X-ray diffraction for semiconductor materials characterization
Overview

• General XRD properties
  – What can be measured
  – Limitations
• Bragg’s law
• Setup
  – Source
  – Stage
  – Detectors
• Examples of measurements on III-nitrides
• Summary
• Homework
My interest in XRD

• Most important characterization tool for epitaxial crystal growth
• I used extensively for Master’s thesis and research
  – Mostly III-nitride semiconductor characterization
• Topic: ‘Novel substrate structures for III-N compound semiconductors’
  – Si, SOI, SiC and graphene substrates
  – Nitrogen polar materials on SiC seems most promising at this point
Properties

• Reasonably fast
  - Basic scans 10min-1h
  - Mapping scans can be long (day)

• Non-destructive to semiconductors (not all materials)

• Readily available. Philips HR-XRD in 4\textsuperscript{th} floor Micronova. Rigaku XRD in Nanotalo

• Probes collective crystal in a ‘large’ area
  - Large probe size compared to AFM or SEM
  - Measured area 1\text{mm}^2 to 1\text{cm}^2. µ-XRD also possible not very common

• Quantitative results
  - Peak position, peak broadening
  - Can be converted to physical properties e.g. lattice constant
Information from XRD

- Crystal quality
  - Can be linked to dislocation density
- Crystal orientation
- Material composition
  - Possible to identify materials. Typically the alloy composition e.g. $\text{Al}_x\text{Ga}_{1-x}\text{N}$
- Lattice constants
- Stress
- Strain
- Relaxation between layers
- Superlattice parameters
  - Thickness, composition, quality
Limitations

- Combination of crystal quality and probed volume needs to be suitable
  - Crystallites need to be oriented and large enough for sufficient correlation length (amount of periodical lattice planes)
- Smooth sample surface
- Flat sample
- What exactly? It depends…
  - Near perfect III-V epitaxy: thickness 10-20 nm
  - III-N epitaxy: 100 nm
  - ALD or sputtering: 1 µm
- High resolution setup or good detection limit for imperfect materials
  - Setup choice
Diffraction

- X-ray wavelength close to atomic spacing (~0.1nm) in crystalline solids
  - Enables diffraction
- Sample acts as a diffraction grating
- Sample atoms' electrons elastically scatter x-rays
- Constructive and destructive interference of scattered x-rays
- Atom arrangement determines reflection angles
- The element determines reflection intensity

- Bragg’s law:
  \[ n\lambda = 2d\sin\theta \]
Diffraction

- \( n \): multiple of wavelength
  - In practice chosen to be 1. Using specific lattice planes or subset of lattices planes.
- \( \lambda \): wavelength
- \( d \): distance of reflecting lattice planes
- \( \theta \): angle of incident beam
  - In this figure the reflection is symmetrical. Incident and reflected angle is the same. Not always the case.

**Bragg’s law:**

\[ n\lambda = 2dsin\theta \]
XRD setup

- X-ray source produces incident beam
- The beam is diffracted by the sample
- Diffraction data is collected by the detector
- Typically sample stage and detector are moved
  - Other geometries are possible
- Slits reduce divergence
X-ray source

• Electrons from heated filament cathode are accelerated by electric field in a vacuum tube
  – 40-60 kV typical
• Electrons hit a metal anode and produce
  – Characteristic x-rays
  – Deceleration radiation ‘Bremsstrahlung’
• Anode material typically Cu
  – Suitable wavelength, K-alpha-1
  – Easy to cool. Thermal conductivity and stability.
• Water cooled
• Stationary or rotating for higher power source
• What is the problem with diffraction using this spectrum? -->
X-ray source

- What is the problem with diffraction using this spectrum? →
  - Divergent
  - Broad in wavelength
- Slits can be used to reduce spread
- Parabolic mirror nearly collimates the beam
- Filter can be used to cut the spectrum, Ni for Cu source
- Monochromator
  - Typically four diffraction events from Ge 400 crystals. Cu K-alpha1 selected.
  - $\lambda = 0.1540560$ nm
  - Divergence close to 10 arc-sec (10/3600 degree)
- Intensity is reduced – setup choice based on sample
HR-XRD setup

- Mirror, monochromator and analyzer crystal added
- 2theta can be measured in an absolute scale
Detectors

- Scintillators
- Pixel detectors
- Accuracy (in angle) vs. speed
  - Also detection limit
- 0D
  - Most accurate with monochromators
  - Low scan speed
- 1D and 2D detectors can increase scan speed and improve detection limit
  - Especially for reciprocal space mapping
Reciprocal space

- Mathematical tool for diffraction analysis
- Reciprocal space vector $\mathbf{S}$ can be calculated from the incident and diffracted beams
- Reciprocal lattice is calculated using the typical translation:

$$b_1 = \frac{\mathbf{a}_2 \times \mathbf{a}_3}{\mathbf{a}_1 \cdot \mathbf{a}_2 \times \mathbf{a}_3},$$

$$b_2 = \frac{\mathbf{a}_1 \times \mathbf{a}_3}{\mathbf{a}_1 \cdot \mathbf{a}_2 \times \mathbf{a}_3} \quad \text{and}$$

$$b_3 = \frac{\mathbf{a}_1 \times \mathbf{a}_2}{\mathbf{a}_1 \cdot \mathbf{a}_2 \times \mathbf{a}_3}.$$
Reciprocal space

- Vector $\mathbf{S}_{hkl}$ in reciprocal space from origin to coordinates $(h,k,l)$
- $\mathbf{S}_{hkl}$ is perpendicular to the real space lattice plane with Miller indices $(hkl)$
- $\mathbf{S}_{hkl}$ is related to the plane distance in real space, $|\mathbf{S}_{hkl}| = 1/d_{hkl}$
- $\mathbf{S}_{hkl} = \mathbf{k}_0 - \mathbf{k}_s$
Scan types

- $\omega$ and $\omega - 2\theta$ are the most used scans in semiconductor characterization.

- $\omega - 2\theta$ scan: $S$ length changed, direction is constant. Lattice planes with different spacing but with same orientation are probed. Different peaks typically created by different materials.

- $\omega$ scan traces an arc in the reciprocal space around the origin. $S$ length is maintained. Lattice planes with same spacing but different orientation. Peak broadening is related to material quality.

- Different geometries bring a suitable reflection into Bragg condition.
ω – 2θ scan

- Symmetrical scan around (002)
- GaN on Si epitaxy
  - Si substrate and five epitaxial layers
ω – scan

• Scan around GaN (002) reflection
• Peak broadening due to material quality
Reciprocal space map

- 2D slice of reciprocal space
- Useful for multilayers
- $S_x$ coordinate related to in plane lattice constant
- $S_y$ coordinate related to the out of plane lattice constant
- Left: strained layer, right: relaxed

\[
Q_x = \frac{1}{2}(\cos(\omega) - \cos(2\theta - \omega)) \quad \text{and} \quad Q_y = \frac{1}{2}(\sin(\omega) + \sin(2\theta - \omega)).
\]
Reciprocal space map

- Reciprocal maps around (002) and (105) reflections
- Similar GaN on Si sample
  - Five epitaxial layers
- Some relaxation between layers
  - Seen only in (105)
Summary

- Important tool for crystal growth characterization
- Crystal quality
- Crystal orientation
- Material composition
- Lattice constants
- Stress
- Strain
- Relaxation between layers
Homework

• 200 nm of AlN has been grown on SiC
• HR-XRD scans of (002) and (102) reflections
• Peak positions ($\theta_{hkl}$) $\theta_{002} = 17.89^\circ$ and $\theta_{102} = 25.06^\circ$. Cu K-alpha-1 wavelength.

1) Calculate lattice constants $a$ and $c$ for the AlN layer
2) Is the AlN layer under compressive or tensile strain?

• Bragg’s law and lattice planes spacing for hexagonal lattice:

$$\frac{1}{d_{hkl}^2} = \frac{4}{3} \frac{h^2 + k^2 + hk}{a^2} + \frac{l^2}{c^2}.$$