X-ray Reflectometry

- **Determination of layer properties such as thickness, density, roughness independently of each other**

  - Liquid, amorphous, poly-crystalline, epitaxial or any combination, prepared on glass, metals, semiconductors or fluids

- **Non-destructive and non-contact method for thickness determination between 2-200nm with a precision of about 0.1 nm**

The method uses a natural property of every matter: the refraction index for X-rays is smaller than unity. This means that the effect of total external reflection is observed for X-rays. This effect is similar to the effect of total reflection, which is known from visible light (e.g. a diver tries to watch the beach from below the water line). The difference is that for X-rays this effect is only observed at very low incident angles, below 1°.
Transmission and reflection

At the boundary of the medium, part of the light will be reflected at angle $\theta_r$ while the other part will be transmitted through the sample at angle $\theta_t$. Snell's law requires that all three beams be in the plane of incidence. The plane of incidence is defined as that plane which contains the input beam, the output beam, and the direction normal to the sample surface.

T and R are defined as the ratio of the light intensity being transmitted or reflected over the incident light intensity on the sample.
x-ray reflectometry

reflection at a ideally flat surface

\[ n_1 \cos \Theta_1 = n_2 \cos \Theta_2 \Rightarrow n_1 \cos \Theta_C = n_2 \]

thin films

\[ d = \frac{\lambda}{2\Delta\theta} \]

interference
The complex refractive index in the x-ray region is slightly less than 1 and is given by

\[ \tilde{n} = 1 - \delta + i\beta \]

\[ \text{dispersion} \quad \text{absorption} \]

For frequencies far greater than the resonance frequencies, \( \nu_0 \), of the atom \( \delta \) can be given by the expression

\[ \delta = \frac{e^2 n_e}{2\varepsilon_0 m (2\pi c)^2} \lambda^2 = \frac{r_0 \lambda^2}{2\pi} \cdot n_e \]

\[ n_e = Z \cdot n_{\text{atom}} \]

\[ n_{\text{atom}} = \frac{N_A}{A} \cdot \rho \]

\[ \delta = \frac{e^2 n_e}{2\varepsilon_0 m (2\pi c)^2} \lambda^2 = \frac{r_0 \lambda^2}{2\pi} \cdot Z \frac{N_A}{A} \cdot \rho \]
We consider reflection at an interface between air, $n_{\text{air}} = 1$ and another material, $n_1 = 1 - \delta$. For incident angles below a critical angle, $\theta_c$, ($\theta < \theta_c$), total reflection occurs. By applying Snell’s law and small angle approximations, the critical angle, $\theta_c$ can be expressed as

$$1 - \delta = \cos \theta_c \approx 1 - \frac{\theta_c^2}{2}$$

$$\Leftrightarrow \theta_c \approx \sqrt{2\delta} = \sqrt{\frac{r_0 \lambda^2}{\pi} N_A \frac{(Z + f')}{A}} \cdot \rho$$
Film density

\[ \theta_c \approx \sqrt{2\delta} = \frac{r_0 \lambda^2}{\pi} N_A \frac{(Z + f')}{A} \cdot \rho \]

Abrupt fall in the reflected intensity
Film thickness

$\theta > \theta_c$

The X-ray beam penetrates inside the film

Reflection therefore occurs at the top and the bottom surfaces of the film. The interference between the rays reflected from the top and the bottom of the film surfaces results in interference fringes which do not depend on the frequency like in the case of optical spectroscopy but are angle dependent.
x-ray reflectometry

Increasing surface roughness leads to a faster decay of reflectivity.
x-ray reflectometry

setup & substrates

setup
• fixed x-ray source (Cu K$_\alpha$)
• $\Theta$-2$\Theta$ geometry (sample/detector)
• monochromator (graphite)
• detector: NaI scintillation counter
• knife edge to align sample

substrates
• silicon wafers (best)
• glass, especially float glass
• polymers (if flat enough)
**x-ray reflectometry**

![Graph showing data analysis](image)

**Data analysis:**
- A model is used to simulate the measured curve on the basis of the Fresnel equations.
- Parameter: electron densities, roughnesses of interfaces, thickness.
x-ray reflectometry

summary

advantages

• direct measurement of geometric thickness (no refractive index needed)
• roughness data over large area
• relatively easy to align
• well-suited for standard substrates (silicon wafers, glass)

disadvantages

• long measurement times (hours)
• theoretical modelling of curve needs experience (many parameters that influence each other)
• cannot be combined with „cells“ (e.g. for solutions)
**ellipsometry**

**principle**

- s- and p-light is reflected differently
- phase: determined by $\Delta$
- amplitude: determined by $\Psi$

$n_3 < n_2 < n_1$
Polarized light is reflected at an oblique angle to a surface
The change to or from a generally elliptical polarization is measured.
From these measurements, the complex index of refraction and/or the thickness of the material can be obtained.
Polarized light

If the electric field vectors describing the propagation of electromagnetic waves are all oriented in one plane, the light is referred to as polarized or, more completely, linearly polarized light.

Of primary interest in ellipsometry is the fact that when linearly polarized light makes a reflection on a surface, there is a shift of the phases of both the components (parallel and perpendicular to the plane of incidence). The shift is, in general, not the same for both, hence an elliptically polarized wave results.

a) Two linearly polarized beams, in phase, add to another linearly polarized beam. b) Two linearly polarized beams, out of phase by 90°; the resultant beam is circularly polarized. If the phase difference is other than 90°, light is elliptically polarized.
Theory

Reflection of light on a surface

Plane polarized waves that are in the plane of incidence are referred as p-waves and plane polarized waves perpendicular to the plane of incidence as s-waves.

The **Fresnel** reflection coefficient $r$ is the ratio of the amplitude of the reflected wave to the amplitude of the incident wave for a single interface. The Fresnel reflection coefficients are different for p and s waves:

\[
\begin{align*}
    r_{12}^p &= \frac{\tilde{N}_2 \cos \phi_1 - \tilde{N}_1 \cos \phi_2}{\tilde{N}_2 \cos \phi_1 + \tilde{N}_1 \cos \phi_2} \\
    r_{12}^s &= \frac{\tilde{N}_1 \cos \phi_1 - \tilde{N}_2 \cos \phi_2}{\tilde{N}_1 \cos \phi_1 + \tilde{N}_2 \cos \phi_2}
\end{align*}
\]
The complex index of refraction

When light passes through a medium, it interacts with it; it is slowed-down and absorbed by matter

\[ \tilde{N} = n - ik \]

where \( n \) is the index of refraction, \( k \) is called the “extinction coefficient”, \( k \) is defined through the absorption coefficient \( \alpha \). In an absorbing medium:

\[ I(z) = I_0 e^{-\alpha z} \quad \text{and} \quad k = \frac{\lambda}{4\pi} \alpha \]

Determines how fast the amplitude of the wave decreases

For dielectric material \( k=0 \) (no of the light is absorbed)
Theory

\[
N_1 \cos \phi_1 + N_2 \cos \phi_2
\]

\[
\frac{N_2 \cos \phi_1 - N_1 \cos \phi_2}{N_2 \cos \phi_1 + N_1 \cos \phi_2}
\]

\[
\frac{N_1 \cos \phi_1 - N_2 \cos \phi_2}{N_1 \cos \phi_1 + N_2 \cos \phi_2}
\]

\[
R^p = |r^p|^2, R^s = |r^s|^2
\]

If both media are dielectric in nature (k=0):

\[
r_{12}^p = \frac{n_2 - 1}{n_2 + 1}, \quad r_{12}^s = \frac{1 - n_2}{1 + n_2}, \quad R^p = R^s = \left(\frac{n_2 - 1}{n_2 + 1}\right)^2
\]

\[
tan \phi_1 = \frac{n_2}{n_1}, \text{ Brewster angle}
\]

\[
n_1 = 1, n_2 = 2, \Rightarrow 63.43^\circ
\]
Reflection and transmission with multiple interfaces

The resultant reflected wave returning to medium 1 is made up of the light reflected directly from the first interface plus all of the transmissions from the light approaching the first interface from medium 2.

The addition of the infinite series of partial waves leads to the resultant and has been derived by Azzam\textsuperscript{1}. The ratio of the amplitude of the resultant reflected wave to the amplitude of the incident wave is given by:

Total reflection coefficient

\[
R^p = \frac{r_{12}^p + r_{23}^p \exp(-i2\beta)}{1 + r_{12}^p r_{23}^p \exp(-i2\beta)} \quad R^s = \frac{r_{12}^s + r_{23}^s \exp(-i2\beta)}{1 + r_{12}^s r_{23}^s \exp(-i2\beta)}
\]

\[
\beta = 2\pi \left( \frac{d}{\lambda} \right) \tilde{N}_2 \cos \phi_2
\]

Film phase thickness

Ellipsometry definitions

Let us define $\delta_1$ as the phase difference between the parallel and perpendicular components of the incident wave. Also, let us define $\delta_2$ as the phase difference between the same components but for the outgoing wave. Delta, $\Delta$, is then defined as:

$$\Delta = \delta_1 - \delta_2$$

Delta (Del) is the change in phase difference between parallel and perpendicular components of the incident wave that occurs upon reflection.

Without regards to phase, the amplitude of both components of the beam may change upon reflection.

$$\tan \Psi = \frac{|R^p|}{|R^s|}$$

$\Psi$ is then the angle whose tangent is the ratio of the magnitudes of the total reflection coefficients.

These definitions lead us to the fundamental equation of ellipsometry.

$$\tan \Psi \exp(i\Delta) = \frac{R^p}{R^s}$$

Now, in principle, given $\tilde{N}_1$, $\tilde{N}_3$, $\phi_1$ (angle of incidence) and $\lambda$, and if $\Delta$ and $\Psi$ are measured, one could calculate $d$, the thickness of a film and $\tilde{N}_3$, its index of refraction. These are tedious calculations, but are easily done by a computer.
Null ellipsometry

We choose our polarizer orientation such that the relative phase shift from Reflection is just cancelled by the phase shift from the retarder.

The angular setting of the QWP and the analyzer could be used to determine the phase shift and attenuation ration. This is conceptual use of these elements. Actual practice is somewhat different.

We know that the relative phase shifts have cancelled if we can null the signal with the analyzer.
advantages

• simple setup (easy to understand & align)
• swollen layer thicknesses can be measured (there is almost no other way to do this)
• segment density profile can be derived
• model-free data analysis by FT

disadvantage

• Fitting procedure