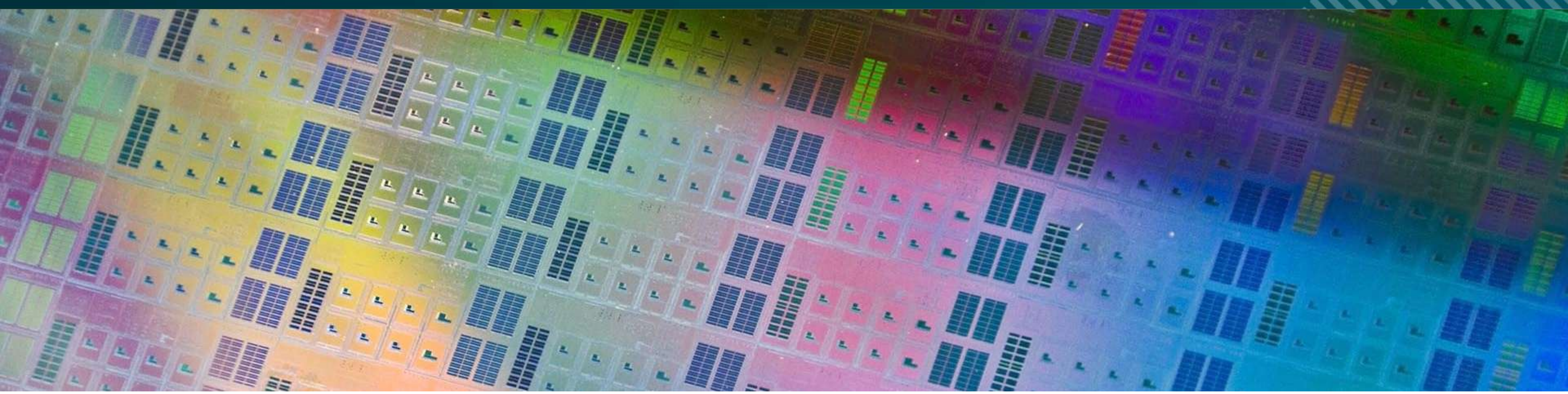
Introduction to Grazing Incidence and X-Ray Reflectivity

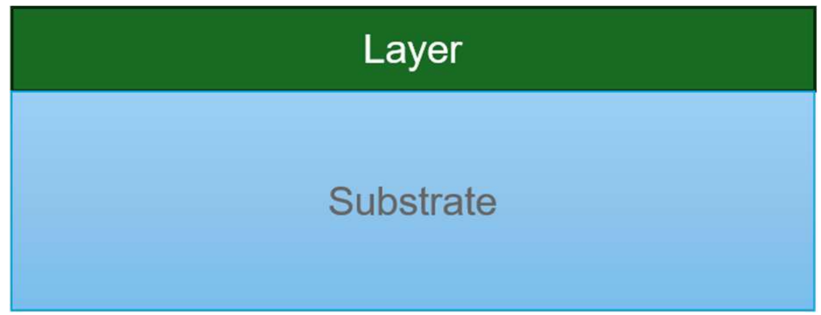
Brad Losey, PhD

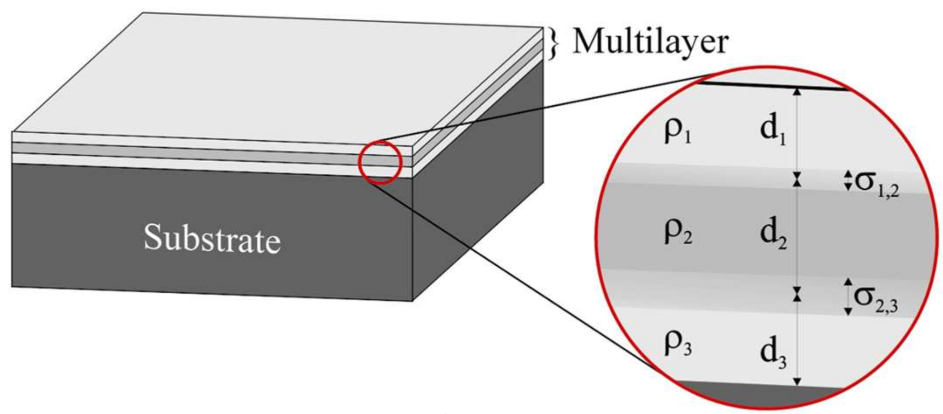


Introduction to Thin Films

Structure

- Characteristics
  - Nanometers to microns thick
  - Used to impart specific electrical, optical, magnetic, or mechanical properties.
- Substrate:
  - Used to control the deposition of the “active” layer(s).
  - Material chosen is based on cost, flatness, rigidity, thermal & electrical conductivity, offcut, etc.
  - Can be single crystal, polycrystalline, amorphous.
  - If there is a crystallographic relationship between the layer and substrate, it is epitaxial.
- Layer:
  - Usually metallic or semiconducting
  - Can be multi-layered

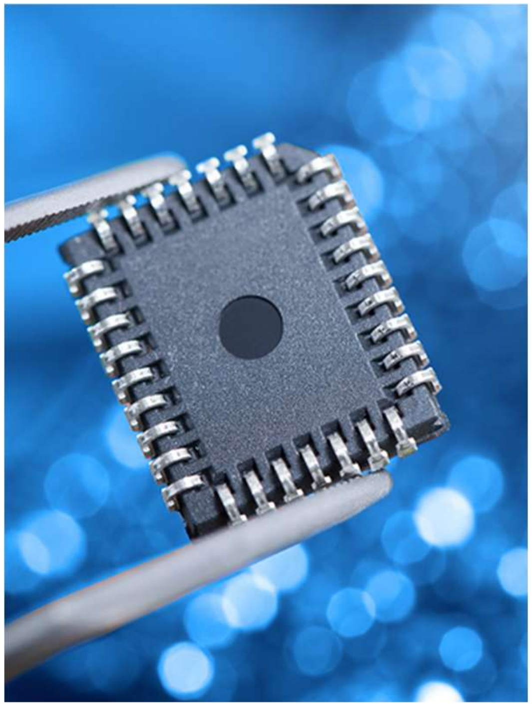




Introduction to Thin Films

Applications

- Microelectronics and Semiconductors
  - Microchips
  - Integrated Circuits
  - Sensors
  - Transistors
  - Displays
- Optics and Photonics
  - Anti-Reflective Coatings
  - Mirrors
  - Optical Filters
- Energy Generation and Storage
  - Photovoltaics
  - Batteries
- Protective Coatings
  - Wear Resistance
  - Biomedical Implants



Introduction to Thin Films

Measurement Techniques

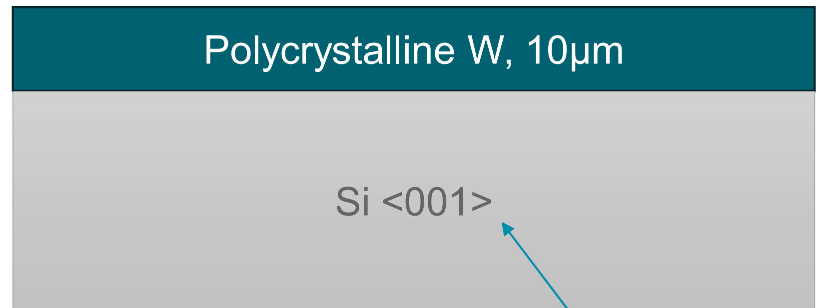
- **Grazing incidence (GI-XRD)**
  - Utilized for phase identification, residual stress, texture, crystallite size/strain and depth profiling
  - Generally restricted to polycrystalline films
  - Parallel beam geometry with a point detector
- **X-ray Reflectivity (XRR)**
  - Utilized to obtain density, thickness and roughness (surface and interface)
  - Can be applied to single crystal, polycrystalline and amorphous films
  - Parallel beam geometry/high-resolution optics with a point detector

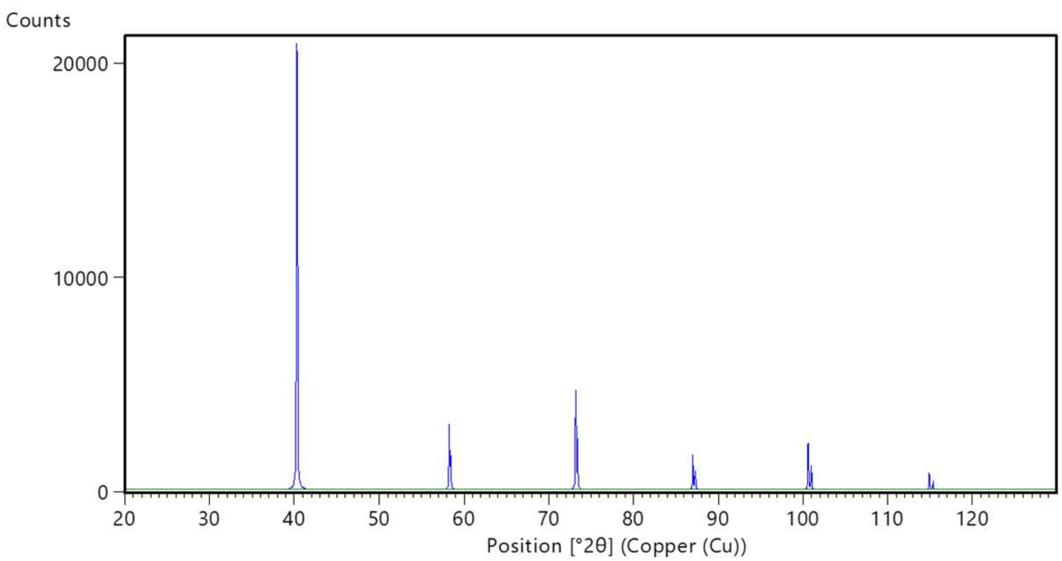
Introduction to Thin Films

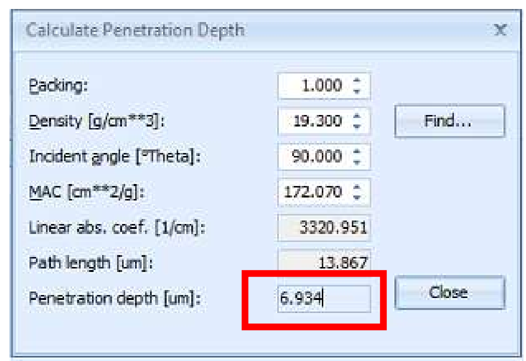
Thickness Considerations

- With this film material and thickness, only the top layer will be observed

Single-phase, tungsten layer



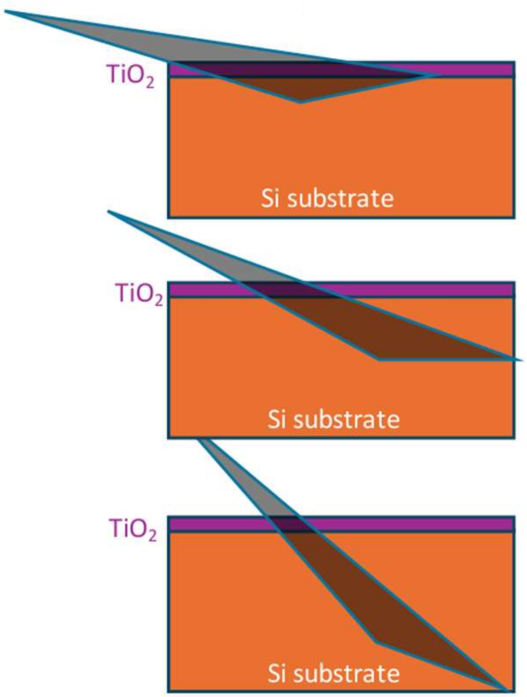




This means it is single crystal silicon with the <001> direction perpendicular to the surface

Introduction to Grazing Incidence

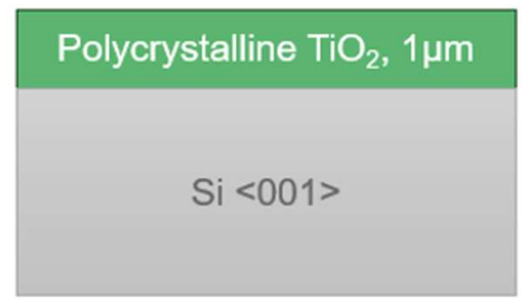
X-Ray Penetration Depth



At 10°2θ (ω=5°)
- 55% of X-rays go through the film and into the Si substrate
- The X-ray beam is 24mm long, the irradiated volume of the film is 0.24mm^3

At 50°2θ (ω=25°)
- 88% of X-rays go through the film and into the Si substrate
- The X-ray beam is 5mm long, the irradiated volume is 0.05mm^3

At 90°2θ (ω=45°)
- 93% of X-rays go through the film and into the Si substrate
- The X-ray beam is 3mm long, the irradiated volume of the film is 0.03mm^3



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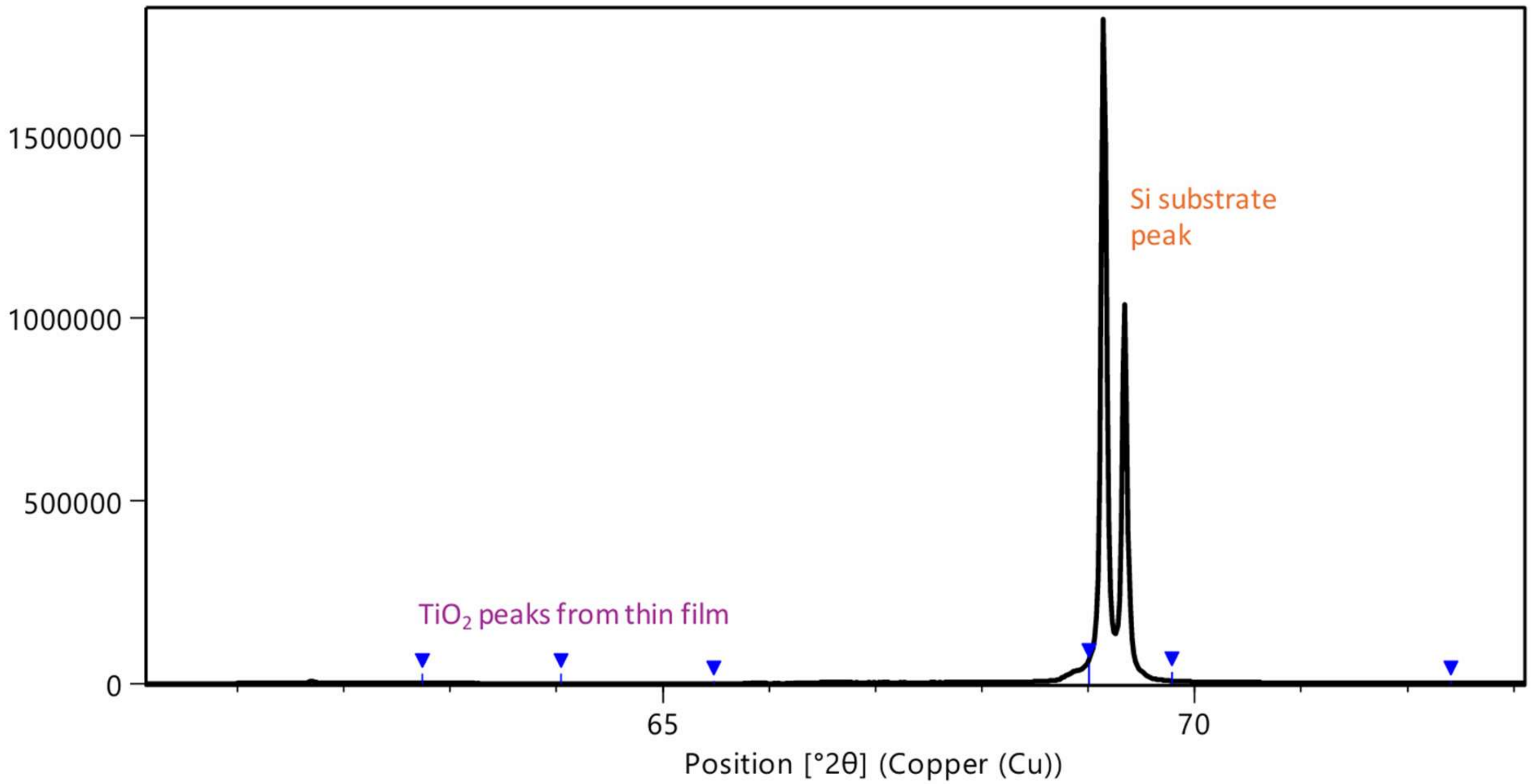
Film Intensity



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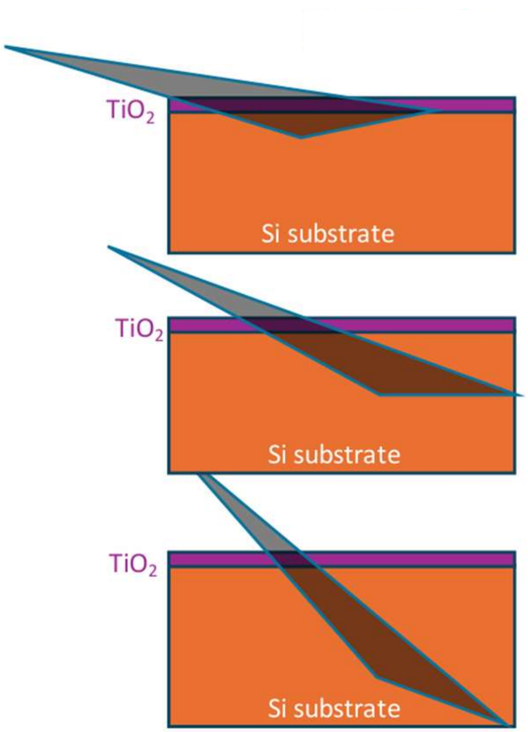
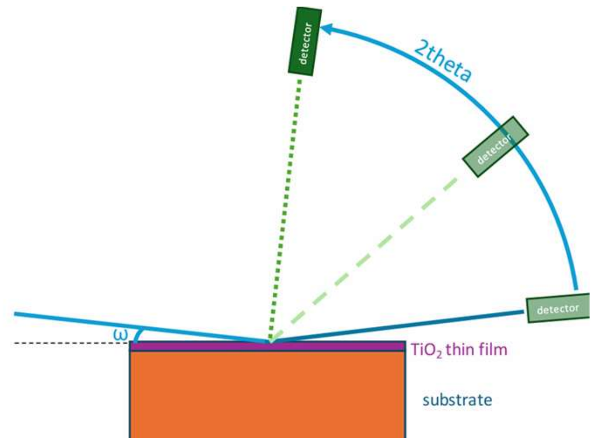
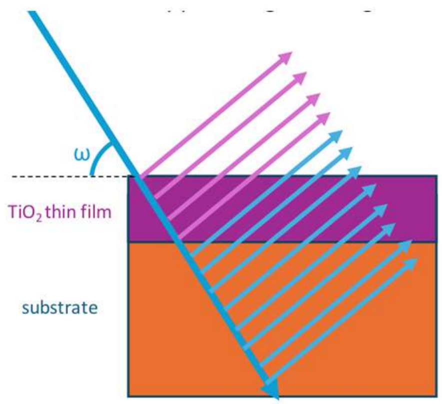
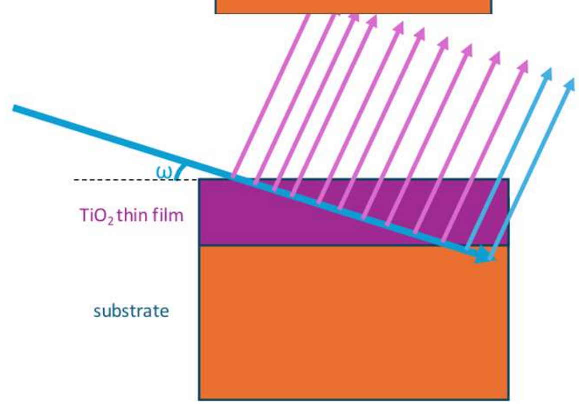
Utilizing Fixed Incidence Angles



- Incident beam held at a fixed low angle
- Improves scattering intensity from the film
- Asymmetric geometry breaks the diffracting condition for single crystals
- Constant beam footprint
- X-ray penetration depth can be controlled or varied for depth profiling





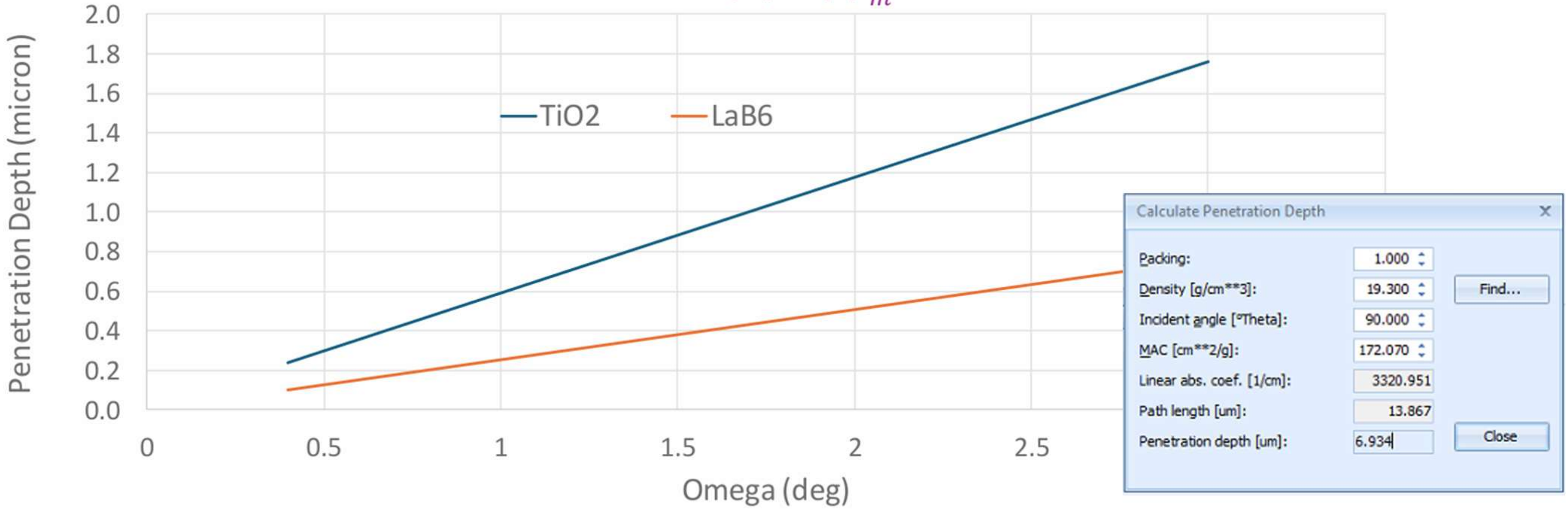


Introduction to Grazing Incidence

Penetration Depth and Film Composition

Depth of penetration calculations vary depending on what % of the X-ray beam you assume is scattered. The plots shows penetration depth calculated for 99.9% X-rays scattered (I_L/I_0=0.001)

$$T = 0.5 * \sin(\omega) * \ln\left(\frac{I_L}{I_0}\right) * \left(\frac{\mu}{\rho}\right)_m * \rho_m$$



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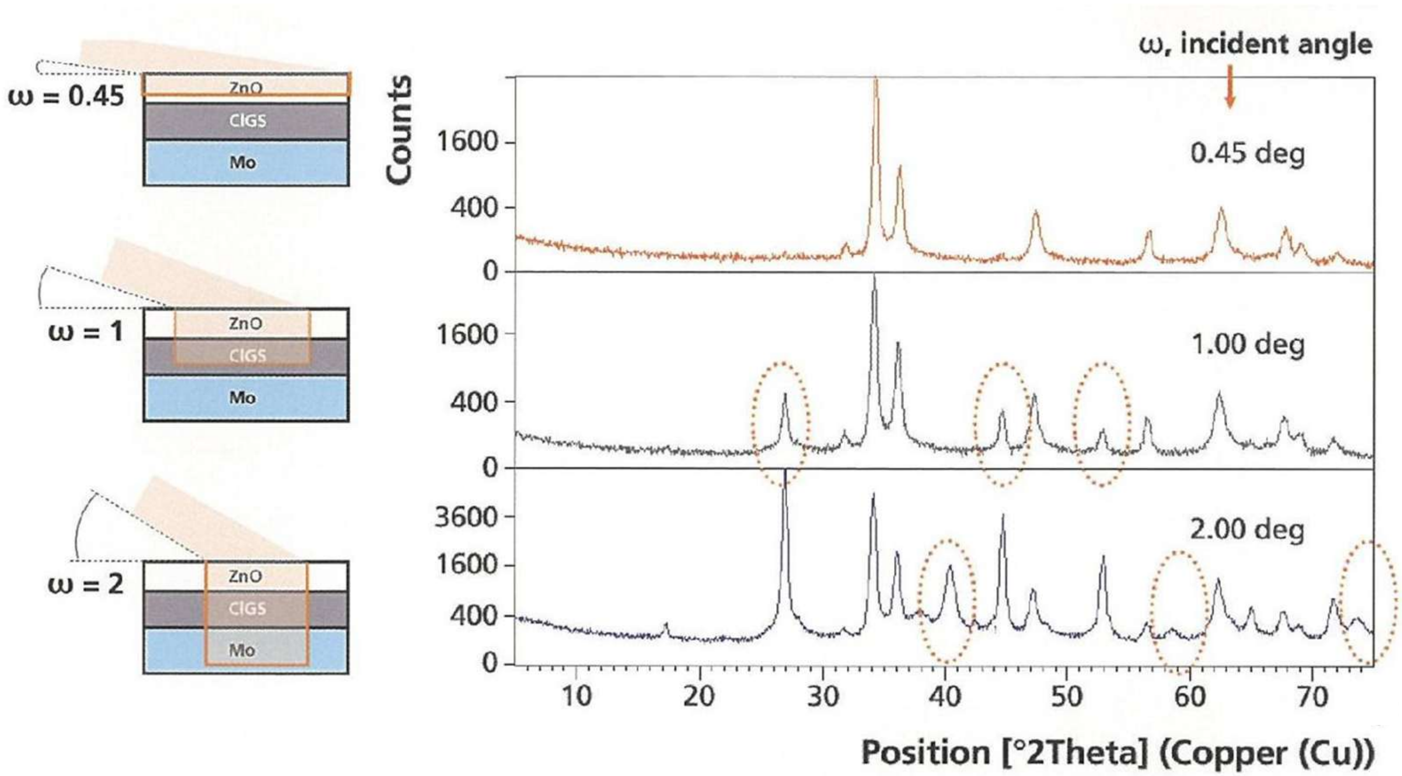
Depth Profiling



Example of depth profiling on a CIGS solar structure, showing the different layers exposed to a parallel X-ray beam in a sequential mode by varying the incident angle.

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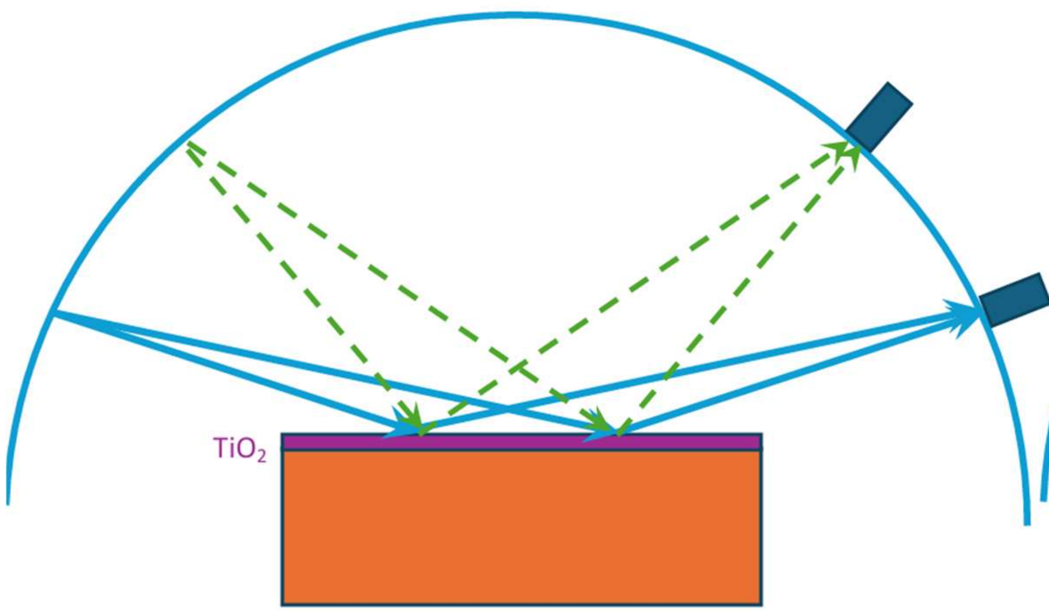
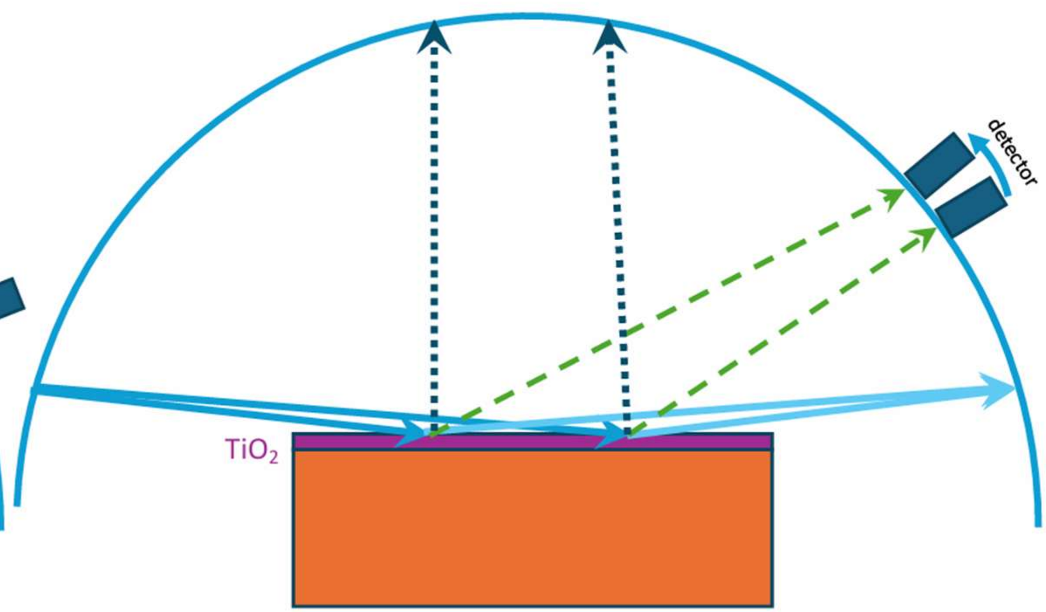
Defocusing Effects

Bragg-Brentano



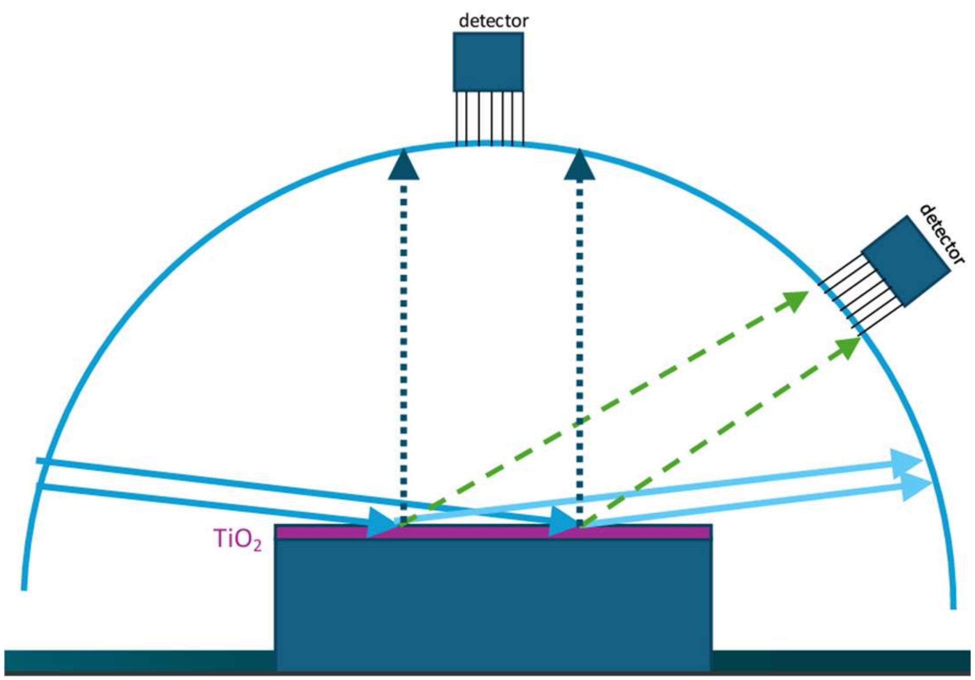
Grazing Incidence

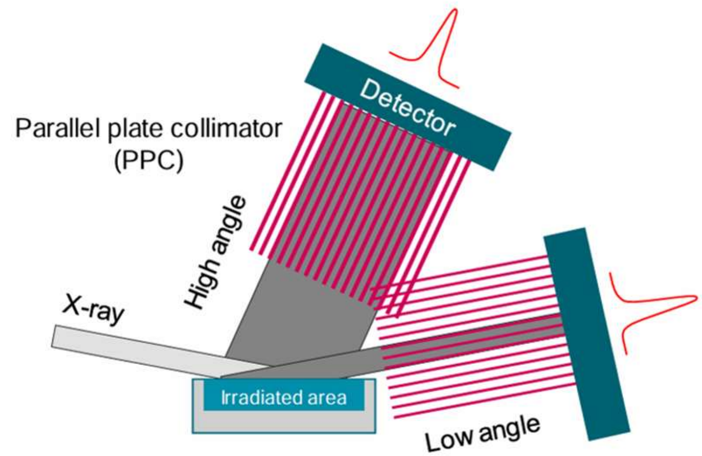


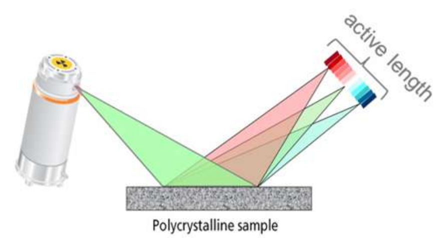
Introduction to Grazing Incidence

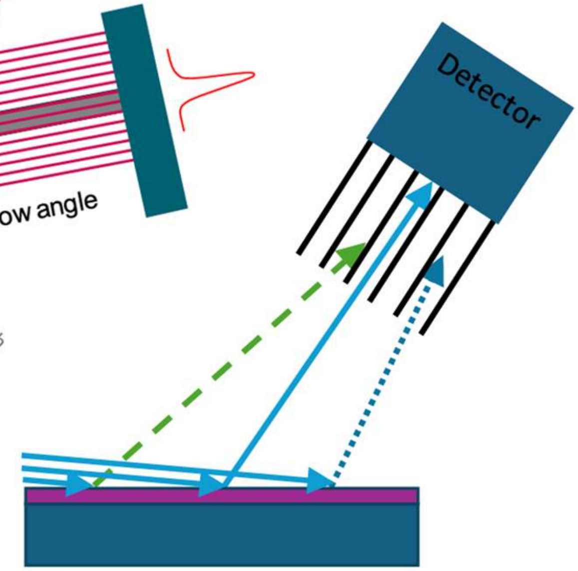
Parallel Beam Geometry

- Parabolic Mirror
- Parallel Plate Collimator
  - Narrow Acceptance Angle
- Point Detector



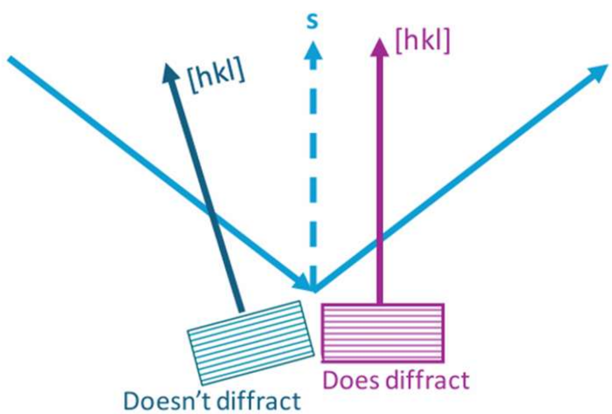




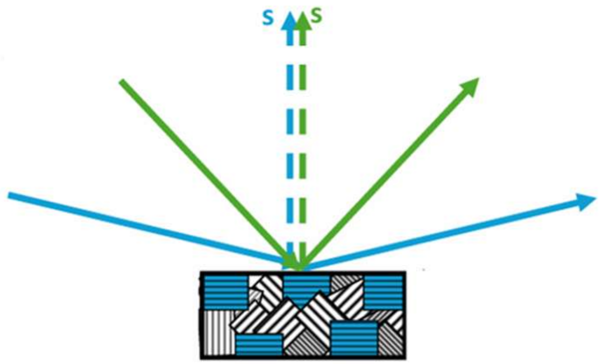


Introduction to Grazing Incidence

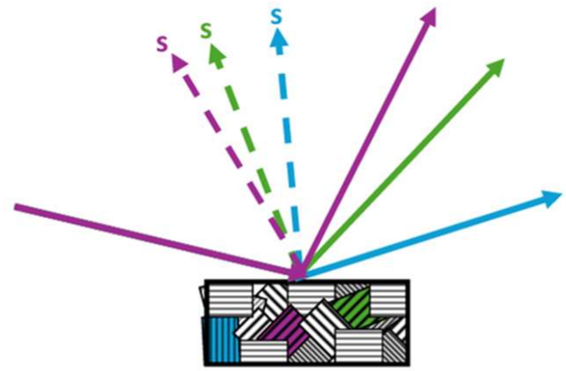
Effect on Preferred Orientation



- In a powder diffractometer, diffraction from a crystal is only observed if:
  - The detector is at the Bragg angle
  - The diffraction vector **s** is parallel to the crystallographic direction **[hkl]**



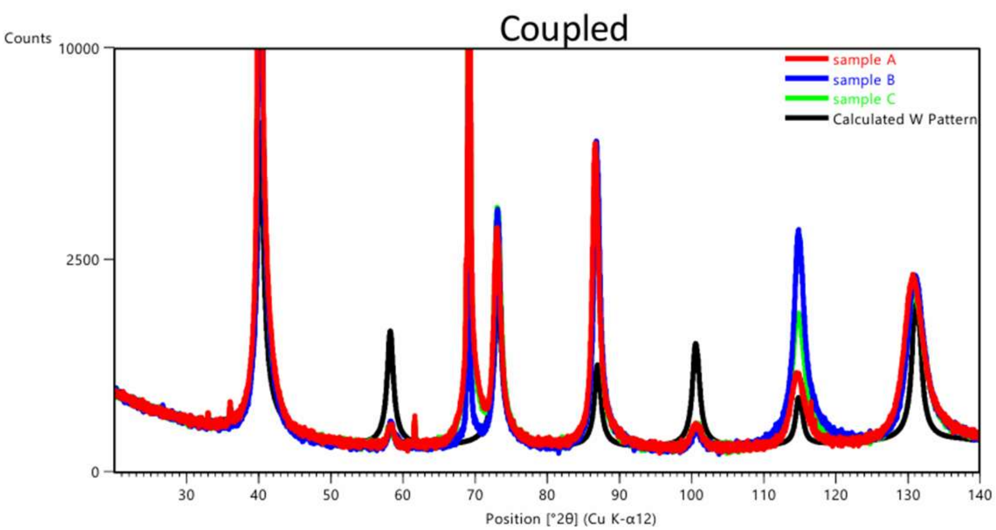
- In Bragg-Brentano geometry, the diffraction vector **s** does not change during the measurement
- Every observed diffraction peak is produced by [hkl] oriented in the sample direction

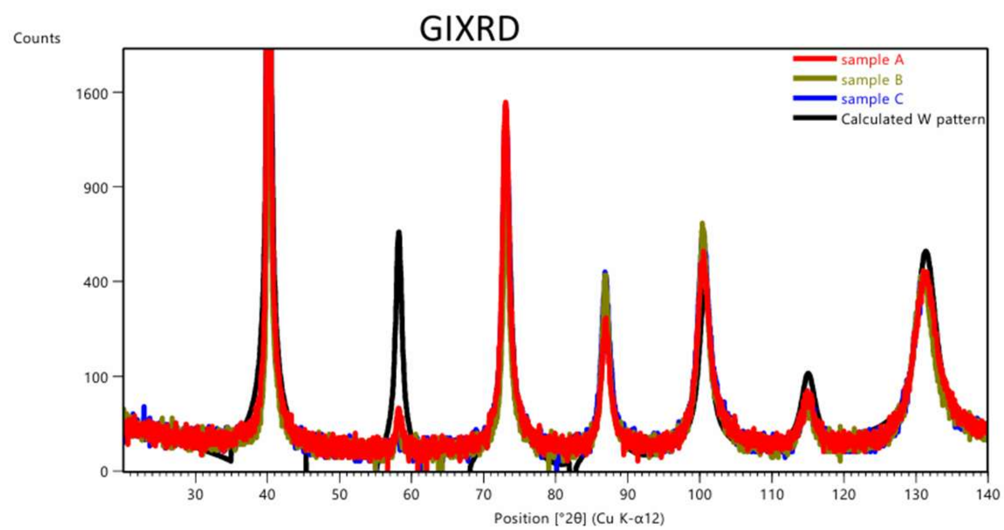


- In GIXRD geometry, the diffraction vector **s** changes with the detector angle **2Theta**
- Every observed diffraction peak is produced by [hkl] oriented in a different direction

Introduction to Grazing Incidence

Effects on Texture





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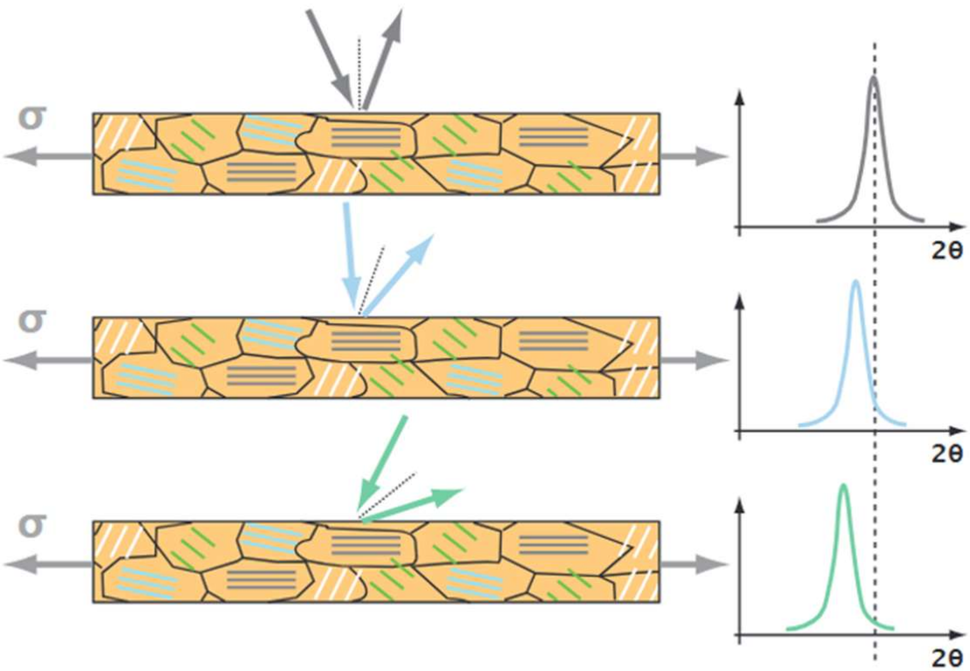
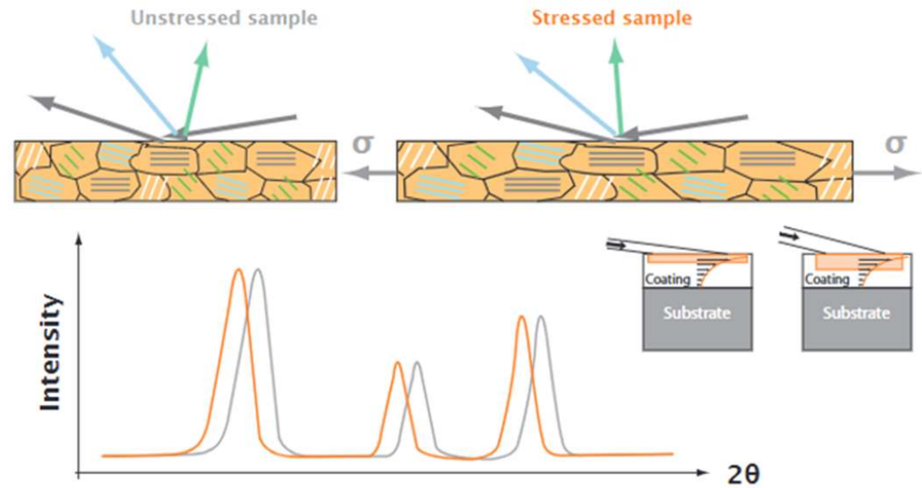
Introduction to Grazing Incidence

Multi-{hkl} Residual Stress

Classic Residual Stress

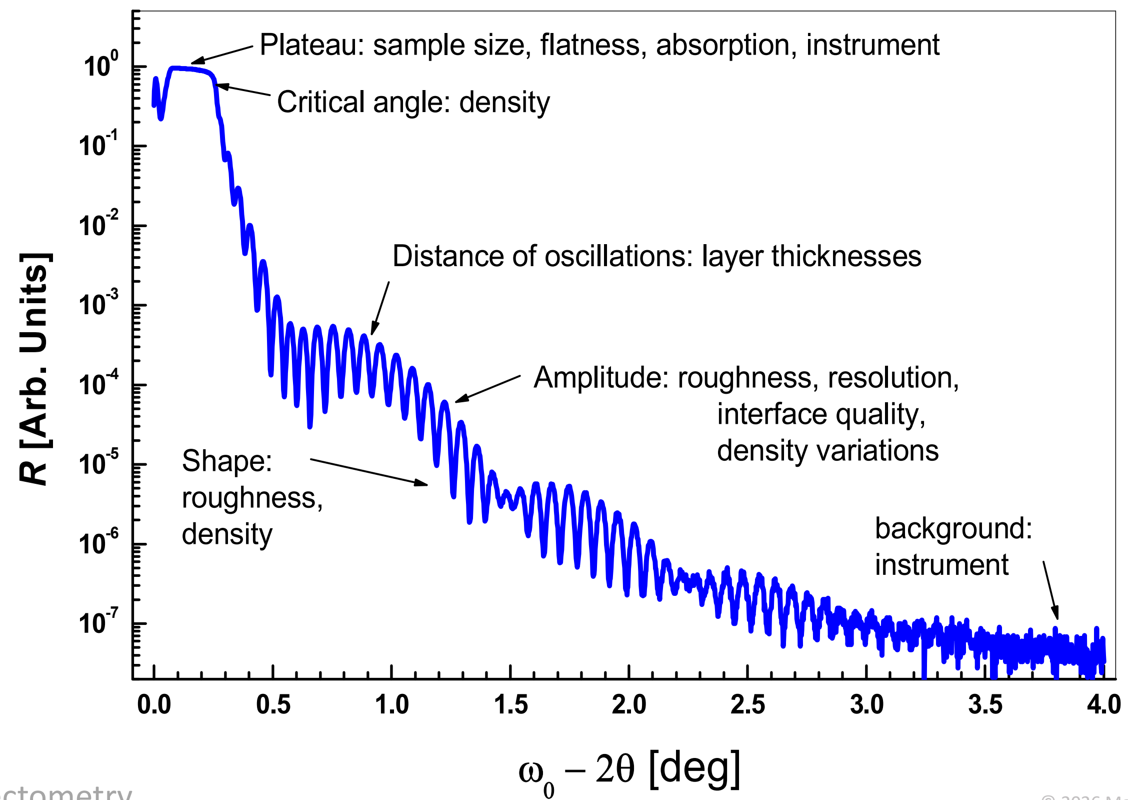


GI Stress



Introduction to X-Ray Reflectivity

Overview of Observed Data



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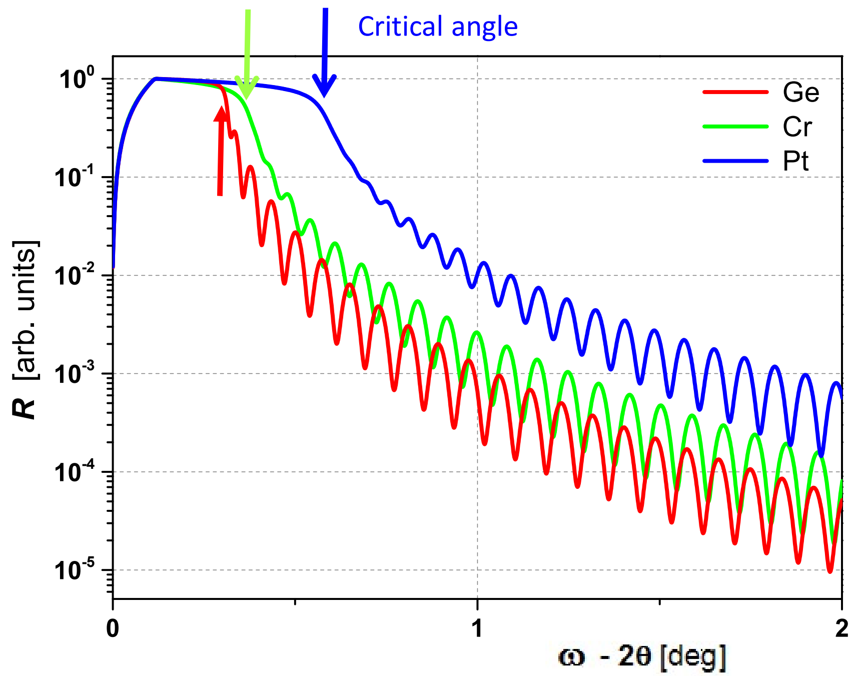
Introduction to X-Ray Reflectivity

Effect of Layer Density

The reflectance of 50 nm single layer on a Si substrate:



Critical angle

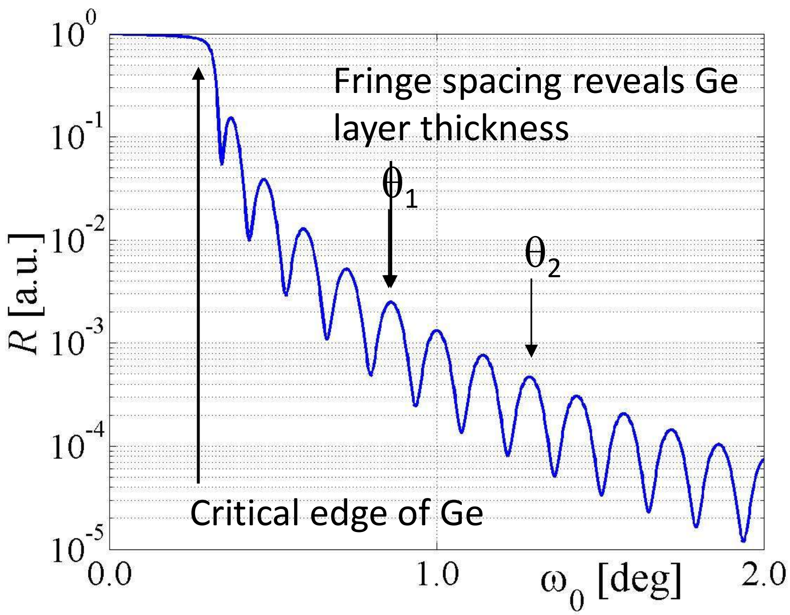
Density

$$\omega_C \sim \sqrt{\rho}$$

$\omega_0 < \omega_c$, total reflection

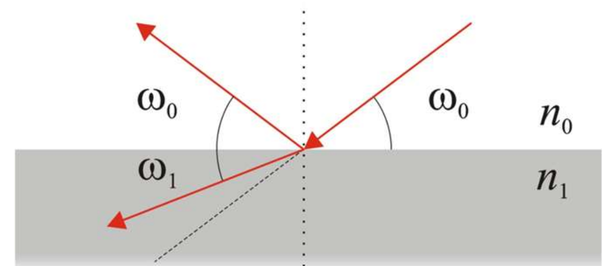
Introduction to X-Ray Reflectivity

Film Thickness

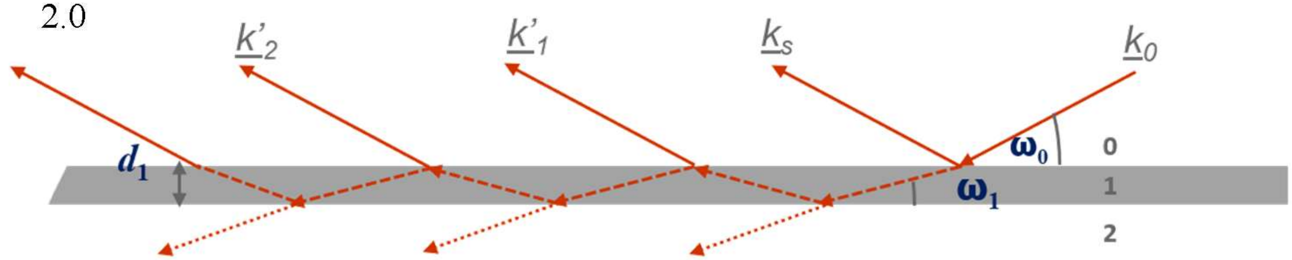


Example: 30 nm Ge layer on Si substrate

$$n_0 \cos\omega_0 = n_1 \cos\omega_1$$

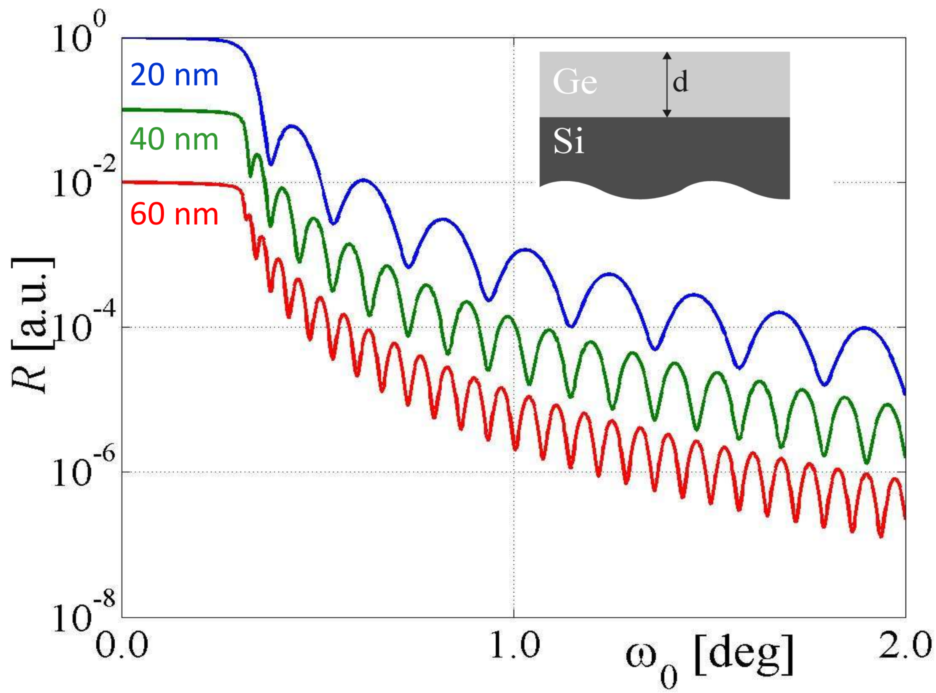


$$d = \frac{M\lambda}{2\left(\sqrt{\theta_1^2 - \omega_C^2} - \sqrt{\theta_2^2 - \omega_C^2}\right)}$$



Introduction to X-Ray Reflectivity

Effect of Layer Thickness



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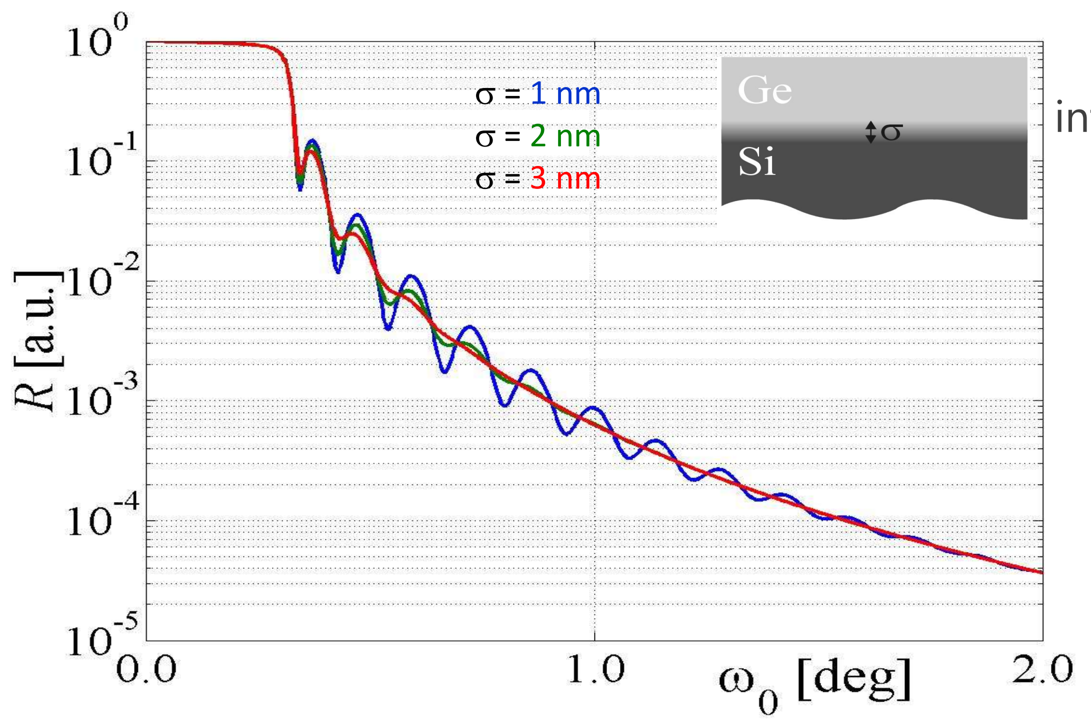
Introduction to X-Ray Reflectivity

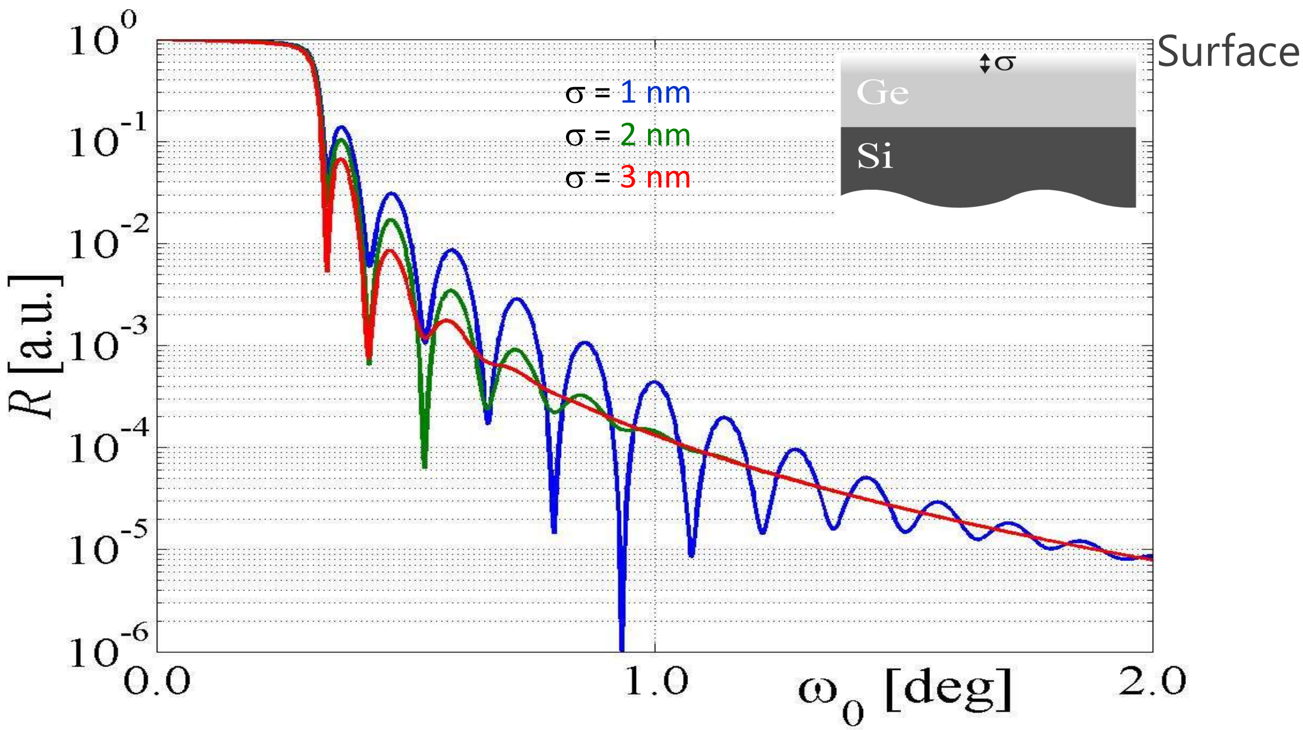
Effect of Layer Roughness



interface

Introduction to X-Ray Reflectivity

Effect of Layer Roughness



Introduction to GI-XRD and XRR

Summary

- Grazing incidence can be used for polycrystalline thin films to obtain a wide range of “standard” powder diffraction information about the surface layer. These include:
  - Phase Identification
  - Crystallite Size and Strain
  - Residual Stress
  - Depth Profiling

- X-Ray reflectivity provides information more specific to the film structure:
  - Density
  - Thickness
  - Surface and Interface Roughness