# 9. Diffraction

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CIME



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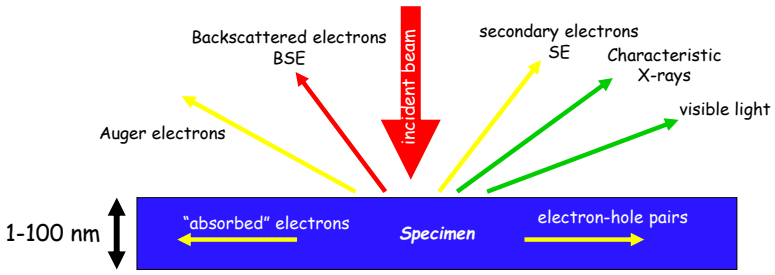
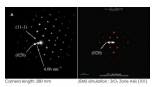
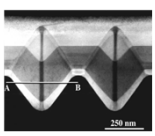
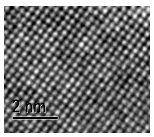
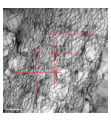
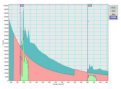
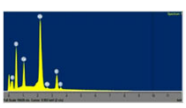
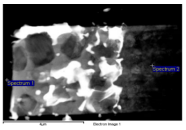
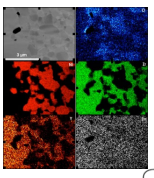
## Signals from a thin sample







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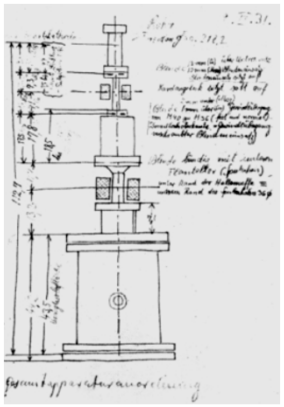
# History

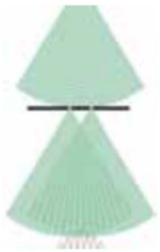
1897: J.J. Thomson: predicts the existence of electrons

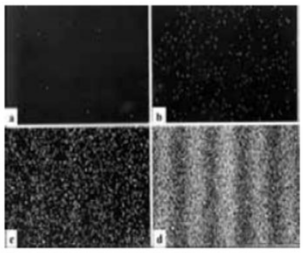
1923: De Broglie: concept of wavelength associated to particles, confirmation by Young's experiment

1927: Busch: focalisation law for magnetic fields, Davisson, Gremer, Thomson: electron diffraction

1931: Ruska, Knoll: first images by electron microscope









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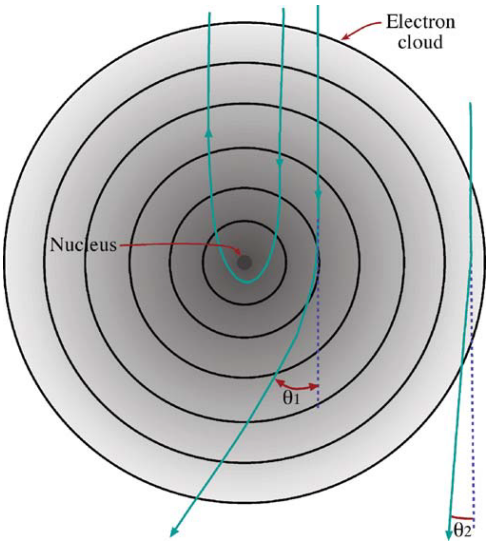
# scattering of electrons by atom(s)




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



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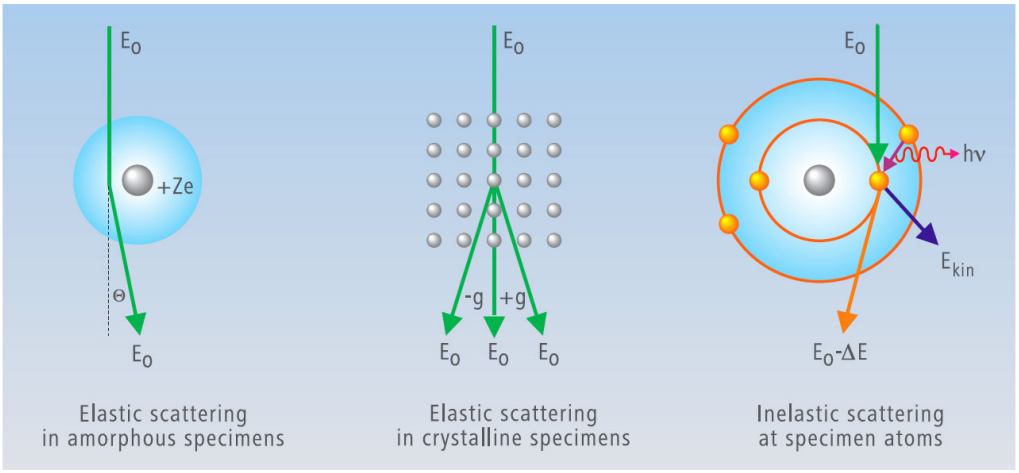
## interaction of high energetic electrons with matter




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)



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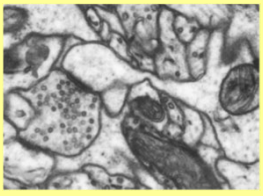
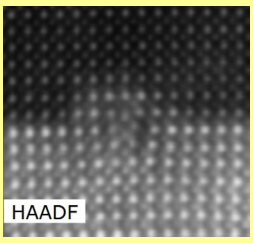
## interaction -> contrast

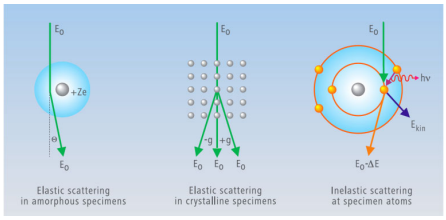
elastic, incoherent



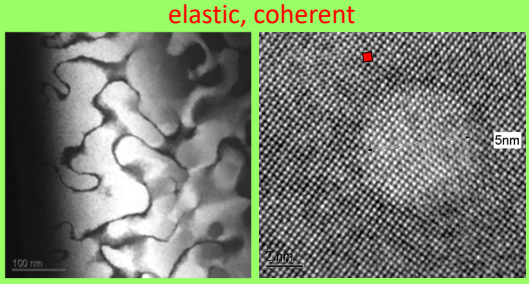
Thin section of mouse brain: mass contrast of stained membrane structures
(G.Knott)




High-resolution STEM image
«z»-contrast



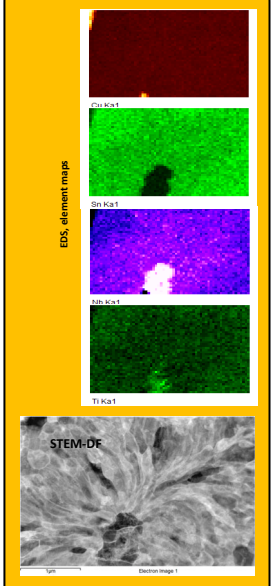

elastic, coherent




Dark field image of
differently ordered
domains:
Diffraction contrast

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

inelastic




Element distribution maps
of Nb3Sn superconductor



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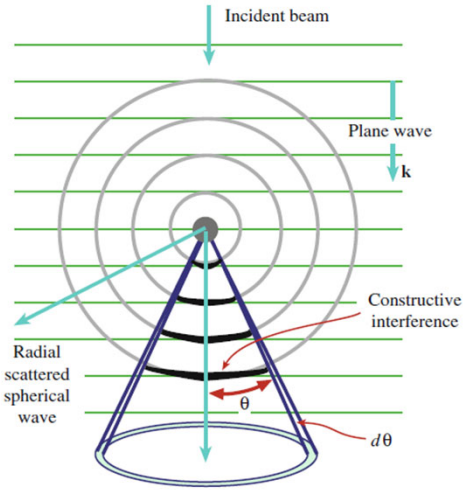
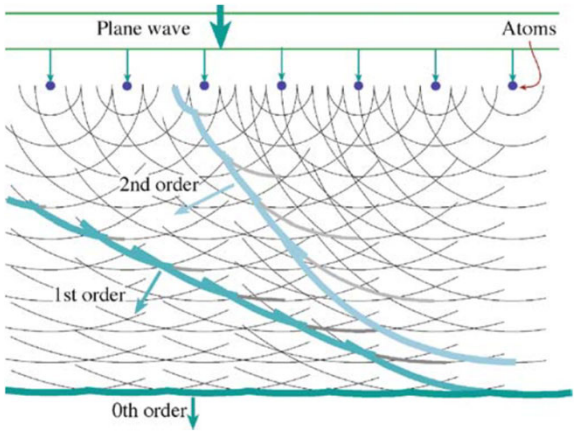
# single atom scattering & Huygens principle




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.



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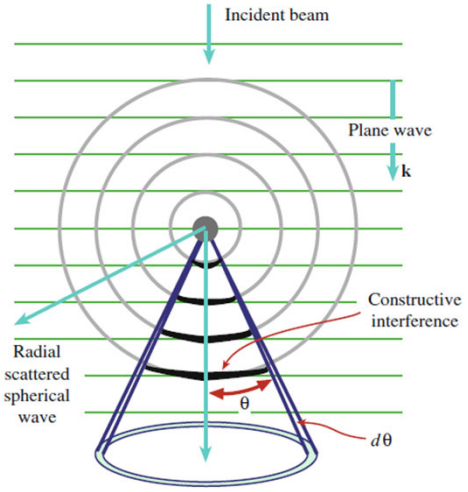
# single atom scattering & Huygens principle

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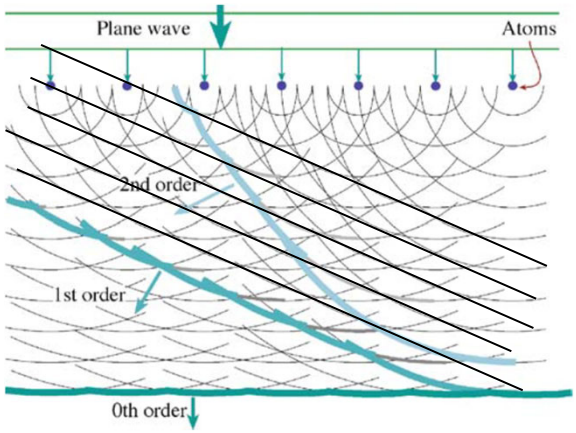


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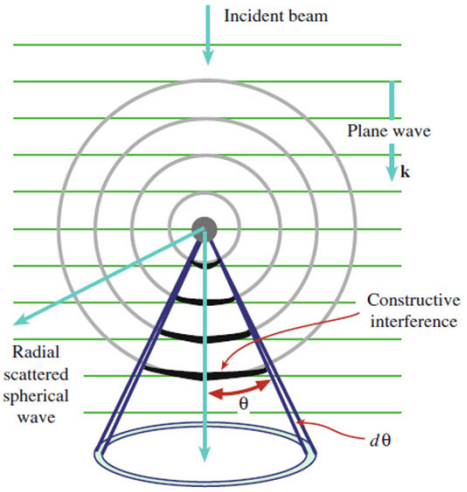


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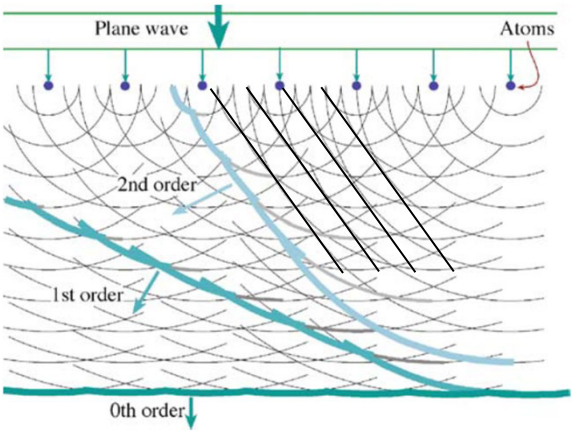


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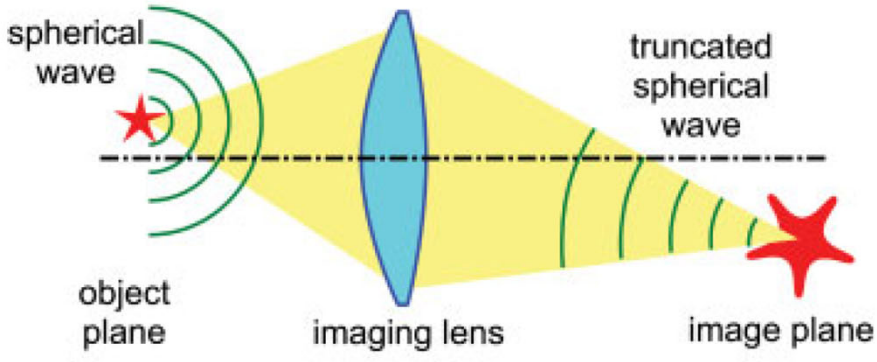
# Abbé's principle in light optics



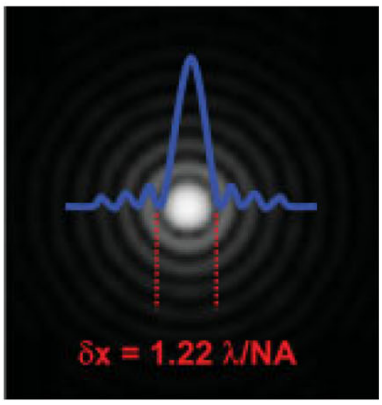




**Figure 21-2:** Image formation of self-luminous objects.

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

x: resolution (point spread function)
λ: wave length
NA: numerical aperture (lens diameter)



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# Abbé: image formation by diffraction

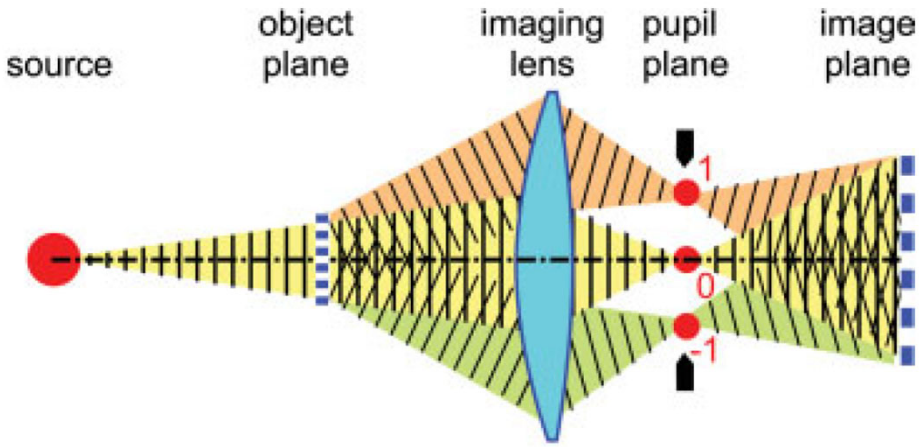


**Figure 21-3:** Abbe Theory of image formation by diffraction and interference.



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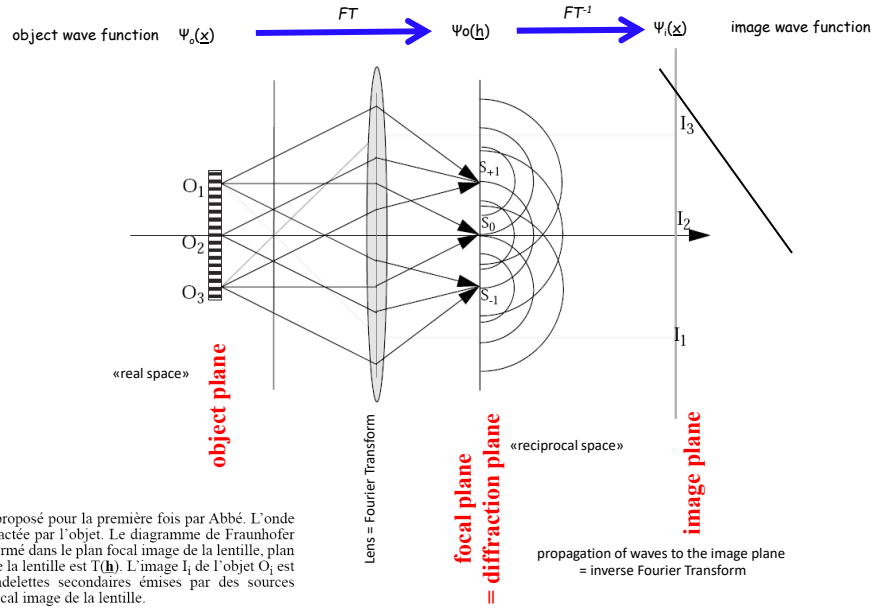
# Abbe's principle




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 $T(\underline{\mathbf{h}})$. 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.



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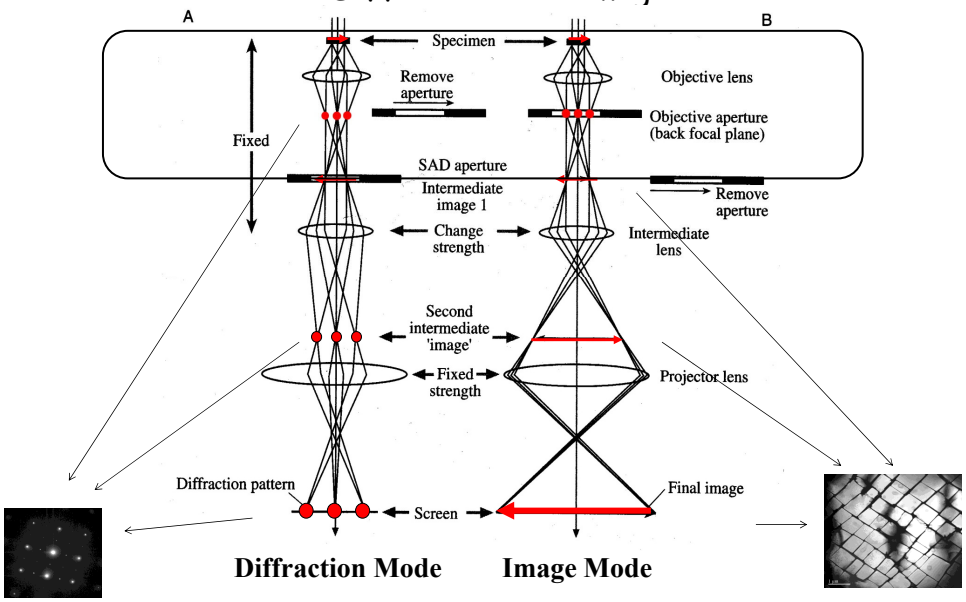
## Two basic operation modes
## Diffraction <-> Image



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## Content

Introduction to electron diffraction

Elastic scattering theory

Basic crystallography & symmetry

Electron diffraction theory

Convergent beam electron diffraction

Kikuchi patterns

X-ray diffraction



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# 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 $e^-$, λ = 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



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# scatter range of electrons, neutrons and X-rays

(99% of intensity lost)

| Elément (masse spécifique) | 4-Be 1.84 g/cm³ | 13-Al 2.7 g/cm³ | 29-Cu 8.93 g/cm³ | 82-Pb 11.3 g/cm³ |
|---|---|---|---|---|
| Rayons X<br>Cu-K$\alpha$ $\lambda$=1.54 Å<br>Mo-K$\alpha$ $\lambda$=0.71 Å | 16 mm<br>83 mm | 0.35 mm<br>3.3 mm | 0.10 mm<br>0.10 mm | 0.017 mm<br>0.034 mm |
| Neutrons thermiques<br>$\lambda \approx 1.08$ Å | 89 m | 6 m | 0.26 m | 14 m |
| Électrons<br>$\lambda$=0.037 Å à 100 kV<br>$\lambda$=0.020 Å à 300 kV | 39 µm | 42 µm<br>~330 µm | 11 µm | 0.6 µm |



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# electron, neutron X-ray scattering

| wave | Wavelength λ | Source | Scattering at | Differentiation of | Sample size |
|---|---|---|---|---|---|
| X-rays | 0.07nm<br>0.15nm | X-ray tubes<br>synchrotrons | Electron cloud | Lattice parameters<br>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 : $10^4$



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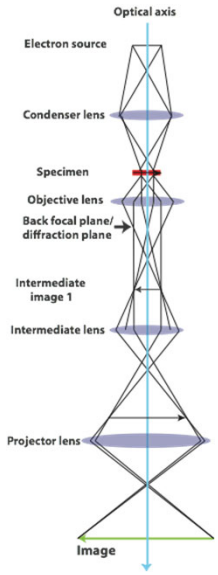
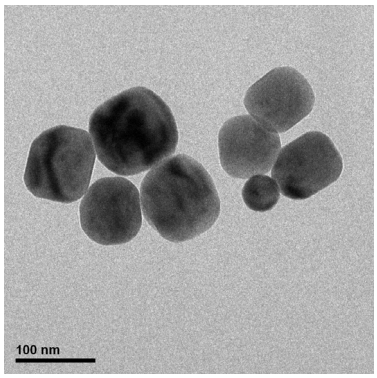
# Image formation




$BaTiO_3$ nanocrystals (Psaltis lab)

Insert selected area aperture to choose region of interest



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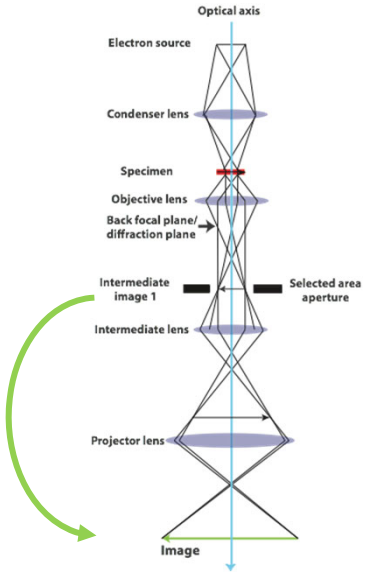
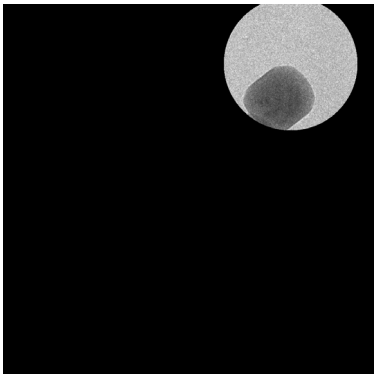
# Image formation



$BaTiO_3$ nanocrystals (Psaltis lab)

Insert selected area aperture to choose region of interest



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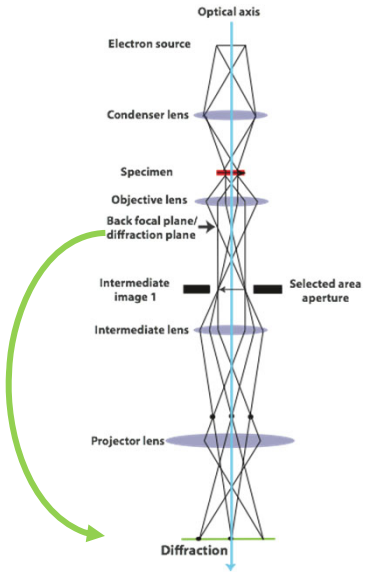
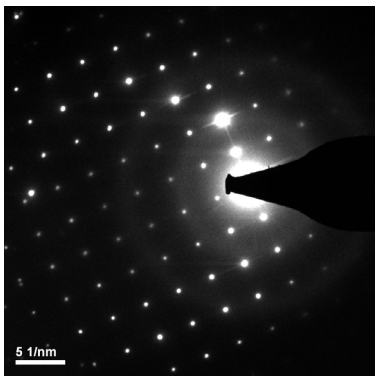
# Take selected-area diffraction pattern




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

Find cubic $BaTiO_3$ aligned on [0 0 1] zone axis



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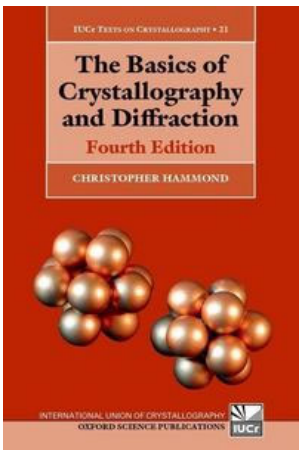
# Basic crystallography & symmetry



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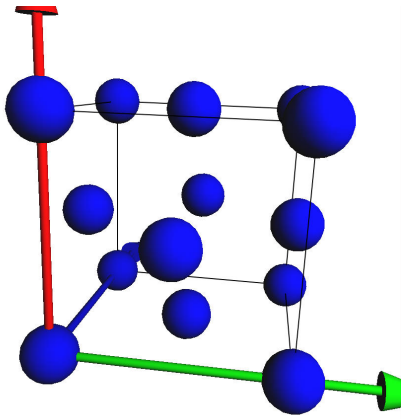
## Crystals: translational periodicity & symmetry



Repetition of translated structure to infinity



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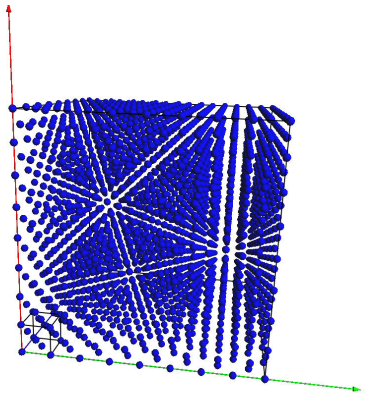
## Crystals: translational periodicity & symmetry



Repetition of translated structure to infinity



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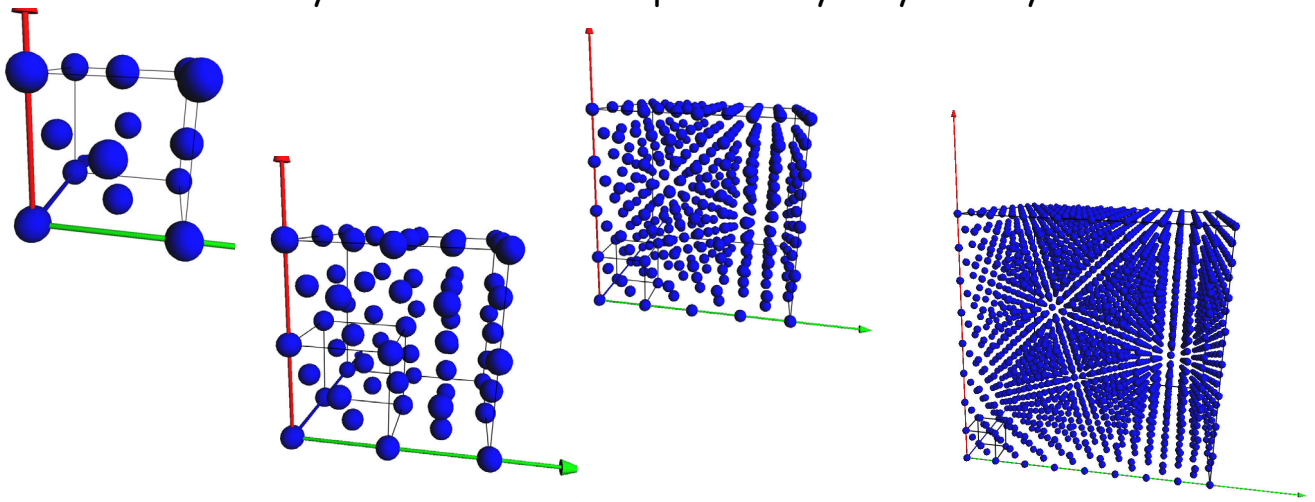

Repetition of translated structure to infinity



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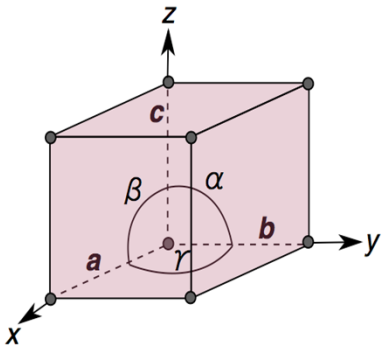
## Crystallography: the unit cell

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: $\alpha = b\hat{}c$; $\beta = a\hat{}c$; $\gamma = a\hat{}b$



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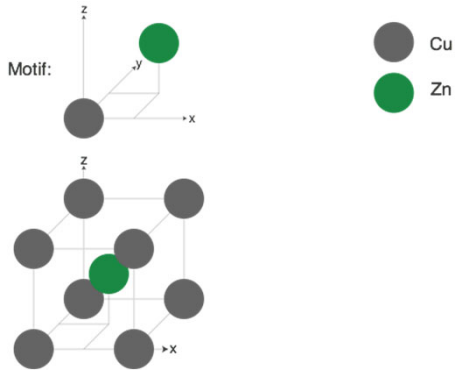
# Building a crystal structure adding atoms

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



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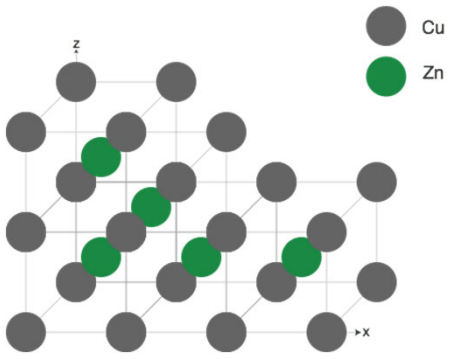
Extend lattice further into space



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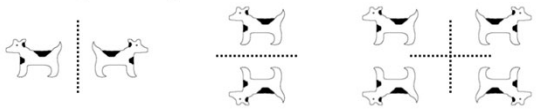
# Introduction to symmetry

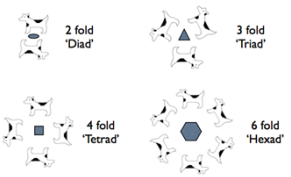
As well as having translational symmetry, nearly all crystals obey other symmetries - i.e. can reflect or rotate crystal and obtain exactly the same structure

Symmetry elements:

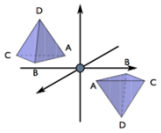
Mirror planes:



Rotation axes:




Centre of symmetry or inversion centre:



Inversion axes: combination of rotation axis with centre of symmetry



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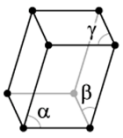
# the seven crystal systems

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

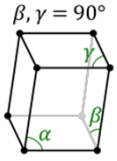
Triclinic

$\alpha,\beta,\gamma \neq 90°$



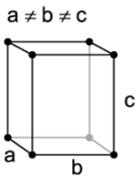

Monoclinic

$\alpha \neq 90°$
$\beta, \gamma = 90°$



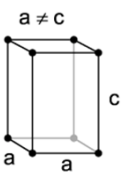

Orthorhombic

$a \neq b \neq c$



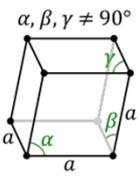

Tetragonal

$a \neq c$

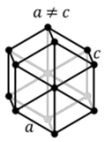



Rhombohedral

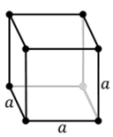
$\alpha, \beta, \gamma \neq 90°$




Hexagonal




Cubic




"primitive unit cells"

Diagrams from www.Wikipedia.org



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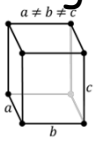
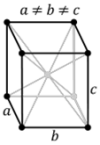
# four possible lattice centerings

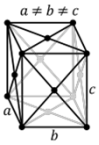
P: Primitive - lattice points on cell corners



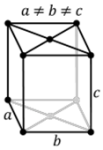

I: Body-centred - additional lattice point at cell centre




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



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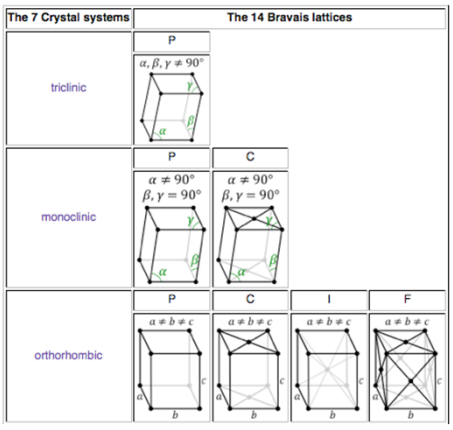
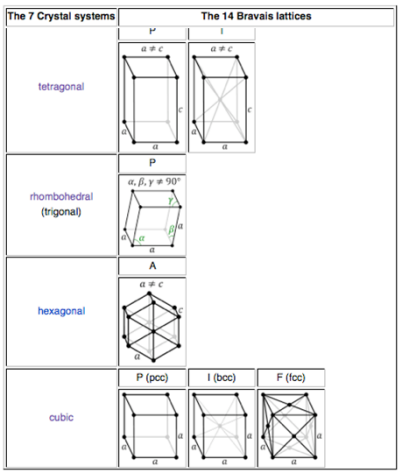
# 14 Bravais lattices

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




Diagrams from www.Wikipedia.org



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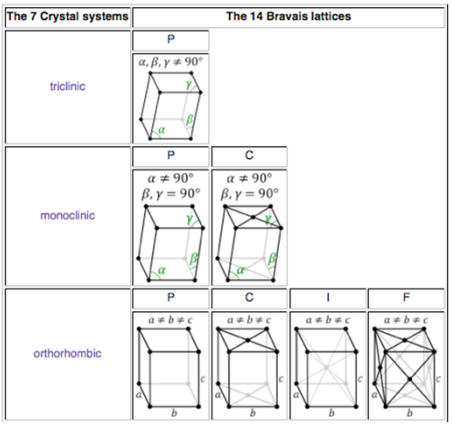
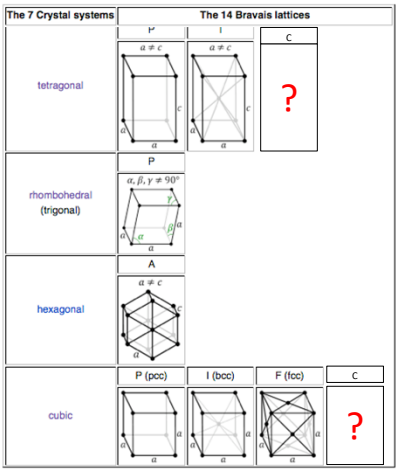
# 14 Bravais lattices

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




Diagrams from www.Wikipedia.org



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# 14 Bravais lattices

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



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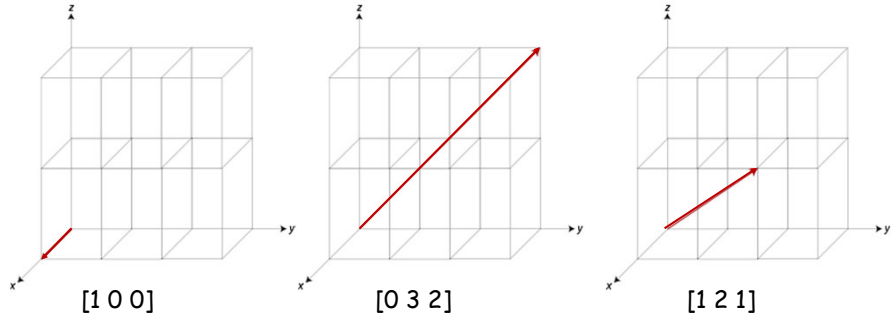
# Crystallography - lattice vectors

A lattice vector t is a vector joining any two lattice points
Written as linear combination of unit cell vectors ***a***, ***b***, ***c***:

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

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

Examples:




[1 0 0] [0 3 2] [1 2 1]

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



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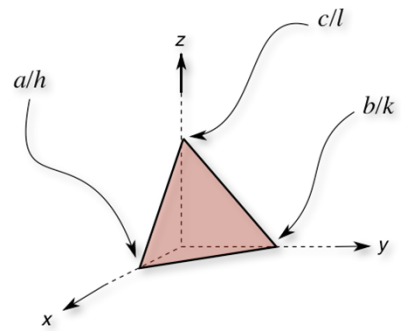
# Crystallography - lattice planes

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,c axes:

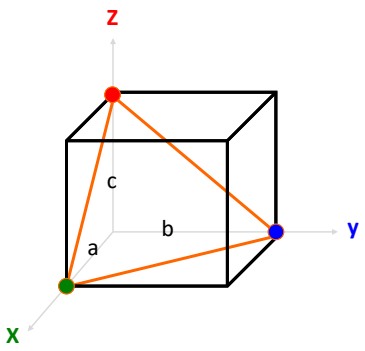
Lattice planes are described using Miller indices (*h k l*) where the *first* plane away from the origin intersects the *x*, *y*, *z* axes at distances:

*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



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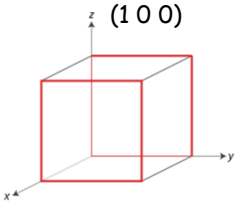
# Crystallography - lattice planes

Sets of planes intersecting the unit cell - examples:

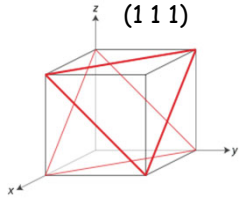
(1 0 0)



intersection at ( a, ∞, ∞ )

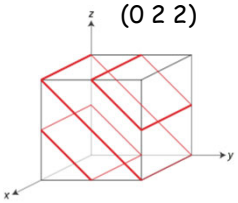
*intersection with coordinate axes (x,y,z) on*
*a/h on the x axis*
*b/k on the y axis*
*c/l on the z axis*

(1 1 1)



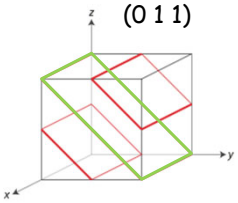
intersection at ( 1*a, 1*b, 1*c )

(0 2 2)



intersection at (∞, 1/2*b, 1/2*c )

(0 1 1)



intersection at (∞, b, c )



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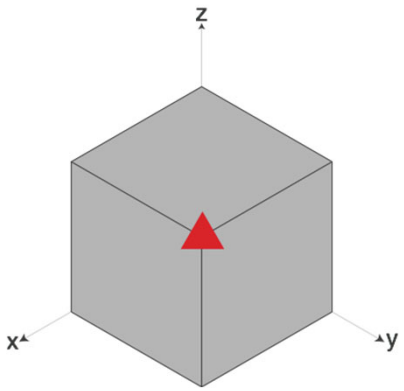
# Lattice planes and symmetry

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)



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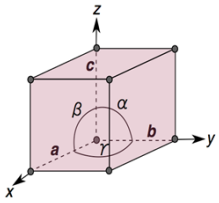
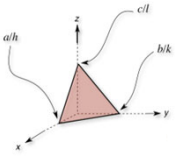
# Introduction to Crystallography

UNIT CELL

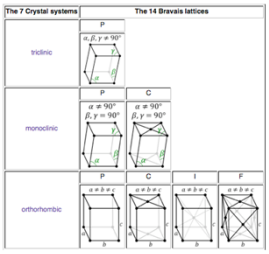



[UVW]: lattice vectors, direction, "zone axes"
(hkl): lattice planes (Miller indices)
{khkl}: equivalent lattice planes




unit cell
decoration
MOTIF






Bravais LATTICE



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# Elastic scattering theory



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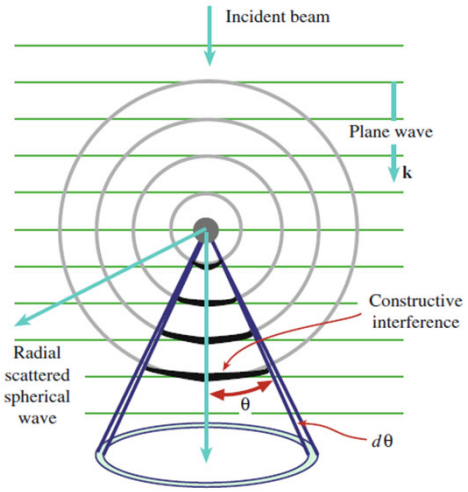
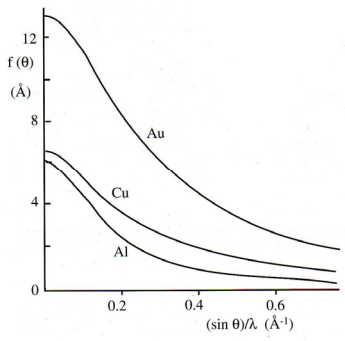
# Scattering theory - atomic scattering factor f(θ)

## 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 ($f_x$)



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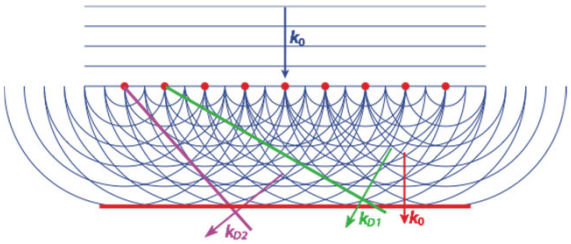
# Scattering theory - Huygen's principle

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!



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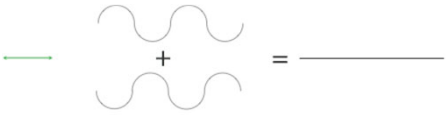
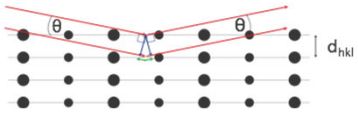
# Diffraction theory - Bragg law

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

$2d_{hkl}\sin\theta = \lambda/2$ - destructive interference





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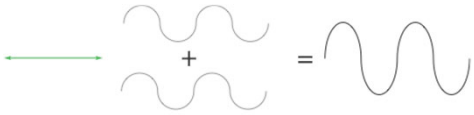
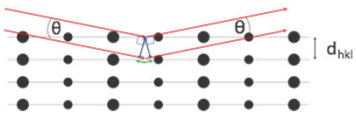
# Diffraction theory - Bragg law

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

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






Electron diffraction: $\lambda \sim 0.001$ nm
therefore: $\lambda << d_{hkl}$
=> small angle approximation: $n\lambda \approx 2d_{hkl}\theta$

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



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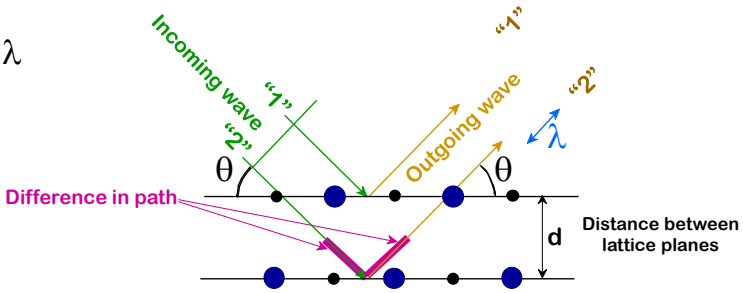
# Bragg's law

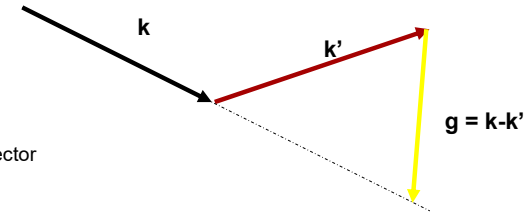
$2 \sin\theta\, d_{hkl} = n \lambda$

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




**Elastic diffraction**

$|\mathbf{k}| = |\mathbf{k'}|$




Periodic arrangement of lattice planes:
**g** : « diffraction vector », reciprocal lattice vector



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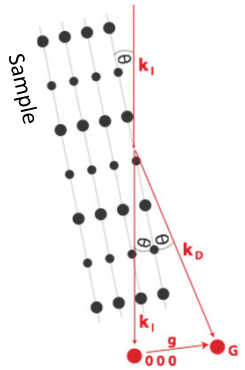
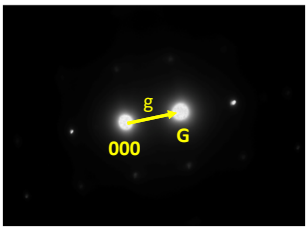
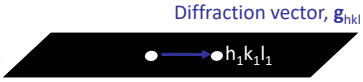
# Diffraction theory - 2-beam condition




Observed diffraction pattern

Diffraction vector, $\mathbf{g}_{hkl}$

Focal plane 


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

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

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



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# Multi-beam scattering condition

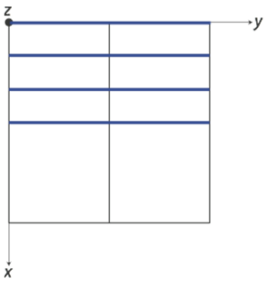
Electron beam parallel to low-index crystal orientation [U V W] = zone axis

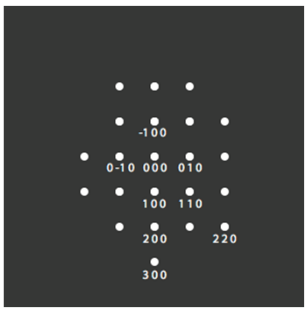
Crystal "viewed down" zone axis is like diffraction grating with planes parallel to e-beam

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 => larger indices (*h k l*)
& greater scattering angle on diffraction pattern from (0 0 0) direct beam



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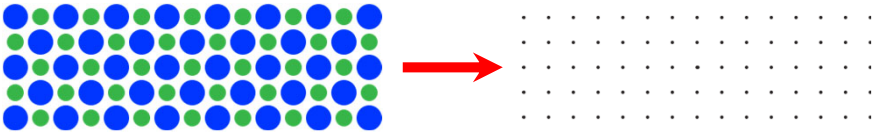
# The reciprocal lattice

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:



Real lattice vector: $\mathbf{r}_n = n_1\mathbf{a} + n_2\mathbf{b} + n_3\mathbf{c}$

Reciprocal lattice vector: $\mathbf{r}^* = m_1\mathbf{a}^* + m_2\mathbf{b}^* + m_3\mathbf{c}^*$

where:

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

**In orthogonal crystal structure:**

**a*** perpendicular to **b** and **c**
= normal of bc plane

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

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

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

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

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

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

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



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# Ewald sphere

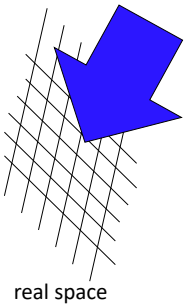
A vector in reciprocal space:

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

diffraction if :

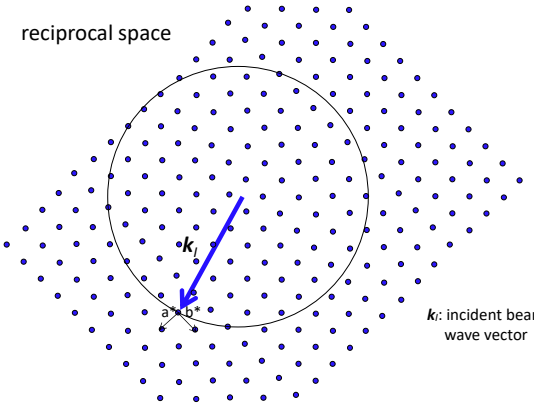
$\mathbf{k}_I - \mathbf{k}_D = \mathbf{g}$ **and** $|\mathbf{k}_I| = |\mathbf{k}_D|$

Bragg **and** elastic scattering



real space

reciprocal space




$k_I$: incident beam wave vector

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

Reciprocal space: sphere radius 1/λ represents possible scattering wave vectors intersecting reciprocal space Ewald sphere

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



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# Ewald sphere

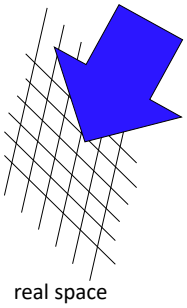
A vector in reciprocal space:

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

diffraction if :

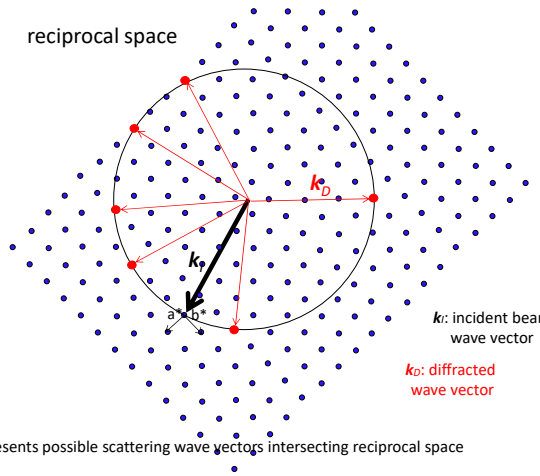
$\mathbf{k}_I - \mathbf{k}_D = \mathbf{g}$ **and** $|\mathbf{k}_I| = |\mathbf{k}_D|$

Bragg **and** elastic scattering



real space

reciprocal space




$k_I$: incident beam wave vector

$k_D$: diffracted wave vector

Reciprocal space: sphere radius 1/λ represents possible scattering wave vectors intersecting reciprocal space

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



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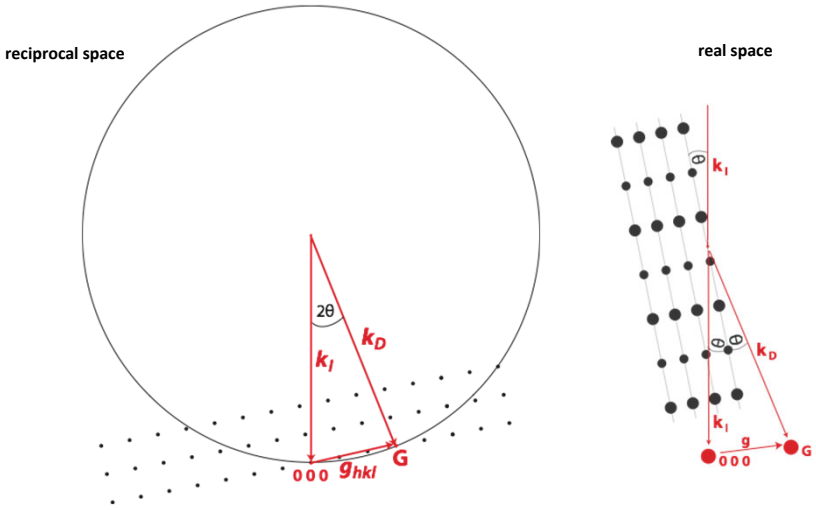
# Ewald sphere in 2-beam condition




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



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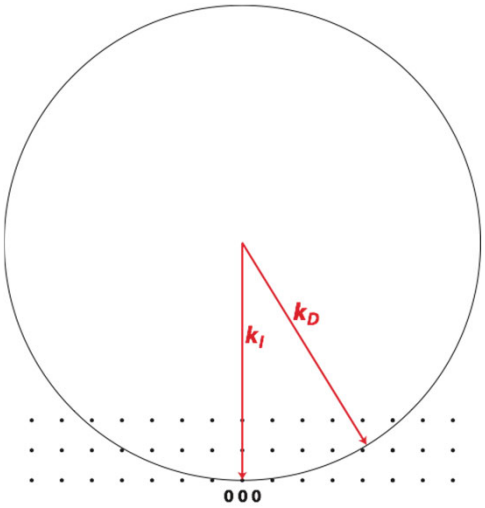
# Ewald sphere and multi-beam scattering scattering




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!



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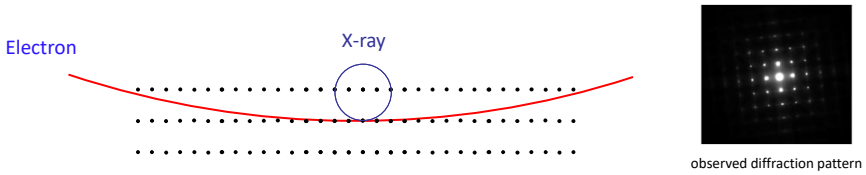
# the reciprocal space
## Almost flat Ewald sphere in electron diffraction

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






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/λ) is much larger for electron diffraction:
**almost flat Ewald sphere**



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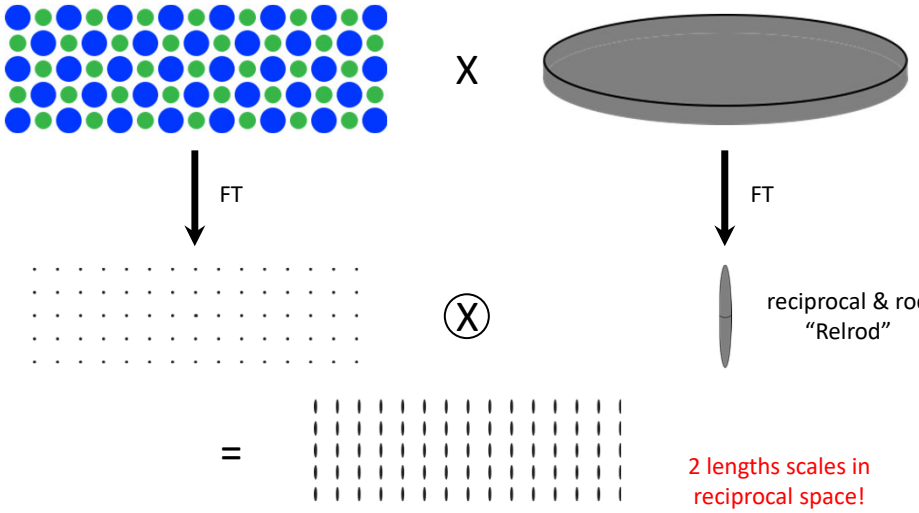
# Fourier transforms and reciprocal lattice

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



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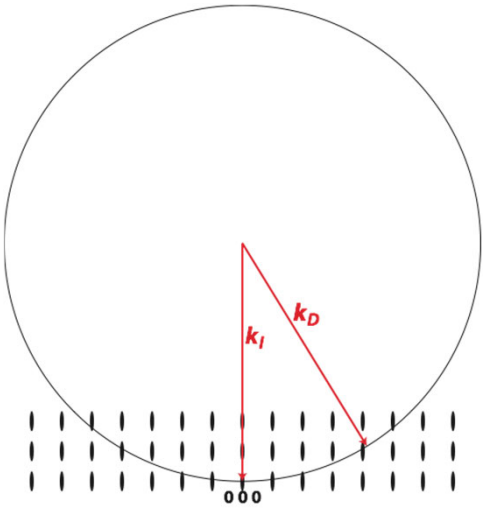
# Ewald sphere intersects Relrods



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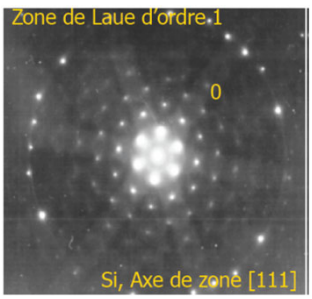
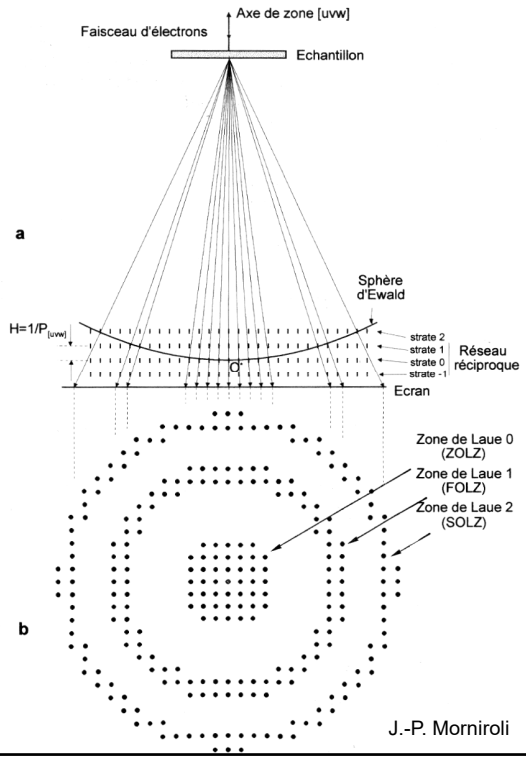
## Laue zone
## "higher order" Laue zones



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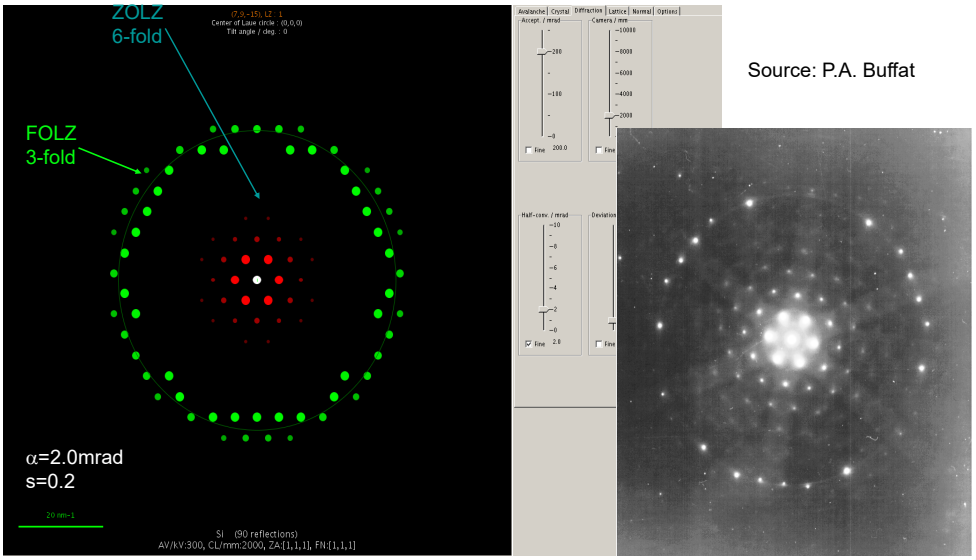
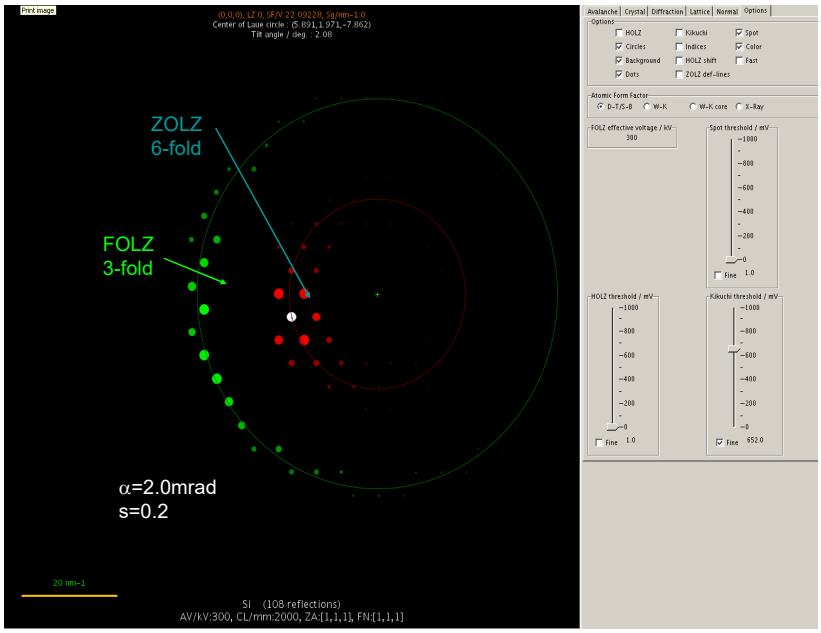
# Sphère d'Ewald : Laue Zones in Si [111], (ZOLZ+FOLZ)




Source: P.A. Buffat



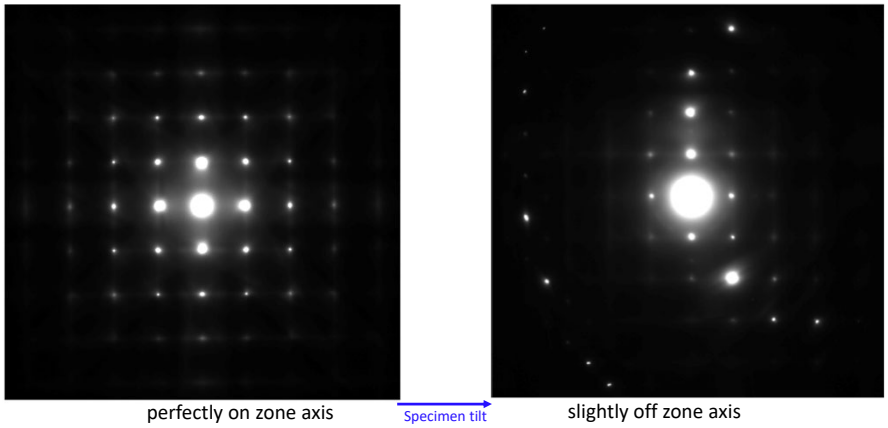
# Sphère d'Ewald : Laue Zones (ZOLZ+FOLZ) échantillon incliné



# 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



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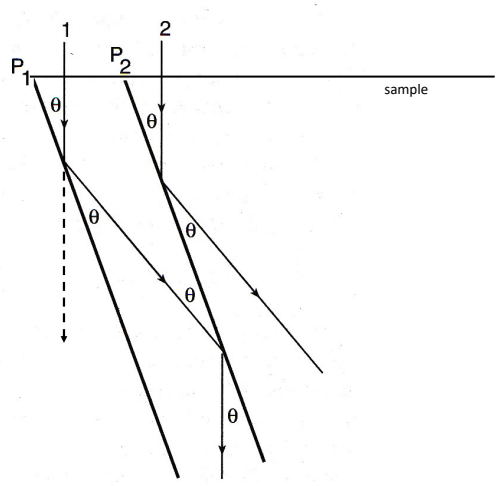
# Dynamical scattering

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



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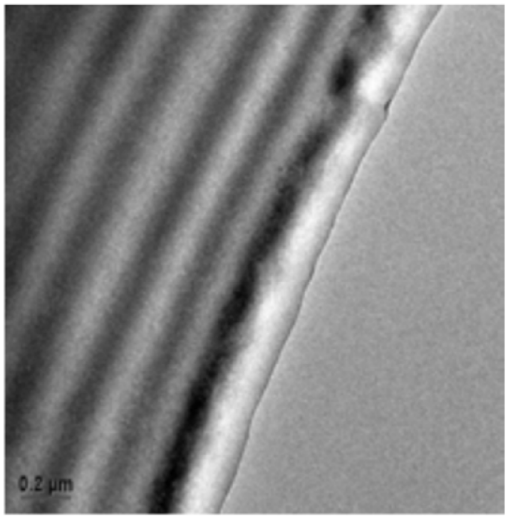
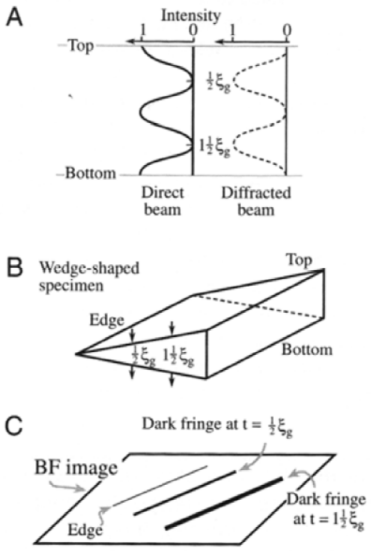
# Dynamical scattering thickness fringes



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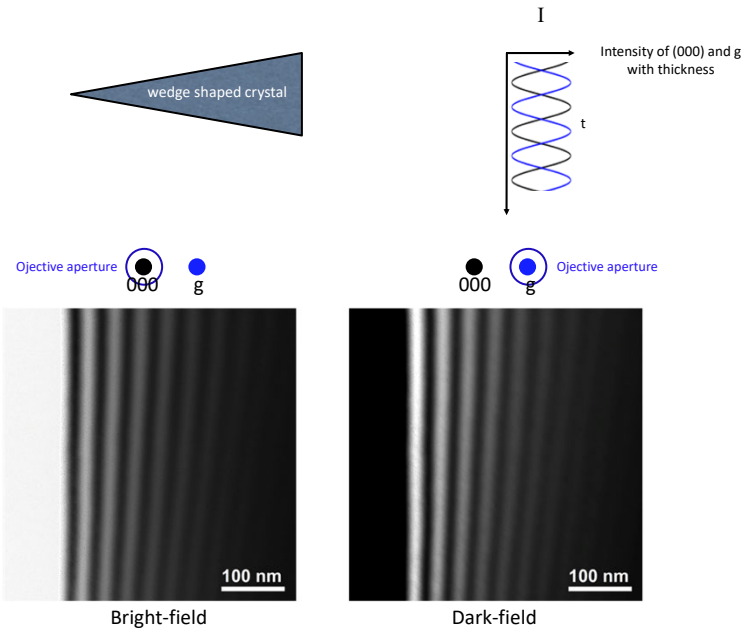
# Dynamical scattering | Thickness fringes



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# Selected-area diffraction phenomena



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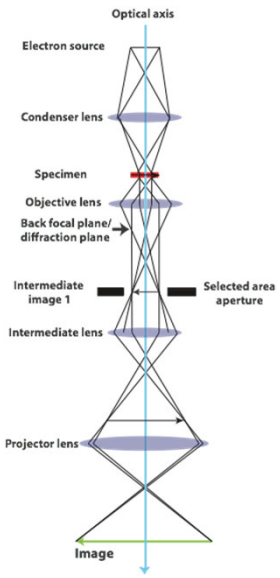
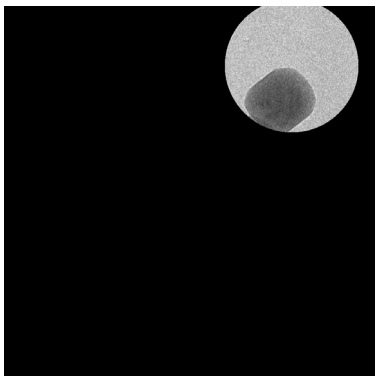
# Image formation



$BaTiO_3$ nanocrystals (Psaltis lab)

Insert selected area aperture to choose region of interest



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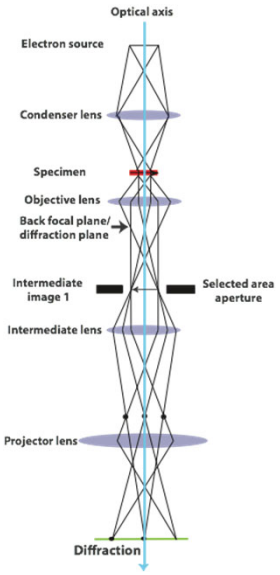
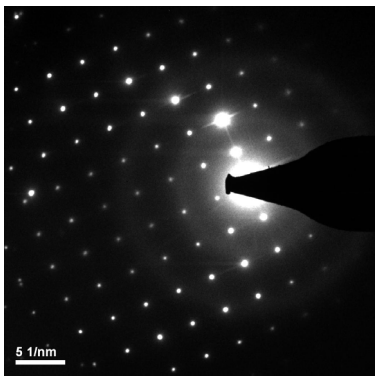
# Take selected-area diffraction pattern




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

Find cubic $BaTiO_3$ aligned on [0 0 1] zone axis



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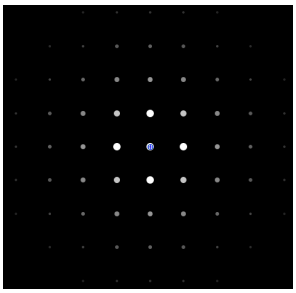
# Symmetry information

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

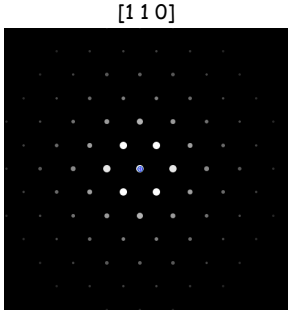
Example: FCC aluminium

[0 0 1]



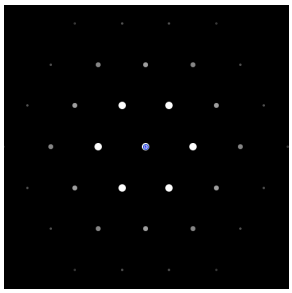
4-fold rotation axis

[1 1 0]



2-fold rotation axis

[1 1 1]



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



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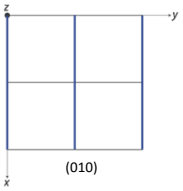
# Weiss zone law

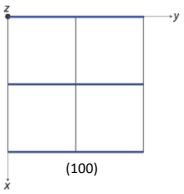
Electron beam parallel to low-index crystal orientation [U V W] = zone axis

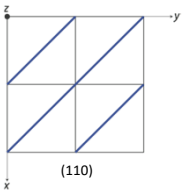
Crystal "viewed down" zone axis is like diffraction grating with planes parallel to e-beam

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

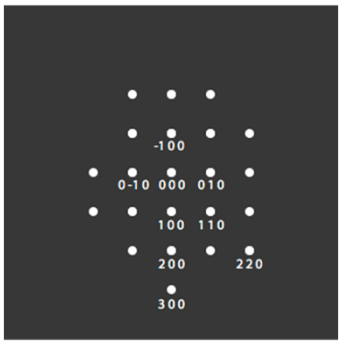



**[001] zone axis**

(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



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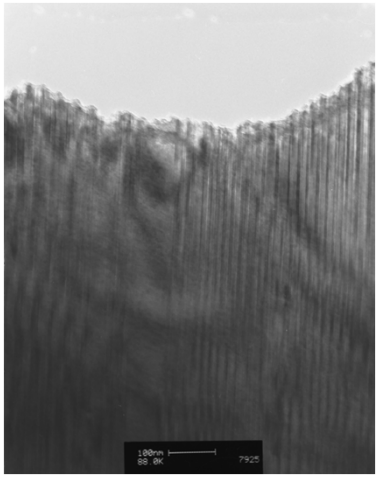
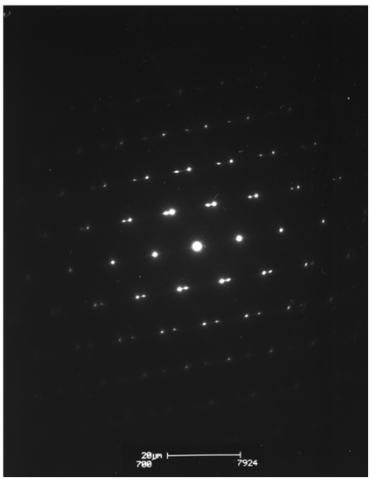
# Twinning in diffraction

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



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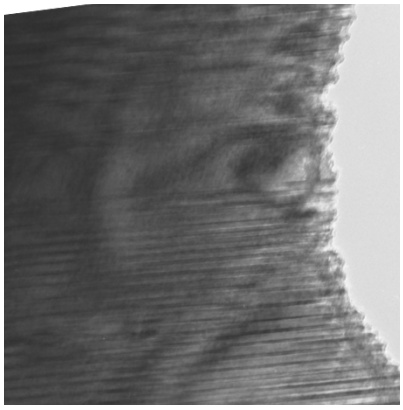
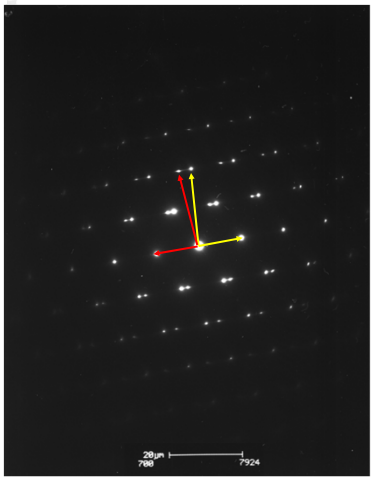
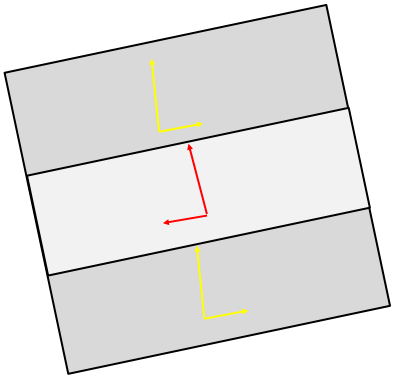
# Twinning in diffraction

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



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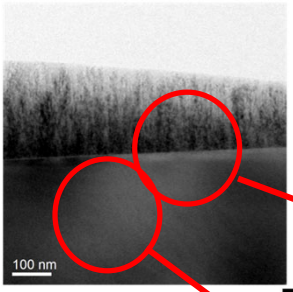
# Epitaxy and orientation relationships

SADP excellent tool for studying orientation relationships across interfaces

Example: Mn-doped ZnO on sapphire

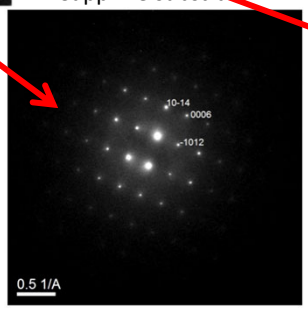


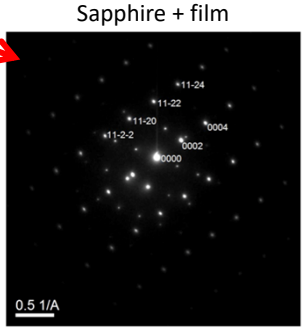

Sapphire substrate

Sapphire + film

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

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



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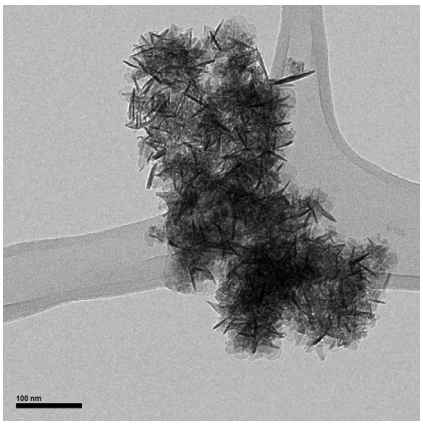
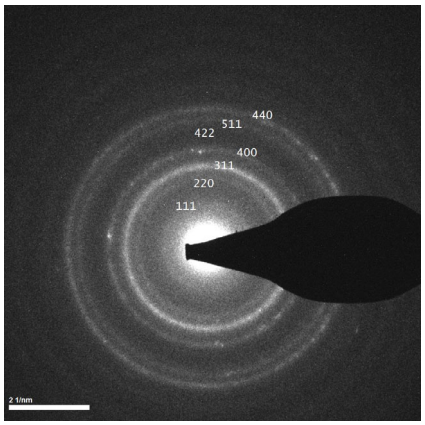
# Ring diffraction patterns

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!



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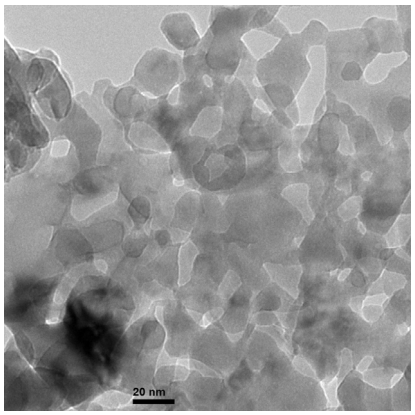
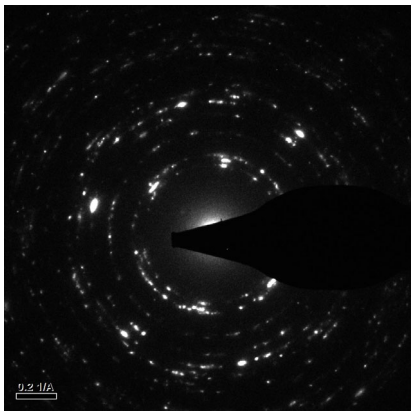
# Ring diffraction patterns

Larger crystals => more "spotty" patterns

Example: ZnO nanocrystals ~20 nm in diameter



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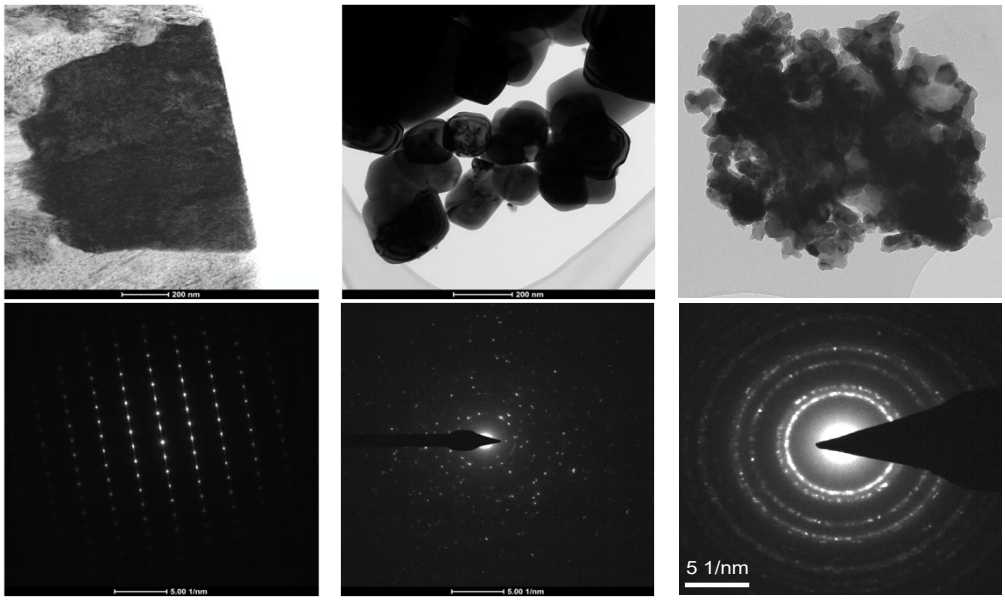
## Applications of electron diffraction






Emad Oveisi



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## Convergent beam electron diffraction



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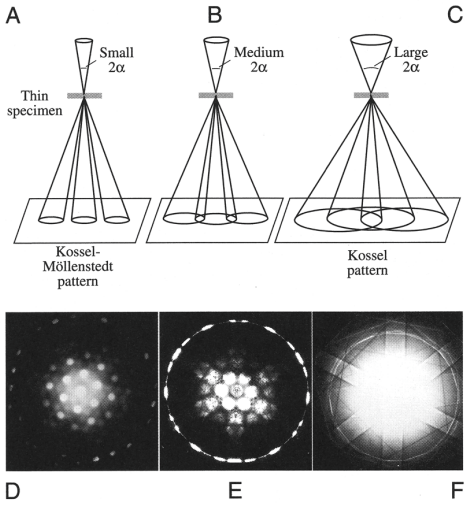
# Convergent beam electron diffraction



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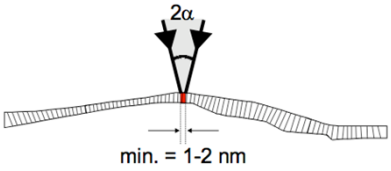
# Convergent beam electron diffraction

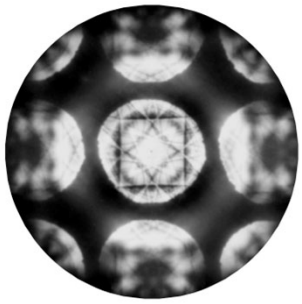
Instead of parallel illumination with selected-area aperture, CBED uses highly converged illumination to select a much smaller specimen region




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:





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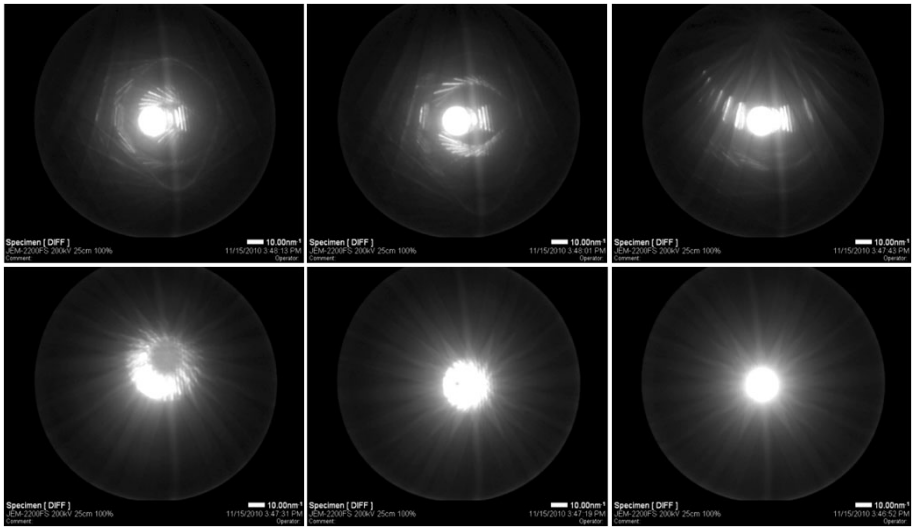
## Convergent beam electron diffraction | To align specimen

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





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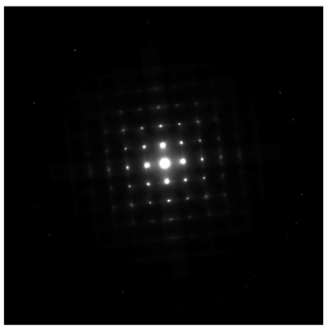
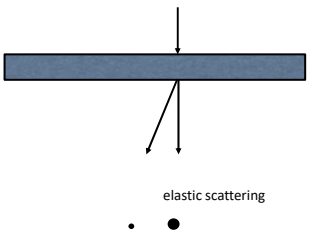
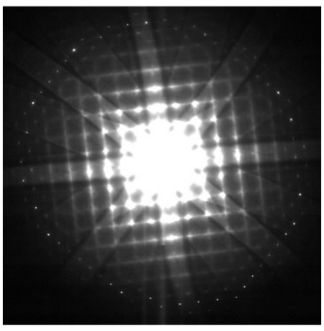
## Kikuchi lines/bands

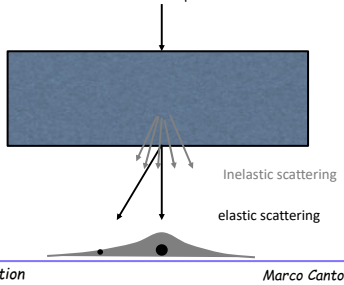


Thin sample



Thicker sample



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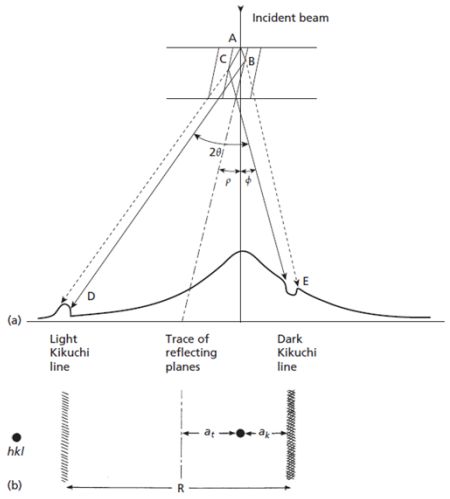
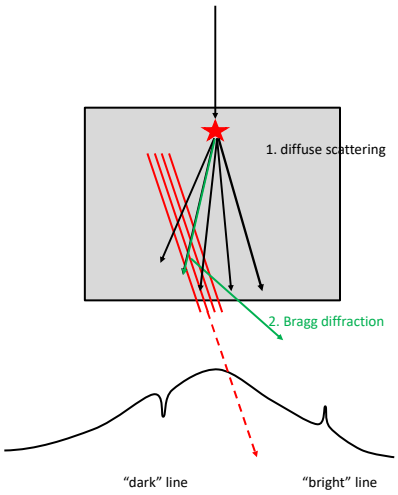
## Diffuse scattering followed by Bragg diffraction



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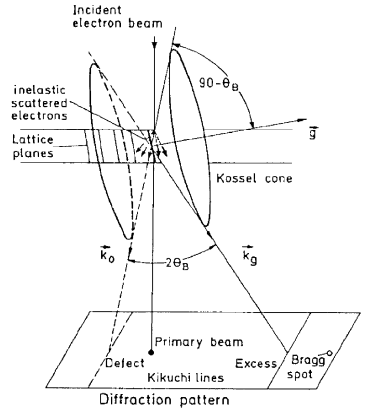
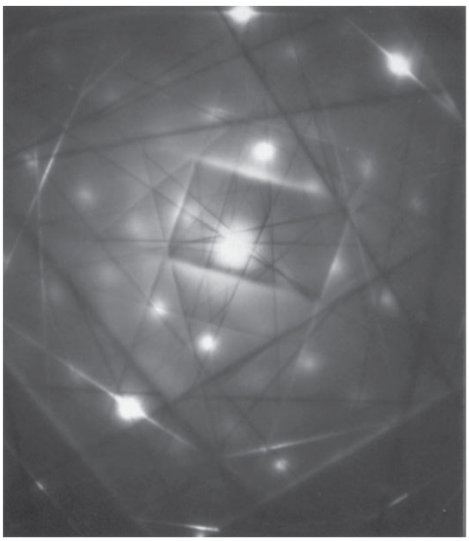
## Kikuchi lines (bands)



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



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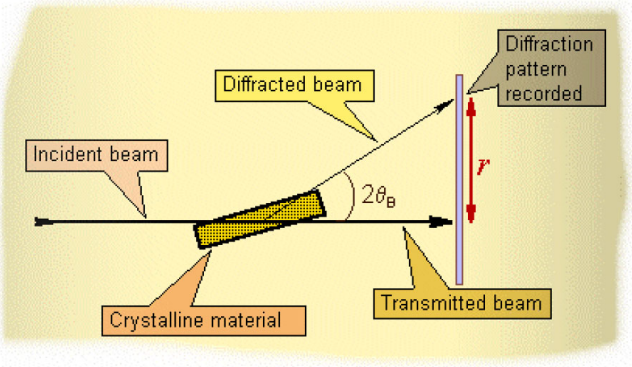
# (powder) X-Rax Diffraction

XRD




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



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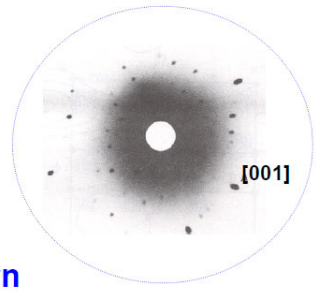
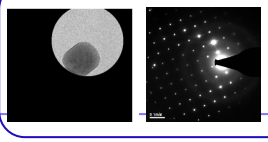
# Laue Method

**Back-reflection Laue**

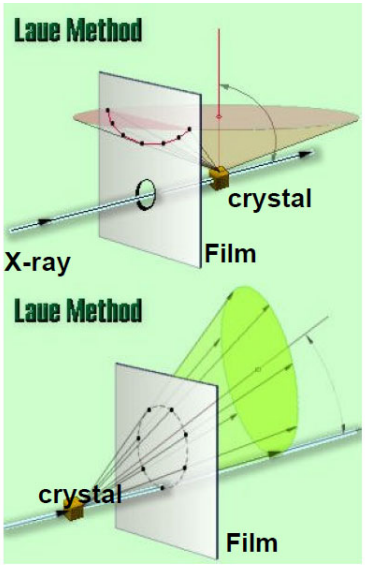



pattern

**Transmission Laue**



In TEM: ~ SAED



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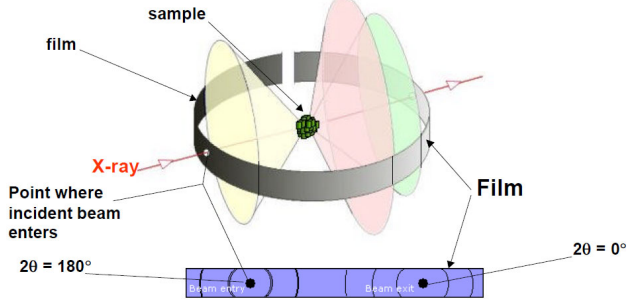
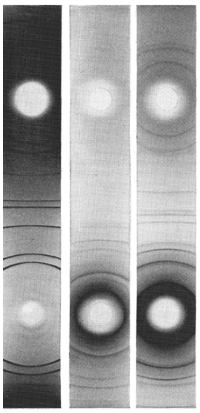
# Debeye-Scherrer

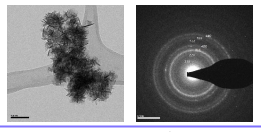
## Detection of Diffracted X-rays
### by Photographic film



**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



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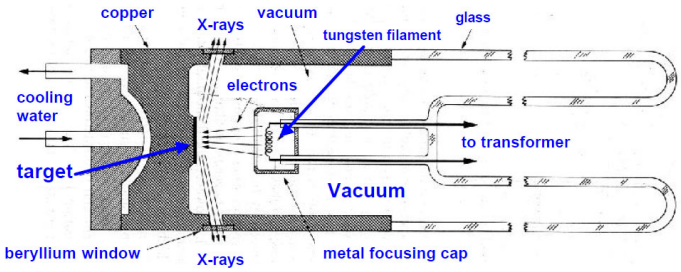
# X-ray tube

## 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.



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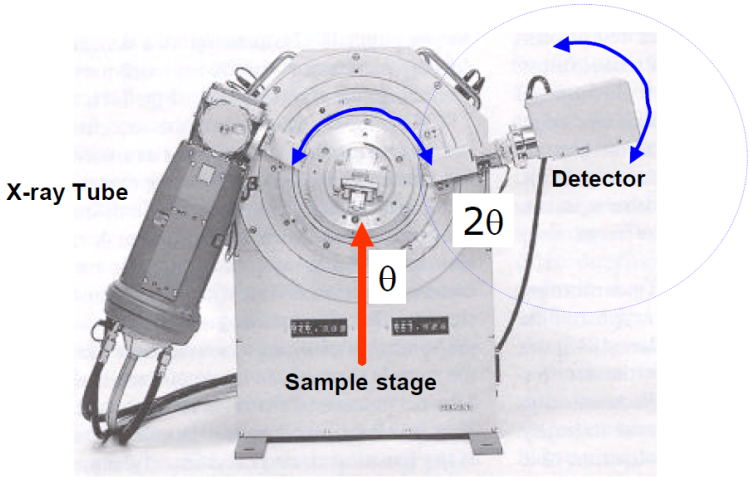
# Diffractometer

## A Modern Automated X-ray Diffractometer




**Cost: $560K to 1.6M**



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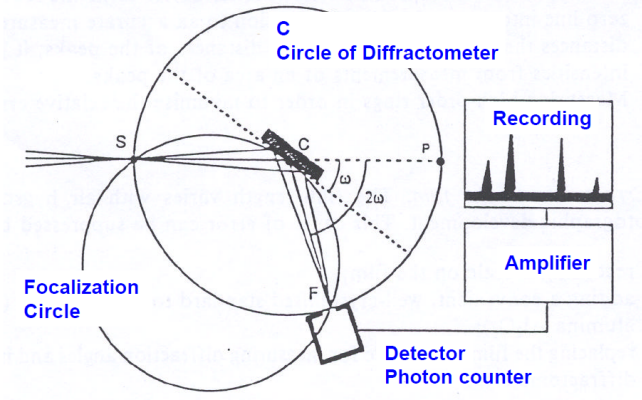
## Detection of *Diffracted* X-rays by a Diffractometer




**Bragg - Brentano Focus Geometry, Cullity**



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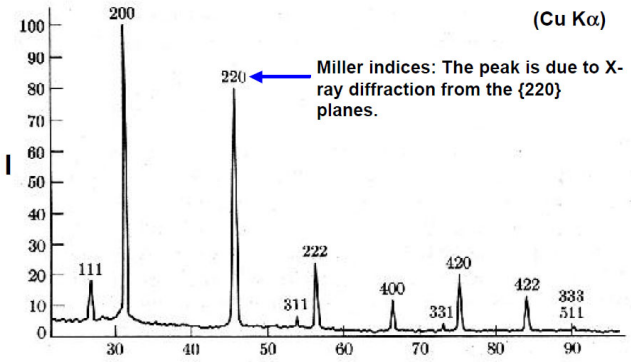
## XRD Pattern of NaCl Powder



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**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).



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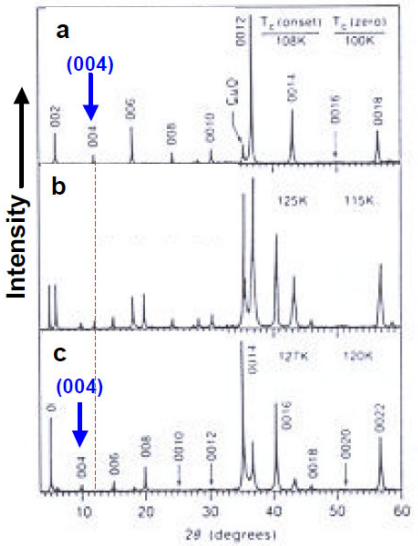


**Diffraction patterns of three Superconducting thin films annealed for different times.**

a. $Tl_2CaBa_2Cu_2O_x$ (2122)
b. $Tl_2CaBa_2Cu_2O_x$ (2122) + $Tl_2Ca_2Ba_2Cu_3O_y$ (2223)
   b = a + c
c. $Tl_2Ca_2Ba_2Cu_3O_y$ (2223)

**CuO was detected by comparison to standards**