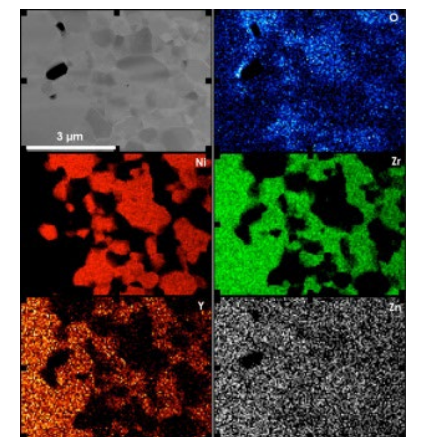
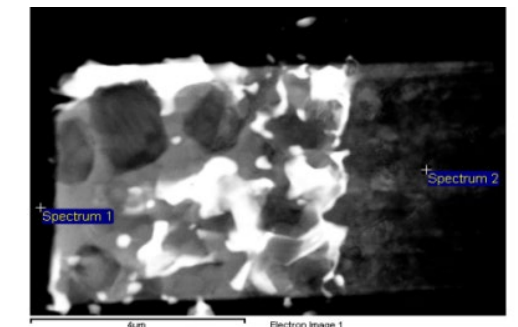
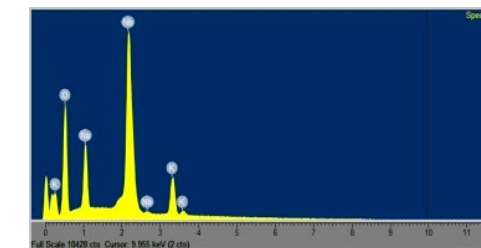
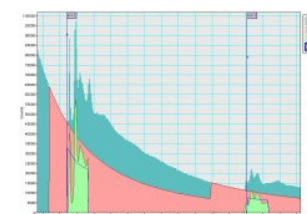
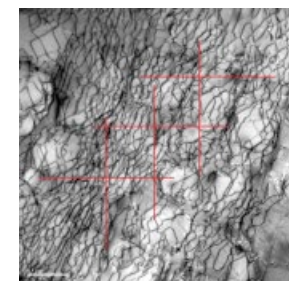
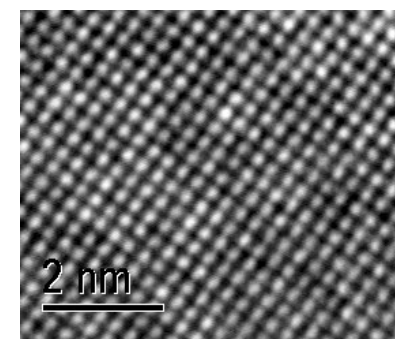
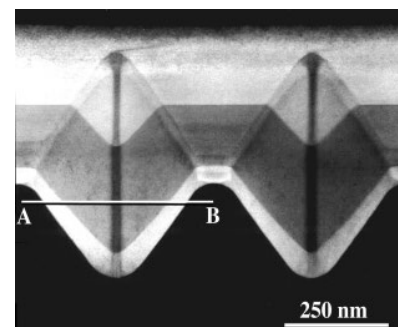
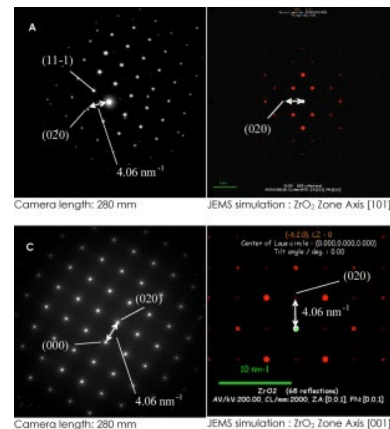
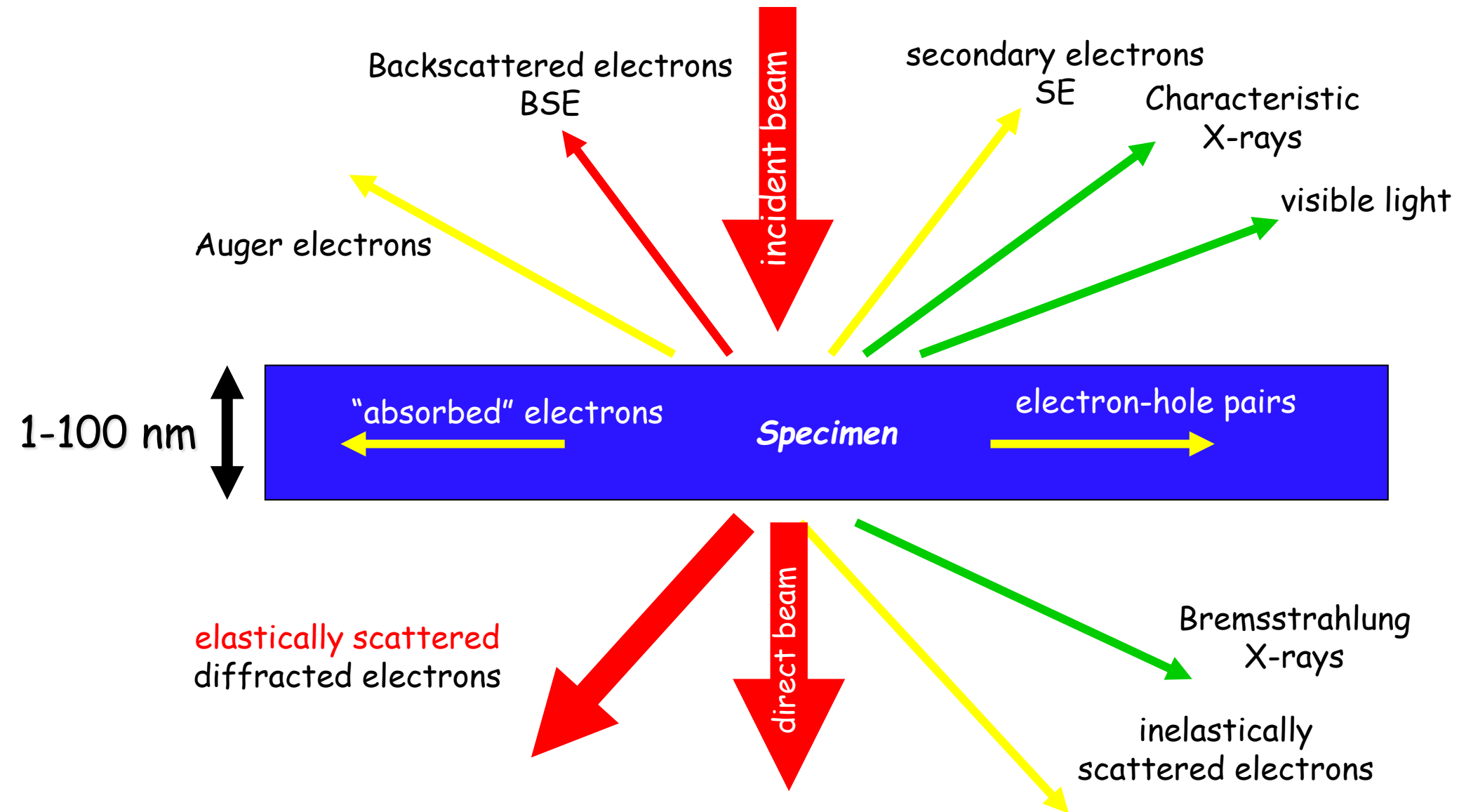


9. Diffraction

Marco Cantoni

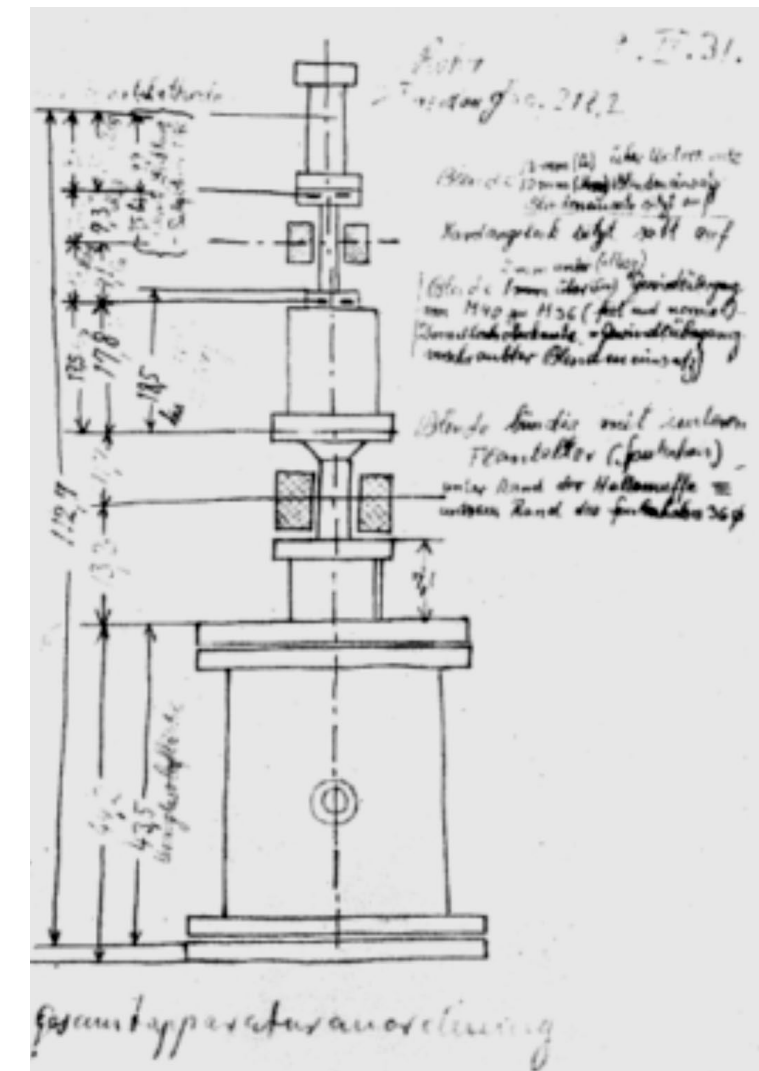
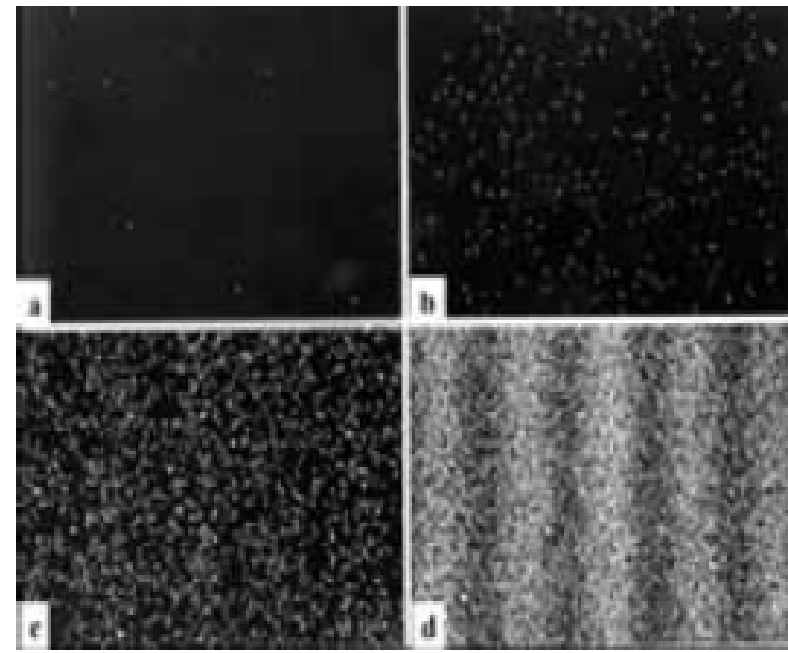
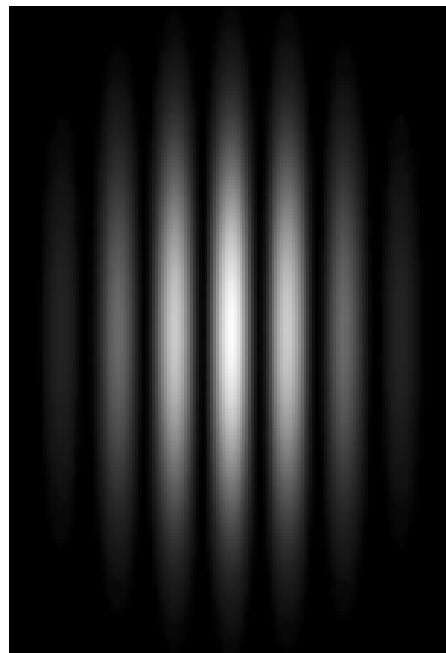
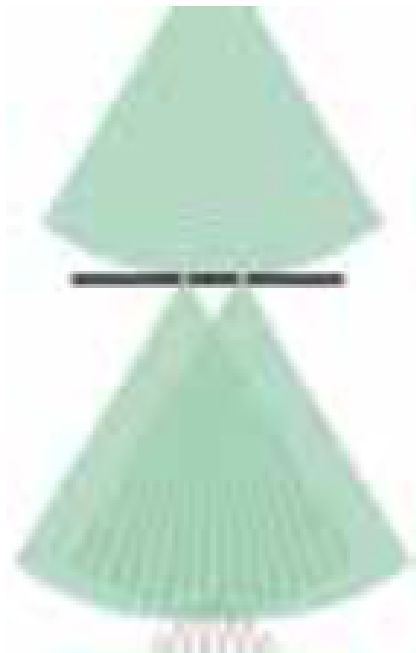
Centre Interdisciplinaire de Microscopie Electronique
CIME

Signals from a thin sample

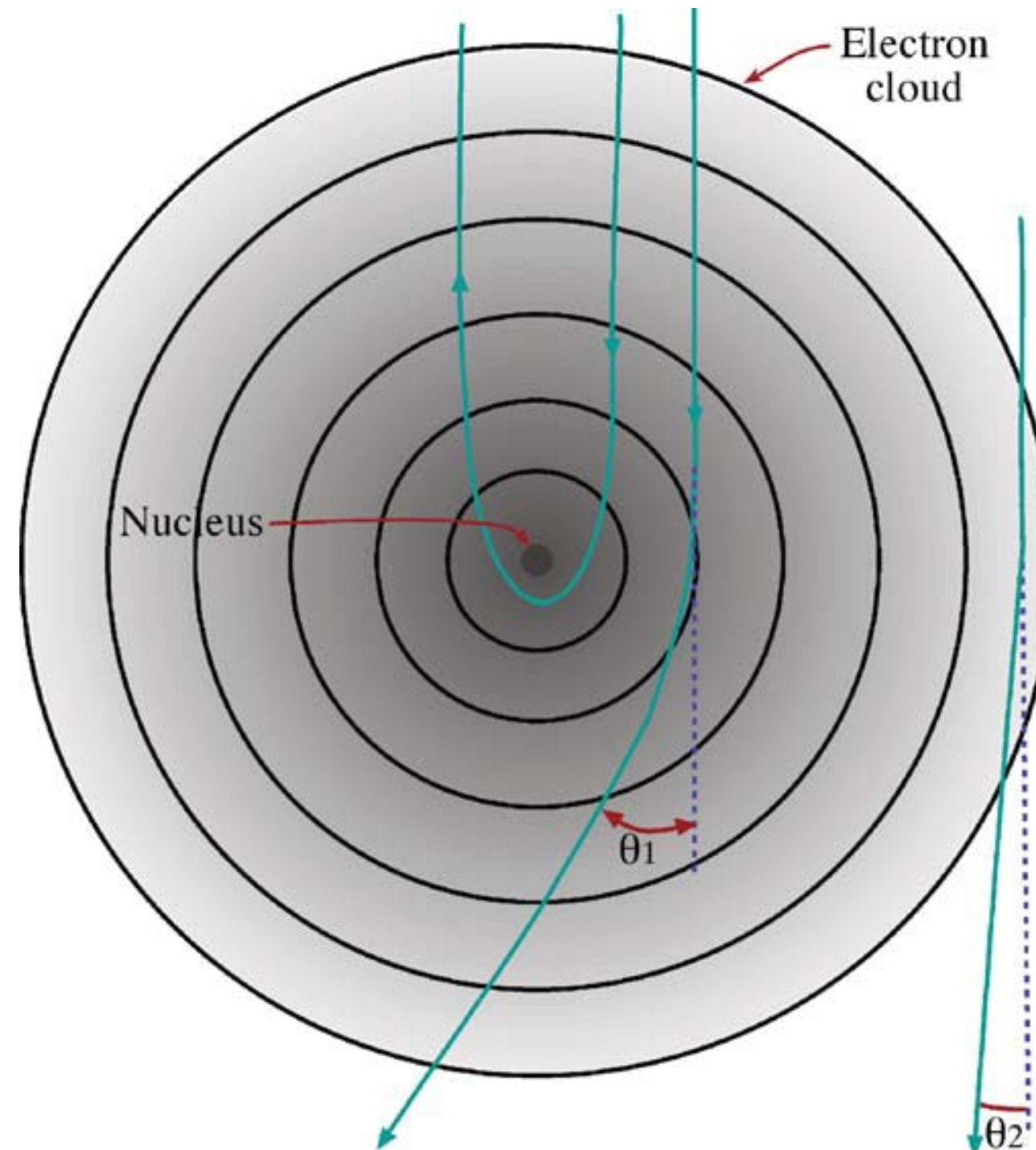


History

- 1897: J.J. Thomson: predicts the existence of electrons
- 1923: De Broglie: concept of **wavelength associated to particles**, confirmation by Young's experiment
- 1927: Busch: focalisation law for magnetic fields, Davisson, Gremer, Thomson: electron diffraction
- 1931: Ruska, Knoll: first images by electron microscope



scattering of electrons by atom(s)

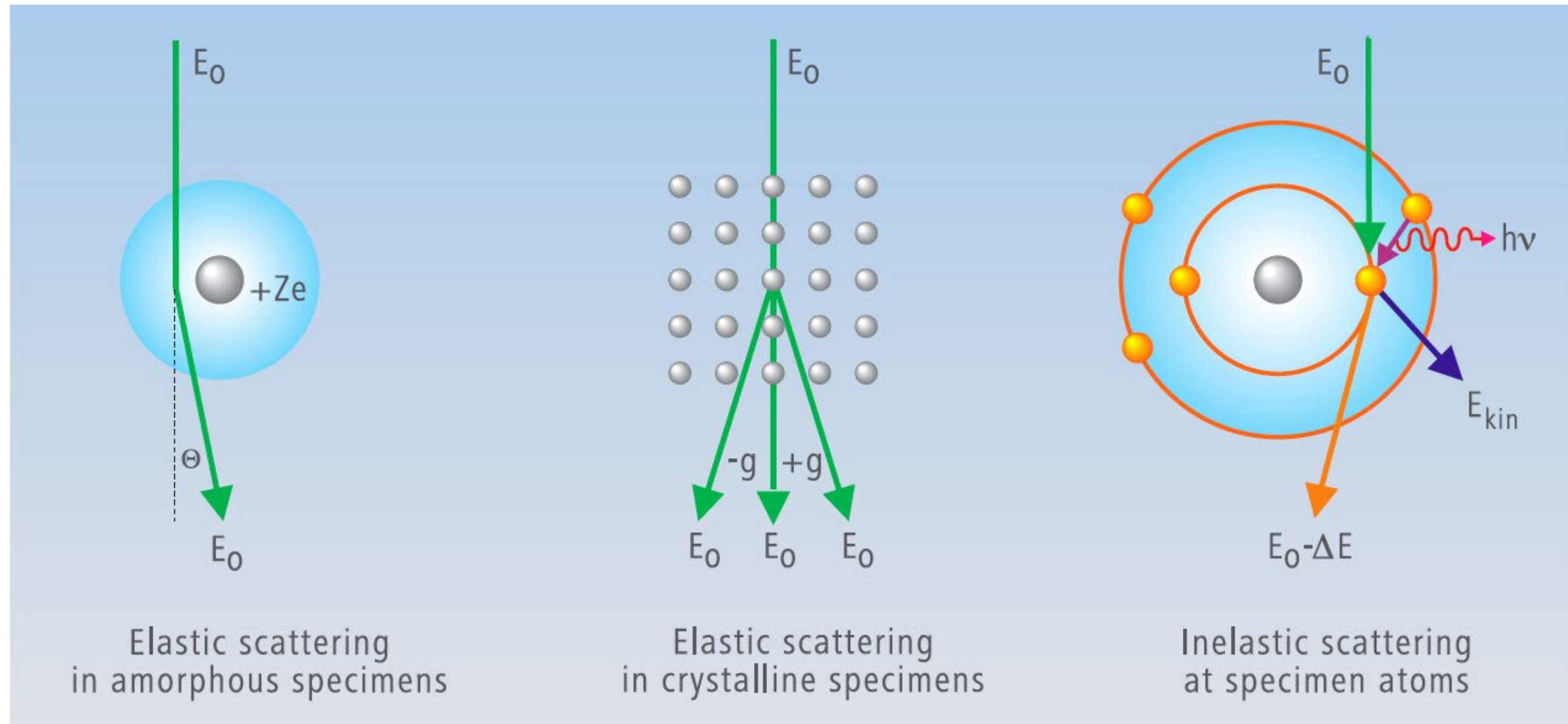


Coulombic interaction with electron cloud:
Low angle scattering (coherent)

Coulombic interaction with nucleus:
high angle scattering (incoherent)

Williams and Carter:
Transmission electron microscopy
Chapter 3: elastic scattering

interaction of high energetic electrons with matter



TEM of biological samples, polymers
STEM

HR-STEM of crystalline samples

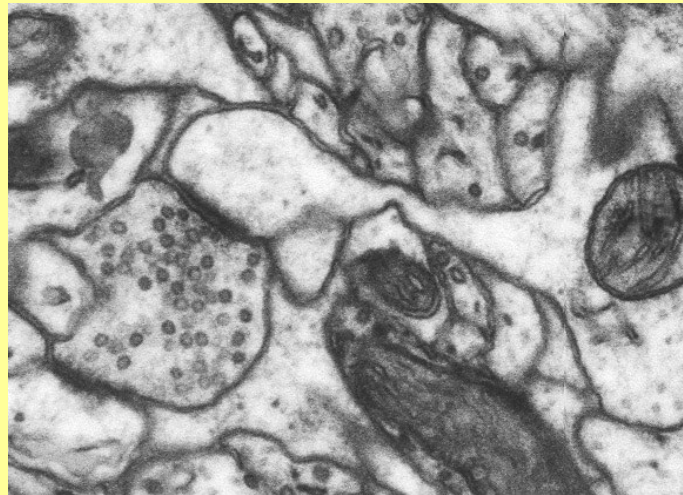
incoherent (e^- "particle")
high angle, Rutherford type

crystalline structure,
defect analysis,
high-resolution TEM
coherent (e^- "wave")
low angle, Huygens
principle

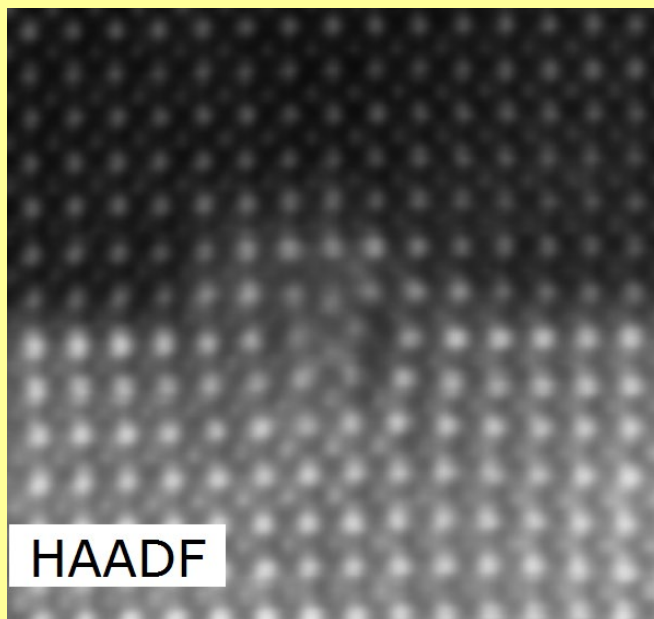
chemical analysis,
Spectroscopy
incoherent, energy loss
(e^- particle)

interaction -> contrast

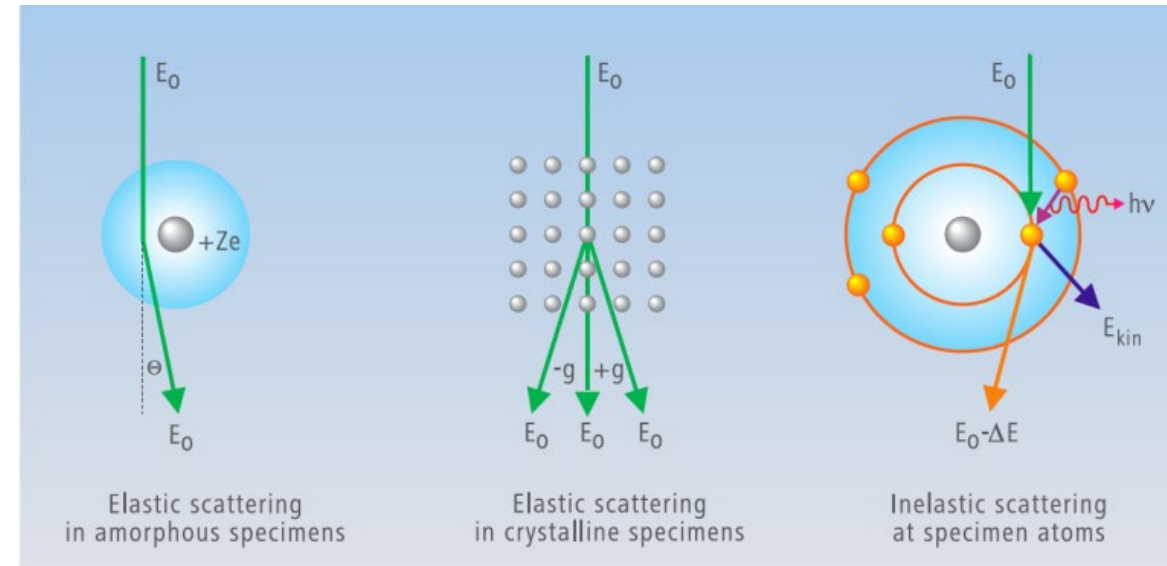
elastic, incoherent



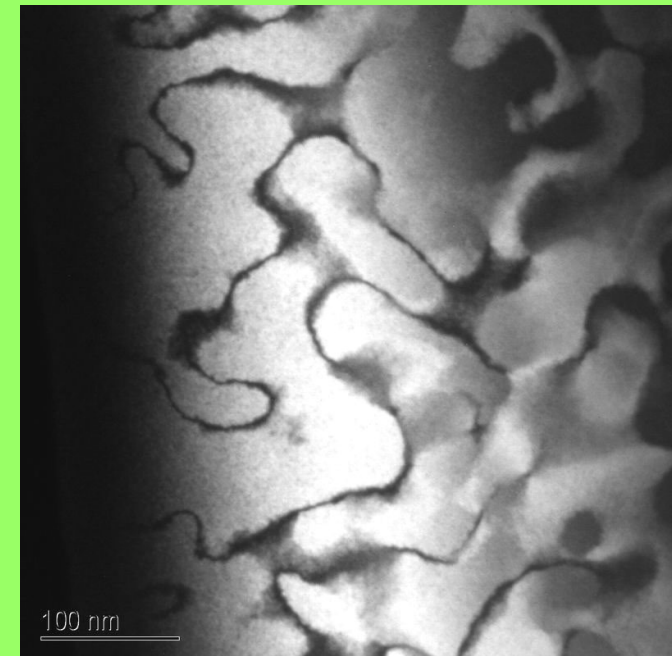
Thin section of mouse brain: mass contrast of stained membrane structures (G.Knott)



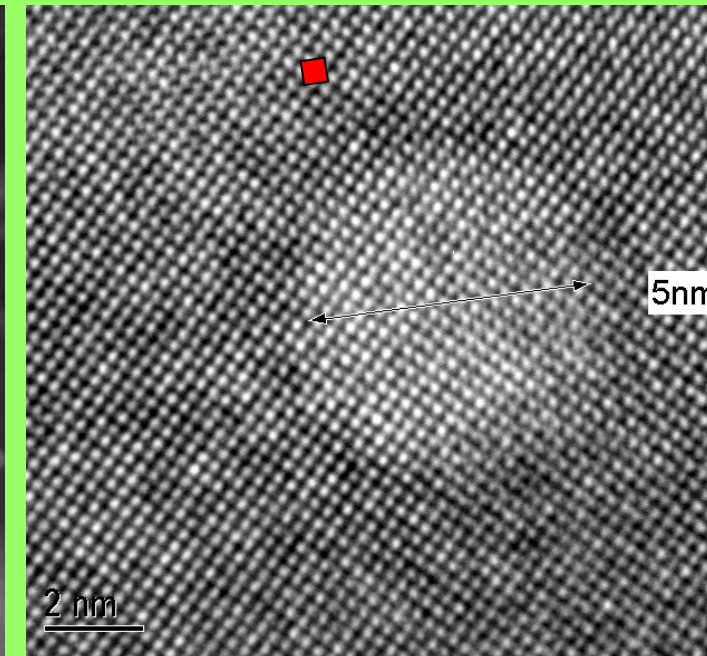
High-resolution STEM image «Z»-contrast



elastic, coherent

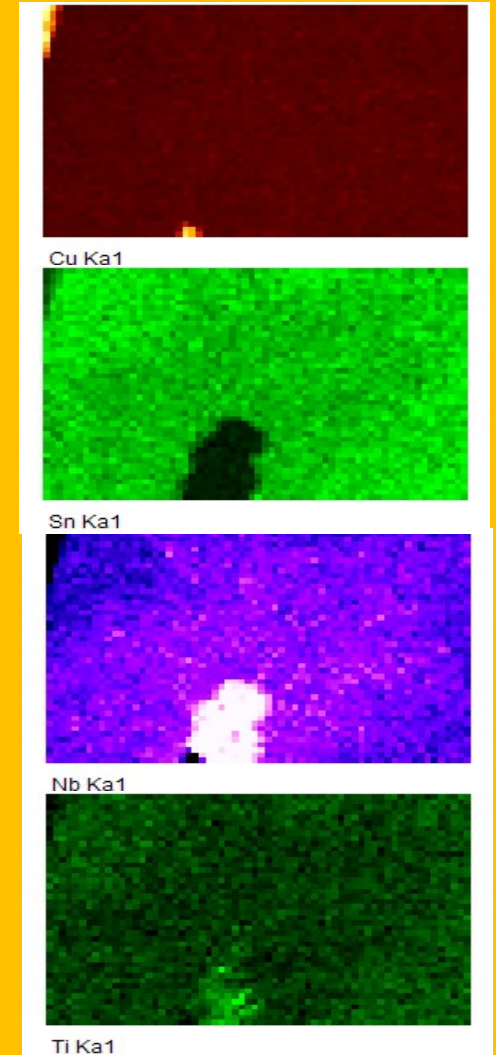


Dark field image of differently ordered domains: Diffraction contrast

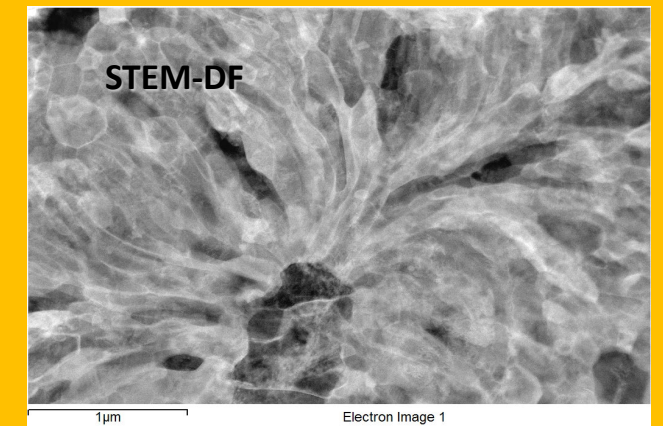


High-resolution image: Image contrast due to interference between transmitted and diffracted beam

inelastic

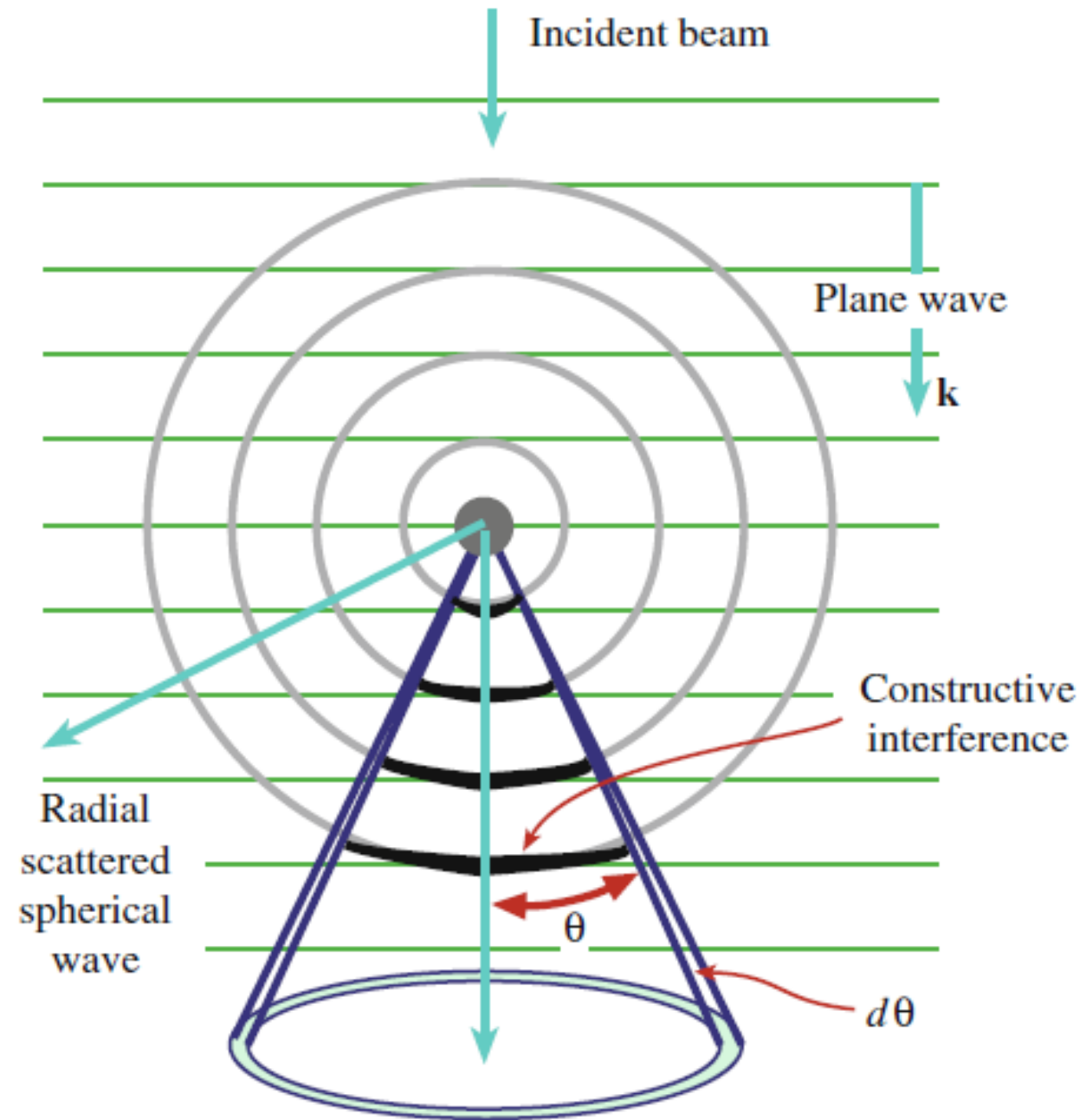


EDS, element maps

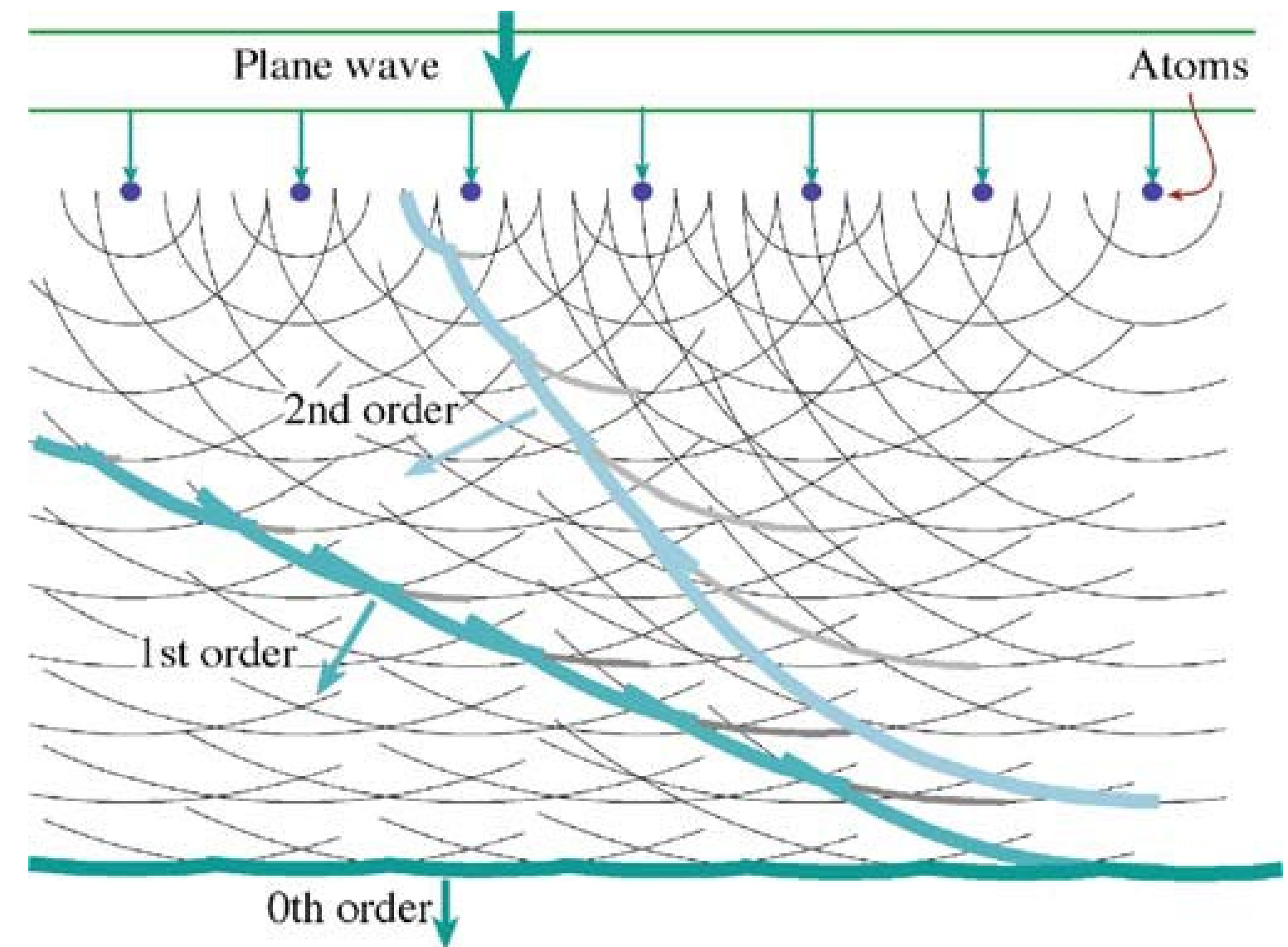


Element distribution maps of Nb₃Sn superconductor

single atom scattering & Huygens principle

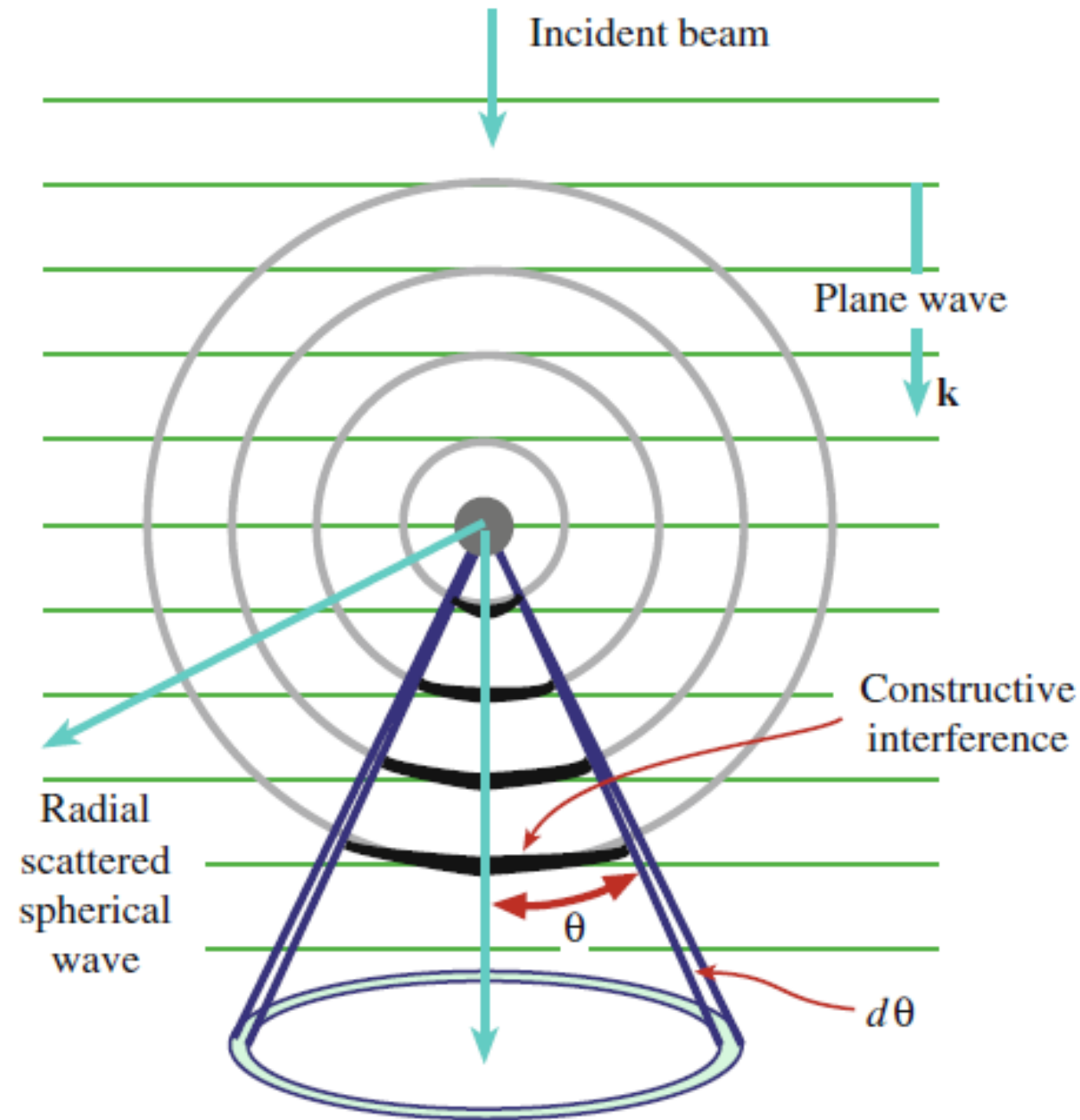


outgoing spherical wave

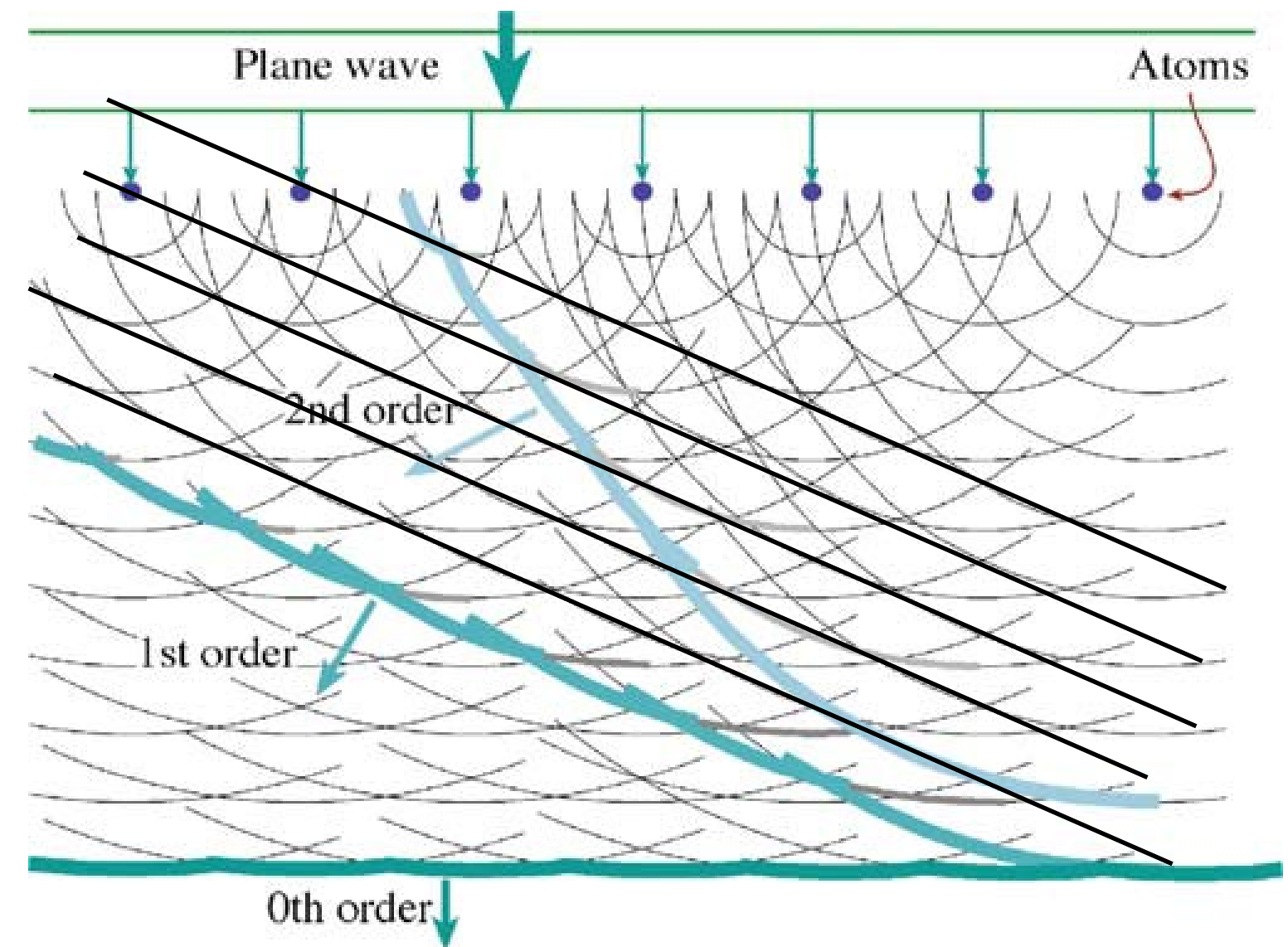


coherent electron wave generates secondary wavelets from a row of scattering centers (e.g., atoms in the specimen). The secondary wavelets interfere, resulting in a strong direct (zero order) beam and several (higher order) coherent beams scattered (diffracted) at specific angles.

single atom scattering & Huygens principle

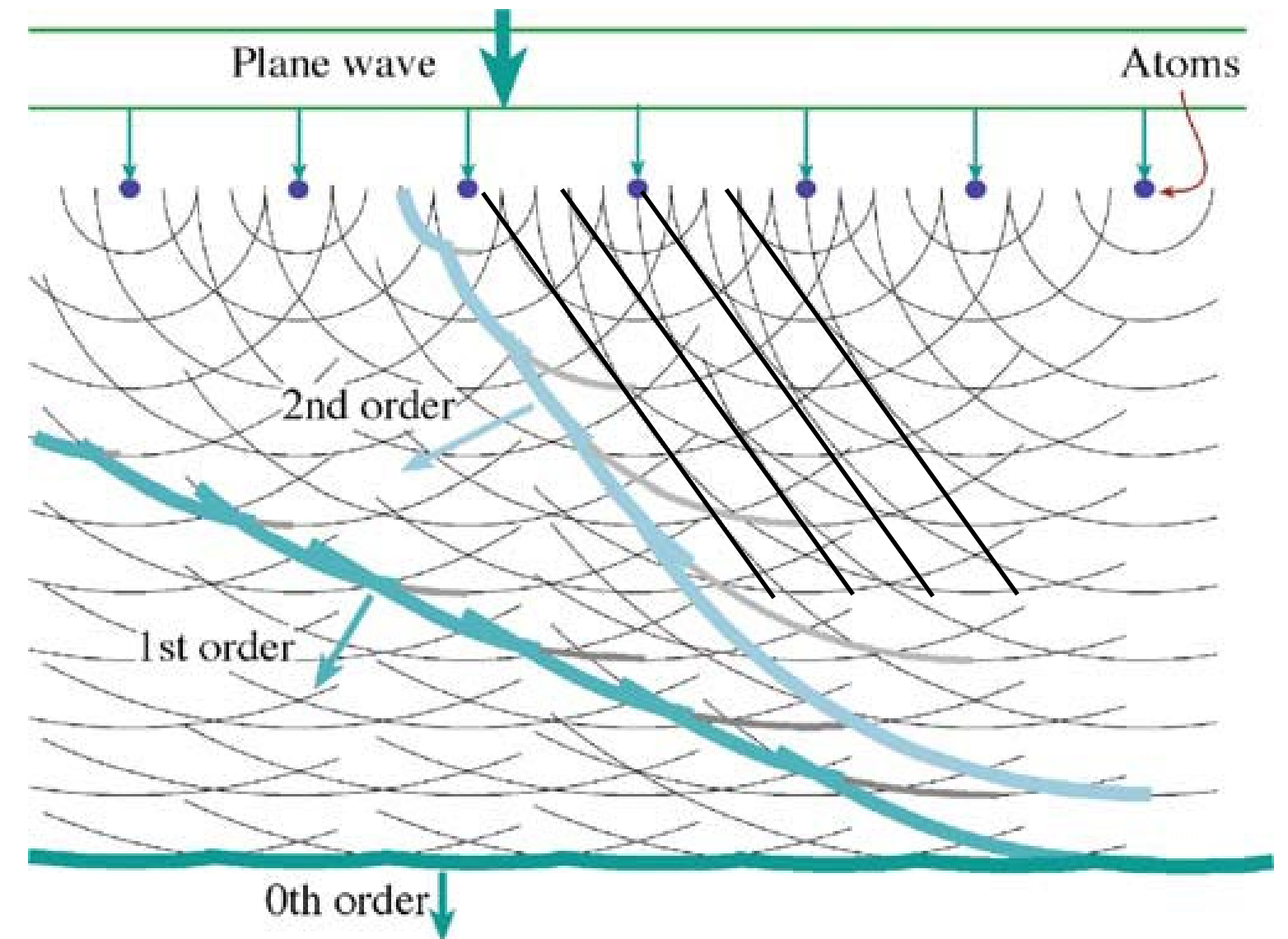
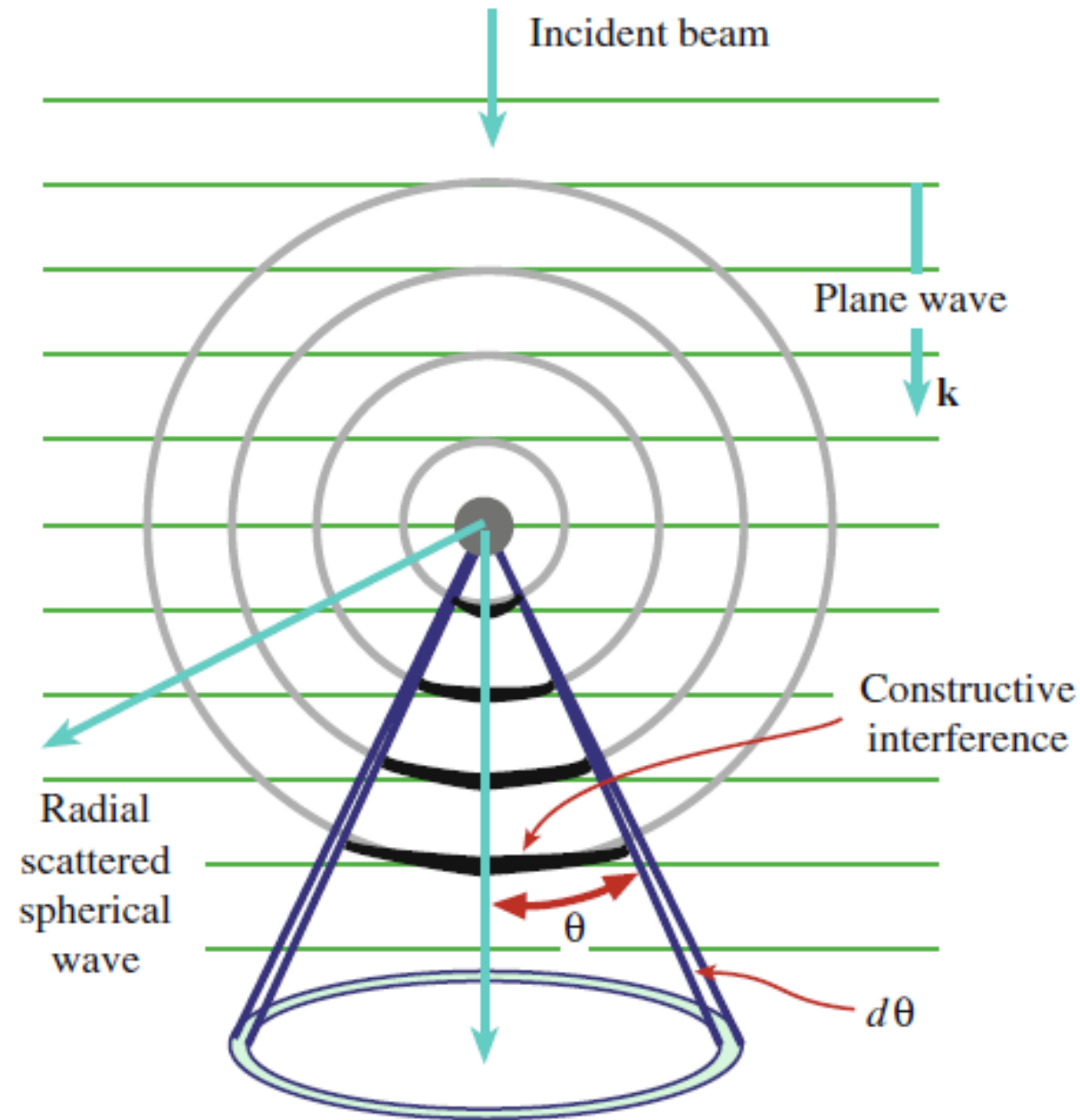


outgoing spherical wave



coherent electron wave generates secondary wavelets from a row of scattering centers (e.g., atoms in the specimen). The secondary wavelets interfere, resulting in a strong direct (zero order) beam and several (higher order) coherent beams scattered (diffracted) at specific angles.

single atom scattering & Huygens principle



coherent electron wave generates secondary wavelets from a row of scattering centers (e.g., atoms in the specimen). The secondary wavelets interfere, resulting in a strong direct (zero order) beam and several (higher order) coherent beams scattered (diffracted) at specific angles.

Abbé's principle in light optics

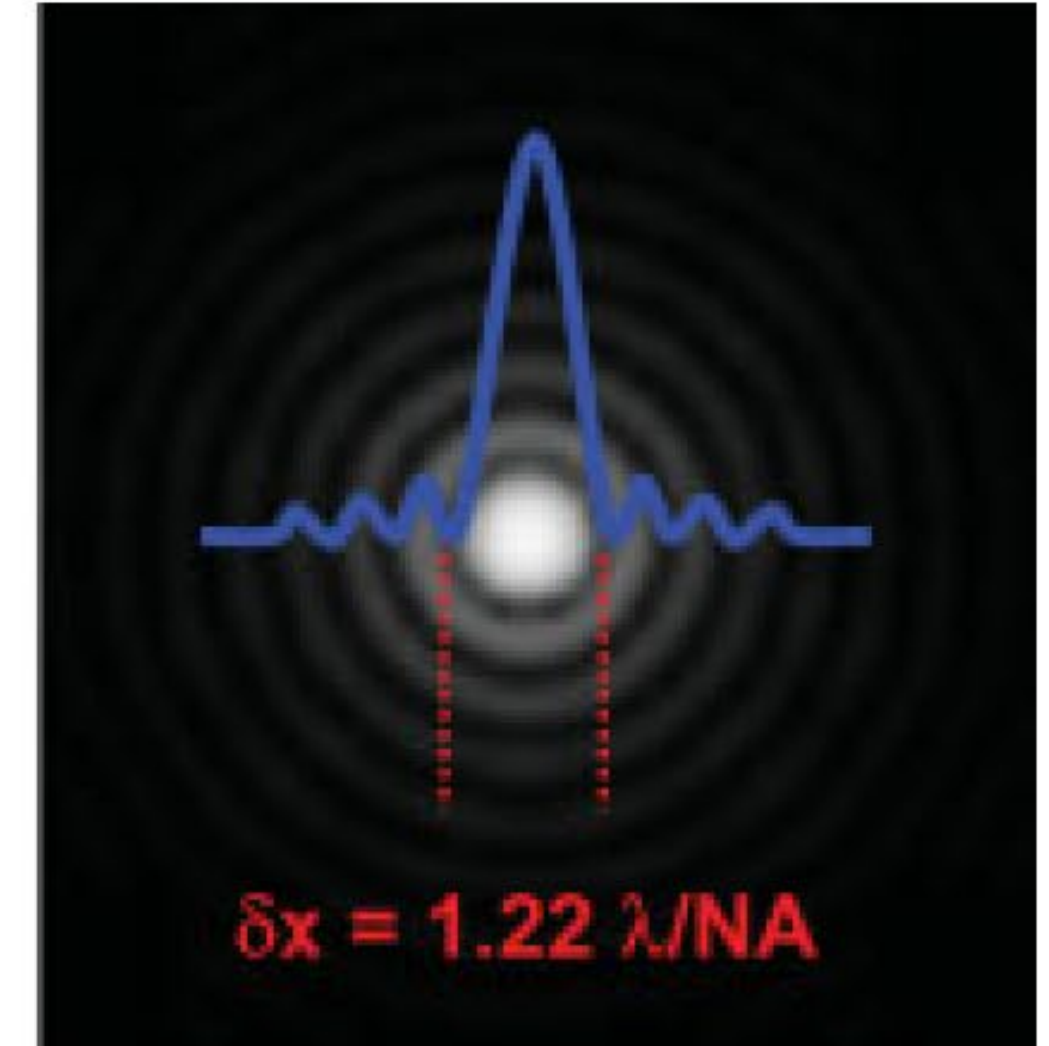
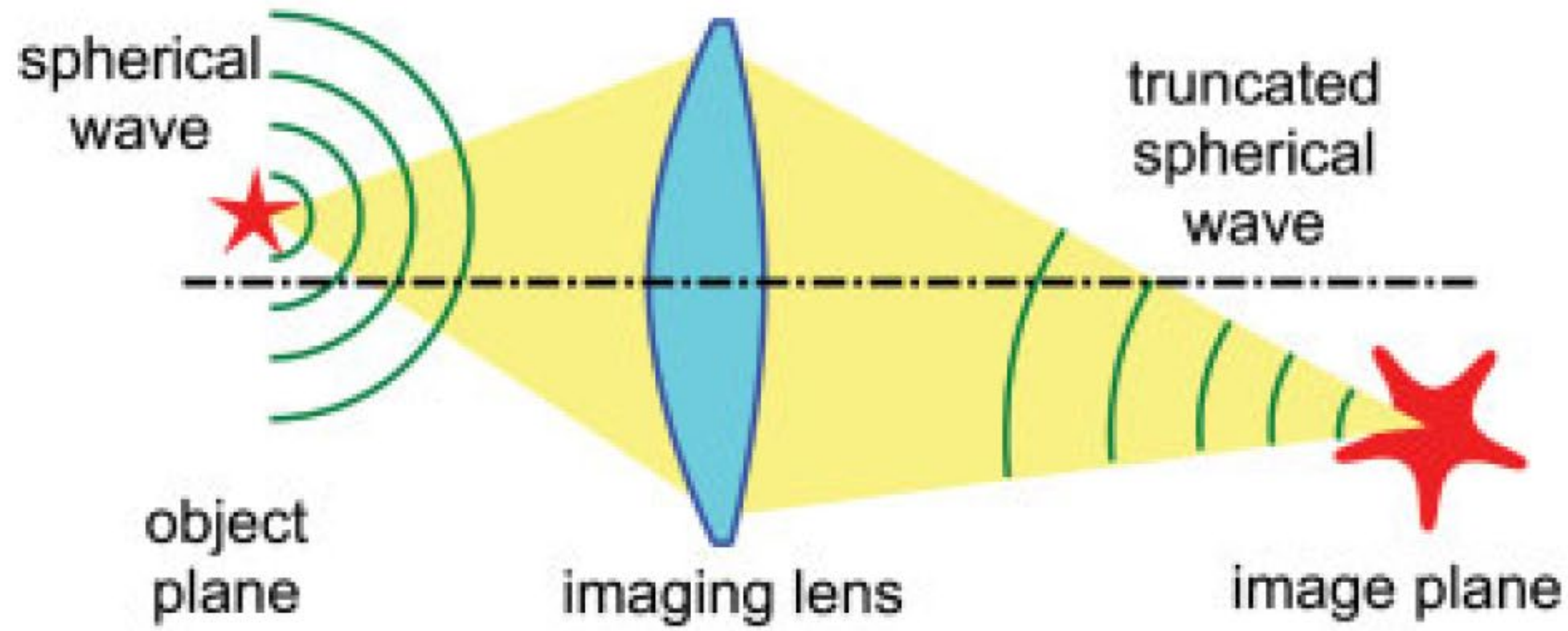


Figure 21-2: Image formation of self-luminous objects.

Handbook of Optical Systems: Vol. 2. Physical Image Formation.

x: resolution (point spread function)

λ : wave length

NA: numerical aperture (lens diameter)

Abbé: image formation by diffraction

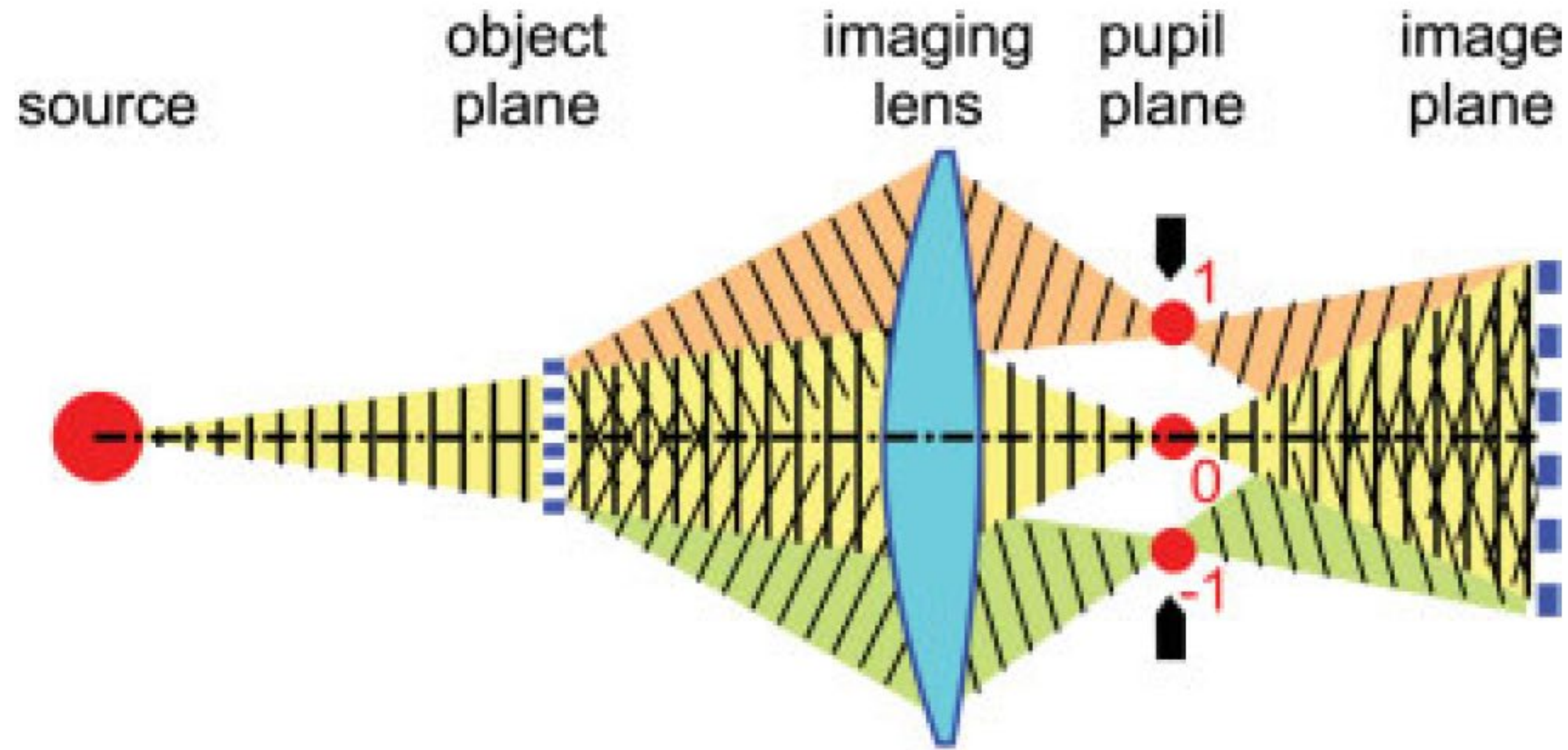
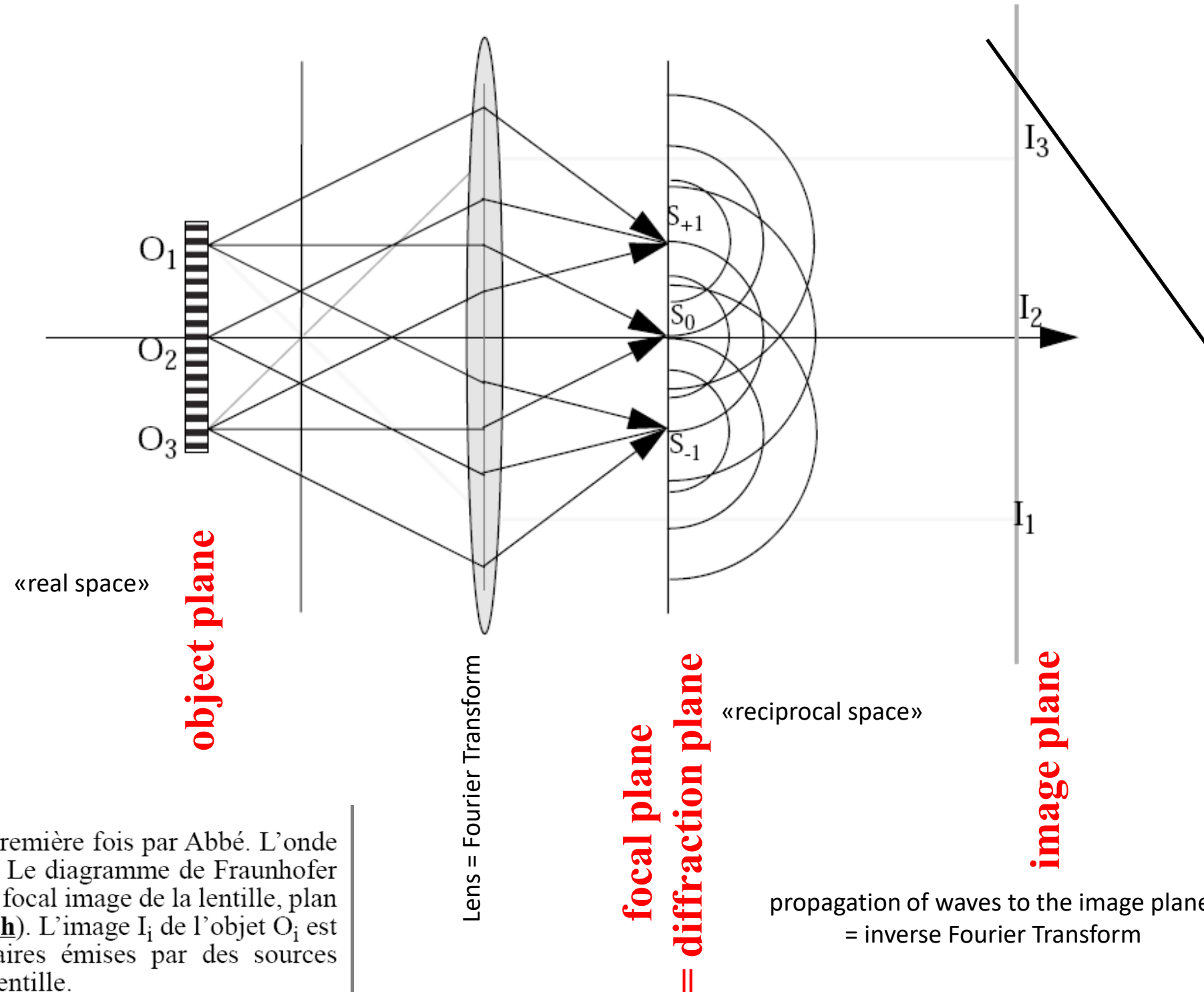


Figure 21-3: Abbe Theory of image formation by diffraction and interference.

Abbe's principle

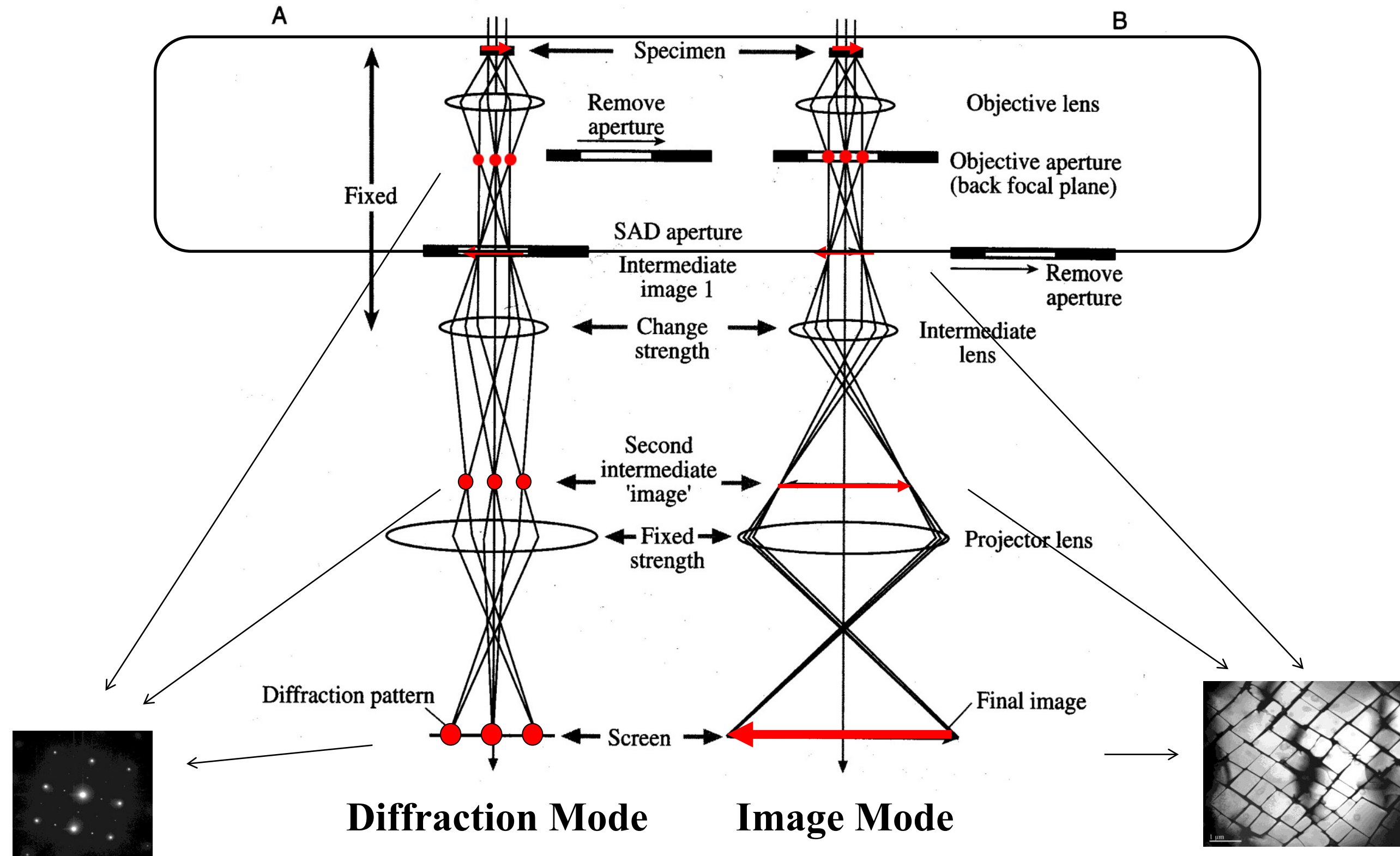
object wave function $\Psi_o(\underline{x})$ $\xrightarrow{FT}$ $\Psi_o(\underline{h})$ $\xrightarrow{FT^{-1}}$ $\Psi_i(\underline{x})$ image wave function



Principe de la formation de l'image proposé pour la première fois par Abbé. L'onde incidente monochromatique est diffractée par l'objet. Le diagramme de Fraunhofer de la fonction d'onde de l'objet est formé dans le plan focal image de la lentille, plan dans lequel la fonction de transfert de la lentille est $T(\underline{h})$. L'image I_i de l'objet O_i est le résultat de l'interférence des ondelettes secondaires émises par des sources ponctuelles S_i placées dans le plan focal image de la lentille.

Two basic operation modes

Diffraction $\leftrightarrow$ Image



Content

Introduction to electron diffraction

Elastic scattering theory

Basic crystallography & symmetry

Electron diffraction theory

Convergent beam electron diffraction

Kikuchi patterns

X-ray diffraction

Why use electron diffraction?

Diffraction: constructive and destructive interference of waves

- wavelength of fast moving electrons much smaller than spacing of atomic planes
=> diffraction from atomic planes (e.g. 200 kV e^- , $\lambda = 0.0025$ nm)
- electrons interact very strongly with matter => strong diffraction intensity
(can take patterns in seconds, unlike X-ray diffraction)
 - spatially-localized information
(≥ 200 nm for selected-area diffraction;
2 nm possible with convergent-beam electron diffraction)
 - close relationship to diffraction contrast in imaging
 - orientation information
 - immediate in the TEM!

diffraction from only selected set of planes in one pattern - e.g. only 2D information

limited accuracy of measurement - e.g. 2-3%

intensity of reflections difficult to interpret because of dynamical effects

Why use electron diffraction?

Diffraction: constructive and destructive interference of waves

- wavelength of fast moving electrons much smaller than spacing of atomic planes
=> diffraction from atomic planes (e.g. 200 kV e^- , $\lambda = 0.0025$ nm)
- electrons interact very strongly with matter => strong diffraction intensity
(can take patterns in seconds, unlike X-ray diffraction)
 - spatially-localized information
(≥ 200 nm for selected-area diffraction;
2 nm possible with convergent-beam electron diffraction)
 - close relationship to diffraction contrast in imaging
 - orientation information
 - immediate in the TEM!

(diffraction from only selected set of planes in one pattern - e.g. only 2D information)

(limited accuracy of measurement - e.g. 2-3%)

(intensity of reflections difficult to interpret because of dynamical effects)

scatter range of electrons, neutrons and X-rays

(99% of intensity lost)

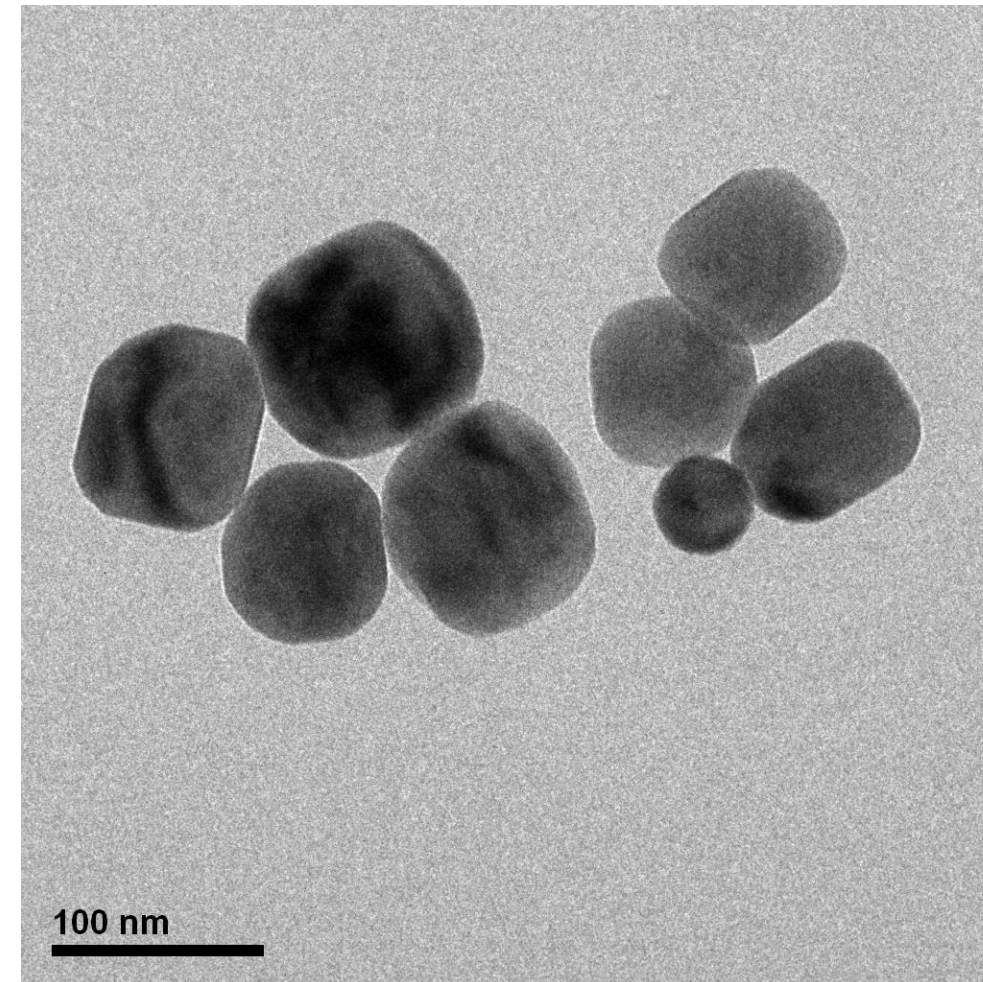
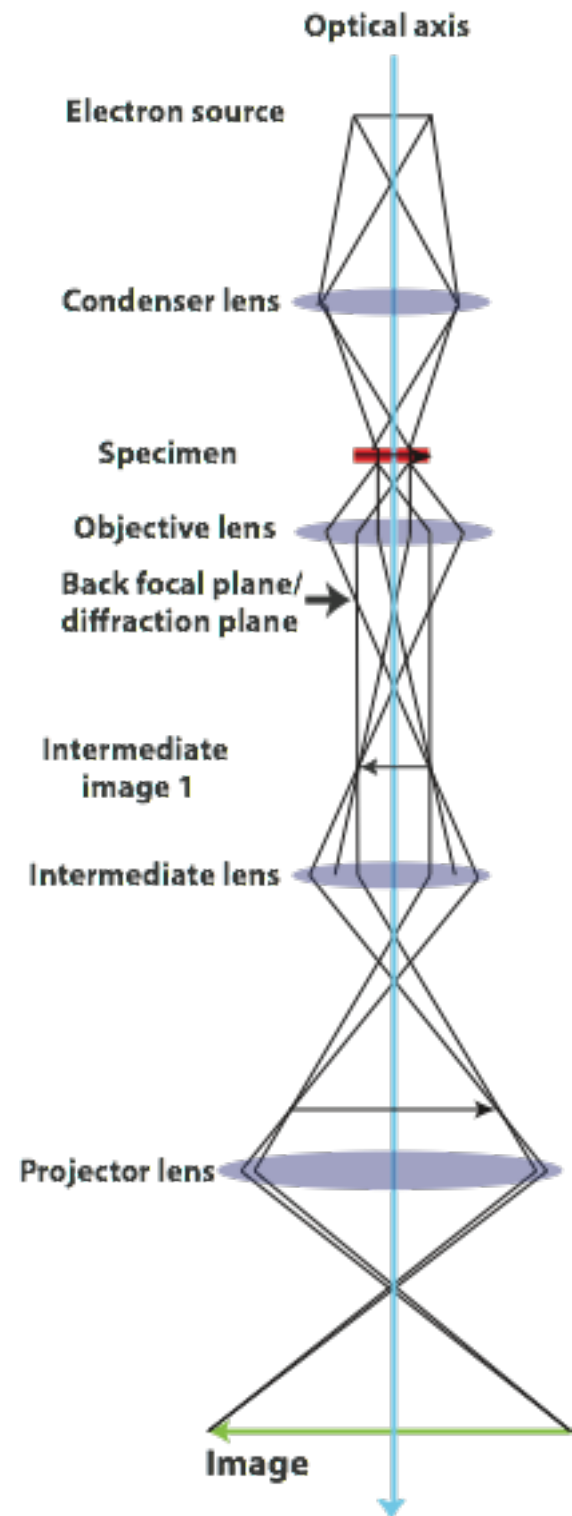
Élément (masse spécifique)	4-Be 1.84 g/cm ³	13-Al 2.7 g/cm ³	29-Cu 8.93 g/cm ³	82-Pb 11.3 g/cm ³
Rayons X Cu-K α $\lambda = 1.54 \text{ \AA}$ Mo-K α $\lambda = 0.71 \text{ \AA}$	16 mm 83 mm	0.35 mm 3.3 mm	0.10 mm 0.10 mm	0.017 mm 0.034 mm
Neutrons thermiques $\lambda \approx 1.08 \text{ \AA}$	89 m	6 m	0.26 m	14 m
Électrons $\lambda = 0.037 \text{ \AA}$ à 100 kV $\lambda = 0.020 \text{ \AA}$ à 300 kV	39 μm	42 μm ~330 μm	11 μm	0.6 μm

electron, neutron X-ray scattering

wave	Wavelength λ	Source	Scattering at	Differentiation of	Sample size
X-rays	0.07nm 0.15nm	X-ray tubes synchrotrons	Electron cloud	Lattice parameters Unit cell	0.1mm
Electrons	2pm @300keV	e guns (SEM/TEM)	Potential distribution (electrons & nucleus)	Lattice parameters, Orientations	0.1um
Neutrons (1)	~0.1nm	Nuclear reactors	Nuclear scattering (nucleus)	LP, isotopes	m
Neutrons (2)	“	“	Magnetic spin (outer electrons)	Oxidation states	m

Scattering power: n : X-ray : e = 1 : 10 : 10⁴

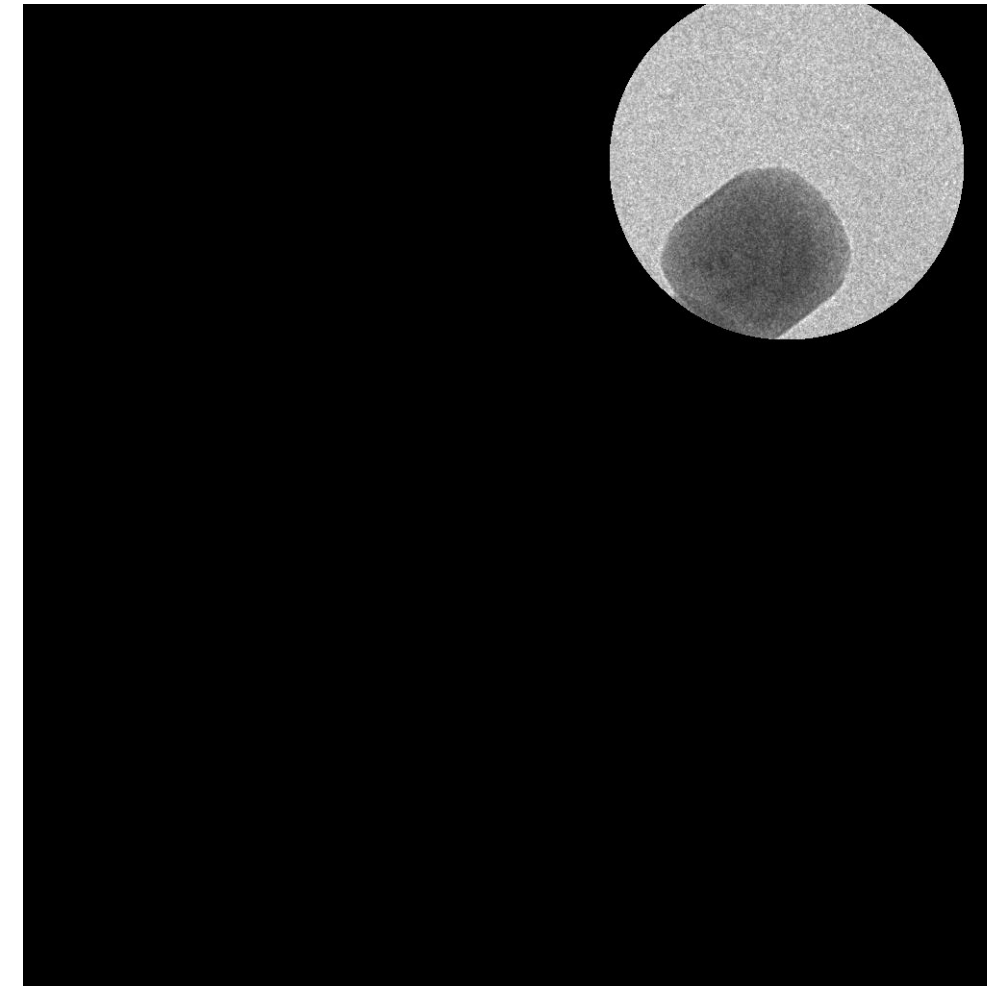
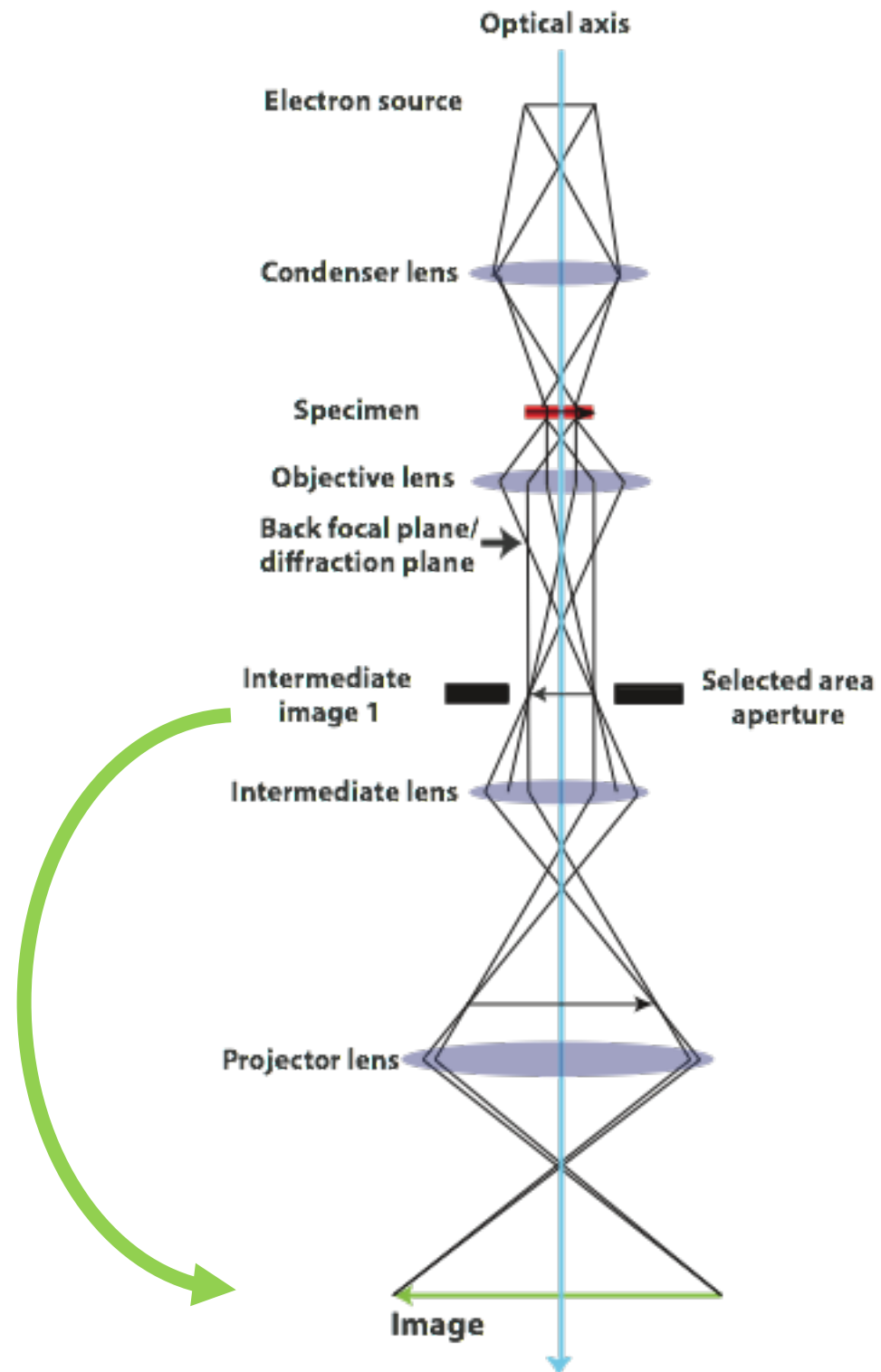
Image formation



BaTiO₃ nanocrystals (Psaltis lab)

Insert selected area aperture to choose region of interest

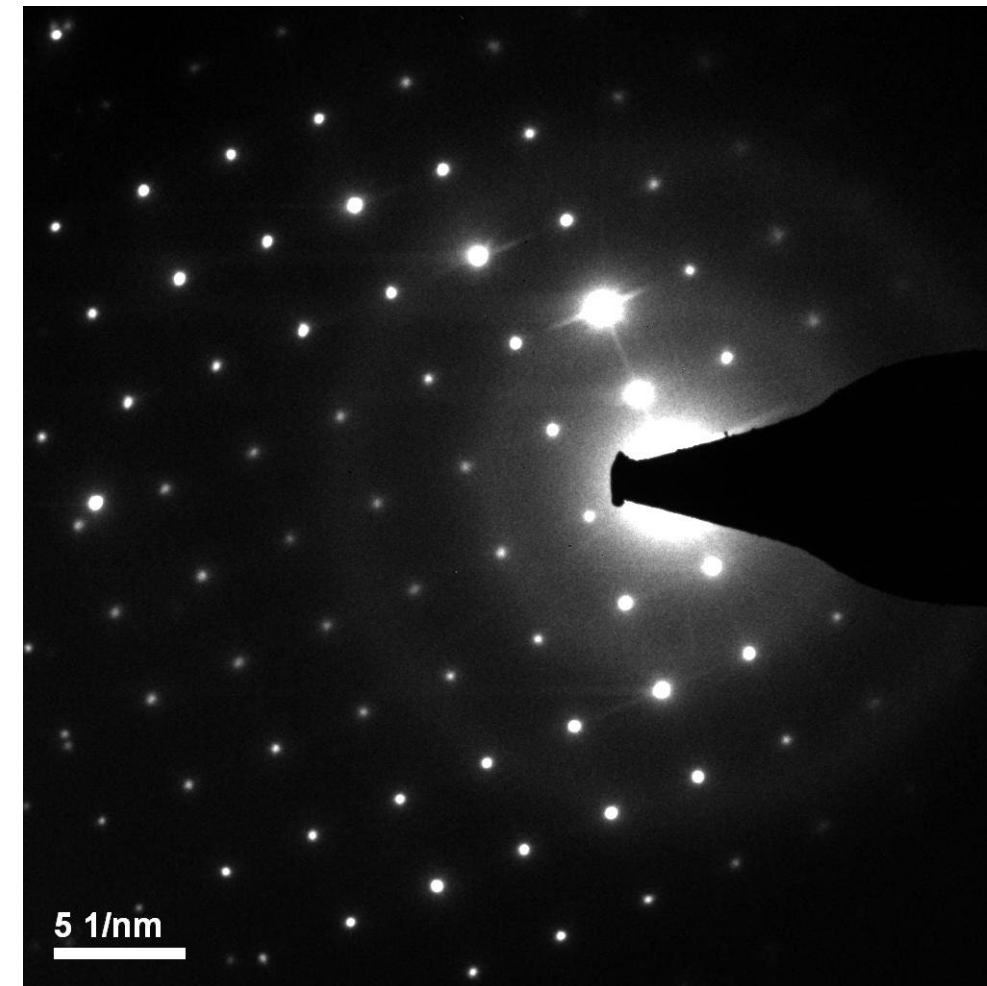
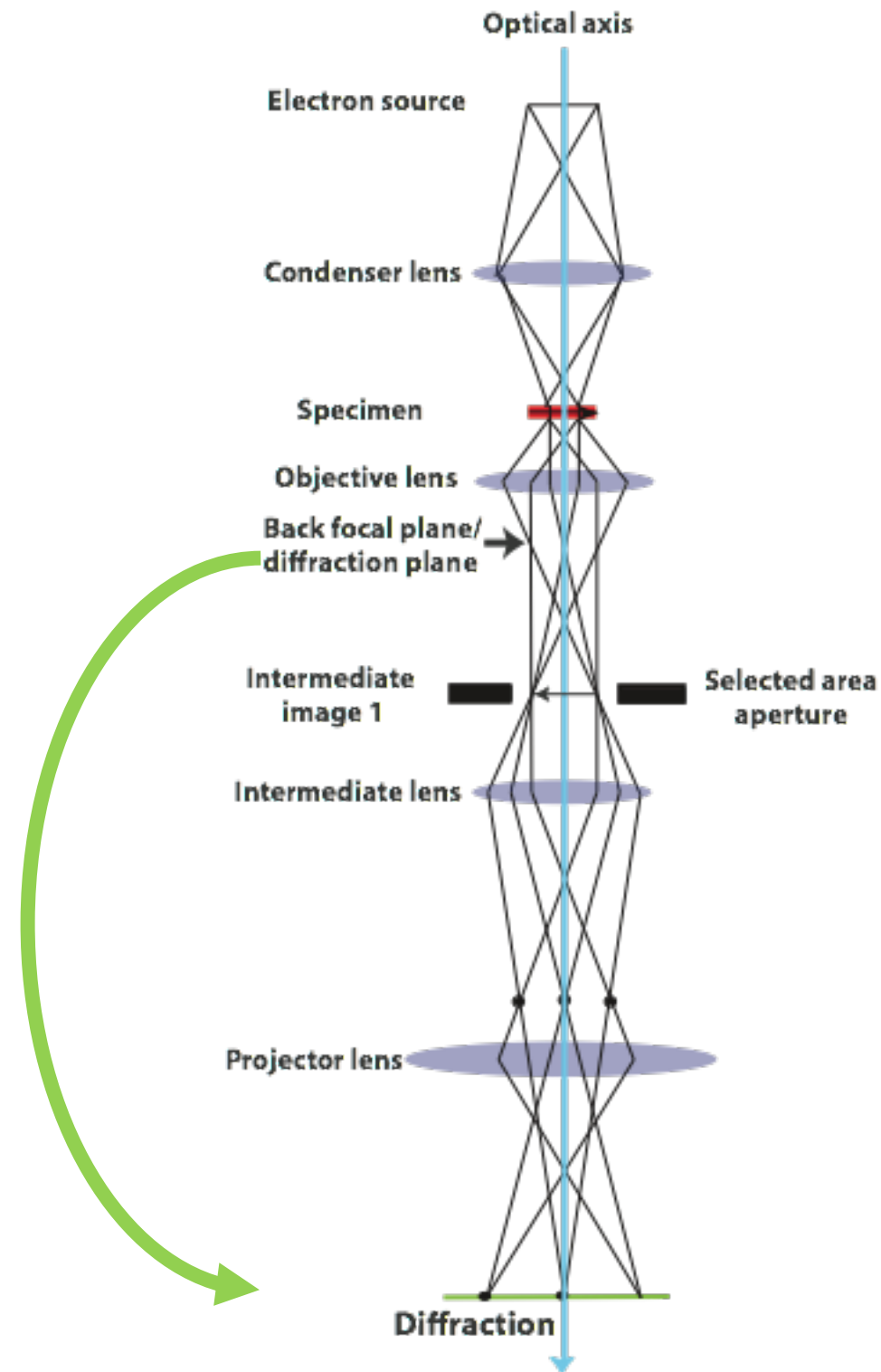
Image formation



BaTiO₃ nanocrystals (Psaltis lab)

Insert selected area aperture to choose region of interest

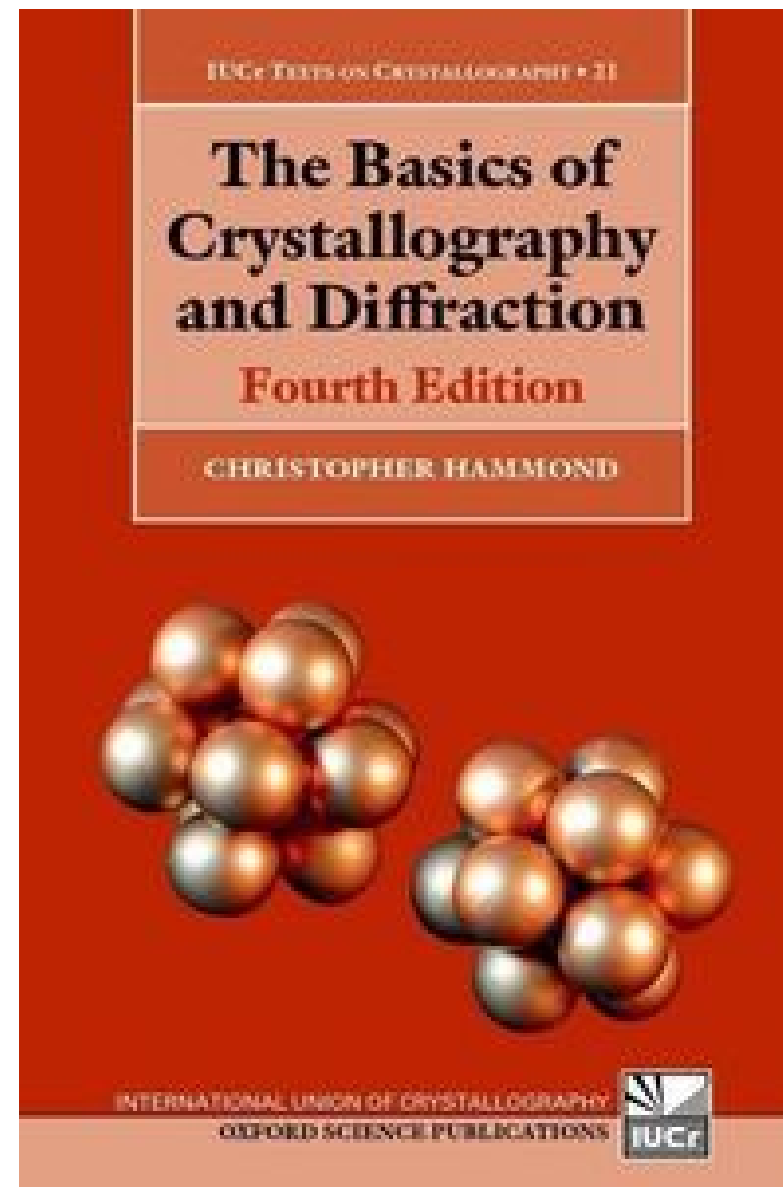
Take selected-area diffraction pattern



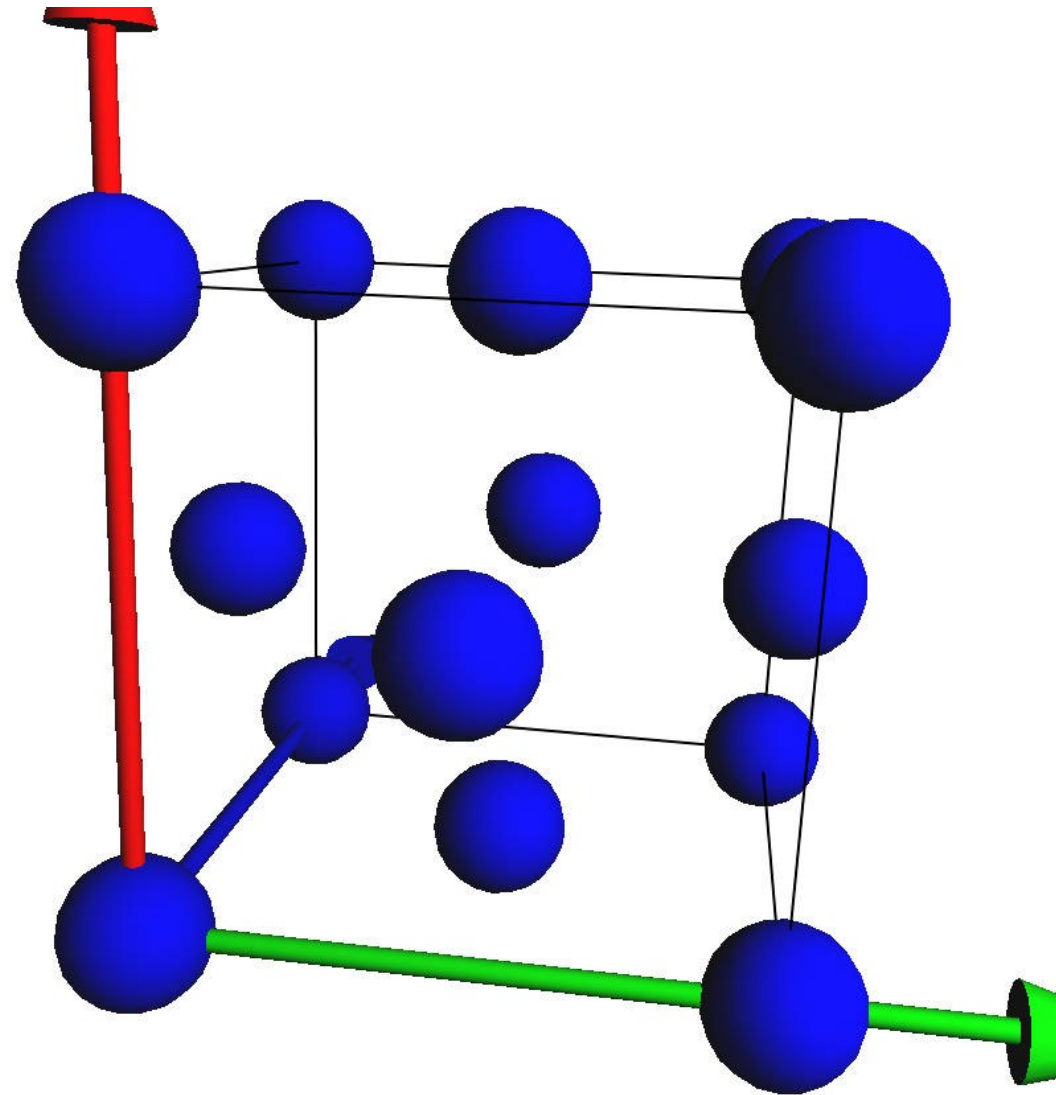
Press “D” for diffraction on microscope console -
alter strength of intermediate lens and focus
diffraction pattern on to screen

Find cubic BaTiO₃ aligned on [0 0 1] zone axis

Basic crystallography & symmetry

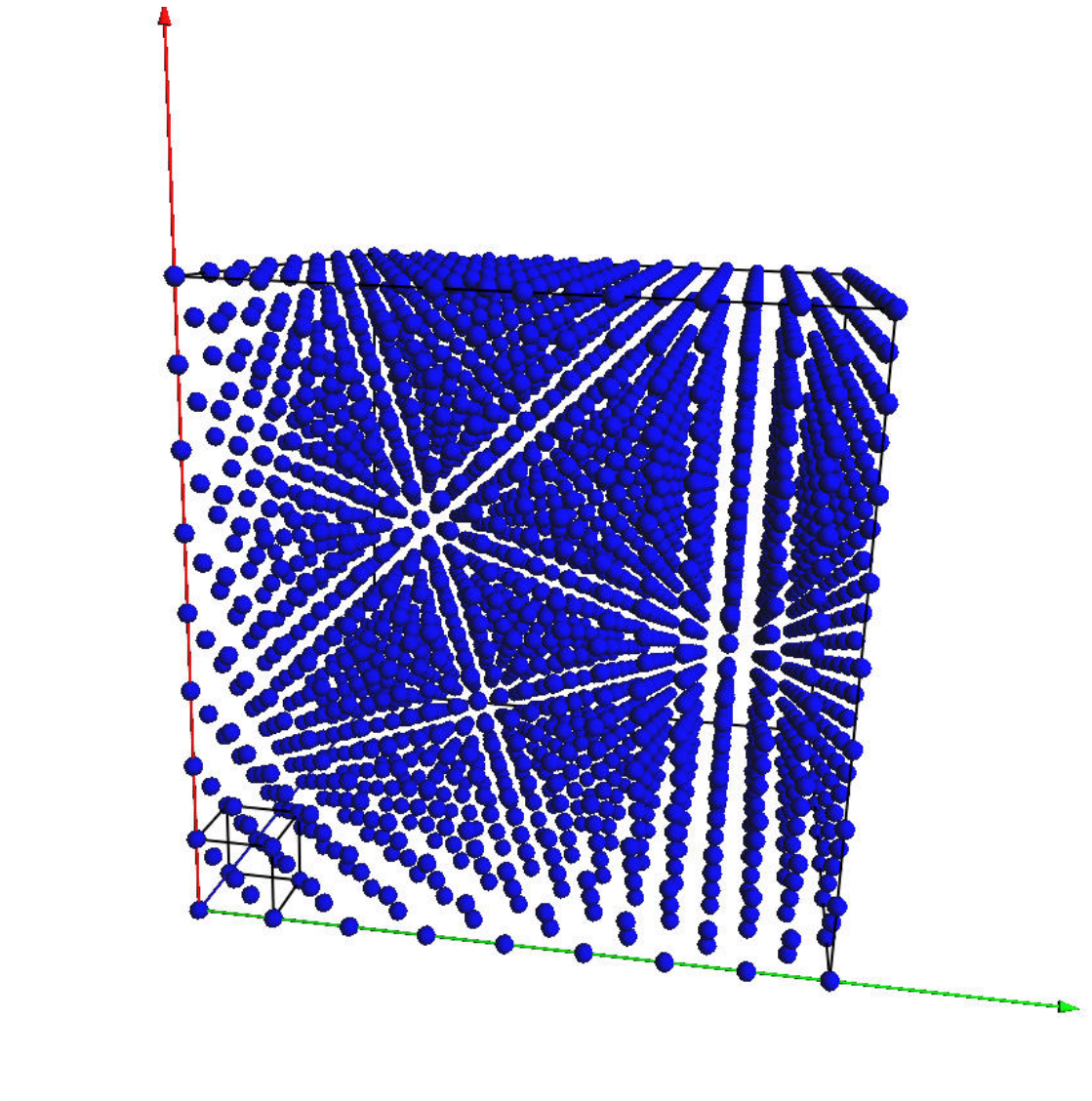


Crystals: translational periodicity & symmetry



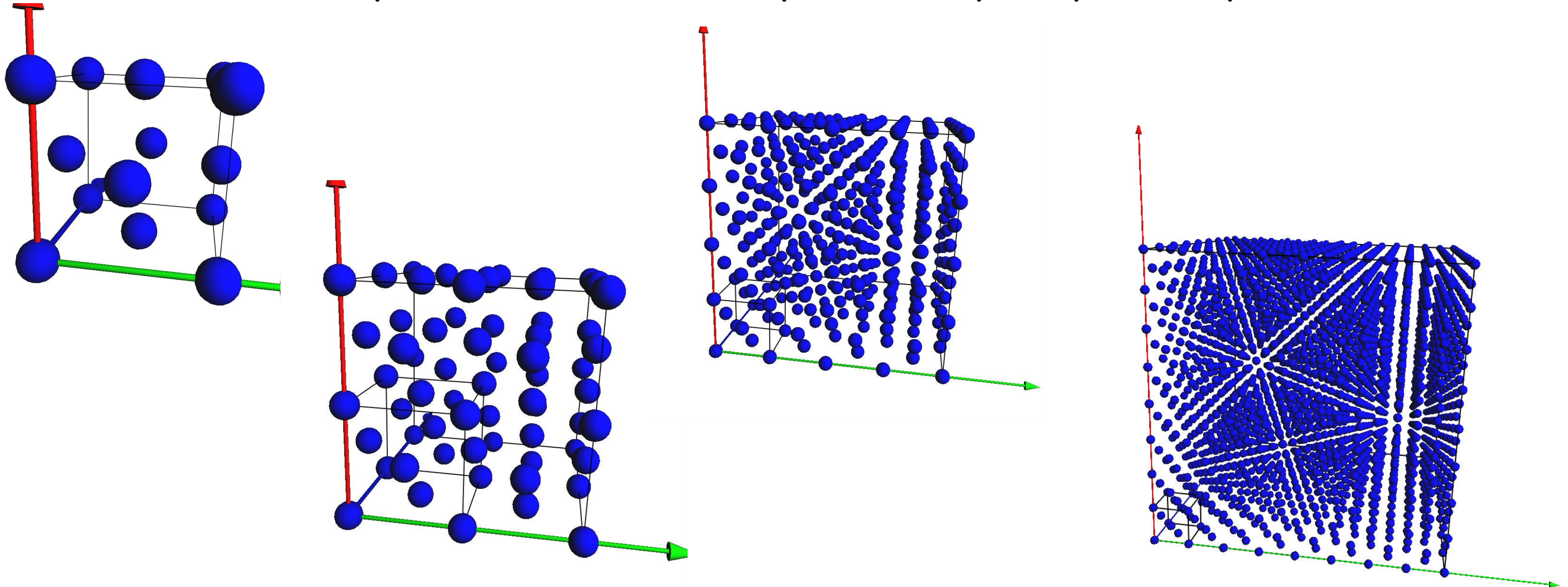
Repetition of translated structure to infinity

Crystals: translational periodicity & symmetry



Repetition of translated structure to infinity

Crystals: translational periodicity & symmetry

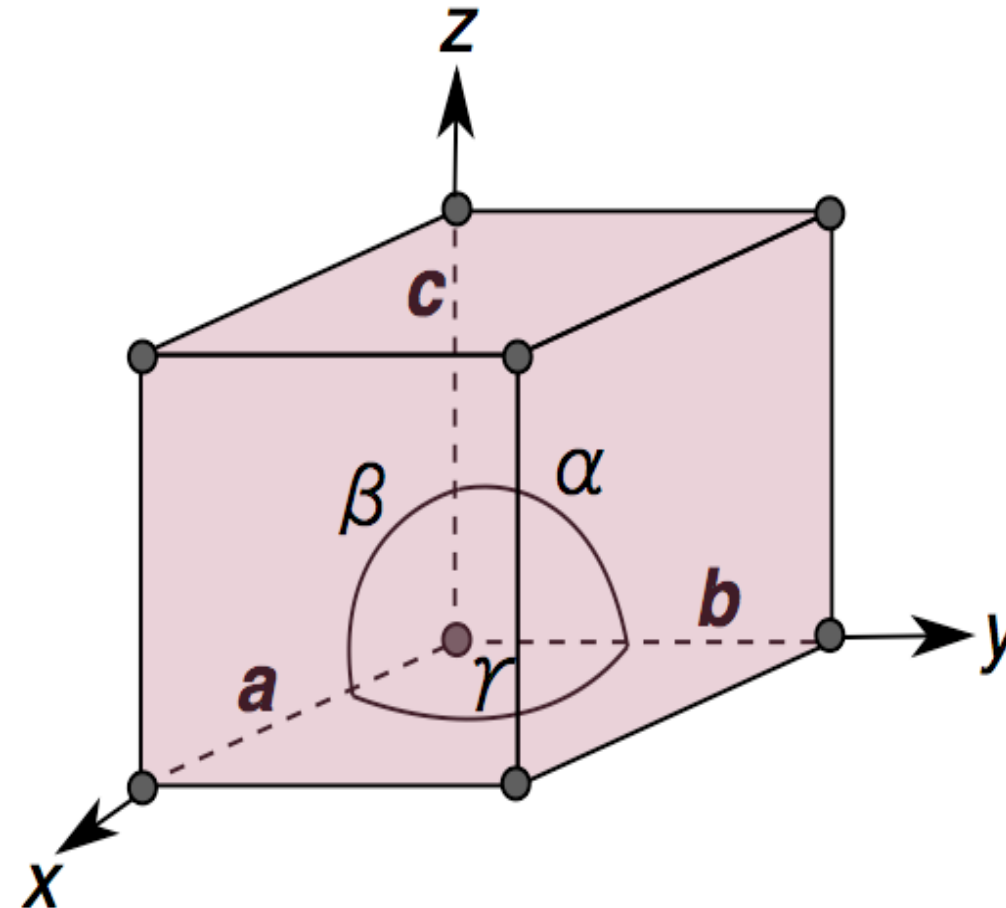


Repetition of translated structure to infinity

Crystallography: the unit cell

Unit cell is the smallest repeating unit of the crystal lattice
Has a **lattice point** on each corner (and perhaps more elsewhere)

Defined by lattice parameters a, b, c along axes x, y, z
and angles between crystallographic axes: $\alpha = \angle b \hat{c}$; $\beta = \angle a \hat{c}$; $\gamma = \angle a \hat{b}$



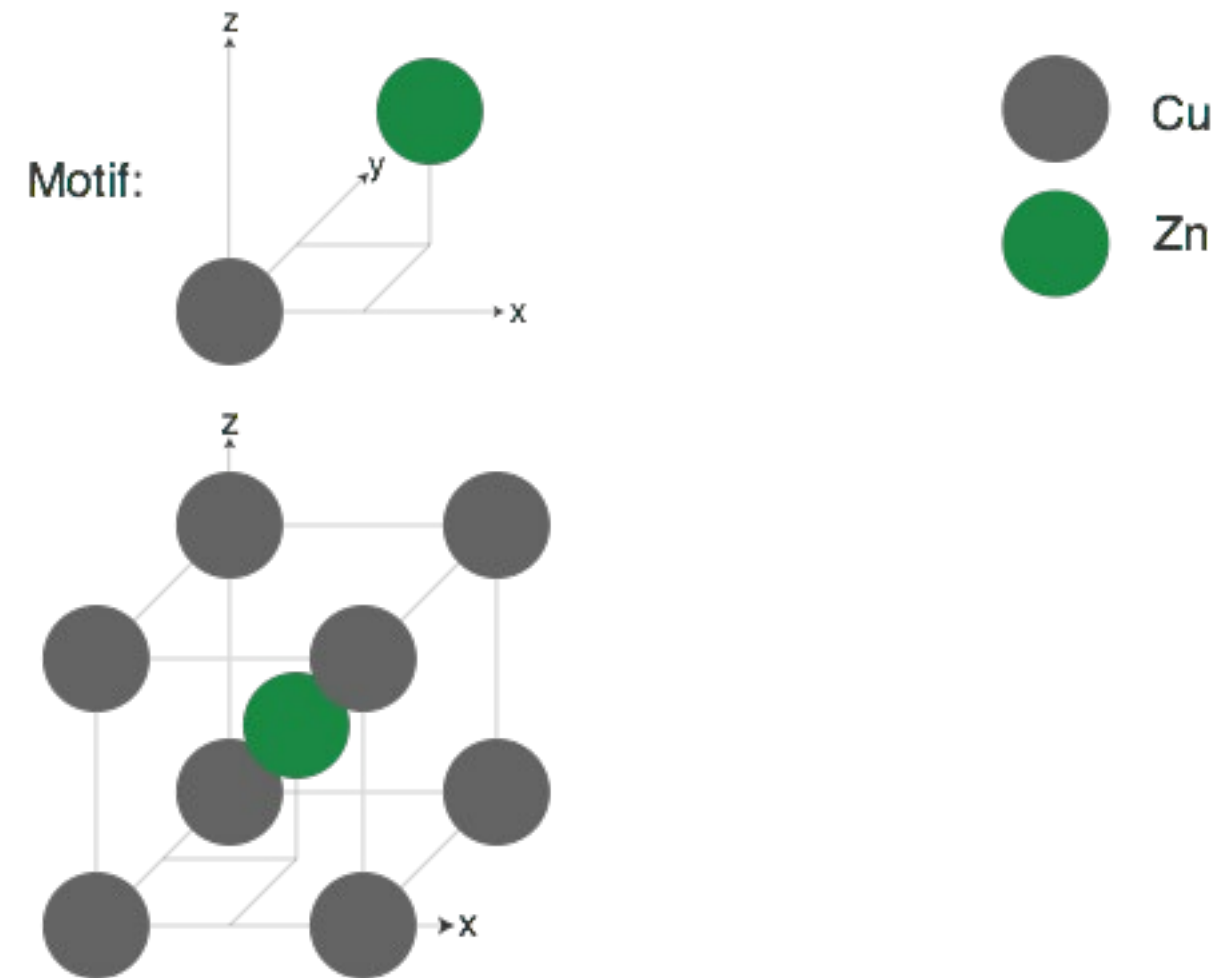
Building a crystal structure adding atoms

Use example of CuZn brass

Choose the unit cell - for CuZn: primitive cubic (lattice point on each corner)

Choose the motif - Cu: $0, 0, 0$; Zn: $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$

Structure = lattice + motif => Start applying motif to each lattice point



Building a crystal structure

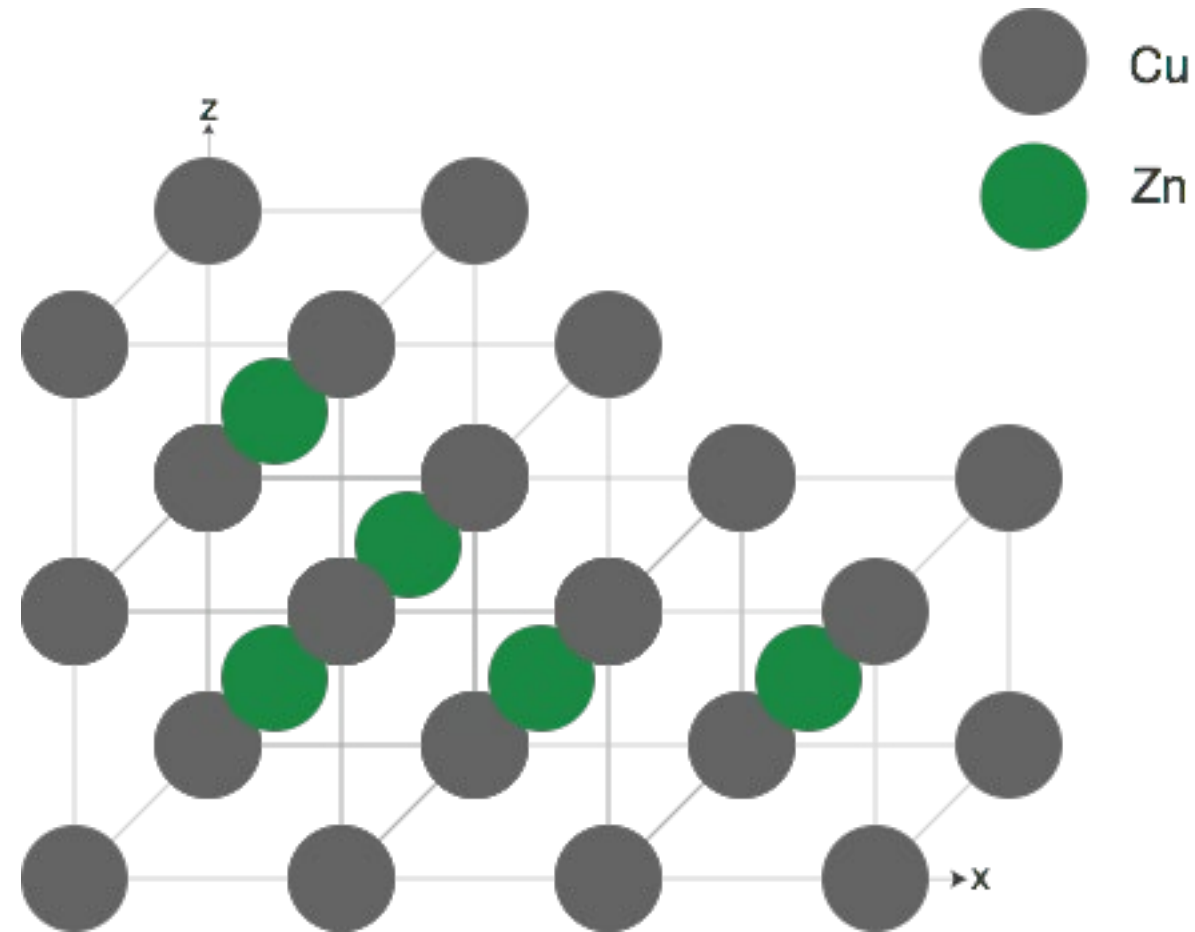
Use example of CuZn brass

Choose the unit cell - for CuZn: primitive cubic (lattice point on each corner)

Choose the motif - Cu: $0, 0, 0$; Zn: $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$

Structure = lattice + motif $\Rightarrow$ Start applying motif to each lattice point

Extend lattice further in to space

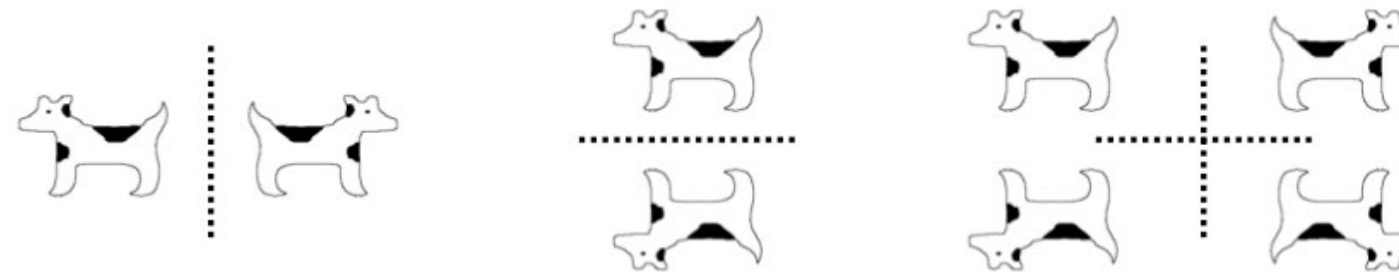


Introduction to symmetry

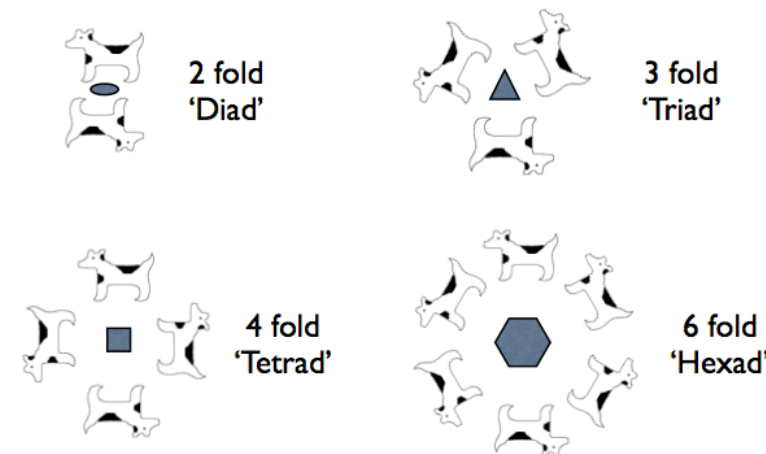
As well as having translational symmetry, nearly all crystals obey other symmetries
- i.e. can reflect or rotate crystal and obtain exactly the same structure

Symmetry elements:

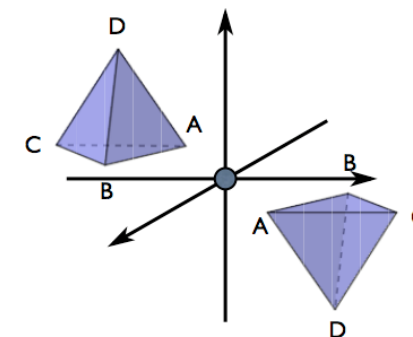
Mirror planes:



Rotation axes:



Centre of symmetry or
inversion centre:



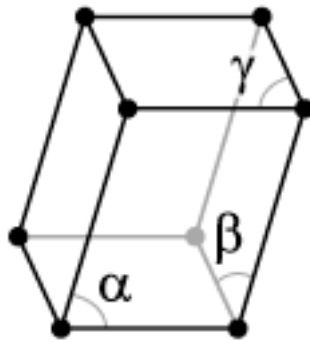
Inversion axes: combination of rotation axis with centre of symmetry

the seven crystal systems

7 possible unit cell shapes with different symmetries that can be repeated by translation in 3 dimensions
=> 7 crystal systems each defined by symmetry

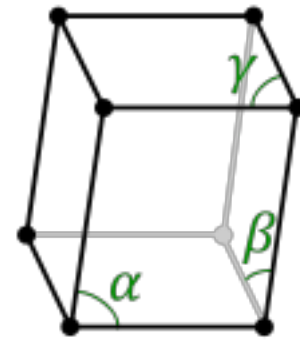
Triclinic

$$\alpha, \beta, \gamma \neq 90^\circ$$



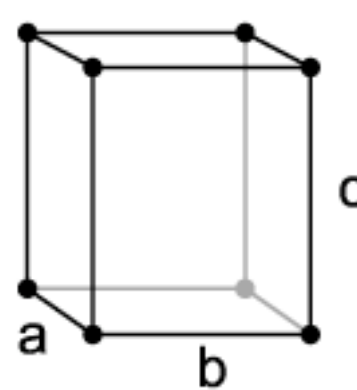
Monoclinic

$$\alpha \neq 90^\circ \\ \beta, \gamma = 90^\circ$$



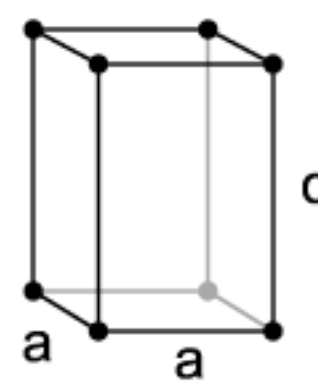
Orthorhombic

$$a \neq b \neq c$$



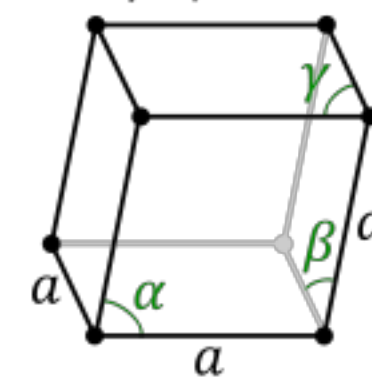
Tetragonal

$$a \neq c$$



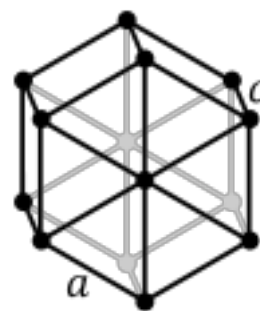
Rhombohedral

$$\alpha, \beta, \gamma \neq 90^\circ$$

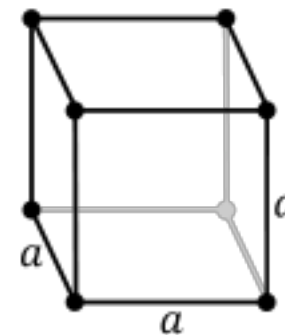


Hexagonal

$$a \neq c$$



Cubic

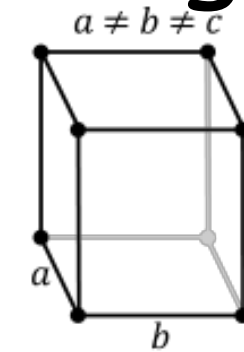


"primitive unit cells"

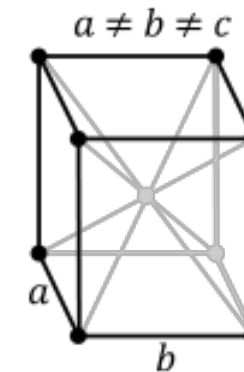
Diagrams from www.Wikipedia.org

four possible lattice centerings

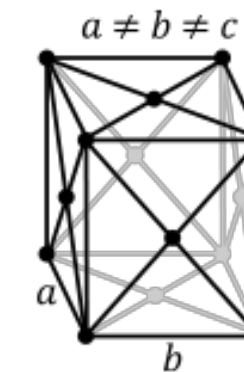
P: Primitive - lattice points on cell corners



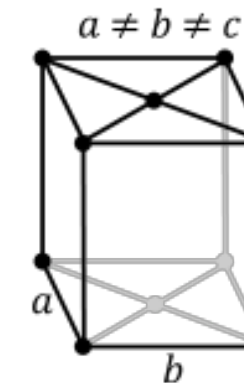
I: Body-centred - additional lattice point at cell centre



F: Face-centred - one additional lattice point at centre of each face



A/B/C: Centred on a single face - one additional lattice point centred on A, B or C face



Diagrams from www.Wikipedia.org

14 Bravais lattices

Combinations of crystal systems and lattice point centring that describe all possible crystals
 - Equivalent system/centring combinations eliminated => 14 (not $7 \times 4 = 28$) possibilities

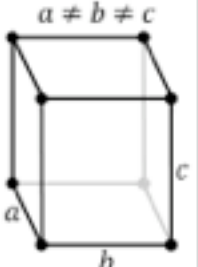
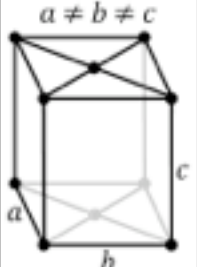
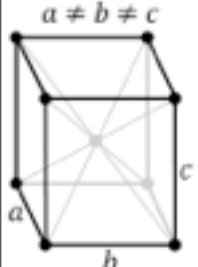
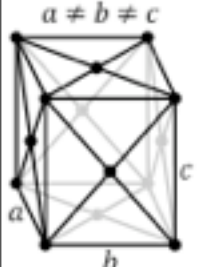
The 7 Crystal systems	The 14 Bravais lattices			
triclinic	P			
	$\alpha, \beta, \gamma \neq 90^\circ$			
monoclinic	P	C		
	$\alpha \neq 90^\circ$ $\beta, \gamma = 90^\circ$	$\alpha \neq 90^\circ$ $\beta, \gamma = 90^\circ$		
orthorhombic	P	C	I	F
	$a \neq b \neq c$	$a \neq b \neq c$	$a \neq b \neq c$	$a \neq b \neq c$
	c	c	c	c

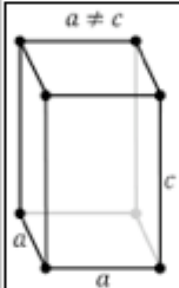
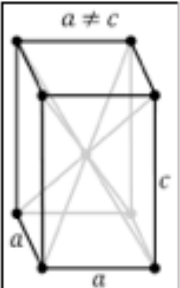
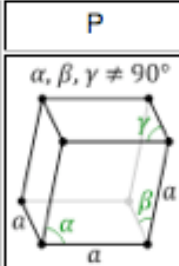
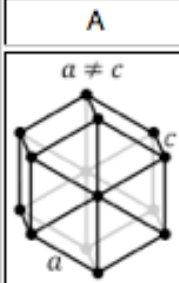
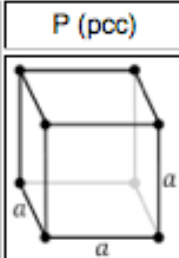
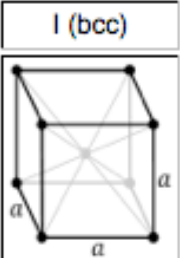
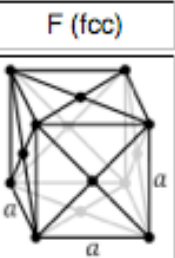
The 7 Crystal systems	The 14 Bravais lattices		
tetragonal	P	I	
	$a \neq c$	$a \neq c$	
rhombohedral (trigonal)	P		
	$\alpha, \beta, \gamma \neq 90^\circ$		
hexagonal	A		
	$a \neq c$		
cubic	P (fcc)	I (bcc)	F (fcc)
	a	a	a

Diagrams from www.Wikipedia.org

14 Bravais lattices

Combinations of crystal systems and lattice point centring that describe all possible crystals
 - Equivalent system/centring combinations eliminated => 14 (not $7 \times 4 = 28$) possibilities

The 7 Crystal systems	The 14 Bravais lattices			
triclinic	P			
	$\alpha, \beta, \gamma \neq 90^\circ$			
monoclinic	P	C		
	$\alpha \neq 90^\circ$ $\beta, \gamma = 90^\circ$	$\alpha \neq 90^\circ$ $\beta, \gamma = 90^\circ$		
orthorhombic	P	C	I	F
	$a \neq b \neq c$	$a \neq b \neq c$	$a \neq b \neq c$	$a \neq b \neq c$
				
	a b c	a b c	a b c	a b c

The 7 Crystal systems	The 14 Bravais lattices			
tetragonal	P 	I 	C ?	
	rhombohedral (trigonal)	P 		
hexagonal		A 		
	cubic	P (fcc) 	I (bcc) 	F (fcc) 

Diagrams from www.Wikipedia.org

14 Bravais lattices

Crystal System	Defining Symmetry (rotation or inversion)	Conventional Unit Cell	Conventional Lattice Types
Cubic	4 triads	$a = b = c$ $\alpha = \beta = \gamma = 90^\circ$	P, I, F
Hexagonal	1 hexad	$a = b \neq c$ $\alpha = \beta = 90^\circ, \gamma = 120^\circ$	P
Trigonal	1 triad	$a = b \neq c$ $\alpha = \beta = 90^\circ, \gamma = 120^\circ$	P, R
Tetragonal	1 tetrad	$a = b \neq c$ $\alpha = \beta = \gamma = 90^\circ$	P, I
Orthorhombic	3 diads	$a \neq b \neq c$ $\alpha = \beta = \gamma = 90^\circ$	P, C, I, F
Monoclinic	1 diad	$a \neq b \neq c$ $\alpha = \gamma = 90^\circ, \beta \geq 90^\circ$	P, C
Triclinic	-	$a \neq b \neq c$ $\alpha \neq \beta \neq \gamma$	P

Crystallography - lattice vectors

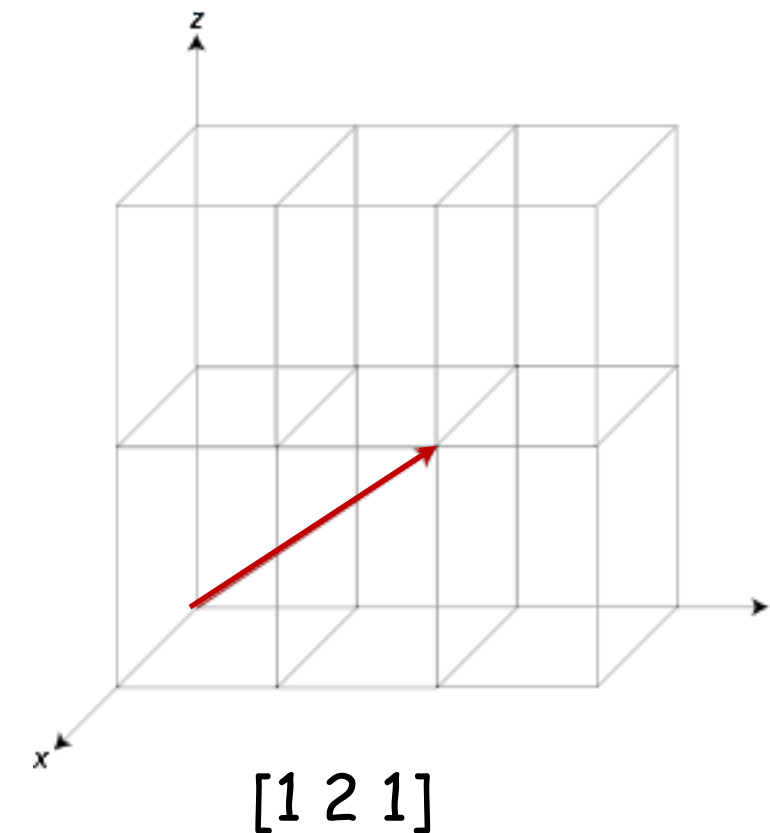
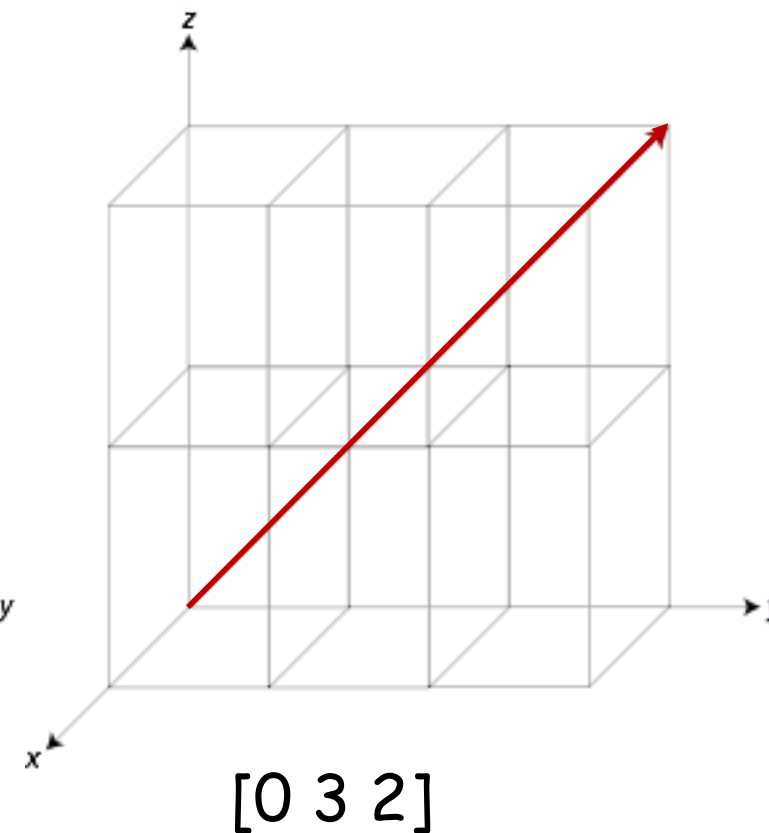
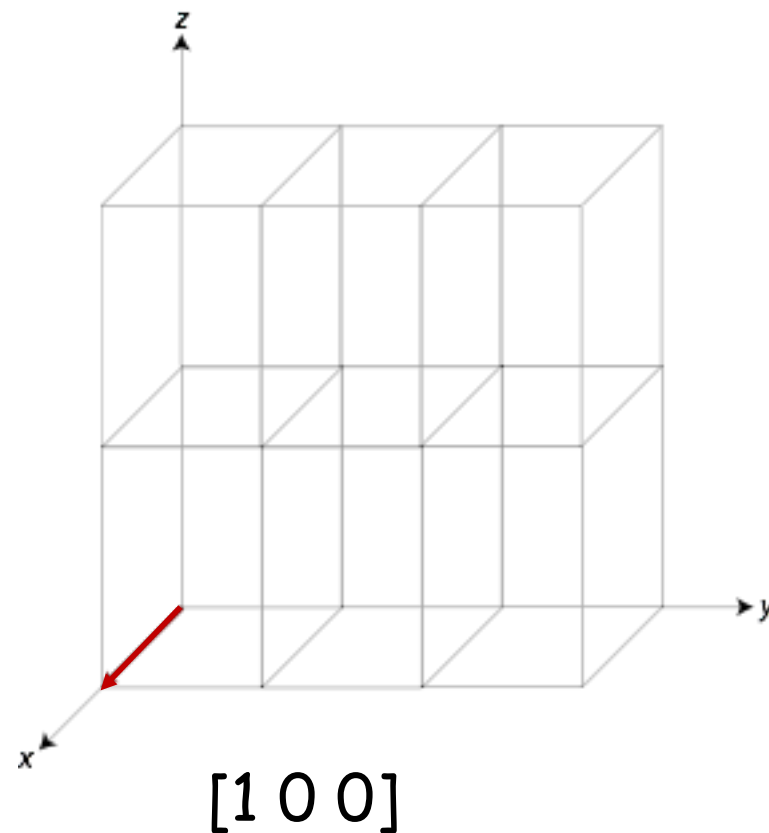
A lattice vector $\mathbf{t}$ is a vector joining any two lattice points

Written as linear combination of unit cell vectors $\mathbf{a}$, $\mathbf{b}$, $\mathbf{c}$:

$$\mathbf{t} = U\mathbf{a} + V\mathbf{b} + W\mathbf{c}$$

Also written as: $\mathbf{t} = [U \ V \ W]$

Examples:



Important in diffraction because we "look" down the lattice vectors ("zone axes")

Crystallography - lattice planes

Lattice plane is a plane which passes through any 3 lattice points which are not in a straight line

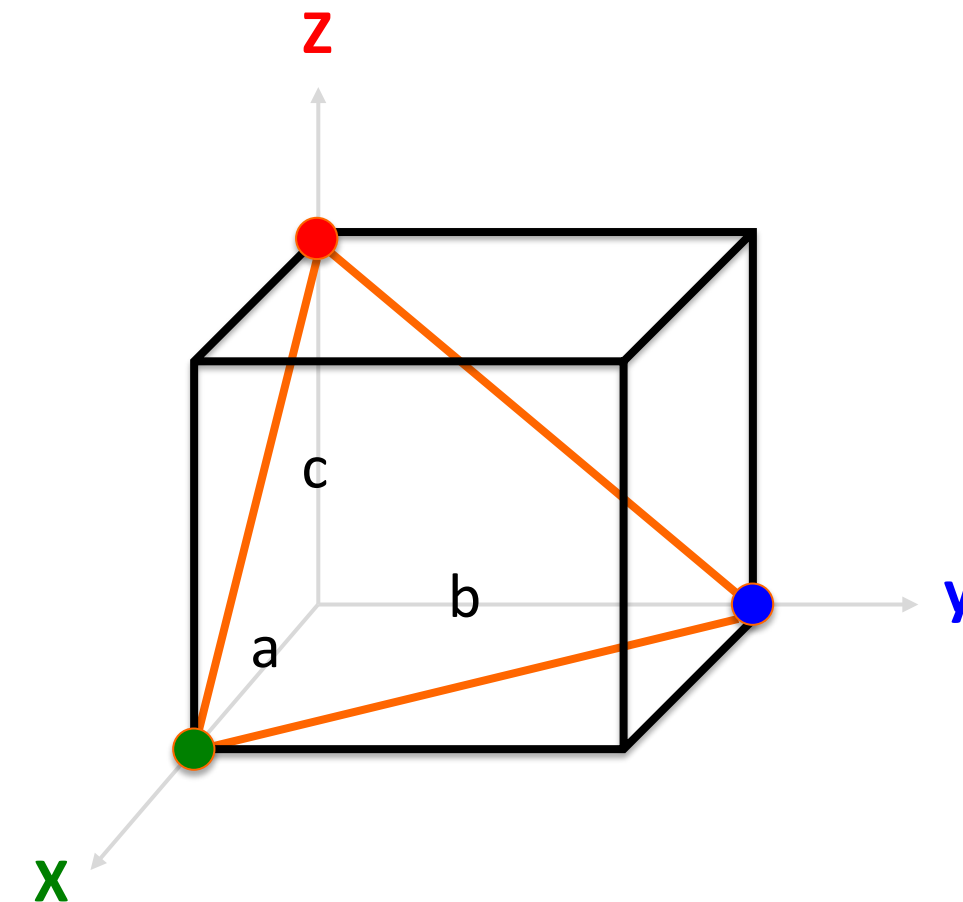
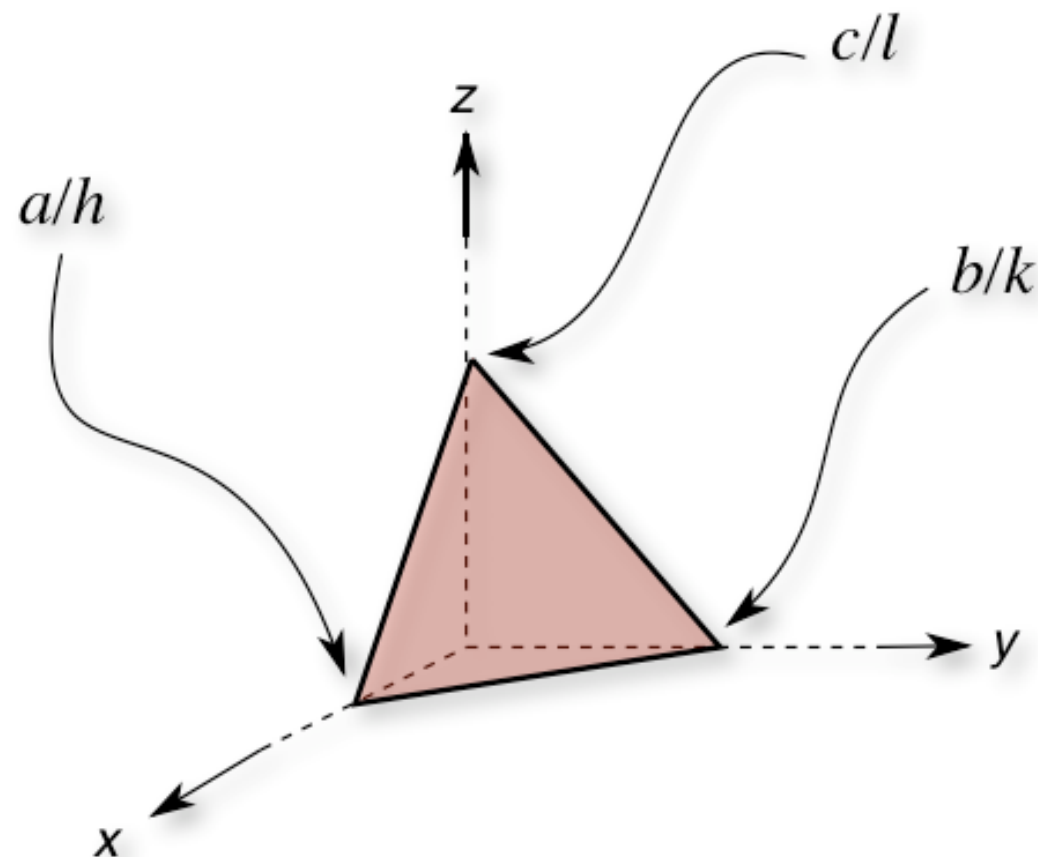
Miller indices ($h\ k\ l$) reciprocal (invers) of 1st intersection with x, y, z axes:

Lattice planes are described using Miller indices ($h\ k\ l$) where the *first* plane away from the origin intersects the x, y, z axes at distances:

a/h on the x axis
 b/k on the y axis
 c/l on the z axis

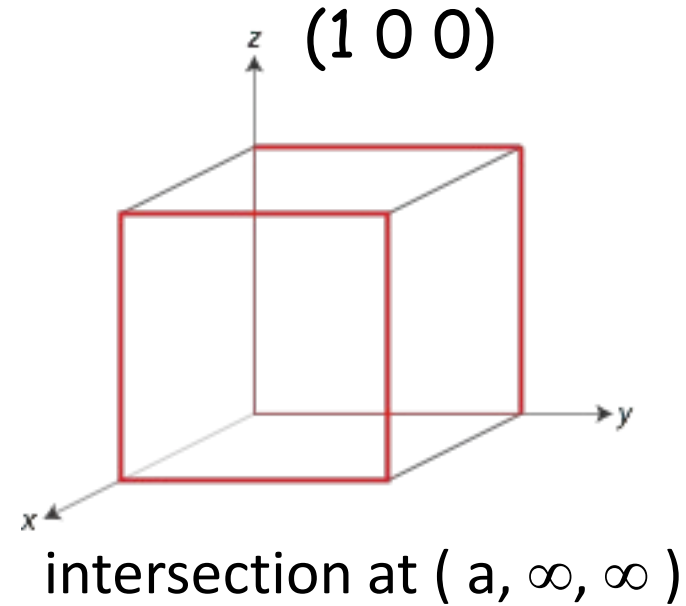
Example: Draw (hkl) = (111) plane

$$a/h = 1, b/k = 1, c/l = 1$$

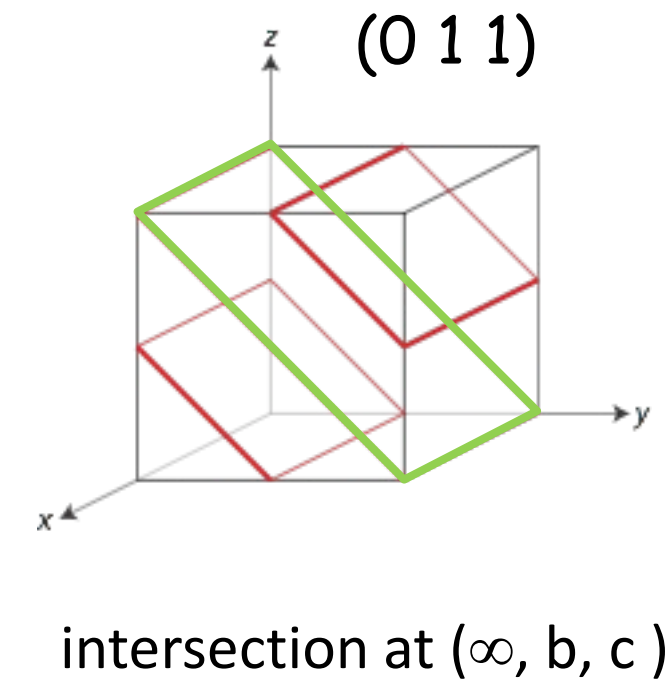
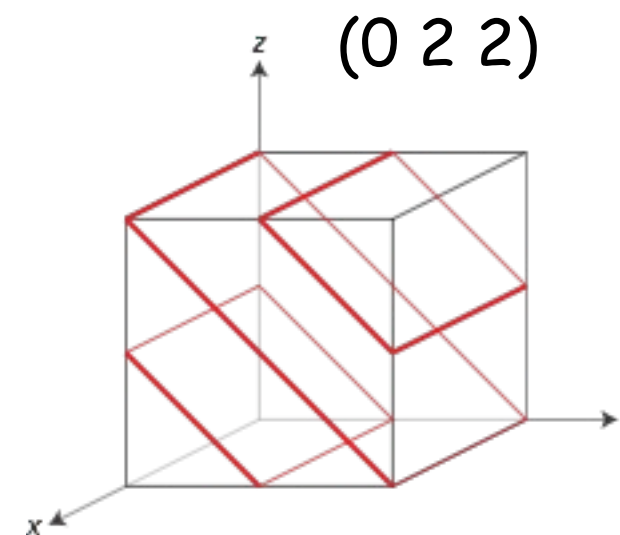
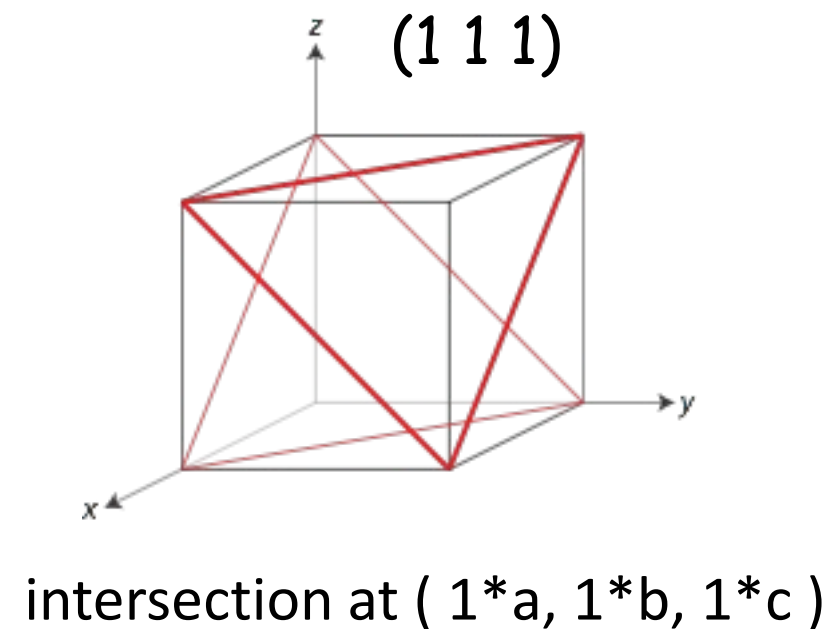


Crystallography - lattice planes

Sets of planes intersecting the unit cell - examples:



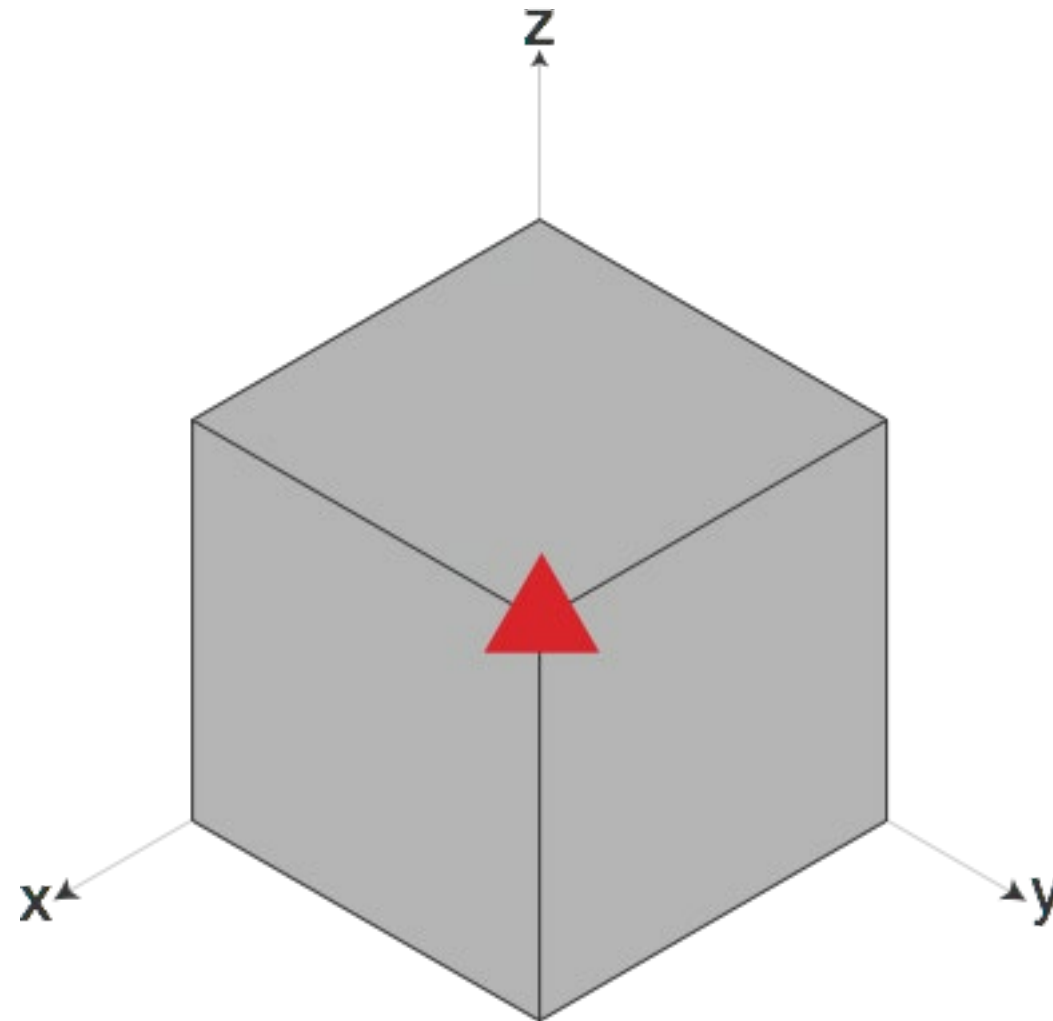
intersection with coordinate axes (x,y,z) on
 a/h on the x axis
 b/k on the y axis
 c/l on the z axis



Lattice planes and symmetry

Lattice planes in a crystal related by the crystal symmetry

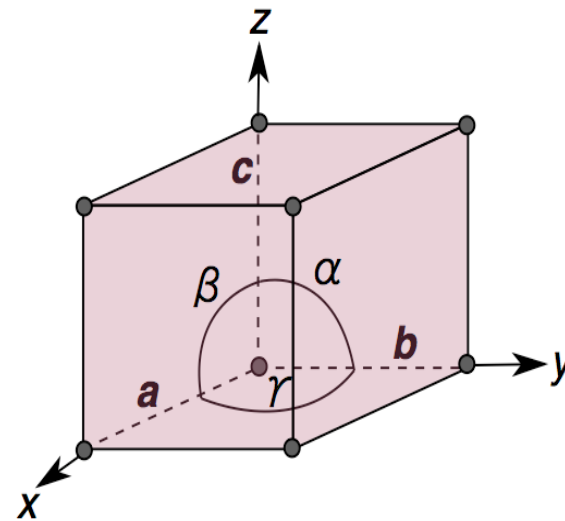
For example, in cubic lattices the 3-fold rotation axis on the $[1\ 1\ 1]$ body diagonal relates the planes $(1\ 0\ 0)$, $(0\ 1\ 0)$, $(0\ 0\ 1)$:



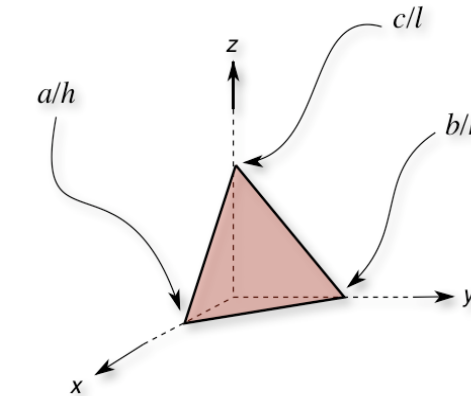
Set of planes $\{1\ 0\ 0\} = (1\ 0\ 0), (0\ 1\ 0), (0\ 0\ 1), (-1\ 0\ 0), (0\ -1\ 0), (0\ 0\ -1)$

Introduction to Crystallography

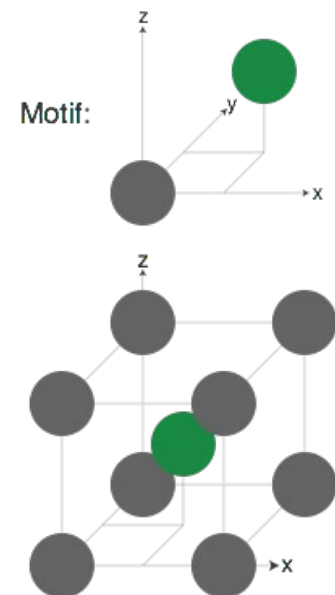
UNIT CELL



[UVW]: lattice vectors, direction, “zone axes”
 (hkl): lattice planes (Miller indices)
 {khkl}: equivalent lattice planes



unit cell
 decoration
 MOTIF



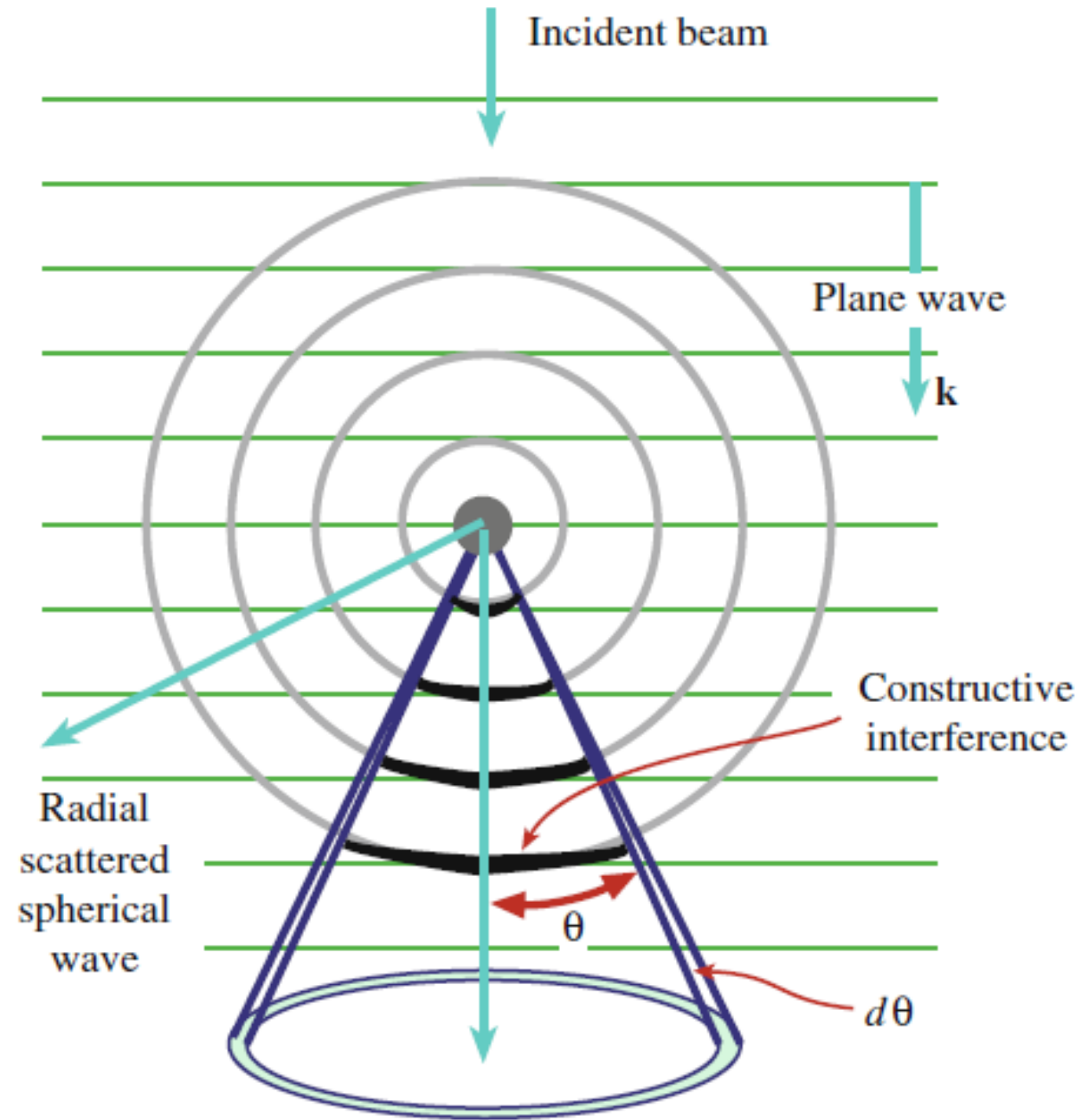
The 7 Crystal systems	The 14 Bravais lattices			
triclinic	P $\alpha, \beta, \gamma \neq 90^\circ$			
monoclinic	P $\alpha \neq 90^\circ$ $\beta, \gamma = 90^\circ$	C $\alpha \neq 90^\circ$ $\beta, \gamma = 90^\circ$		
orthorhombic	P $a \neq b \neq c$	C $a \neq b \neq c$	I $a \neq b \neq c$	F $a \neq b \neq c$

Bravais LATTICE

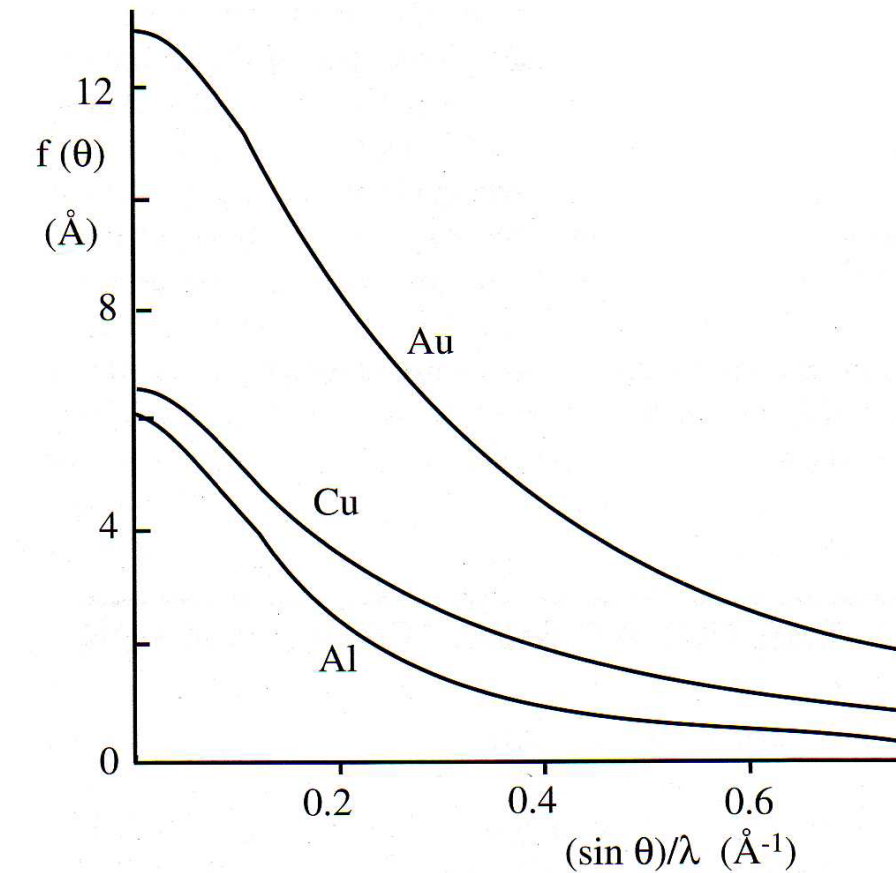
Elastic scattering theory

Scattering theory - atomic scattering factor $f(\theta)$ single atom

Consider coherent elastic scattering of electrons from atom



$$\psi(\mathbf{r}) = e^{ikz} + f(\theta) \frac{e^{ikr}}{r},$$



Atomic scattering factor for electrons

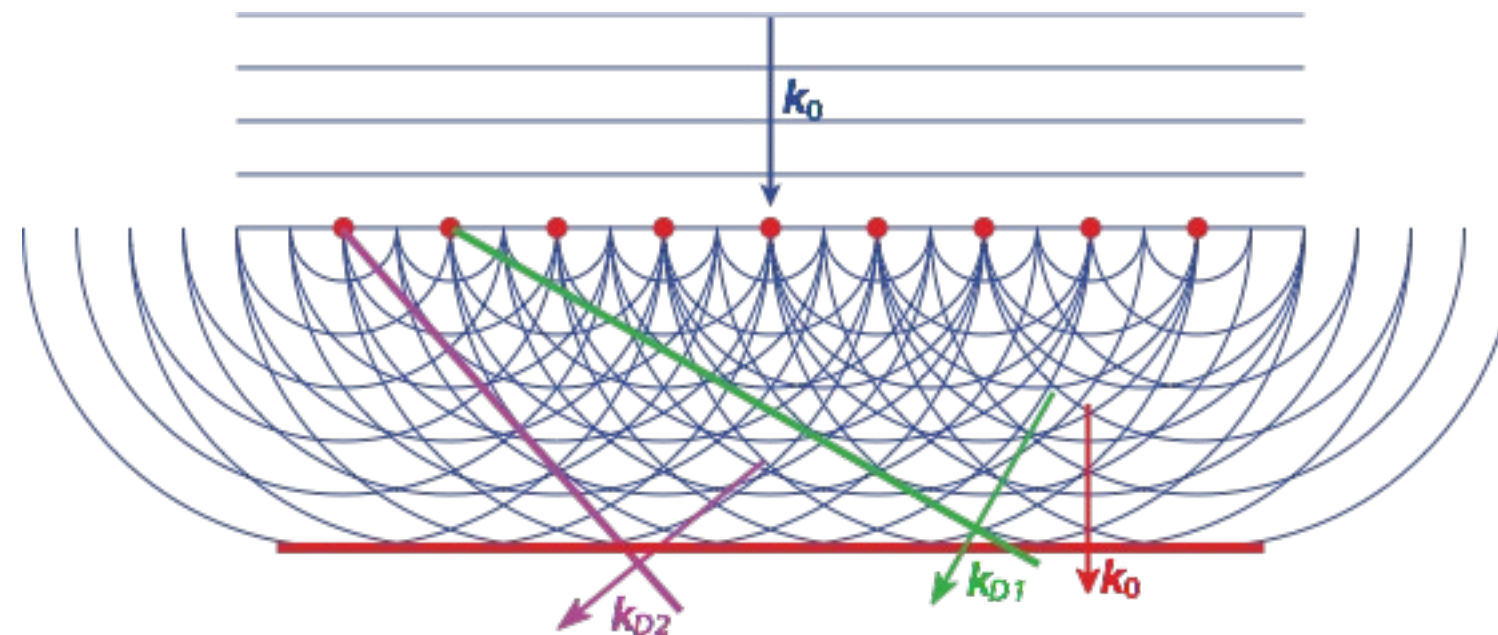
$$f(\theta) = \frac{\left(1 + \frac{E_0}{m_0 c^2}\right)}{8\pi} \left(\frac{\lambda}{\sin \frac{\theta}{2}}\right) (Z - f_x)$$

The **Mott-Bethe formula** is used to calculate electron form factors from X-ray form factors (f_x)

Scattering theory - Huygen's principle

Periodic array of scattering centres (atoms)

Plane electron wave generates secondary wavelets



Secondary wavelets interfere =>
strong direct beam and multiple orders of diffracted beams from constructive interference

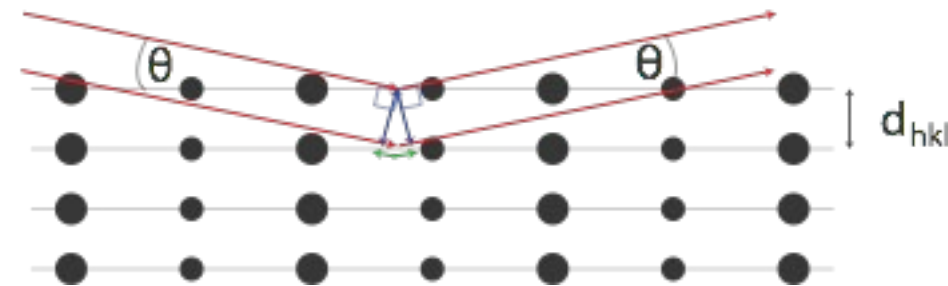
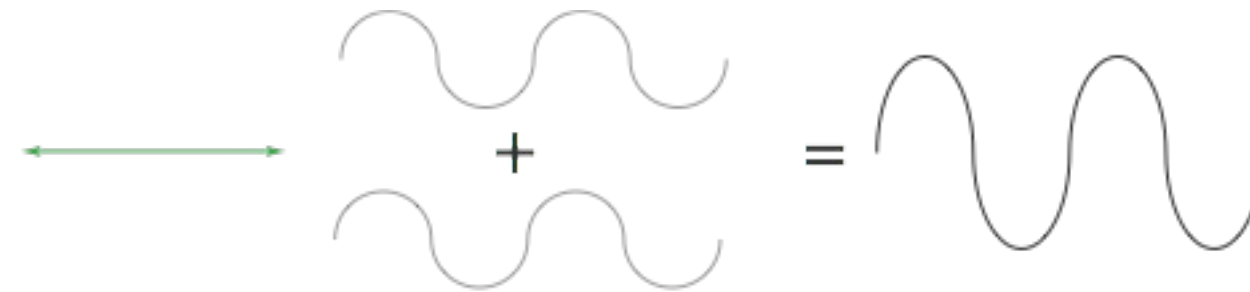
Atoms closer together => scattering angles greater

=> **Reciprocity!**

Diffraction theory - Bragg law

Path difference between reflection from planes distance d_{hkl} apart = $2d_{hkl}\sin\theta$

=> Bragg law:
 $n\lambda = 2d_{hkl}\sin\theta$



Electron diffraction: $\lambda \sim 0.001 \text{ nm}$

therefore: $\lambda \ll d_{hkl}$

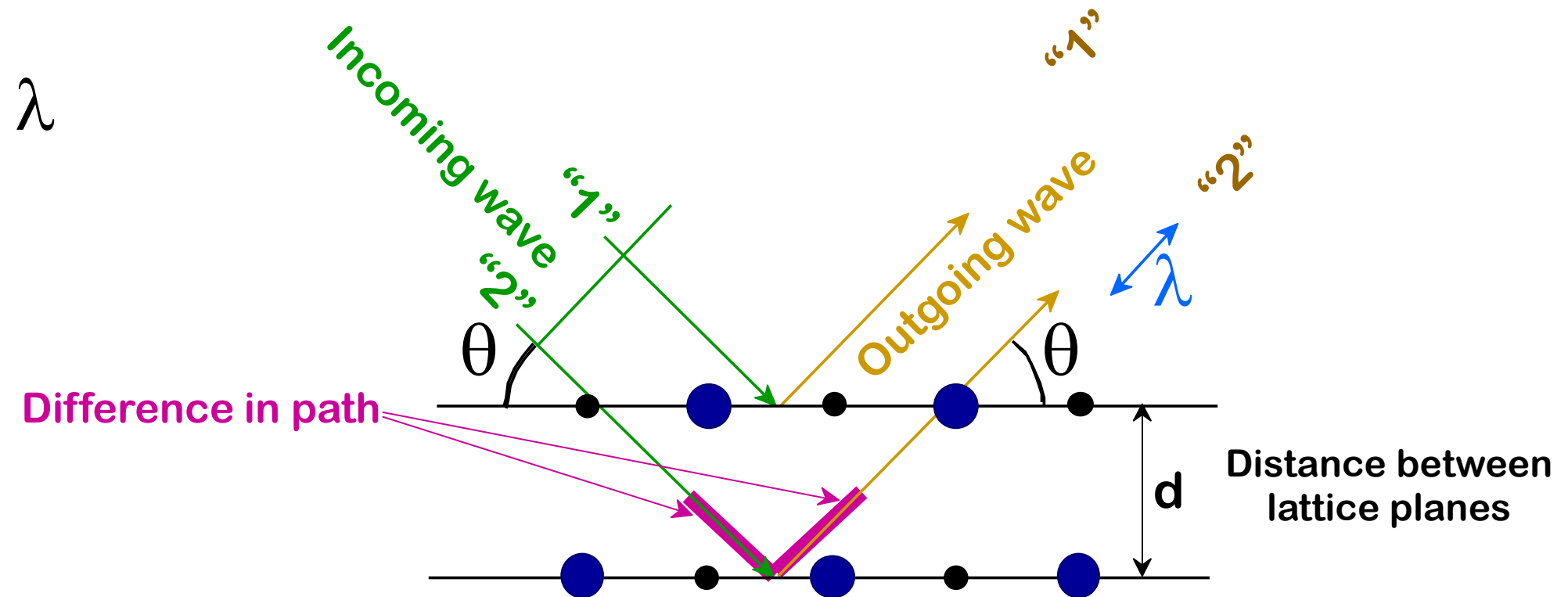
=> small angle approximation: $n\lambda \approx 2d_{hkl}\theta$

Reciprocity: scattering angle $\theta \sim d_{hkl}^{-1}$

Bragg's law

$$2 \sin\theta d_{hkl} = n \lambda$$

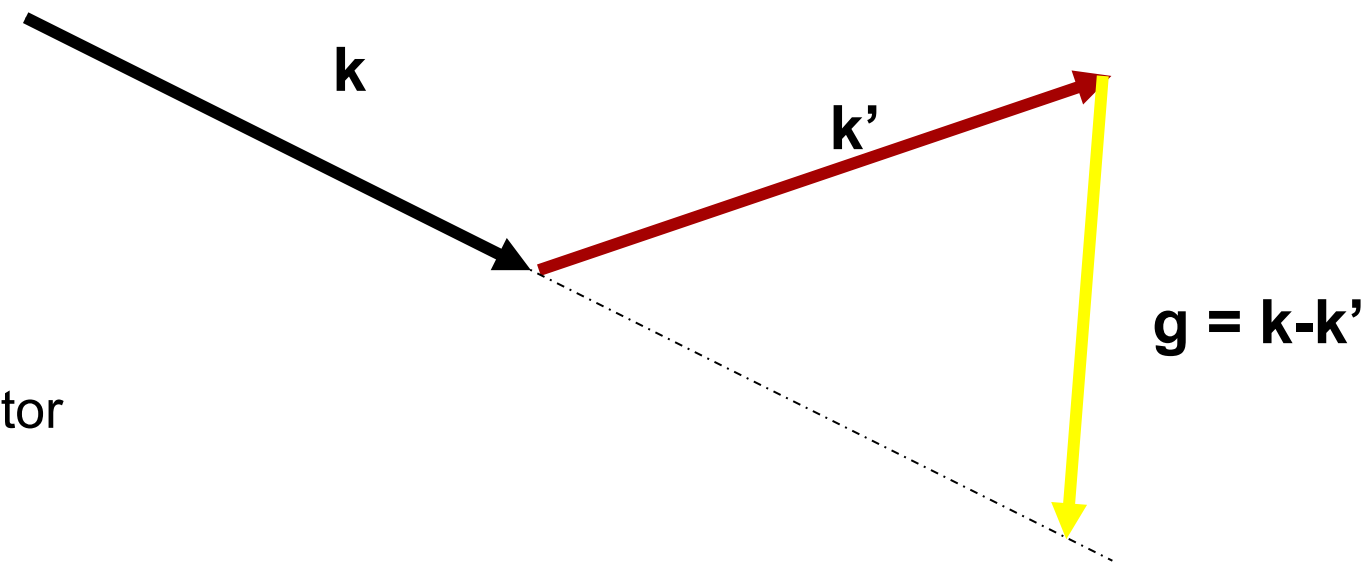
$$d_{hkl} = n \lambda / 2 \sin\theta$$



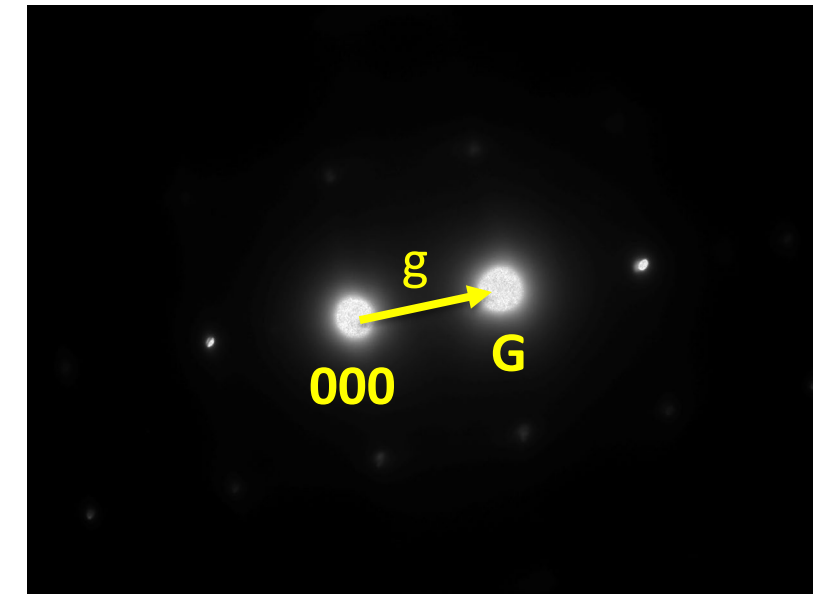
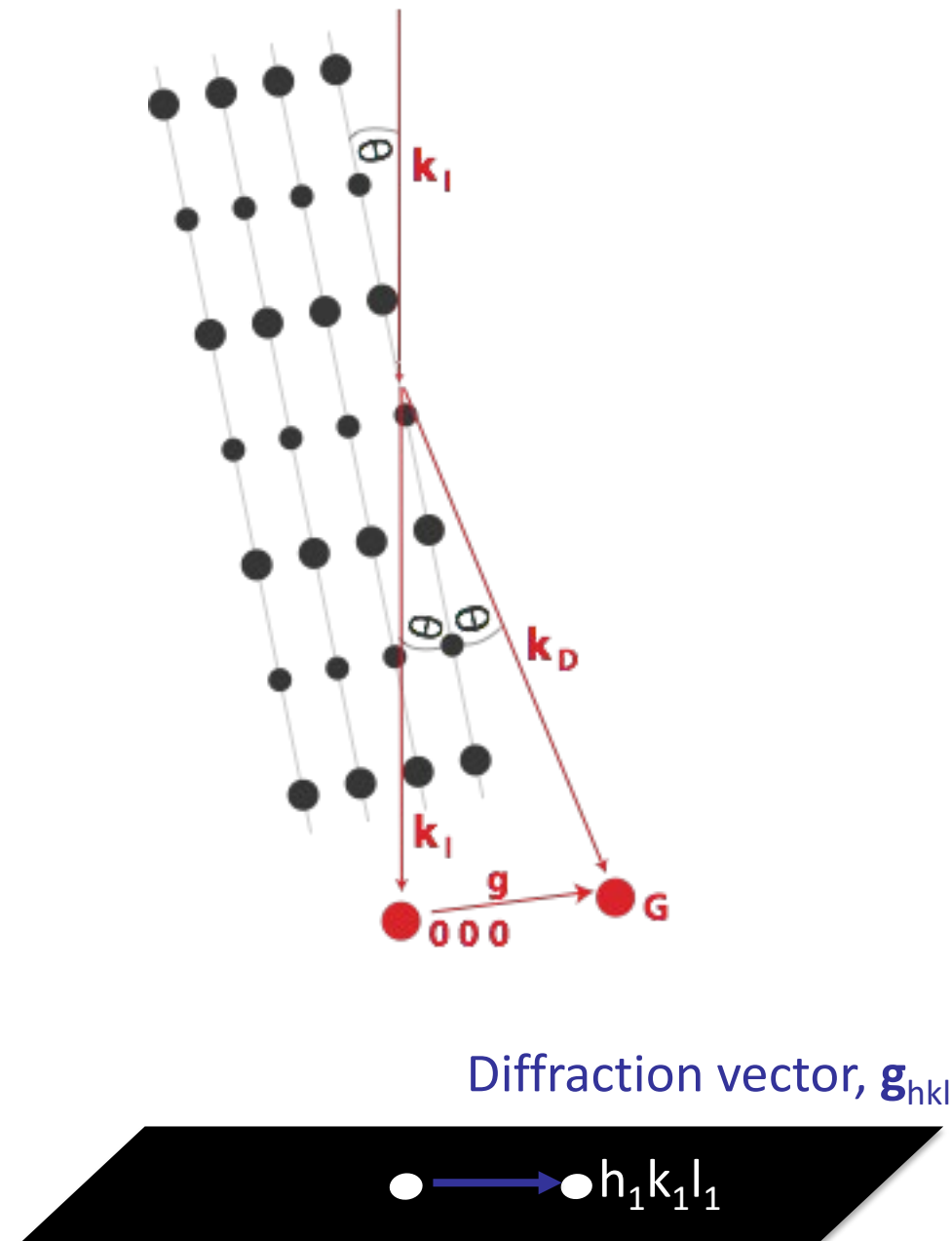
Elastic diffraction

$$|k| = |k'|$$

Periodic arrangement of lattice planes:
 $\mathbf{g}$: « diffraction vector », reciprocal lattice vector



Diffraction theory - 2-beam condition



Observed diffraction pattern

$$2\theta_B = \frac{\lambda}{d_{hkl}}$$

$$|g_{hkl}| = \frac{1}{d_{hkl}}$$

2-beam condition: strong scattering from single set of planes

Multi-beam scattering condition

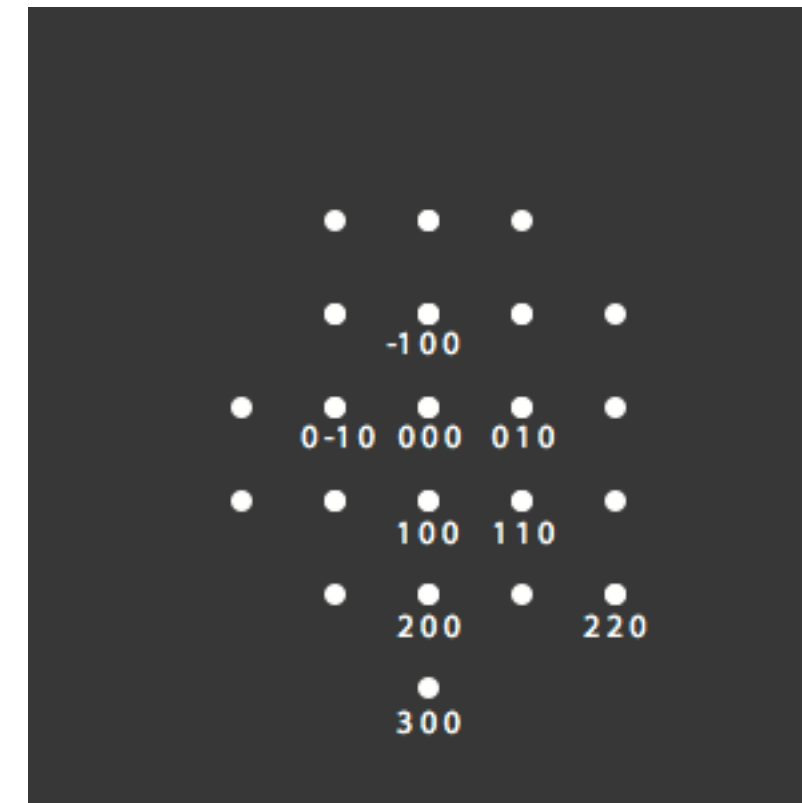
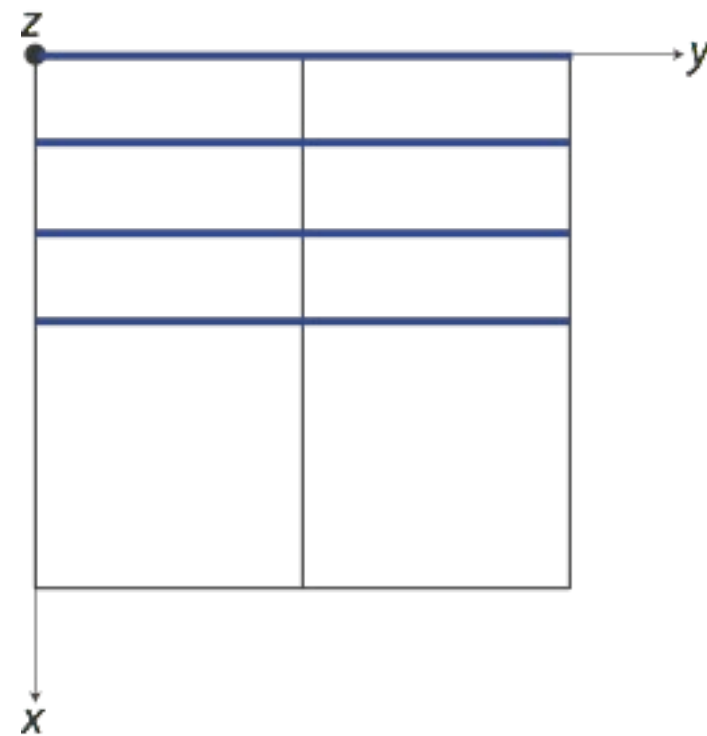
Electron beam parallel to low-index crystal orientation $[U\ V\ W]$ = zone axis

Crystal "viewed down" zone axis is like diffraction grating with planes parallel to e-beam

In diffraction pattern obtain spots perpendicular to plane orientation

Example: primitive cubic with e-beam parallel to $[0\ 0\ 1]$ zone axis

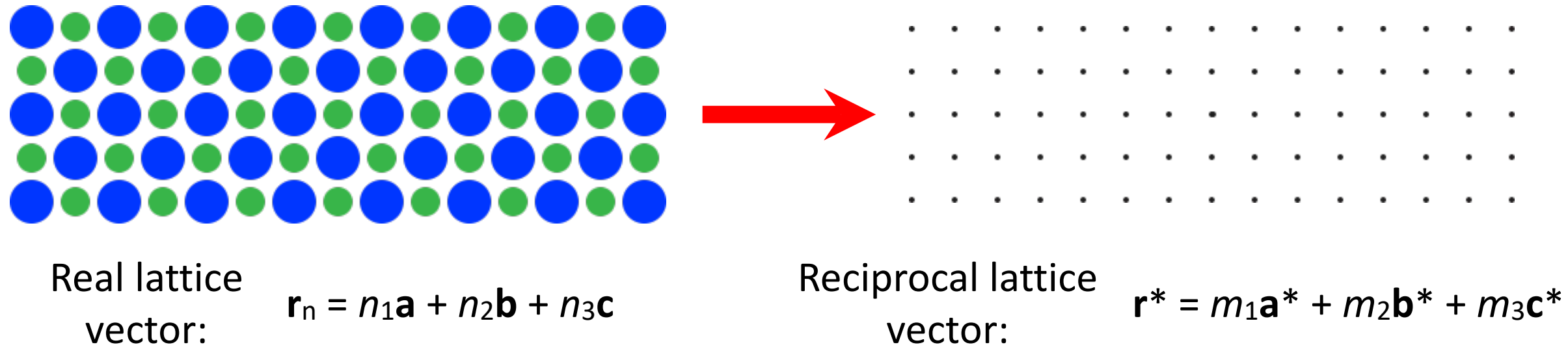
2 x 2 unit cells



Note reciprocal relationship: smaller plane spacing $\Rightarrow$ larger indices $(h\ k\ l)$
& greater scattering angle on diffraction pattern from $(0\ 0\ 0)$ direct beam

The reciprocal lattice

In diffraction we are working in “reciprocal space”; useful to transform the crystal lattice in to a “reciprocal lattice” that represents the crystal in reciprocal space:



where:

$$\mathbf{a}^* \cdot \mathbf{b} = \mathbf{a}^* \cdot \mathbf{c} = \mathbf{b}^* \cdot \mathbf{c} = \mathbf{b}^* \cdot \mathbf{a} = \mathbf{c}^* \cdot \mathbf{a} = \mathbf{c}^* \cdot \mathbf{b} = 0$$

In orthogonal crystal structure:

$$\mathbf{a}^* \cdot \mathbf{a} = \mathbf{b}^* \cdot \mathbf{b} = \mathbf{c}^* \cdot \mathbf{c} = 1$$

$$[\mathbf{a}]: \text{nm}, [\mathbf{a}^*]: \text{nm}^{-1}$$

$\mathbf{a}^*$ perpendicular to $\mathbf{b}$ and $\mathbf{c}$
= normal of bc plane

i.e. $\mathbf{a}^* = (\mathbf{b} \wedge \mathbf{c}) / V_c$ V_c : volume of unit cell

$$|\mathbf{a}^*| = 1 / d_{100}$$

For scattering from plane $(h \ k \ l)$ the diffraction vector:

$$\mathbf{g}_{hkl} = h\mathbf{a}^* + k\mathbf{b}^* + l\mathbf{c}^*$$

Plane spacing: $d_{hkl} = \frac{1}{|\mathbf{g}_{hkl}|}$

Ewald sphere

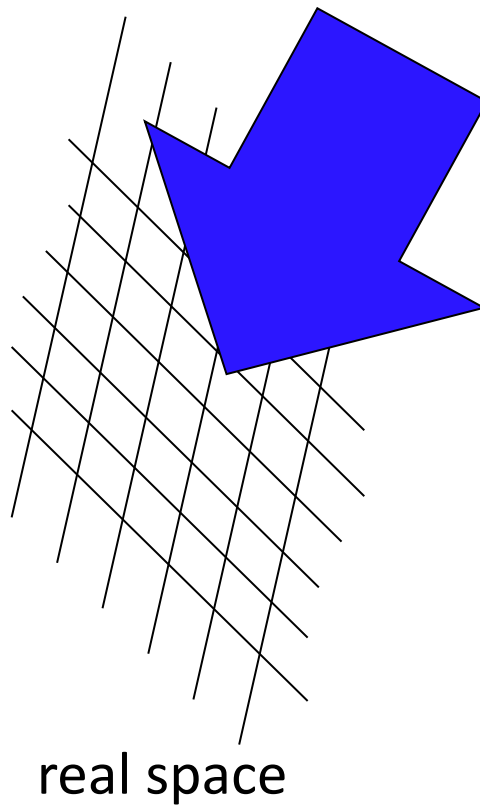
A vector in reciprocal space:

$$\mathbf{g}_{hkl} = h \mathbf{a}^* + k \mathbf{b}^* + l \mathbf{c}^*$$

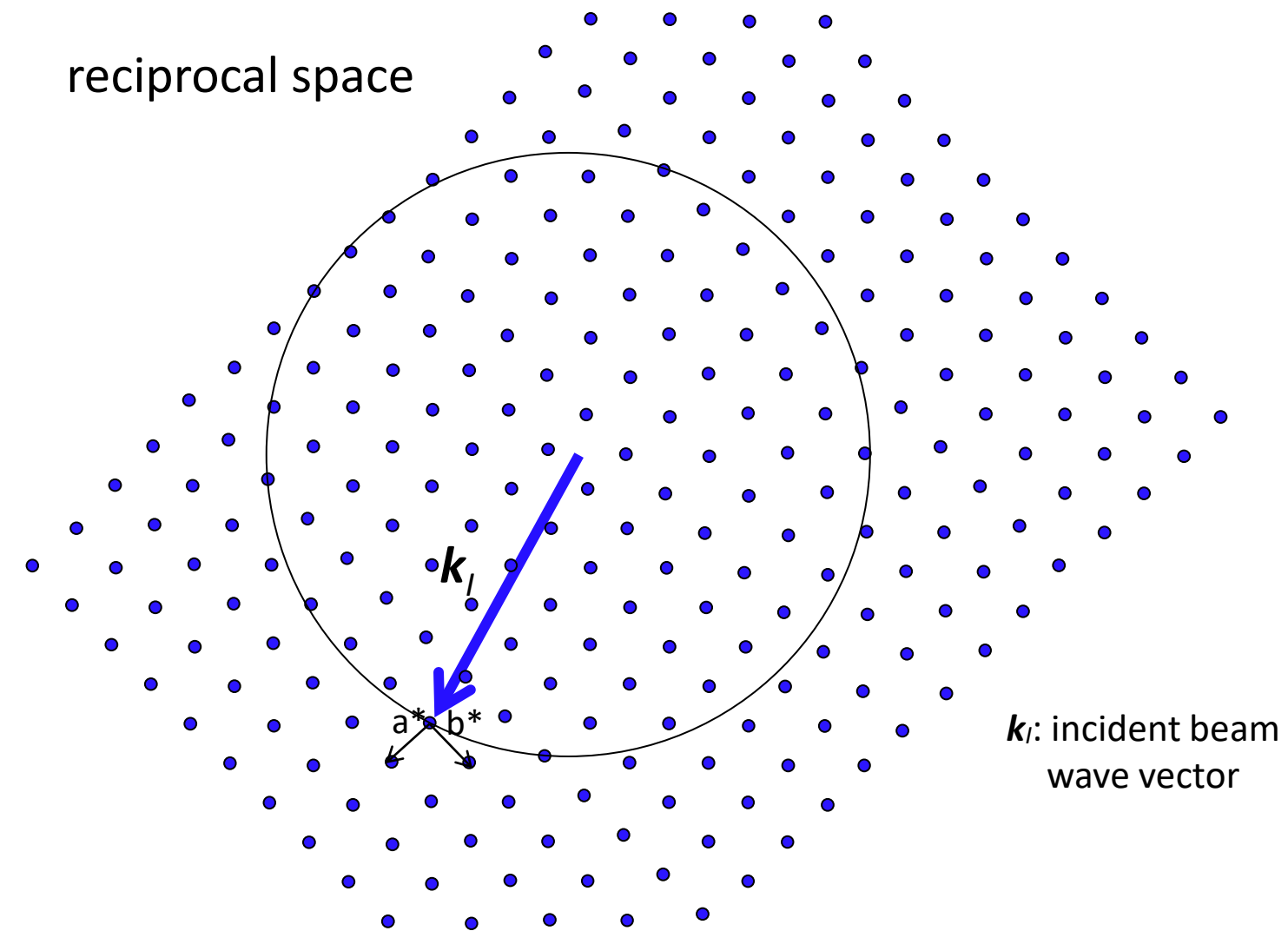
diffraction if :

$$\mathbf{k}_i - \mathbf{k}_D = \mathbf{g} \quad \text{and} \quad |\mathbf{k}_i| = |\mathbf{k}_D|$$

Bragg and elastic scattering



Bragg: $d_{hkl} = \lambda/2 \sin\theta = 1/|\mathbf{g}|$



Reciprocal space: sphere radius $1/\lambda$ represents possible scattering wave vectors intersecting reciprocal space
Ewald sphere

Electron diffraction: radius of sphere very large compared to reciprocal lattice
 $\Rightarrow$ sphere circumference almost flat

Ewald sphere

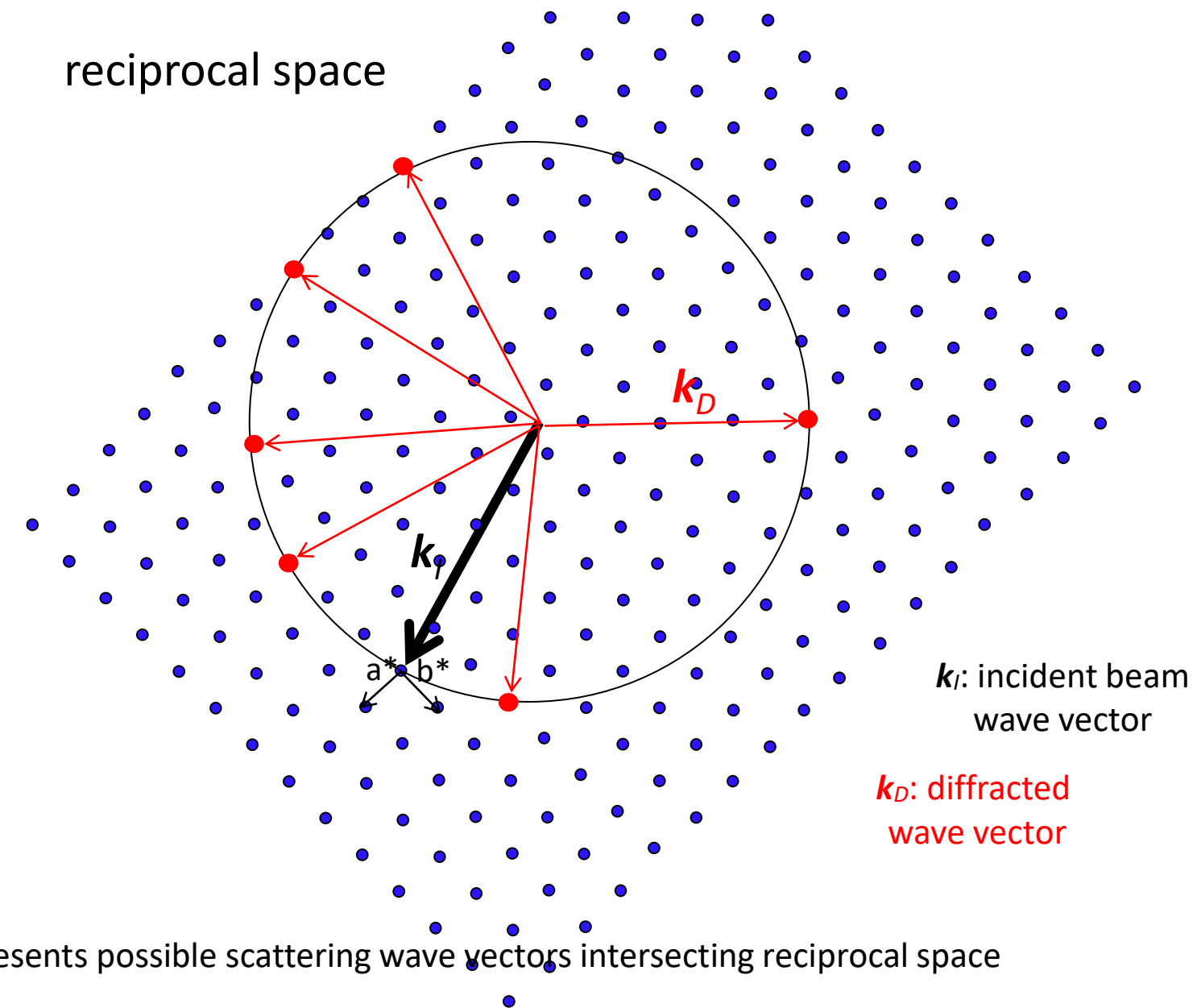
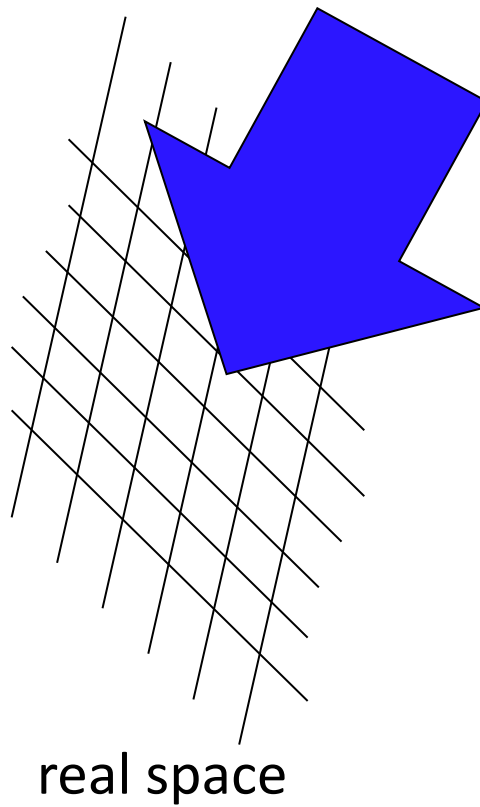
A vector in reciprocal space:

$$\mathbf{g}_{hkl} = h \mathbf{a}^* + k \mathbf{b}^* + l \mathbf{c}^*$$

diffraction if :

$$\mathbf{k}_i - \mathbf{k}_D = \mathbf{g} \quad \text{and} \quad |\mathbf{k}_i| = |\mathbf{k}_D|$$

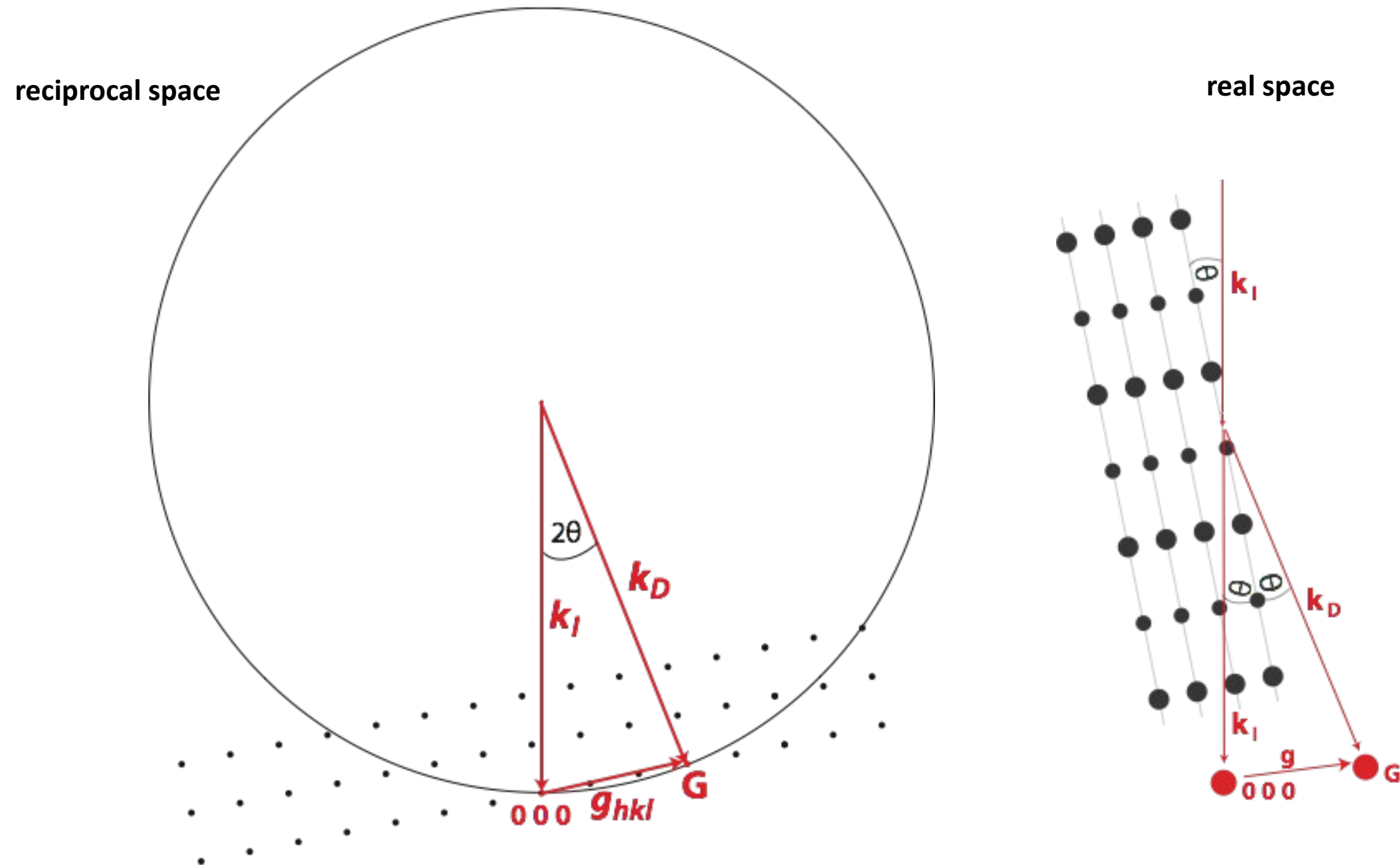
Bragg and elastic scattering



Reciprocal space: sphere radius $1/\lambda$ represents possible scattering wave vectors intersecting reciprocal space

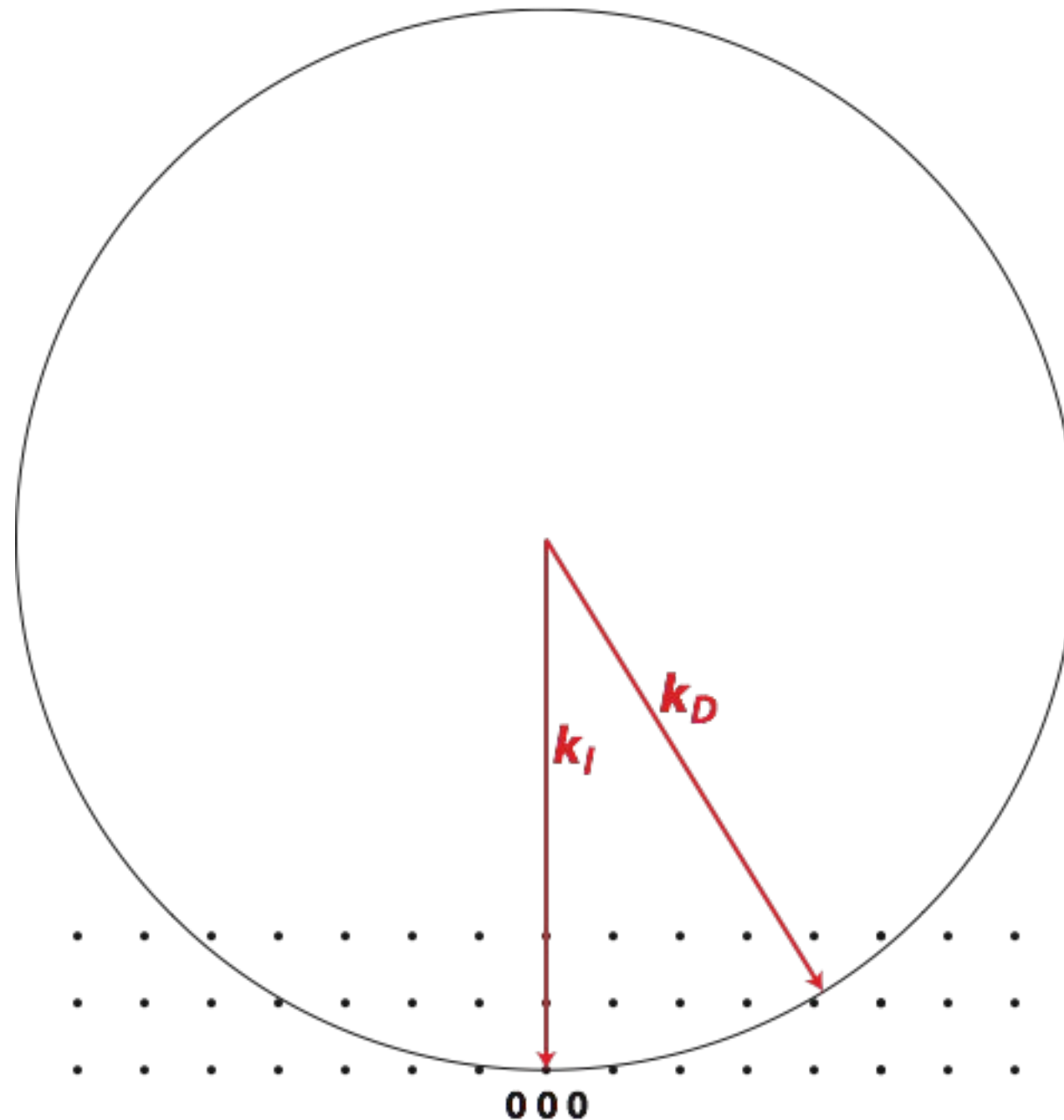
Electron diffraction: radius of sphere very large compared to reciprocal lattice
=> sphere circumference almost flat

Ewald sphere in 2-beam condition



2-beam condition with one strong Bragg reflection corresponds to Ewald sphere intersecting one reciprocal lattice point

Ewald sphere and multi-beam scattering



Assume reciprocal lattice points are infinitely small

With crystal oriented on zone axis, Ewald sphere may not intersect reciprocal lattice points

However, we see strong diffraction from many planes in this condition

Because reciprocal lattice points have size and shape!

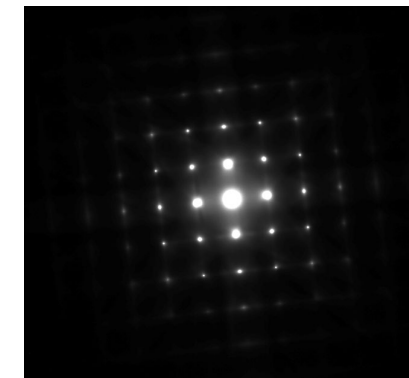
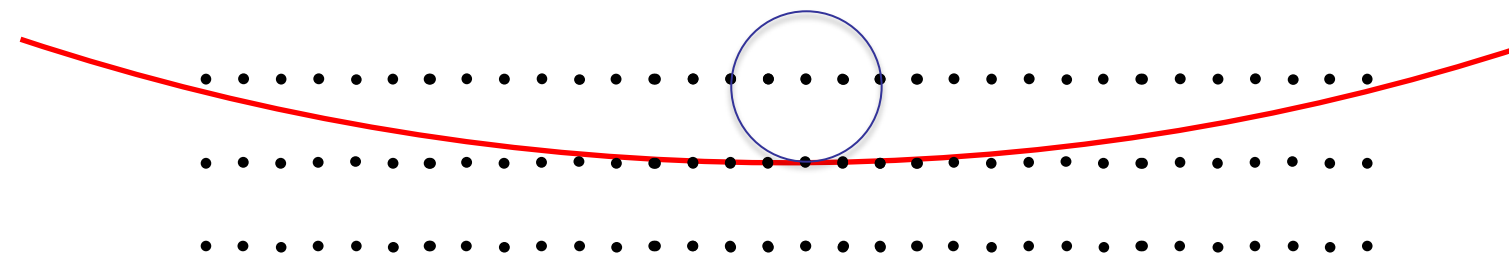
the reciprocal space

Almost flat Ewald sphere in electron diffraction

there are two main differences between x-ray and electron diffraction

Electron

X-ray



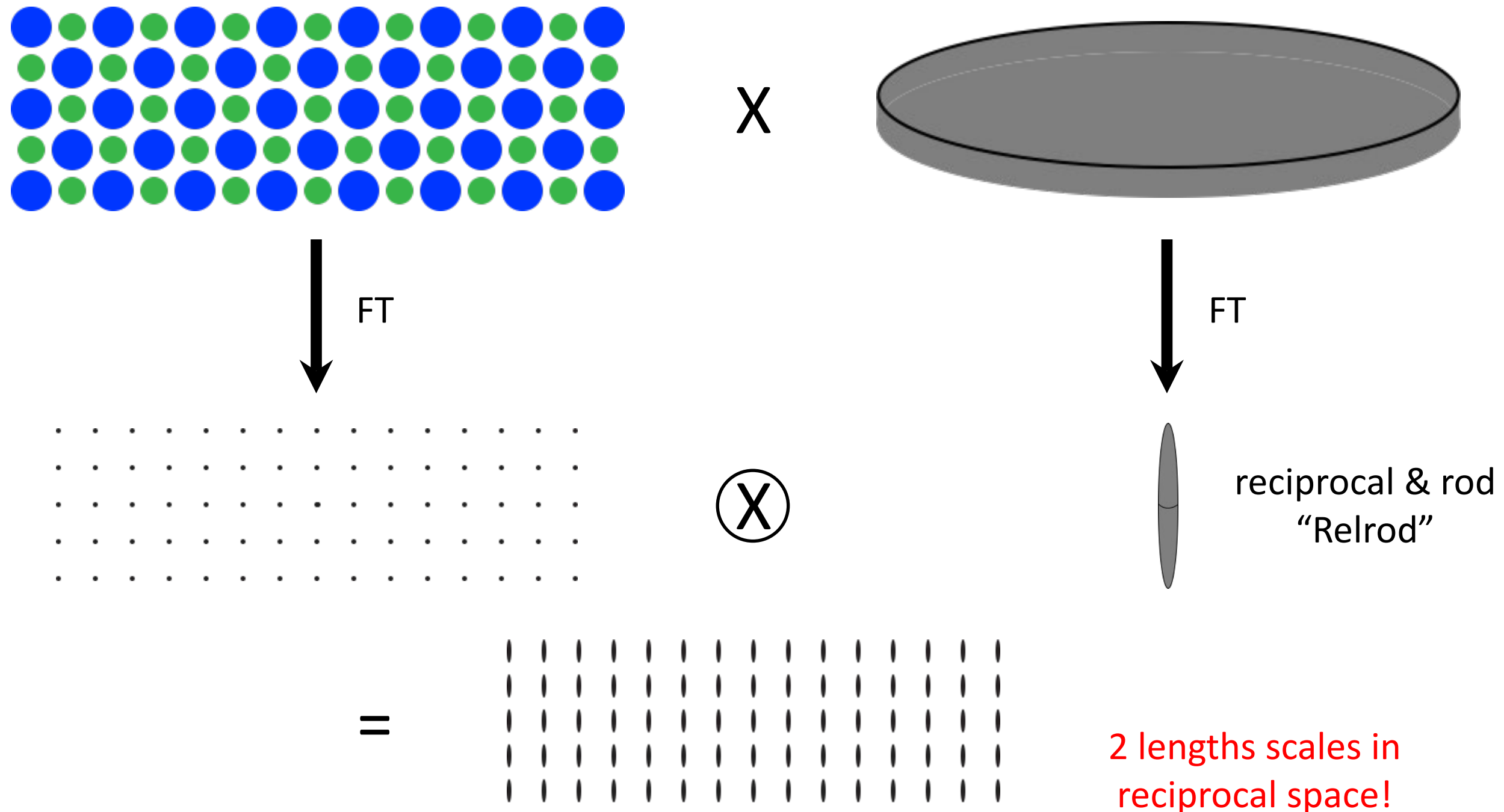
observed diffraction pattern

The energy of the electron beam (typically 80-300 keV) is far higher than that of a typical x-ray beam and so the wavelength of radiation is much smaller.

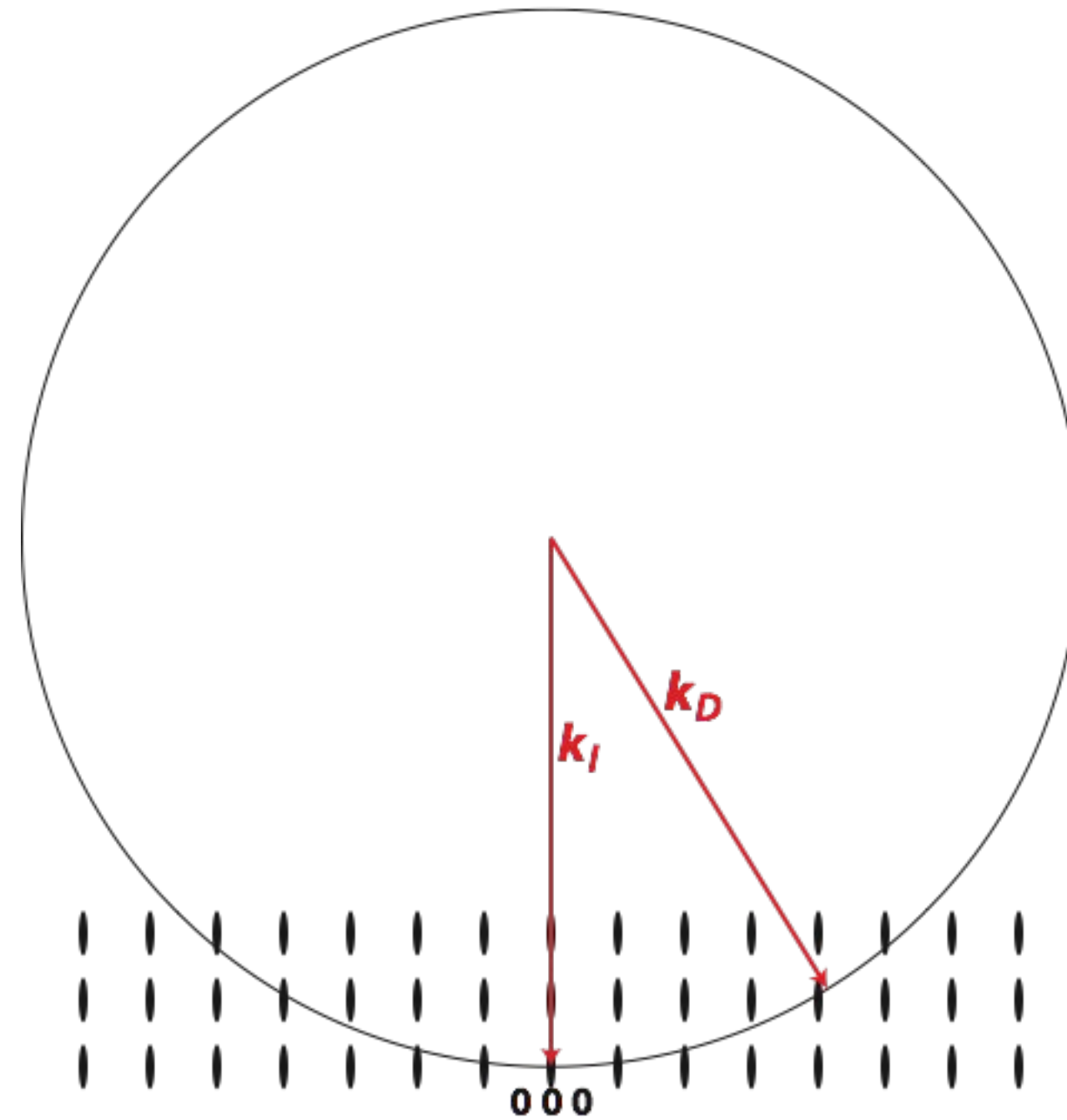
This means that the Ewald sphere radius ($= 1/\lambda$) is much larger for electron diffraction:
almost flat Ewald sphere

Fourier transforms and reciprocal lattice

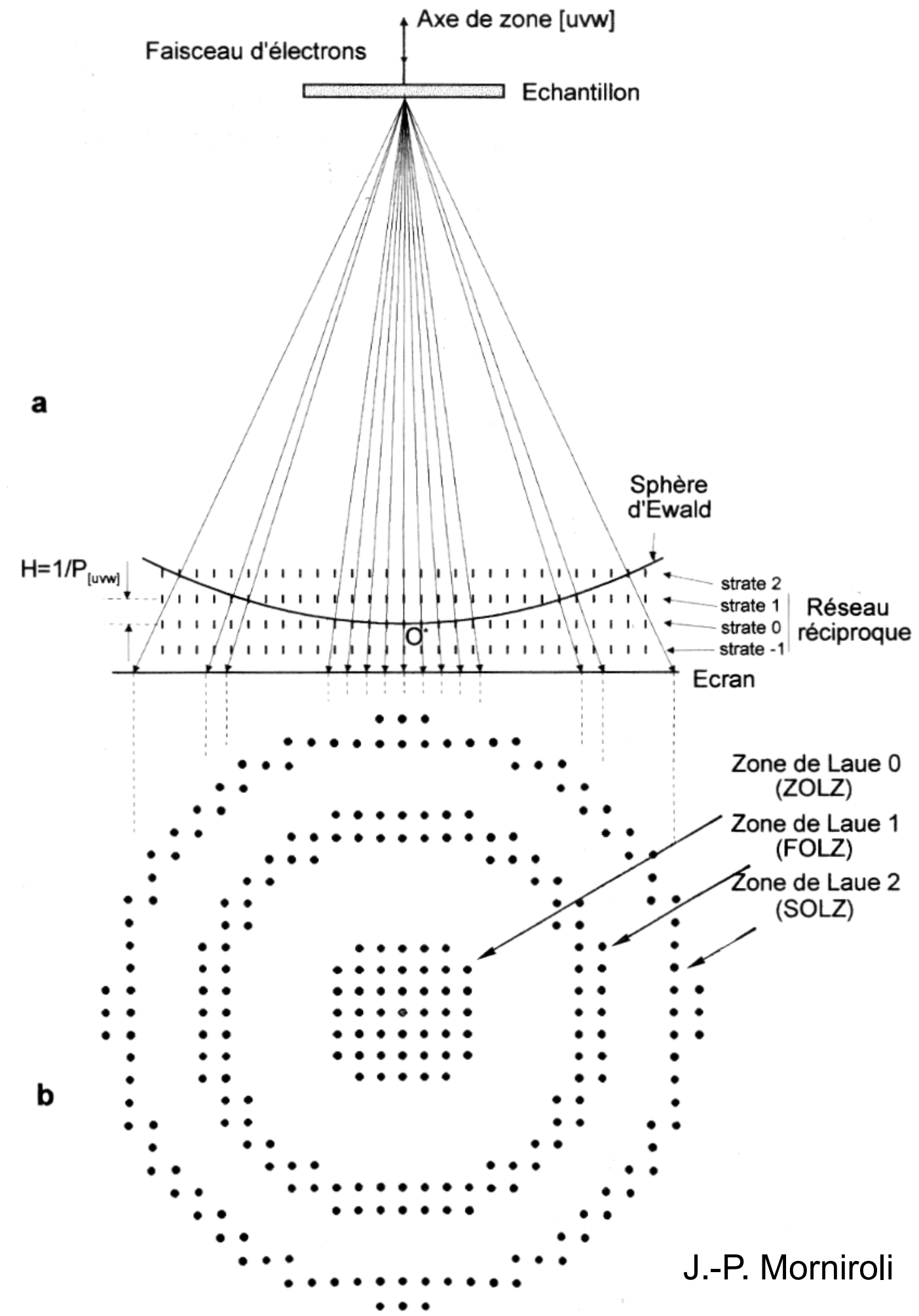
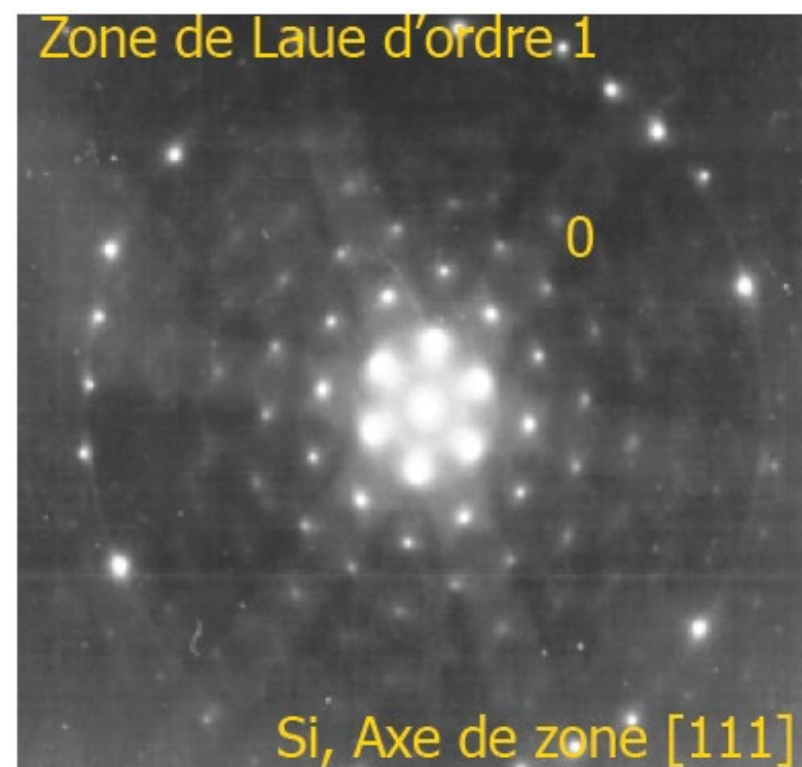
Real lattice is not infinite, but is bound disc of material with diameter of selected area aperture and thickness of specimen - i.e. thin disc of material



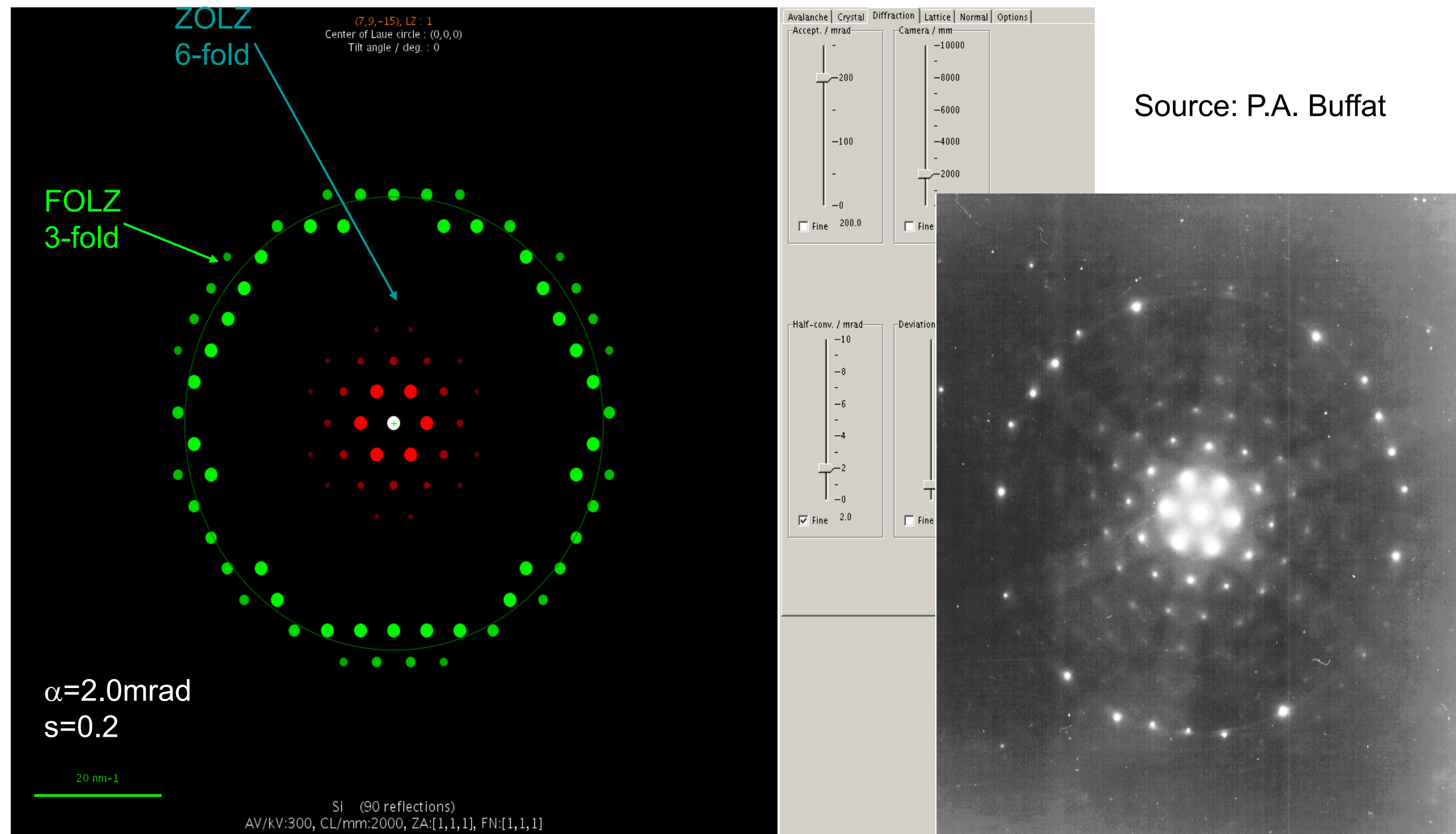
Ewald sphere intersects Relrods



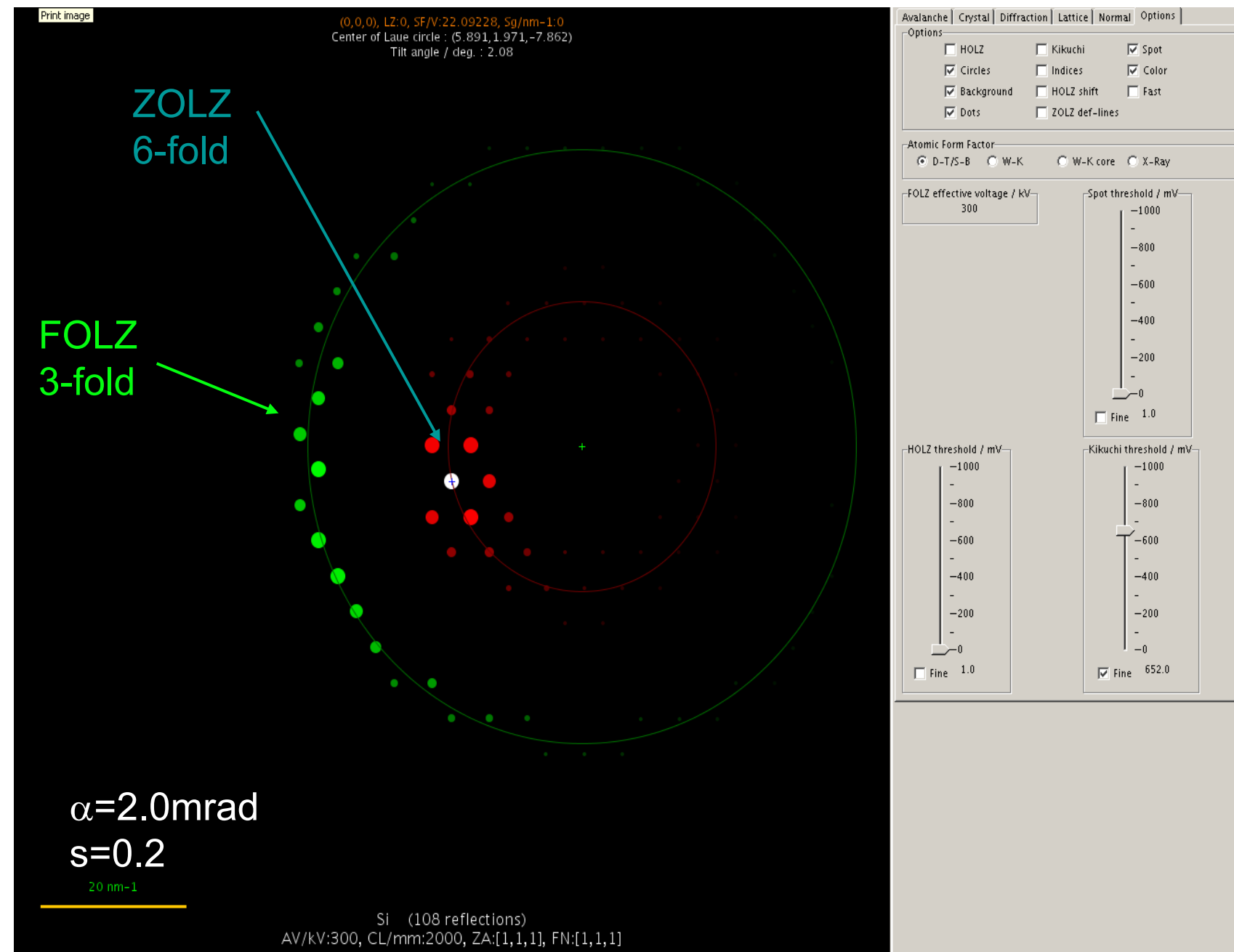
Laue zone "higher order" Laue zones



Sphère d'Ewald : Laue Zones in Si [111], (ZOLZ+FOLZ)



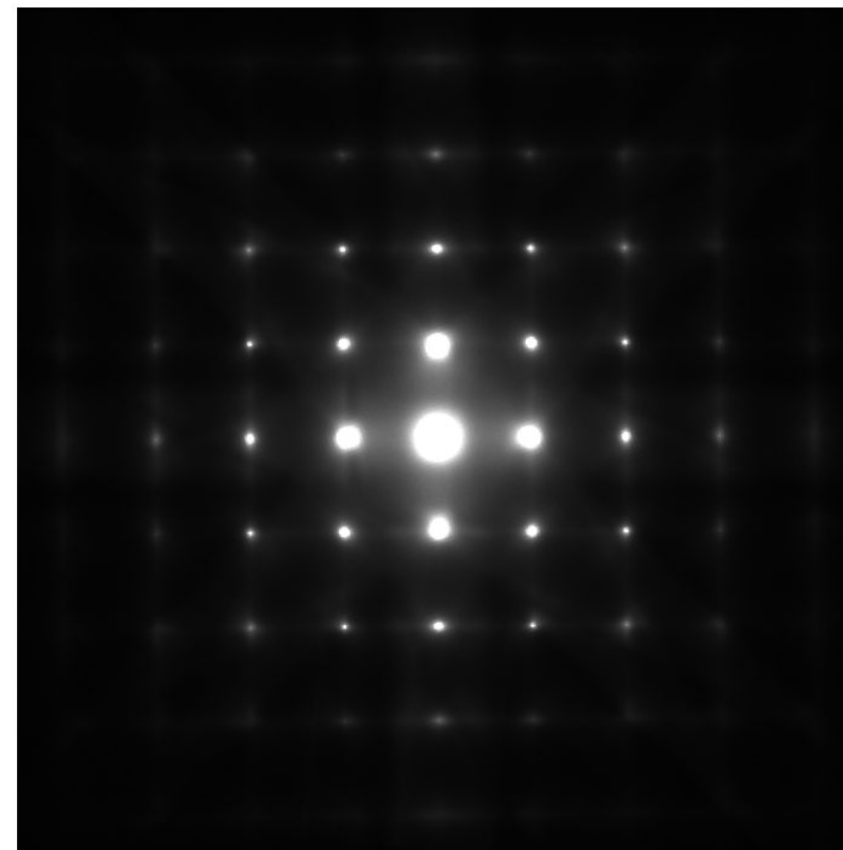
Sphère d'Ewald : Laue Zones (ZOLZ+FOLZ) échantillon incliné



the reciprocal space tilting away from zone axis

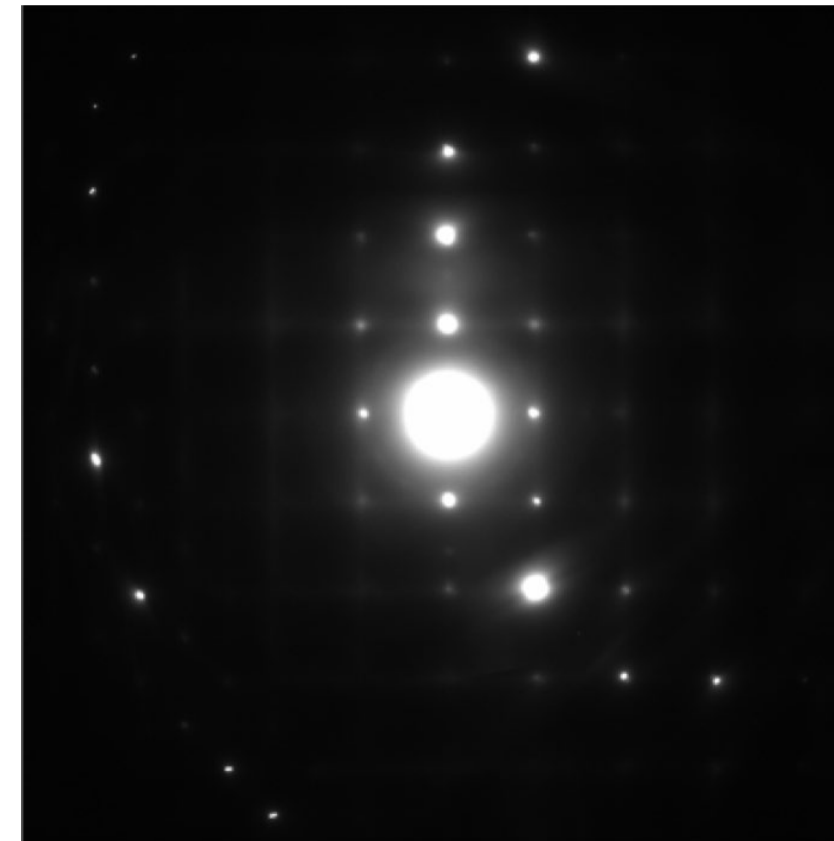
Relaxation of Bragg condition due to the "rod" shape of reflections:

When we tilt the specimen away from a zone axis we still see reflections for planes that are not in exact Bragg condition. The diffractions spots will not "move" with tilt. They will "fade away" as the Ewald sphere intersections change



perfectly on zone axis

Specimen tilt



slightly off zone axis

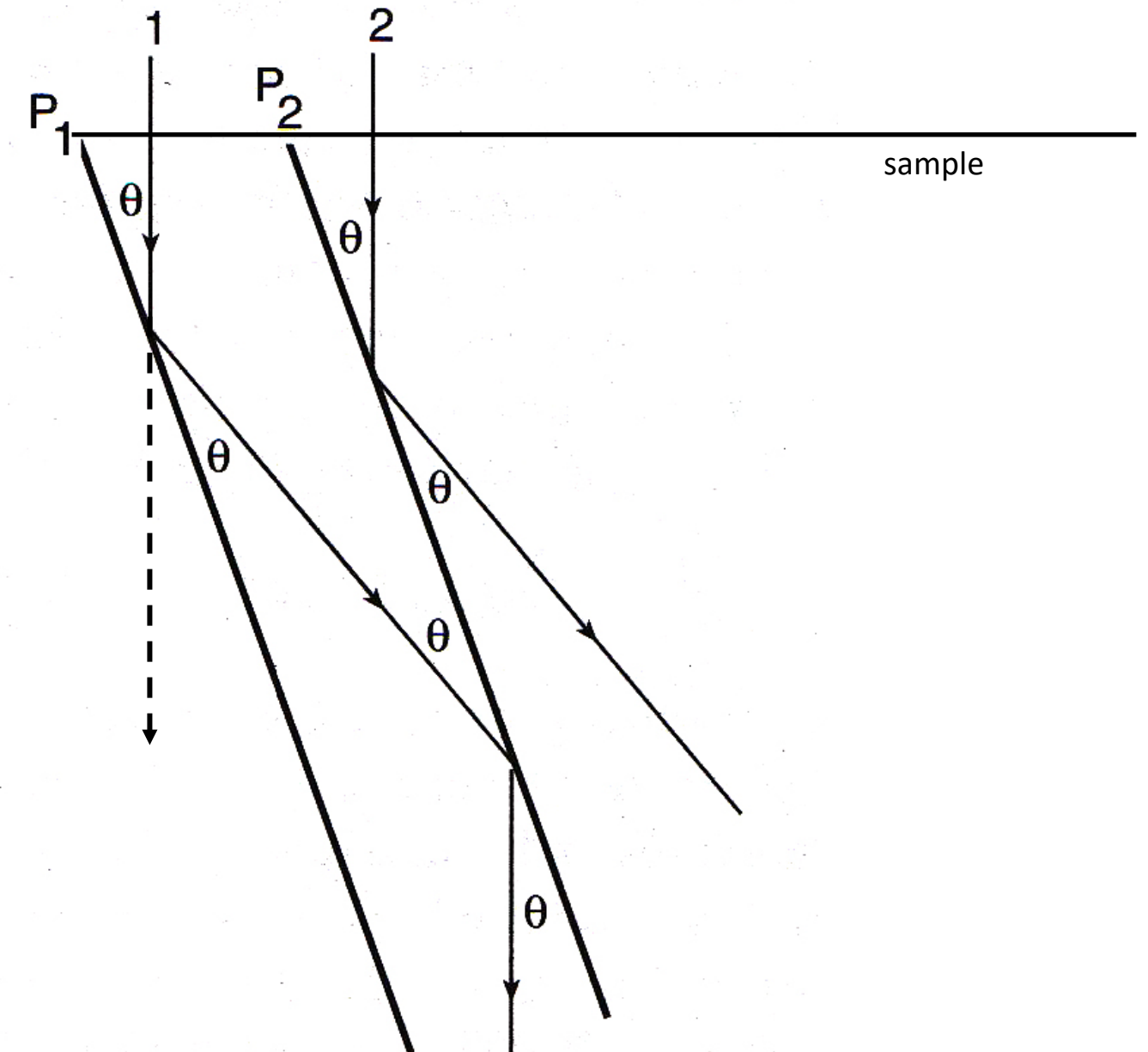
Dynamical scattering

For interpretation of intensities in diffraction pattern, single scattering would be ideal
- i.e. "kinematical" scattering

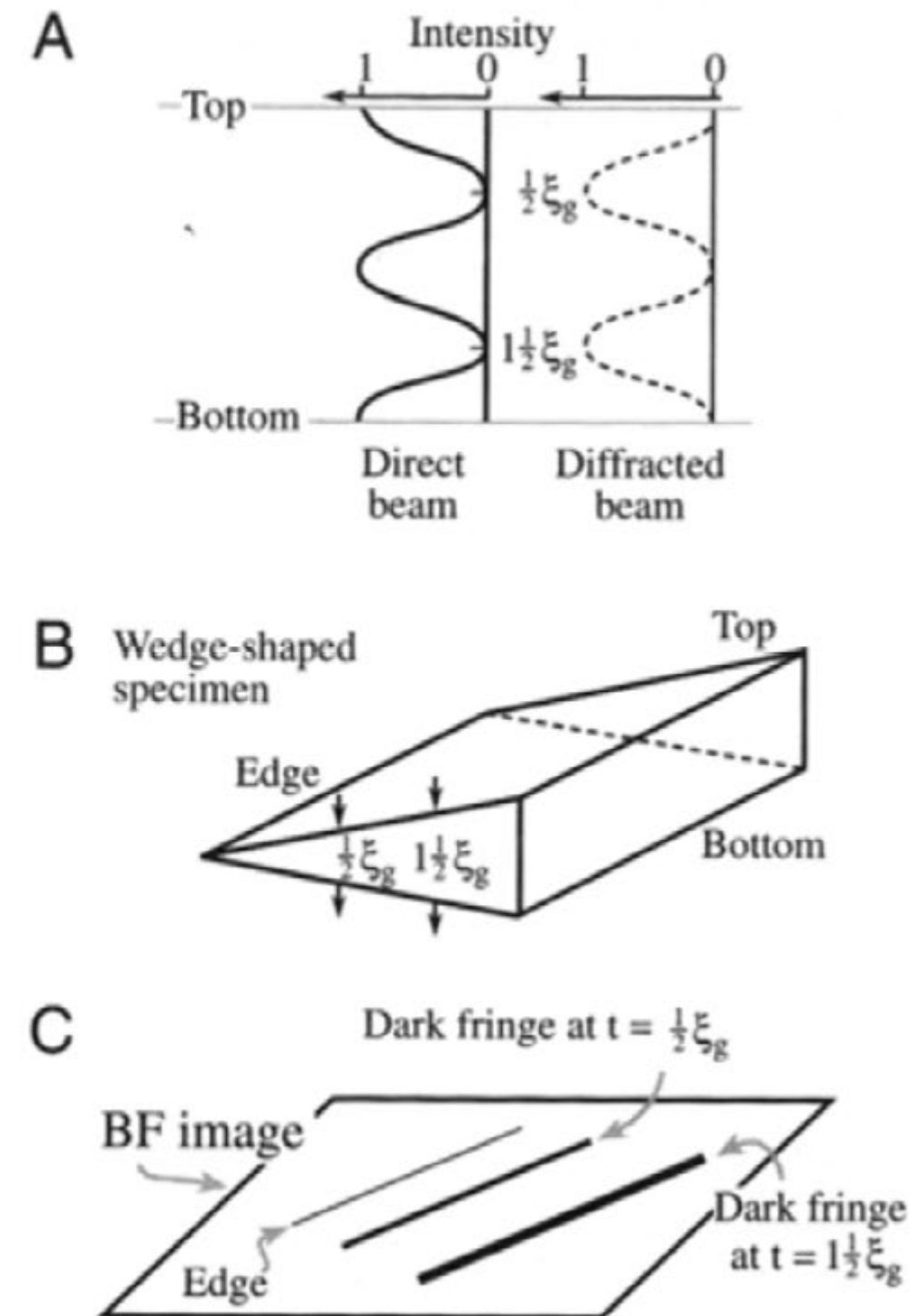
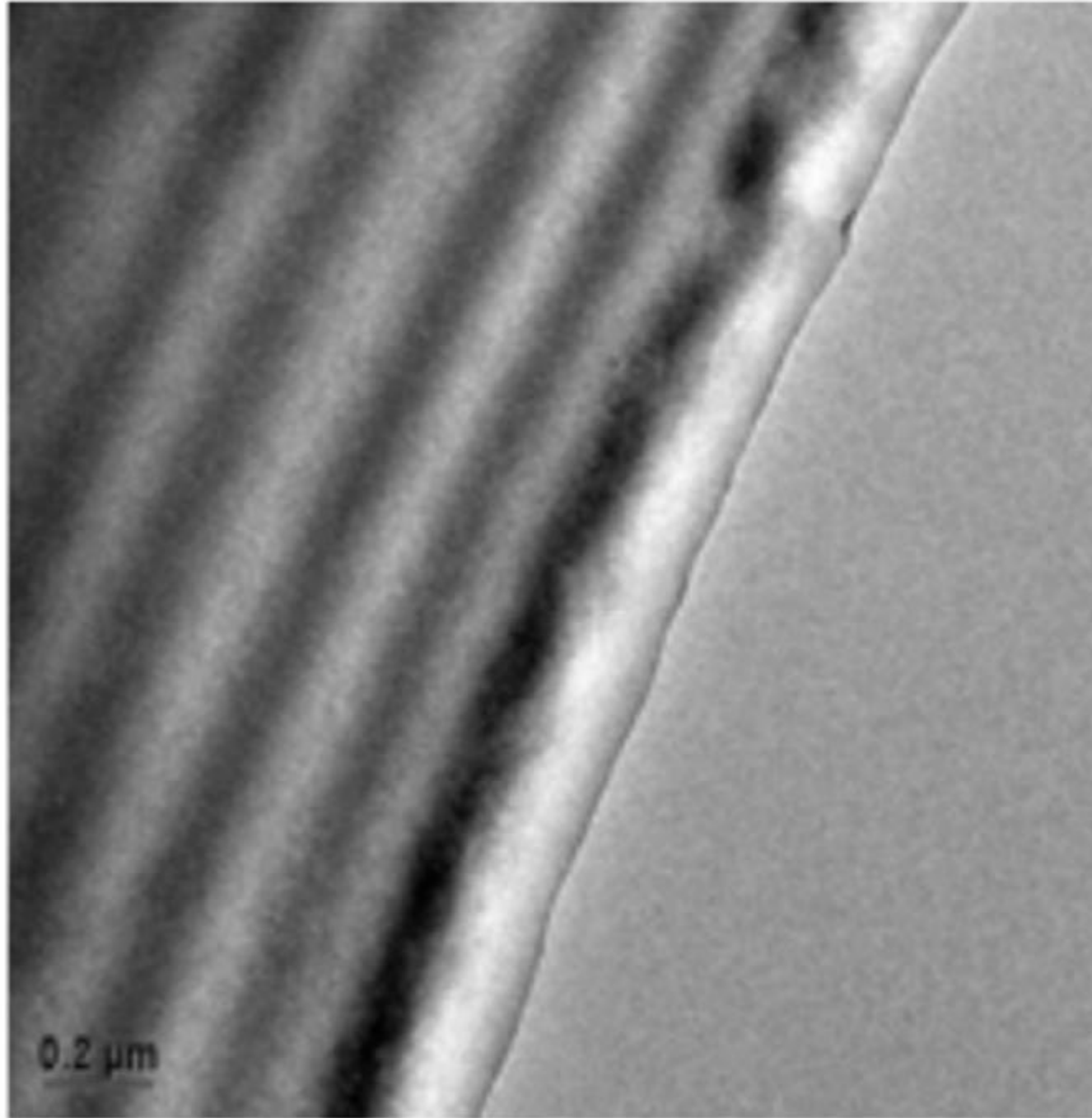
However, in electron diffraction there is often multiple elastic scattering:
i.e. "dynamical" behaviour

This dynamical scattering has a high probability because a Bragg-scattered beam is at the perfect angle to be Bragg-scattered again (and again...)

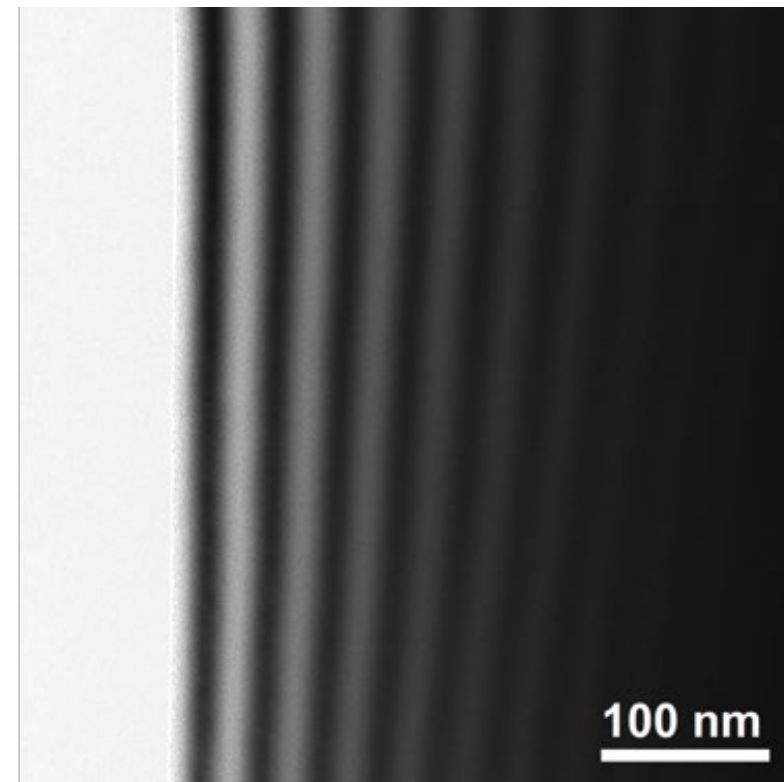
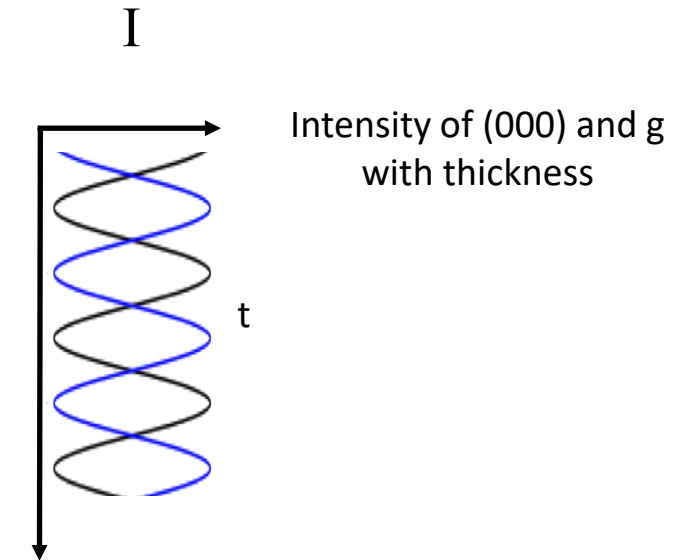
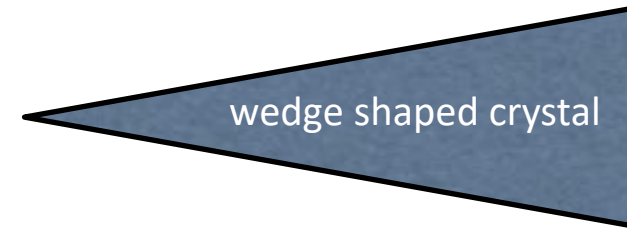
As a result, scattering of different beams is not independent from each other



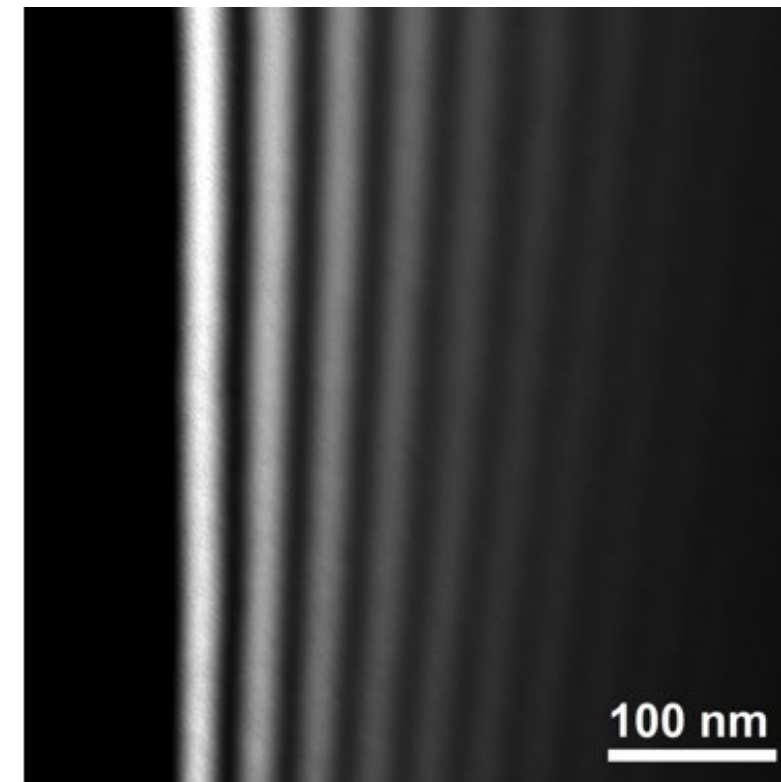
Dynamical scattering thickness fringes



Dynamical scattering | Thickness fringes



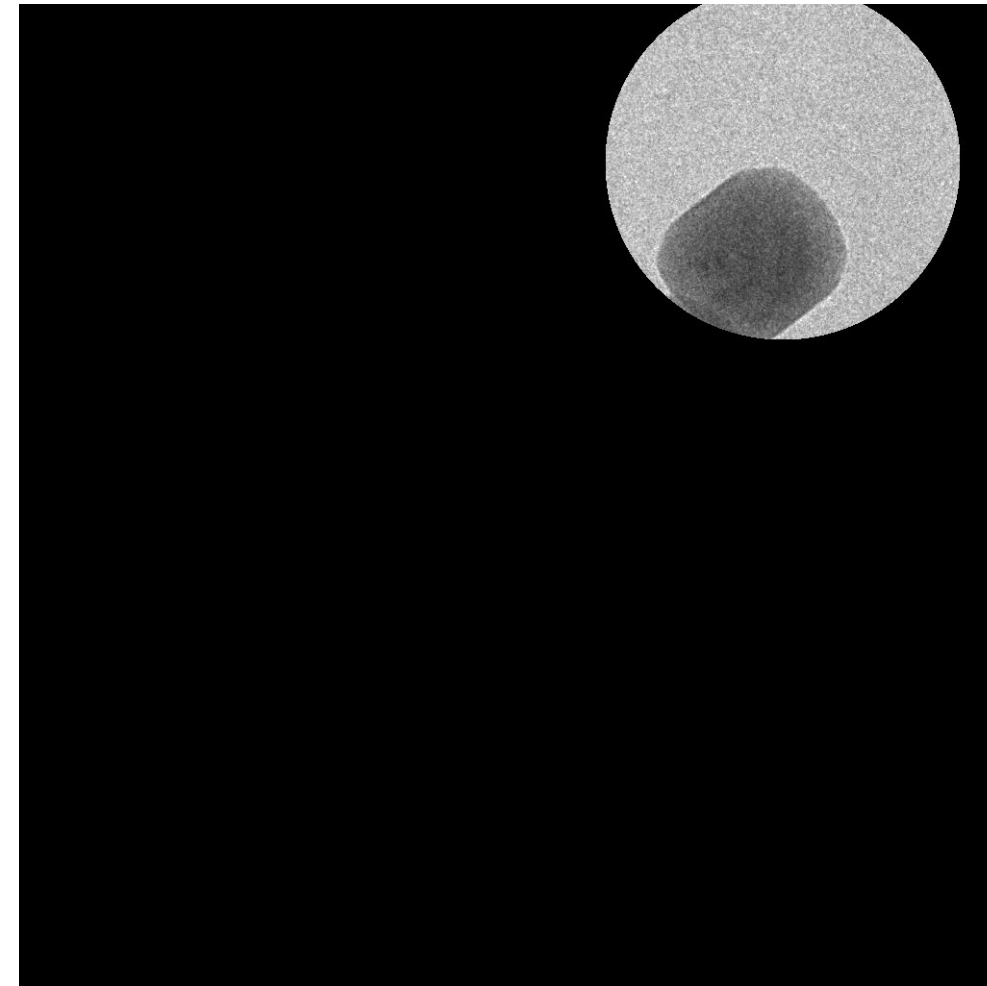
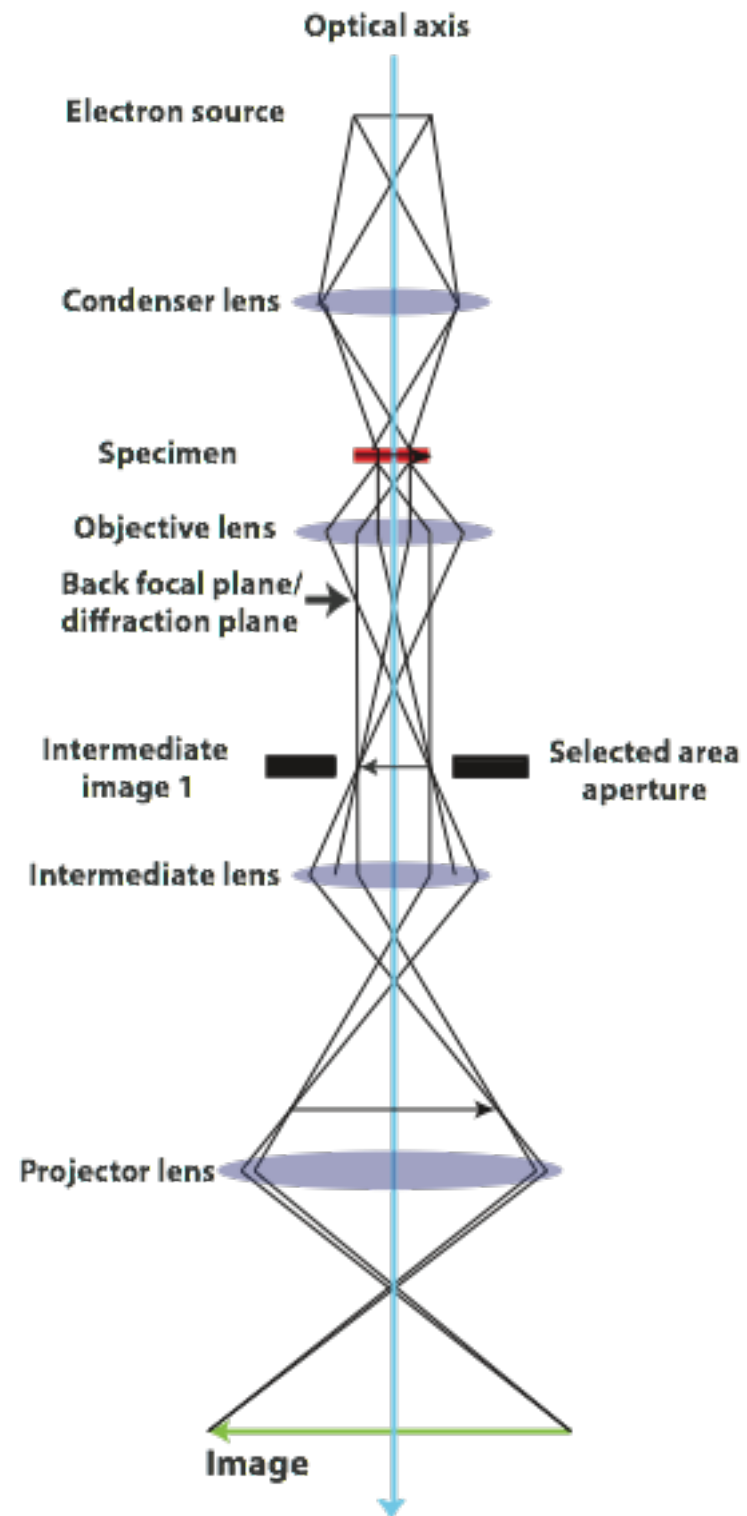
Bright-field



Dark-field

Selected-area diffraction phenomena

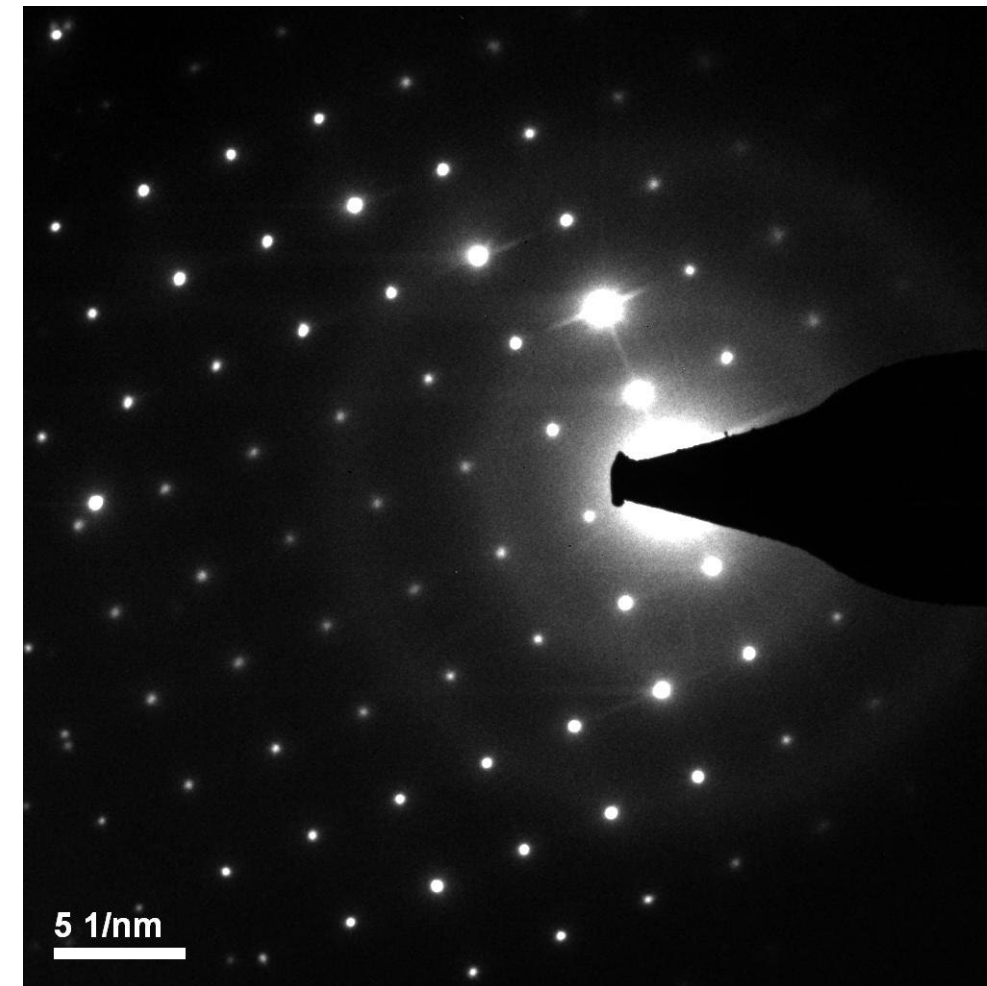
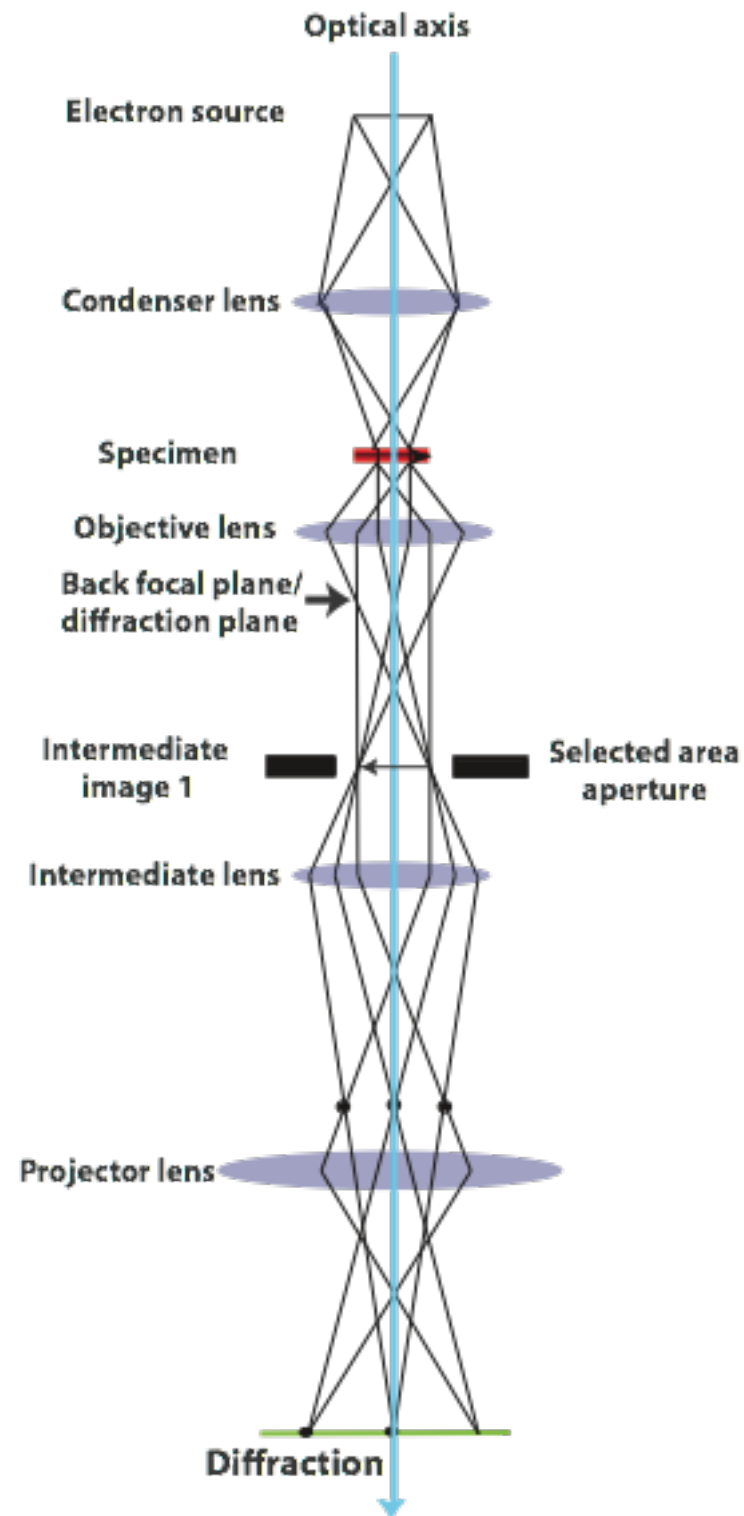
Image formation



BaTiO₃ nanocrystals (Psaltis lab)

Insert selected area aperture to choose region of interest

Take selected-area diffraction pattern



Press “D” for diffraction on microscope console -
alter strength of intermediate lens and focus
diffraction pattern on to screen

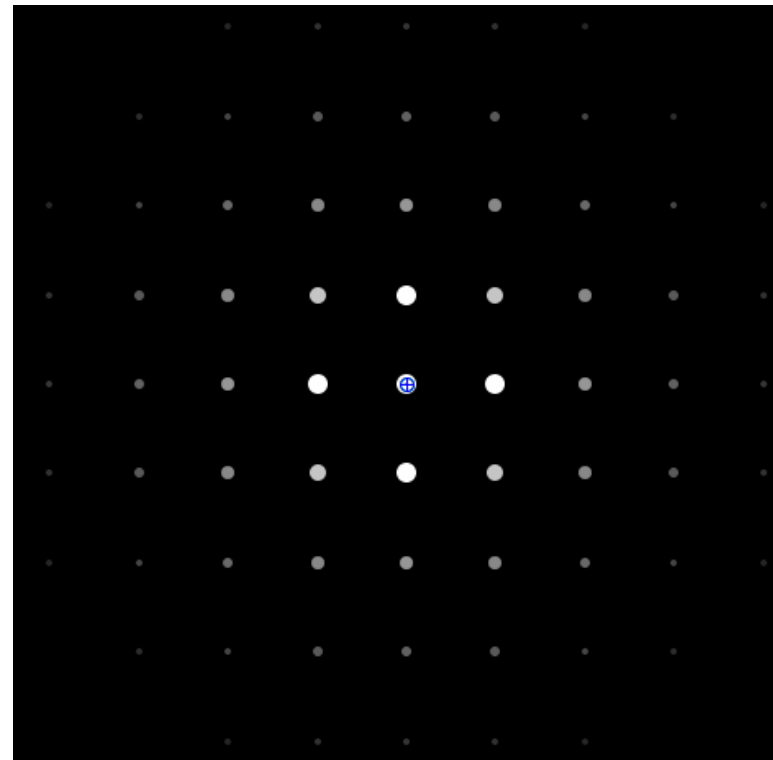
Find cubic BaTiO₃ aligned on [0 0 1] zone axis

Symmetry information

Zone axis SADPs have symmetry closely related to symmetry of crystal lattice

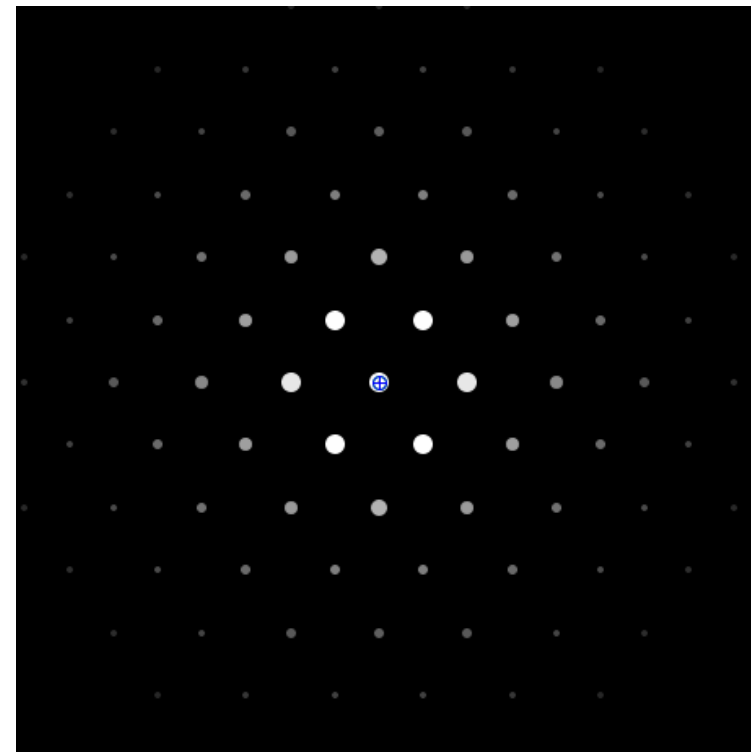
Example: FCC aluminium

$[0\ 0\ 1]$



4-fold rotation axis

$[1\ 1\ 0]$

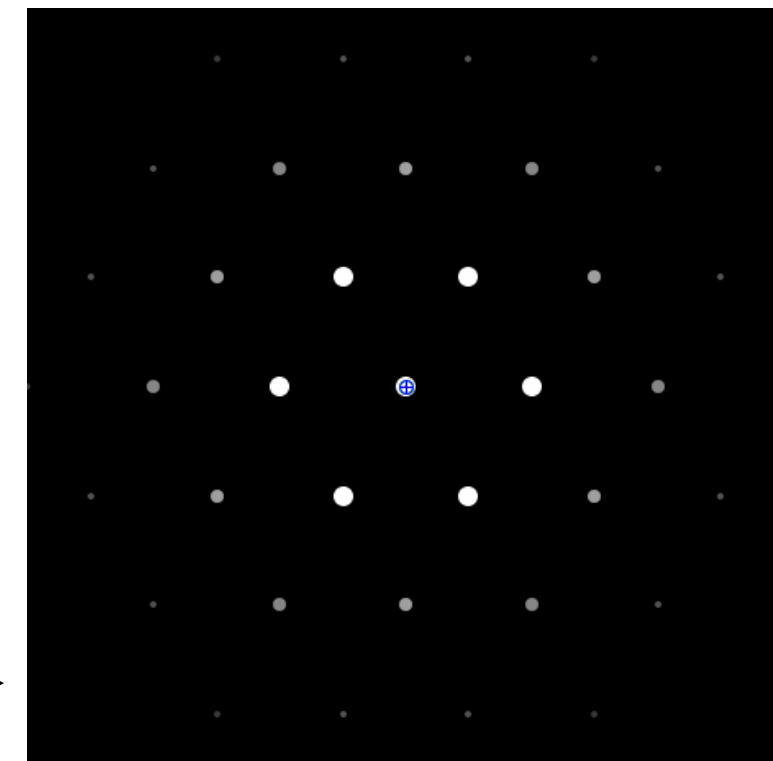


2-fold rotation axis

6-fold rotation axis - but $[1\ 1\ 1]$ actually 3-fold axis
Need third dimension for true symmetry!



$[1\ 1\ 1]$



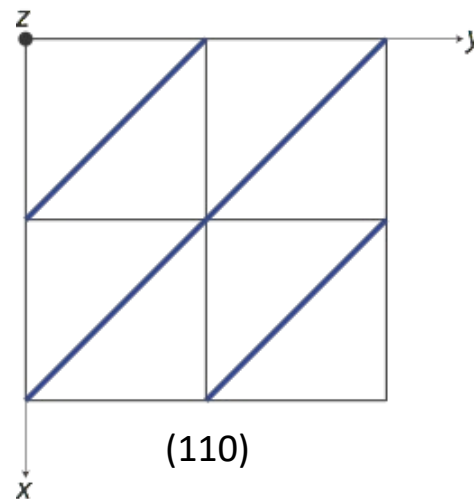
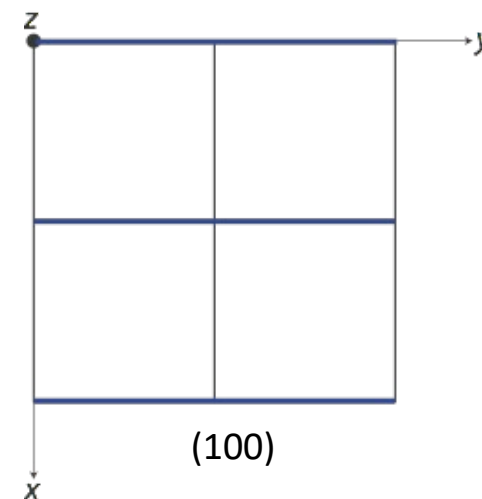
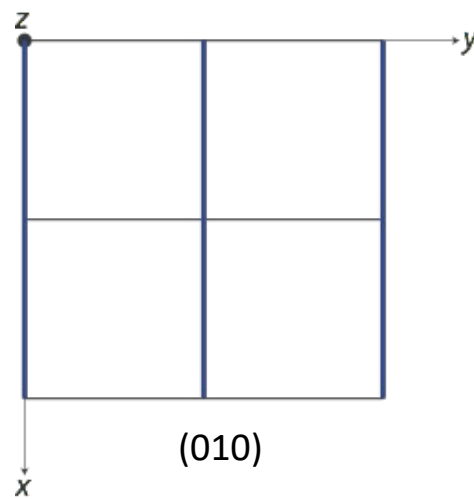
Weiss zone law

Electron beam parallel to low-index crystal orientation $[U\ V\ W]$ = zone axis

Crystal "viewed down" zone axis is like diffraction grating with planes parallel to e-beam

In diffraction pattern of orthogonal lattices spots appear perpendicular to plane orientation

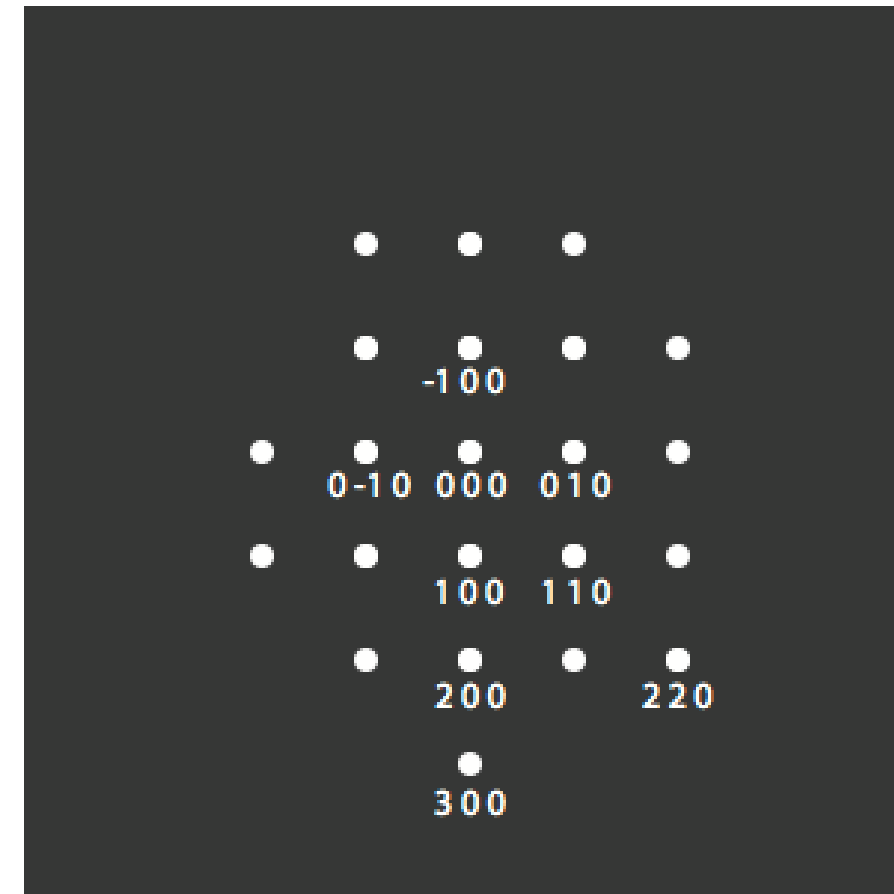
Example: primitive cubic with e-beam parallel to $[0\ 0\ 1]$ zone axis



[001] zone axis

$$(100): 1*0 + 0*0 + 0*1 = 0$$

$$(110): 1*0 + 1*0 + 0*1 = 0$$

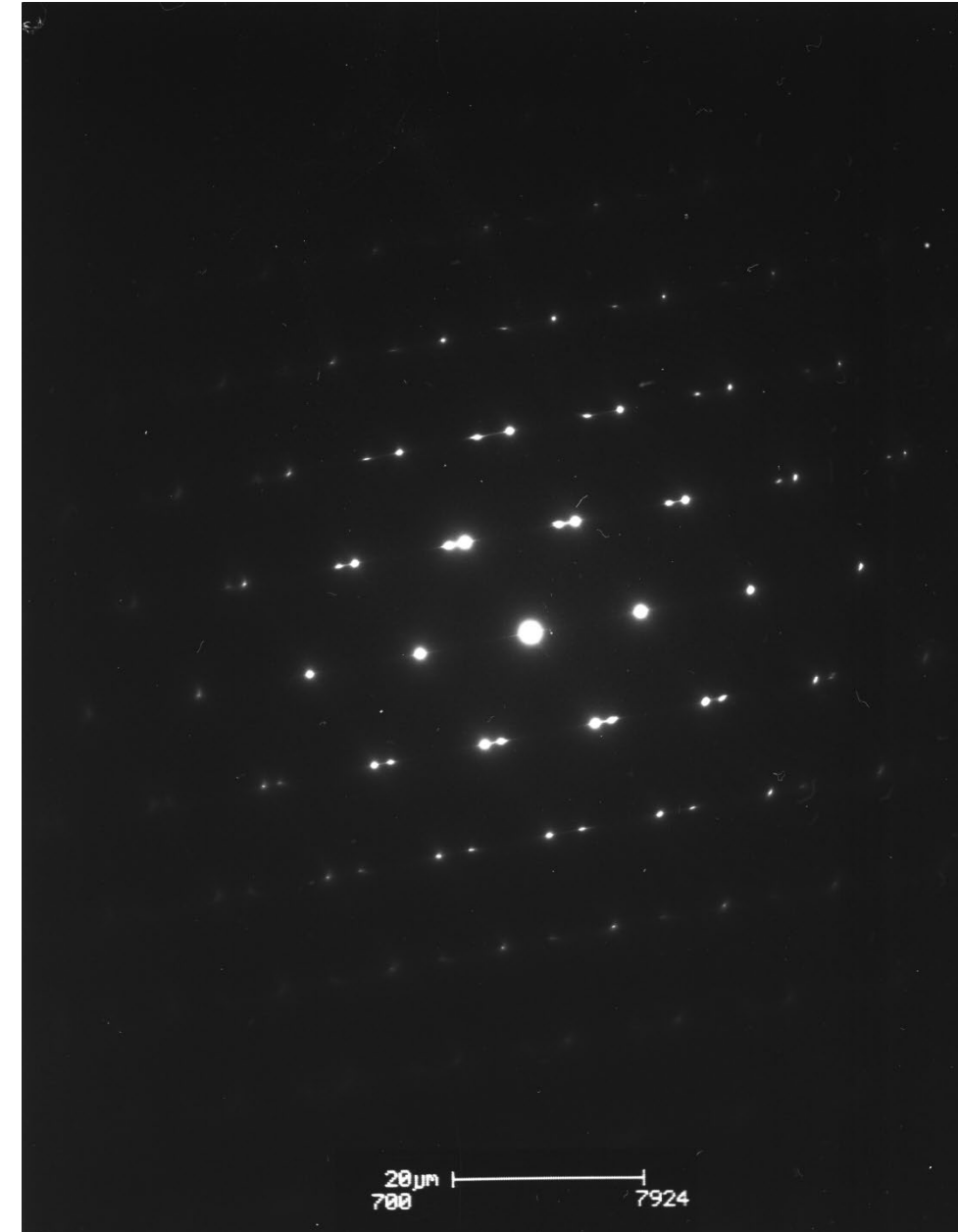
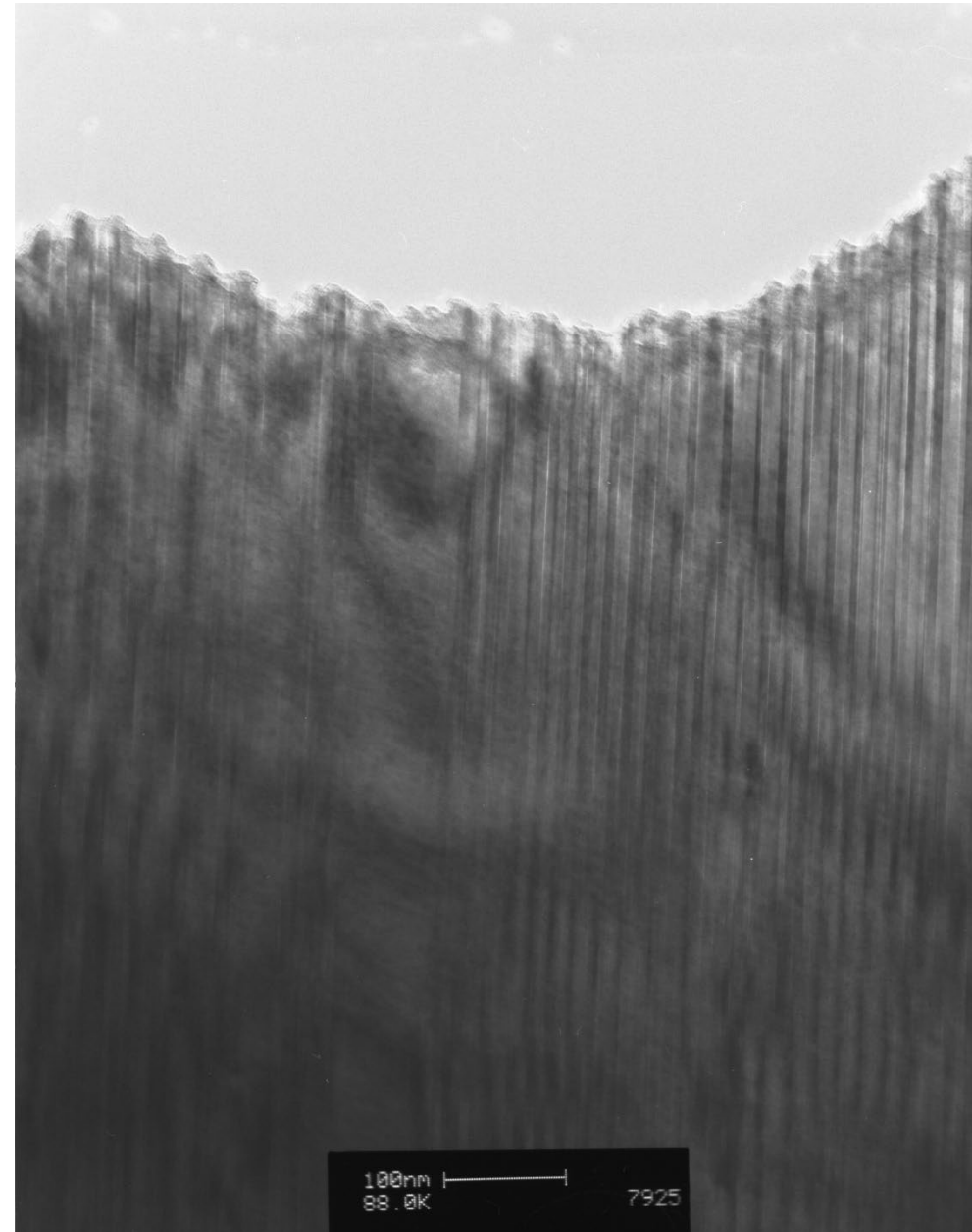


Weiss Zone Law obeyed in indexing ($hU + kV + lW = 0$)

The intersection of (010) and (100) lattice planes: $[001]$ direction = zone axis

Twinning in diffraction

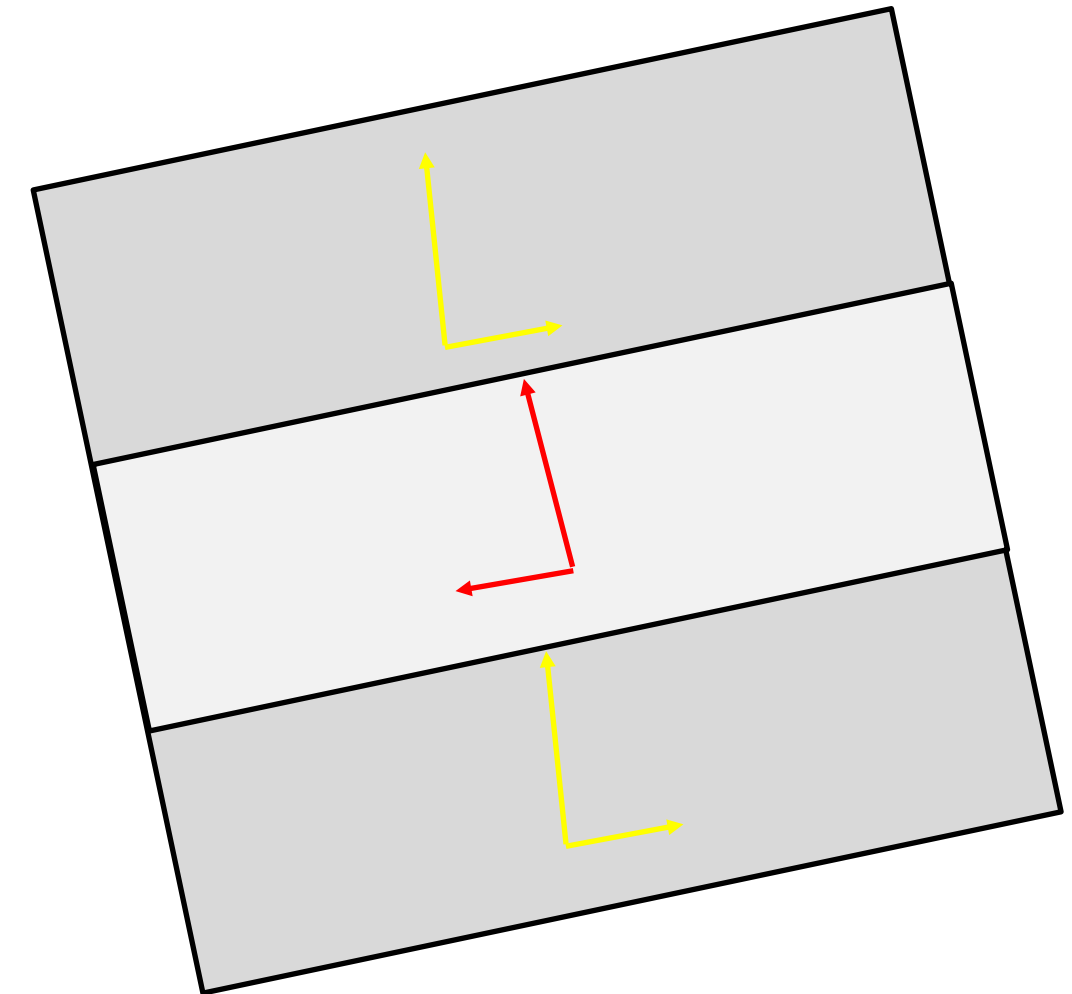
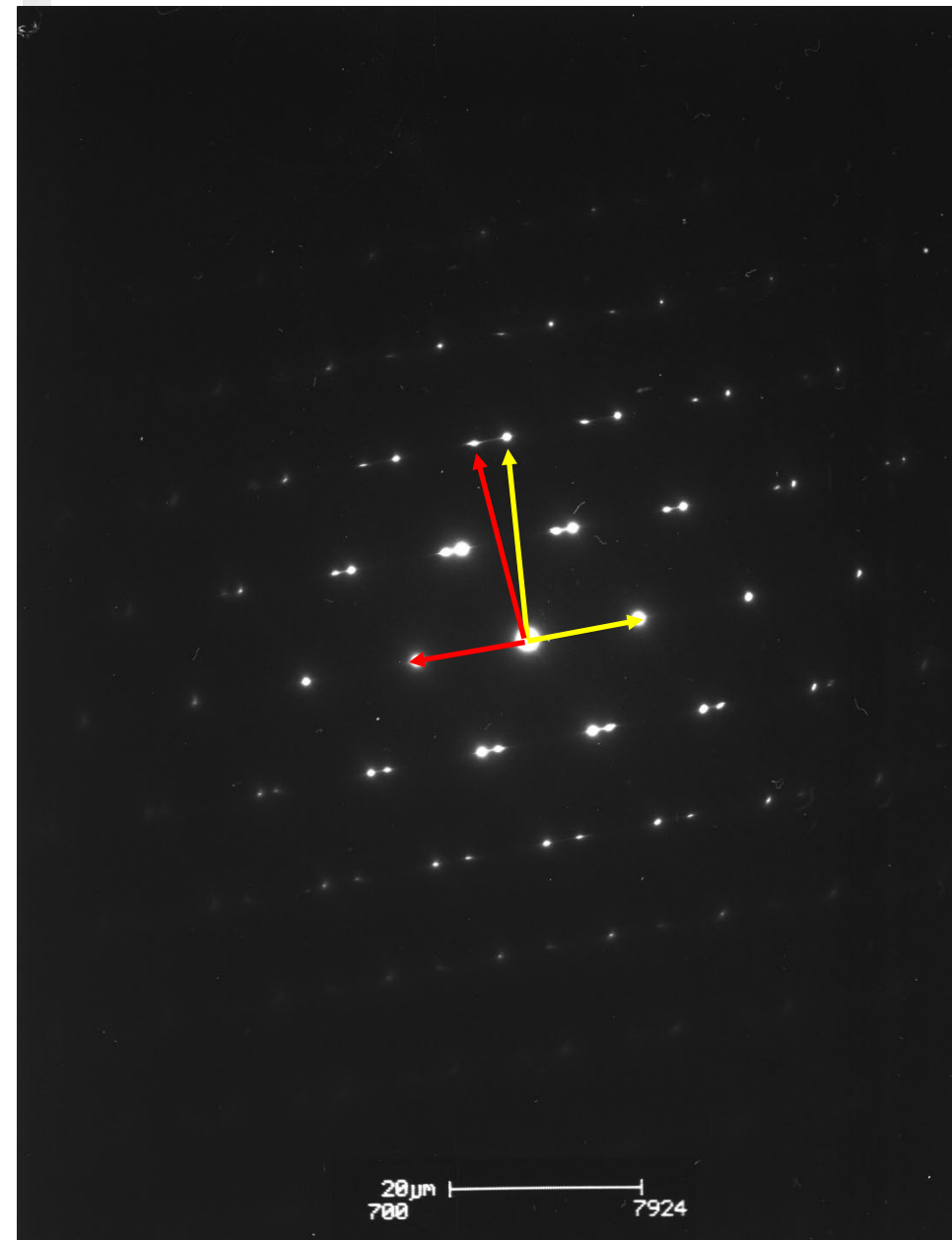
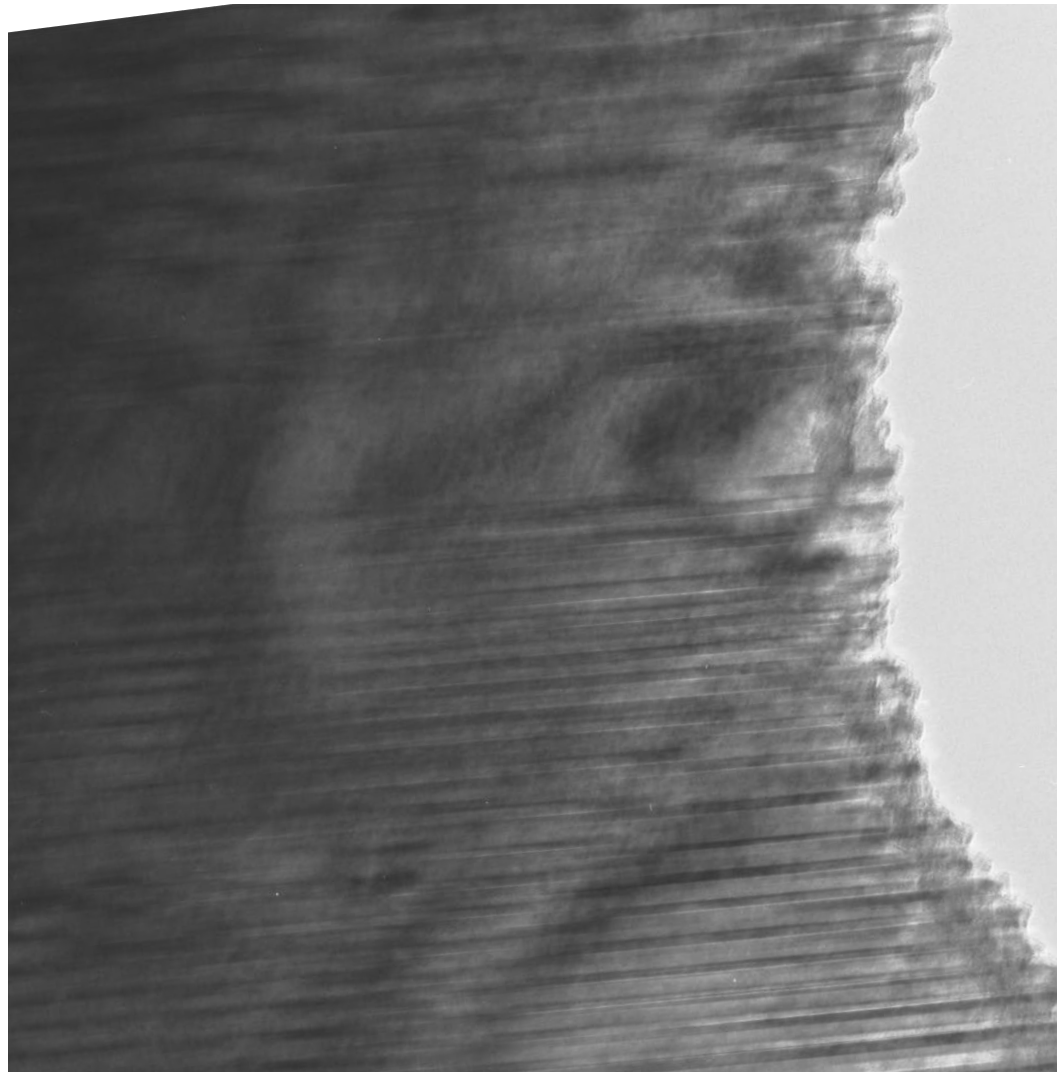
Example: Co-Ni-Al shape memory FCC twins observed on $[1\ 1\ 0]$ zone axis
(1 1 1) close-packed twin planes overlap in SADP



Images provided by Barbora Bartová, CIME

Twinning in diffraction

Example: Co-Ni-Al shape memory FCC twins observed on $[1\ 1\ 0]$ zone axis
close-packed twin planes overlap in SADP

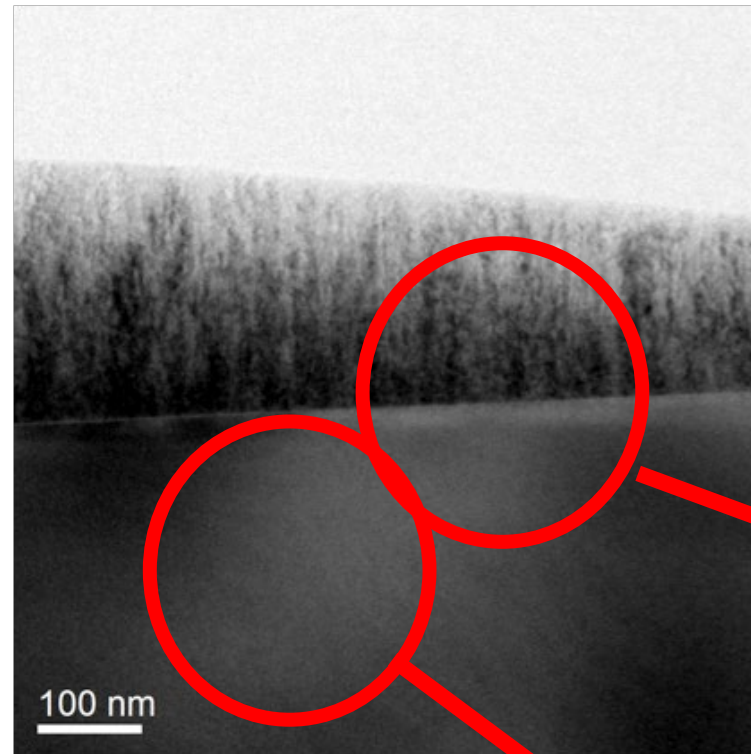


Images provided by Barbora Bartová, CIME

Epitaxy and orientation relationships

SADP excellent tool for studying
orientation relationships across
interfaces

Example: Mn-doped ZnO on sapphire

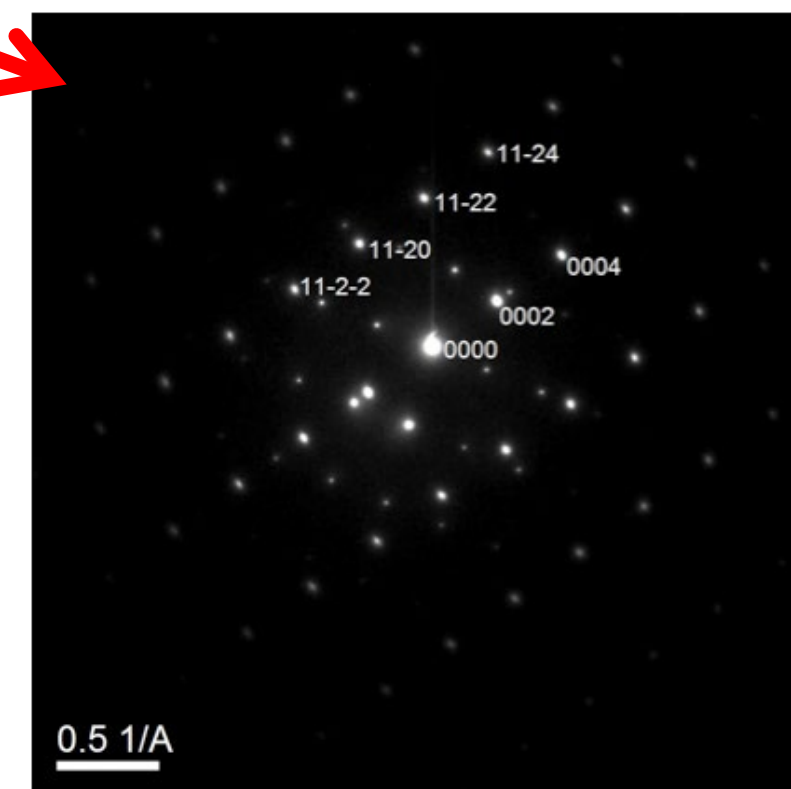
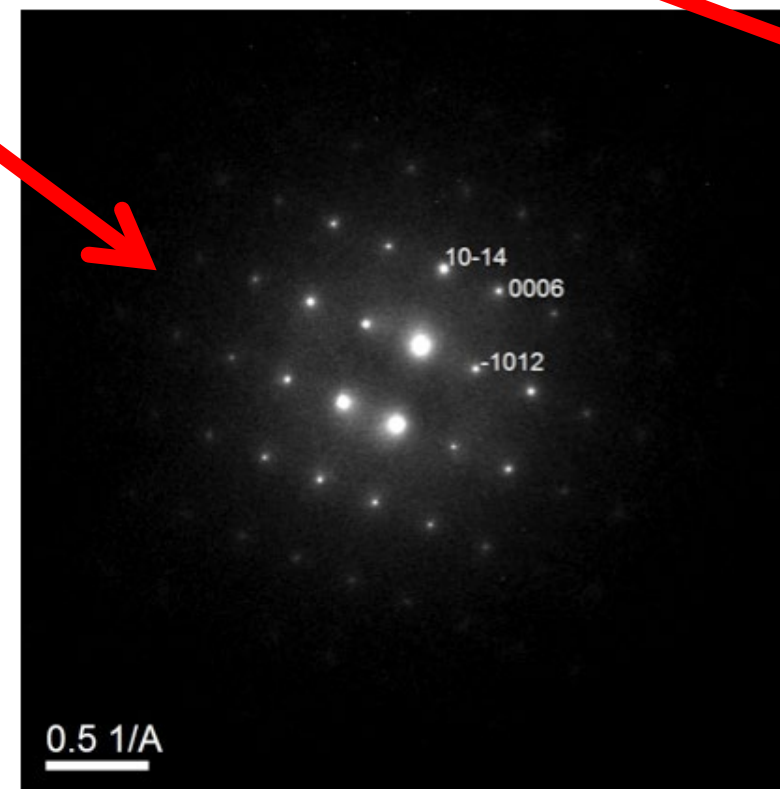


Sapphire substrate

Sapphire + film

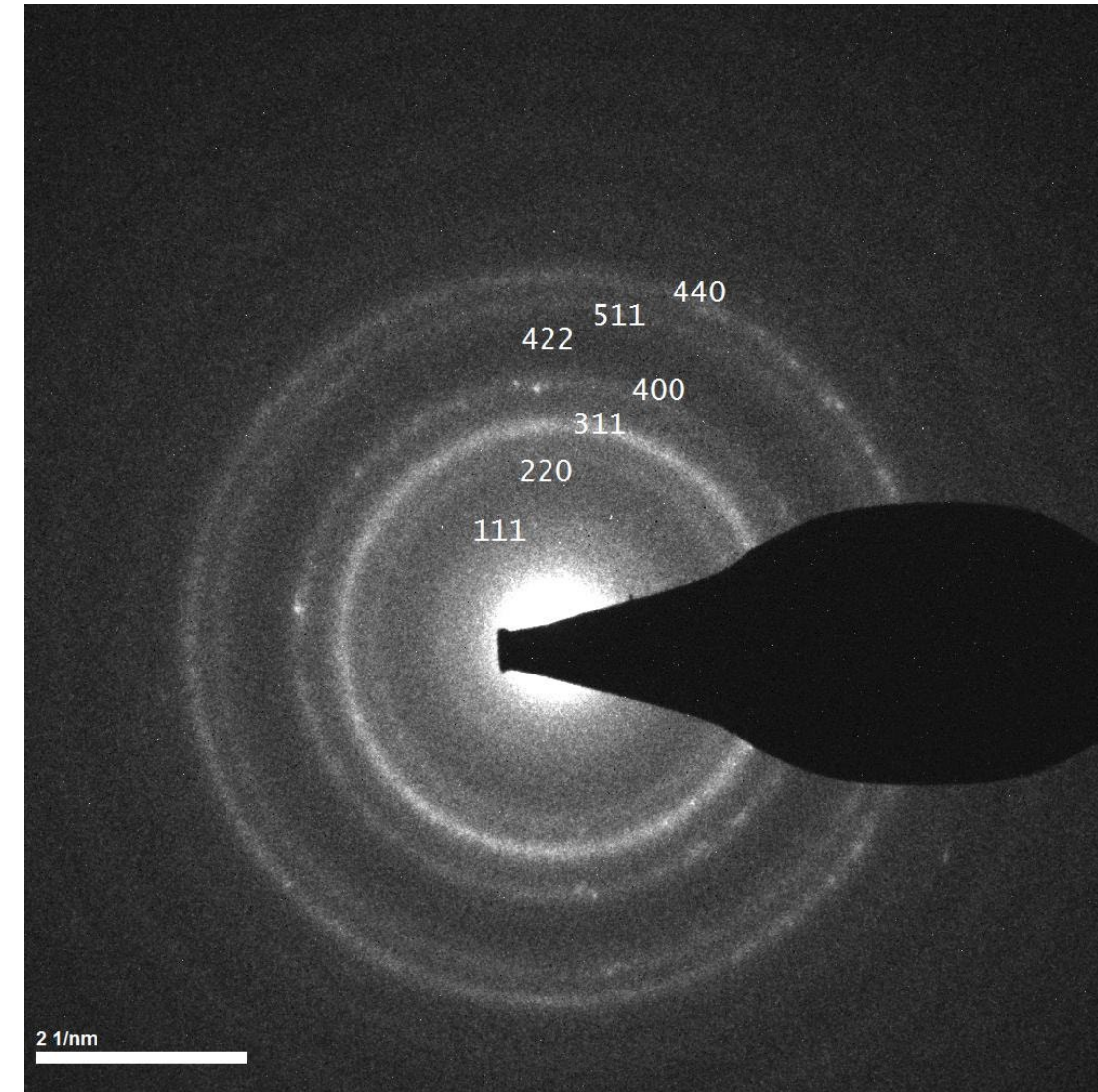
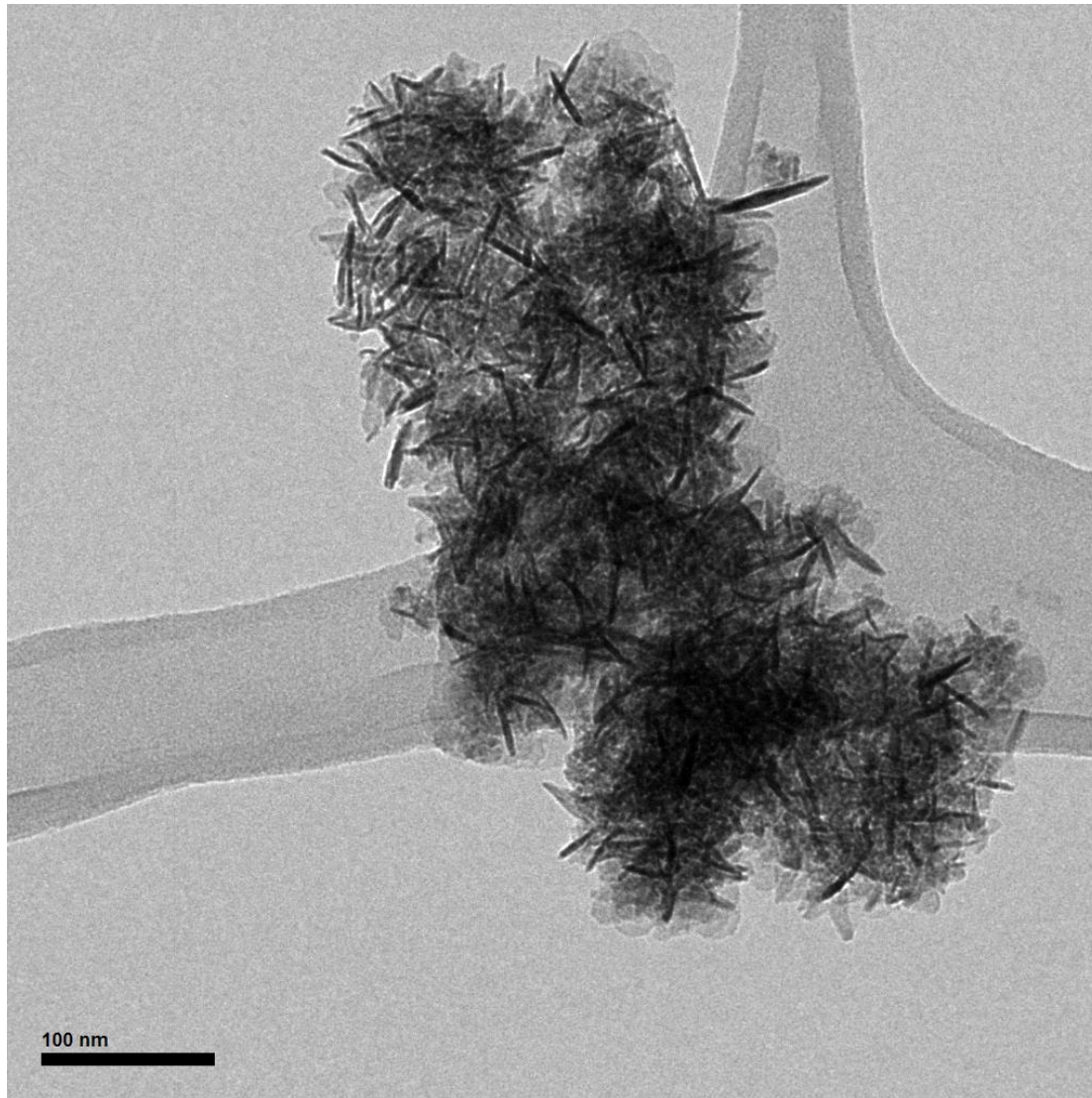
Zone axes:
 $[1\ -1\ 0]_{\text{ZnO}} // [0\ -1\ 0]_{\text{sapphire}}$

Planes:
 $c\text{-plane}_{\text{ZnO}} // c\text{-plane}_{\text{sapphire}}$



Ring diffraction patterns

If selected area aperture selects numerous, randomly-oriented nanocrystals,
SADP consists of rings sampling all possible diffracting planes
- like powder X-ray diffraction

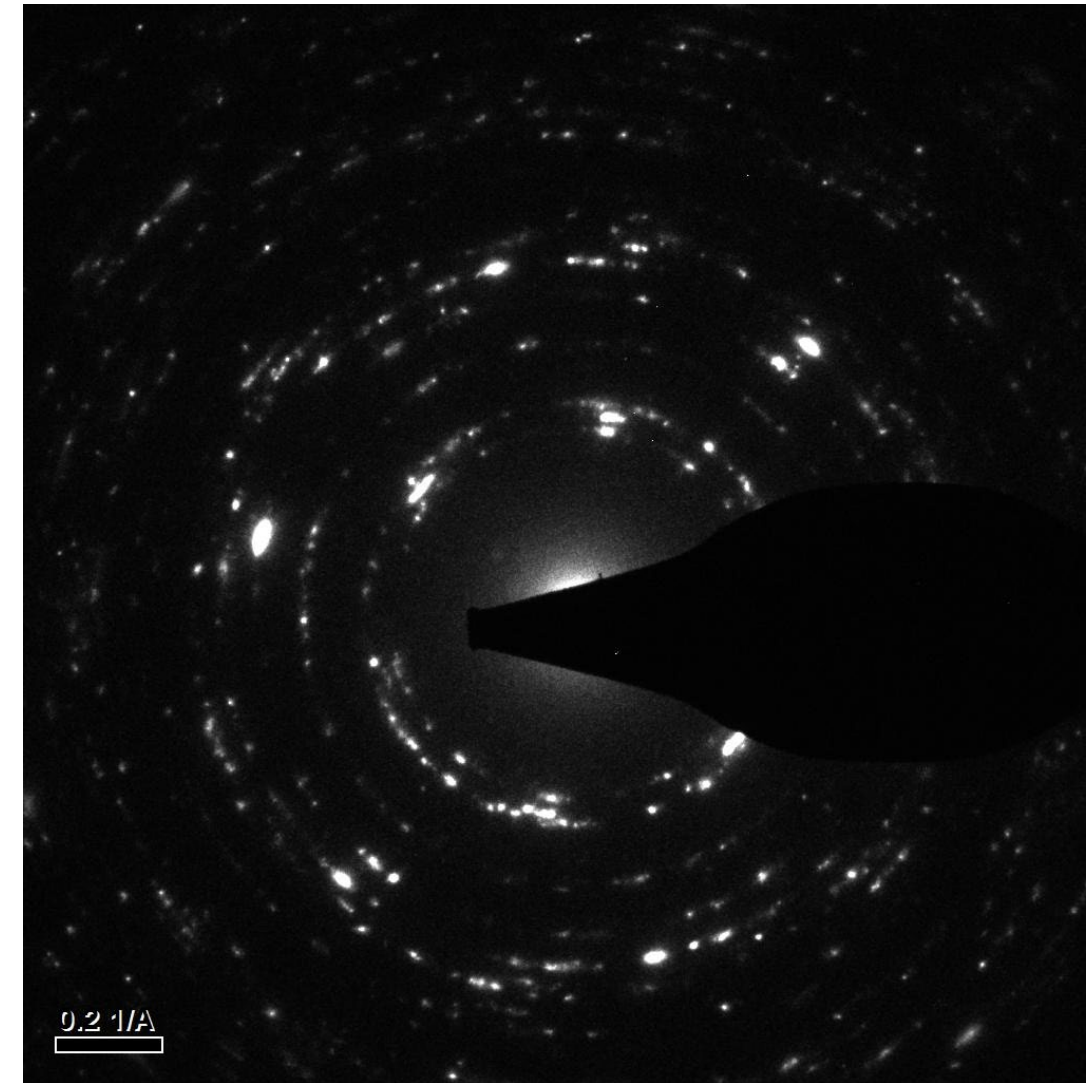
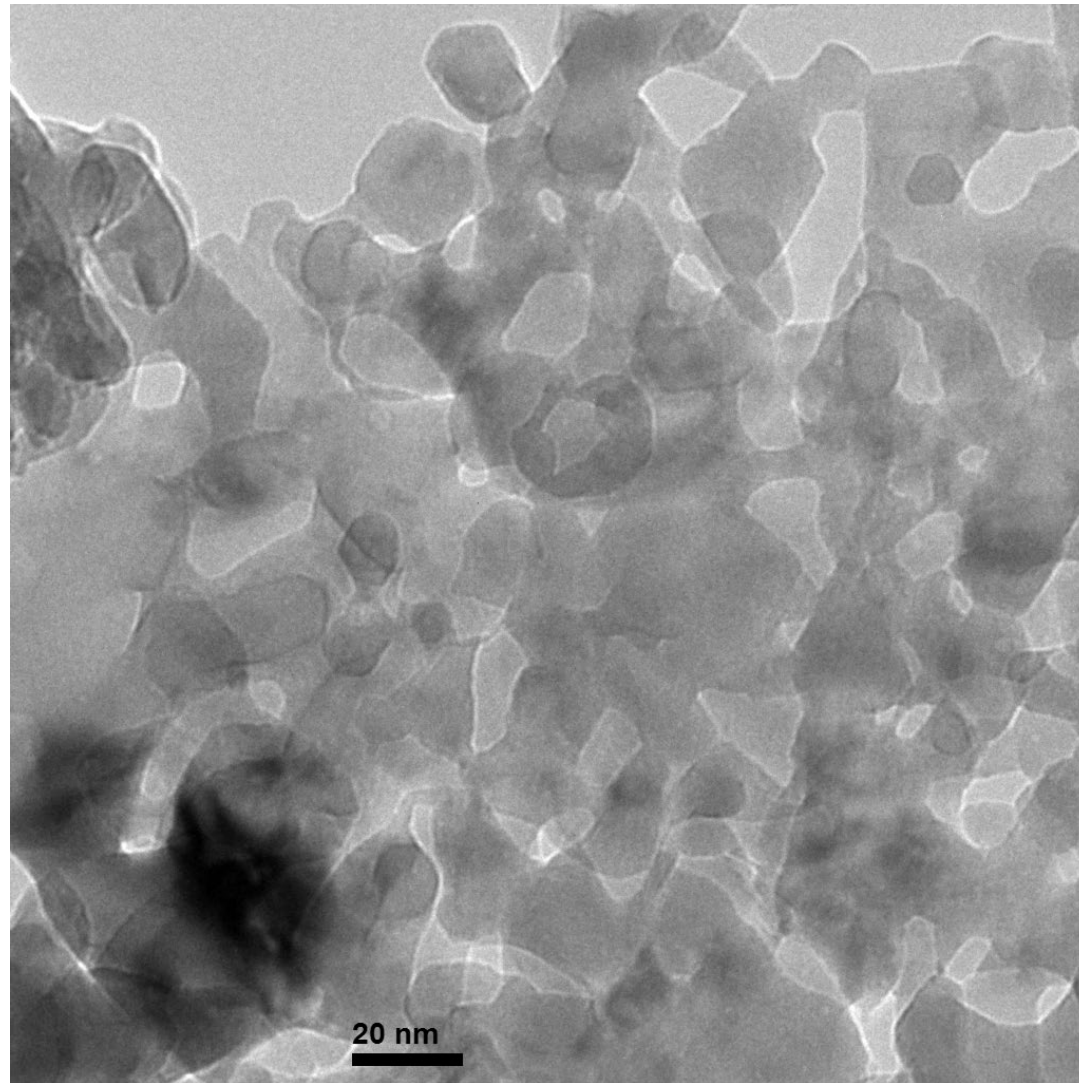


Example: "needles" of contaminant cubic MnZnO_3 - which XRD failed to observe!

Ring diffraction patterns

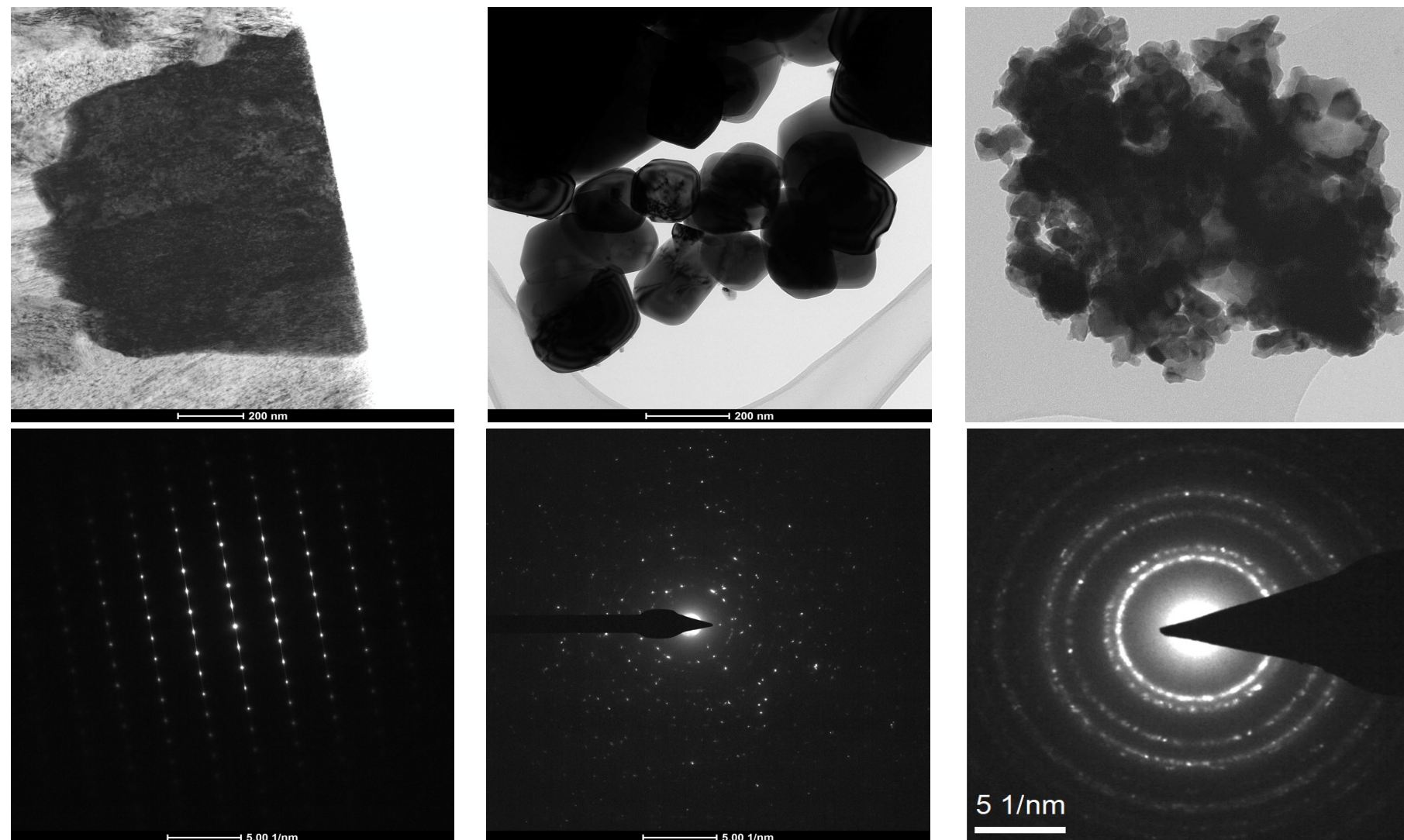
Larger crystals => more "spotty" patterns

Example: ZnO nanocrystals ~20 nm in diameter



Applications of electron diffraction

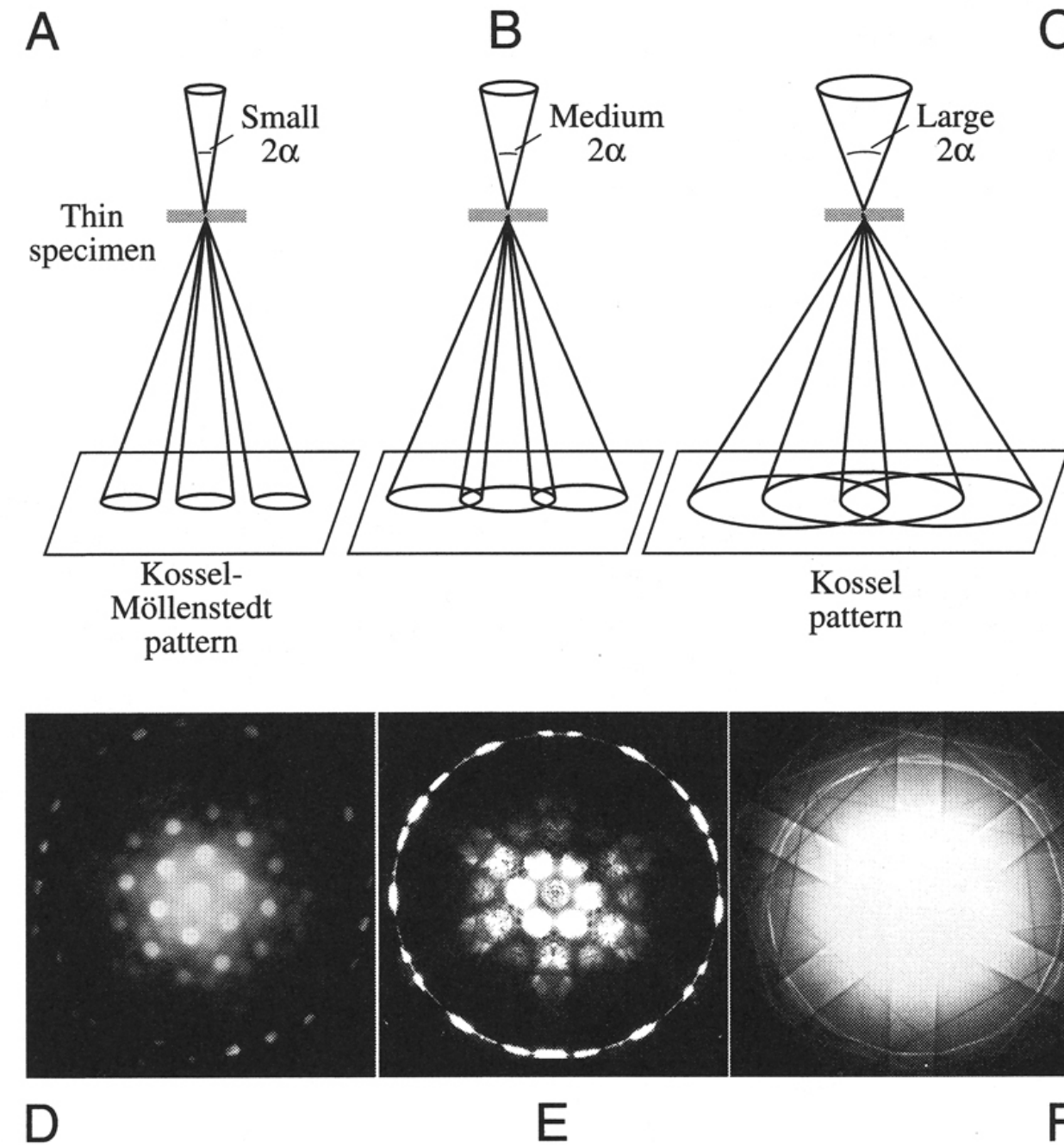
Introduction à la microscopie électronique: Diffraction



Emad Oveisi

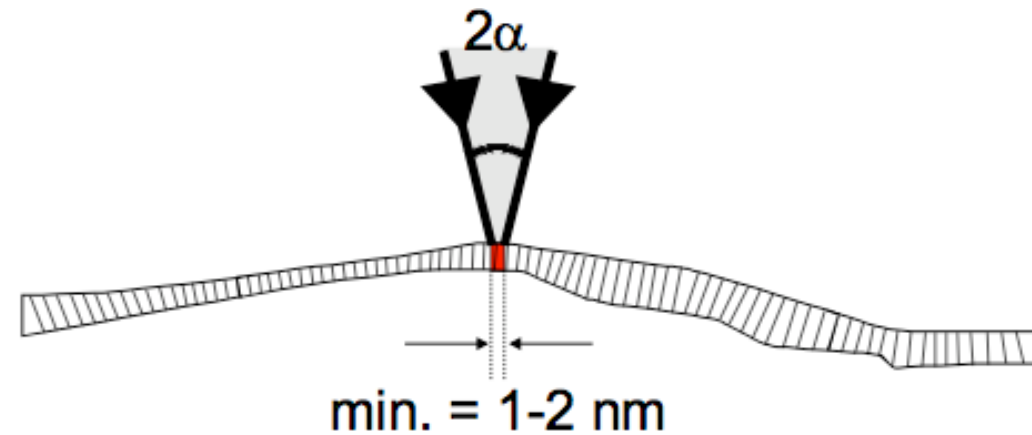
Convergent beam electron diffraction

Convergent beam electron diffraction



Convergent beam electron diffraction

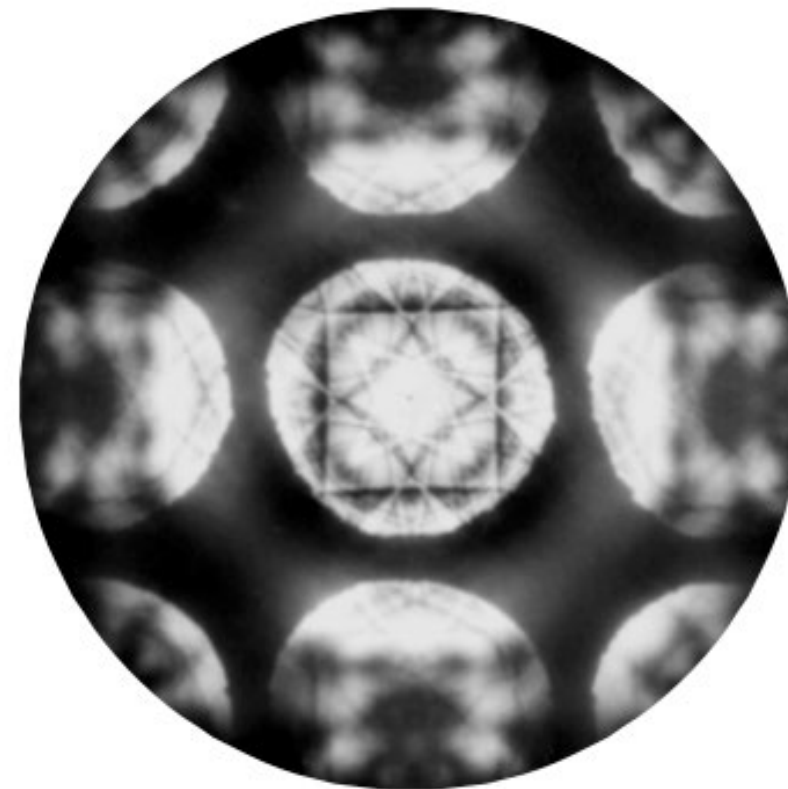
Instead of parallel illumination with selected-area aperture, CBED uses highly converged illumination to select a much smaller specimen region



Small illuminated area =>
no thickness and orientation variations

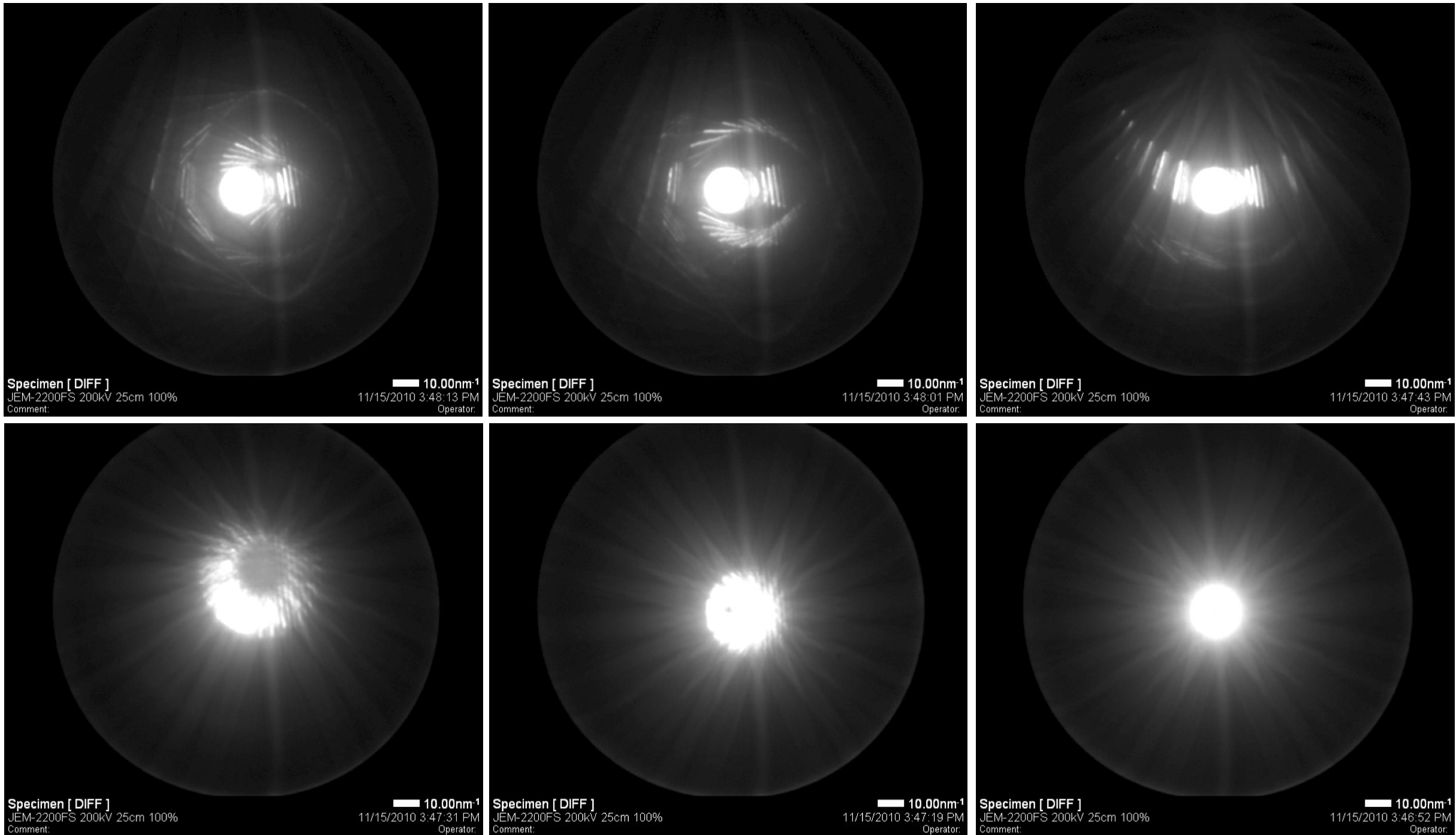
There is dynamical scattering, but it is useful!

Can obtain disc and line patterns
"packed" with information:

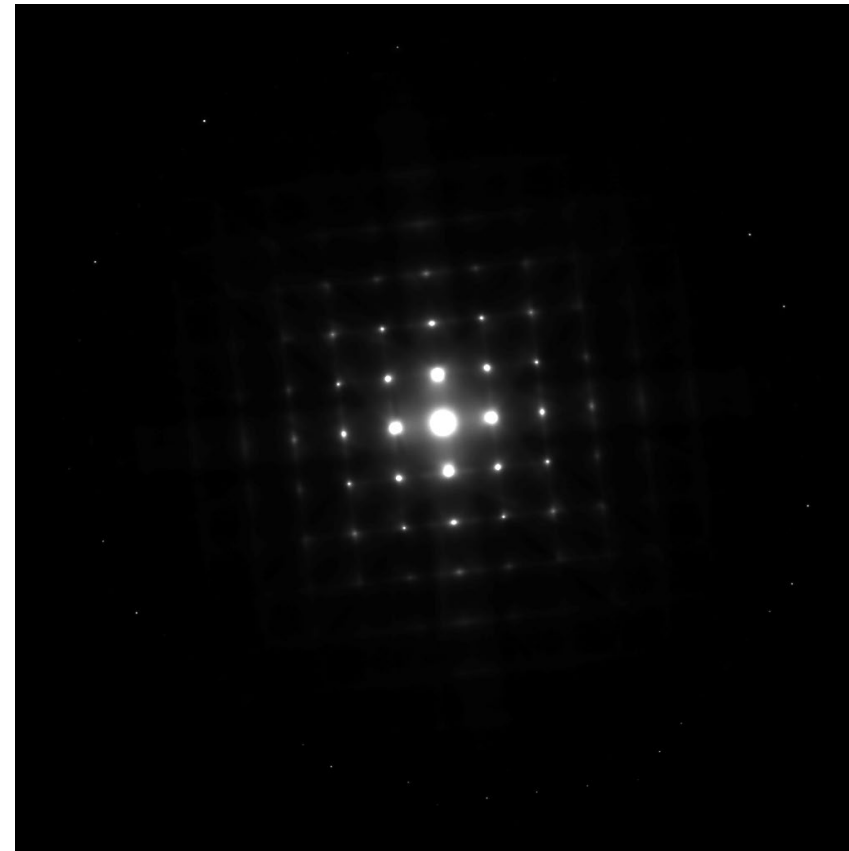


Convergent beam electron diffraction | To align specimen

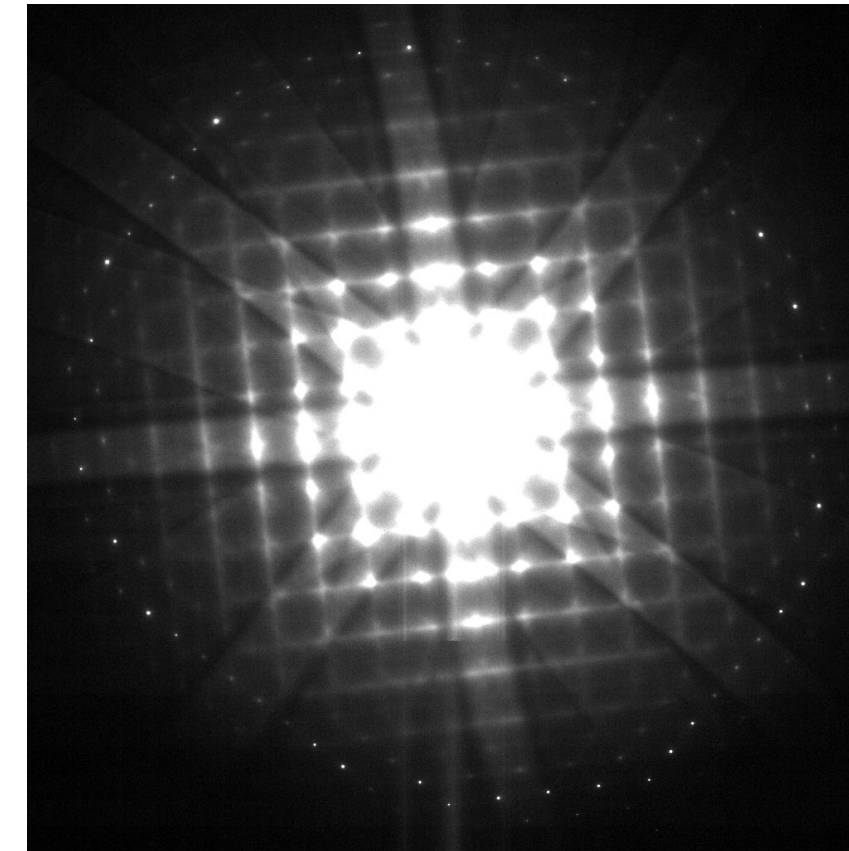
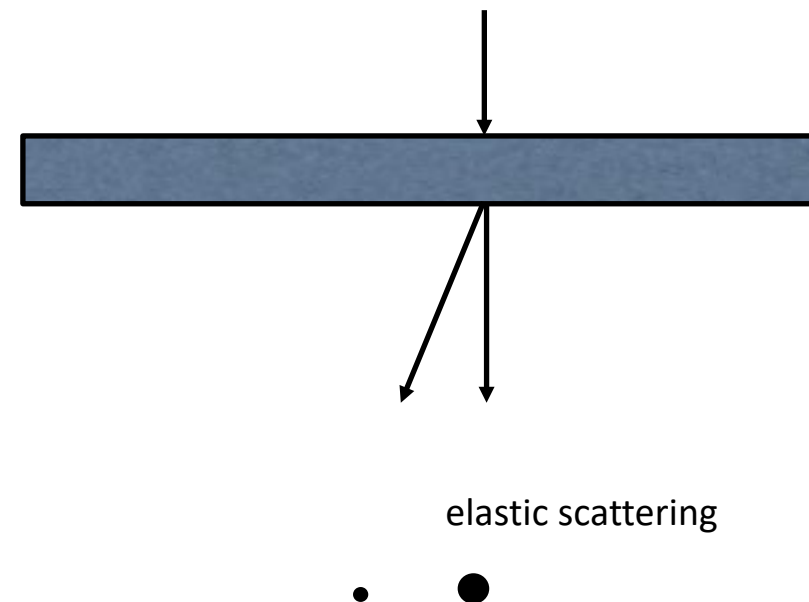
ZnO thin-film sample; TEM beam focused to spot on sample



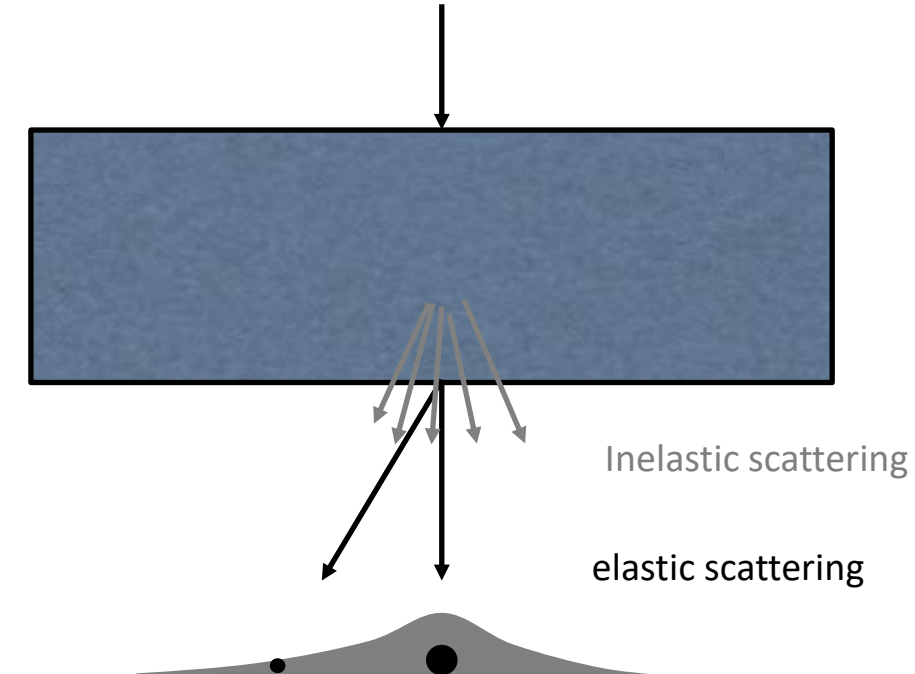
Kikuchi lines/bands



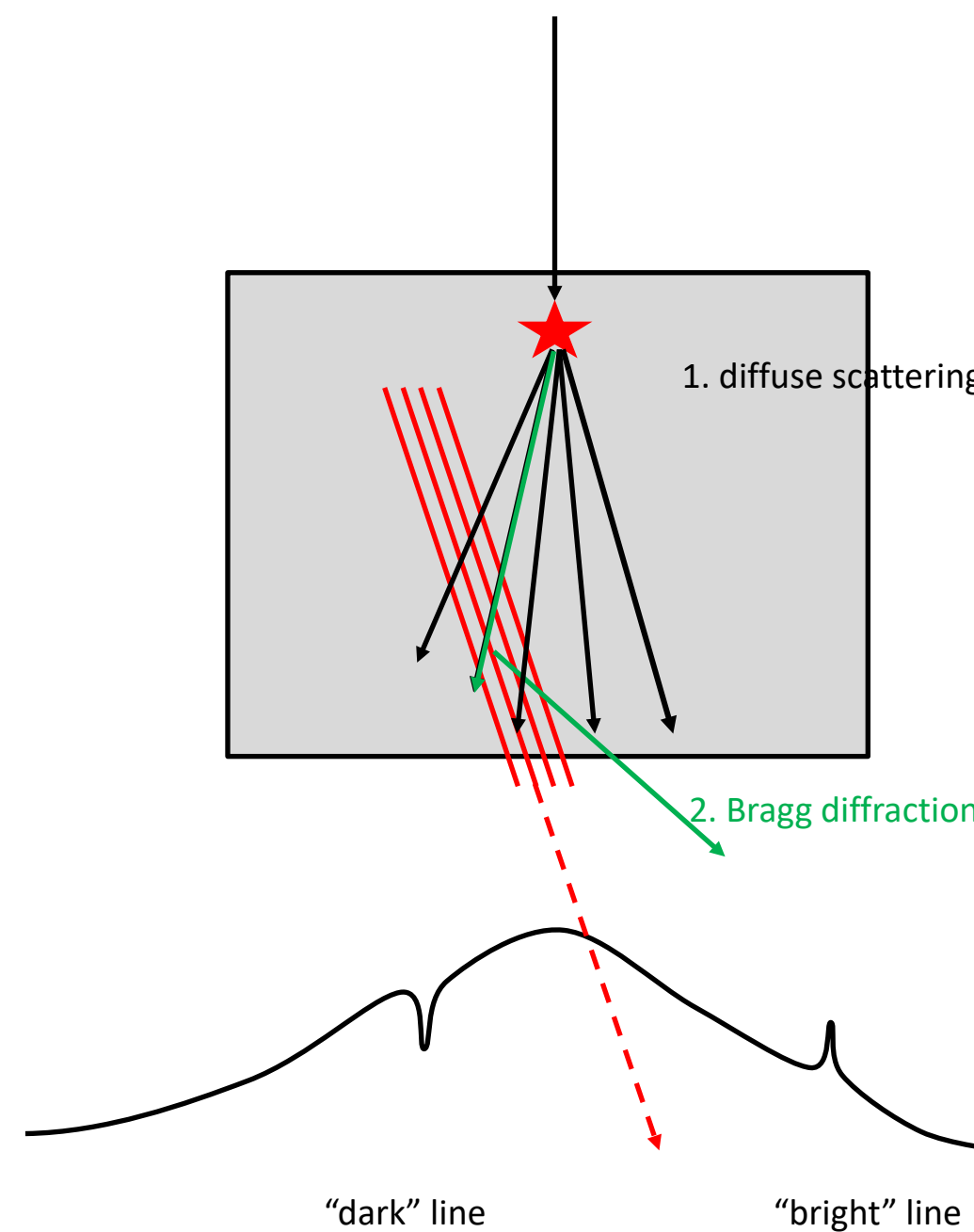
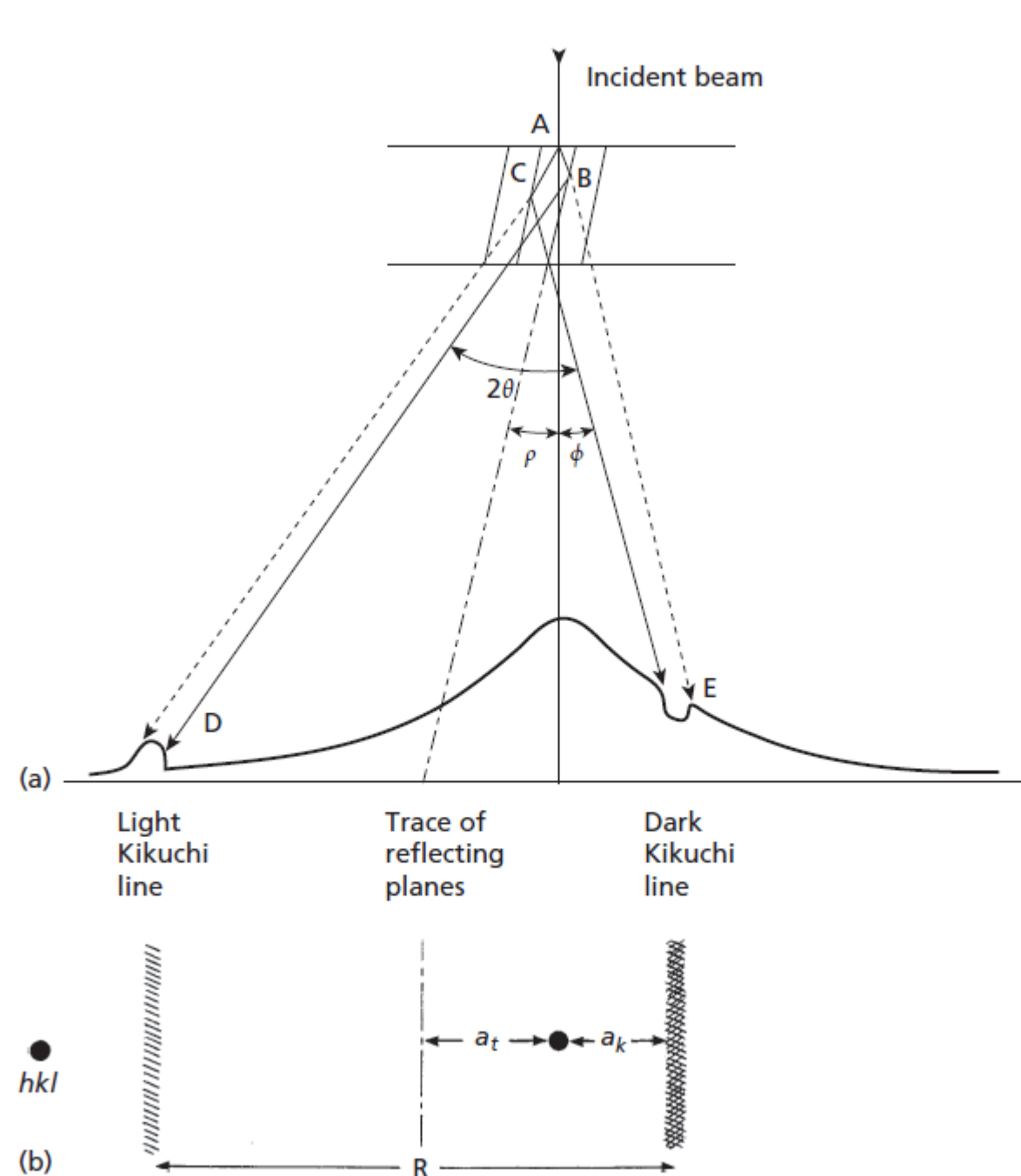
Thin sample



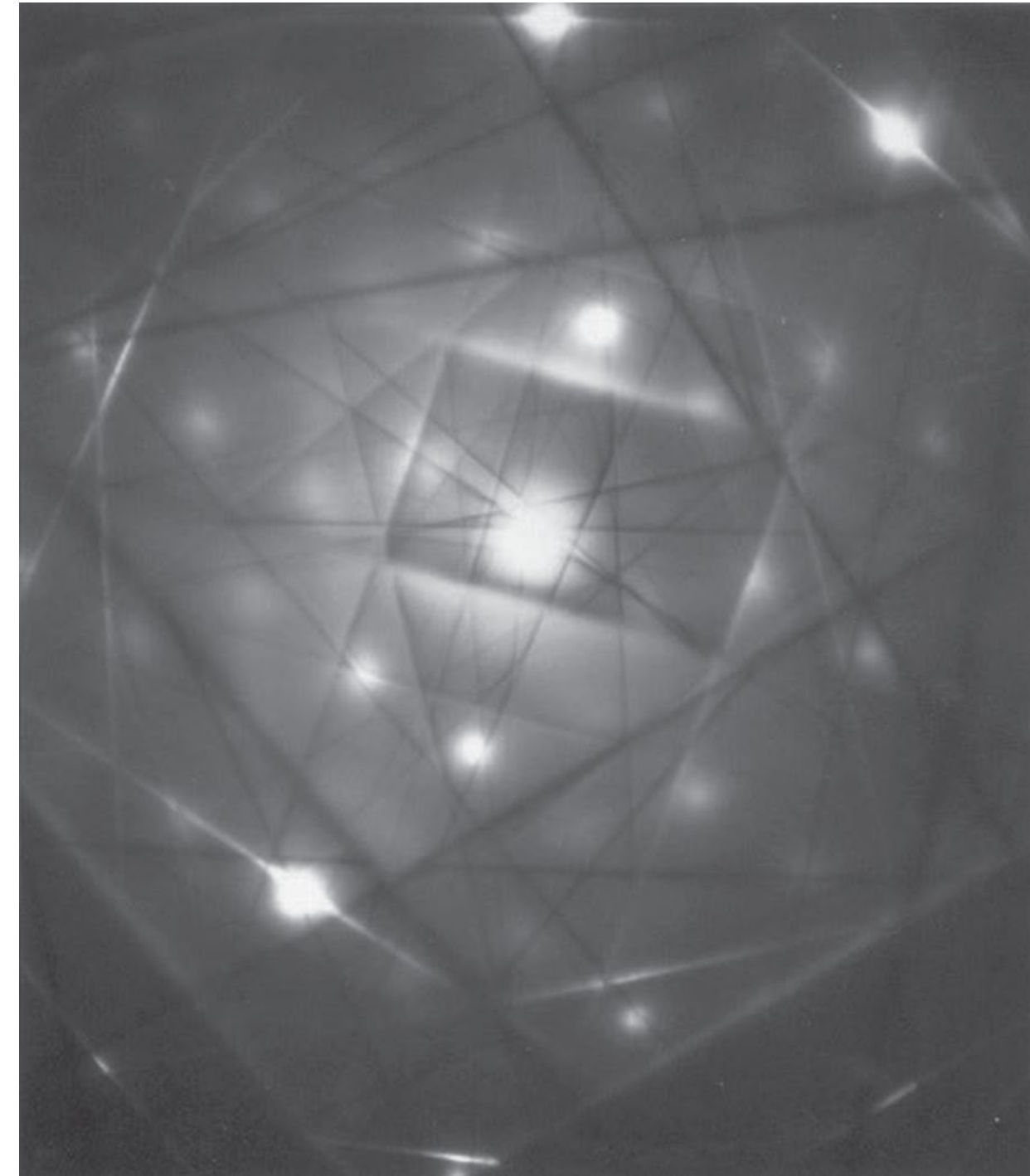
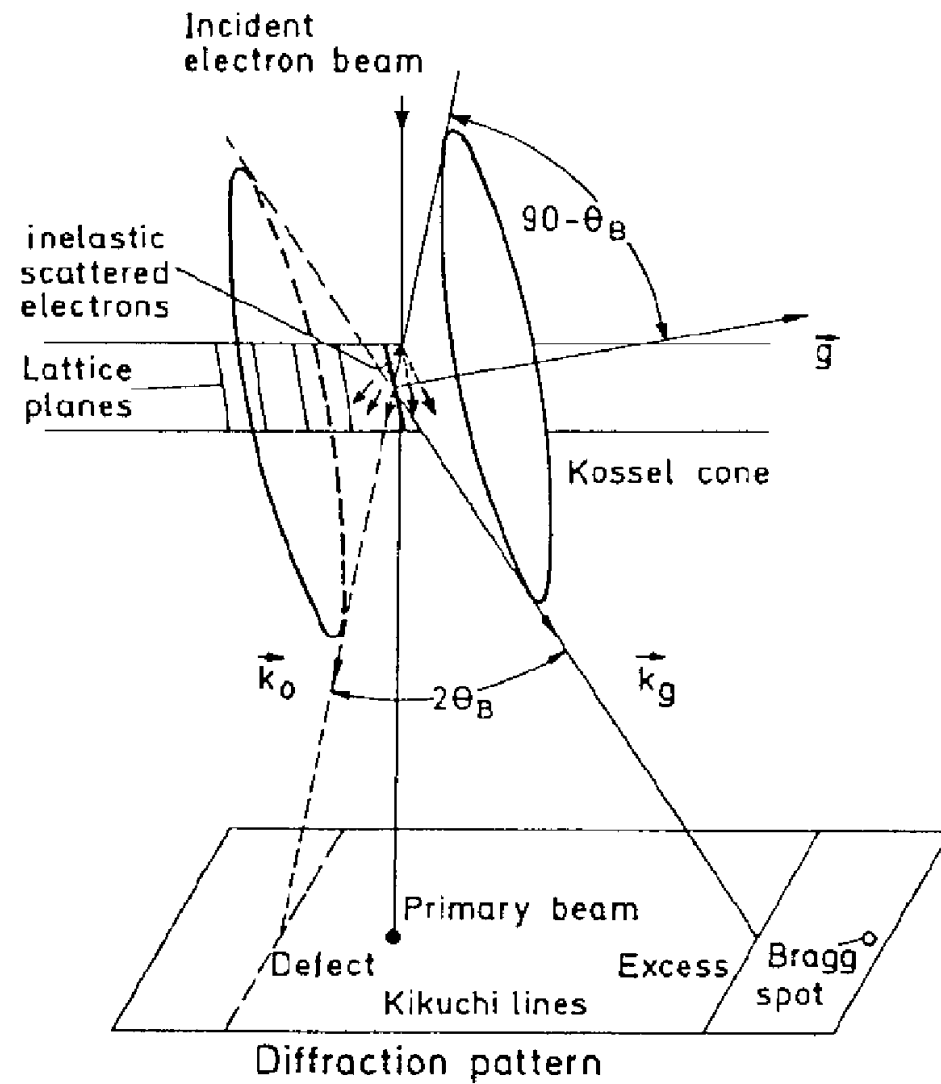
Thicker sample



Diffuse scattering followed by Bragg diffraction



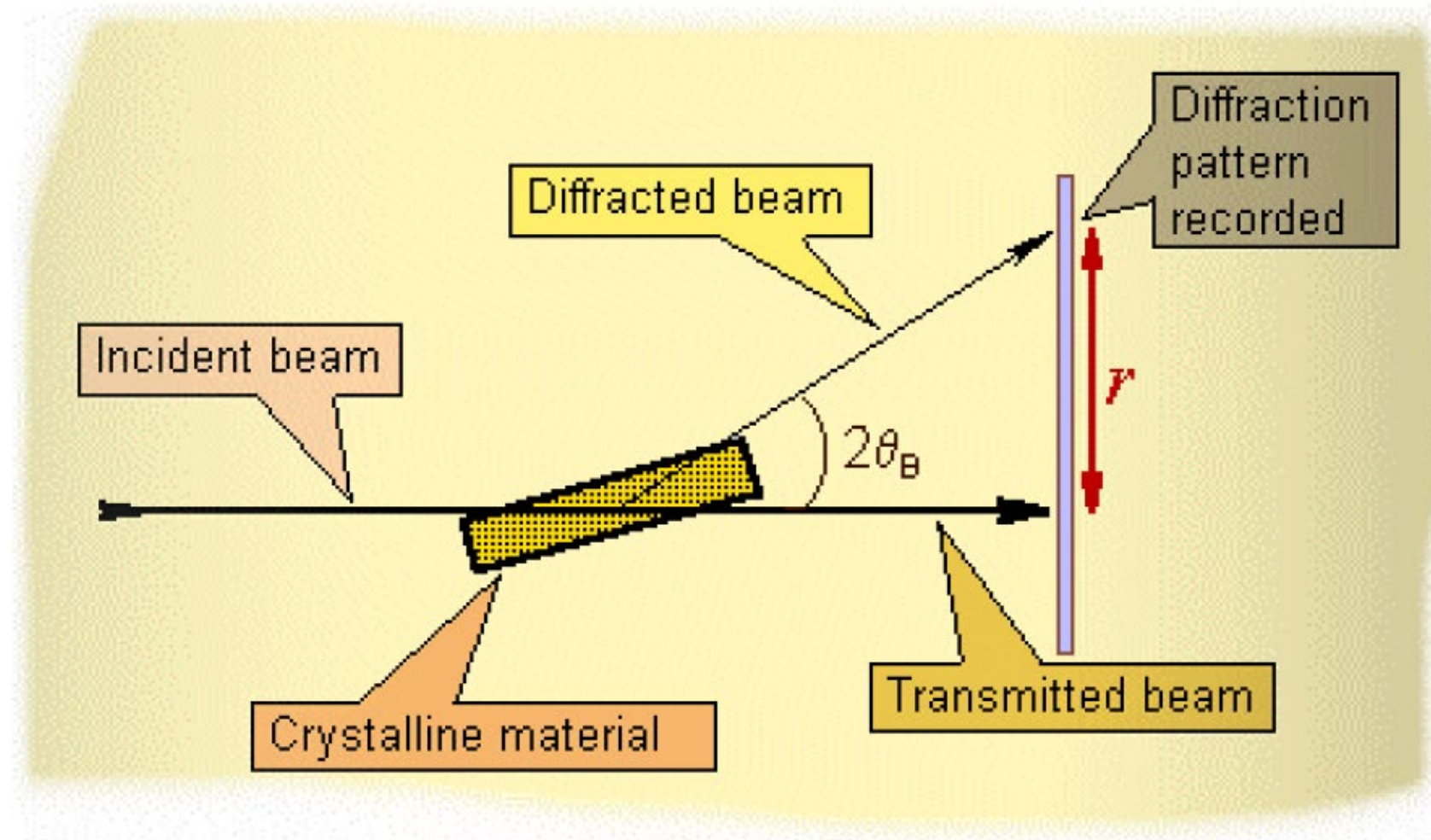
Kikuchi lines (bands)



Thick sample: inelastic scattering followed by elastic scattering (= diffraction)

(powder) X-Ray Diffraction

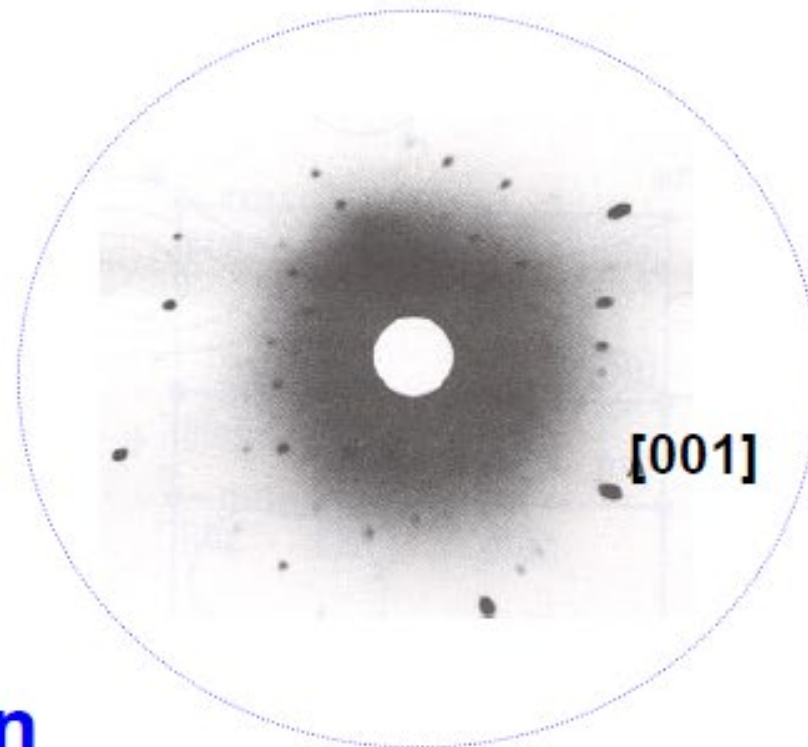
XRD



www.micro.magnet.fsu.edu/primer/java/interference/index.html

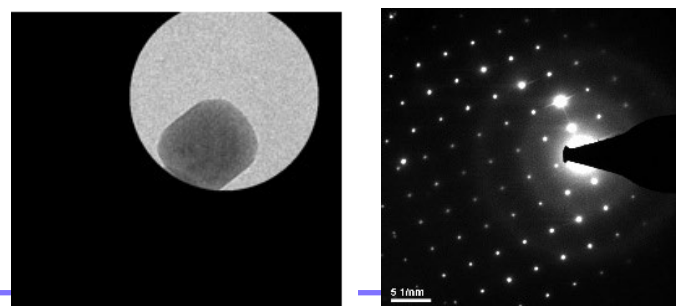
Laue Method

Back-reflection Laue

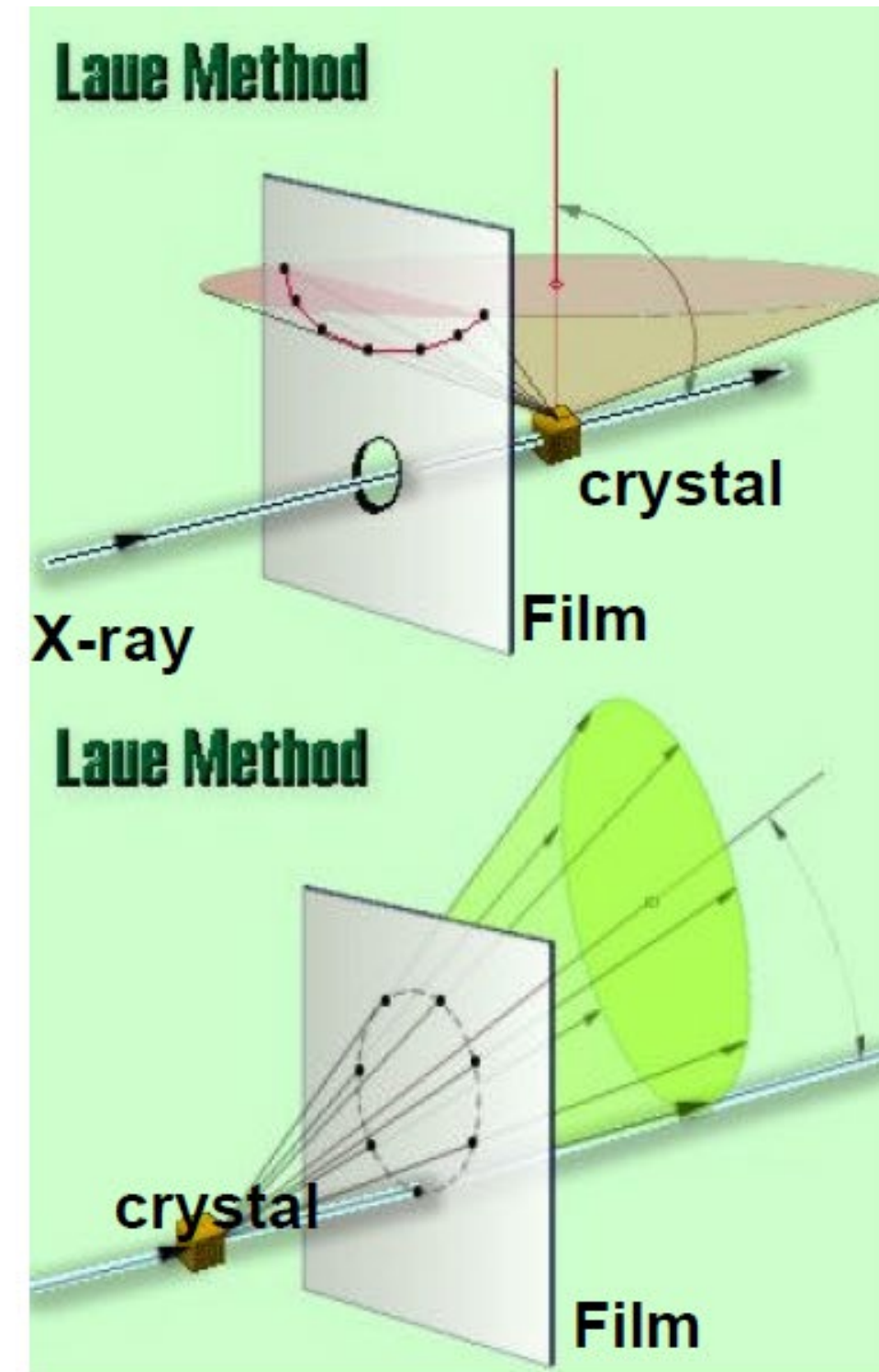


pattern

Transmission Laue

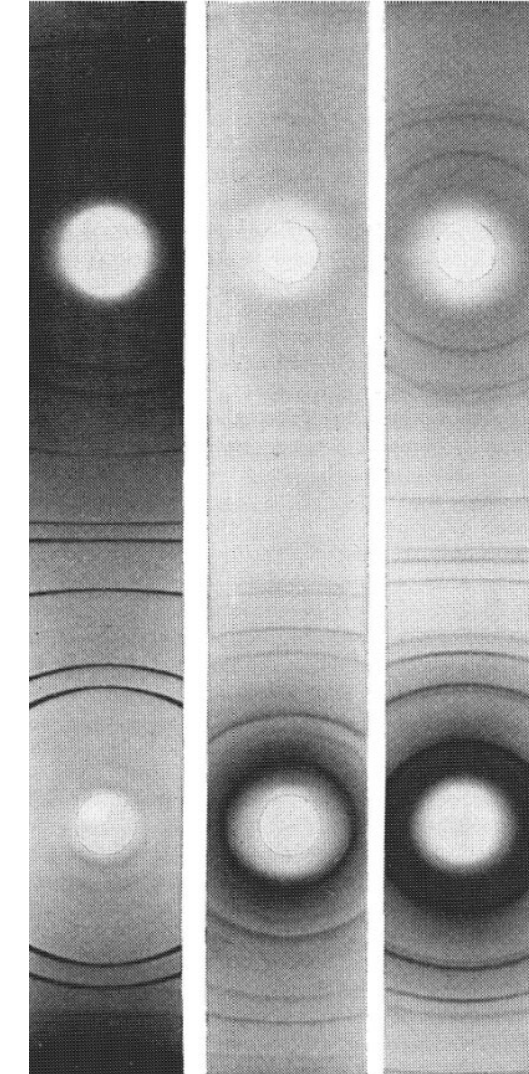
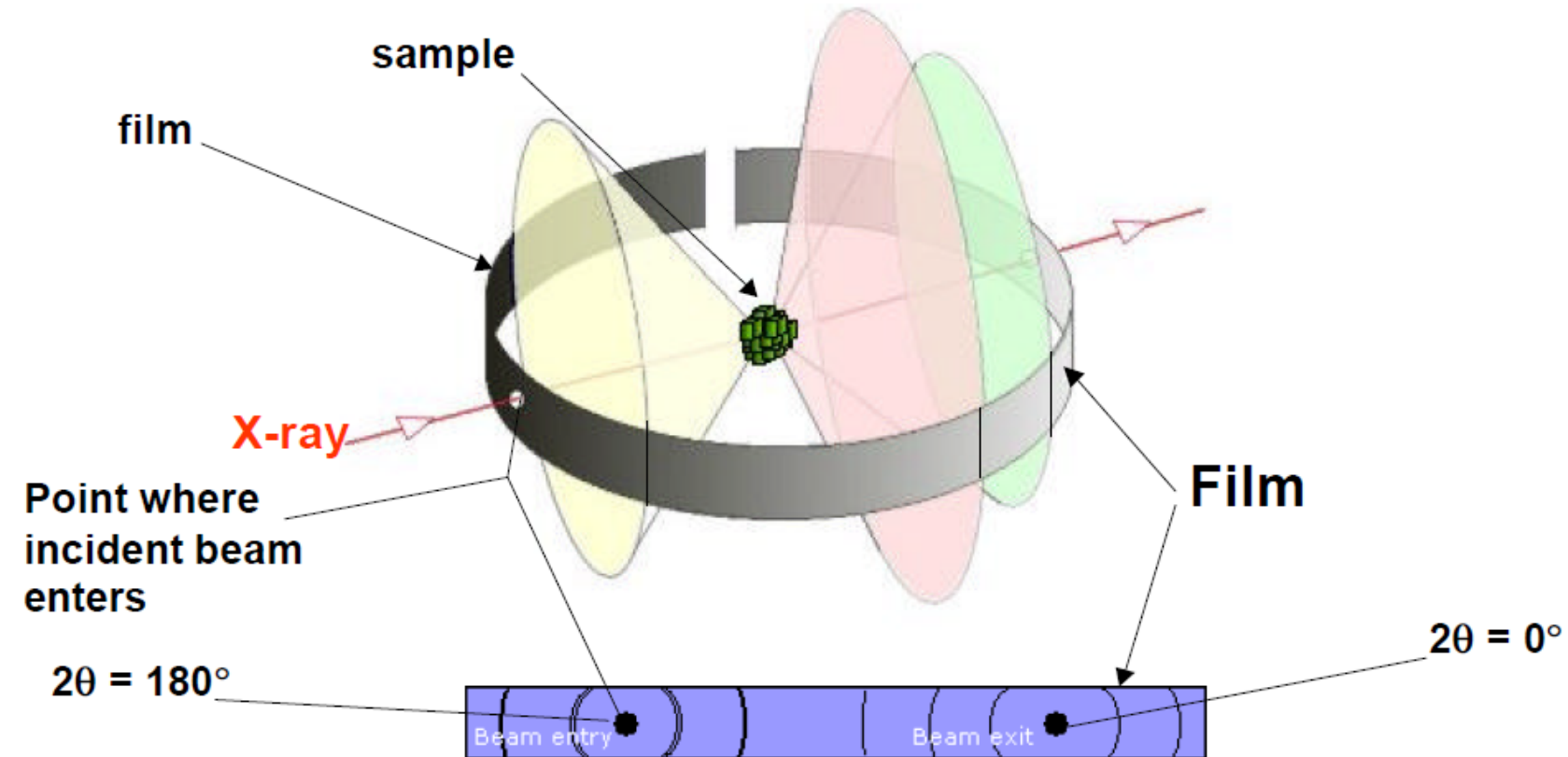


In TEM: ~ SAED



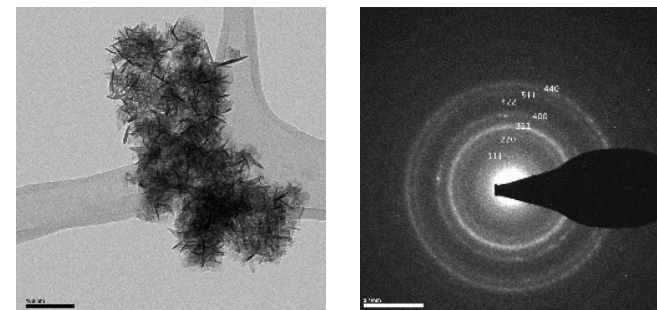
Debye-Scherrer

Detection of Diffracted X-rays by Photographic film



Debye - Scherrer Camera

A sample of some hundreds of crystals (i.e. a powdered sample) show that the diffracted beams form continuous cones. A circle of film is used to record the diffraction pattern as shown. Each cone intersects the film giving diffraction lines. The lines are seen as arcs on the film.

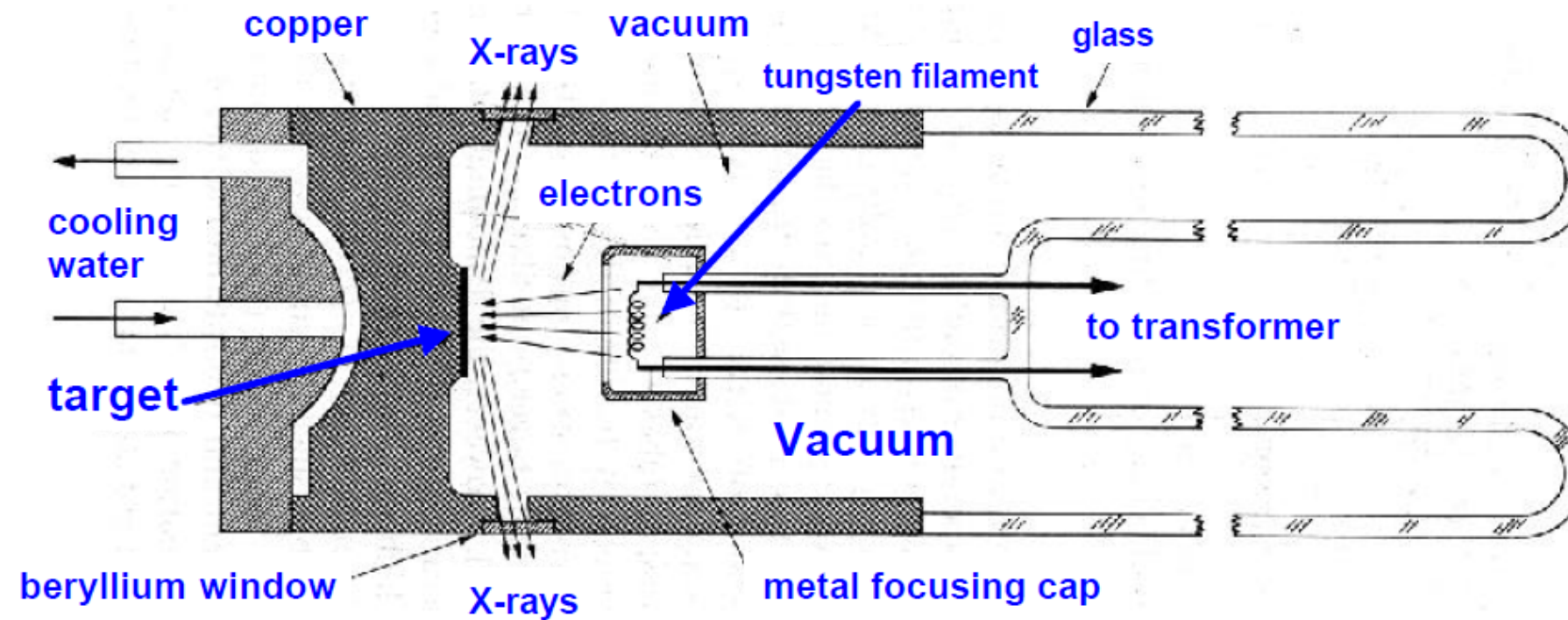


In TEM: ring pattern
of fine powder

X-ray tube

3.0 Production of X-rays

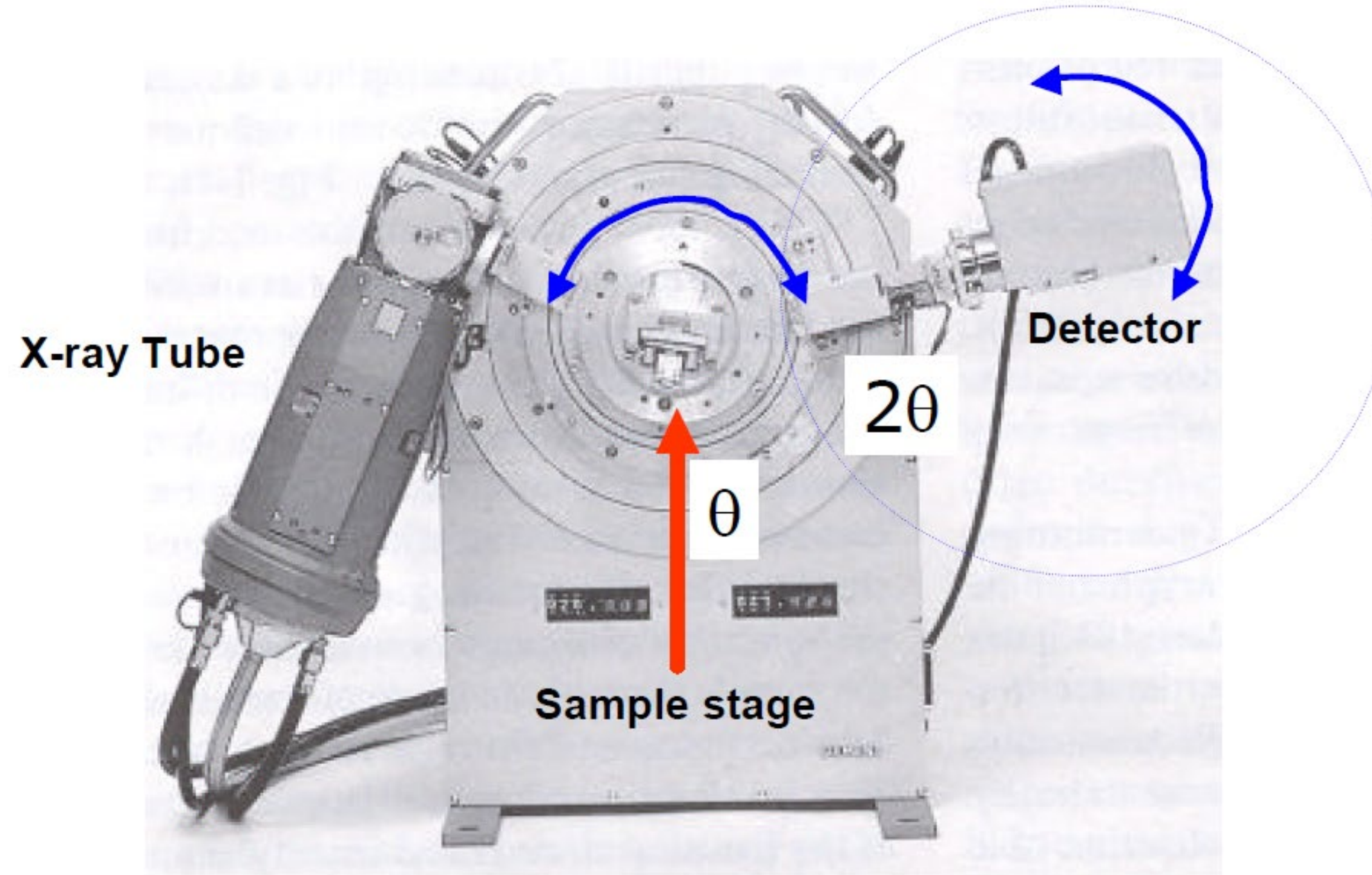
Cross section of sealed-off filament X-ray tube



X-rays are produced whenever high-speed electrons collide with a metal target. A **source of electrons** – hot W filament, a **high accelerating voltage** between the cathode (W) and the anode and a **metal target**, **Cu**, Al, Mo, **Mg**. The anode is a water-cooled block of Cu containing desired target metal.

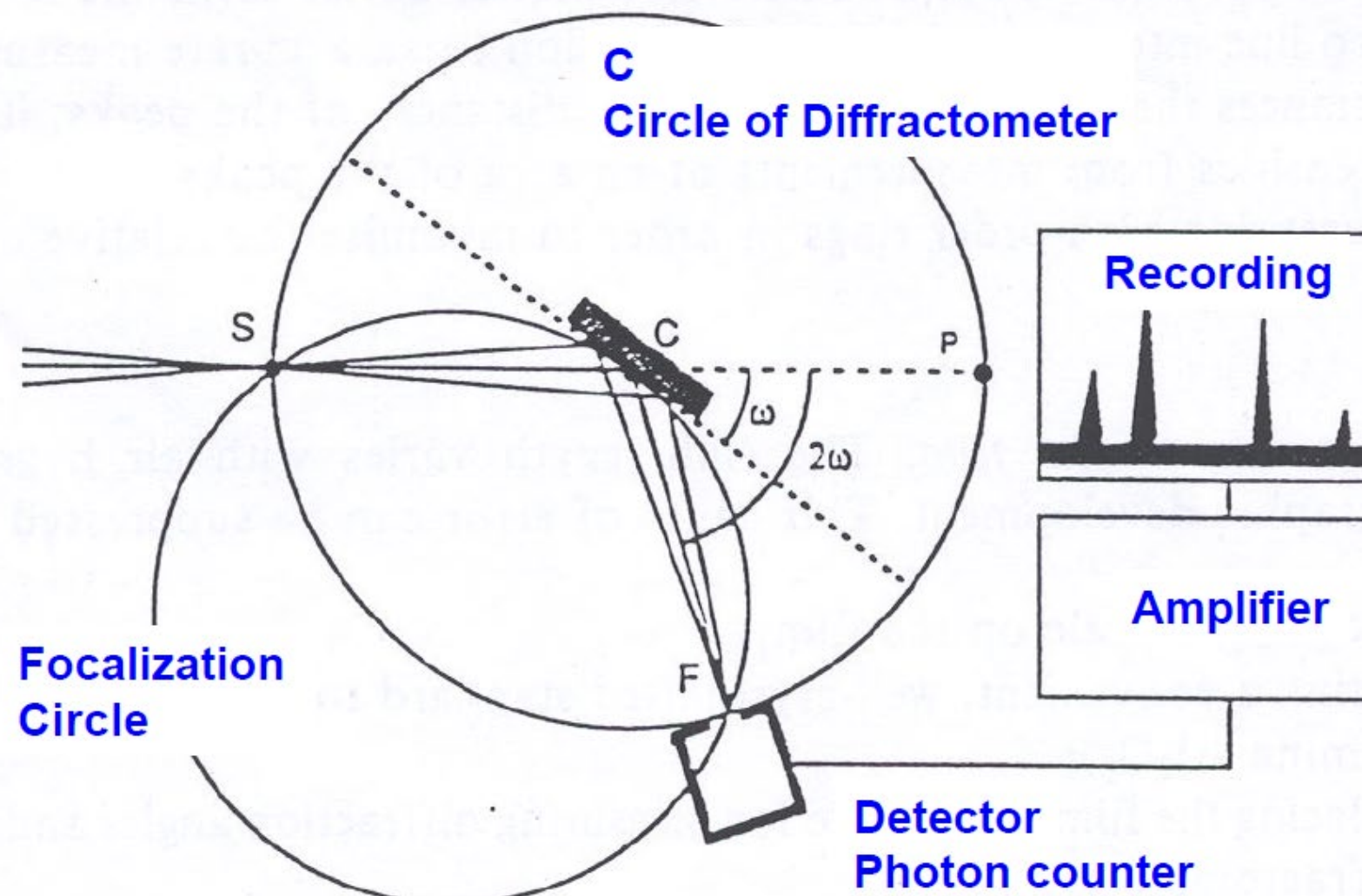
Diffractometer

A Modern Automated X-ray Diffractometer



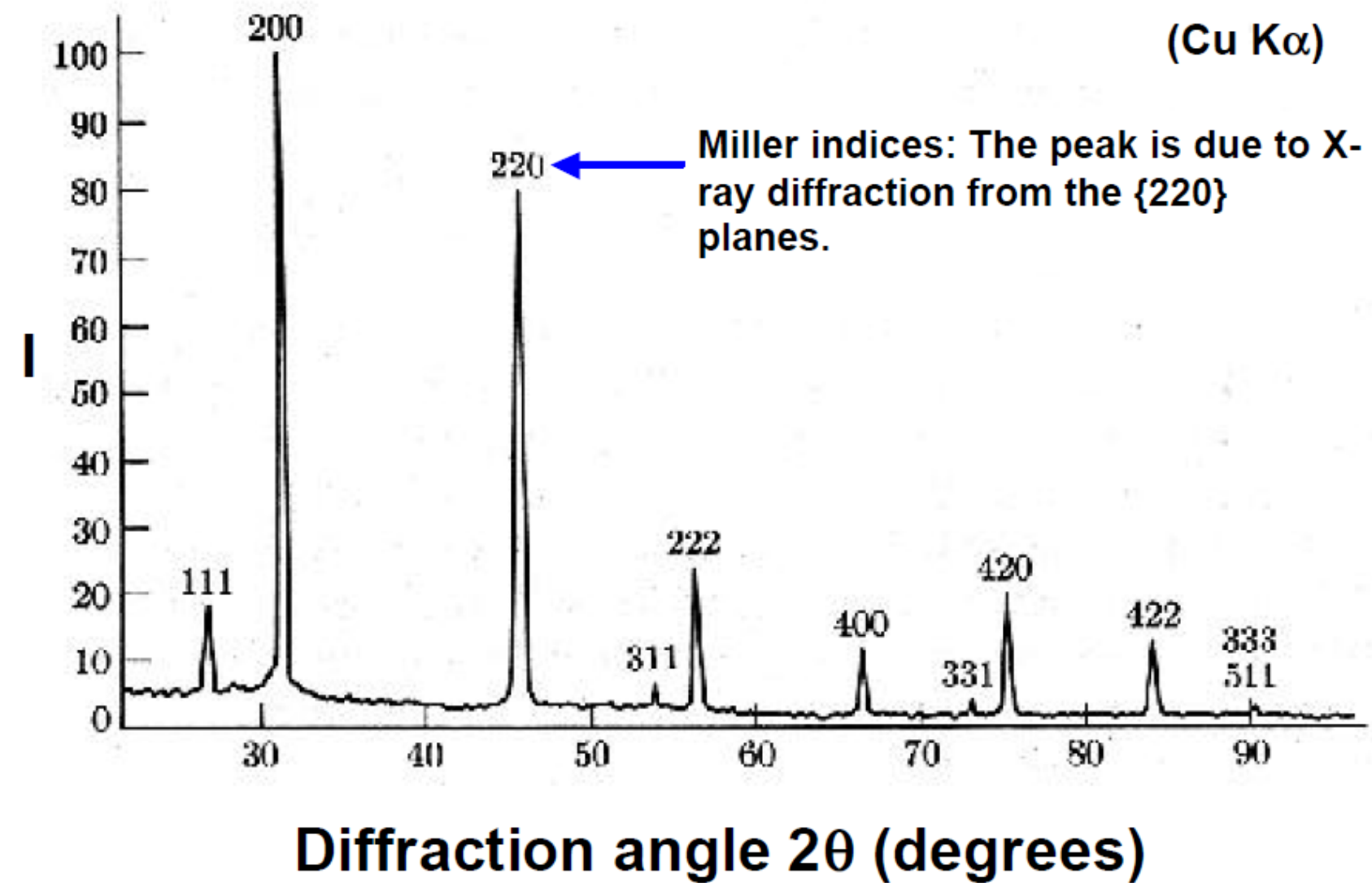
Cost: \$560K to 1.6M

Detection of *Diffracted* X-rays by a Diffractometer



Bragg - Brentano Focus Geometry, Cullity

XRD Pattern of NaCl Powder



One of the most important uses of XRD!!!

- Obtain XRD pattern
- Measure d-spacings
- Obtain integrated intensities
- Compare data with known standards in the JCPDS file, which are for random orientations (there are more than 50,000 JCPDS cards of inorganic materials).

