720, 641	7.280			
22	332	4.690	×			54	721, 633, 552	7.349	×		
23	—	—				55	—	—			
24	422	4.899	×	×	×	56	642	7.483	×	×	×
25	500, 430	5.00				57	722, 544	7.550			
26	510, 431	5.099	×			58	730	7.616	×		
27	511, 333	5.196		×	×	59	731, 553	7.681		×	×
28	—	—				60	—	—			
29	520, 432	5.385				61	650, 643	7.810			
30	521	5.477	×			62	732, 651	7.874	×		
31	—	—				63	—	—			
32	440	5.657	×	×	×	64	800	8.00	×	×	×

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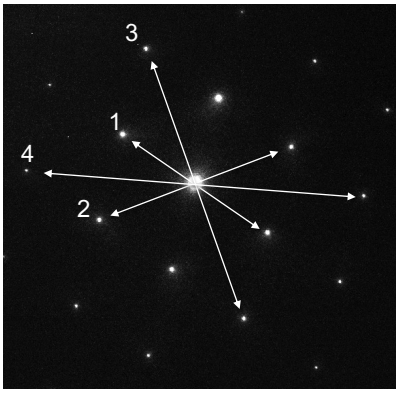
Indexing using d-spacing Ratios

	R_i	R_i/R_1	Allowed hkl	$\frac{\sqrt{h_i^2+k_i^2+l_i^2}}{\sqrt{h_0^2+k_0^2+l_0^2}}$
1	130.6	1	111	1
2	154.0	1.18	200	1.15
3	213.0	1.64	220	1.63
4	253.2	1.93	311	1.92



Si {110}

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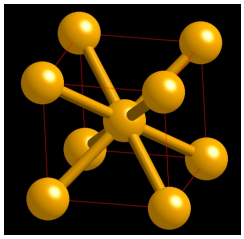

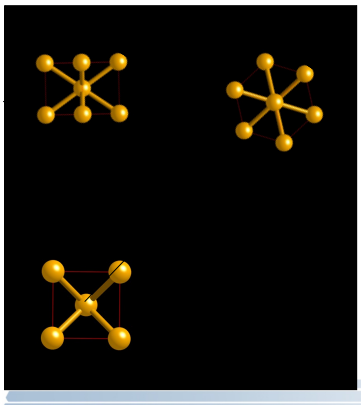

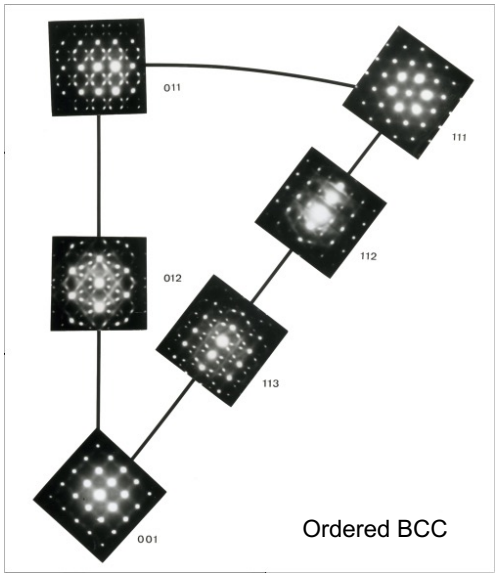

Ordered BCC

Diffraction Space is related to Real Space

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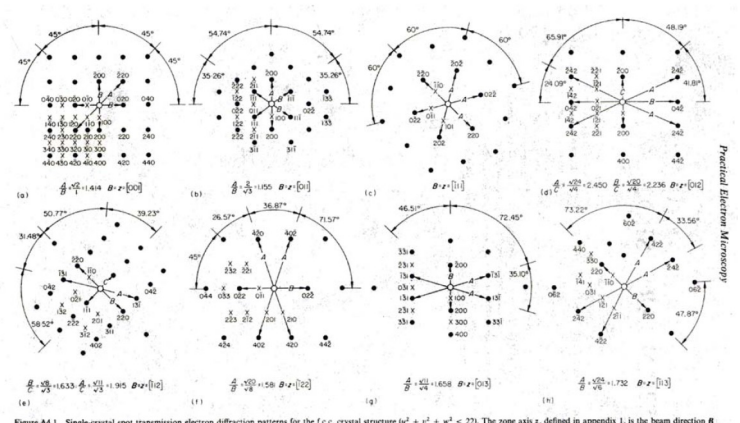

Figure A4.1 Single-crystal spot transmission electron diffraction patterns for the f.c.c. crystal structure ($u^2 + v^2 + w^2 < 22$). The zone axis z, defined in appendix 1, is the beam direction **B** defined in section 2.7.2, as indicated. The crosses in one quadrant of the diagram indicate the positions of the spots for the ordered f.c.c. ($L1_2$) unit cell. The complete pattern may be generated by repeating these spots in the remainder of the pattern and indexing using the addition of vectors, see section 2.7.2.1.

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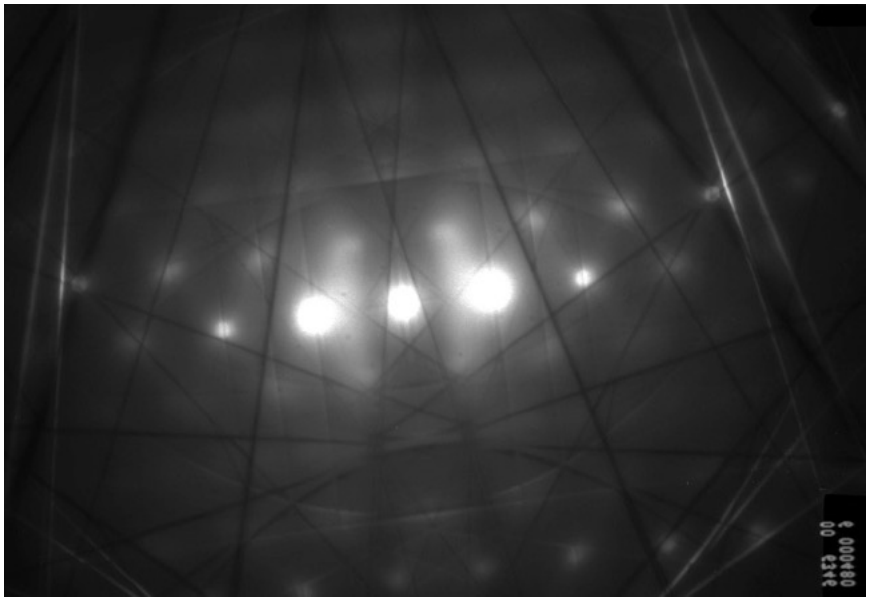
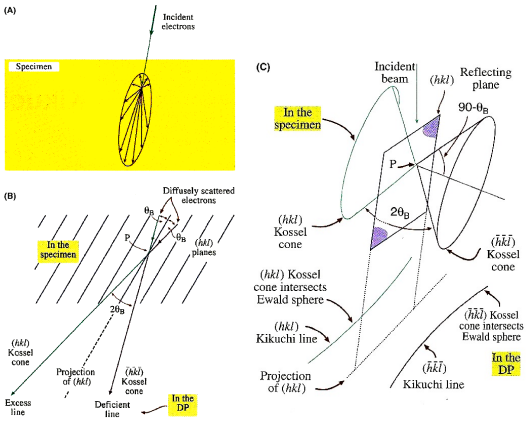
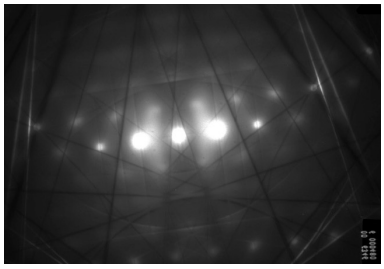
Kikuchi Lines

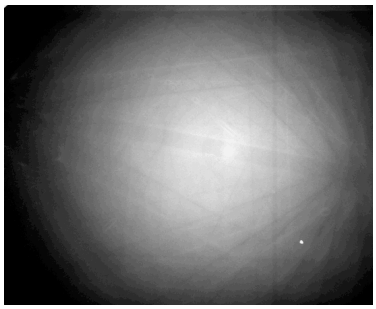


40

Kikuchi Lines



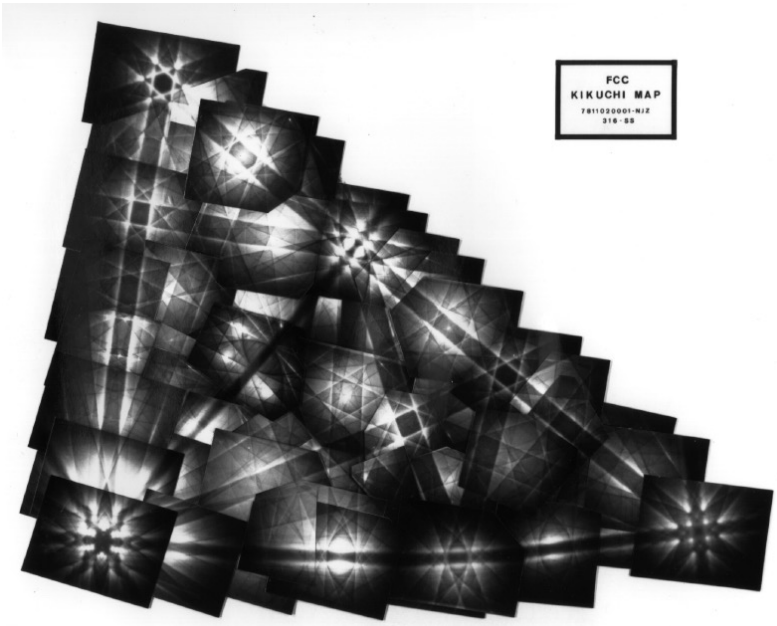




- Inelastically scattered electrons are Bragg Diffrated by lattice planes
- Range of orientation of scattered electrons gives rise to two cones of Diffraction
- Projection of the cones creates a “pair” of Kikuchi lines

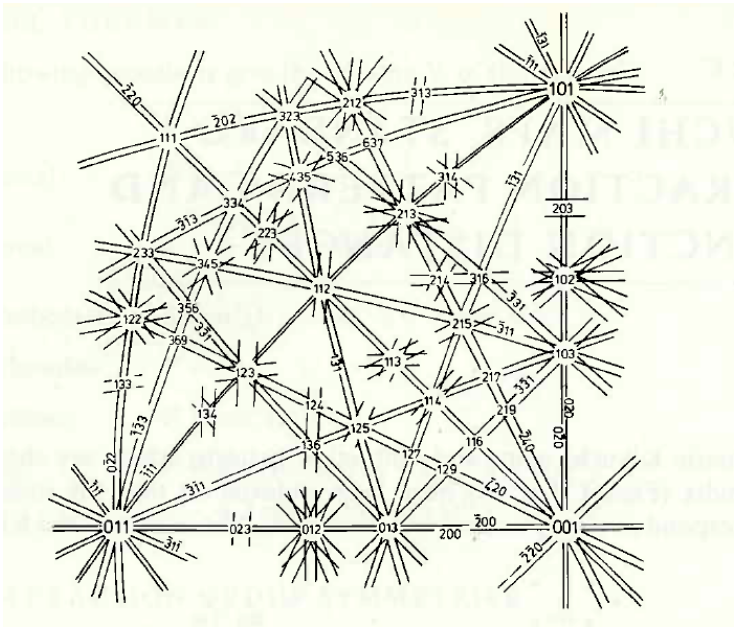
41

41



42

42



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Single-title holder
Double-tilt holder
Low-background holder
Heating holder
Cooling holder
Cryo-transfer holder

Specimen Holders

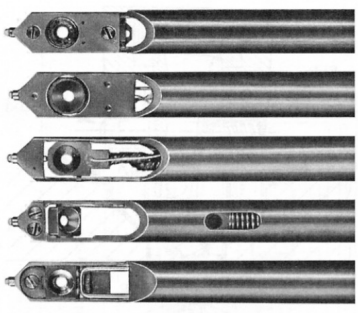

Figure 8.8. Examples of different designs for the side-entry holder. From the top, they are: a rotation holder, a heating holder, a cooling holder, a double-tilt holder, and a single-tilt holder.

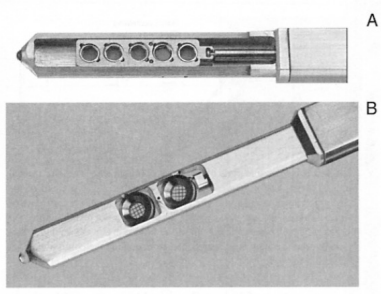

Figure 8.9. Multiple-specimen holders. (A) Five-specimen single-tilt and (B) two-specimen double-tilt.

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How do Apertures & Condensor Lens affect Diffraction

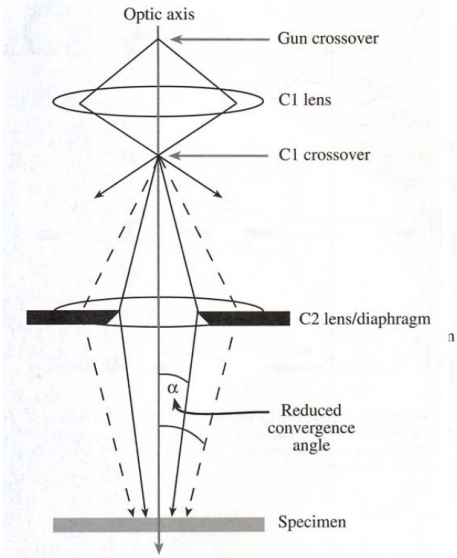

Figure 9.2. Effect of the C2 aperture on the beam coherence: a smaller aperture creates a more parallel, more coherent beam.

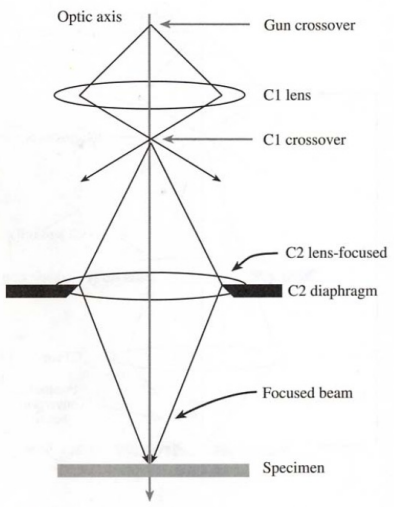

Figure 9.3. A focused C2 lens illuminates a small area of the specimen with a nonparallel beam.

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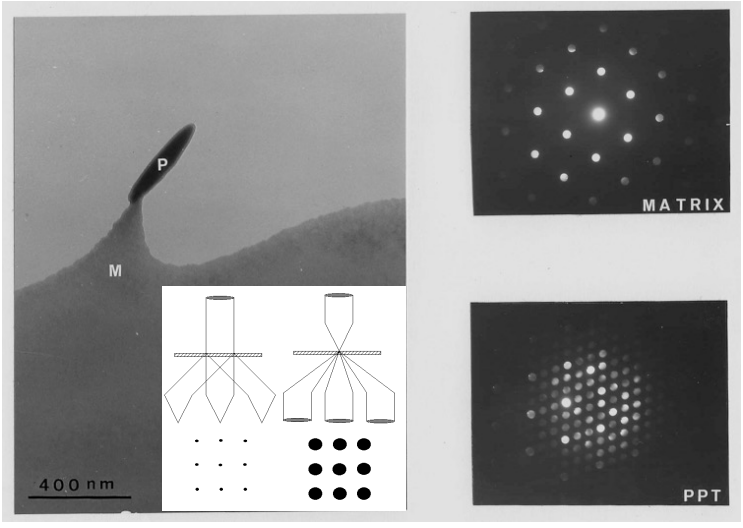
45

Convergent/Parallel Beam Electron Diffraction



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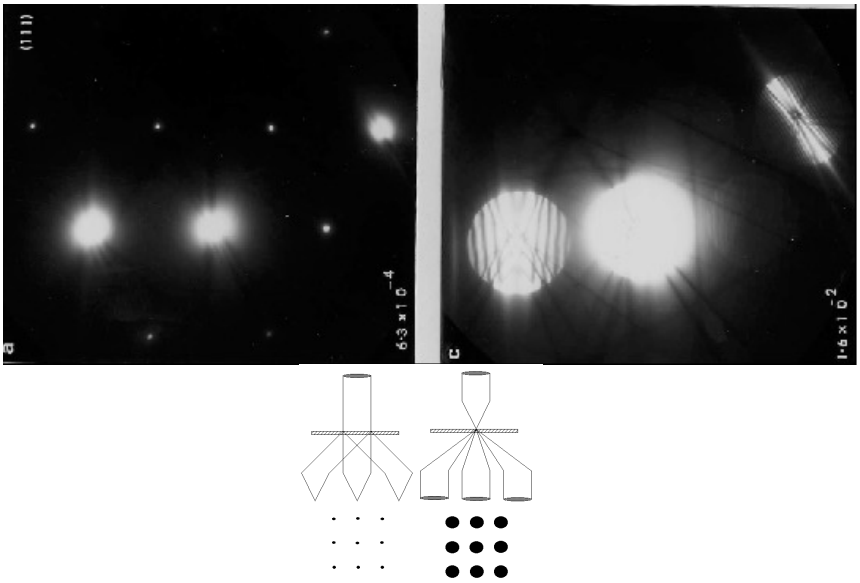
Nearly Parallel | Non-Parallel / Convergent Beam



47

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Obtaining ~ Parallel Illumination

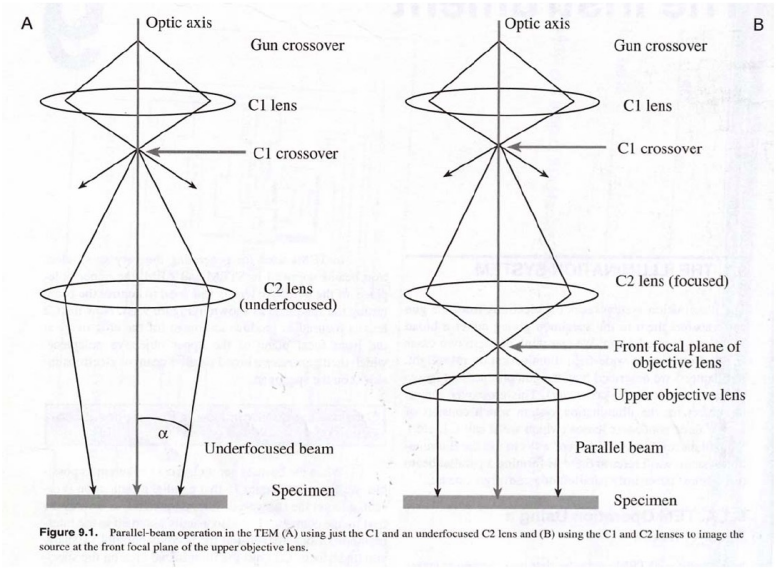

Figure 9.1. Parallel-beam operation in the TEM (A) using just the C1 and an underfocused C2 lens and (B) using the C1 and C2 lenses to image the source at the front focal plane of the upper objective lens.

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Place an aperture in the Image Plane this defines an area back projected onto the specimen

Selected-Area Diffraction

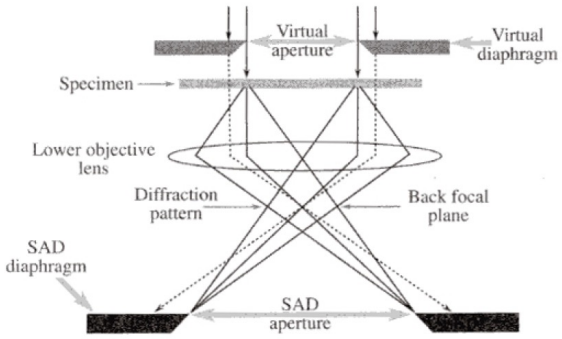

Figure 9.13. Ray diagram showing SAD pattern formation: the insertion of an aperture in the image plane results in the creation of a virtual aperture in the plane of the specimen. Only electrons falling inside the dimensions of the virtual aperture at the specimen will be allowed through into the imaging system. All other electrons will hit the SAD diaphragm.

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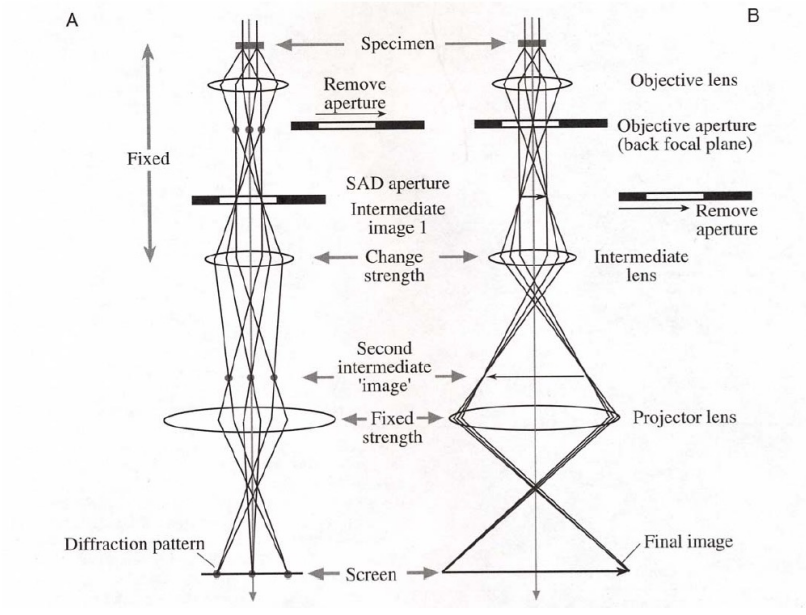

50

Why CBED? Why not SAD?

Limits of Conventional SAD

Conventional SAD uses an aperture to define the area from which the pattern is to be recorded. The aperture is placed in the image plane of the objective lens to create a virtual aperture in the specimen plane (Le Poole 1947). The spatial resolution in SAD is limited by both spherical aberration and the ability of the operator to focus the aperture of the and the image in the same plane. The error in area selection U is given by:

$$U = C_s(2\theta_B)^3 + D2\theta_B$$

where: C_s *= spherical aberration coefficient*

θ_B *= Bragg angle*

D= minimum focus step.

The result is that the theoretical lower limit of area selection is ~0.5μm (in practice governed by aperture size).

J.B. Le Poole, Philips Tech. Rundsch 9 (1947) 33.

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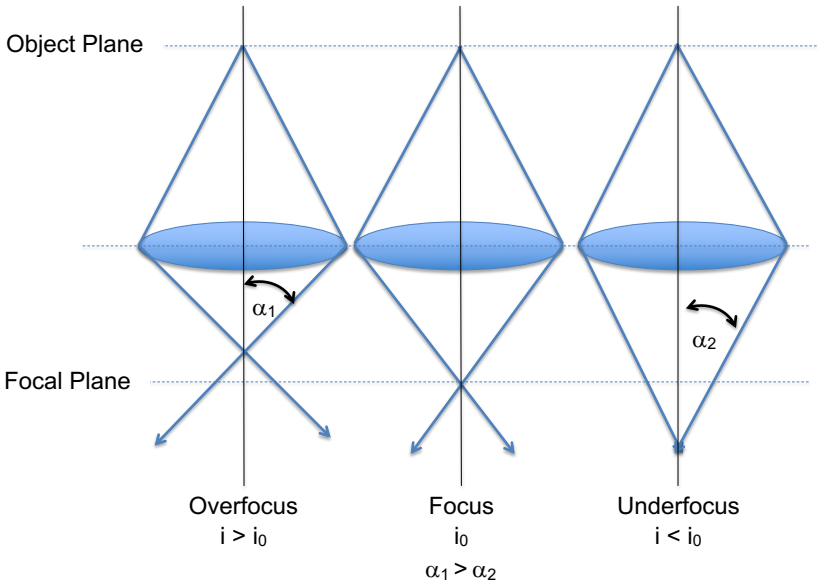
51



Underfocus is more “parallel” than Overfocus

52

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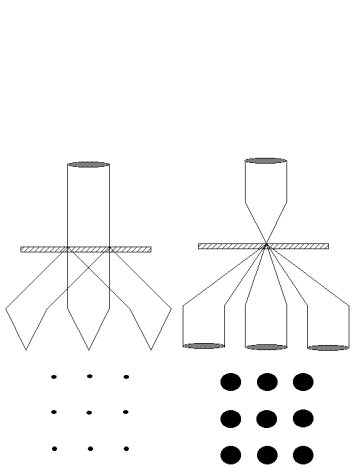

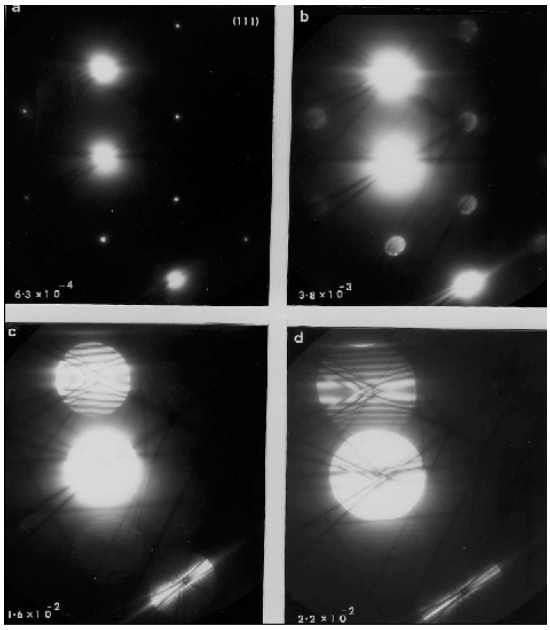

Notice, loss of definition of weak reflections !

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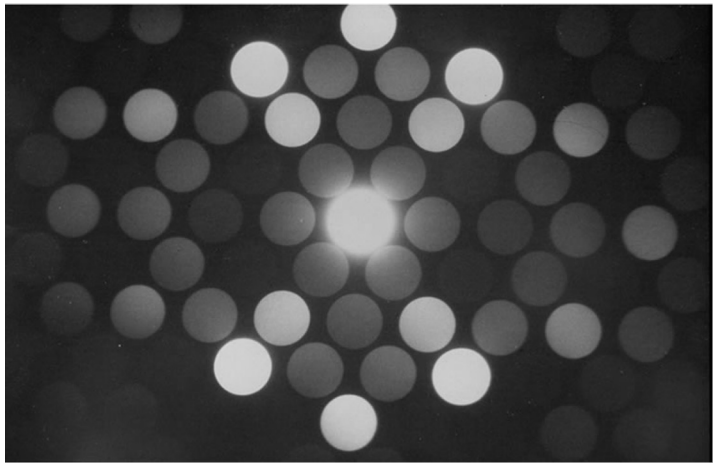
$M_{23}C_6$ [110]



Eades / Bristol Group

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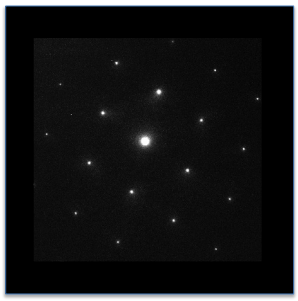
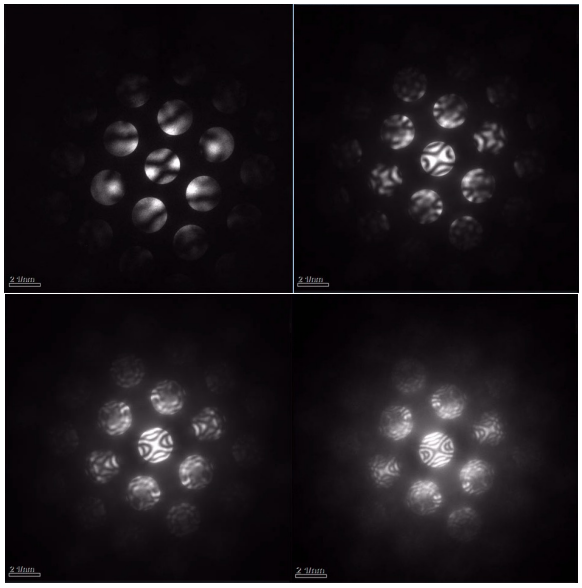
Silicon
{110}

Structure in CBED patterns is Dependant upon Extinction/Thickness





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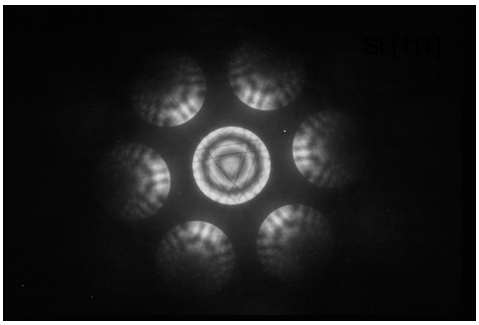
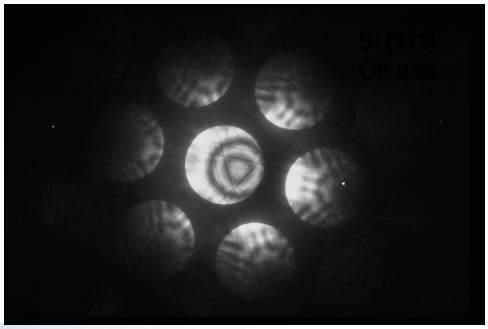


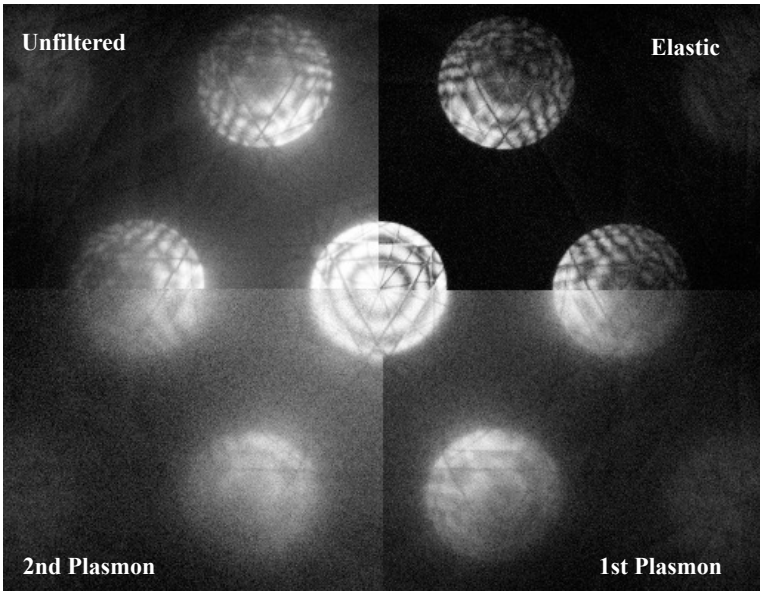
CBED is extremely sensitive to orientation and is the best way to set the exact orientation



56

56

Silicon 111 ZAP- Energy Filtered



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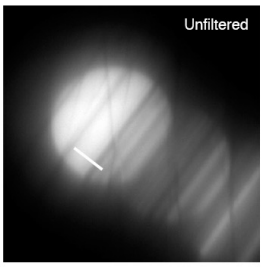
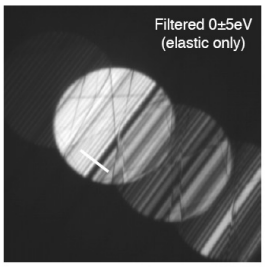
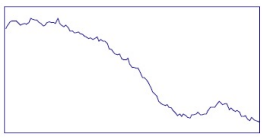
Thick Specimens needed for Kikuchi Patterns
and Intensity oscillations in the CBED disks
But…. this also introduces diffuse scattering

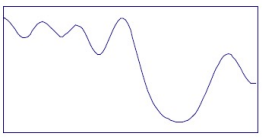


Silicon [110]





essential for
quantitative CBED



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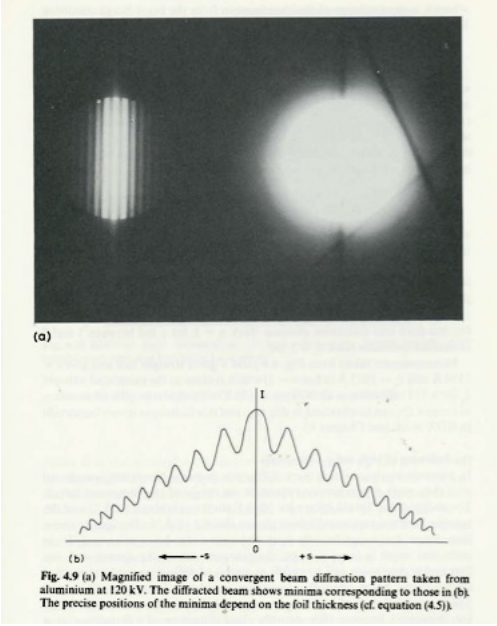

Fig. 4.9 (a) Magnified image of a convergent beam diffraction pattern taken from aluminium at 120 kV. The diffracted beam shows minima corresponding to those in (b). The precise positions of the minima depend on the foil thickness (cf. equation (4.5)).

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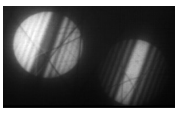
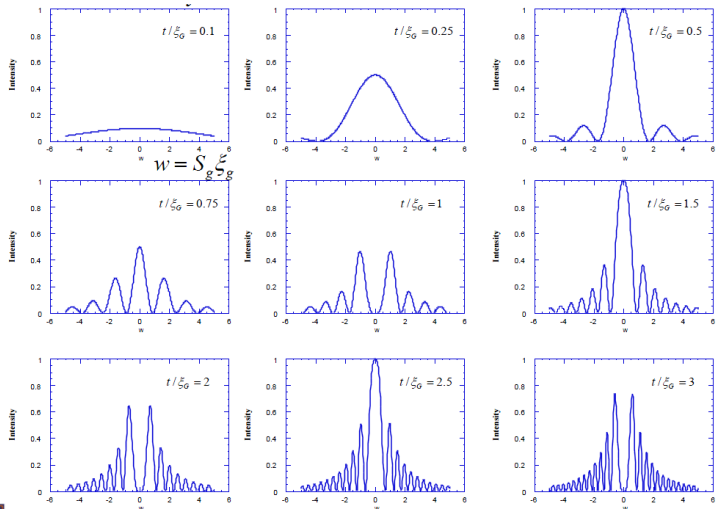


Calculated Intensity in Diffraction Disk
as a function of relative thickness
under two Beam Dynamical Conditions



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Theoretical Considerations for Thickness Measurement

Convergent beam diffraction discs are maps of diffracted intensity as a function of incident wave angle and therefore have a direct correspondence to a rocking curve. In the two-beam approximation the rocking curve for the diffracted intensity $|\phi|^2$ is given by (Hirsch et al. 1965):

$$|\phi|^2 = \sin^2 \beta\pi\Delta kz \qquad (1)$$

Where:

$$\beta = \tan^{-1}\left(\frac{1}{s\xi_g}\right) \quad \Delta k = \frac{\sqrt{1+(s\xi_g)^2}}{\xi_g}$$

s is the deviation parameter, ξ_g is the extinction distance and **z** the foil thickness.

"Electron Microscopy of Thin Crystals", Hirsch et al (1965).

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Differentiation with respect to s reveals that the minima of $|\phi|^2$ in equation (1) obey the relationship:

$$\Delta kz = \text{integer} \qquad (2a)$$

and the maxima obey the relationship:

$$\tan \pi\Delta kz = \pi\Delta kz \qquad (2b)$$

Also, s=0 is always either a maximum or minimum. Kelly et al. (1975) expressed equation (2a) as:

$$\left(\frac{s_i}{n_k}\right)^2 = -\left(\frac{1}{\xi}\right)^2\left(\frac{1}{n_k}\right)^2 + \left(\frac{1}{z}\right)^2 \qquad (3a)$$

It is evident that a plot of $\left(\frac{s_i}{n_k}\right)^2$ against $\left(\frac{1}{n_k}\right)^2$ in a two-beam condition yields a straight line with intercept $\left(\frac{1}{z}\right)^2$ and slope of $\left(\frac{1}{\xi}\right)^2$. This is the basis of the CBED thickness measurement technique that is now well known.

P.M. Kelly et al., Phys. Stat. Sol. (1975)A31, 771.

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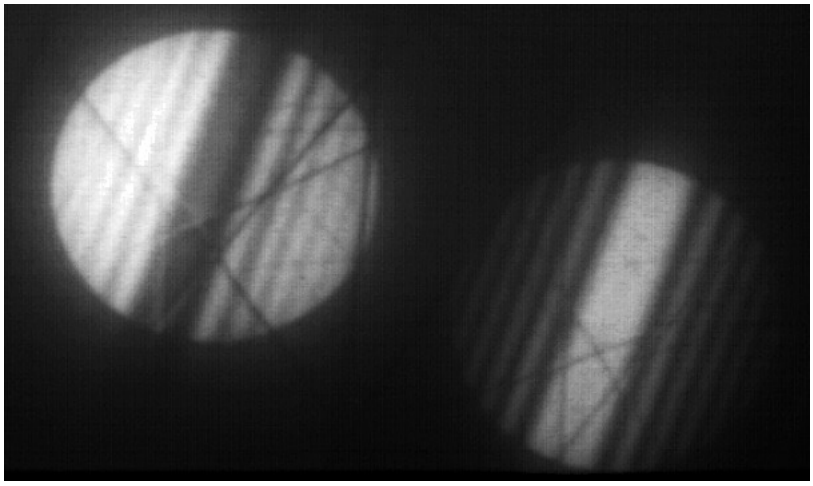

Si <220> @ 200 kV
a=0.543 nm
t~ 280 nm ξ ~ 125 nm

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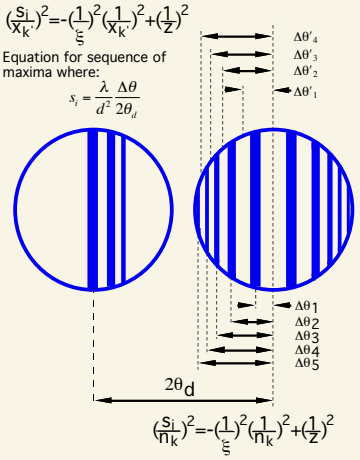
$$\left(\frac{S_i}{x_{k'}}\right)^2=-\left(\frac{1}{\xi}\right)^2\left(\frac{1}{x_{k'}}\right)^2+\left(\frac{1}{z}\right)^2$$

Equation for sequence of maxima where:

$$s_i=\frac{\lambda}{d^2}\frac{\Delta\theta}{2\theta_d}$$



$$\left(\frac{S_i}{n_k}\right)^2=-\left(\frac{1}{\xi}\right)^2\left(\frac{1}{n_k}\right)^2+\left(\frac{1}{z}\right)^2$$

Equation for sequence of minima where:

$$s_i=\frac{\lambda}{d^2}\frac{\Delta\theta}{2\theta_d}$$

$$\left(\frac{S_i}{n_k}\right)^2=-\left(\frac{1}{\xi}\right)^2\left(\frac{1}{n_k}\right)^2+\left(\frac{1}{z}\right)^2$$

Points for minima open squares

Intercept $\frac{1}{z^2}$

Slope $=-\frac{1}{\xi^2}$

$$\left(\frac{S_i}{x_{k'}}\right)^2=-\left(\frac{1}{\xi}\right)^2\left(\frac{1}{x_{k'}}\right)^2+\left(\frac{1}{z}\right)^2$$

Points for the maxima filled circles

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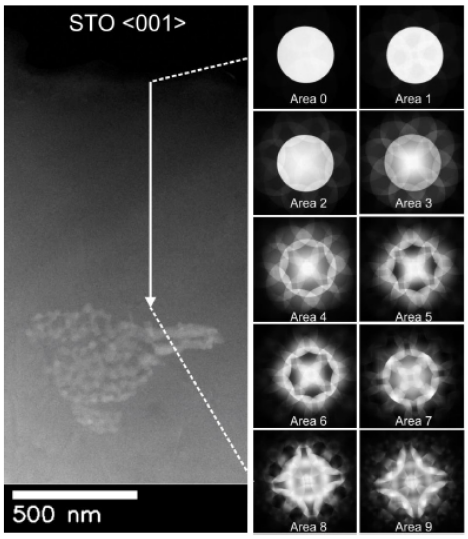
Position averaged CBED (PACBED) – crystal thickness measurement

- PACBED is a simple technique for crystal thickness measurement
- Scan over more than one unit cell
- Acquire a CBED pattern while doing so – this will average the pattern over an entire unit cell
- This replicates a large probe which averages over many unit cells – yet retains the overlaps from intermediate convergence angle
- Energy filter if you can, this will make things sharper

Generally done in STEM mode as you scan over an area



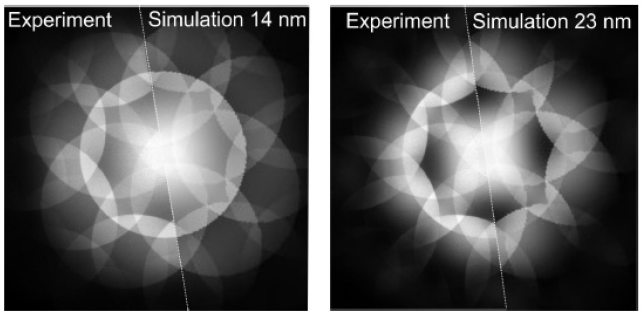
A specimen of STO – and a series of experimental PACBED patterns from increasing thickness

Le Beau et al, Ultramicroscopy 110, p118 (2010)

65

Semi-quantitative measurement of thickness

- Compare simulated patterns with experimental patterns via visual inspection
- Simulation of incoherent patterns is fast (100 DP's can take a few minutes)
- Good packages for this include JEMS and μSTEM*
- Accuracy is on the order of 1 nm or so for intermediate thicknesses



Comparison between two experimental and simulated PACBED patterns

* http://tcmp.ph.unimelb.edu.au/mustem/muSTEM.html

But only measures Crystalline Thickness

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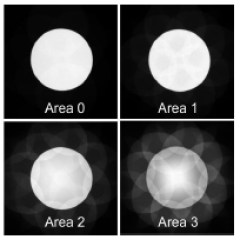
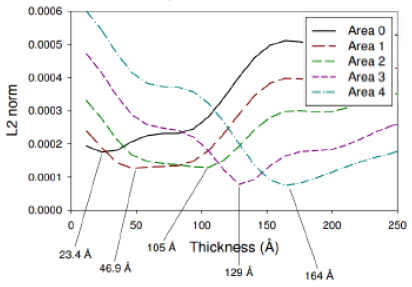
Quantitative measure of crystal thickness…

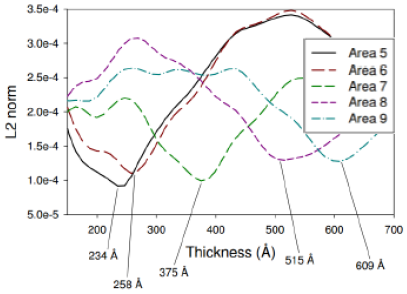
Matching PACBED's by eye becomes challenging for thin crystals

Currently matches are purely subjective

Least squares match (L2 norm) made to simulated PACBED patterns allows objective, repeatable thickness determination







L2 norm match to simulations of experimental PACBED patterns, curve minima indicates most likely match

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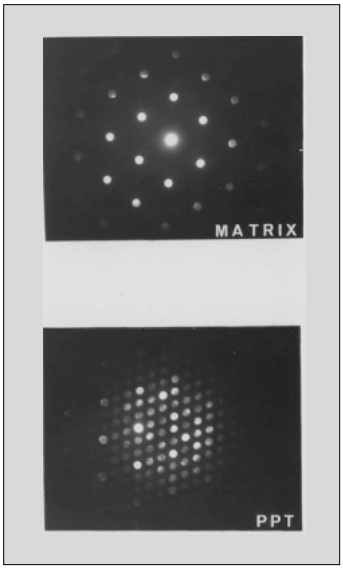
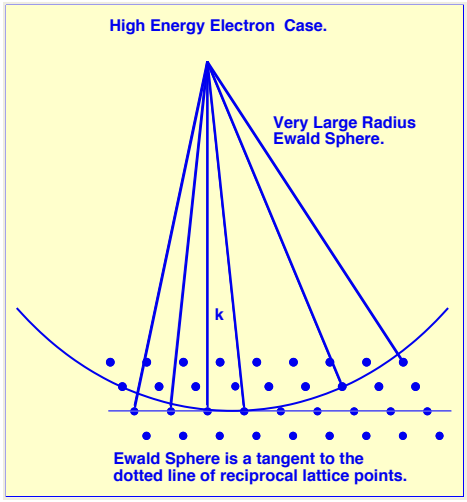


Diffracted Intensity Falls off with angle because of Ewald Sphere



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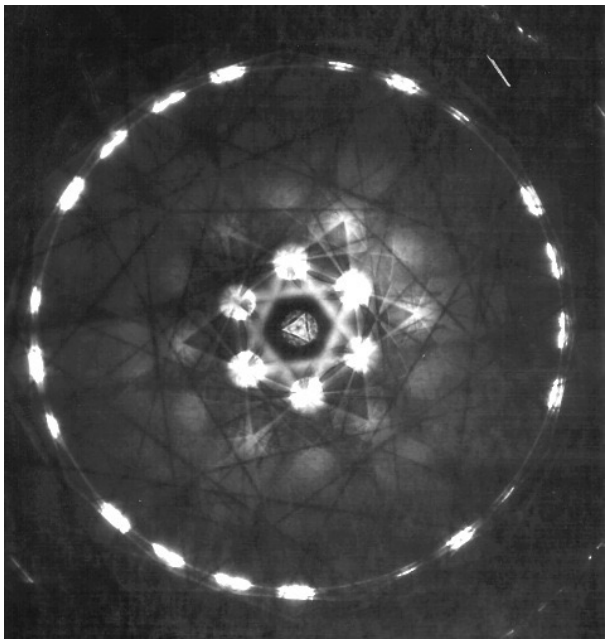
68



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Laue Zones

- At a zone-axis orientation, the reflections in the diffraction pattern break up into zones called Laue zones
- The central zone is called the zero-order Laue zone
- The first ring is called the first-order Laue zone - and so on
- The first-order, second-order, third order (and so on) are known collectively as the higher-order Laue zones

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HOLZ

- HOLZ is the acronym for higher-order Laue zone
- The rings of reflections outside the central, zero-order Laue zone are the HOLZ
- Because the narrow, dark, straight lines in the bright field disc are associated with diffraction into a HOLZ reflection, they are known as HOLZ lines
- Do not confuse HOLZ with HOLZ lines

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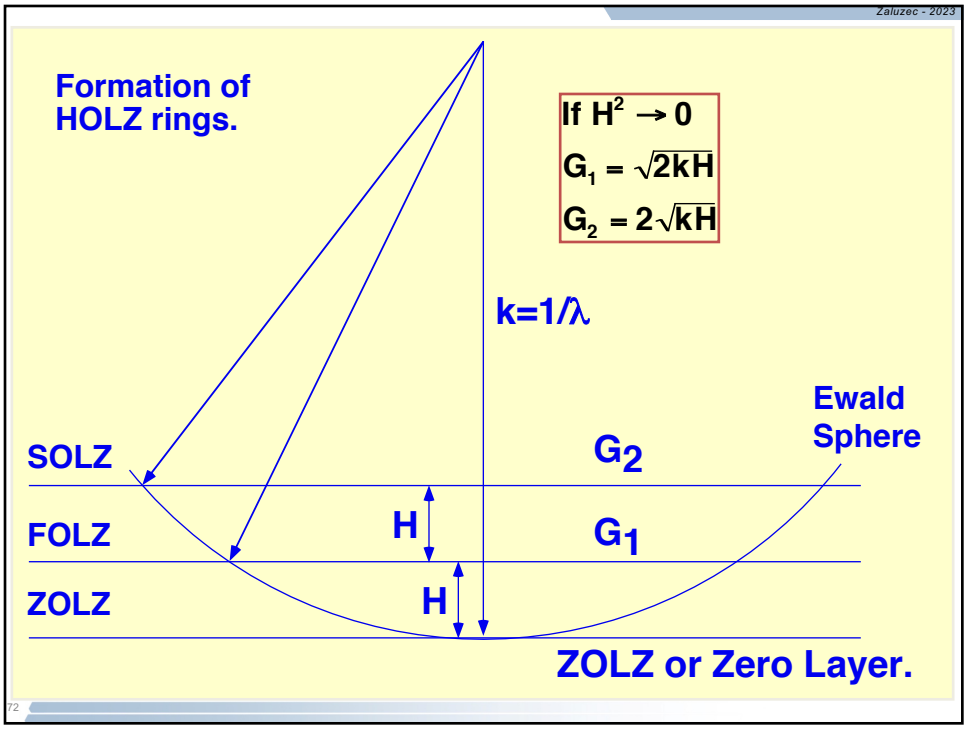

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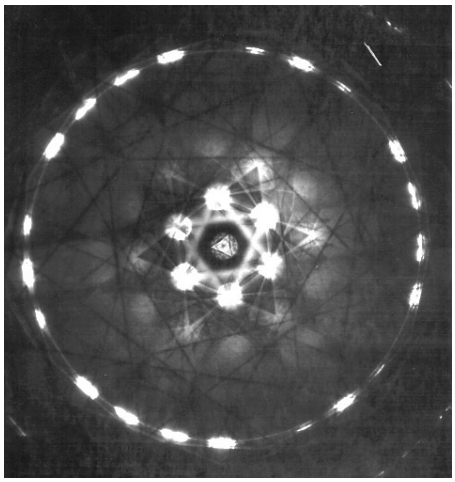
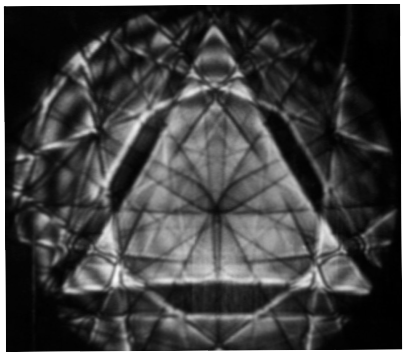
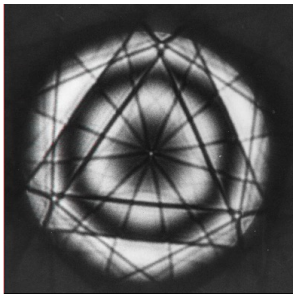
Details of CBED Holz lines are sensitive
to both crystal structure and operating conditions

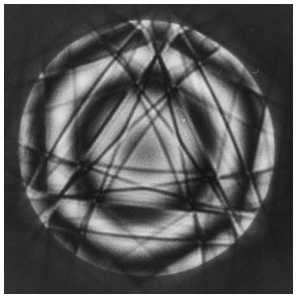




73

Details of CBED Holz lines are sensitive
to both crystal structure and operating conditions





113 kV

100 kV

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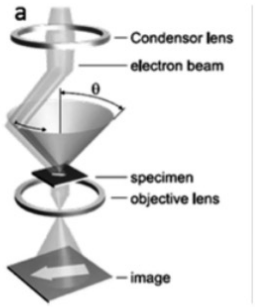
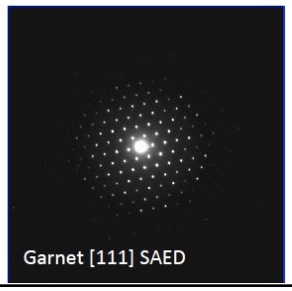
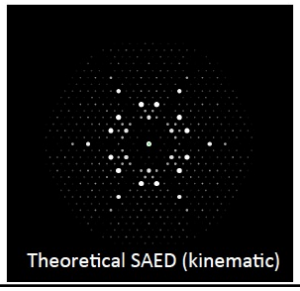
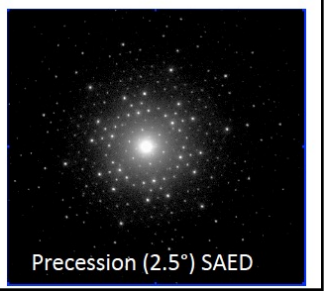
Precession diffraction

- SAED patterns are difficult for automated indexing
  - Dynamical diffraction
- Precession patterns are more kinematical
  - Can use diffraction intensities.
- Deflection coils used to tilt and rotate the beam in a cone with apex in the sample
  - Precession angle from ~ 0.5° to 3°




Garnet [111] SAED


Theoretical SAED (kinematic)


Precession (2.5°) SAED

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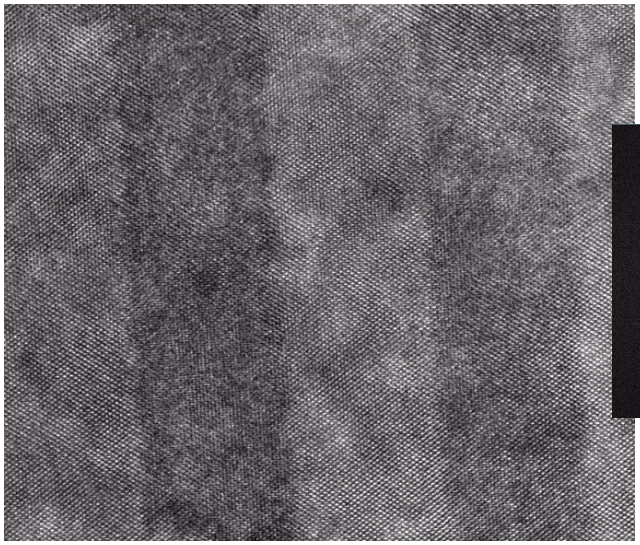
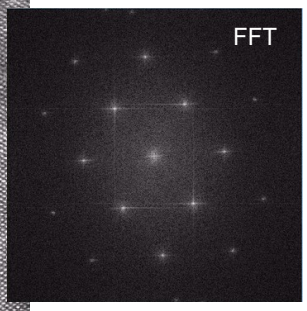
*FFT of an Image is **NOT** a Diffraction Pattern !!!*





It is a Power Spectrum of the frequencies in the Image

Si/Ge MultiLayer

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