EEE 6212
Semiconductor Materials

Lecture 5: Electron diffraction

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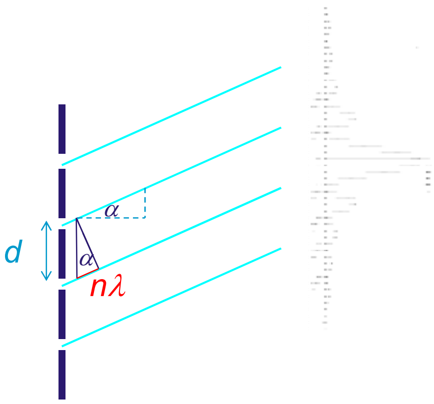
- Bragg’s Law: the crystal as a diffraction grating
- Ewald’s sphere
- diffraction of light, X-rays and electrons
- general evaluation of diffraction patterns
- spot patterns, CBED & Kikuchi patterns

Diffraction by grating

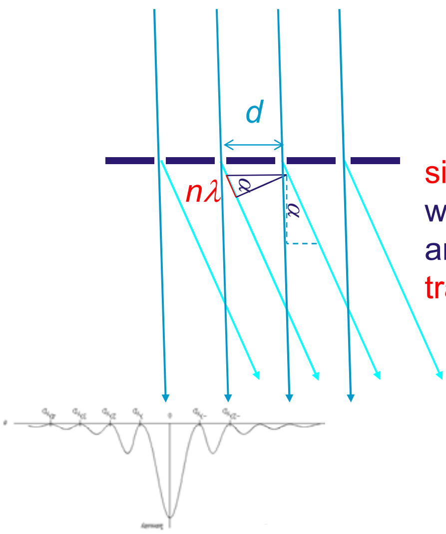


diffraction maxima occur due to constructive interference between neighbouring rays if

$\sin\alpha = n\lambda/d$

where $n \in \mathbb{N}$ and λ=wavelength

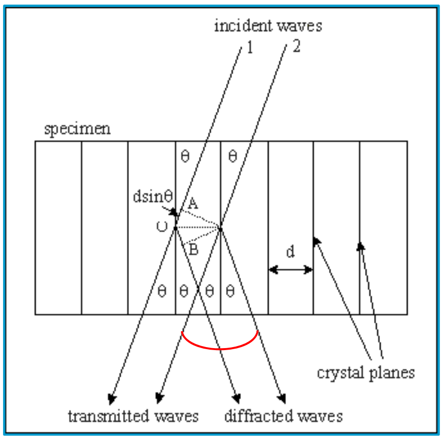
Diffraction by grating



$\sin\alpha = n\lambda/d$

where $n \in \mathbb{N}$ and λ=wavelength and α is the angle between transmitted and diffracted beam

Bragg's Law



optical path difference between diffracted waves 1 and 2 is $2d\sin\theta$.

If $2d\sin\theta = n\lambda$ then the waves are in-phase and interference with each other constructively;

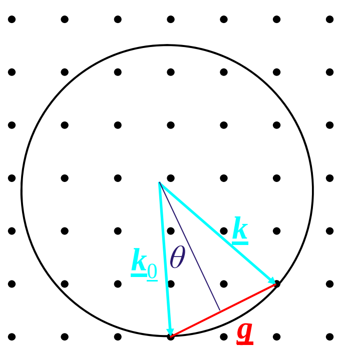
therefore the conditions for diffraction is:

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

BRAGG'S LAW

where 2θ is the angle between the transmitted and the diffracted beam, hence **the crystal acts like a diffraction grating with $\alpha=2\theta$!**

Ewald's sphere construction



construct so-called 'reciprocal lattice' with points of all crystal reflections, then draw circle with radius $k_0=1/\lambda$ and determine the directions for the incoming beam $\boldsymbol{k}_0$ and the scattered beam $\boldsymbol{k}$. Diffraction then occurs only if difference is a reciprocal lattice point, i.e.:

$\underline{\boldsymbol{k}}-\underline{\boldsymbol{k}}_0=\underline{\boldsymbol{g}}$

from sketch follows:

$\sin\theta = n(g/2)/k_0 = n\lambda/(2d)$

same result as Bragg's Law!

Comparison of wavelengths and angles

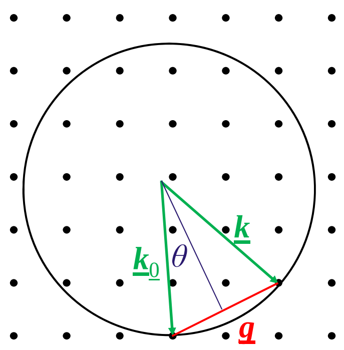
example: consider order n=1 and a lattice spacing of d=0.2nm

radiation	wavelength	wavevector	diffraction angle	
	$\lambda=hc\,[2eVE_0+(eV)^2]^{-1/2}$	$k_0=1/\lambda$	θ =arcsin $n\lambda/(2d)$	
	[nm]	[nm^{-1}]	[mrad]	[°]
light (f=5×10^{14}Hz)	600	0.0017	-	-
X-rays (Cu K_α, hf=8041eV)	0.1542	6.485	3957	22.67
electrons (200keV)	0.00251	399	6.275	0.3595
neutrons (0.025eV*)	0.1809	5.528	4693	26.89

* thermal energy

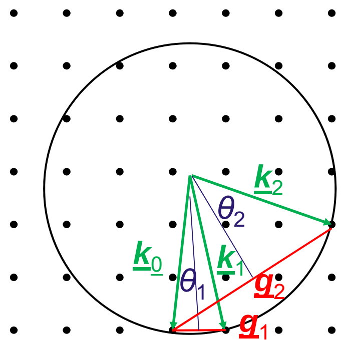
Fast X-rays and slow neutrons have wavelengths similar to atomic spacings and as they are not strongly absorbed, diffraction is strong.

Comparison in reciprocal space



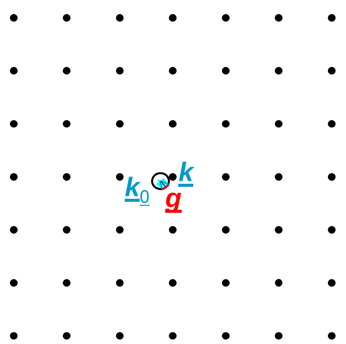
Wavelengths similar to lattice spacings mean that the wavevectors $\underline{\boldsymbol{k}}$ and $\underline{\boldsymbol{k}}_0$ are about as long as the reciprocal lattice vector $\underline{\boldsymbol{q}}$, namely ~$1/d_{hkl}$. As a result, the Ewald's sphere cuts through a number of lattice points. Whether a **few or many reflections** will be excited depends on the direction of incidence.

Comparison in reciprocal space



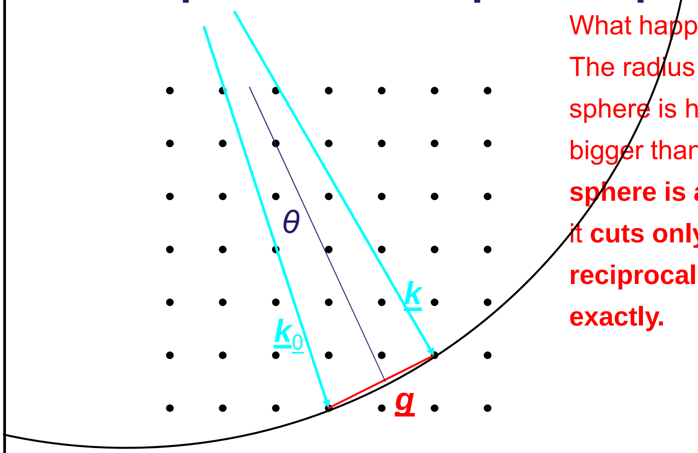
Wavelengths similar to lattice spacings mean that the wavevectors $\underline{k}$ and $\underline{k}_0$ are about as long as the reciprocal lattice vector $\underline{g}$, namely ~$1/d_{hkl}$. As a result, the Ewald's sphere cuts through a number of lattice points. Whether a **few or many reflections** will be excited depends on the direction of incidence, as shown here for simultaneous excitation of $\underline{g}_1$ and $\underline{g}_2$.

Comparison in reciprocal space



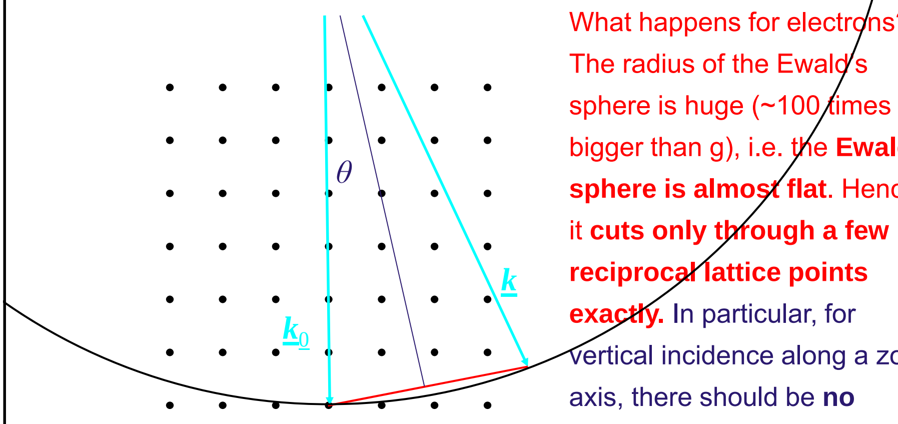
What happens for visible light? The radius of the Ewald's sphere is too small to cut through any lattice points: **no diffraction**.

Comparison in reciprocal space



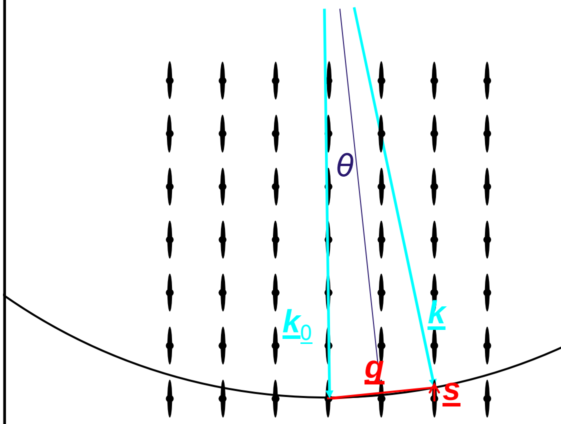
What happens for electrons? The radius of the Ewald's sphere is huge (~100 times bigger than g), i.e. the **Ewald's sphere is almost flat**. Hence, it **cuts only through a few reciprocal lattice points exactly.**

Comparison in reciprocal space



What happens for electrons? The radius of the Ewald's sphere is huge (~100 times bigger than g), i.e. the **Ewald's sphere is almost flat**. Hence, it **cuts only through a few reciprocal lattice points exactly.** In particular, for vertical incidence along a zone axis, there should be **no** diffraction at all!

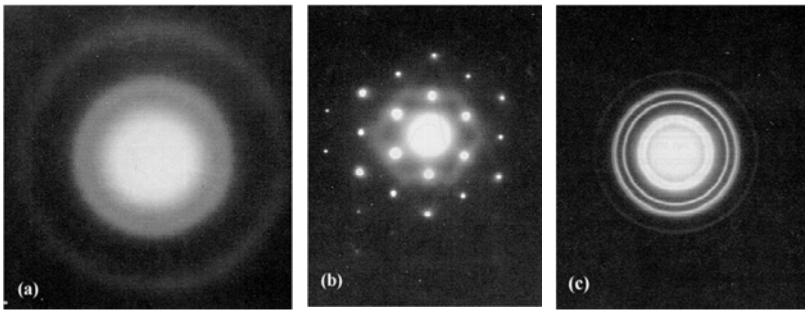
Introduction of reciprocal lattice rods



However, as electrons are strongly absorbed in matter, foils for transmission electron microscopy must be very thin. As a result, the reciprocal lattice is transformed from a set of points to a set of **reciprocal lattice rods**, with extension perpendicular to the foil plane. **In effect, Bragg's law can be approximately fulfilled for a number of reflections within a so-called systematic row.**

$\underline{s}=\underline{k}-\underline{k}_0-\underline{g}$ is called excitation error.

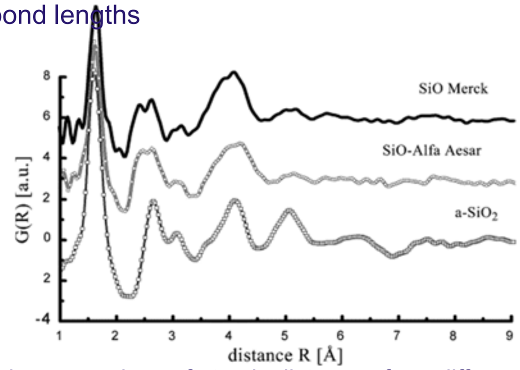
Selected-area electron diffraction (SAED) in the TEM



selected-area electron diffraction patterns obtained from three different materials: (a) amorphous carbon film, (b) Aluminium single crystal and (c) poly-crystalline gold; images courtesy of IM Ross, Univ. Sheffield

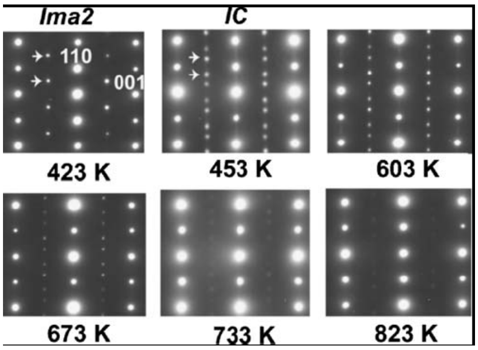
Radial distribution functions of amorphous materials

Principle: record diffraction pattern, get radial profile by line averaging along azimuth angle, subtract background, Fourier transform to real space: peaks = bond lengths



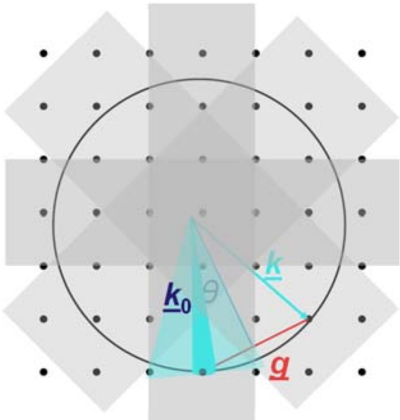
Example: comparison of atomic distances from different oxides
K. Schulmeister and W. Mader, J. non-cryst. Solids 320 (2003) 143-150

Study of phase transitions as function of temperature



Example: Ima2 – IC – P4/mbm phase transition in <110> $Ba_2NdNb_3Ti_2O_{15}$ bronze
I. Levin *et al.*, Appl. Phys. Lett. 89 (2006) 122908

Kikuchi bands



For convergent beam illumination or multiple inelastic scattering **in a thick specimen**: replace arrow for direction of incidence by a **cone** and obtain a **Kikuchi pattern with bright ('excess') and dark ('deficiency') lines** instead of a spot pattern. The pattern's symmetry contains detailed crystallographic information.

Electron back-scatter diffraction (EBSD) in the SEM

A single automated EBSD run can provide rather complete characterisation of the microstructure: it **maps the local sample orientation** (down to **~20nm resolution** if the voltage is reduced to ~5kV) from which one can construct maps of

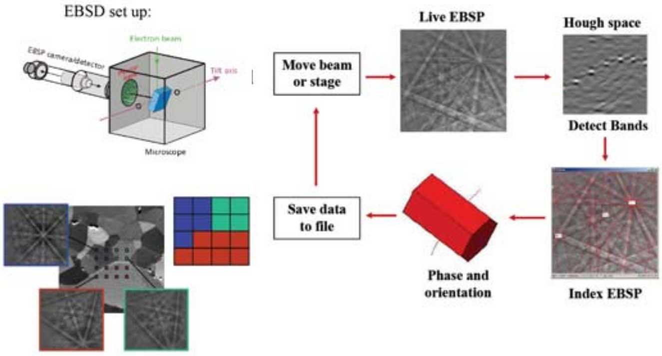
- phase distribution
- grain size distribution

and thus measure also

- grain misorientations
- intra-granular deformation

Prerequisite: a very clean surface (produced e.g. by electro-polishing, etching, ion beam milling)

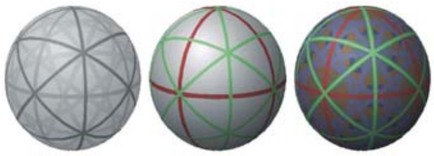
Indexing & Automation



images courtesy of HKL Inc.

Body and Face centred cubic Iron

bcc



Plane	Intensity	No.
{110}	100%	x6
{200}	51%	x6
{112}	32%	x12
{220}	23%	x6
{013}	17%	x12
{222}	13%	x4
{123}	10%	x24

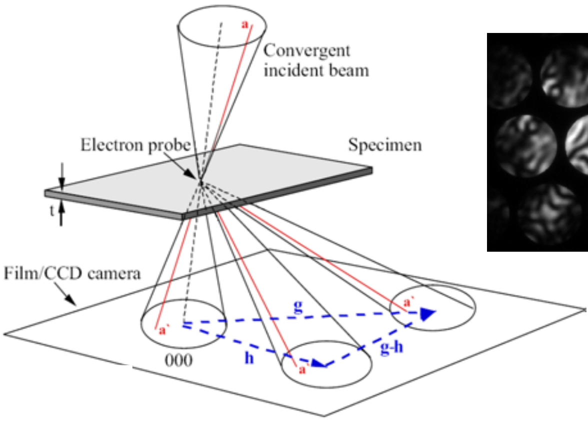
fcc

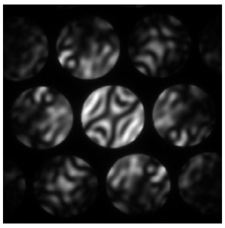
Plane	Intensity	No.
{111}	100%	x4
{200}	77%	x3
{220}	38%	x6
{113}	26%	x12
{222}	23%	x4
{400}	16%	x3
{133}	13%	x12
{240}	12%	x12

bcc h+k+l=2n (i.e. no {111})
fcc h, k, l all odd or all even

images courtesy of HKL Inc.

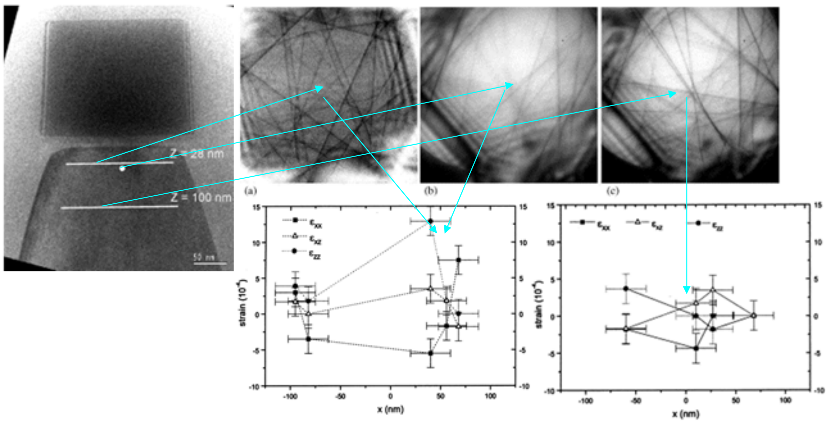
Convergent-beam electron diffraction (CBED) in the TEM





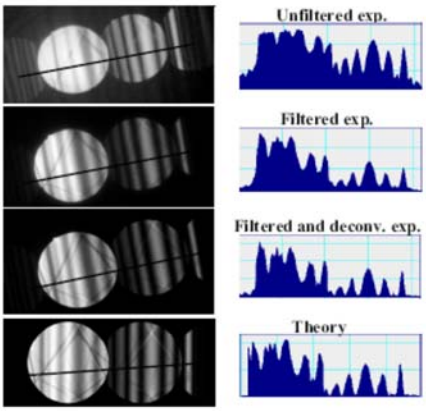
advantages: very small probe possible, each point in the discs refers to one incident beam direction (have multiple directions)
disadvantages: contamination and radiation damage; complicated modelling

local strain measurements



Example: strain measurement in a shallow trench isolation structure with ~10nm spatial resolution;
A. Armigliato *et al*., Mater. Sci. Semicond. Process. 4 (2001) 97-99

electron crystallography



Principle: record diffraction pattern energy filtered, remove detector point spread function, then get by fit to dynamical simulations in iterative manner:

- lattice and atomic positions
- structure factors
- **electron charge density** (by FFT)
- Debye-Waller-factor and absorption

R. Holmestad, Europ. School on Advanced TEM measurement techniques for materials science, St. Aygulf, France, 25 Sept. -7 Oct. 2005

Summary

- Bragg's law of diffraction states $\sin\theta = n\lambda/(2d)$.
- This is identical to diffraction from an optical grating of spacing *d* for total angle of deflection $\alpha=2\theta$.
- Ewald's sphere construction in reciprocal space yields Bragg's law.
- Crystals diffract fast X-rays or slow neutrons strongly.
- Electrons are diffracted only for thin foils (TEM) or from surfaces (SEM, RHEED). Bulk material absorbs electrons.
- Spot diffraction patterns hence tell is about symmetry, (space group), lattice spacings and angles (reciprocal dimensions, hence also strain) and structure factors.