X-ray Reflectivity Measurements of III-V Superlattices

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X-ray Reflectivity

Difference from X-ray Diffraction
• Low-angle incident beam $\theta < 12^\circ$
• Specular reflection from film surfaces

What can be measured
• Film thicknesses
  • Thickness gradient in SL
• Film Refractive index
  • Ordering of layers
• Roughness
  • Lateral correlation of roughness
  • Cross-plane roughness correlation
Reflectivity of substrate

1. Incident angle < Total reflection critical angle
   All incident X-rays are reflected.

2. Incident angle = Total reflection critical angle
   Incident X-rays propagate along the sample surface.

3. Incident angle > Total reflection critical angle
   Incident X-rays penetrate into the material by refraction.

Fig. 2. Reflectivity curve of Si.
Interference pattern emerges between substrate and film reflections

Critical angle dependent on film thickness!!

Fig. 4. Reflectivity of Au film on Si substrate.

Film

Substrate

Yosaka 2010
Electron density is our contrast mechanism

- Refractive index is determined by electron density

- Peak height diminishes as film density approaches the substrate density $\rho_{\text{Si}} = 2.33 \text{ g/cm}^3$

- Critical angle also changes with surface film density

Fig. 5. X-ray reflectivity curves of Au, Cu and SiO$_2$ film on Si substrates (film thickness is 20 nm). Yosaka 2010
Surface and interface roughness decrease specular intensity and hide interference fringe patterns.

**Fig. 6.** X-ray reflectivity curves of Si substrates with two different values of surface roughness.

**Fig. 7.** X-ray reflectivity of Si substrate differences with interface roughness (Film thickness is 20 nm).
Fourier method for determining layer thicknesses

\[
\cos\left(\frac{4\pi d}{\sqrt{\sin^2(\theta - 2\delta)}}\right) \quad d = \text{layer thickness} \\
\delta = \text{refractive index}
\]

Yosaka 2010

**Fig. 12.** X-ray reflectivity curve of monolayer film (left) and the corresponding extracted oscillation curve (light).

**Fig. 13.** Profile after Fourier transformation.

Fourier Transform
Determine lateral roughness correlation from Yoneda wings

Types of roughness models: specular, fractal, staircase, and castellation
Real data fitting example: strain-balanced InAlAs/InGaAs superlattice

Vanishes for identical layer thicknesses and roughness
Layer thickness gradient causes peak broadening at high angles
1Å Surface oxide features visible at low angles

- Data
- With Surface Oxide
- No Surface Oxide

counts/s

$\theta$ (degrees)
Roughness greatly reduces peak intensity at high angles
Anomalous high-angle peaks in scan!
Superlattice diffraction peaks clearly extend into the reflectivity scan range.
What can be measured

Film thicknesses
- Thickness gradient in SL

Film Refractive index
- Ordering of layers

Roughness
- Lateral correlation of roughness
- Cross-plane roughness correlation

Reference for a good reflectivity introduction: