Thin Film Analysis

X-ray Reflectometry

Joachim. F Woitok
Almelo, The Netherlands
Outline

• Basic principles
• Information content
• Experimental set-ups
• The role of roughness
• Diffuse scattering
Film Properties of Interest:

• geometry (thickness, …)
• topography (roughness, sub-micro structures)
• chemistry (elements, composition, bonds)
• crystallography (structure, texture, defects)
• mechanical prop. (stress, hardness, density)
• optical prop. (colour, refr. index, scattering)
• electrical prop. (conductivity, permittivity, …)
Basic Principle of X-ray Reflectivity

refractive index

\[ n = 1 - \delta + i\beta \]

\( \delta \approx 10^{-6} \)
\( \delta \sim \rho \)

\[ n_1 \cdot \cos \theta_1 = n_2 \cdot \cos \theta_2 \]
Basic Principle of X-ray Reflectivity

The refractive index is given by:

\[ n = 1 - \delta + i\beta \]

where \( \delta \approx 10^{-6} \) and \( \delta \sim \rho \).

For the reflection, we have:

\[ n_1 \cdot \cos \theta_1 = n_2 \cdot \cos \theta_2 \]
Basic Principle of X-ray Reflectivity

refractive index

\[ n = 1 - \delta + i\beta \]

\[ \delta \approx 10^{-6} \]
\[ \delta \sim \rho \]

total external reflection

\[ \cos \theta_C = n_2 \]

\[ \theta_C = \sqrt{2\delta} \]
Basic Principle of X-ray Reflectivity

refractive index

\[ n = 1 - \delta + i\beta \]

\( \delta \approx 10^{-6} \)
\( \delta \sim \rho \)

total external reflection

\[ \cos \theta_C = n_2 \]
\[ \theta_C = \sqrt{2\delta} \]
Information Depth

![Graph showing the relationship between $z_0$ (Å) and $\psi_i/\psi_c$.]
### Numerical Examples

<table>
<thead>
<tr>
<th>Material</th>
<th>Density [g/cm$^3$]</th>
<th>$\delta \cdot 10^6$</th>
<th>$\theta_C$ [$^\circ$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>2.32</td>
<td>7.57</td>
<td>0.223</td>
</tr>
<tr>
<td>SiO$_2$</td>
<td>2.26</td>
<td>7.33</td>
<td>0.219</td>
</tr>
<tr>
<td>GaAs</td>
<td>5.32</td>
<td>14.52</td>
<td>0.309</td>
</tr>
<tr>
<td>Si$<em>{0.88}$Ge$</em>{0.12}$</td>
<td>2.74</td>
<td>8.52</td>
<td>0.236</td>
</tr>
<tr>
<td>Cu</td>
<td>8.96</td>
<td>242.9</td>
<td>0.413</td>
</tr>
</tbody>
</table>
Reflectivity Curve

Reflectivity vs. Omega/2Theta (degrees)

- Si
- SiO₂ (50 Å)
- Cu (50 Å)
**X-ray Reflectometry**

**alternative methods:**
- density: RBS, refractive index (-)
- thickness: optical methods, TEM (-)
- roughness: interferometry, AFM, profilometry (only surfaces)

**reflectometry:**
- density: ± 1-2 %
- thickness: ± 0.5- 1 % (max. about 1000 nm)
- roughness: model dependent (reproducibility 3%)
Information Content

- Plateau: sample size, flatness, absorption, instrument
- Critical angle: density
- Distance of oscillations: layer thicknesses
- Amplitude: roughness, resolution, interface quality, density variations
- Shape: roughness, density
- Background: instrument
Thickness Oscillations (Single Layer)

\[ \theta^2 = \theta_c^2 + \frac{\lambda^2}{4d^2} \nu^2 \]
Thickness Analysis

X’Pert Reflectivity 1.1

Fourier Transform

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Typical Low Resolution Experimental Set-up
Other Possible Set-ups

1. **Attenuator**
   - X-ray tube (line focus)
   - Soller slits
   - Beam mask
   - Divergence slit
   - Thin layers

2. **Soller slits**
   - X-ray mirror
   - Attenuator
   - Thin layers

3. **Parallel plate collimator**
   - Slit 0.1 mm
   - Flat crystal Monochromator (Graphite)
   - Detector

Δω = 0.05°
Δω = 0.04°
Choice of Set-ups for X-ray Reflectivity

- Thickness up to 100 nm (fringe separation 0.04°):
  - divergence slit / X-ray mirror
    Kα doublet ($\Delta \lambda / \lambda$: 2.5 $\times 10^{-3}$)
  - receiving slit / parallel plate collimator

- Thickness more than 100 nm (<1000nm):
  - primary monochromator ($\Delta \lambda / \lambda$: 3 $\times 10^{-4}$)
  - analyzer crystal / secondary mirror
Sample Illumination

Illuminated length (mm) vs. Incident angle

- Beam width 1.2 mm
- Beam width 0.2 mm
Sample Alignment

- samples should be **LARGE** and **FLAT**
- sample should bisect incident beam when $\omega=0$ and $2\theta=0$
- adjust $\psi$ to give minimum peak widths
Sample Alignment

Alignment should **always** be carried out whilst the sample is reflecting.
XRR on Undulated Samples

Rocking curves used for alignment

Curved sample

Irregular sample

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Alignment (2)

Problem:
Curved/undulating/irregular surfaces cause rocking curve broadening, deformation - preventing accurate adjustment
Alignment (3)

Solution:
Add a beam knife
The Beam Knife
Example Non-ideal Surface

Ta$_2$O$_5$ on SiO$_2$
High Resolution Set-up – Thick Layers
Thick layers: Effect of Optics

- PDS - PASS/PRS
- Hybrid 2-bounce - mirror
- Hybrid 4-bounce - TA
- Mirror+(220)a - TA
Thick Layer – The ‘Limit’
Data Evaluation XRR

- Measured data → Min($X^2$) → Full pattern simulation
- density, thickness, roughness

Automatic fitting

substrate

layer #1
layer #2
...
layer #N
Model Based on X-ray Diffraction
Best Fit Simulation

counts/s
100M
10M
1M
100K
10K
1K
100
10
1
0.1
0 1 2 3 4 5 6
Omega/2Theta (°)

Si cap
SiGe
SiGe
Si substrate
Effect of Roughness

Reflectivity vs. Omega/2Theta (degrees)

Cu (100 Å)
Si

σS
σL
0 0
5 0
5 5
5 10 [Å]

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Roughness – Density Gradient

- gradient erf
- roughness
**Diffuse Scattering**

- X-ray scattering which is not specular
- Information about scale of roughness parallel to interface (lateral correlation)
- Particularly useful for multilayers (vertical correlation)
- Use $\omega$ scans at fixed $2\theta$ or offset $\omega/2\theta$ scans or $2\theta$ at fixed $\omega$
- Or use 2-D scans
The Fractal Interface Model

Suitable for molecular films:
Surface Roughness Parameters

\[ \sigma = \text{(RMS) roughness} \quad h = \text{Hurst parameter} \]
\[ L = \text{correlation length} \quad D = 3 - h = \text{Fractal dimension} \]

h=0.9  
D=2.1

h=0.5  
D=2.5

h=0.3  
D=2.7
Suitable for an off-cut semiconductor:

\[ \frac{d}{l} = \sin \gamma \]
The Castellated Interface Model

Suitable for grown/deposited semiconductors/metals:

\[ l_2 \quad d \quad l_1 \]
SiO$_2$(185Å)/Si
Effect of Roughness Parameter

Cu (100Å)

Si

σs  σL  L
0   0   10000
5   0   10000
5   5   10000
5   10  1000
5   10  1000

Å
Other Dynamical Effects

![Graph showing dynamical effects with log intensity on the y-axis and Omega (degrees) on the x-axis. The graph compares Cu (500 Å) and Si samples with specified parameters: σ: 15 Å, L: 1000 Å, and 2 Å.](image)
Measured

- **SiO₂**
- **CrO₂**
- **Si substrate**
Reflectivity and Diffuse Scattering

Counts/s

Omega (°)

100M
10M
1M
100K
10K
1K
100
10
1
0.1

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Roughness Parameters

L: 45±10nm, h:0.5
Choosing the Appropriate Model

... Metallic multilayer using the fractal model:

\[ \xi = 300 \text{ nm}, \ h = 0.2, \text{ and } \sigma = 1.5 \text{ nm} \]

\[ 2\theta = 1.79^\circ \]

\[ 2\theta = 2.34^\circ \]
... Metallic multilayer using the castellated model:

\[ l_1 = l_2 = 20 \text{ nm}, \ d = 2.0 \text{ nm} \ (\approx \sigma) \]

Distributions of heights and widths
Diffuse X-ray Scattering

TiN(330Å)/Ti(235Å)/SiO2(950Å)/Si
Roughness Models Periodic Multilayers

- no vertical replication
- roughening
- smoothing
- identical replication
Diffuse Scattering From Periodic Multilayers

2θ - ω < θc

ω < θc

regions of resonant diffuse scatter
superlattice reflections

incident beam from right

2θ < ω
ω < 0

Qy
Qx
Reflectivity Curve Multilayer

Mirror (1/16)  
50x[Si/Mo] on Si

Period: 6.6 nm
Multilayer Structure
Simulation
Conclusion

Reflectivity for reliable and fast determination of layer thicknesses and densities

Non-destructively, no limitation to sample crystallinity

Detection of very thin layers (down to 1 nm)

Sample size should be larger than 1x1cm²

Long measurement time (0.5-15h)

Complex evaluation, multi parameter fit