

Introduction to High Resolution X-Ray Diffraction of Epitaxial Thin Films

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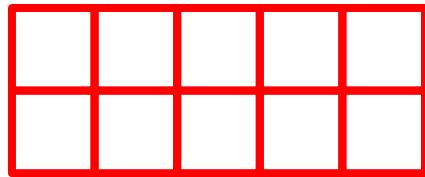
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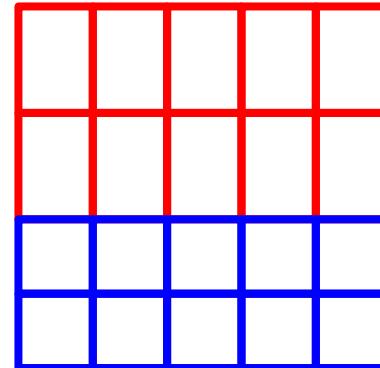
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What is an epitaxial film?

- Traditionally, an epitaxial film is a lattice-matched semiconductor thin film grown on a semiconductor single crystal substrate
 - The lattice of the film is nearly identical to the lattice of the substrate
 - When the film grows, its lattice changes (strains) in order to match the lattice of the substrate
 - The atomic bonding across the substrate and film is “perfectly” matched



The lattice of the **film (red)** is almost the same as the **substrate (blue)**



The lattice of the **epitaxial film (red)** distorts to minimize the strain energy where it bonds to the **substrate (blue)**

A greater variety of functionally epitaxial films are now produced on a regular basis

- A common modern definition for epitaxy is “a single crystal layer that grows with a particular orientation determined by the single crystal substrate”
 - This definition does not require the film and substrate to be lattice matched, but they must still be similar enough to interact and have a defined relationship
- A liberal definition of epitaxy would be “Any film which resembles a single crystal in its lattice structure and properties”
 - This definition lessens the importance of the relationship between the film and substrate. This definition would consider a single crystal layer grown on a glass (amorphous) substrate to still be epitaxial, whereas other definitions would not.
- Definitions depending on the relationship between the film and substrate:
 - Homoepitaxial film: the film and substrate are the same material
 - Heteroepitaxial film: the film and substrate are different materials
 - Some definitions would still require the film and substrate to have similar structures so that they are lattice matched



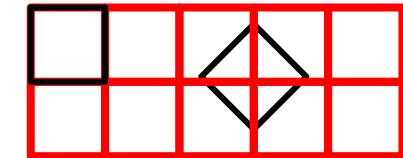
There are functionally two different types of heteroepitaxial thin films

- In traditional heteroepitaxial thin films, the film and the substrate have very similar crystal structures and lattice parameters
 - There is a strong relationship between the film and substrate, in terms of:
 - Bonding across the interface
 - Geometric similarity between their crystal structures and orientation
 - Examples: AlGaInP on GaAs, Si(Ge) on Si,
- Many newer types of heteroepitaxial thin films sometimes include films with different crystal structures than the substrates
 - The film and substrate structures might belong to different crystal systems: for example, a cubic film growing on a hexagonal substrate
 - The geometric relationship between the film and substrate is more complex
 - These films tend to have less lattice strain and higher defect concentration, particularly mosaicity, because the relationship between the film and substrate is weaker.
 - Examples: GaN on Al_2O_3 , BiFeO_3 on LaAlO_3 , $\text{Pb}(\text{Zr}, \text{Ti})\text{O}_3$ on MgO
 - *I have not yet found a good term to describe these types of films*

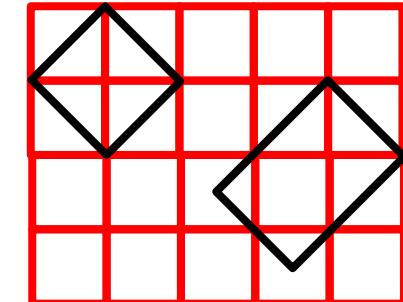


There are different modes of epitaxy depending on the geometric relationship between the film and substrate structures

- **Commensurate:** the primitive film lattice coincides with all symmetry equivalent substrate lattice points
- **Coincident:** the lattice points of the film coincide with some, but not all, of the equivalent substrate lattice points
 - Type I: every lattice point of the film coincides with substrate lattice points. However, not every substrate lattice point has a coincident film lattice point. The film tends to match with lines of substrate lattice points
 - Type II: only some of the film lattice points lie on substrate lattice lines.
- **Incommensurate:** even though the thin film has grown as an effective single crystal without any grain boundaries, there is no direct correlation between the film lattice and the substrate lattice



Different examples of commensurate epitaxy, showing how the film lattice (black) correlates to the substrate lattice (red)



Different examples of coincident epitaxy, showing how the film lattice (black) correlates to the substrate lattice (red)

HRXRD and XRR are both used to study thin films and benefit from the same optics, so we often consider them together

HRXRD can measure:

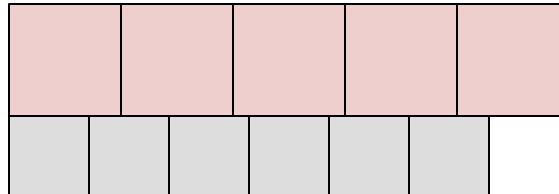
- Structural Information
 - Composition
 - Thickness
 - Superlattice period
- Defects
 - Mismatch
 - Relaxation
 - Misorientation
 - Dislocation Density
 - Mosaic Spread
 - Curvature
 - Inhomogeneity
 - Surface Damage

XRR can measure:

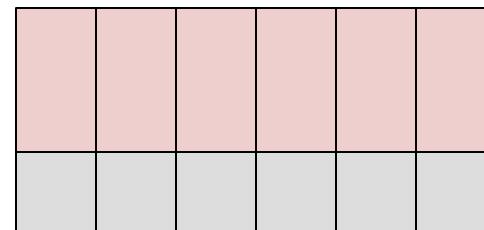
- Thickness
- Surface and Interface Roughness
- Density or composition of the topmost layer



Relaxation (Lattice Strain)



Relaxed Film



Strained Film

- If the film is mismatched to the substrate, then the film might be strained so that the lattice parameters in the lateral direction (ie within the plane of the film) are forced to match the lattice parameters of the substrate
- This distorts the unit cell of the film
 - A formerly cubic unit cell is now tetragonal
- Determine the degree of relaxation
 - No relaxation (fully strained)- the lateral lattice parameters of the film are strained to be identical to the substrate
 - Fully relaxed- the lateral lattice parameters of the film are equal to the bulk values– they have not been distorted at all

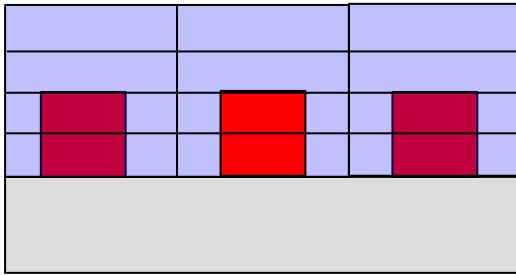
Dislocations

- Interface dislocations may form to relieve lattice strain between a film and substrate with a large amount of mismatch
- Slip dislocations are created by plastic deformation due to thermal or mechanical strain in the layer
 - Slip dislocations create a broadened rocking curve and diffuse scatter
 - In the Hirsch model, Dislocation density $\rho = \frac{\beta^2}{9b^2}$
 - β is the broadening of the rocking curve in radians
 - b is the Burgers vector in cm

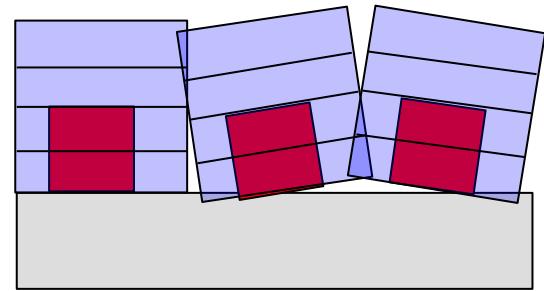


Mosaic Spread

- Mosaicity is created by slight misorientations of different crystals as they nucleate and grow on the substrate. When the crystals join, they form low energy domain boundaries.

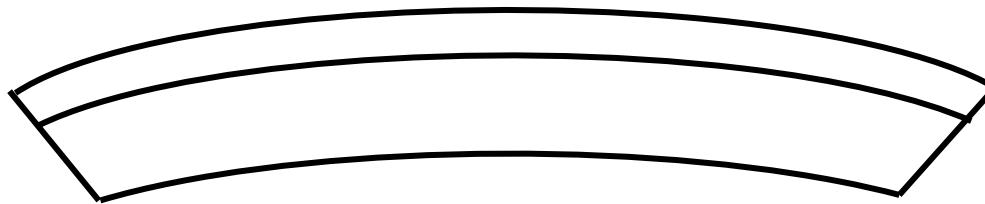


In the ideal case, each nuclei (red) is perfectly oriented. When the crystals grow and meet, there is perfect bounding between the crystallites and therefore there is no grain boundary



If the nuclei (red) are slightly misaligned, then low angle domain boundaries will be formed.

Curvature



- The film and substrate may become slightly curved rather than perfectly flat
 - This may be the result of deposition process, thermal expansion mismatch between the film and substrate, etc

Inhomogeneity or Gradients

- Both compositional and strain gradients can be identified and quantified.



There are several levels between films with perfect epitaxy or ideal polycrystalline randomness

Perfect Epitaxy	Single crystal film in perfect registry with a substrate. There are no defects in the film or the substrate.
Nearly perfect epitaxy	Single crystal film in nearly perfect registry with a substrate. Both film and substrate contain a low concentration of defects. Most defects are dislocations in the film.
Textured epitaxial*	Film consists of mosaic domains in nearly perfect registry with the substrate. All domain boundaries are very low angle/low energy. There is nearly perfect bonding across domain boundaries.
Strongly textured polycrystalline	Film consists of grains with nearly perfect preferential orientation of all principle axes. This orientation is often strongly correlated to the substrate. Misorientation parameter for texture is small.
Textured polycrystalline	Film consists of grains with a preferred orientation for 3 principle axes or only along 1 axis out-of-plane.
Polycrystalline	Film consists of randomly oriented grains.
Amorphous	Film does not have long-range crystalline order.



What techniques can be used to learn what type of information about these films

	Thickness	Composition	Lattice Strain/ Relaxation	Defects	Orientation	Residual Stress	Crystallite Size
Perfect Epitaxy	XRR, HRXRD	HRXRD, RC	Assume 100%	Assume none	HRXRD	--	--
Nearly perfect epitaxy	XRR, HRXRD	HRXRD, RC	HRXRD	RC	HRXRD	--	--
Textured epitaxial*	XRR, HRXRD	HRXRD	HRXRD, IP-GIXD	RC	HRXRD	--	--
Strongly textured polycrystalline	XRR	XRPD, IP-GIXD	IP-GIXD	XRPD, IP-GIXD	IP-GIXD, PF	IP-GIXD	XRPD, IP-GIXD
Textured polycrystalline	XRR	XRPD, GIXD or IP-GIXD	--	XRPD, GIXD OR IP-GIXD	PF	Psi	XRPD, GIXD
Polycrystalline	XRR	XRPD, GIXD	--	XRPD, GIXD	PF	Psi	XRPD, GIXD
Amorphous	XRR	--	--	--	--	--	--

XRR- X-Ray Reflectivity

HRXRD- High Resolution XRD using coupled scan or RSM

RC- Rocking Curve

XRPD- Bragg-Brentano powder diffraction

GIXD- grazing incidence XRD

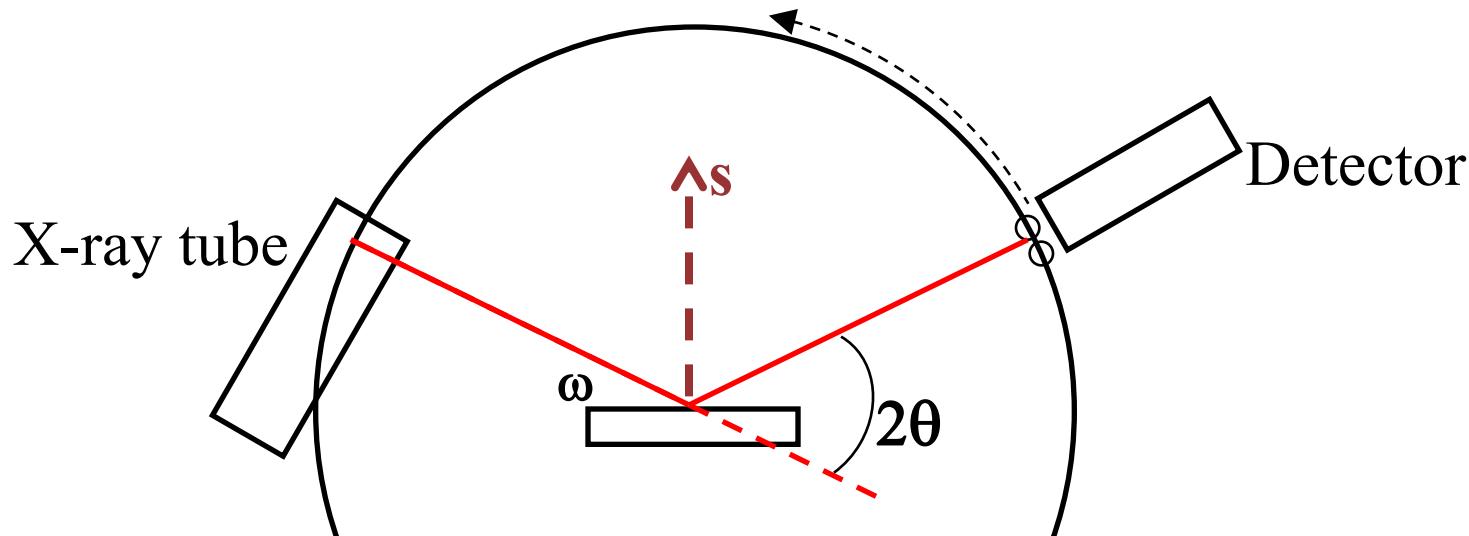
IP-GIXD- in-plane grazing incidence XRD

PF- pole figure

Psi- $\sin^2\psi$ using parallel beam



HRXRD data usually measures scattered X-ray intensity as a function of omega and/or 2theta



- 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. Omega
 - A **Detector Scan** plots X-ray intensity vs. 2Theta without changing Omega.
 - A **Coupled Scan** is a plot of scattered X-ray intensity vs 2Theta, but Omega also changes in a way that is linked to 2Theta so that $\text{Omega} = \frac{1}{2} * 2\text{Theta} + \text{offset}$
 - A coupled scan is used to measure the Bragg diffraction angle

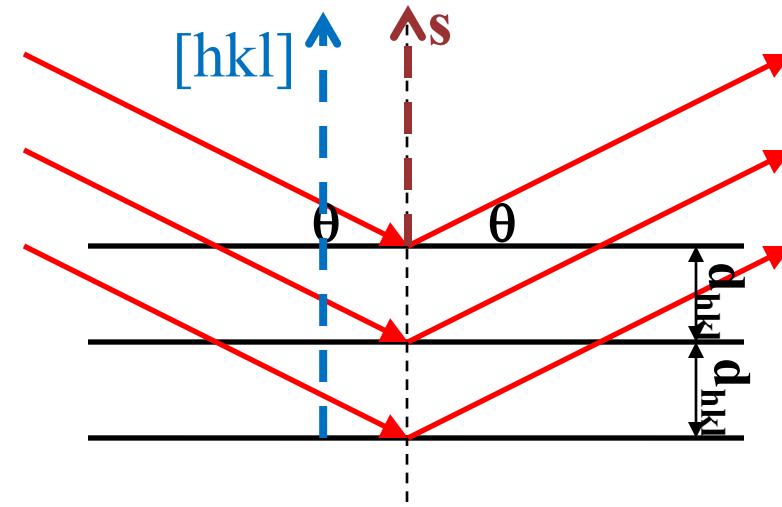
Diffraction patterns are produced by the coherent scattering of light by atoms in crystalline materials.

- Diffraction occurs when each object in a periodic array scatters radiation coherently, producing concerted constructive interference at specific angles.
- Atoms in a crystal are a periodic array of coherent scatterers.
 - The wavelength of X rays are similar to the distance between atoms.
 - Diffraction from different planes of atoms produces a diffraction pattern, which contains information about the atomic arrangement within the crystal
 - The strong peak of intensity that is produced by the coherent scattering of the atomic arrangement in a crystal is called the Bragg diffraction peak.
- The substrate and film layers can be considered to produce separate plan waves
 - These plane waves from the substrate and each film layer will interact, 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.



Bragg's law is a simplistic model to understand what conditions are required for diffraction.

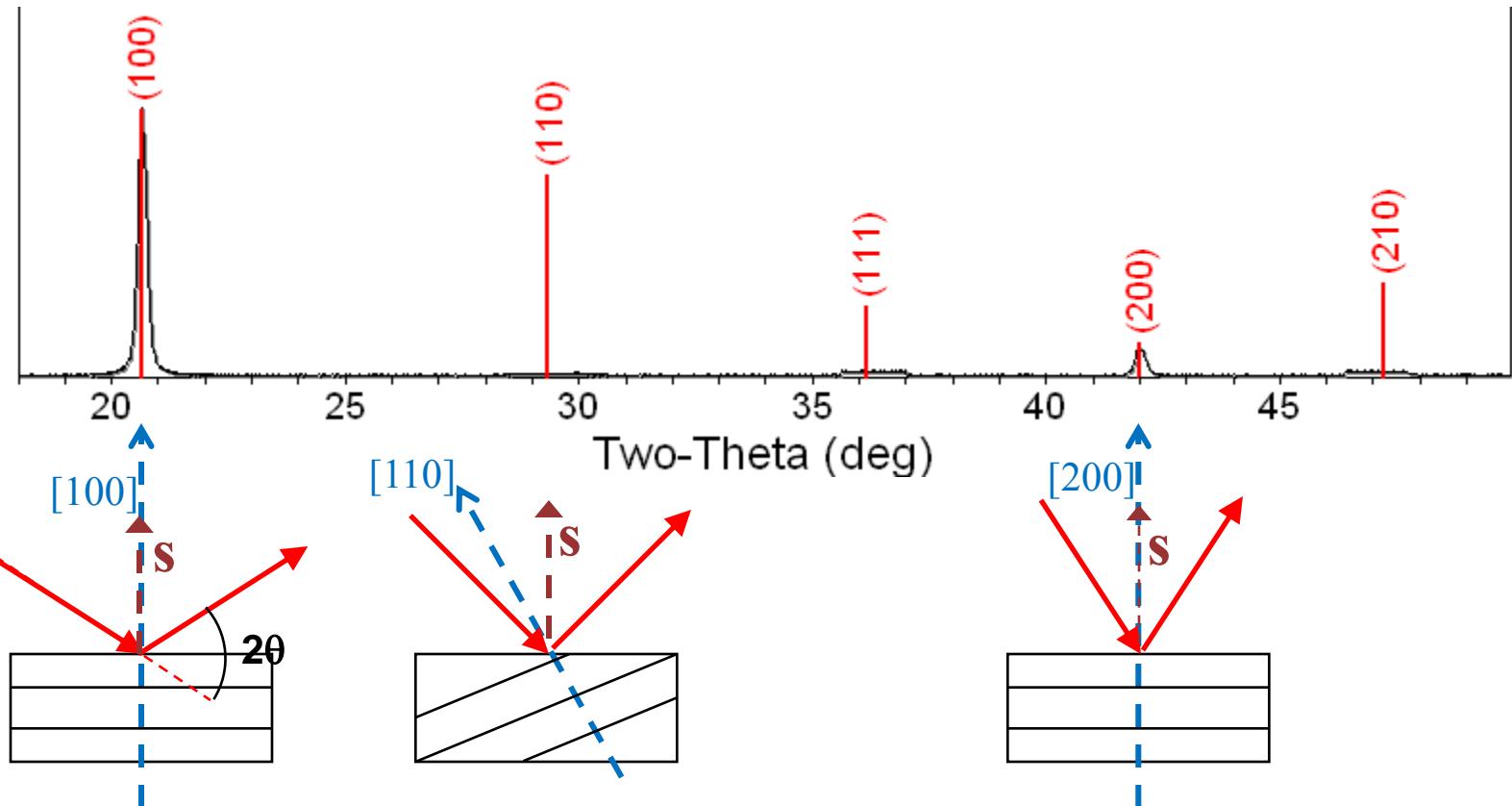
$$\lambda = 2d_{hkl} \sin \Theta_B$$



- For parallel planes of atoms, with a space d_{hkl} between the planes, constructive interference only occurs when Bragg's law is satisfied.
 - In our diffractometers, the X-ray wavelength λ is fixed.
 - Consequently, a family of planes produces a diffraction peak only at a specific angle θ .
 - **The space 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 a plane of atoms
 - Diffraction vector s: the vector that bisects the angle between the incident and diffracted beam



A coupled scan of a single crystal produces only one family of Bragg peaks in the diffraction pattern.



At $20.6^\circ 2\theta$, Bragg's law fulfilled for the (100) planes, producing a diffraction peak.

The (110) planes would diffract at $29.3^\circ 2\theta$; however, they are not properly aligned to produce a diffraction peak (the perpendicular to those planes does not bisect the incident and diffracted beams). Only background is observed.

The (200) planes are parallel to the (100) planes. Therefore, they also diffract for this crystal. Since d_{200} is $\frac{1}{2} d_{100}$, they appear at $42^\circ 2\theta$.

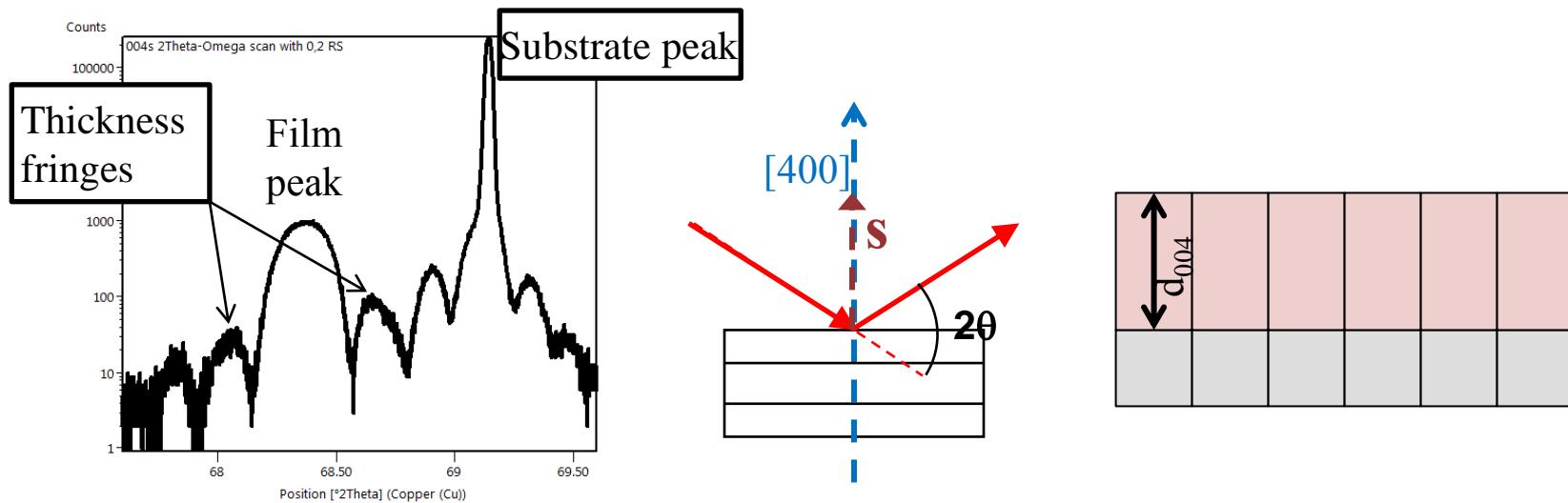


HRXRD can be used to measure several characteristics of epitaxial thin films

- Rocking curves are primarily used to study defects such as dislocation density, mosaic spread, curvature, misorientation, and inhomogeneity
 - In lattice matched thin films, rocking curves can also be used to study layer thickness, superlattice period, strain and composition profile, lattice mismatch, ternary composition, and relaxation
- Coupled scans are used to study lattice mismatch, ternary composition, relaxation, thickness and superlattice period
 - Lattice mismatch, composition, and relaxation all affect the position of the 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
- Reciprocal space maps provide the most complete amount of information and are necessary for the analysis of strained films
- X-Ray Reflectivity can give information on
 - Thickness, interface roughness, and composition or density
 - XRR works with non-epitaxial and even non-crystalline thin films



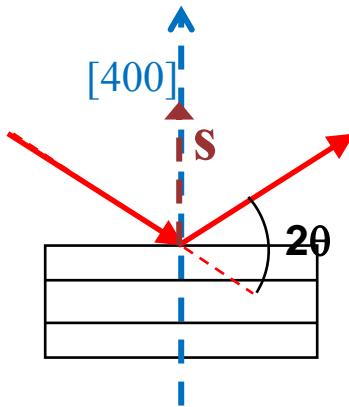
A coupled diffraction scan will give information on the d-spacing and thickness



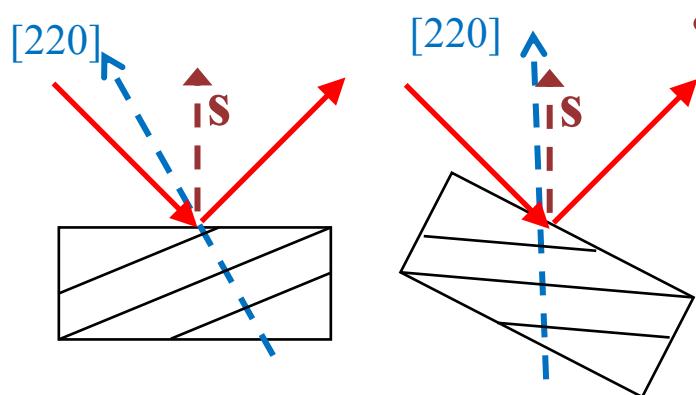
- Coupled scan: omega and 2-theta change in coupled manner so the direction being measured (ie diffraction vector, \mathbf{s}) does not change
- The peak position will give the d-spacing for the Bragg peak
 - This will provide information on anything that changes the lattice parameter of the unit cell, such as *composition* or *strain/relaxation*
 - If both parameters are unknown, then multiple measurements will be required to calculate their values
 - This will only provide measurement of the lattice parameter in one direction
- The width of the film's Bragg peak can be used to quantify the film thickness
 - The thickness fringes can also be used to quantify the film thickness



The sample can be tilted to measure Bragg peaks with different crystallographic directions

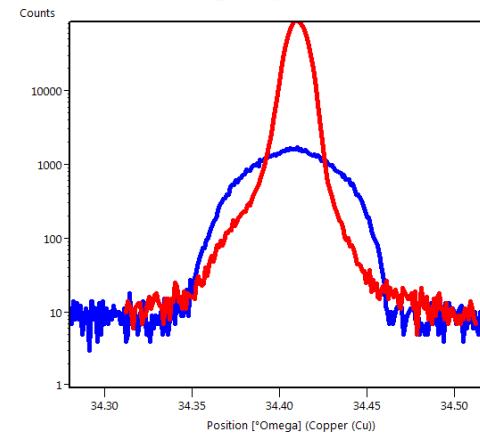
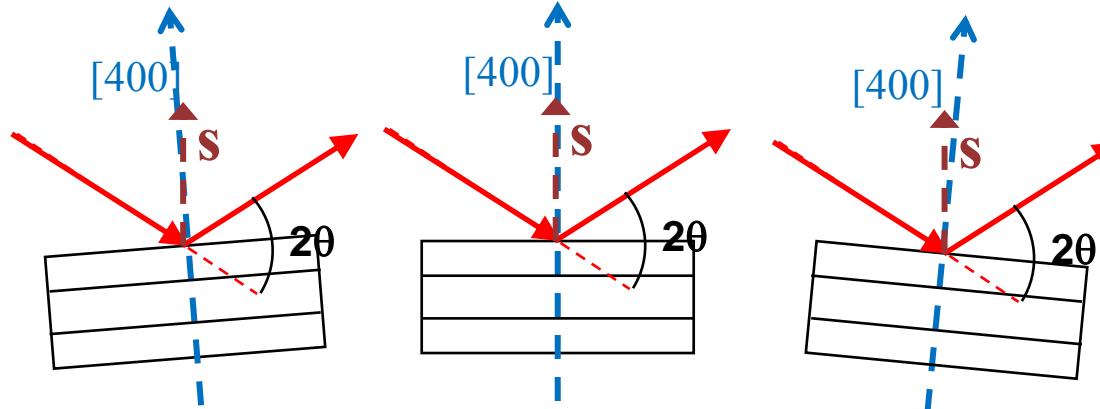


- A symmetric scan requires that $\omega = \frac{1}{2} 2\Theta$, so that the diffraction vector \mathbf{s} will be normal to the surface of the sample

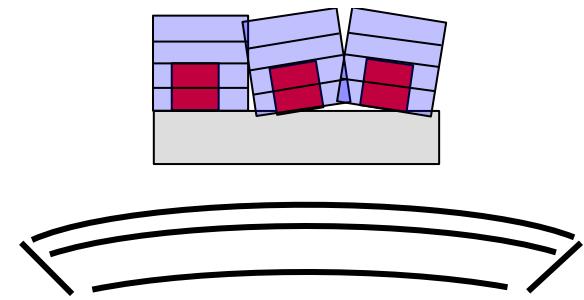


- In order to measure different crystallographic directions in the sample, the sample can be tilted
 - This is called an asymmetric scan

A rocking curve (omega scan) produces observed intensity from planes that are not perfectly parallel



- In a rocking curve, the detector is set at a specific Bragg angle and the sample is tilted.
- A perfect crystal will produce a very sharp peak, observed only when the crystal is properly tilted so that the crystallographic direction is parallel to the diffraction vector s
 - The RC from a perfect crystal will have some width due to instrument broadening and the intrinsic width of the crystal material
- Defects like mosaicity, dislocations, and curvature create disruptions in the perfect parallelism of atomic planes
 - This is observed as broadening of the rocking curve
 - The center of the rocking curve is determined by the d-spacing of the peaks



Double-Axis vs Triple-Axis Diffractometry

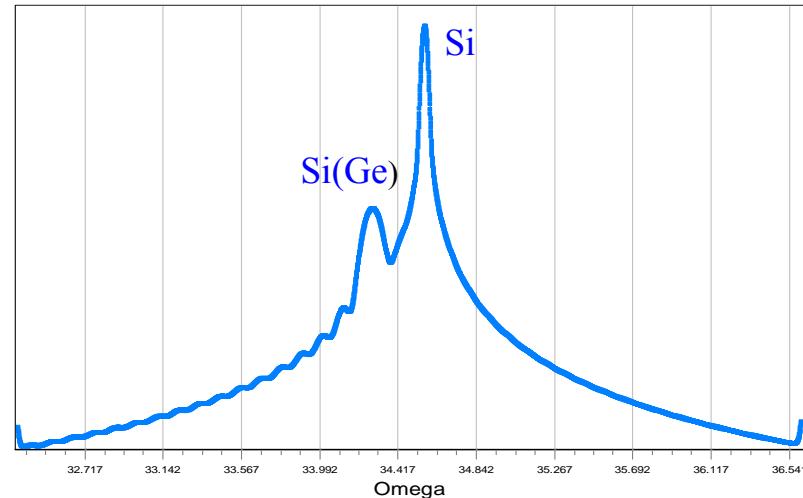
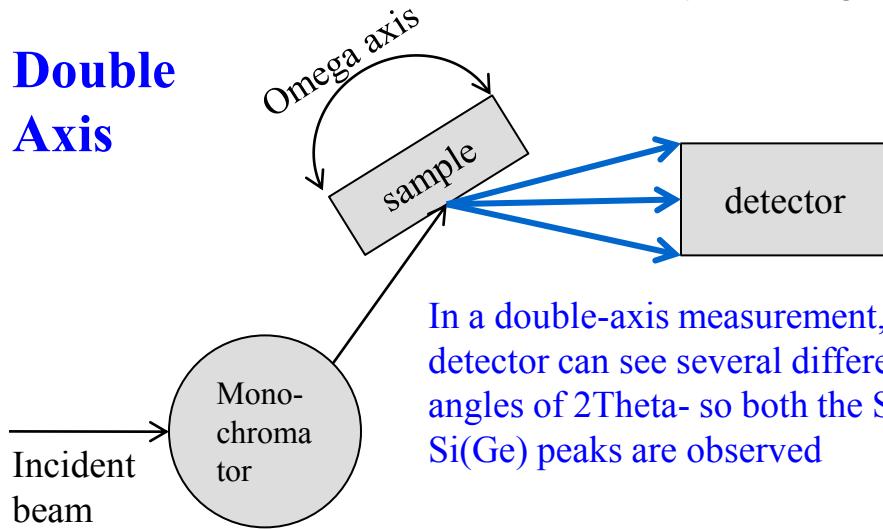
- There are two instrumental configurations for HRXRD
- In both experiments, the monochromator provides a conditioned beam
- In the double-axis experiment, the detector does not discriminate between different diffraction angles 2theta.
 - All Bragg angles are measured simultaneously (over a limited range)
 - The sample is rotated about its omega axis (changing the incident angle) to produce a Rocking Curve (intensity vs omega)
- In the triple-axis experiment, a slit or analyzer crystal determines the angular acceptance of the detector.
 - While a rocking curve (intensity vs omega) can be measured, it is more common to collect data by using a coupled scan
 - As the sample is rotated about omega, the detector is rotated at twice the rate so that $2\text{Theta}=2*\text{Omega}$, producing a coupled omega-2theta scan
 - Reciprocal space maps are collected by collecting coupled scans at different omega offsets, where $2\text{Theta}=2*\text{Omega} - \text{offset}$
 - This separates the effects of strains and tilts on the measurement and permits the measurement of diffuse scatter



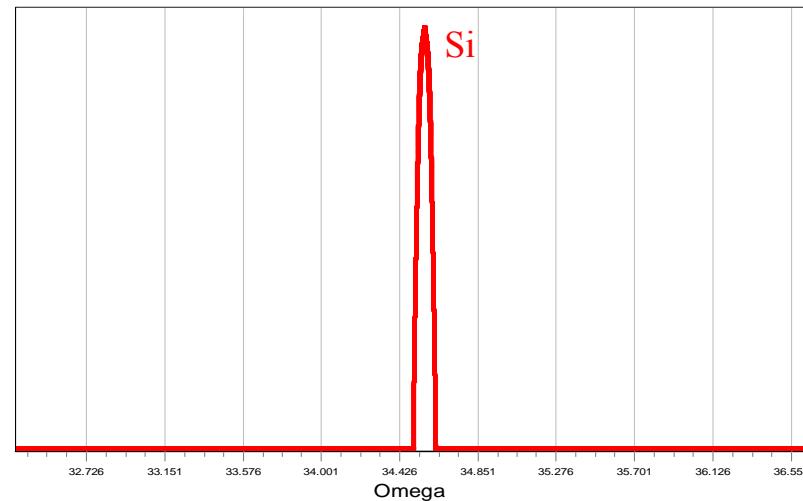
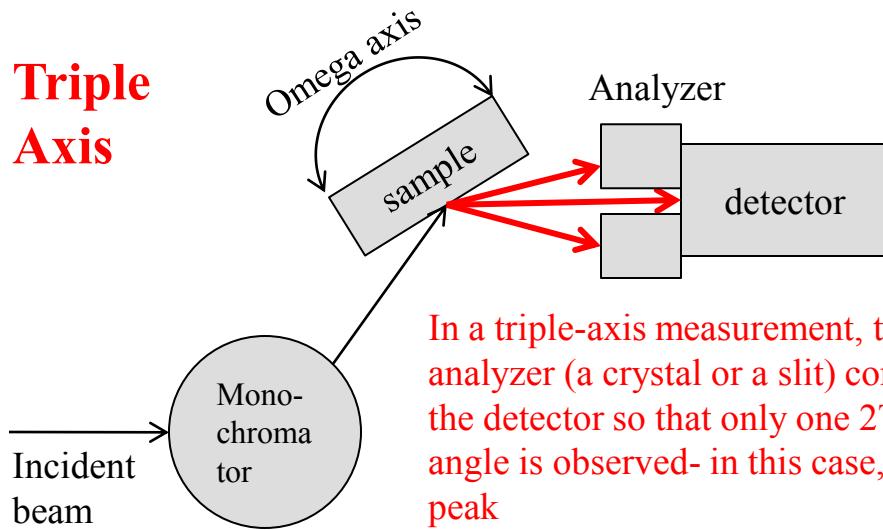
Rocking Curve Schematic: double- vs triple-axis

A Si(Ge) film on Si was scanned by rotating omega while keeping the detector stationary

Double Axis



Triple Axis



Rocking Curve with Double-Axis

- Traditional double-axis diffractometry uses a single reference crystal to condition the X-ray beam.
 - This crystal is the same material as the sample
 - The rocking curve is a correlation of the rocking curves of the two crystals
 - very sensitive to strains and strain gradients in the specimen
- The reference crystal configuration is not practical for a research environment
 - Instead, use a 2x or 4x monochromator to provide a more highly conditioned beam to the sample
 - This allows the rocking curve to be collected by a wide variety of materials
- Double-axis rocking curves are easily simulated using fundamental X-ray scattering theory



Double-axis rocking curves work best for lattice matched thin films

- A range of Bragg angles are collected simultaneously without angular discrimination
 - The detector can only cover a limited angular range with suffering defocusing effects
 - The Bragg angle of the substrate and film peaks should be about the same
- The rocking curve peak position is determined by the Bragg angle and the tilt of the planes
 - Diffraction is observed when $\Omega = \frac{1}{2} * 2\Theta + \text{Tilt}$
 - Differences in the Bragg angle (differences in the d-spacing of the crystallographic planes) are resolved by differences in the rocking curve peak position
 - Differences in tilt of the crystallographic planes are also resolved by differences in the rocking curve peak position
 - Tilts and dilations cannot be distinguished using rocking curves
- The rocking curve width and shape is a product of the material and defects
 - A perfect crystal has an intrinsic width (FWHM) for that material
 - Different planes of a crystal also have different intrinsic peak widths
 - Defects cause the rocking curve to broaden beyond the intrinsic width for the Bragg peak
 - Multiple defects are separated by measuring multiple rocking curves, indentifying systematic trends:
 - Between symmetric and asymmetric scans
 - Rotating the sample
 - Changing the beam size
 - Changing beam position



Triple-Axis coupled omega-2Theta scans

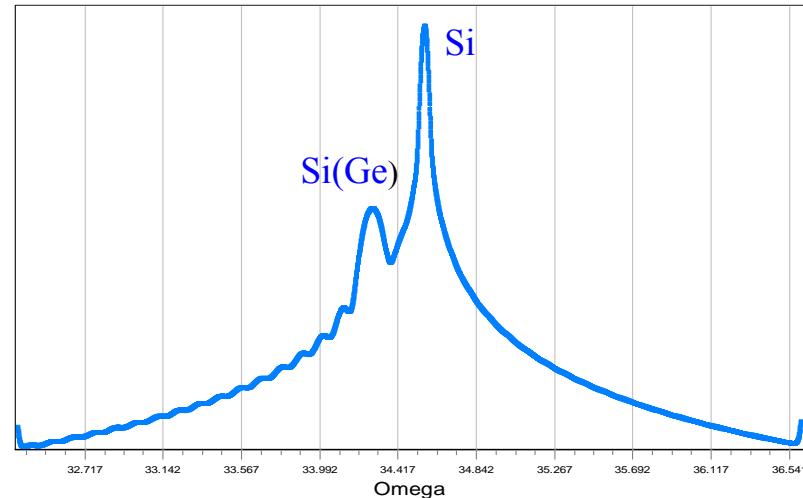
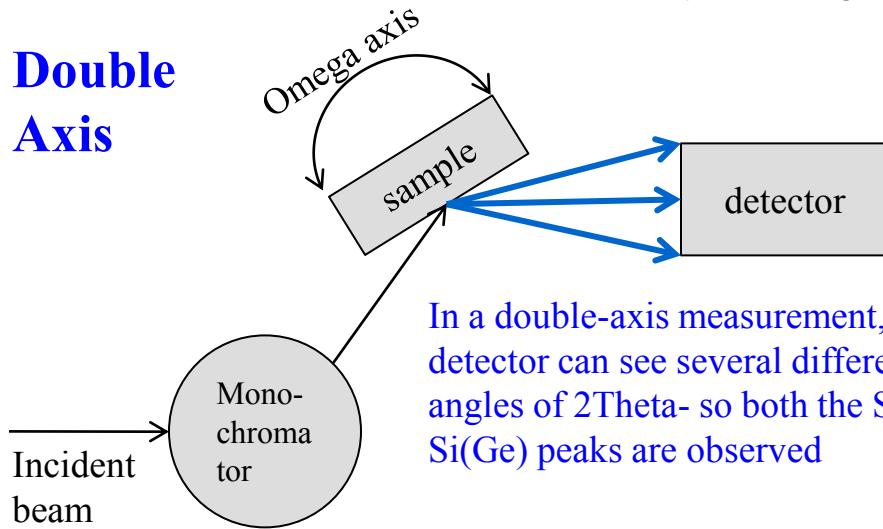
- The triple axis diffractometer observes a narrowly defined region of 2Theta angle
 - Use a slit with an opening between 3deg to 0.1deg
 - Use an analyzer crystal with an effective opening of ??
- A rocking curve collected on a triple-axis diffractometer will observe data for one specific diffraction peak, rather than all diffraction peaks within a certain range
 - You can measure tilt independent of strain (dilation) and get defect information for each individual layer
 - Tilt and strain could not be independently resolved using a single double-axis rocking curve
- A single coupled scan is resolving differences in the d-spacing values of the crystallographic planes
 - d-spacing responds to mismatch, composition, relaxation
 - Can resolve these contributes whereas rocking curve cannot
 - Triple-axis provides much better resolution of multilayers with modest amount of defects (threading dislocations, etc) compared to double-axis
- A common strategy is to collect an omega-2theta scan, identify peak positions, then collect the rocking curve for each diffraction peak



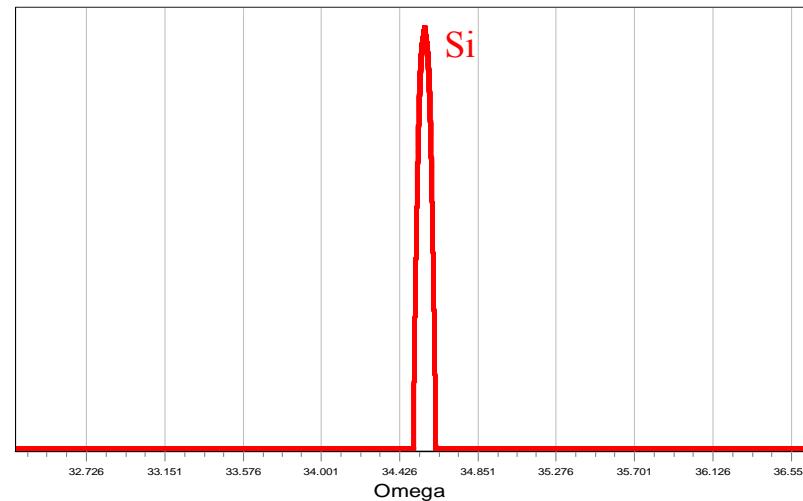
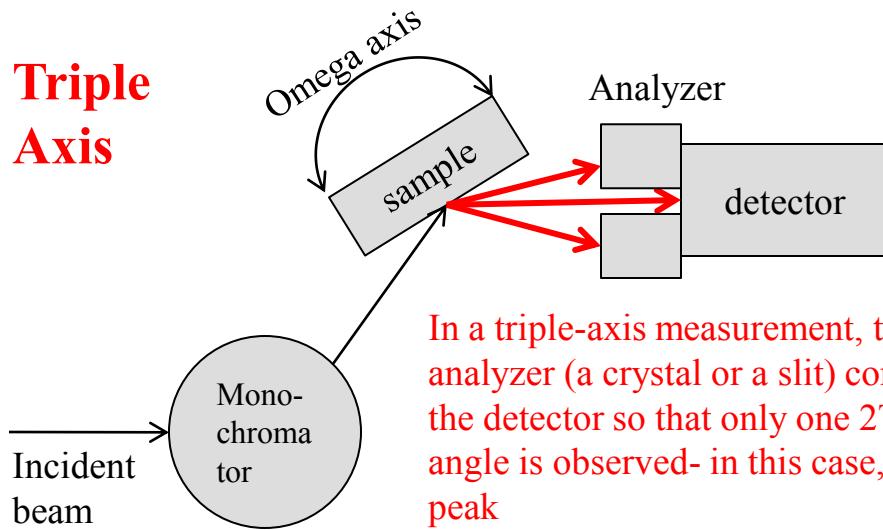
Rocking Curve Schematic: double- vs triple-axis

A Si(Ge) film on Si was scanned by rotating omega while keeping the detector stationary

Double Axis



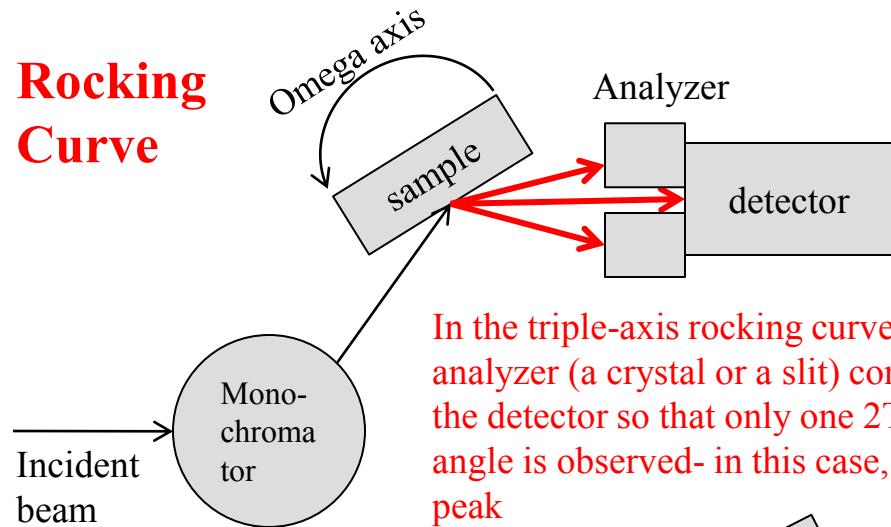
Triple Axis



Triple-Axis Rocking Curve vs Coupled Scan

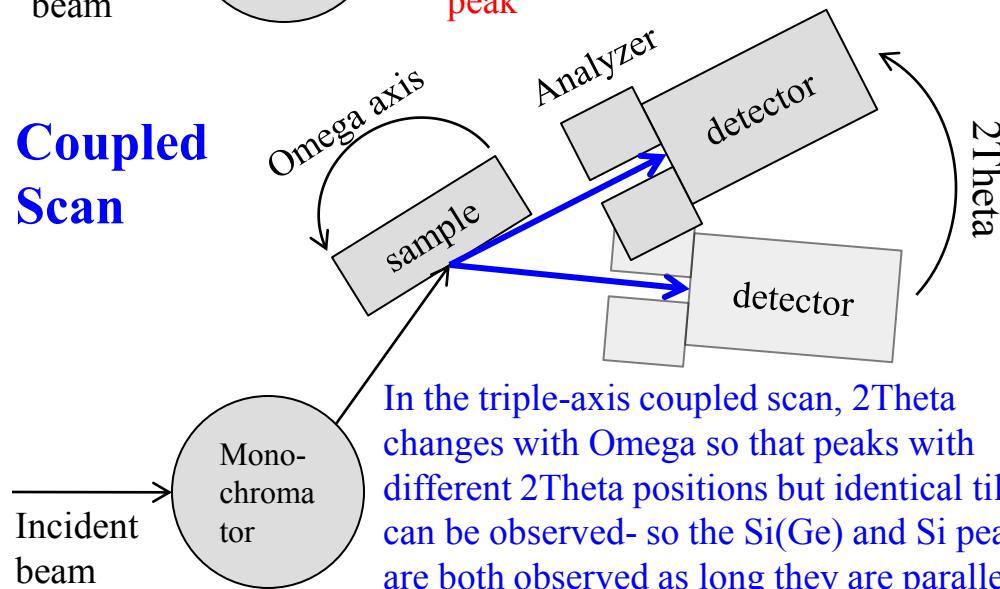
A Si(Ge) film on Si was scanned using a rocking curve and a coupled Omega-2Theta Scan

Rocking Curve

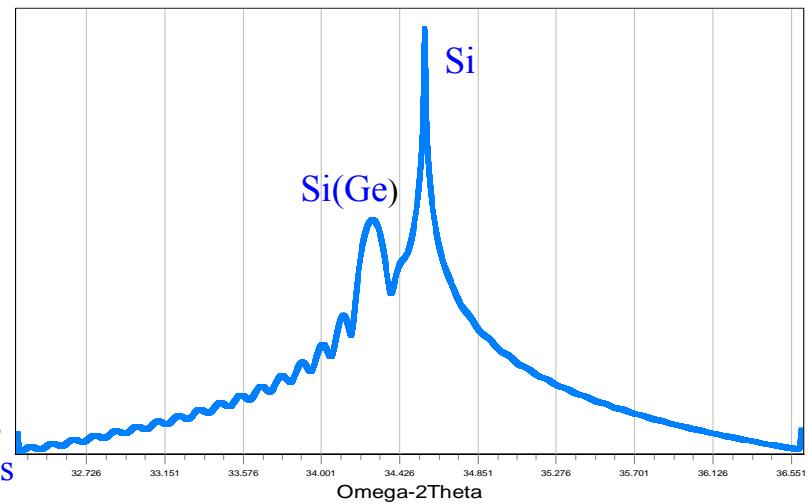
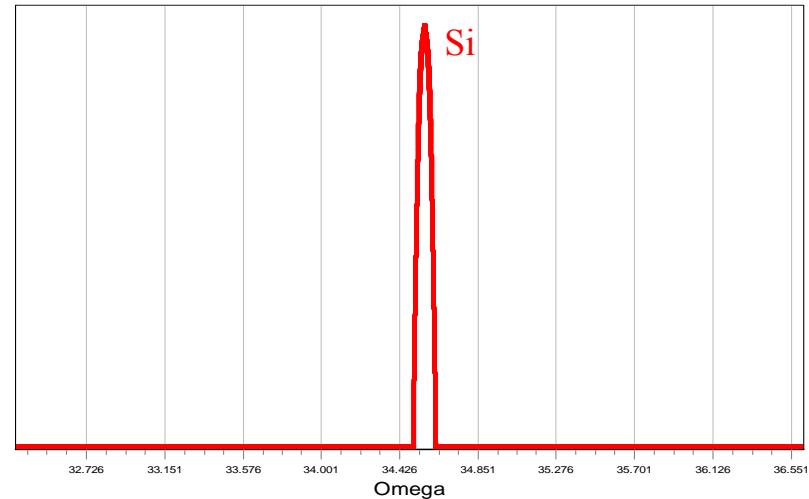


In the triple-axis rocking curve, the analyzer (a crystal or a slit) constrains the detector so that only one 2Theta angle is observed- in this case, the Si peak

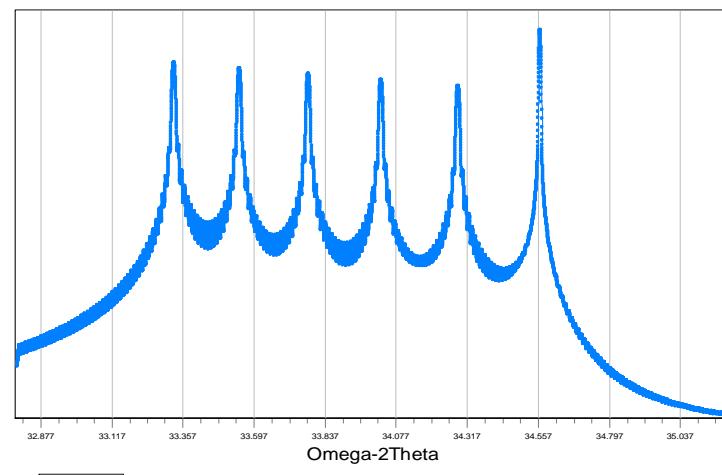
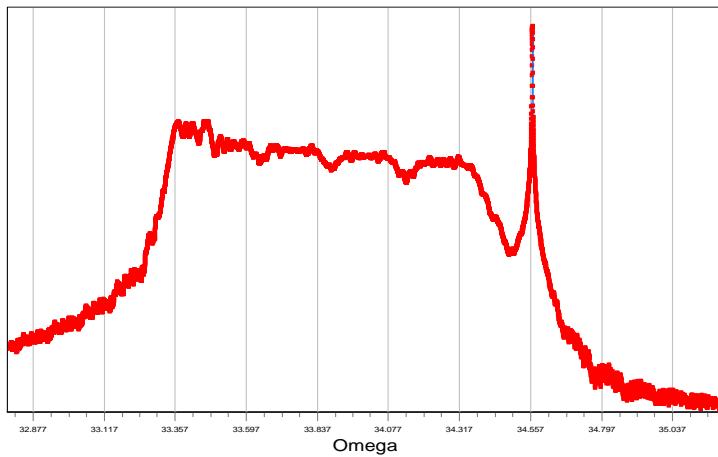
Coupled Scan



In the triple-axis coupled scan, 2Theta changes with Omega so that peaks with different 2Theta positions but identical tilts can be observed- so the Si(Ge) and Si peaks are both observed as long they are parallel



The Triple-Axis Coupled Scan Allows you to discern more complicated detail in a measurement



- The double-axis rocking curve a Si wafer coated with 5 slightly relaxed Si(Ge) layers of varying Ge concentration
- The Ge concentrations were 10, 20, 30, 40, and 50%.
- Each Ge layer was 500nm thick.
- The triple-axis coupled Omega-2Theta scan of the same Si wafer coated with 5 slightly relaxed Si(Ge) layers of varying Ge concentration
- A rocking curve in triple-axis mode can be collected for each individual peak to determine the tilt variation of each individual Si(Ge) layer



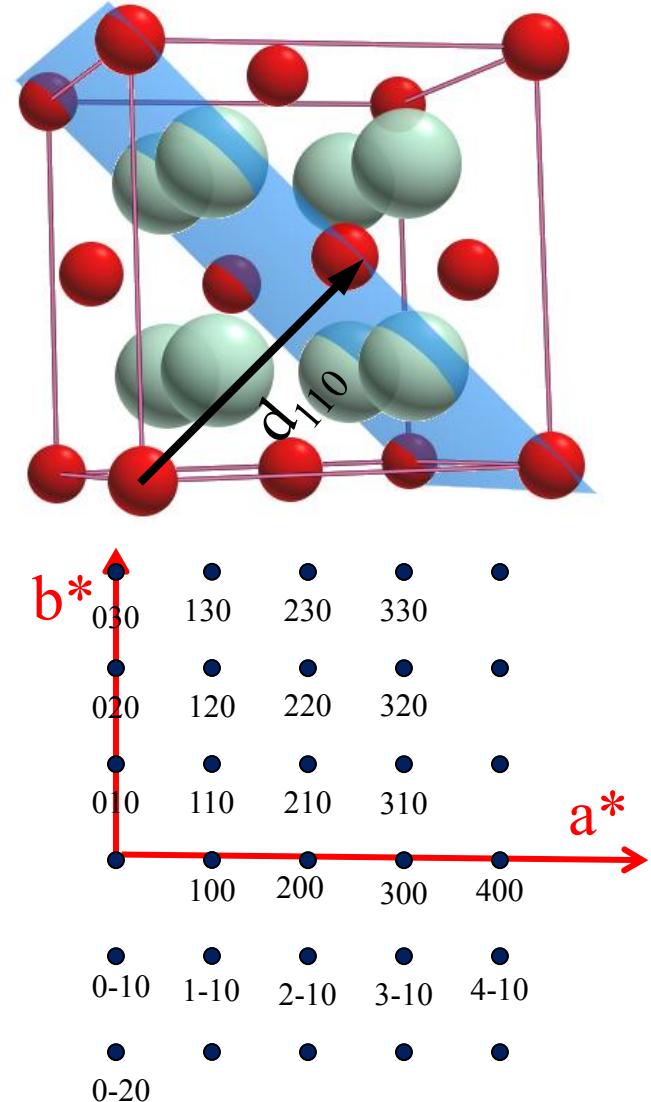
Triple-Axis Diffractometry: coupled scans vs Reciprocal Space Maps

- Coupled scan collects data as omega-2Theta or 2Theta-omega
 - The detector angle 2Theta is moved at twice the rate as the sample rotation about omega
 - $2\text{Theta} = 2 * \text{omega} + \text{tilt}$
 - This will observe peaks with different Bragg angles, but only for one specific tilt
 - If the epilayers are tilted with respect to the substrate, then a single coupled scan cannot observe both substrate and film peaks
 - In order to observe possible data, must collect coupled scans for a range of tilts: this is the Reciprocal Space Map
- The Reciprocal Space Map collects several omega-2Theta coupled scans, but each coupled scan is collected with a slightly different tilt (offset) in the omega direction
 - When the scan is collected, 2Theta still moves at twice the rate as the sample rotation so that $2\text{Theta} = 2 * \text{Omega} + \text{tilt}$
 - The tilt value is slightly different for each coupled scan that is collected
 - This is equivalent to what we did on the previous slide when we collected the rocking curve for each Si(Ge) peak that we observed— instead the reciprocal space map produces a complete map of Omega-2Theta vs Tilt (omega)

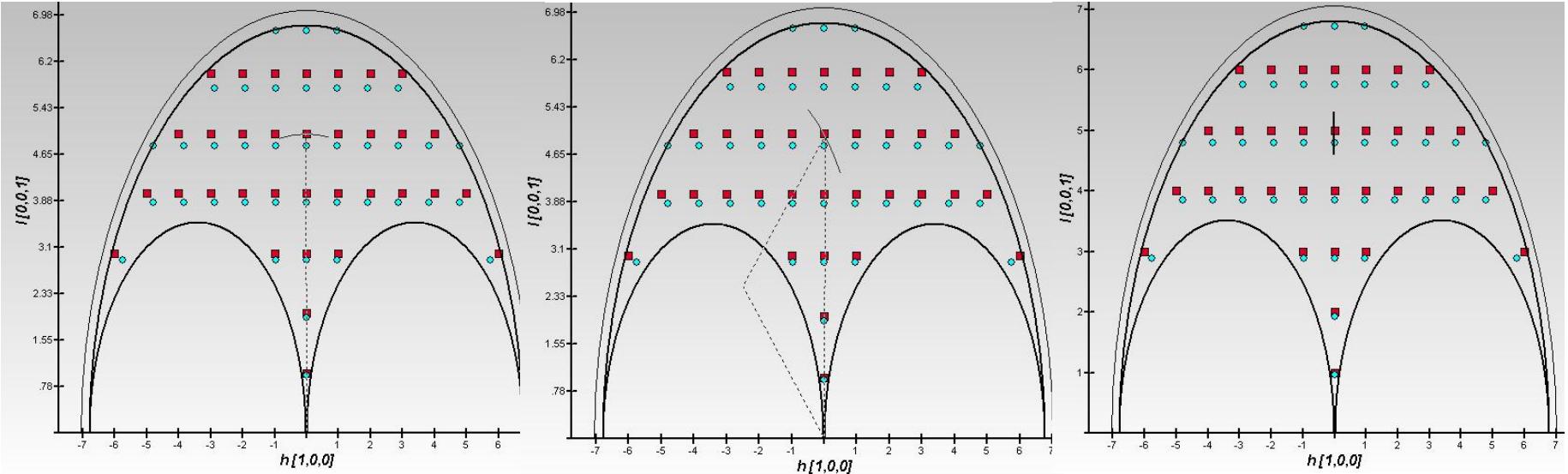


Defining Reciprocal Space

- d_{hkl} is the vector drawn from the origin of the unit cell to intersect the first crystallographic plane in the family (hkl) at a 90° angle
- The reciprocal vector is $d^*_{hkl} = 1/d_{hkl}$
- In the reciprocal lattice, each point represents a vector which, in turn, represents a set of Bragg planes
- Each reciprocal vector can be resolved into the components:
 - $d^*_{hkl} = ha^* + kb^* + lc^*$



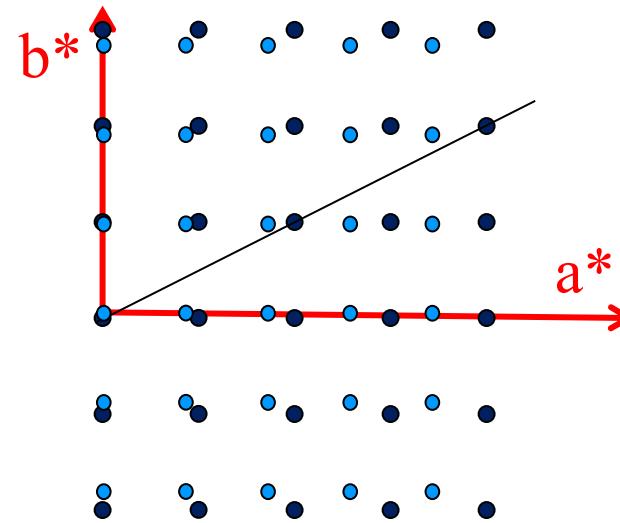
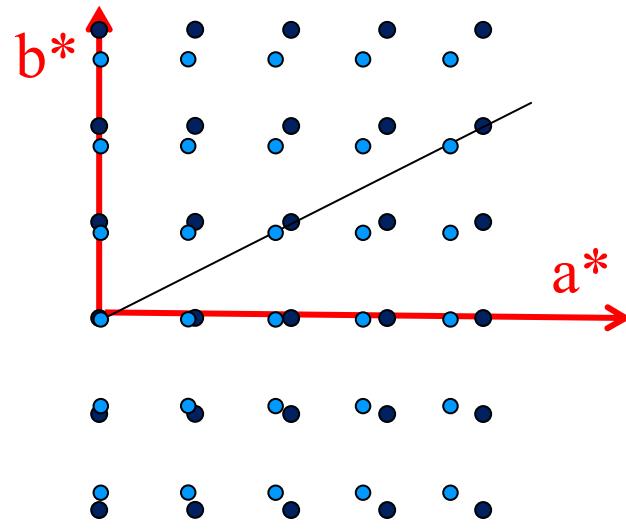
Different scan types cover different regions of reciprocal space



- The rocking curve (omega scan) is an arc centered on the origin
- The detector scan (2theta scan) is an arc along the Ewald sphere circumference
- The couple scan (2theta-omega scan) is a straight line pointing away from the origin

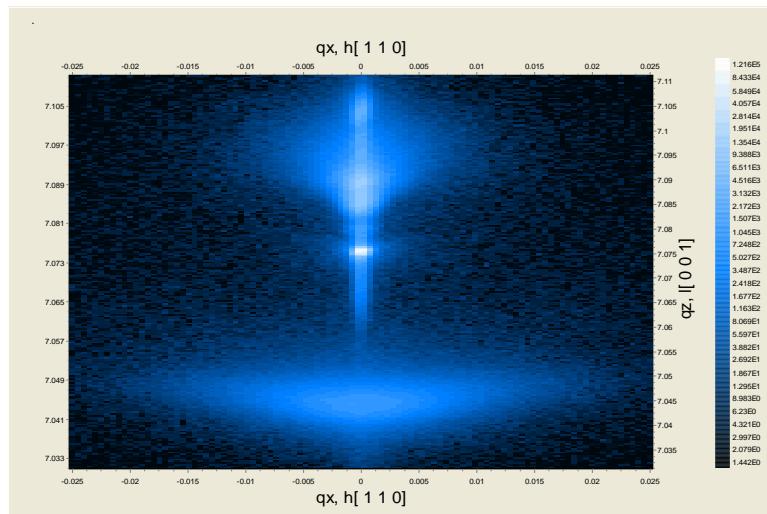
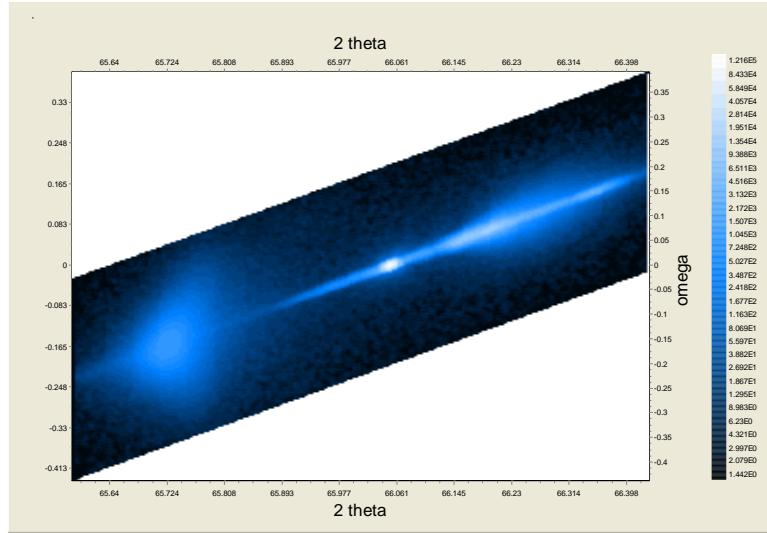


Effects such as strain will shift reciprocal lattice points, preventing the collection of data with a single scan



- The reciprocal space map uses multiple scans in order to observe both the film and substrate peaks

Converting to reciprocal space units



- The sample was a (001) oriented wafer; the (004) Bragg was mapped
 - The [004] direction is normal to the plane of the wafer
 - [110] is a lateral direction (ie the direction within the plane of the film)
- Position in q_z correlates to the d-spacing of the peak
- Position in q_x correlates to tilt of planes
- Map of the symmetric Bragg peak can be used to separate tilts and strain
 - To separate composition/mismatch and strain, need to map an asymmetric peak

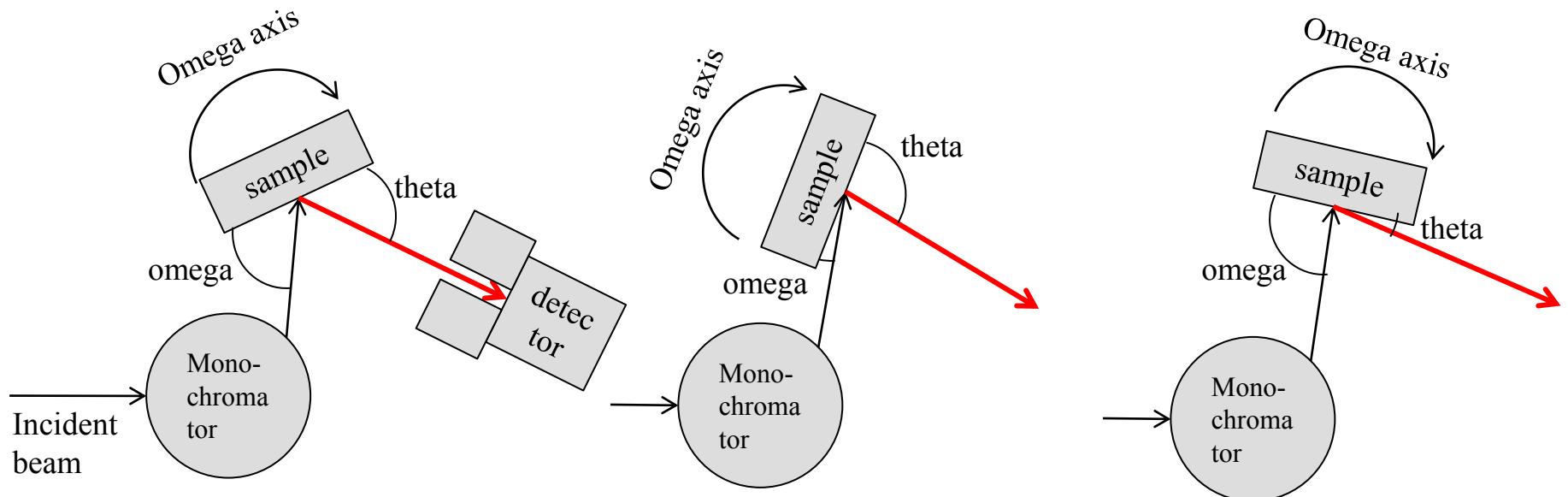


Symmetric vs Asymmetric

- One family of planes is parallel or nearly parallel to the surface of the sample.
 - These are the only planes examined 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 $2\Theta=2*\Omega+\text{tilt}$
 - 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 (summarized later)

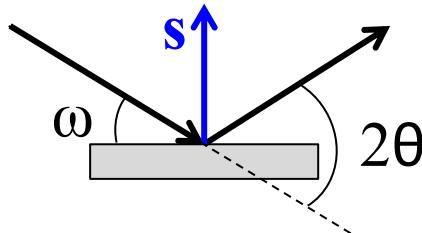


Symmetric vs Asymmetric

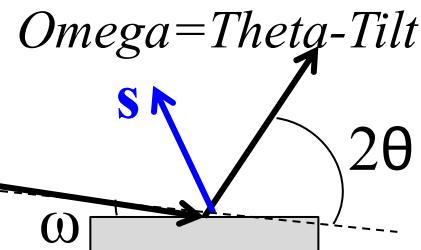


Symmetric Scan

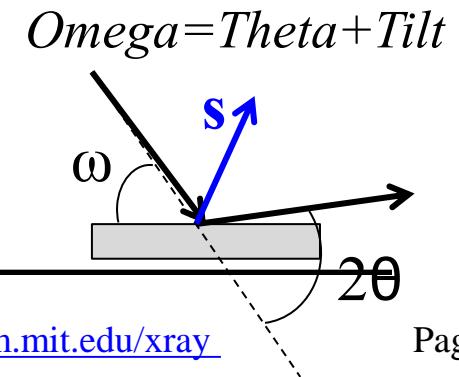
$\Omega = \theta$



Asymmetric Scan
Grazing Incidence (-)



Asymmetric Scan
Grazing Exit (+)



Our Triple Axis Machine

- Incident beam optics
 - Mirror only (for XRR)
 - Mirror + Ge(022)x4 asymmetric monochromator
 - Mirror + Ge(044)x4 symmetric monochromator
 - Could be tuned to Ge(022)x4 symmetric monochromator
 - Slits to control the height and width the X-ray beam
- Receiving-Side Optics
 - Motorized receiving slit + point detector
 - Ge(022)x3 Analyzer Crystal + point detector
 - Linear Position Sensitive Detector- point mode or 1D mode
 - PSD in high dynamic range configuration (90deg mount) and point mode with manual receiving slit for XRR



HRXRD requires an Incident Beam Monochromator

- If the incident beam contains both $K\alpha 1$ and $K\alpha 2$ radiation, much of the important information from the film will be lost
- The incident beam must also have very low divergence
 - The source profile of the X-ray beam will obscure broadening of the rocking curve caused by defects in the epilayer
- The best signal is produced when the divergence of the incident X-ray beam matches the quality of the film
 - An X-ray beam with very low divergence will scatter with low efficiency from a highly distorted film
 - For example, Si-Ge multilayers often have some relaxation in each layer, which also produces a small amount of threading dislocations. A lower resolution (more divergence) monochromator will give a stronger signal than a high resolution (less divergence) monochromator from such a sample ... without compromising resolution.



Values comparing Bruker monochromators when coupled with a Goebel Mirror

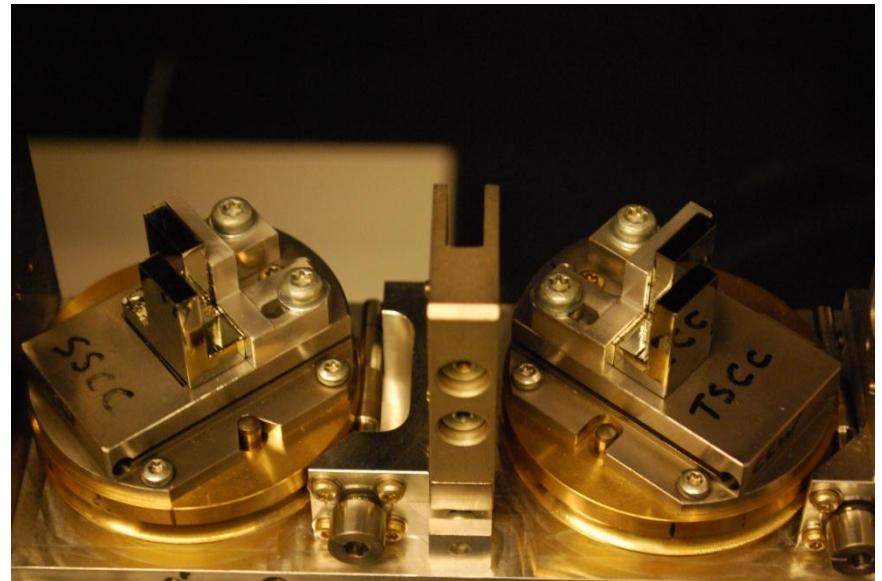
Monochromator	Divergence (arc-seconds)	Beam Intensity (cps)	FWHM of Si(022) (°)
None (mirror only)	108"	170,000,000	0.07
Ge(022)x4 symmetric	12"	4,500,000	0.0035
Ge(022)x4 asymmetric	25"	18,000,000	0.008
Ge(044)x4 symmetric	5"	150,000	0.0015

- The mirror refocuses the divergent beam into a pseudo-parallel beam, producing less divergence and an intensity gain
 - The pseudo-parallel beam from the mirror interacts more efficiently with the monochromator, producing a stronger incident X-ray beam
- A 2-bounce monochromator gives good intensity and peak shape, but requires a slit to define the spectral bandpass—so not all K α 2 is removed
 - We use a 4-bounce monochromator instead—we lose some incident beam intensity, but have a better quality beam with no K α 2 and better collimation (ie resolution)
- Using Ge instead of Si for the monochromator yields higher intensity, but costs more and has high losses from polarization
 - The asymmetric design reduces the polarization losses to give higher intensity



This image shows a 4-bounce Ge monochromator

- Each pair of diffracting crystals is channel-cut from a single piece of Ge
 - This prevents misorientation between the pair of crystals
- Two sets of channel-cut crystals are used
 - The orientation between these two sets must be precisely aligned to get a usable X-ray beam
- Slits are used control the width of the beam entering the first channel-cut crystal and to control the width in-between the two sets of channel-cut crystals



Historical- why omega-2Theta and why regard as mismatch/relaxation

- HRXRD started as rocking curve (omega scans) using double-axis instruments
- When triple axis developed to do coupled scans of omega and 2theta, it was referenced as omega-2theta
 - In powder diffraction, it is referenced as 2theta-omega
- Mismatch/Relaxation
 - Starting assumption is that you want fully strained lattice matched epitaxial thin films
 - Therefore, mismatch and relaxation are regarded as “defects”
 - Mismatch and partial relaxation may be desired for ternary films, but the analysis software will still often regard them as defects



**PEAK POSITIONS ARE AFFECTED BY
RELAXATION/STRAIN, COMPOSITION, AND TILT**

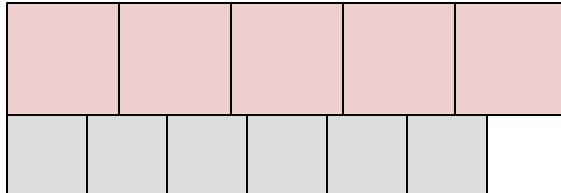


Massachusetts Institute of Technology

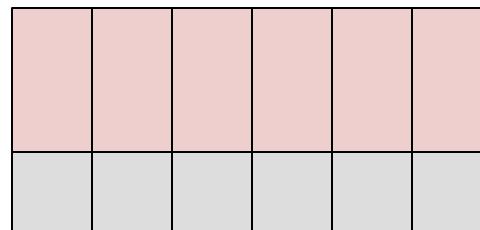
<http://prism.mit.edu/xray>

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Relaxation



Relaxed Film

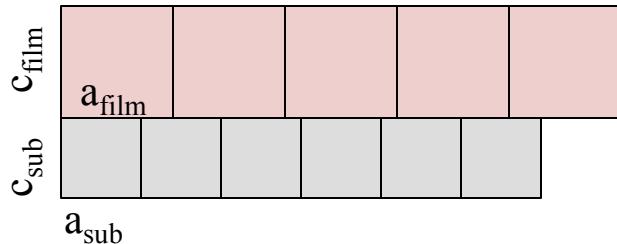


Strained Film

- If the film is mismatched to the substrate, then the film might be strained so that the lattice parameters in the lateral direction (ie within the plane of the film) are forced to match the lattice parameters of the substrate
- This distorts the unit cell of the film
 - A formerly cubic unit cell is now tetragonal
- Determine the degree of relaxation
 - No relaxation (fully strained)- the lateral lattice parameters of the film are strained to be identical to the substrate
 - Fully relaxed- the lateral lattice parameters of the film are equal to the bulk values– they have not been distorted at all

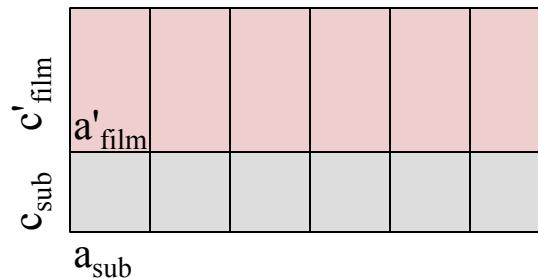
Relaxation

Relaxed Film



- $a_{\text{sub}}=c_{\text{sub}}$
- $a_{\text{film}}=c_{\text{film}}$
- $a_{\text{film}} \neq a_{\text{sub}}$
- $(001)_{\text{sub}} // (001)_{\text{film}}$
- $(101)_{\text{sub}} // (101)_{\text{film}}$
- Difference in Bragg angles b/w film and substrate is by splitting of peaks in the Rocking Curve and multiple peaks in Coupled Scan
- Asymmetric coupled scans show Bragg diffraction from both film and substrate

Strained Film

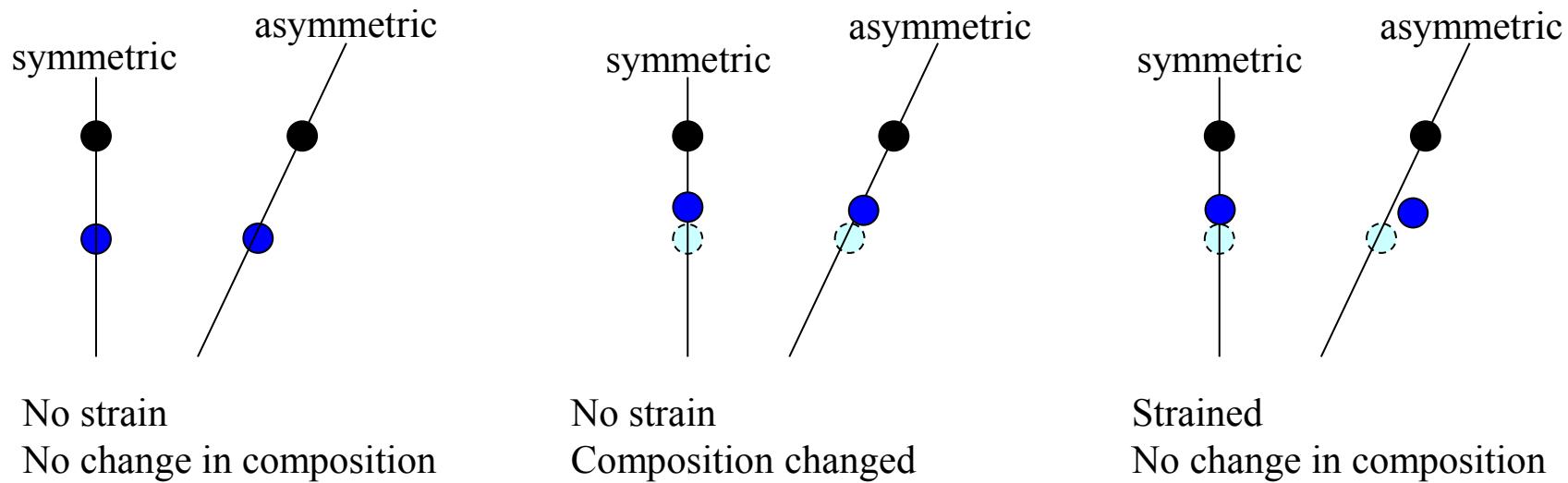


- $a_{\text{sub}}=c_{\text{sub}}$
- $a'_{\text{film}} \neq c'_{\text{film}}$
- $a'_{\text{film}}=a_{\text{sub}}$
- $(001)_{\text{sub}} // (001)_{\text{film}}$
- $(101)_{\text{sub}}$ not parallel $(101)_{\text{film}}$
- The Bragg angle for (001) shifts from its theoretical position, seen in rocking curve and coupled scans
- Asymmetric coupled scan shows a film peak or substrate peak, but not both because they are not parallel
- Separation between peaks in Rocking curves changes with the scan geometry (GE vs GI vs sym)

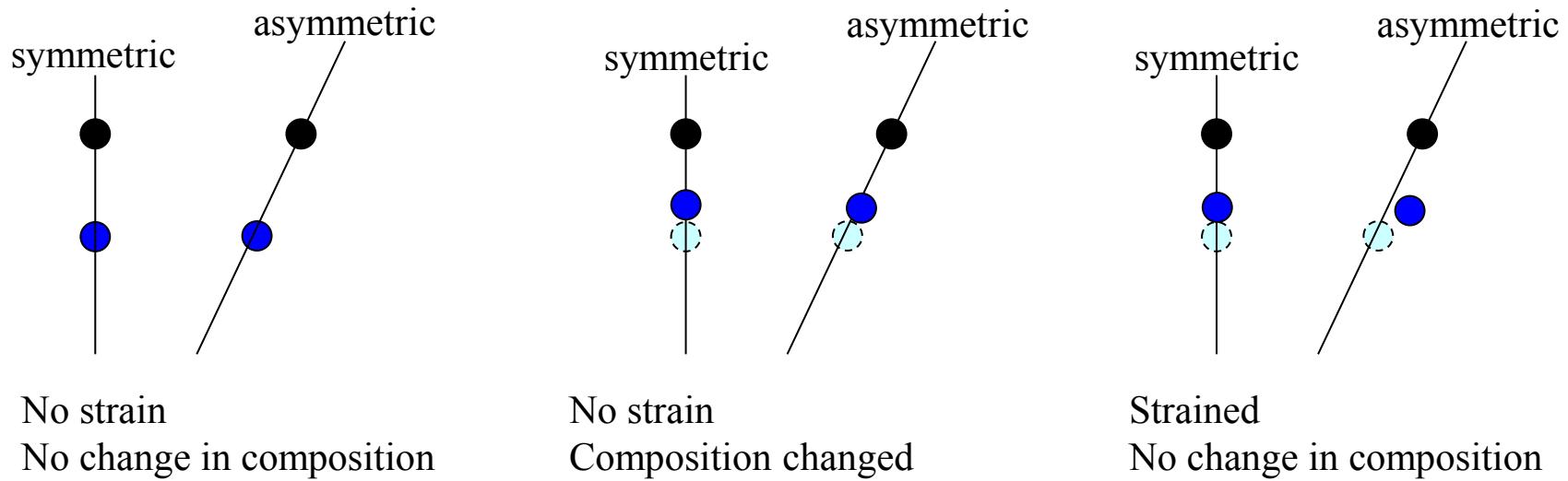


Composition

- In substitutional solid solutes, the composition can vary
- Changes in the composition will change the lattice parameters, which will change d_{hkl} and therefore the Bragg peak positions
 - Unlike relaxation, changes in composition will not change lattice tilts

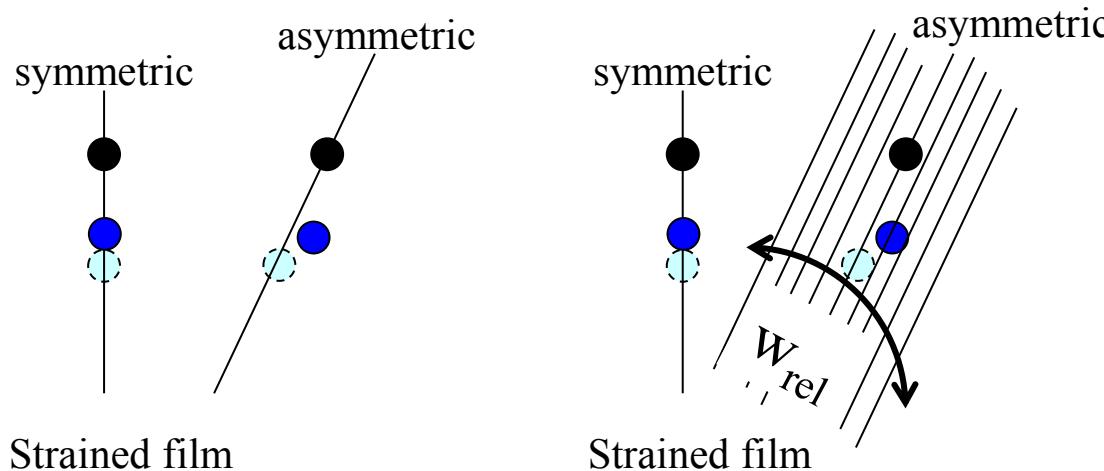


Symmetric scans cannot distinguish between strain and compositional changes



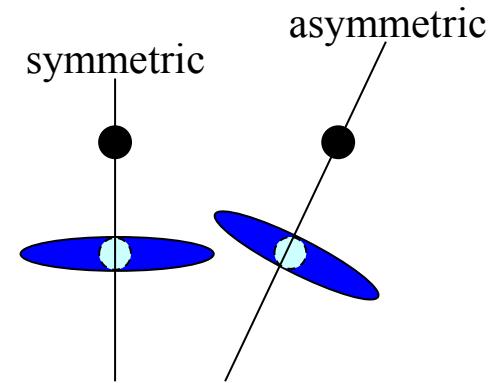
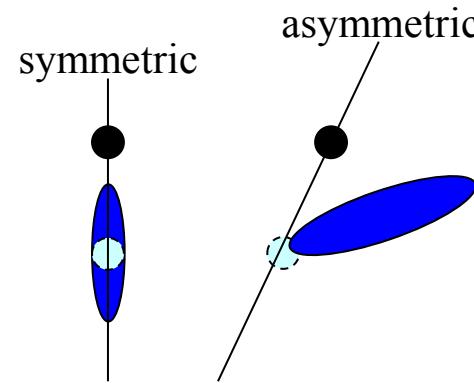
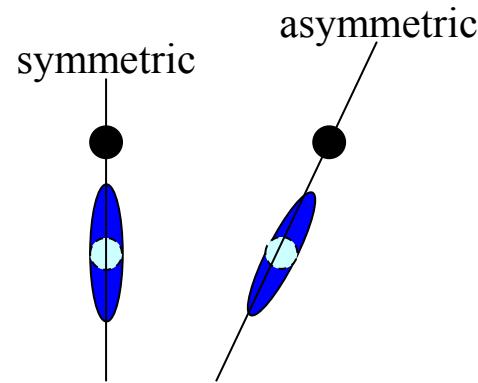
- In the symmetric scan, strain and compositional changes produce similar peak shifts
- In order to quantify both strain and composition, must combine a symmetric scan with an asymmetric scan

If the film is highly strained, a single coupled asymmetric scan produce usable data

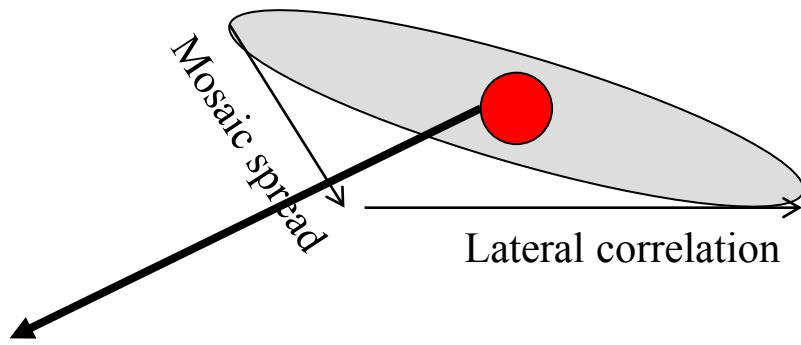


- The typical way to collect reciprocal space maps is to vary relative omega and collect multiple 2theta-omega coupled scans

Defects and gradients can produce spreading of the reciprocal space point



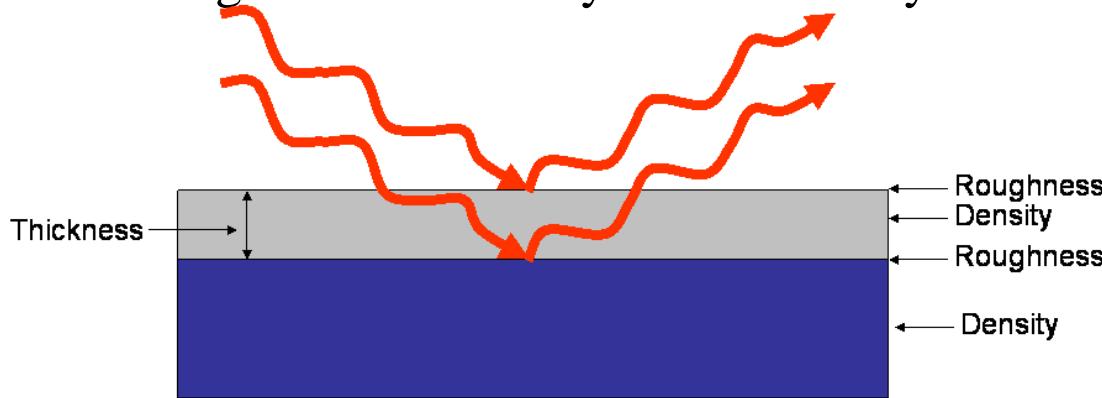
Mosaic Spread can be quantified by measuring the broadening of the lattice point in reciprocal space



- The amount of broadening of the reciprocal lattice point that is perpendicular to the reflecting plane normal can be attributed to mosaic spread
- The peak broadening parallel to the interface can be attributed to lateral correlation length

X-Ray Reflectivity (XRR)

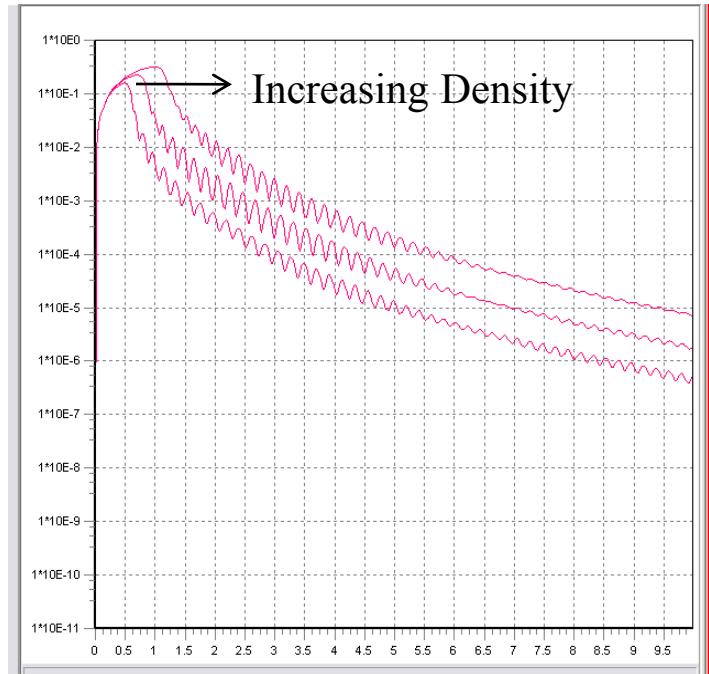
- The same equipment that is optimized for HRXRD can also be used for XRR analysis of thin films.
- X-ray waves reflecting from each different surfaces in a multilayer thin film.
 - The multiple reflected waves interfere with each other, producing a reflectivity curve
 - The XRR scan can be used to determine the density, thickness, and roughness of each layer in a multilayer thin film.



This image is taken from training materials provided by Bruker AXS

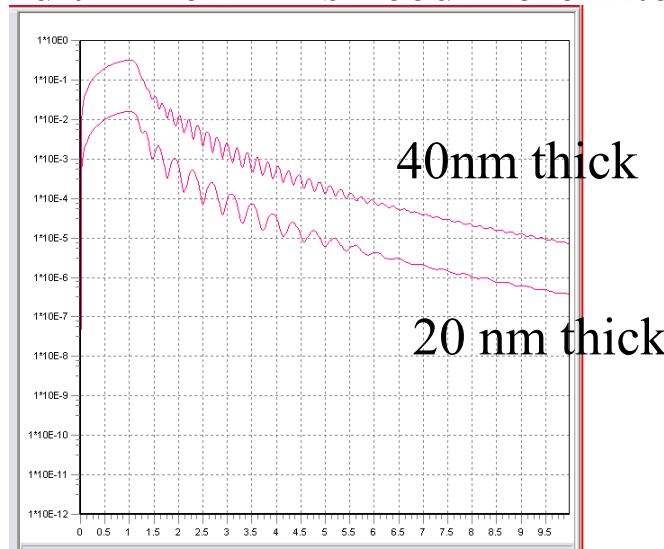
The critical angle is a function of the density and composition of the layer

- Below the critical angle, θ_C , the X-ray beam is completely reflected (total external reflection)
- The critical angle for a layer is a function of its electron density
 - This is a convolution of density and composition
 - If one is known, the other can be determined using XRR
 - For example, for a given composition, as the density of the film increases the critical angle θ_C often increases.



The distance between interference fringes is a function of the thickness of the layers

- Interference fringes are created by the phase difference between X-rays reflected from different surfaces
- The distance between the fringes is inversely proportional to the thickness of the layer
 - Because of this, thicker films need better resolution (use a monochromator) and thinner films need more intensity (use only the mirror)



Roughness determines how quickly the reflected signal decays

- Roughness causes X-rays to be scattered rather than reflected
 - This produces a decay in the reflected beam intensity
 - The loss of beam intensity increases with Theta
- A rougher surface produces more diffuse scatter, causing the reflected beam intensity to decay more with Theta
 - The diffuse scatter can be measured to look for order in the roughness of the film.

