EEE 6212
Semiconductor Materials

Lecture 7: X-ray diffraction

Outline of L7: X-ray Diffraction

- Bragg's Law of diffraction
- Debye-Scherrer geometry (transmission)
- Laue geometry (reflection)
- epitaxy & strained layers
- rocking curves
- coupled scans
- phenomena that can be measured
- Summary

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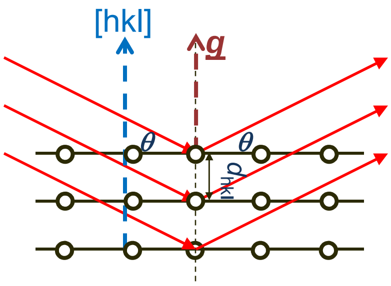
Bragg Diffraction



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

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Bragg Diffraction

- For parallel planes of atoms, with a spacing d_{hkl} between the planes, constructive interference only occurs when Bragg's Law is satisfied.
- The X-ray wavelength λ is fixed.
- Family of planes produces a diffraction peak only at a specific angle θ.
- The spacing between diffracting planes of atoms determines peak positions.
- Additionally, the plane normal [hkl] must be parallel to the diffraction vector **s**.
- plane normal [hkl]: the direction perpendicular to an (hkl) plane of atoms
- diffraction vector ***g***: the vector that bisects the angle between the incident and diffracted beam

X-ray diffraction

- Each object in a periodic array scatters radiation coherently - constructive interference at specific angles produces spots.
- Atoms in a crystal are a periodic array of coherent scatterers.
  - The wavelength of X-rays is similar to distance between atoms.
  - Diffraction from different planes of atoms produces a diffraction pattern, which contains information about the atomic arrangement within the crystal.
- The substrate and film layers can be considered to produce separate plane waves
  - These plane waves from the substrate and each film layer will interfere, producing additional peaks of intensity that will contain microstructural (rather than atomic) information
- X Rays are also reflected, scattered incoherently, absorbed, refracted, and transmitted when they interact with matter.

Comparing types of radiation

Table 1.1 Characteristics of electron, neutron and X-radiations

Characteristic	Electrons	Neutrons	X-rays
Absorption	high	very low	low
Penetration	<1 μm	~cm	~mm
Rocking curve width	degree	<arc second	arc seconds
Strain sensitivity	10^{-3}	10^{-7}	10^{-7}
Spatial resolution	1 nm	30 μm	1 μm
Destructive?	yes	no	no
Cost	high	very high	medium
Convenience	good	poor	good

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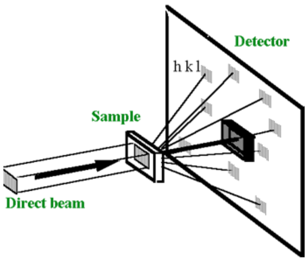
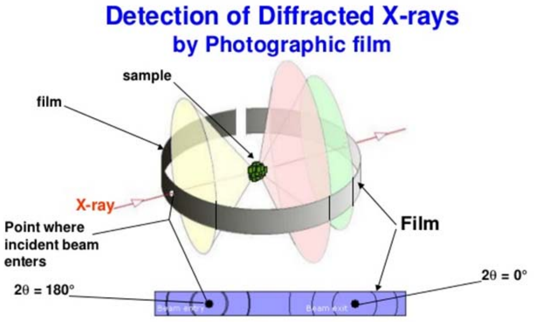
X-ray diffraction in transmission geometry: Debye-Scherrer camera



note: 2D detector that can record whole diffraction pattern



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.

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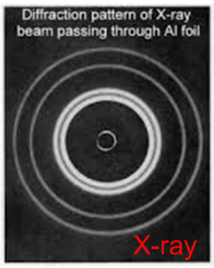
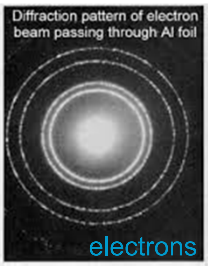
Comparison of different diffraction patterns

application to poly-crystalline aluminium (Al)



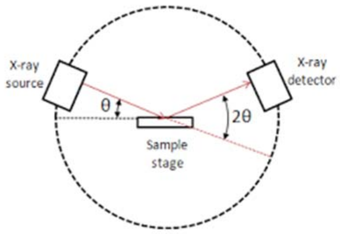


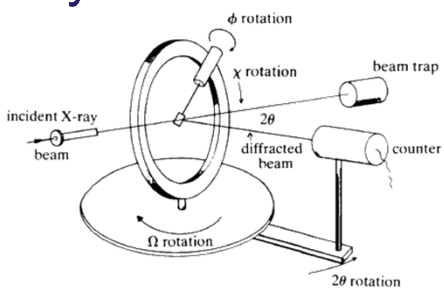
from: ECSE.rpi.edu

application to single crystalline rock salt (NaCl)

from: www.public.asu.edu

X-ray diffraction in reflection: the Laue geometry





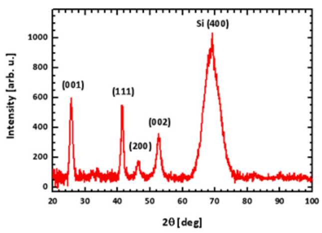
top view

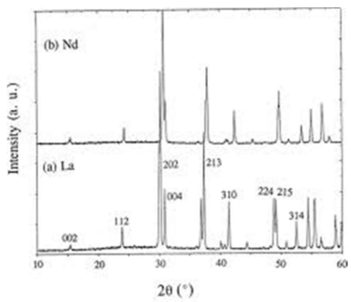
perspective side view

note: point detector that can record only intensity, hence need to scan (θ,2θ)

schematic view of 4-circle diffractometer; including the angles between the incident ray, the detector and the sample.
from: serc.carleton.edu

Comparison of X-ray scans from different powder specimens





wide peaks indicating poor crystal quality
from: www.ifj.edu.pl

narrow peaks indicating good crystal quality in X-ray diffraction patterns of (a) $BaLa_2ZnO_5$ and (b) $BaNd_2ZnO_5$.
From: nvlpubs.nist.gov

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X-ray diffraction in reflection: the Laue geometry (animated)

https://commons.wikimedia.org/wiki/File%3AKappa_goniometer_animation.ogg

Animation shows the five motions possible with a four-circle kappa goniometer. The crystal is mounted for measurements so that it may be held in the X-ray beam and rotated. The rotations about each of the four angles φ, κ, ω and 2θ leave the crystal within the X-ray beam, but change the crystal orientation. The detector (red box) can be slid closer or further away, allowing higher resolution data to be taken (if closer) or better discernment of the Bragg peaks (if further away).

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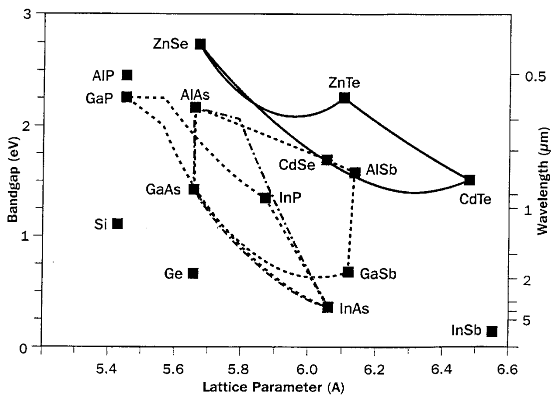
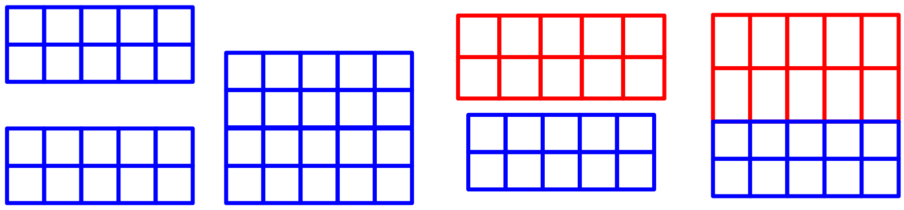
Semiconductor materials



A crystal is grown epitaxially upon a substrate.
First consider a “thick” layer (order of ~> 1μm thick).

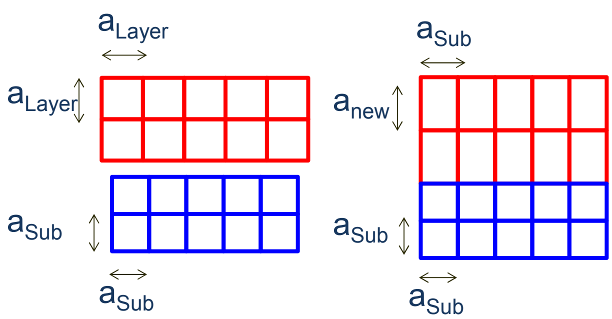
Epitaxy

Definition: Layer-by-layer deposition of atoms upon a substrate



homo-epitaxy hetero-epitaxy

Strained Layers

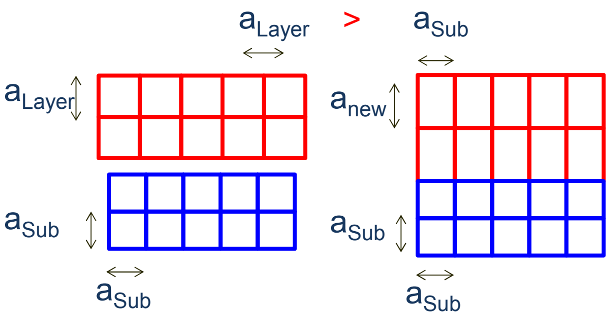


strain parallel to plane of substrate (in-plane)

$\varepsilon_{//} = \Delta a/a$
$= (a_{Layer} - a_{Sub})/a_{Sub}$

assume same space group, atomic arrangements, slightly different lattice parameters and elastic parameters

Strained Layers



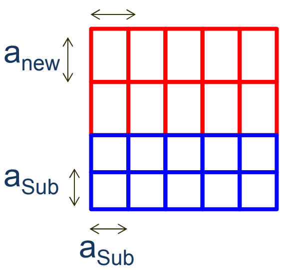
strain parallel to plane of substrate (in-plane)

$\varepsilon_{//} = \Delta a/a$
$= (a_{Layer} - a_{Sub})/a_{Sub}$

> 0, i.e. layer is squeezed in-plane and so is in compression

assume same space group, atomic arrangements, slightly different lattice parameters and elastic parameters

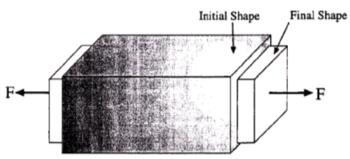
Poisson's ratio



$$\nu = \frac{\text{transverse strain}}{\text{longitudinal strain}} = -\frac{\varepsilon_t}{\varepsilon_a} = -\frac{\frac{\Delta D}{D_o}}{\frac{\Delta L}{L_o}}$$

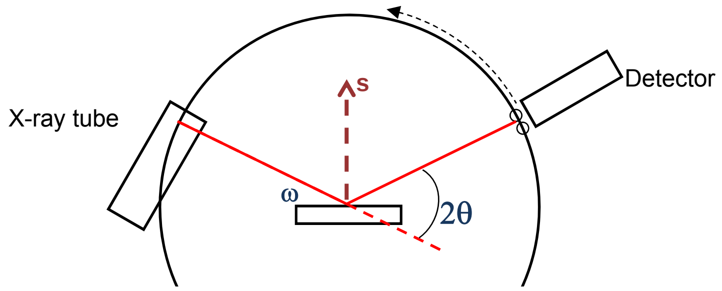
$= -s_{12}/s_{11}$
$\approx$ 0.3 for many semiconductors

$\varepsilon_{//} = -\varepsilon_{perp}/\nu$



Compression/tension in-plane results in deformation out-of plane.
The Poisson ratio ν is determined by stiffness coefficients of material.

Angles in X-Ray diffractometry



The incident angle, ω, is defined between the X-ray source and the sample.
The diffracted angle, 2θ, is defined between the incident beam and detector angle.
Type of scans:

A **Rocking Curve** is a plot of X-ray intensity vs. ω (omega)
A **Detector Scan** plots X-ray intensity vs. 2θ (theta) without changing ω.
A **Coupled Scan** is a plot of scattered X-ray intensity vs 2θ, where ω also changes in a way that is linked to 2θ so that $\omega = ½ (2\theta)$ + offset
A coupled scan is used to measure the Bragg diffraction angle.

Rocking Curve & Coupled Scans

- **Rocking curves are primarily used to study defects** such as dislocation density, mosaic spread, curvature, mis-orientation, and inhomogeneity
  - In lattice matched thin films, rocking curves can also be used to study layer thickness, super-lattice period, strain and composition profile, lattice mismatch, ternary composition, and relaxation.
- **Coupled scans are used to study lattice mismatch, ternary composition, relaxation, thickness and super-lattice period.**
  - Lattice mismatch, composition, and relaxation all affect the positions of each Bragg peak. A single coupled scan can be used to study the film ***if*** only one of these is unknown- otherwise, multiple coupled scans are required for analysis.

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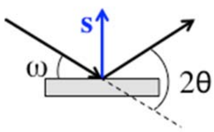
Symmetric and Asymmetric

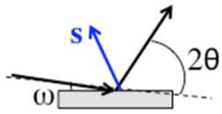
symmetric

$\omega = \theta$



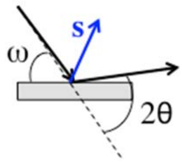
asymmetric
grazing incidence

$\omega = \theta -$ specimen tilt



asymmetric
grazing exit

$\omega = \theta +$ specimen tilt



commonly use symmetric 004 scans in III-V compounds with ω~32°

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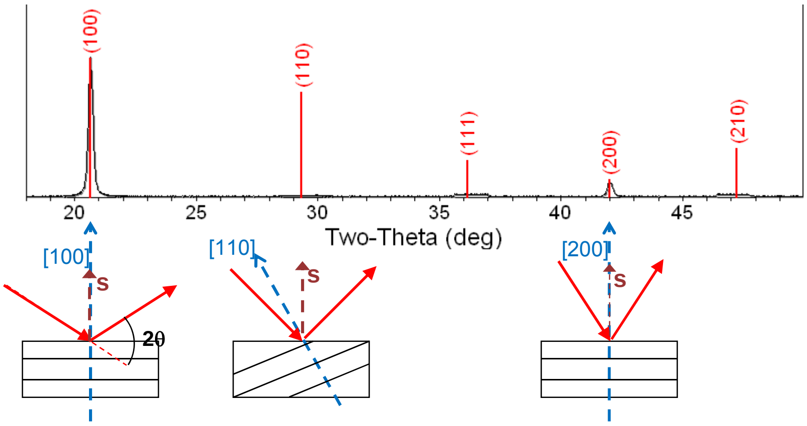
Symmetric, Asymmetric (2)

- Families of planes parallel or nearly parallel to the surface of the sample are investigated in a symmetric scan.
  - The sample is not tilted, so $2\theta = 2\omega$
- Other planes can only be observed by tilting the sample
  - Asymmetric scans are used to collect these other planes by tilting the sample about omega, so $2\theta = 2\omega$ +tilt
  - The sample can be tilted two ways:

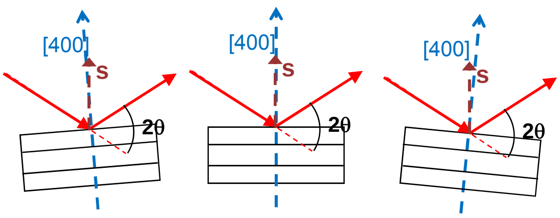
    Grazing incidence (-) tilts the sample towards a lower omega value

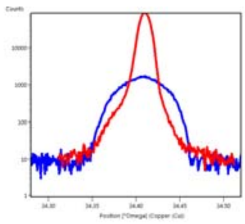
    Grazing exit (+) tilts the sample towards a higher omega value
- Several properties can only be determined by collecting both symmetric and asymmetric scans.

Coupled Scan – Single Crystal



Rocking Curve -Symmetric





A perfect single crystal will produce a very sharp peak, observed only when the crystal is properly tilted so that the crystallographic direction ***g*** is parallel to the surface normal ***s***.
The rocking curve from a perfect crystal will have some finite width due to instrument broadening and the intrinsic width of the crystal material.

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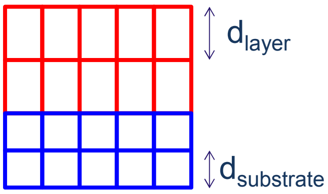
Mismatch

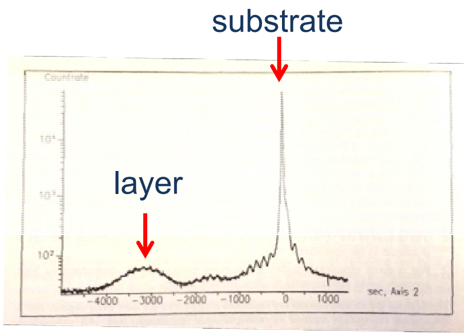


$d_{layer}\sin\theta_{layer}=d_{sub}\sin\theta_{sub}$

.....

$\delta d/d = -\delta\theta \cot\theta_{sub}$

splitting of layer and substrate peak due to different perpendicular lattice specing in rocking curve, independent of sample rotation



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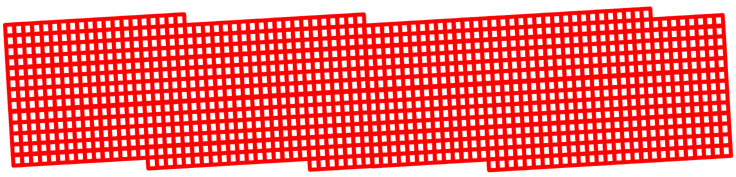
Misorientation

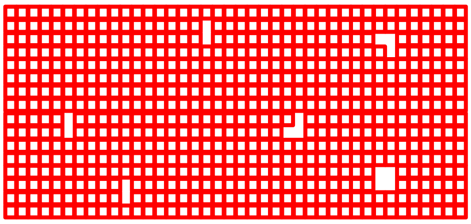


Some substrates are intentionally mis-oriented by a few degrees to provide **surface steps at regular intervals**.

Some homo-epitaxy results in mis-oriented epitaxy upon a vicinal substrate

evidence: variation in rocking curve on rotation of the sample

Defects

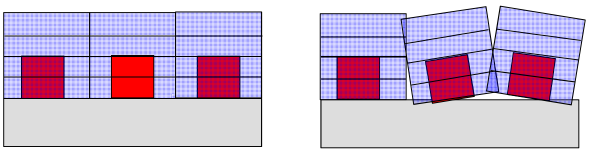


Disruption to crystal perfection **broadens diffraction peaks**.

No shift of peak with beam position.

If the broadening is invariant with beam size – assume defect density is uniform over wafer surface

Mosaicity



Differently oriented grains **also broaden diffraction peaks**.

No shift of peak with beam position, but broadening increases with beam size up to mosaic cell size (assumes smallest beam smaller than mosaic size)

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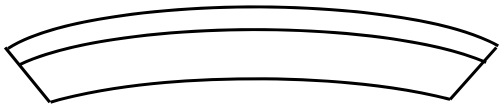
Curvature



Differently oriented regions (with regard to beam) broaden diffraction peaks.

Peak shifts systematically with beam position on curved surface.

Broadening increases linearly with beam size.

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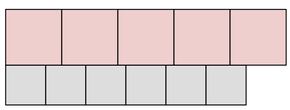
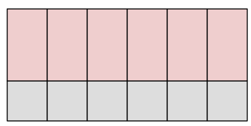
Relaxation



fully relaxed film



fully strained film

For strained layers **partial relaxation can occur**.

Changes splitting of peaks – comparing fully relaxed with fully strained

Compare symmetric and asymmetric reflections to determine degree of relaxation

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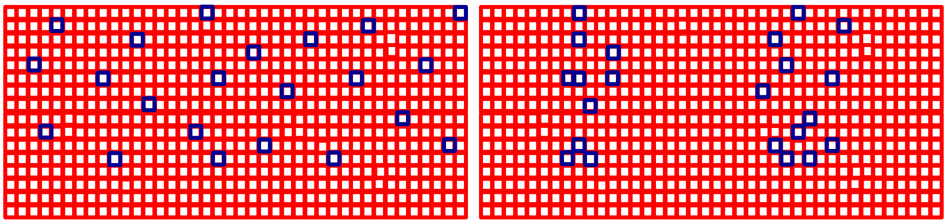
Inhomogeneity



If beam size is smaller than regions of inhomogeneity then position of beam gives different effects – individual characteristics can be mapped.

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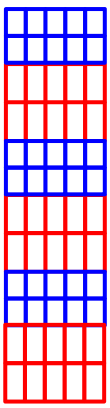
Thin Layers - Superlattices



no longer “thick” layers

now have a structure like an etalon or nano-scale diffraction grating (Newton’s rings, etc…)

perform coupled scans (omega -2theta)

Additional frequency component in diffraction pattern – c.f. Fourier transform, sidebands… see lab course!

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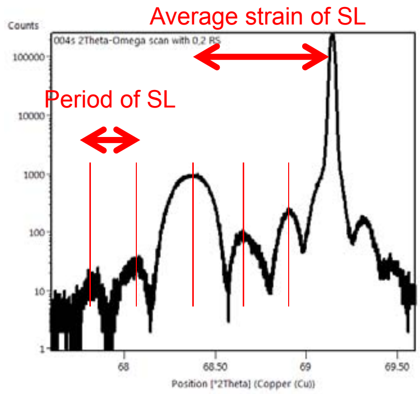
Thin Layers - Superlattices



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Summary

- principle of X-ray diffraction
- Debye-Scherrer vs. Laue geometry
- rocking curves
- coupled scans
- information on lattice parameters and related phenomena